Krishna K. Pandey · V. Ramakantha
Shakti S. Chauhan · A.N. Arun Kumar
Editors

Wood is Good

Current Trends and Future Prospects in Wood Utilization

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Preface

Wood, with its unparalleled versatility, is a fabulous gift of nature. Having a wide range of applications, it has played an important role in the progress of human civilization. Greater technological innovations may further render wood as the single most important natural resource in the times to come. In the current scenario of global climate change, unlike any other non-renewable material, wood can be sustainably produced, processed, and converted into a range of products with the least of carbon footprints. Wood is Good.

Wood is a highly variable and complex material. The inherent variability in wood between species, within a species, and also within a tree poses challenges in its processing and utilization. Short-rotation plantation-grown timber has been further added to this challenge. In this backdrop, the Institute of Wood Science and Technology (IWST), Bengaluru, India, organized an international conference on "Wood is Good: Current Trends and Future Prospects in Wood Utilization" on November 21–23, 2014.

The Institute of Wood Science and Technology is a premier research institute under the aegis of Indian Council of Forestry Research and Education (ICFRE) of the Ministry of Environment, Forests and Climate Change, Government of India. With a specialized team of scientists, the institute has been carrying out frontier research in wood identification, processing, wood composites, wood modification, wood energy, wood quality, and tree improvement. The conference provided a platform to academicians, researchers, and industry professionals across the globe to present and discuss the recent innovations, trends, and future prospects. More than 100 research papers covering a wide range of topics were deliberated in the conference.

This book is a collection of selected papers presented during the conference. The papers are grouped in five major themes, namely wood properties and variability, wood protection, wood-based composites, wood utilization pattern, and wood and climate change.

We are thankful to the Ministry of Environment, Forests and Climate Change, Government of India, for the support rendered. We are grateful to the Director General, ICFRE, for his guidance and encouragement. We express our sincere

gratitude to all our colleagues at IWST for their immense help. We duly acknowledge the contribution of all the authors and reviewers.

Bengaluru, India

Krishna K. Pandey
V. Ramakantha
Shakti S. Chauhan
A.N. Arun Kumar

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About the Editors

Dr. Krishna K. Pandey is a senior scientist at the Institute of Wood Science and Technology, Bengaluru, India, having more than 30 years of research experience. He was postdoctoral researcher at Kyoto Institute of Technology, Japan; and senior researcher at Helsinki University of Technology, Finland. Dr. Pandey has published more than 50 papers in peer-reviewed journals. He is an elected fellow of International Academy of Wood Science and a recipient of National Award for Excellence in Forestry Research by the Indian Council of Forestry Research and Education, Government of India. Presently, he is a member of Environmental Effects Assessment Panel of UNEP.

Dr. V. Ramakantha was the Director of the Institute of Wood Science and Technology, Government of India, and he retired as Principal Chief Conservator of Forests, Manipur, India. He is well known in the fields of Forestry Research and Education, Wildlife Conservation, and *Ex-situ* Conservation of flora and fauna. As a prolific writer, he is an author for two books and has published several articles in peer-reviewed journals and magazines. He was the chairman of the Committee on National Strategy and Working Scheme for the Management of Red Sanders (*Pterocarpus santalinus*). Dr. Ramakantha is a fellow of the Society for Applied Biotechnology and a Guest Faculty in several reputed institutions in India. He has received international recognition in the field of wildlife photography.

Dr. Shakti S. Chauhan is a senior scientist at the Institute of Wood Science and Technology, Bengaluru, India, with more than 20 years of research experience. He was a Ph.D. scholar, postdoctoral researcher, and FAO fellow at University of Canterbury, Christchurch, New Zealand. Dr. Chauhan is recognised internationally for his work on wood quality assessment and wood polymer composites. He has published more than 50 papers in peer-reviewed journals and co-authored two books on wood science. He is a reviewer for several international journals in the field of wood and material science. He is a member, Committee for Timber Storage, Bureau of Indian Standards. Dr. Chauhan received Appreciation Award for his contribution in the field of wood technology, conferred by the Honourable Governor of Karnataka.

Dr. A.N. Arun Kumar is a senior scientist of the Institute of Wood Science and Technology, Bengaluru, India. Having research experience of more than two decades, he has published several articles in peer-reviewed international journals and has also been a reviewer for many high-impact journals on forestry. Dr. Arun has carried out pioneering research work on heartwood and oil variation in the Indian Sandalwood (*Santalum album*), the most valued tree species of India. His extensive research on growth and wood quality variability of several commercially important tree species such as *Pterocarpus santalinus, Chloroxylon swietenia, Hardwickia binata and Melia dubia* is a significant contribution in the field of tree improvement.

Part I
Wood Properties and Variability

Optimizing Wood Utilization Based on Whole Tree Inherent Property Maps

Mathew Leitch and Scott Miller

Abstract The forest industry is in a state of change currently in Canada. The past strengths of the industry have been a steady supply of high-quality lumber and paper products for over a century. More recently, as we move to second-growth forests and plantations, we are recognizing that wood properties have changed and are extremely variable across the landscape. To this end, the government of Ontario created a Forest Resource Inventory to better deal with the resource and supply a map of the resource to be used by industry in their management planning practices. This has lead government and industry to realize the inventory of basic parameters such as species composition and general heights which is not adequate in today's global economy and markets. Competition from other regions of the world has forced the industry in Canada to look at how we do business and how we can remain competitive in global markets with fast-growing southern hemisphere plantations of pines and eucalypts. In order to better utilize every stick of wood we cut, a better knowledge of the inherent wood properties for trees and whole landscapes is required. In addition, a method of collecting this information efficiently and cost-effectively is also required where data are collected and stored without losses or errors being introduced to the database system. This paper presents a method of both collecting the data accurately while bringing human error to a minimum and nondestructively lessening the cost of collecting the data. This method represents a new way of looking at inventory where inherent properties dictate the inventory and how we assess the forest across a landscape. We have shown that we can produce landscape property maps using a newly developed Wood Science App that both collects data in the field and controls it but also is linked to the information database in our laboratory eliminating the need for people to enter data. In addition, the App controls the testing equipment in the laboratories,

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which removes another level of potential error in the database system. The whole mapping system developed in the Lakehead University Wood Science & Testing Facility (LUWSTF) is designed to allow a better system both to test wood properties in the laboratories and also to collect field information in a nondestructive manner to map wood properties across the landscape. This will not only assist current industries in managing their forest resources but also act as a promotional tool for attracting new investment into the region.

Keywords 3D mapping · Eastern larch · Nondestructive testing · Wood properties · Wood utilization

Introduction

Where We Are Now

In 2013, the Canadian forest industry contributed $19.8 billion to Canada's GDP and directly employed 216, 500 people (Natural Resources Canada 2014). The forest industry in Canada and many parts of the world has been struggling for the last several years due to the global down turn that occurred in around 2004–2008 (Ontario Ministry of Natural Resources 2012). The forest sector in Canada was particularly hard hit due to the sudden down turn in the US housing market, where the USA also represents roughly 80% of the export market for Canadian forest exports (Industry Canada 2009). It was clearly realized in Canada that we needed to look at the industry as a whole and begin making some changes. One of the changes seen as a means of protecting the industry from such a severe financial hit is to make the industry more competitive and expand to diversify the industry so we are not so dependent on commodity products such as sawn lumber and pulp. This is particularly important when you look at the changes in the operating surplus of the wood products industry sector in Ontario that was at $1.9 billion in 2004 and at $808 million by 2008 (Ontario Ministry of Natural resources 2012). During this same period, investment into the industry dropped from $1.2 billion to $857 million (Ontario Ministry of Natural resources 2012). In order to move from a commodity-based industry in Northern Ontario where lumber and pulp and paper are the mainstays to a more diversified industry including secondary processing such as value-adding industries and industries working in the bio-economy, more detailed information about the resource is required. The value-added sector in Ontario was worth $12.2 billion in 2004 and $9.1 billion in 2008 (Ontario Ministry of Natural resources 2012). Much of this capacity is located in the Southern reaches of the province while much of the Northern part of the province is commodity-based.

The current industry relies on the provincial Forest Resource Inventory to manage the parcel of land (forest tenure license) to supply their mills. This inventory is now seen as insufficient and inaccurate to allow industry to maximize its efficiency and competitiveness. This is particularly important as Ontario alone has 72 million hectares of forestland, which makes up 16% of Canada's forest (Ontario Ministry of Natural Resources (OMNR) 2009). Many studies have looked at the competitiveness of our lumber industries (Nautiyal and Singh 1985; Constantino and Townsend 1986); however, no one has looked at increasing competitiveness through improving the inventory to increase efficiency and therefore competitiveness. The Ontario Wood Supply Strategy has identified "Quality of Wood Supply Information" as one of two issues that need to be addressed, with improving the forest inventory and increasing utilization of available wood as two means of fixing this issue (OMNR 2004). This report also encourages integration between mills, which is also an idea that lends itself to the research presented here where collaboration between mills combined with better information about the resource will increase productivity, efficiency, profits, and competitiveness.

Where We Need to Be

In order to improve our knowledge of the inventory, the LUWSTF at Lakehead University began a whole-tree-mapping program in 2007 where initially under-valued commercial tree species were studied and their inherent properties mapped on the ecosites across the landscape where they are present (Miller et al. 2013). This program led to an expansion where we included the main commercially harvested trees that were more relevant to industry. Once we began this process, we realized many inefficiencies in the whole resource inventory system that needed attention. In order to complete our intensive tree and landscape property maps, we needed to address issues such as field data collections, data transfer, data storage and data retrieval, essentially data management optimization (DMO). This issue lead to a new project to develop a Wood Science App to collect and control data both in the field and in the laboratory. This then led to the requirement of a data management system in the laboratories that worked with the field system. Finally, a system was required to allow using the data and sharing it easily across networks.

The remainder of this paper will discuss mapping tree properties; evidence of the value in mapping tree properties; Wood Science App and data management system; data sharing and reporting; and landscape wood property maps.

How We Get There

The goal of this research was to create a database that stored all pertinent information required to maximize the utilization of species across the landscape and to

ensure the best and highest value product could be produced. In addition, the goal was to create landscape maps to identify where a species grew across the landscape and how its properties varied based on site conditions (i.e., ecosites). This led to an issue in the industry and research field, which was how to deal with large amounts of data. This is a particularly difficult issue as most field work involves field notes or tablets that eventually have to be re-entered into a database by hand or some sort of transfer of data. This is fine as long as the person inputting the data is taking care to be accurate, but also requires that the data collected in the field are also accurate. This is an issue everywhere where data are collected and then require someone to move it to another storage place, such as a database system. Then of course is how you control the data in a database so it is accessible easily and quickly.

Our approach to solving these issues of property mapping, large amounts of data and how to use our resources was to incorporate all levels into one system where field collections, material processing, testing and then data analysis, and storage are all controlled in a similar manner using one system.

Mapping Tree Properties

Mapping inherent tree properties is an area that is growing in order for countries to better utilize the forest resources they have and to encourage new investment by being able to display not only the inventory from the point of view of species and volumes but also to display the wood quality expected across landscapes. To accomplish this is an incredible undertaking in a country like Canada that is expansive with many different forest types (Natural Resources Canada 2014). The LUWSTF began a whole tree inherent property-mapping program in 2007 to increase the opportunities for under-valued commercial species in Northern Ontario, Canada. This program began as a destructive tree-harvesting program to gain the inherent properties we required to map the species at the tree level and at the landscape level (Miller et al. 2012; Leitch et al. 2012). This required a shift in how we measure tree properties to include defects as well as all parts of the tree (radially and axially) instead of the traditional standards tests that had samples collected in the N, S, E, W azimuth directions at breast height. Figure 1 displays the quadrant sample labeling system where all parts of the stem are tested radially and axially using this quadrant system. This intensive destructive system requires at least 3 large trees are collected from each ecosite across the landscape. Figure 2 displays the level of measurements used in destructive sampling to create whole tree property maps (Miller 2010).

The importance of mapping properties is becoming more important as global competition increases and countries realize that utilizing the forest resources they have should include maximizing value, which can be attained through understanding the resource, its properties and how they can best be utilized in industry.

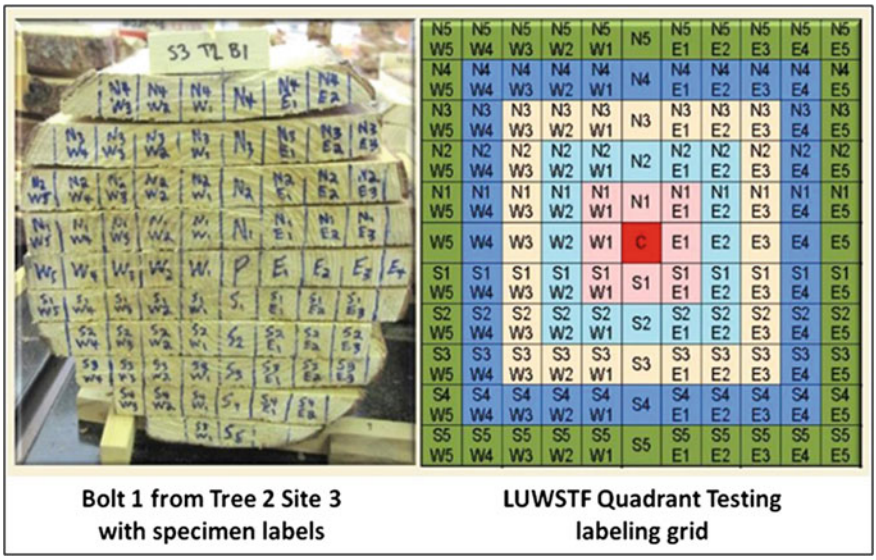


Fig. 1 Quadrant system for labeling sample logs for mapping tree properties developed in the LUWSTF

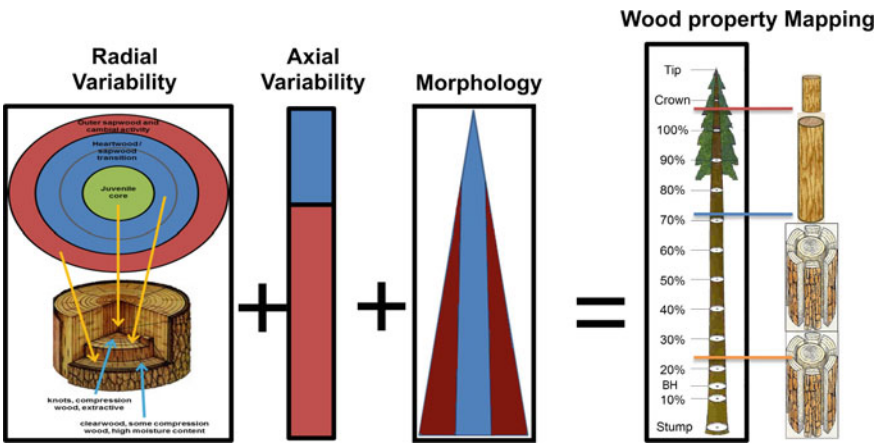


Fig. 2 Tree property maps include radial and axial variability plus morphology to create wood property maps

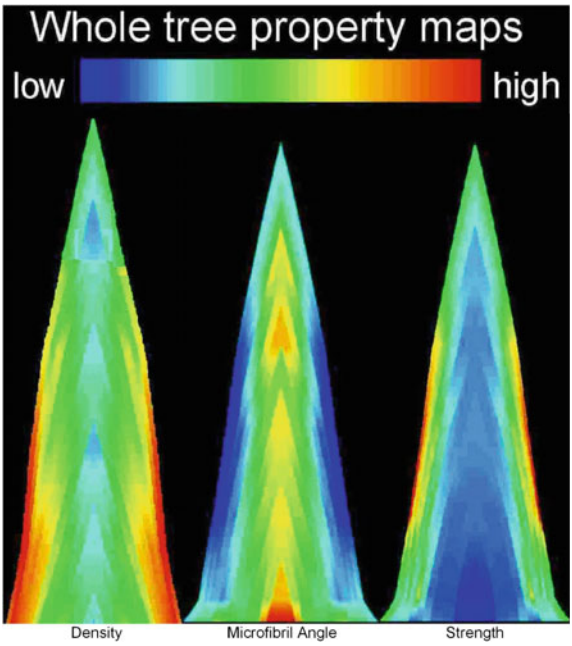


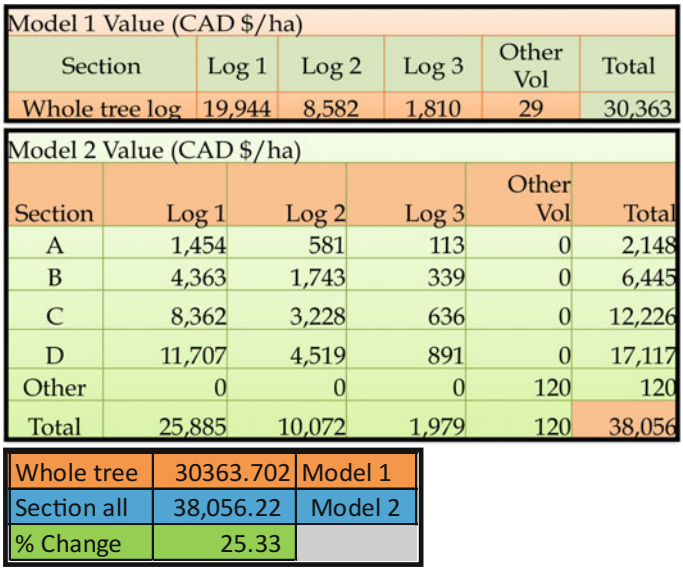
Fig. 3 Property maps of eastern larch for density, microfibril angle, and strength radially and axially in the stem on one of the ecosites larch grows on

Studies looking at aspects of wood properties within trees are present in the literature (i.e., Wilhelsson et al. 2012; Torquato et al. 2014, Cortini et al. 2011; Lessard et al. 2014; Xiang et al. 2014). Many, however, look at individual trees or one location, few look at landscape mapping of tree/wood properties (Lessard et al. 2014).

In the LUWSTF, we began mapping individual trees for each ecosite at the start of this program and have developed new ways to use this information to create landscape property maps. Figure 3 displays some tree maps for a particular property on one of the ecosites where eastern Larch grows in Northwestern Ontario (Miller 2010). These maps for individual trees are the bases for our landscape property-mapping program. Following these studies is also when data control was recognized as an issue and led to our data storage and handling systems.

Evidence of the Value in Mapping Tree Properties

We have shown clearly in an initial study that by recognizing the inherent properties of a tree and sending the appropriate parts of that tree to the right processing facility, the value of every tree cut can be increased by up to as much as 25–30% (Fig. 4; Leitch et al. 2011). On a landscape scale, these can amount to huge



Model 1 Value (CAD $/ha)

Section	Log 1	Log 2	Log 3	Other Vol	Total
Whole tree log	19,944	8,582	1,810	29	30,363

Model 2 Value (CAD $/ha)

Section	Log 1	Log 2	Log 3	Other Vol	Total
A	1,454	581	113	0	2,148
B	4,363	1,743	339	0	6,445
C	8,362	3,228	636	0	12,226
D	11,707	4,519	891	0	17,117
Other	0	0	0	120	120
Total	25,885	10,072	1,979	120	38,056

Whole tree	30363.702	Model 1
Section all	38,056.22	Model 2
% Change	25.33	

Fig. 4 Buck optimization software to display how sending logs to the right mill to optimize value creates an increase in value of the tree over traditional methods of milling

increases in the value of every tree cut and processed; however, it does require mills to integrate and cooperate with respect to resource sharing.

Wood Science App and Data Management

The LUWSTF has developed a data storage and handling system that allows easy transfer of all field data and laboratory-produced data to be transferred and stored easily and efficiently with little opportunity for human error.

LUWSTF and Central Computer Services Inc. (CCS) developed the Wood Science Data Management Application (WS App.), Lakehead University Natural Resource Management Network-Assisted Storage (LUNRM-NAS), Wood Science Intranet (lacie) and Lakehead University Forest Resource Analysis Database (LU-FRAD), to assist in the management and analysis of forest resource inventory data. LUNRM-NAS and lacie provide the infrastructure for data management of LU-FRAD, from data capture and storage to data sharing and collaboration through web-based ftp and cloud functions.

The Forest Resource Analysis Database (FRAD) initiative strives to develop technologies and processes which will assist resource managers, manufactures, communities, and government agencies to identify opportunities to increase the value of the forest resource and forest products.

The WS App. is a database program, which provides the interface for data collection, manipulation, analysis, storage, and reporting. Field collection activities are completed using the WS App. on computer tablets, which are then uploaded to lacie when wood samples are brought to LUWSTF for testing. Lacie then allows the WS App. to import test data directly from test equipment so that the continuity of merging field data with the corresponding destructive testing results is assured. Once data collection has been completed, the WS App. transfers the project database to LU-FRAD for all further activities.

In addition to wood properties mapping of standing timber, the WS App. links forest products testing to forest inventory, thus allowing for cost–benefit analysis of forest products optimization and end-use prediction testing. The functionality of the WS App. and the adaptability of the program were tested in the summer of 2013 in partnership with industry and other agencies. The results of these studies are currently being published.

The NAS/lacie system for storage is a system where by the data collected in the field or laboratories through the WS App. are processed and stored in the NAS. An intranet with WiFi was established as the backbone of the data storage system. Lacie is a central electronic storage devise needed to allow automatic data export from multiple testing machines in the laboratories to the central data storage system (LU-FRAD).

The data in the NAS are then accessible to anyone who is registered and has access to this system. All research partners have a secure folder specific to them on the NAS and can access it anywhere in the world from a computer. The WS App. has been Beta-tested on 6 projects so far, and all test partners have a folder on the NAS to access and move data around. The LU-FRAD system is therefore able to take captured data from the NAS and create 3D models of the forest resource with customized label metrics. Gerema Software, which is embedded into the WS App., is a tool that is used for reporting and query functions. It is able to set modeling attributes like product design criteria for resource optimization and end-use forecasting as well as follow changes in the resource from standing timber through to processing and finished products. The LU-FRAD can also produce landscape maps for land-use planning activities and total resource optimization including abilities to integrate management of both timber and nontimber products and services. An additional ability of the WS App. is that it can upload other laboratories/agencies databases, point cloud data, any comma or tab delimitation data for incorporation into LU-FRAD. The system is also designed to allow expansion into other fields of research. For example, the WS App. can be used to create data for optimization studies and valuation studies using 3D optimization software allowing estimations of standing forest values and lumber volumes per grade; using maps for attracting investment; fiber property maps for pulp and rayon mills; advancing the forest resource inventory; product development and resource monitoring to name a few.

Data Sharing and Reporting

The NAS is a powerful tool for data sharing, and the WS App. and LU-FRAD are powerful tools for dealing with data and creating reports. Sharing data securely is a top priority in research and with partners. The NAS allows partners to be able to go online and access a secure folder in the NAS specific to the partner to either place information in the folder or take information we have put there. Each partner has a specific password to gain access to their folder. This is particularly useful for these types of studies where data files can be very large and greater than email systems allow, the NAS has a 3-terabyte capacity to move data so size is not an issue.

The reporting functions of the App allow for very specific reports to be produced from the entire database or whole file reports. Reports incorporate standard methodologies for reporting, for example, site quality, harvestable timber volume, and wood product grades and are all based on legally defendable statistical methods. An example of a Standard Report could be a Wood Properties Plot Summary as shown in Fig. 5. Query reporting allows a more focused search of the data to suit report needs specifically from a large database (Fig. 6). Tabular export is a reporting function that allows simple access and transfer of raw data (Fig. 7).

In addition to these functions, the WS App. can mine every study within LU-FRAD for data. For example, we can make a query that tells the WS App. to incorporate the following studies or all studies for site attributes or tree height and

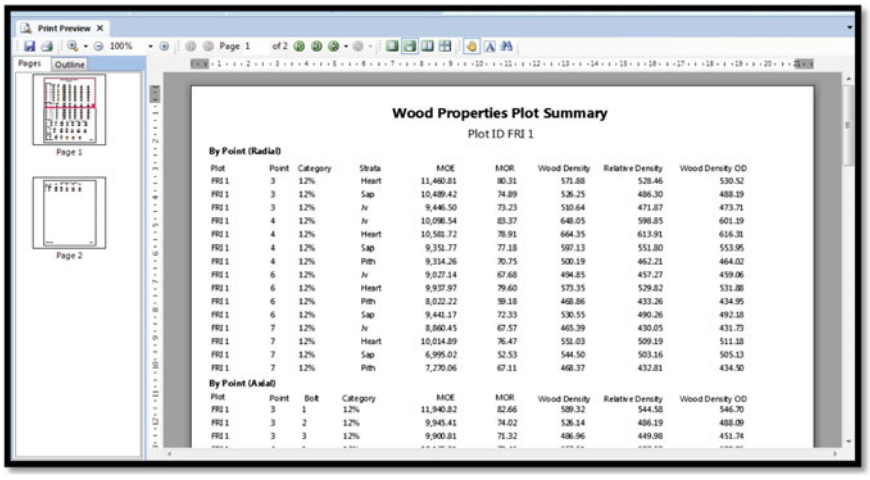


Fig. 5 Example of a standard report produced from LU-FRAD

Query

Fields	Tree	Disk	GrowthRings	X1	X2	Y1	Y2	Z1	Z2
Table	tblTreeStem	tblTreeStem	tblTreeStem	tblTreeStem	tblTreeStem	tblTreeStem	tblTreeStem	tblTreeStem	tblTreeStem
Sort									
Show	☑	☑	☑	☑	☑	☑	☑	☑	☑
Criteria			4						
Or									

Report

Results

Tree	Disk	GrowthRings	X1	X2	Y1	Y2	Z1	Z2
1	4	70	14.1	-14.1	13.4	-13.4	1.3	1.35
27	4	70	10.9	-10.9	11.35	-11.35	1.3	1.35
33	4	68	7.1	-7.1	6.35	-6.35	1.3	1.35
48	4	75	12.75	-12.75	12.45	-12.45	1.3	1.35
58	4	70	13.5	-13.5	13.65	-13.65	1.3	1.35
169	4	75	13.35	-13.35	13.35	-13.35	1.3	1.35
172	4	63	11.15	-11.15	11.15	-11.15	1.3	1.35
200	4	60	6.8	-6.8	6.65	-6.65	1.3	1.35
269	4	69	14	-14	14	-14	1.3	1.35
287	4	68	15.15	-15.15	15.15	-15.15	1.3	1.35
364	4	67	12.35	-12.35	12.2	-12.2	1.3	1.35
401	4	68	11.1	-11.1	11.3	-11.3	1.3	1.35
429	4	70	14.45	-14.45	13.6	-13.6	1.3	1.35
441	4	67	14.05	-14.05	13.35	-13.35	1.3	1.35
448	4	64	6.7	-6.7	6.5	-6.5	1.3	1.35
474	4	68	7.65	-7.65	6.85	-6.85	1.3	1.35
500	4	69	13.75	-13.75	13.75	-13.75	1.3	1.35
511	4	69	13.95	-13.95	13.6	-13.6	1.3	1.35
534	4	68	15	-15	14.6	-14.6	1.3	1.35
537	4	66	12.35	-12.35	11.6	-11.6	1.3	1.35

Rows: 20

Fig. 6 Example of a query and subsequent report produced from LU-FRAD

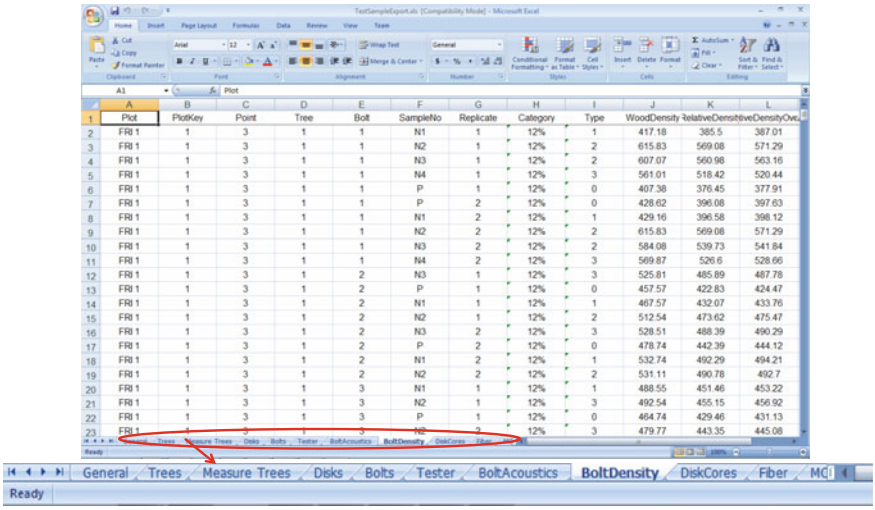


Fig. 7 Example of a tabular export for transfer of raw data from LU-FRAD

diameter and report on the findings by ecosite and/or species, making LU-FRAD a growing mega database over time.

Landscape Wood Property Maps

The final landscape wood property maps display the properties of the trees individually as well as across the landscape. Figure 8 displays a simple tree map with the attributes juvenile and mature wood zones marked on it. This is a 3D map that is fully controllable on a computer where it can be rotated to look at the stem features from any angle. Figure 9 displays a map created in ArcMap that displays the variability of specific gravity in Eastern Larch in Northwestern Ontario. At a stand level, there will be some variation; however, it is very apparent the average specific gravity of the species varies significantly across the landscape. More studies have looked at tree properties (Morrow et al. 2013; Bendtsen and Senft 1986; Duchesne 2006; Mvolo et al. 2015), how to rapidly measure properties (Evans et al. 1995) and

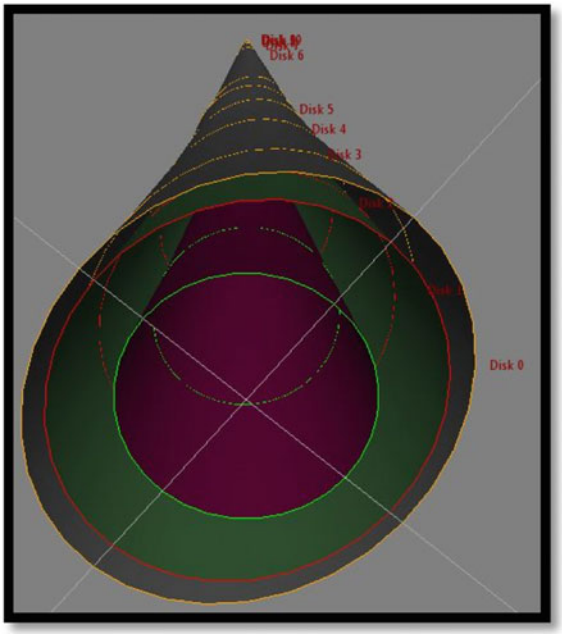


Fig. 8 3D mapping of wood properties showing the juvenile core and the outer mature wood on a measured tree

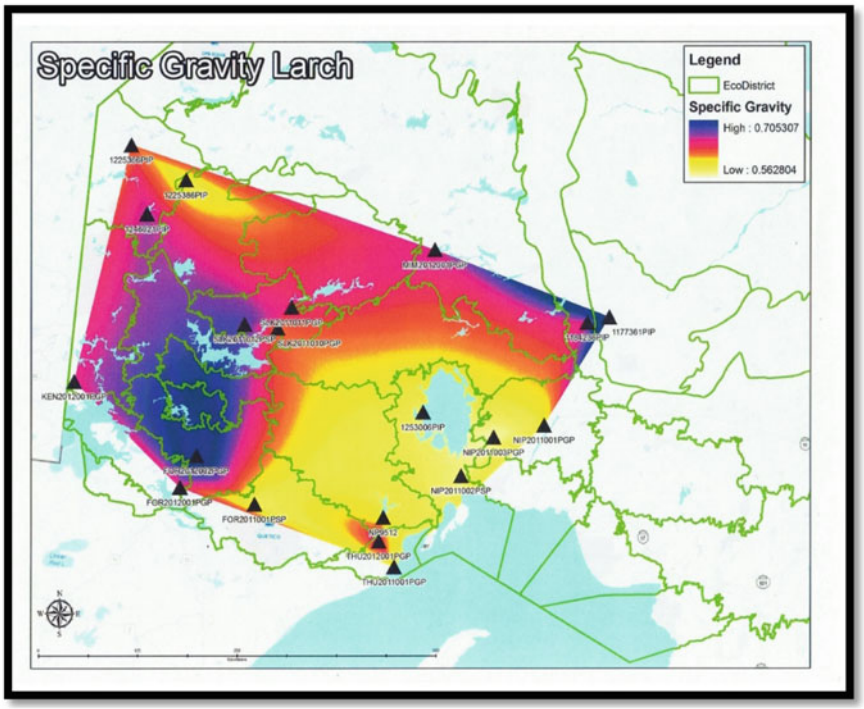


Fig. 9 Map for Eastern Larch specific gravity showing the variation across the landscape in Northwestern Ontario

how to use this to optimize utilization (Lundqvist 2001); however, none look at the landscape level and at the intensity of our data collection system. The full landscape maps will look like Fig. 10 where polygons of stands with the same properties will be identified and labeled with the lumber grades that are found on that site. Figure 10a is a polygon created using the existing Forest Resource Inventory where a large area has been identified as one polygon. When you look at Fig. 10b, it can be seen that through intensive landscape property mapping using our WS App., more detail about the resource becomes apparent where a once single polygon has become 3 polygons of very different wood properties.

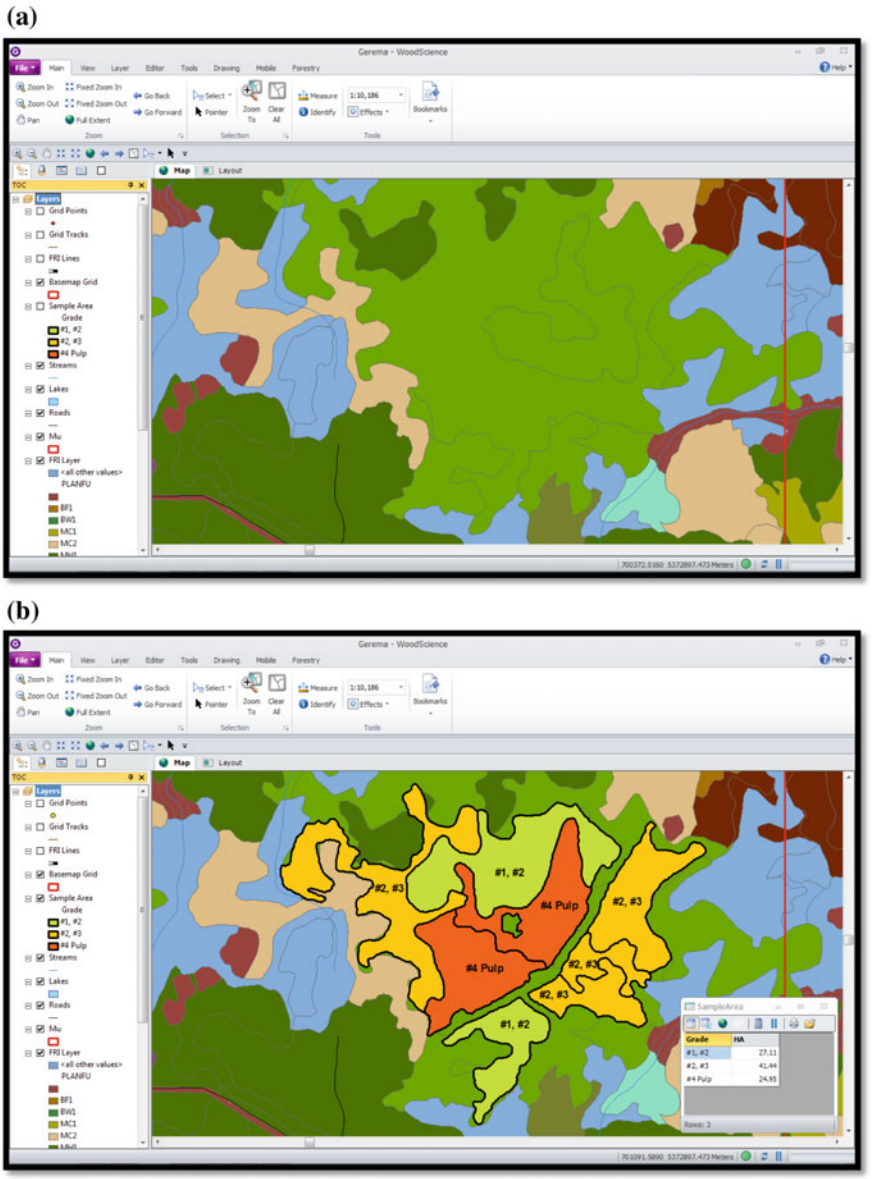


Fig. 10 Landscape map of **a** traditional inventory where the block is classified as one type, and **b** our mapping based on wood properties and re-labeling based on NLGA lumber Grades

Conclusions

The LUWSTF has developed a very efficient system of collecting, controlling, and creating reports for resource inventory research. The system eliminates many issues that exist in research where field and laboratory work create huge data files that need to be put in a storage system that allows easy entry of data and easy recovery of that data for further research or reporting. The LUWSTF Wood Science App, LU-FRAD, and the LU-NAS all combine and work together to allow this efficient system to control data. The opportunities in the forest resource inventory area are enormous both for government to have a good description of the forest resource including wood quality and also for industry to more effectively manage their holdings and react to market demand more efficiently. The ability to recognize where the wood quality varies across the landscape is very useful to allow Canadian producers to be more competitive in a global market where time to meet market demand is critical. In addition, other opportunities to utilize this system are many and the commercialization of our Wood Science App will hopefully help others in their activities.

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Screening Corewood of Pine for Wood Properties

M. Sharma, J.C.F. Walker and Shakti S. Chauhan

Abstract Conventional breeding requires long breeding cycles which limits selection intensity due to large tree sizes and high costs associated with assessing properties. In contrast very early selection offers a short cycle and assesses corewood which is the poorest wood in the tree. Young *Pinus radiata* trees were deliberately leant to produce separately opposite wood (OW) and compression wood (CW). The two distinct wood types were evaluated for dynamic modulus of elasticity, basic density, longitudinal shrinkage and volumetric shrinkage. A subset was also characterised for microfibril angle and mechanical properties using dynamic mechanical analyser to understand property–structure relationships in OW and CW. The wood properties of the two wood types differ significantly. We observed, for example, higher stiffness and density of CW which implies that selection in a nominally vertical stem would inadvertently result in a biased selection in favour of trees that happened to have abundant CW. This is avoided by focusing on the properties of OW in leant stems.

Keywords Radiata pine · Early selection · Compression wood and opposite wood

Introduction

Conventional tree breeding of radiata pine examines the wood quality in 8–11-year-old trees. Ostensibly this is desired as it gives a high age–age correlation between properties at selection age and at the harvest age. However, outerwood properties are of little concern. Apiolaza et al. (2013) argued that the main problem in radiata pine is the high per cent of poor-quality corewood, formed in the first 5–10 growth rings. Corewood has undesirable properties such as low basic density, high

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microfibril angle (MFA), high longitudinal shrinkage, short tracheids, high lignin and low cellulose content. It is this wood that needs to be improved to an acceptable level. For example, the current price differential between box grade material (the worst of the corewood) and utilitarian grades for framing (MGP10) is much greater than that between utilitarian grades and premium grades for engineering (MGP12) and finishing purposes found in outerwood (Apiolaza et al. 2013). Focusing on corewood permits working with young trees and justifies very early selection (<3 years). Shorter breeding cycles should outweigh any lower accuracy in early selection (Apiolaza 2009). Moreover, it is more practical to, consider whether cost, timescale or logistics, characterise a large population of small-diameter trees at age <3 years than a small population of large-diameter 8–11-year-old trees.

The large variation in stiffness in young radiata pine and its degree of genetic control (Lindström et al. 2004; Dungey et al. 2006) mean that stiffness can be improved by screening at a young age. There is a practical difficulty, namely the intermittent and uncertain presence of compression wood (CW) in young trees (Nakada 2007). Although CW is associated with leaning trees, even vertical stems can have significant amounts of CW, though severity and amount of CW is higher in leaning trees (Donaldson et al. 2004). Burdon (1975) examined 12-year-old *Pinus radiata* on four sites in New Zealand and estimated that 30–45% of the stem volume contained mild to severe compression wood. Deliberately leaning young trees separates the two wood types, resulting in pure CW on the underside and pure opposite wood (OW) on the upper side of the leant stem. These can be assessed separately for range of wood quality parameters.

Non-destructive acoustic testing in 8–11-year-old tree (Kumar et al. 2002; Matheson et al. 2008) is widely used in breeding programs. In young trees, due to their small relative size and poor form, the problem of CW is exaggerated and a non-destructive test will not be ideal, because it is not possible to determine the percentage of CW in an intact tree and the results of screening can mislead. This paper used acoustic resonance to characterise wood properties of CW and OW in short lengths of stemwood cut from leaning trees. The results were referenced against a more fundamental property MFA, which is considered as the main determinant of stiffness and stability (Cave and Walker 1994). At high MFAs, which occur in corewood and CW, the effect of the cell-wall matrix on wood properties was predicted to be particularly pronounced (Yamamoto and Kojima 2002; Xu et al. 2011). Therefore, storage modulus and damping coefficient (tanδ) were studied using DMA to understand the structure–property relationship and explore the possibilities of gathering information about matrix composition and its influence on wood properties.

Materials and Methods

A multi-family radiata pine progeny trial was established at Amberley, Canterbury, New Zealand, on non-irrigated flat land in 2007. In August 2008, all trees were staked and leant at an angle of $\sim 30°$. Trees were low pruned at a regular interval to

get a clear bole stem. The trees were harvested in June 2010, and a 300-mm-long clear stemwood sample was extracted immediately above the root collar.

The full experimental procedures are described in Chauhan et al. (2013). First, 120-mm-long bolt from the base of each tree was ripped and trimmed to give two 100 mm long samples (three sides sawn leaving the cambial surface as a slightly curved fourth face): one sample came from the upper side of the lean yielding OW and the other sample from the lower side yielding CW. Samples were dried at 35 °C until they reached constant weight, corresponding to a moisture content (MC) of *ca.* 5%. Density (ρ) was calculated from dry weight ($\sim$5% MC) and volume of the sample. Stiffness (hereafter referred to as $MOE_{dynamic}$) was determined by the product of the resonance acoustic velocity squared (V^2) measured using WoodSpec (developed by Industrial Research Ltd) and the previously determined wood density:

$$MOE_{dynamic} = V^2 \rho \quad (1)$$

A subset of 163 specimens, taken uniformly across the stiffness spectrum, was used in a further study of MFA and dynamic mechanical analysis (DMA). A 10 mm thick strip adjacent to the bark was used to prepare two specimens: one 10 × 5 × 1.5 mm (longitudinal × tangential × radial) for X-ray diffraction and a second 40 × 10 × 1.5 mm (L × T × R) for DMA.

The X-ray diffractometer at the School of Biological Sciences, University of Auckland, comprised of: a Rigaku (Tokyo, Japan) MicroMax-007HF generator outputting 40 kV and 30 mA; a rotating copper anode; an Osmic (Michigan, USA) VariMax-HF mirror and a Mar (Norderstedt, Germany) 345dtb detector. The beam was orientated in the radial direction of the wood samples with exposure set to three minutes. Samples were positioned at a distance of 200 mm from the detector. An open source software "The Area Diffraction Machine Version" was used to integrate the intensity of the diffraction profile of 002 planes of cellulose. Cave and Robinson's method (Cave and Robinson 1998) was used to interpret the integrated diffraction profiles.

For DMA, samples were conditioned in a humidity control box over calcium nitrate (i.e. at 50% relative humidity) at 25 °C which corresponds to 9% moisture content. Density of samples was calculated using mass and dimensions of the sample. A DMA Q800 with a humidity control chamber from TA Instruments with single-cantilever bending mode was used with 17.5 mm distance between the clamping midpoints. Samples were clamped on their tangential face with clamping torque of $\sim$1 Nm. A displacement of 15 μm amplitude was applied at 1 Hz frequency at a constant temperature (25 °C).

Results and Discussion

The two wood types differ significantly in measured wood properties (Table 1). Compression wood is characterised by higher basic density and longitudinal shrinkage and lower acoustic velocity squared and volumetric shrinkage.

The mean basic density at ~5% moisture content was ~544 kg/m^3 for CW and 365 kg/m^3 for OW. Donaldson et al. (2004) reported a mean density 572 kg/m^3 in CW and 477 kg/m^3 in OW in leaning 18-year-old *Pinus radiata* trees. The low density of OW in the present study can be attributed to the juvenile nature of the samples which are characterised by low percentage of latewood, thin tracheid cell walls and large lumens.

The high dry acoustic velocity in OW compared to CW at ~5% moisture content could be attributed to a lower microfibril angle in OW. The difference in their mean acoustic velocities was 6%, so only a small difference in MFAs is to be expected. Indeed the mean MFAs in OW and CW differed by only 5 (Fig. 1). The MFA range in OW was 28°–48° which is consistent with Donaldson (1993). The range for CW was from 36° to 53°. There was good correlation between dry acoustic velocity^2 (V^2) measured on the principal 100 mm sample of OW and the MFA values on the smaller piece taken from the larger sample, although the same relationship was not as strong in CW (Fig. 2). The results not only validate the acoustic technique but also demonstrate that being a bulk measurement it adds further benefits in screening young trees.

The mean dynamic modulus was 3.40 GPa in CW and 3.03 GPa in OW (Table 1) for three-year-old trees. The slightly higher stiffness in CW, arising from its greater density, highlights the necessity to lean the trees as high stiffness in straight trees might be due to randomly distributed CW rather than due to stiffer OW.

Longitudinal shrinkage in OW and CW was comparable to previously reported results (Dadswell and Wardrop 1949; Ying et al. 1993). In this study and previous studies (Harris and Meylan 1965; Wooten et al. 1967) even at comparable MFA,

Table 1 Summary statistics of opposite wood and compression wood properties at 5% moisture content in young three-year-old *Pinus radiata* trees

Variables	Opposite wood		Compression wood	
	Mean	CV%	Mean	CV%
Basic density (kg/m^3)	365	7.1	544	10.4
Dry density (kg/m^3)	419	6.7	591	10.8
Dry acoustic velocity^2 (km/s)^2	7.23	9.7	5.77	8.0
Dynamic modulus (GPa)	3.03	12.3	3.40	10.6
Longitudinal shrinkage (%)	1.01	29.4	3.34	19.1
Volumetric shrinkage (%)	12.7	17.6	8.0	12.75

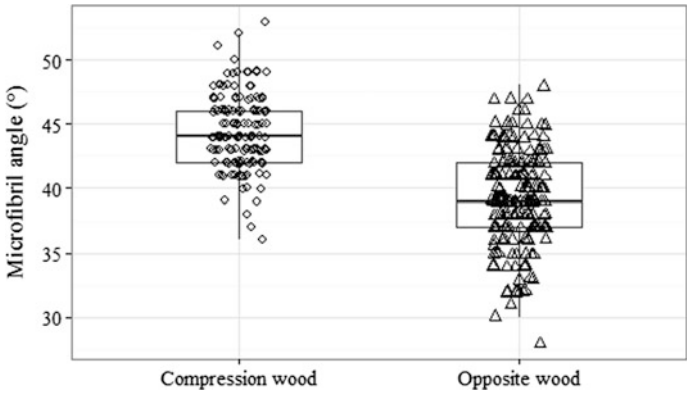

Fig. 1 Boxplot representing variation in MFA in compression wood and opposite wood in young *Pinus radiata*

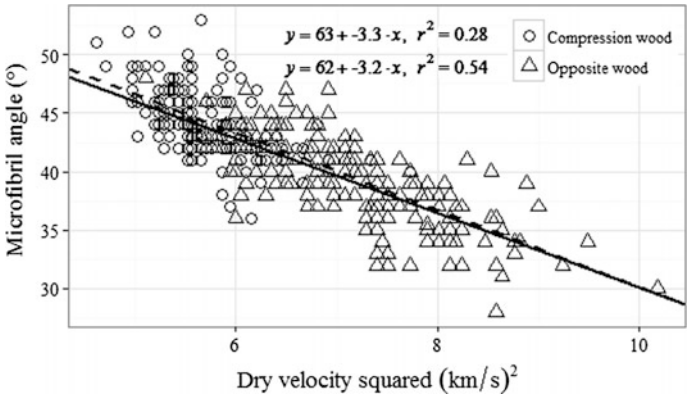

Fig. 2 Relationship between MFA and acoustic velocity squared in compression wood and opposite wood in young *Pinus radiata*

CW shrinkage is three times greater than opposite wood in longitudinal direction. Harris (1977) suggested that in addition to MFA, tracheid wall thickness is also responsible for high longitudinal shrinkage in CW, while Floyd (2005) noted that galactose content correlated with high longitudinal shrinkage. In CW the lignified secondary layer ($S2_{(L)}$) is characterised by a high level of (1-4)-β-galactan content (Altaner et al. 2010). Thus, along with physical and structure factors the galactan content in CW might contribute to the high longitudinal shrinkage in CW.

In opposite wood stiffness ranges from 1.94 to 4.90 GPa, and longitudinal shrinkage ranges from 0.15 to 1.91%, suggesting that there are possibilities to select for stiffness and stability at a young age. Moreover, there was strong negative correlation between stiffness and longitudinal shrinkage in opposite wood ($R = -0.68$). Thus, stiff opposite wood is also dimensionally stable.

The mean volumetric shrinkage for CW was 8%, and for OW it was 12.7%. Theoretically volumetric shrinkage increases with density, but high volumetric shrinkage in OW could be attributed to the fact that the thin walls in OW have collapsed while drying. Collapse was noticed in samples with low density. Comparatively low volumetric shrinkage in denser CW can be explained by its anatomical structure. The absence of S3 wall results in the cell wall shrinking towards the primary cell wall, i.e. increasing the size of the lumen without affecting the outer dimensions of the tracheid (Kelsey 1963; Harris and Meylan 1965).

The DMA analysis revealed that two wood types differed significantly in their damping coefficients (tanδ) (Fig. 3). Lower damping in CW was consistent with an earlier study (Brémaud et al. 2013). As MFA is positively correlated with tanδ, lower damping in CW is likely to be caused by the different proportions or structures of the cell-wall polymers, or the anatomical features of the tracheids. Brémaud et al. (2013) suggested that the abundance and more condensed nature of lignin with fewer methoxy groups in CW could be the reason for its lower damping.

The correlation between mechanical properties measured using DMA and other wood properties is given in Table 2. In OW, there was a significant positive correlation between MFA and tanδ suggesting that chemical composition alone is not the reason for tanδ variation. In CW tanδ did not correlate with MFA. The reason could be the low variation in MFA in CW. Thus, it might be possible to predict chemical composition particularly lignin percentage using tanδ values in CW.

In spite of the modest sample size in the DMA work, there is good correlation between storage modulus measured by DMA and dynamic MOE measured by acoustics (Fig. 4). Therefore, acoustics being faster and a bulk measurement is a better option to screen young wood for stiffness.

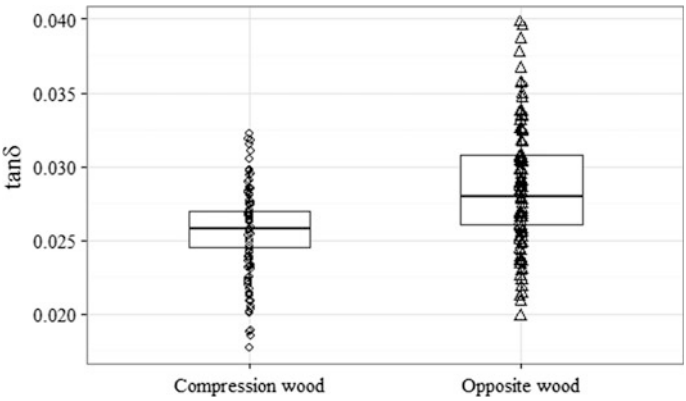

Fig. 3 Boxplots representing variation in tanδ measured at 25 °C, 1 Hz and 9% moisture content in compression wood and opposite wood in young *Pinus radiata*

Table 2 Pearson correlation matrix (with *p* values in parentheses) for wood properties of young *Pinus radiata*

Variable	Basic density (kg/m^3)	Acoustic velocity^2 $(km/s)^2$	Dynamic modulus (GPa)	MFA (°)	tanδ	Storage modulus (GPa)
Basic density (kg/m^3)		0.23 (0.03)	0.68 (<0.01)	−0.02 (0.87)	−0.26 (0.09)	0.55 (<0.01)
Acoustic velocity^2 $(km/s)^2$	−0.36 (<0.01)		0.85 (<0.01)	−0.74 (<0.01)	−0.53 (<0.01)	−0.67 (<0.01)
Dynamic modulus (GPa)	0.73 (<0.01)	0.34 (<0.01)		−0.53 (<0.01)	−0.50 (<0.01)	0.79 (<0.01)
MFA (°)	0.28 (<0.01)	−0.61 (<0.01)	−0.14 (0.17)		0.47 (<0.01)	−0.50 (<0.01)
Tanδ*	0.05 (0.62)	−0.27 (<0.01)	−0.11 (0.29)	0.16 (0.12)		−0.61 (<0.01)
Storage modulus[a] (GPa)	0.42 (<0.01)	−0.24 (<0.01)	0.61 (<0.01)	−0.08 (<0.01)	0.27 (<0.01)	

Opposite wood data above the diagonal, compression wood lies below the diagonal

[a]Measured at 25 °C, 1 Hz and 9% MC; other variables were measured at 20° and 5% MC

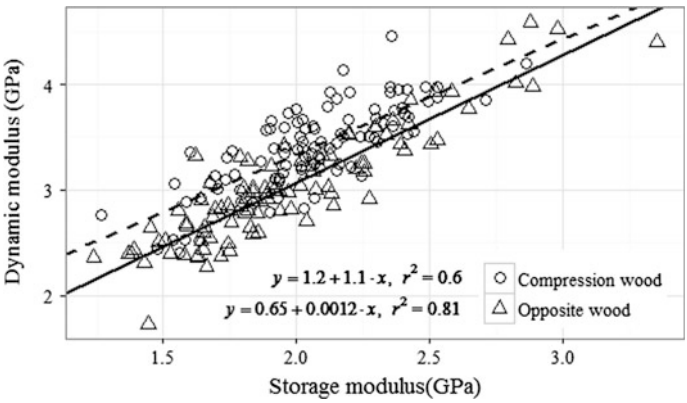

Fig. 4 Relationship between storage modulus measured at 25 °C, 1 Hz and 9% moisture content and dynamic modulus measured at room temperature and 5% moisture content in compression wood and opposite wood of young *Pinus radiata*

Conclusions

The wood types differ significantly in all the measured properties. Large variations in phenotypic stiffness (1.94–4.90 GPa) and longitudinal shrinkage (0.15–1.91%) within opposite wood suggest that it is possible to improve corewood quality by selecting at young age. It can be done much faster and economically by using resonance acoustic technique.

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Imaging Spiral Grain in *Pinus radiata* with X-ray Microtomography

Jimmy Thomas and David A. Collings

Abstract A new method was developed to visualise spiral grain in 1-year-old *Pinus radiata* trees by tracking the orientation of resin canals which follow the grain. Complete serial transverse sections were imaged at high resolution (2400 dpi) with a professional flatbed scanner using circular polarised transmitted light. Circular polarised light was created by arranging linear polariser sheets and quarter wave-retarder plates at specific angles. These caused the resin canals to appear as black dots against the bright background of birefringent tracheids. ImageJ macros were used to align the images, and a series of image processing steps were applied to detect and map the location of the canals. Only resin canals were identified, and when shown as white dots in the resultant image stack, they could be used to generate a 3D view of spiral grain using the ImageJ '*3D Viewer*' plug-in. These 3D visualisations showed the organisation of resin canals and confirmed the rapid onset of spiral grain, with the near-vertical grain adjacent to the pith generally reorienting to a strong left-handed spiral within the first year of growth. Using the SkyScan 1172 X-ray microtomography system, tomograms of the remaining portions of the wood specimens were collected with resolutions of 2–3 μm per pixel and converted to transverse section images. Processed images and 3D visualisations showed a similar view of resin canal orientation and spiral grain as compared to the scanner method. These methods provided new insights into our understanding on the formation of spiral grain.

Keywords Circular polarised light · Image analysis · Radiata pine · Resin canals · Spiral grain · X-ray microtomography

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Introduction

This paper describes a series of novel image acquisition and analysis techniques that create three-dimensional (3D) visualisations and measurements of spiral grain in *Pinus radiata* (radiata pine). Spiral grain is the inclination of fibres to the vertical axis of the tree. In pines, it leads to a reduction in the strength of sawn timber (Cown et al. 1991) and badly affects the surface smoothness (Sepúlveda 2001) resulting in downgrade in timber quality and the rejection of sawn boards (Johansson et al. 2001). Further, over 90% of drying problems in *Pinus radiata* are related to twist which can occur, in part, because of spiral grain (Cown et al. 1996). In Europe, the annual loss caused by distortion due to spiral grain in dried timber was estimated to nearly €1 billion (Tarvainen 2005).

Spiral grain is traditionally measured by the scribe test (Northcott 1957) or using destructive techniques like radial splitting (Brazier 1965) and can be observed at a cellular level through serial sectioning (Bannan 1966; Hejnowicz 1961; Włoch et al. 2002). There are, however, few approaches for studying changes in grain at the cellular level across large areas of tissue. Modern approaches to investigate spiral grain include confocal microscopy, computed tomography and digital imaging coupled with image analysis. Although confocal microscopy was used for measurements of interlocked grain in a tropical hardwood, *Hopea odorata* (Ogata and Fujita 2005), our own experiments with confocal microscopy were unsuccessful due to limited depth of field and poor laser penetration. Instead, we have developed two novel methods to visualise spiral grain. The first approach visualised resin canals, the tubular intercellular ducts that occur in most conifers (IAWA Committee 2004; Rao and Juneja 1992), with a desktop scanner adapted to image with circular polarised light. Our observations confirmed that the canals run parallel to the tracheids and that measurements of their orientations in 3D reconstructions reveal the spiral grain angle of wood. It is also demonstrated that X-ray microtomography (μCT), an emerging, non-destructive technique for obtaining internal characteristics of various materials including timber, give similar observations of the resin canals. Although these investigations revealed the grain at a near-cellular level, by studying the entire pine tree stem, overall observations of grain could be made. This information at cellular level will be essential to understand the incremental changes in tracheid orientation that contribute to the formation of spiral grain.

Materials and Methods

Serial Sectioning and Imaging Spiral Grain by Polarised Light Scanning

Eight-month-old trees that had been grown either vertically or tilted at 30–45° for several months were used for these experiments. Stem sections (25 mm long, 8–

10 mm in diameter) had a vertical line scored along the surface as a baseline from which to measure grain angle and also to act as a reference mark during image reconstructions, and were fixed in FAA. After washing, 60-µm-thick, complete transverse sections were cut using a sledge microtome (HM400, Microm, Walldorf, Germany) and mounted on slides in glycerol. Serial sections (typically 72 sections covering 4.5 mm) were scanned at 2400 dpi with a professional flatbed scanner (Epson Perfection V700 Photo) modified to run with circular polarised light. Slides were placed between crossed sheets of linear polarising film that surrounded paired sheets of quarter wave-retarder film angled at 45° (all from Edmunds Optics, Singapore) (Fig. 1a). This meant that the wood sections were imaged with circularly polarised light and that whereas the secondary cell walls of the tracheids rotated the polarised light so that it would pass through the second polariser film, background light whose polarity remained unmodified would be eliminated from the image (Fig. 1b). To generate 3D reconstructions, the background of each serial section image was filled with white to enable a uniform thresholding, and the images were aligned manually in Photoshop. Subsequent image processing was completed using ImageJ. The stack of images was fully aligned with the plug-ins '*StackReg*' and '*Cumulative Rotation*'. Uniform thresholding was applied, and the '*Analyse particles*' function run to detect the resin canals with restrictions (size = 25–500 square pixels, circularity = 0.5–1.0), and recording specific measurements ('*Area*', '*Stack position*' and '*Centroid*', the location of the object's centre). An output file generated by the '*Show Masks*' option showing the location of the canals (Fig. 1c) was used to generate a 3D image of spiral grain using the plug-in '*3D Viewer*', while an image processing algorithm written in MATLAB was used to string together resin canals between sections and to measure the orientation of the resin.

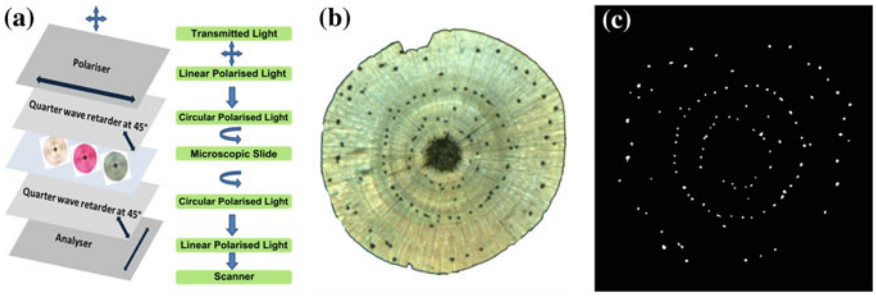


Fig. 1 Imaging and detection of resin canals: **a** imaging technique with circularly polarised light, **b** a cross section imaged with circular polarised light, demonstrating even lighting across the sample, and **c** resin canals, detected using ImageJ macros, shown as *white dots*

X-ray Microtomography Imaging of Spiral Grain

A high-resolution X-ray microtomography system (SkyScan1172, Kontich, Belgium) was used to image the same stems from which serial sections had been cut so that direct comparisons could be made between X-ray tomography and serial sectioning approaches. Dried whole stems were mounted vertically on the specimen holder and imaged with X-rays and a high-sensitivity digital CCD camera at 0.3° increments through a rotation of 180°. These raw X-ray images were processed through the SkyScan software to produce a series of transverse sections (tomograms) from the raw X-ray images. Resin canals were detected in the tomograms using a similar approach to that used for serial sections (although the stack alignment plug-ins were not required), 3D images reconstructed, measurements of grain attempted in MATLAB.

Results and Discussion

Imaging Stem Transverse Sections with a Flatbed Scanner

Our new scanning technique uses a combination of polarisers (Arpin et al. 2002) and quarter wave-retarders (Higgins 2010) to create high-contrast images that can be used for image analysis. This approach replicates conventional polarised light microscopy but, by using a desktop scanner, can image large areas that would be difficult with microscopy. The approach requires the use of circular rather than linear polarised light, because linear polarisation generates on-axis reductions in intensity (the 'Maltese cross' effect) which confounds automated image analysis.

Numeric Analysis and Quantification of Spiral Grain Using MATLAB

ImageJ identified the location of resin canals in each section and generated 3D representations of the grain which showed increased spiralling at locations further away from the central pith (Fig. 2a). The locations of the resin canals could be strung together into chains using an algorithm in MATLAB, which also calculated the location and angle of these chains and represented the chains as arrows. The arrowheads represent the canal at the top of the stem while the beginning of arrow is the canal at other end of the stem. Thus, the arrow lengths indicate the magnitude of the grain, and the arrow direction the grain handedness (Fig. 2b). From these calculations, graphs could be developed that showed resin canal angle (grain angle) as a function of the distance from the centre of the tree (Fig. 2c).

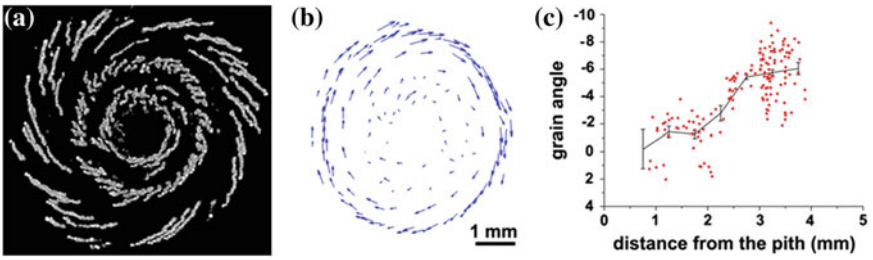
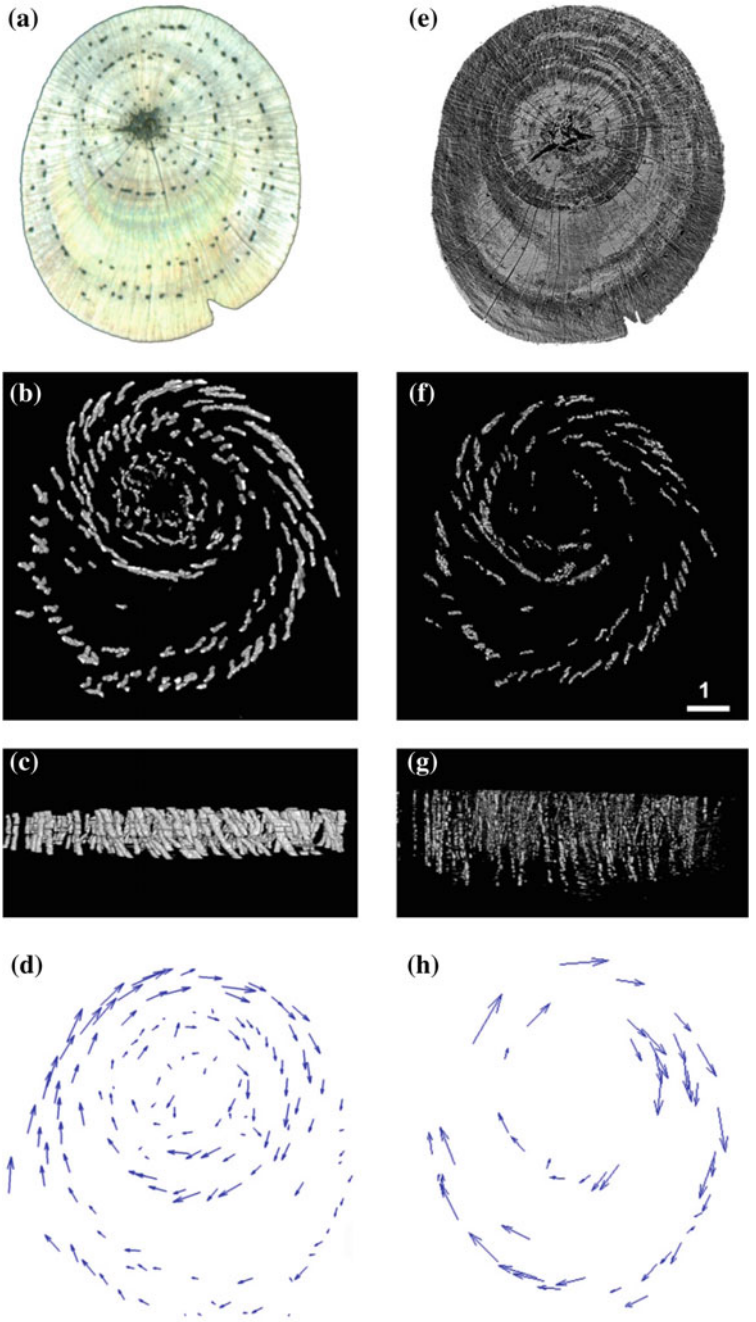


Fig. 2 3D visualisation of resin canals in a single tree, quantification of spiral grain using MATLAB. **a** Three-dimensional visualisation of resin canals and spiral grain using ImageJ. **b** Output from MATLAB showing grain from the detected resin canals, and **c** individual measurements for resin canal angles (*dots*) and average grain angle from pith to periphery

In all vertical trees, the grain was nearly straight or weakly right-handed near the pith, while the grain became more left-handed away from the pith, as has previously been reported for radiata pine (Cown et al. 1991). When five vertical trees were analysed, the average grain angle determined by the orientation of the resin canals was 'positive' near the pith (0–4°) which meant that a right-handed grain existed in that area. The average grain angle progressively decreased to zero at the middle of the stem, and reversal of grain was observed towards the periphery where the left-handed twist ranged between zero and 8°.

X-ray Microtomography and Grain Angle Measurements

X-ray microtomography generated stacks of calculated sections (Fig. 3e) that closely matched transverse sections cut from adjacent locations in the same stem and imaged with the circular polarised light images (Fig. 3a) with resin canals detectable in both image stacks in ImageJ (Fig. 3b, f). However, not all of the resin canals identified in reconstructions from the polarised light images could be identified in reconstructions from the tomograms. The ImageJ detection routine was optimised for the relatively large-sized 'canals' detected by circular polarised light that have several layers of parenchyma cells with primary cell walls that only weakly rotate polarised light, thus appearing dark against the bright background of the tracheid secondary walls. By X-ray microtomography, however, the cell walls of these parenchyma cells were still imaged which meant that the observed size of the canals seen was considerably smaller. This meant that the ImageJ detection of the canals was compromised and that MATLAB had fewer canal locations to string together to generate overall grain angles (Fig. 3f, h). In 3D reconstructions of the resin canals, viewed from side on, the left-handed twist exhibited by the resin canals in both the samples was clearly visible (Fig. 3c, g) although the angle of the canals (i.e., the grain) was magnified sixfold in the reconstructions from the polarised light because


(a)
(e)
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(f)
1
(c)
(g)
(d)
(h)

◀ **Fig. 3** Comparison of grain detection using polarised light scan and X-ray tomography: **a** circular polarised light scan image of a tilted tree, **b** 3D reconstruction of resin canals imaged with circular polarised light, **c** a side view of the 3D reconstruction in **b** showing left-handed resin canals, **d** grain map derived from the resin canals detected in **b**, **e** an X-ray tomogram at a location adjacent to the section in **a**, **f** 3D reconstruction of resin canals imaged with X-ray tomography, **g** a side view of the 3D reconstruction in **f** showing left-handed resin canals, **h** grain map derived from the resin canals detected in **f**. Not as many resin canals were detected by X-ray tomography compared to circular polarised light

the z-resolution (60 μm-thick sections) was six times higher than the scanner resolution (10 μm).

The use of X-rays to investigate grain at a cellular level, and over large areas of tissue, has been limited to the soft X-ray investigations of interlocked grain in *Acacia* (Ogata et al. 2003). However, no three-dimensional reconstructions were attempted in that study. Our study is, therefore, the first attempt to look at grain across an entire stem but considering patterns at a cellular level. Radiata pine normally shows the initial development of left-handed grain in a symmetric pattern around the stem. Tilted trees, however, formed compression wood in their lower halves and the organisation of the canals around the pith showed a remarkable asymmetry. There were fewer canals in the compression wood region, and these appeared to be much straighter than the twisted canals found elsewhere. Those canals in the opposite wood regions showed similar characteristics of canals in the vertical trees.

Conclusions

Our study has confirmed the early onset of left-handed spiral grain in vertically-grown radiata pine seedlings, a pattern that is typical of other pines. Left-handed grain develops in the corewood, followed by a reduction in the angle of the grain and sometimes the formation of right-handed wood. Using both X-ray microtomography and reconstructions based on serial sections, it is shown that the resin canals near to the pith showed lower spiralling, whereas the canals near to the cambium were highly spiralled in a left-handed direction. These methods were also used to detect grain patterns in tilted trees forming compression wood. While the fundamental difference between the control and tilted trees was that there were fewer canals in the lower side of the tilted trees and more wood formation, our analysis of these tilted trees does suggest that the grain is affected by the presence of compression wood being much straighter than in the opposite wood. These novel methods for detecting grain, using polarisers and the quarter wave-retarder plates on a scanner, provided a better way of image acquisition for the subsequent image analysis, and 3D reconstruction of internal structure and comparisons to X-ray microtomography will provide new insights into the formation of spiral grain.

Acknowledgements We thank Professor John Walker and Drs. Luis Apiolaza and Shakti Singh Chauhan (School of Forestry, University of Canterbury) for the trees used in this study and for their encouragement, and thank Dr. Jonathan Harrington (Scion Ltd, Rotorua, New Zealand) for help with MATLAB. JT gratefully acknowledges Ph.D. funding from Scion Ltd.

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Rapid Measurement of Density of Wood in Progeny Trial of *Acacia mangium* Willd. Using Resistograph—A Nondestructive Method

A.R. Uthappa, Maheshwar Hegde, P. Karthick Kumar, B. Gurudev Singh and R.S. Prashanth

Abstract Wood quality is an important factor considering the end users requirement. However, the traditional selection process which emphasizes mainly on growth traits may have negative effect on wood quality parameters. Wood density is an important attribute which is positively correlated with many wood quality parameters. The inclusion of wood density into breeding programs necessitates to find quick and inexpensive selection method that is capable of providing reasonably accurate estimates of wood density on standing trees without causing significant injury during data collection. Wood density is traditionally determined by the volumetric method that is accurate but expensive for large-scale sampling as it often involves destructive wood sample collection by cutting the trees. Efficacy of Resistograph (Rinntech® 4452-S) in quick measurement of wood density in standing trees was tested here. This hand-held device measures the drilling resistance of a needle driven into trees electronically. Seventy-five trees belonging to 25 families in a progeny trial of *Acacia mangium* were considered for the study. The Resistograph drilling resistance was taken at breast height on trees sampled, and at same height wood discs were collected to measure the wood density following volumetric method. The observations were subjected to correlation and simple linear regression analysis. The correlation between family mean resistance and the family mean wood density was higher ($r = 0.89$) than the individual tree correlation ($r = 0.81$). A weak, negative and nonsignificant correlation ($r = -0.08$) between moisture content and resistance indicated wood moisture content had no relationship with drilling resistance readings of Resistograph.

Keywords *Acacia mangium* · Wood density · Resistograph

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Introduction

Wood density is the ratio of the oven-dry weight of wood to its green volume and it is the best single predictor of wood quality for various end uses (Zobel and Van Buijtenen 1989) and is therefore often assumed to be equivalent to wood quality (Zhang et al. 1993). It is a reliable and easy-to-measure trait, which significantly affects wood suitability for different end uses. In addition to that, wood density can be manipulated by genetic improvement and silvicultural practices (Zobel and Van Buijtenen 1989). Generally, wood density is strongly correlated with other wood properties such as strength and stiffness. It is also one of the most important parameters affecting paper pulp qualities (Blair et al. 1976). Therefore, wood density is one of the very important traits to include in tree breeding programs because it is under high degree of genetic control and also has good economic value (Sprague et al. 1983).

Acacia mangium—a fast growing, evergreen leguminous tree, native of Australia, Papua New Guinea and Indonesia, has been introduced to India during 1980s. Under favourable conditions, it grows up to 30 m height and 40 cm in diameter at breast height (DBH) in 14–15 years of age. This species normally prefers acidic soils and can grow even soils with pH as low as 4.2. The main uses of this species are for timber, paper and pulp, erosion control, shade or shelter, nitrogen fixation, ornamental and intercropping. The density of *A. mangium* wood is reported to vary from 422.5 to 572.5 kg/m^3 at 12% moisture content (Dhamodaran and Chacko 1999).

The volumetric method, by which wood density is normally determined, requires extracting wood samples from trees at various heights and measuring the green volume and mass of dry wood in the laboratory after oven drying (ASTM 1985). This method is generally accurate, but it is both time-consuming and destructive as large number of trees need to be felled and also it is expensive for a tree improvement program that requires large number of trees required to be tested quickly. Several instruments like torsiometer, electronic micro densitometry and Pilodyn wood tester have been used to substitute the traditional standard methodology of wood density testing (Moura et al. 1987). Among these instruments, Pilodyn is commonly used for measuring wood density of live trees (Costa-e-Silva et al. 2000), but it also has its own drawbacks (Koch and Fins 2000). The Pilodyn requires removal of bark before driving the nail into trees and it is not recommended for through-the-bark measurements (Koch and Fins 2000).

A new device called Resistograph was considered for studying the efficacy of the instrument in determining the wood density nondestructively and rapidly in large number of trees in a 2½-year-old progeny trial of *A. mangium*. The Resistograph measures the drilling resistance offered by the wood at various points along the drilling path (bark to pith region) of the small needle. The study was conducted with

the objective of determining the relationship between the Resistograph readings and mean wood density of the *A. mangium* families and thereby facilitating selection of superior trees based on wood density using Resistograph readings.

Materials and Methods

A progeny trial of *A. mangium* established by the Institute of Forest Genetics and Tree Breeding, Coimbatore during August 2011 at Palode in Kerala, India, was selected for the study. This progeny trial consisted of 125 families collected from different sources like first-generation seed orchards, provenance stands and plantations and was planted in Latinized row–column design with four replications. For this study, 75 trees belonging to 25 families were sampled out for wood sample collection. Growth data of the sampled tress were recorded. Based on the diameter at breast height, the radius was worked out and the selected trees were drilled at breast height up to the core (radius length) using the Resistograph (Rinntech® 4452-S) and resistance was recorded. The same trees were harvested, and wood discs (0–15 cm height) were collected at 1.37 m height for wood density estimation. The green volume of the wood samples was measured by water displacement method. Oven-dry weight was measured from the same sample by drying it in a well-ventilated oven at 103 ± 2 °C until the wood samples achieved constant weights. As the samples contained high moisture content, the wood samples were dried up to 64 h in the oven. The constant weight hypothesis was tested by weighing the samples at regular intervals. The samples were weighed immediately after being taken out of the drying oven. For the field study, Resistograph model Rinntech® 4452-S was used. DECOM software version 2.3.3 supplied with the instrument was used in transferring the Resistograph data from the instrument to the computer. Using DECOM software, it is possible to measure the resistance at various regions along the needle path like bark, sapwood and heartwood regions. Using this software, the Resistograph readings at different regions were obtained viz. resistance readings with bark (resistance II) and excluding bark region (resistance III) (Fig. 1).

The volumetric wood density and Resistograph values of different families were subjected to correlation analysis. The linear regression model was used to examine the strength and nature of relationships between wood density and the Resistograph readings (% drilling resistance).

Results and Discussion

The diameter at breast height of the sampled trees varied from 5.16 to 13.28 cm, with a mean of 8.87 cm. The total height ranged from 5.5 to 14 m, with a mean of 9.83 m.

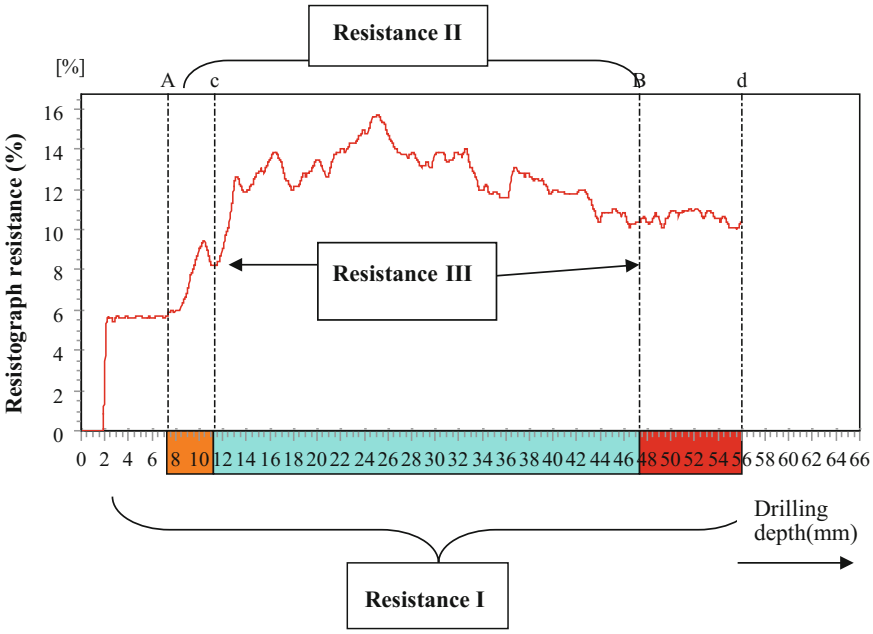

Fig. 1 Resistograph profile at different regions, namely, whole reading (*resistance I*), truncated with bark to pith (*resistance II*) and without bark (*resistance III*)

The mean wood density values varied considerably among all the 25 families (Table 1). The mean density values ranged from 0.270 to 0.479 g/cm^3. The overall mean wood density for all the families was 0.396 g/cm^3 with a standard deviation of 0.067. The wood density values were subjected to one-way ANOVA. The results revealed that there were significant differences among the families (Table 1). The significantly maximum higher wood density was recorded in the family no. 41 (0.479 g/cm^3) followed by family no. 95 (0.473 g/cm^3). The minimum wood density was recorded in the family no. 50 with a value of 0.270 g/cm^3.

The mean resistance values ranged from 3.5 to 10.3%. The total mean resistance for all the families was 7.111% with a standard deviation of 2.096. The resistance values were subjected to one-way ANOVA. The results revealed that there were significant differences among the families (Table 1). The maximum and significantly higher resistance was recorded in the family no. 41 (10.3%) followed by family no. 95 (9.4%). The minimum and significant lower resistance was observed in the family no. 50 with a value of 3.5% which was similar to that of trends observed in wood density.

Wood density and Resistograph resistance had a high positive correlation (Fig. 2). The correlation between resistance and wood density estimated by volumetric method was 0.814. The resistance reading values of each tree were positively related to its wood density, and the coefficient of determination (R^2) in simple linear regression analysis was 0.662. The relationship between the Resistograph and wood

Table 1 Familywise mean wood density, mean resistance (Resistograph readings), standard deviation, standard error and Duncan's test of significance of means ($P < 0.05$)

Sl. no.	Families numbers	Wood density		Drilling resistance (Resistograph reading)	
		Mean	Std. error	Mean	Std. error
1	50	0.270^{a}	0.011	3.500^{a}	0.666
2	53	0.318^{ab}	0.020	6.267^{abcde}	0.186
3	76	0.337^{abc}	0.023	5.267^{ab}	0.437
4	69	0.338^{abc}	0.012	5.167^{ab}	0.578
5	29	0.347^{abcd}	0.019	6.600^{abcde}	1.514
6	30	0.349^{abcd}	0.009	5.467^{abc}	0.348
7	16	0.355^{abcde}	0.012	5.733^{abcd}	0.393
8	103	0.364^{abcde}	0.023	6.433^{abcde}	0.801
9	96	0.378^{bcdef}	0.028	7.067^{bcdef}	0.649
10	65	0.381^{bcdef}	0.004	7.100^{bcdef}	0.351
11	31	0.383^{bcdef}	0.076	7.067^{bcdef}	2.285
12	83	0.384^{bcdef}	0.011	6.167^{abcde}	0.694
13	27	0.388^{bcdef}	0.005	6.933^{bcdef}	0.467
14	84	0.420^{bcdef}	0.008	8.833^{cdef}	0.233
15	24	0.421^{bcdef}	0.028	7.500^{bcdef}	0.945
16	1	0.429^{cdef}	0.049	8.833^{cdef}	0.968
17	49	0.430^{cdef}	0.039	7.200^{bcdef}	0.814
18	34	0.432^{cdef}	0.046	7.533^{bcdef}	1.497
19	108	0.437^{cdef}	0.037	8.033^{bcdef}	0.470
20	72	0.438^{cdef}	0.009	8.900^{cdef}	0.681
21	107	0.439^{cdef}	0.020	7.033^{bcdef}	1.545
22	60	0.448^{def}	0.062	6.333^{abcde}	1.189
23	12	0.459^{ef}	0.023	9.100^{def}	1.601
24	95	0.473^{f}	0.022	9.400^{ef}	1.498
25	41	0.479^{f}	0.012	10.300^{f}	0.814
	Total	0.396	0.008	7.111	

Note Means with same letters are not significantly different

density has been represented in Fig. 2. High and significant correlation between the mean level of the Resistograph resistance and density of wood has been reported earlier (Rinn et al. 1996). Strong relationships have been reported between the volumetric- and Resistograph-based densities by a number of authors: $R^2 > 0.8$ (Rinn et al. 1996), $r = 0.82$ (Ceraldi et al. 2001), $r = 0.76$ (Lin et al. 2003), $R^2 = 0.74$ (El-Kassaby et al. 2011). Studies by Gantz (2002) revealed that Resistograph measures were moderately correlated with density data (from 0.30 to 0.78). In another study, Isik and Li (2003) found that Resistograph was useful for quickly and accurately assessing wood density in a loblolly pine (*Pinus taeda* L.) progeny trial.

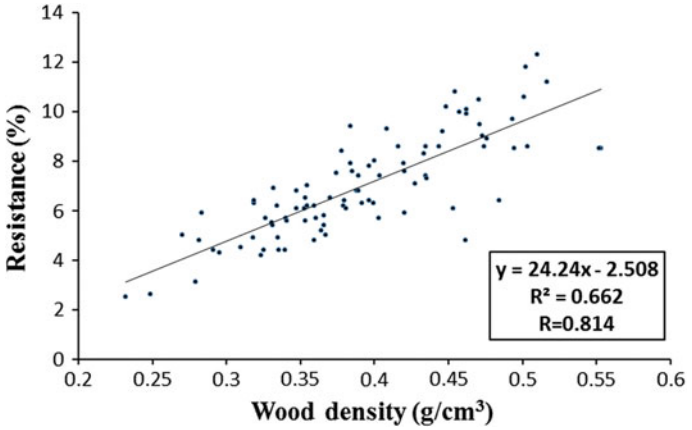

Fig. 2 Wood density determined by volumetric method were plotted against the Resistograph readings (resistance) at the individual tree level in *Acacia mangium*

Relationship Between Wood Density and Resistance Readings at Various Regions (from Bark to Pith)

To investigate the possibility of improving the correlation between Resistograph readings and wood density, the readings at various regions, namely I—the actual field drilling readings, II—the Resistograph readings from bark to pith region measured from the graph (Fig. 1) and III—sapwood to pith region and excluding bark region, were taken for correlating with wood density.

The relationship between wood density and resistance measured through various resistance readings did not vary much, and all the three readings were positively correlated with the wood density (Table 2).

Table 2 Correlation and linear regression models for truncated reading without bark (resistance III), truncated reading with bark (resistance II) and whole reading (resistance I) with wood density

Parameters	Correlation	R^2	Regression equations
Resistance III & wood density	0.81	0.66	$y = 28.12x - 2.291$
Resistance II & wood density	0.80	0.63	$y = 24.70x - 1.749$
Resistance I & wood density	0.81	0.66	$y = 24.24x - 2.508$

Correlation Between Family Mean Resistance and Family Mean Wood Density Values

The correlation between family mean resistance and the family mean wood density was higher (0.89) than the individual tree correlation (0.81). The linear simple regression coefficient (R^2) was 0.792. The relationship between the mean Resistograph resistance and mean wood density has been represented in Fig. 3 with the regression equations. Higher correlation between family mean wood density and drill resistance readings has been reported by Gwaze and Stevenson (2008). Studies in a shortleaf pine (*Pinus echinata*) revealed that at the individual tree level, the linear relationship between wood density and drill resistance (amplitude) was weak and positive (R^2 = 0.23) but was stronger (R^2 = 0.47) at the family mean level.

In another study Isik and Li (2003) reported lower individual tree-based correlations compared to family mean phenotypic relationship between density and amplitude. Bouffier et al. (2008) reported high correlation between the Resistograph and density data (R^2 = 0.93) on familial level. The Resistograph can be effectively used to rank *A. mangium* families for wood density based on amplitude (resistance) values alone as there is high family mean correlation between density and amplitude.

Relationship Between Moisture Content in Green Wood and Resistograph Readings

The moisture content in the sampled wood varied from 61 to 175% with a mean value of 120.82%. Very high moisture content in wood was because of lower age of the trees (2½ year) and collection of the samples during rainy season. The

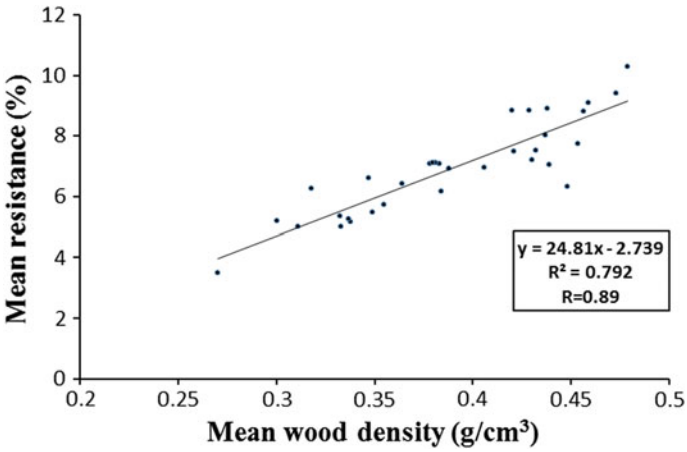

Fig. 3 Simple linear regression between family mean wood density and the family mean Resistograph readings (resistance I)

correlation studies between moisture content and resistance revealed a weak, negative and nonsignificant correlation (−0.08) indicating wood moisture content had no relationship with drilling resistance readings of Resistograph in the current study. However, the moisture content and wood density had significant but low negative correlation (−0.27). Juvenile wood, with thinner cell walls and larger lumens, leads to higher moisture content (Zobel and Sprague 1998). Generally, *A. mangium* species has high moisture content. Peh and Khoo (1984) reported 119% moisture in wood grown in Sabah, Malaysia. In contrary, Shanavas and Kumar (2006) reported that moisture content of 49.46% in mangium wood grown in Kerala, India. It is evident that the age of the tree and season of sample collection play an important role in determining wood moisture content. Negative correlation of wood density with moisture content has been reported in many species (Ofori and Brentuo 2010).

Conclusion

Wood density is the best single predictor of wood quality, and it greatly affects wood suitability for different end uses. Therefore, it is of vital importance to incorporate this trait among existing selection criteria; however, this task brings about a need for finding a fast, reliable, nondestructive, and inexpensive tool for the assessment of live standing trees. Conventional method of wood density estimation necessitates either felling trees and collection of wood discs or extracting increment cores from standing trees and then estimating the wood density in the laboratory. This destructive, expensive and time-consuming method of wood sampling restricts its usage in tree improvement programs. Since the Resistograph readings have a strong positive correlation with wood density, it has a great potential to quickly assess the wood density of trees in progeny trials/breeding populations and then to select trees for wood density in *A. mangium*. The device is easily portable, relatively simple to use in the field, and it causes minimum injuries to trees as bark removal is not required. We found that hundreds of trees can be drilled in single day. The lower-ranking individuals/families can be culled early, or the higher ranking individuals/families can be selected at younger age to achieve faster progress in improvement of wood density in breeding programs. In a clonal selection program, individuals can be selected for wood density at juvenile age itself.

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Eliminating Growth-Stresses in *Eucalyptus*: A Scoping Study with *E. bosistoana* and *E. nitens*

M. Sharma, J.C.F. Walker and Shakti S. Chauhan

Abstract Tropical hardwoods dominate the market for fine timbers. In contrast, plantation-grown eucalypts, whether in temperate or tropical regions, are grown largely for pulpwood. Addressing log end-splitting and distortion of timber due to growth-stress is necessary if eucalypts are to succeed in solid wood markets. This scoping study compared growth-strain in 2-year-old *Eucalyptus nitens* using both traditional strain gauges and a novel, rapid slit-opening method. Subsequently only the splitting test was used to screen 2-year-old *Eucalyptus bosistoana*. The splitting test offers a cheap and fast, yet repeatable and rigorous selection method to screen very young trees and so opens the way to create a new landrace of low growth-strain winners.

Keywords Early selection · Eucalyptus · Growth-strain

Introduction

Hardwoods have the advantage of not forming corewood (as traditionally understood), although Panshin and De Zeeuw (1980) note "*As a general rule the low quality of corewood is more marked in conifers than in hardwoods*" and so ought to be better suited to short rotations. Yet fast-growing eucalypts have been planted principally for energy and pulpwood. Only recently silvicultural regimes have been adapted for solid products (Acosta et al. 2008).

The constraint on the use of eucalypts as sawlogs is the unpredictable yet certain fact that some good-looking sawlogs will split before processing, or their timber will distort or move off the saw or boards will warp on drying (Dahman 1975; Poole et al. 2013). These problems arise from the huge "between-tree" variability in

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growth-stress generated continuously in the cambium during the entire life of the tree: the wood forming in the cambium is laid down in tension and wants to contract, and as a consequence the wood near the pith experiences increasing compressive stress and wants to elongate. The resulting radial gradient, from axial tension at the cambium to axial compression at the pith, is generally greater in small diameter trees/logs than in larger diameter trees/logs which is why long rotations are favoured when growing sawlogs (Haslett 1988).

There is no direct way to measure growth-stress in trees or logs. Instead, growth-stress is calculated indirectly by measuring growth-strain resulting from the release of stress (Nicholson 1971; Kikata 1972; Archer 1978). The strain values themselves are useful in predicting the stability of timber. Muneri and Leggate (Muneri and Leggate 2000) predicted bow distortion on sawing from the longitudinal growth-strain measurements in 4-year-old *Eucalyptus pilularis*. Okuyama et al. (2004) and Valencia et al. (2011) found that high longitudinal growth-strain was a significant indicator of increased board end-splitting.

Growth-strain (and associated log end-splitting, warp, collapse, checking and brittle heart) imposes substantial processing costs. These characteristics vary greatly between trees and fortunately are highly heritable with good later-age correlations, so the opportunity exists to breed eucalypts with low growth-strain, which will be better suited to solid wood processing (Murphy et al. 2005; Apiolaza 2009; Potts et al. 2011).

Selection of a low growth-strain breeding population—alongside but not secondary to other objectives such as growth and form—is achievable. This scoping study shows how this might be done. It involves measuring growth-strain with the traditional strain gauge as well as the "splitting test" in 2-year-old *Eucalyptus nitens* and *Eucalyptus bosistoana*. The purpose is to establish cheap and fast, yet repeatable and rigorous selection methodology to create a sub-population of winners. The first experiment with *E. nitens* (seed of uncertain origin) sought to validate early selection procedures using the "splitting test", while a second experiment with *E. bosistoana* ought to demonstrate between- and within-family variability and so the viability of mass screening to create a new landrace consisting of a commonwealth of low growth-strain individuals.

The theory behind the splitting test (Fig. 1) has been examined by Chauhan (2008, 2009), Chauhan and Entwistle (2010) and Entwistle et al. (2014). The relevant, simplified equation is:

$$\varepsilon = k_1 \frac{YD^2}{L^2} \quad \text{or} \quad y = k_2 \frac{\varepsilon L^2}{D} \tag{1}$$

where ε is the growth-strain, k is a constant, Y is the opening between the two half-round prongs, D is the mean stem diameter under bark and L is the length of the sawn slit.

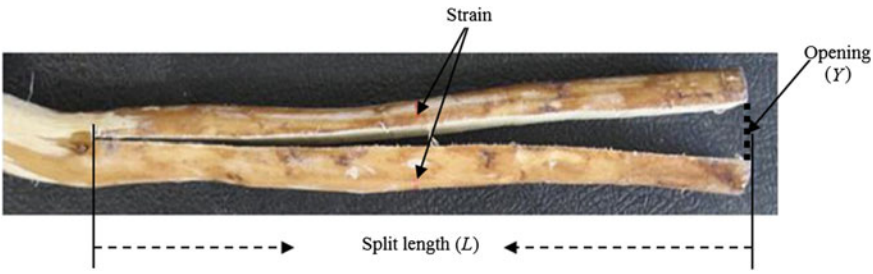

Fig. 1 Diagram representing the different parameters used to measure strain in a spilt log

Material and Methodology

Eucalyptus nitens seedlings of unknown origin were procured from a local nursery and planted in 75-L bags and trickle irrigated to maintain uniform growth conditions. After three months of planting, a group of stems were tilted at an angle of about 20° with the help of garden stake. The remaining trees were allowed to grow vertically. To ensure the vertical growth, the stems were tied with garden stakes. All the stems were pruned to give a clear segments of >600 mm length above the root collar for wood quality testing. Two years after planting, 40 leaning and 40 vertically straight trees were selected for analysis of wood quality. The selected trees were harvested, and clearwood segments were extracted from each tree. The leaning stems are expected to produce tension wood on the upper side of the lean. The upper and lower sides were marked prior to felling. The clearwood stem lengths were taken to the laboratory, debarked and end-trimmed. A pair of strain gauges were glued (opposite sides) at the mid-point of the segment. On the leaning stems, strain gauges were attached to the tension and opposite wood sides. The strain gauges were connected to a strain meter in quarter bridge configuration. The inherent growth-stresses were released by sawing along the pith, and the corresponding surface strains were recorded with the strain meter, while the stem opening, *Y*, as a result of outward bending of the two half-round prongs, was measured (Chauhan and Walker 2011). The saw cut perpendicular to the lean segregated tension wood from opposite wood in the leaning stems. For small diameter stems, the slit length was shorter; in larger diameter stems the slit was longer. The derived openings in Tables 1 and 2 were normalized for slit length using Eq. (1). The first study with *E. nitens* demonstrated the validity of the split-log test in measuring growth-strains in very young stems. In the follow-up study, only splitting method was adopted to assess the magnitude of growth-strains. Wood basic density, volumetric shrinkage and dynamic modulus of elasticity were determined from small wood segments cut from the two half-round sections. Resonance frequency of longitudinal vibration was obtained for each sample in air-dried condition, and dynamic modulus of elasticity was determined using the following Eq. 2:

Table 1 Summary statistics of wood properties in 2-year-old vertical and leaning *E. nitens*

	Vertical		Leaning	
	Mean	CV%	Mean	CV%
Under bark diameter (mm)	34.0	21.6	35.5	11.7
Opening, normalized for a 250 mm slit length (mm)	3.84	42.4	8.15	40.9
Derived strain ($\times 10^{-6}$)	700	41.6	1652	41.4

			Tension wood		Opposite wood	
			Mean	CV%	Mean	CV%
Strain gauge measured strain ($\times 10^{-6}$)	460	46.2	913	48.0	1322	62.4
Basic density (kg m^{-3})	451	6.2	497	7.2	449	5.1
Air-dry (12% MC) dynamic modulus of elasticity (GPa)	6.4	10.5	13.4	28.6	4.7	15.1
Volumetric shrinkage, green to 12% MC (%)	14.1	16.5	22.5	29.5	11.91	30.1

Experimental procedures are described in Chauhan et al. (2013). The normalized stem opening is corrected to take account of kerf thickness (see Entwistle et al. 2014)

Table 2 Summary statistics of wood properties in 2-year old vertically grown *E. bosistoana* (n = 200, 20 families)

Parameters	Mean	CV%
Under bark diameter (mm)	28.9	15.9
Opening of slit (mm)	1.68	126
Derived strain ($\times 10^{-6)}$	258	118.6
Basic density (kg m^{-3})	615	4.5
Oven-dry dynamic modulus (GPa)	7.0	19.4
Volumetric shrinkage (%) (green to 0% MC)	24.4	14.1

Material tested came from ongoing seed collections in Australia and came from different areas within the species' natural distribution

$$\mathrm{DMoE} = 4L^2 f^2 \rho \tag{2}$$

where L is the sample length, f is the fundamental frequency and ρ is the air-dry density of wood.

In the second study, wood quality variability was assessed in young stems of *E. bosistoana* which were grown vertically in planter bags. The sourced sapling in these trials was of known genetic origin, and the study aimed at understanding the variability in growth-stresses within and between families and identifying families with lower stresses. The families came from New Zealand Dryland Forest Initiatives (NZDFI's) ongoing seed collection in Australia (Millen et al. 2009). A total of 200 trees of 2 year age (20 half-sib families with 10 reps) were tested in December 2010—trees within each replication block were planted in randomized "single-tree plots".

Results and Discussion

E. nitens *Trial*

All measured wood properties of *E. nitens* are summarized in Table 1. Strain gauge values (and values for other properties) on opposite sides of the stem for vertically grown *stems* were identical and therefore averaged. Average values for vertical trees were significantly less than for leaning trees (Table 1). The average growth-strain measured on opposite sides of the stem using strain gauges ranged from 43 to 970 $\times$ 10^{-6} in straight stems and from 270 to 2517 $\times$ 10^{-6} for leaning stems. Thus, there is little risk in any selection procedure of ranking leaning stems ahead of vertical stems, i.e. selection will not be biased in favour of leaning stems. The outward bending of two half-round prongs on slitting the stem along the length is the outcome of the stress relaxation on both sides of the stems, and therefore, the strain derived from Eq. 1 represents the sum of the strains on two sides. Therefore, the derived strain value was divided by two to get the average strain value in the stem.

Wood basic density of tension wood side was about 13% higher than opposite wood density; however, dynamic modulus of elasticity and volumetric shrinkage in samples from tension wood side were nearly 185 and 100% higher than opposite wood. These results are in congruence with those observed on one-year-old leaning stems of *Eucalyptus regnans* (Chauhan and Walker 2011) wherein the dynamic modulus of wood samples from tension wood side (upper side of the lean) was nearly threefold as compared to opposite wood (lower side of the lean). Interestingly, the mean growth-strains on the opposite side were higher than tension wood side which is in contrast to the general perception reported in the literature and therefore needs to be further investigated.

The relationship between growth-strain measurements from strain gauges and those derived from the opening, Y, between the two half-round prongs is plotted in Fig. 2. A very strong linear association ($R^2 = 0.93$) between the measured growth-strain and that derived from the opening, Y, in the splitting test allows screening of genotypes with low strain by slitting alone. The slitting method, though destructive, provides a practical approach to screen several thousands of trees at very young age (<age 2) to create a new low growth-strain landrace for quality timber resources.

E. bosistoana *Trial*

Having established the practical viability of the splitting test, it would be prudent to scope its application in the same way that Murphy et al. (2005) scoped selection of low growth-strain provenances and families in 9-year-old *Eucalyptus dunnii.* This was the purpose of the second study using half-sib families of *E. bosistoana* drawn

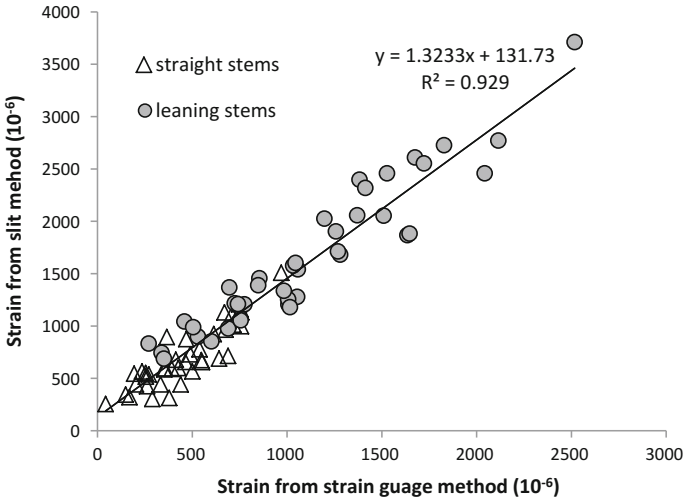

Fig. 2 Measured growth-strain using strain gauges versus derived growth-strain from slitting method

from its natural range of coastal Victoria and New South Wales (Jovanovic and Booth 2002). The measured wood properties are summarized in Table 2. Among the properties, opening on slitting and correspondingly the derived strain were highly variable. Wood basic density exhibited the least variability. It was observed that many individual stems did not exhibit any opening on slitting. Overall, it was observed that the magnitude of growth-strains in stems of *E. bosistoana* was much lower (258×10^{-6}) than that measured in *E. nitens* stems of unknown genetic origin (700×10^{-6}).

The variability in the growth-strain values within and between families is shown in Fig. 3. There is sufficient within-family variability to find one or more low growth-strain individuals in every family. It was observed that 8 individual stems in family code 1 did not exhibit measurable opening on slitting, and therefore, the derived strain was "0" for these stems. Similarly families 6 and 14 had six individuals, families 2, 5, 7 and 11 had five individuals that exhibited zero strain value, whereas families 18 and 20 had no individuals with zero strain and also had the highest average strain among the families.

For wood quality, first target should be low growth-strain. In an early study with *E. nitens* (Chauhan and Walker 2004) and in this study, there was no correlation between growth-strain measured using strain gauges and either stem diameter or MOE. Therefore, one can select individuals for low growth-strain and from this sub-population select individual with faster diameter growth and/or higher MOE.

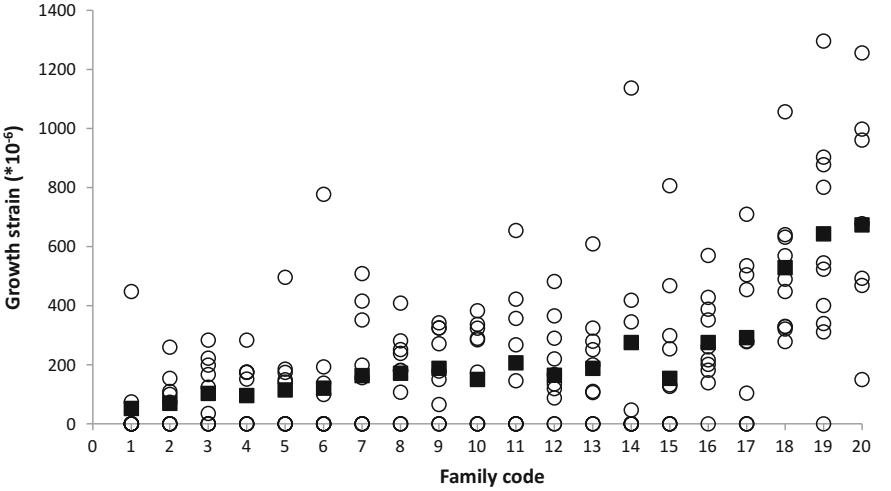

Fig. 3 Growth-strain variability within and between 20 families of *E. bosistoana*. A simple selection criterion might involve propagation of coppice from the regenerating stems for those individuals with low strain. The *filled squares* represent the mean strain for families

Conclusion

Low growth-stress should be one of the main objectives of breeding eucalyptus for high-quality timber alongside tree growth and form. The splitting test is a fast and economic method of screening for growth-stresses in young trees. The large variability in this study suggests that there are possibilities to breed trees with low growth-stresses without sacrificing for other wood properties.

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Longitudinal Growth Strains in *Melia dubia*

Sandhya Sharma, Santosh Sumbali, Pankaj Aggarwal and Shakti S. Chauhan

Abstract Logs of many fast-grown tree species develop end splitting immediately after felling, and timber from such logs suffers from excessive warping and twisting on sawing. Such distortions are attributed to the presence of high magnitude of inherent growth stresses. This results in low recovery of quality sawn material and therefore devalues a tree species for timber purposes. For the efficient utilization of any timber species, prior knowledge on the intensity and distribution of growth stresses in trees is vital. *Melia dubia* is increasingly gaining popularity as the plantation species because of its fast growth and is becoming a preferred species for plywood manufacturing. For ensuring the suitability of the species as a timber for other applications, the propensity of growth stresses needs to be known. Since inherent growth stresses cannot be measured directly, *in lieu* longitudinal growth strains are measured by relieving the stresses. In this paper, magnitude of longitudinal growth strain measured in trees and logs of *M. dubia* from three age groups growing in different locations are presented. The longitudinal growth strains were measured using wire strain gauge method. The magnitude of growth strain was ranging from 56 to 730 microstrains. The observed growth strains are in the range which does not raise a serious concern in processing and utilizing this species as timber.

Keywords Growth stress · Growth strain · *Melia dubia* · Plantation · Timber

Introduction

Plantation forestry is rapidly becoming a major source for solid wood, wood-based composites and engineered wood. Consequently, there has been a significant emphasis on obtaining higher value wood products from fast-grown plantation timbers. One of the important concerns with such material is the enormous

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variability in wood quality between and within species. Many plantation species like *Eucalyptus* spp., *Acacia auriculiformis*, *Nothofagus* spp. are prone to have relatively high magnitude of growth stresses, excessive shrinkage, processing difficulties, etc., limiting their utility for solid wood products. Understanding the natural variability of such wood quality traits and their influence on product qualities plays a critical role in effective utilization of such timbers.

High magnitude of longitudinal growth stresses has a significant implication in recovery and processing of high quality timber. Though the longitudinal growth stresses are inherent to all trees, their magnitude varies between species to species and also within species. These stresses cause ring shake and brittle heart in standing trees, end splitting in logs within a short span of felling a tree, severe warping in timber immediately after sawing and twisting and cracking in timber during drying (Kubler 1987; Yang and Waugh 2001; Chauhan and Aggarwal 2011). Growth stresses have become a major wood quality criterion in selection of superior genotypes and also assessing the economic values of logs for timber in eucalyptus species (Henson et al. 2004). Growth stress measurement in standing trees and logs is still a challenge, and generally growth strains are determined by releasing the stresses. The measured strains are considered to be the indicator of stress levels within the elastic limits as stresses are proportional to stains.

Melia dubia is an indigenous to India and is generally found in moist deciduous forests. It is also found to grow in Sri Lanka, Malaysia, Java, China and Australia (Saravanan et al. 2013). Traditionally, the wood from the species was used mainly for timber, fuel wood, agricultural implements, etc. The species has rapidly emerged as an important commercial wood for plywood and light structural applications (Parthibhan et al. 2009). Further faster growth, coppicing ability and adaptability to variety of soil conditions have incited significant interest of farmers and forestry entrepreneurs in growing the species as an agro and farm forestry timber species. Consequently, large-scale plantations of this species have been raised in several parts of India. Recognizing the importance of the species for solid wood applications, efforts are being made to assess the variability in anatomical features (Swaminathan et al. 2012), variability in wood properties (Sharma et al. 2013), suitability of using the wood for alternate applications like pulping (Saravanan et al. 2013), the genetic diversity in this species (Kumar et al. 2013) and selection of superior genotypes based on wood traits (Chauhan and Arunkumar 2014). The magnitude of growth stresses in this species has not been assessed so for. For ensuring the suitability of the species as a timber for solid wood applications, the propensity of growth stresses needs to be known.

In this paper, magnitude of longitudinal growth strain measured in trees and logs of *M. dubia* from three age groups growing in different locations are presented. In addition, the trees were also measured for girth at breast height, acoustic velocity and pilodyn penetration.

Materials and Methodology

The study was carried out in three *M. dubia* plantations in Punjab (4-year-old plantation in Dasuya, 8-year-old plantation in Mathewada, Ludhiana, and 11-year-old plantation in Palanpur, Mohali) and one 7-year-old plantation in Yeshwanthpur, Kolar district, Karnataka, India. In each plantation, 10–12 trees were randomly selected for growth strain measurements. The selected trees were measured for girth at breast height, pilodyn penetration and acoustic velocity.

Pilodyn measurement: A 6-joule pilodyn (PILODYN 6 J FOREST) tool was used to measure pilodyn pin penetration at the breast height. For the measurement, a small window was created by removing the bark at the point of measurement and a single pilodyn reading, i.e., the depth of penetration of the flat-nosed pin into the wood was recorded with an accuracy of 1 mm.

Acoustic velocity measurement: Acoustic velocity in standing trees was measured using Fakopp make stress wave timer tool. Two probes were inserted in wood vertically in-line through the bark at an angle of 45° to the trunk surface facing each other. The distance between the probes was 1 m, and the lower probe was inserted at 30 cm above the ground. It was ensured that the transducer probes (needles) penetrate the bark to reach the wood beneath to give the true transit time of stress wave through wood. Accurate transit time measurement requires a stable and firm attachment of the transducers to the wood. The pin-type probe acts as a wave guide and also provides good coupling of the transducer with the wood that allows sound to propagate despite the presence of bark. The start probe was gently tapped with a light hammer (about 100 gm) to induce stress waves in the wood. The arrival time of the stress wave at the receiver (end) probe was recorded with an accuracy of 1 μs. For each tree, five measurements of stress wave transit time were taken. Stress wave velocity (V) was determined using the following equation (Eq. 1).

$$V = \frac{\text{Distance between probes}}{\text{Transit-time}} \tag{1}$$

Growth strain measurement: Longitudinal growth strains were measured using the wire strain gauge method as described by Chauhan and Aggarwal (2011). In this method, a small window of the bark was removed at the breast height to expose the wood surface. The surface moisture present at the wood surface was wiped using cotton. A 350-Ω wire strain gauge (5-mm guage length) was glued to the wood surface using cyanoacrylate-based adhesive. The adhesive was allowed to cure for about 30 min to get effective bonding of the gauge to the wood surface. The lead wires of the strain gauge were connected to the strain meter (TC-32K Tokyo-Sakkai make) in the quarter-bridge configuration, and the initial strain value recorded. Thereafter, wood fibers were slit above and below the strain gauge using a hand drill machine releasing the tensile stress in the fibers. The cut slots were about 20 mm wide and about 20–25 mm deep. The edge of the slot close to the gauge

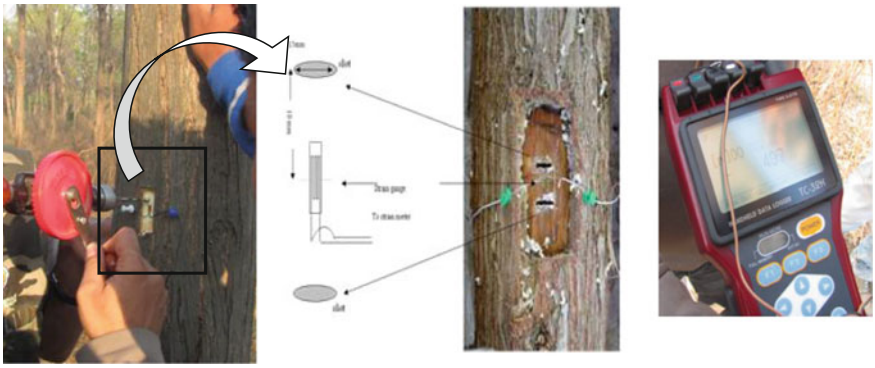

Fig. 1 Strain measurement using wire strain gauge technique

was about 15 mm from the centerline of the gauge. As the slots are made, cut fibers contract longitudinally and expanded transversely. Any strains observed at the wood surface due to stress relaxation are reflected by the strain gauge, and the same was recorded by strain meter. The configuration of holes and strain gauge is shown in Fig. 1.

Results and Discussion

The descriptive statistics of the tree girth, growth strain, pilodyn and acoustic velocity for different locations are given in Table 1. There was a significant variation in girth at breast height (GBH) and growth strain within the plantations. As expected, the girth was lowest for 4-year-old plantation and highest for 11-year-old plantation. On an average, the girth increment of the trees in Punjab was about 7–8 cm per year and about 6 cm/year in Yeshwanthpur plantation of Karnataka. The longitudinal growth strains were varying from 56×10^{-6} to 730×10^{-6} across the locations and age classes. Longitudinal growth strains were lowest in 11-year-old Palanpur plantation of Punjab (272×10^{-6}), and the highest was in 8-year-old Mathewada plantation (371×10^{-6}). However, one-way analysis of variance indicated no significant difference in growth strain levels in all the plantations. It is clearly evident that the growth strain values in this species are nearly uniform irrespective of the significant differences in the age of the trees. Also, the strain values are much lower than 700×10^{-6}, which is considered be the threshold strain beyond which growth stress-related defects like end splitting in logs and warping in timber become prominent (Kubler 1987). This suggests that growth stress may not be of any serious concern in this species.

Pilodyn penetration was found to be the highest in Palanpur plantation and lowest in Dasuya plantation. Higher penetration indicates lower wood basic density. Chauhan and Arunkumar (2014) have shown a strong negative relationship

Table 1 Descriptive statistics of various parameters measured for different plantations

Sl. no.	Location and plantation age	GBH (cm)	Strain (10×10^{-6})	Pilodyn penetration (mm)	Acoustic velocity (km/s)
1	Dasuya (Punjab) (4 years)	36.78[a] (14.33%)	302.30[a] (67.88%)	15.44[a] (9.70%)	3.41[a] (5.48%)
2	Mathewada (Punjab) (8 years)	55.48[b] (13.54%)	371.10[a] (45.08%)	17.72[b] (7.60)	3.42[a] (4.48%)
3	Palanpur (Punjab) (11 years)	70.38[c] (22.80%)	272.60[a] (26.87%)	19.25[c] (8.32%)	3.72[b] (4.78%)
4	Yeshwanthpur (Karnataka) (7 years)	43.65[a] (20.56%)	352.80[a] (57.38%)	15.65[a] (8.25%)	3.99[c] (3.62%)

Value in the parenthesis is coefficient of variation. Values with the same superscript letter are not significantly different at $\alpha = 0.05$

between pilodyn penetration and wood basic density in *M. dubia* with coefficient of determination of 0.77. There was no significant difference in pilodyn penetration for Dasuya plantation of Punjab and Yeshwanthpur plantation from Karnataka. To ascertain the density differences, 5-mm-diameter increment cores were extracted from few selected trees in each plantation and wood basic density was determined from the green volume and over dry weight of core as per the standard formula (Walker 2006). The wood basic density was 400, 383, 370 and 401 kg/m^3 of Dasuya, Mathewada, Palanpur and Yeshwanthpur plantations, respectively. The density values are in the same order as the pilodyn penetration.

The acoustic velocity exhibited very little variation within plantations as evident by low coefficient of variation. Among the plantations, acoustic velocity was ranging from 3.41 to 3.99 km/s and was highest in Yeshwanthpur plantation of Karnataka. Acoustic velocity was same for Dasuya and Mathewada plantation. The acoustic velocity (V) is related to the dynamic modulus of elasticity (DMoE) of wood in the following form (Eq. 2).

$$\text{DMoE} = \rho \times V^2 \tag{2}$$

where ρ is the wood density at the time of velocity measurement. When measured in standing trees, square of acoustic velocity is directly proportional to the modulus of elasticity as the green density of wood remains nearly constant irrespective of differences in basic density (Walker 2006).

Measurement of acoustic velocity in standing trees and correspondingly determination of modulus of elasticity without any destructive sampling provide an opportunity to estimate the growth stress levels in trees. The mean green density of wood was about 850 kg/m^3. The dynamic modulus of elasticity was determined for all the trees using Eq. 2. Growth stress was estimated by multiplying DMoE with

growth strain. The growth stress was varying from 0.5 to 9.00 MPa in the studied trees. Among the locations, the mean growth stress was found to be 2.97, 3.14, 3.78 and 4.43 MPa for Dasuya, Mathewada, Palanpur and Yeshwanthpur plantations, respectively. High stress values for Yeshwanthpur plantation are attributed to high acoustic velocity observed in the trees in this plantation. The variation in acoustic velocity may significantly influence the stress values. The scatter diagram (Fig. 2) shows the relationship between strain and stresses.

Low level of stresses in trees with low MoE of wood may yield high growth strain. Trugilho and Oliveira (2008) estimated growth stresses in *Eucalyptus dunnii* by measuring dynamic MoE in samples extracted from the tree and found to be in the range of 15–25 MPa. The stress values estimated for *M. dubia* are far lower than the reported on eucalyptus species. Similarly, growth stresses were determined for 10-year-old *E. globulus* from dynamic MoE and longitudinal strains (Yang and Ilic 2003). They also observed a large variation in growth strain (CV-62%) and small variation in dynamic MoE (CV-18%) that resulted in a strong linear correlation between growth strain and growth stress.

A Pearson's correlation analysis was performed to examine the interrelationship between various parameters (Table 2). The correlation was carried out for pooled data across locations and age class. There was a positive correlation between girth

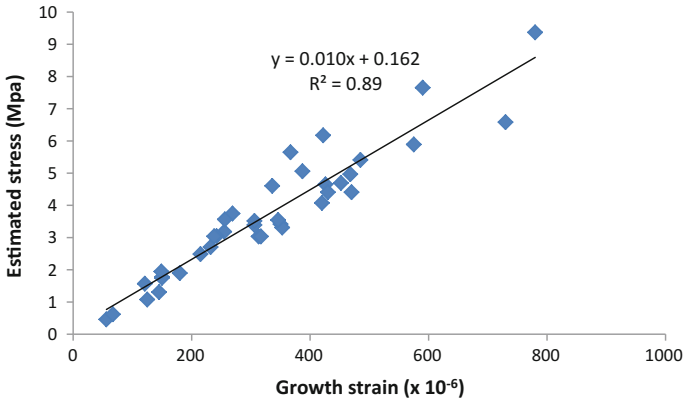

Fig. 2 Relationship between measured strain and estimated stress

Table 2 Correlation between various parameters

Parameter	Girth	Strain	Pilodyn	Acoustic velocity
Girth	1			
Strain	−0.27	1		
Pilodyn	0.55**	−0.05	1	
Acoustic velocity	0.02	0.03	−0.15	1

**Significant at $\alpha = 0.001$

at breast height and pilodyn penetration, indicating a negative association of girth with wood density. Growth strain and acoustic velocity had no relationship with any of the parameters.

The absence of any relationship of growth strains with other wood traits like wood density, acoustic velocity, dynamic MoE and tree girth has also been reported on other species like *Eucalyptus tereticornis* (Chauhan and Aggarwal 2011), 8-year-old *E. nitens* (Chafe 1990), 10-year-old *E. cloeziana* (Muneri et al. 1999) and 4-year-old *E. pilularis* (Muneri and Leggate 2000), *E. nitens* (Chauhan and Walker 2004). The absence of any relationship between pilodyn penetration and acoustic velocity suggests that there may not be any association between these two parameters in case of *M. dubia*. The results are in congruence with the results reported by Chauhan and Arunkumar (2014) on 9-year-old plantation of *M. dubia*.

Conclusions

Low level of growth strain in *M. dubia* in the studied plantation of different age and from different locations implies that the growth stress-related concerns may not be observed in processing and utilization of this species for solid wood applications, provided the density and modulus of elasticity are acceptable for such applications. The absence of any relationship between other wood traits like acoustic velocity (indicator of MoE), pilodyn penetration (indicator of wood density) and GBH (growth indicator) provides an opportunity in tree improvement in this species for improved wood quality traits.

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Immunofluorescence Localization of β-(1-4)-D-Galactan and Xylans in Tension Wood and Normal Wood Fibres of *Leucaena leucocephala*

S. Pramod, Kishore S. Rajput and Karumanchi S. Rao

Abstract The distribution pattern of β-(1-4)-D-galactan and xylans in the fibres of tension wood and normal wood of *Leucaena leucocephala* was investigated using immunofluorescence microscopy. Light microscopic observation of tension wood fibres revealed that they are characterized by the presence of typical non-lignified, tertiary wall layer. LM-5 labelling of normal fibres showed weak labelling from the cell wall, indicating less concentration of β-(1-4)-D-galactans or masking effect due to co-polymerization of other polymers. Both less substituted (LM-10) and highly substituted (LM-11) xylans found to be distributed more in the secondary wall region, while compound middle lamella showed weak labelling. In tension wood fibres, LM-5 labelling revealed strong signals from tertiary wall layer, indicating that gelatinous layer contains more amount of β-(1-4)-D-galactans. The lignified secondary wall and compound middle lamellae showed weak labelling with LM-5. Labelling of xylans with LM-10 showed intense fluorescence signals from the lignified secondary wall, while gelatinous layer showed weak labelling, indicating less concentration of xylans in the gelatinous layer. In conclusion, in addition to the generalized concept that the G-layer in tension wood fibre is rich in cellulose, our results showed that constitution of pectins and xylans is also different compared to the secondary wall of normal wood fibres.

Keywords Immunolabelling · β-(1-4)-D-galactan · Xylan · Tension wood · Normal wood · *L. leucocephala*

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Introduction

Formation of reaction xylem is an important developmental aspect during the growth of trees due to their imperative functions in movements and regulating their form. In angiosperms, the reaction xylem called tension wood is characterized by a modified secondary wall structure of fibres, reduction in size and frequency of vessels and an increased proportion of fibres compared to those of normal wood (Jin and Kwon 2009). Tension wood forms an attractive experimental system for exploring the development and biochemical pathways of secondary wall formation as well as control mechanism of the carbon flux into lignin, cellulose and holocellulose (Kwon 2008). Tension formation has been attributed to growth stress and is demonstrated to be influenced by gravity (Linden 2005) and plant hormones (Pramod et al. 2012). Growth stresses result from the superposition of two kinds of stress: support stress and maturation stress. The former is an elastic response to the increasing load of the wood and shoots supported by the tree, while the latter originates during the process of wood formation (Clair et al. 2006). When the tension wood develops in response to gravitational stimulus, it generates a strong tensile force, which can pull a large inclined stem into a vertical or near vertical position, along the grain of the upper side of the living stem (Yamamoto et al. 2002).

Among the different cell types of angiosperms xylem, fibres are characterized by thick secondary wall that provides the mechanical strength to the stems and branches of the trees. Therefore, changes in the structure and chemical composition of fibres believed to be a major factor determining balancing mechanism of trees. Tension wood is characterized in some species by the fibres with an additional gelatinous layer (G-layer) which is deposited on the lumen side of the cell wall, partly replacing the secondary cell wall. The G-layer consists mainly of highly crystalline cellulose (Daniel et al. 2006), although other components such as hemicelluloses (Nishikubo et al. 2007), arabinogalactan protein (Lafarguette et al. 2004) or lignin (Joseleau et al. 2004) may also be present. The anatomical and biochemical features of tension wood have been demonstrated through extensive studies during past few decades. However, there are few studies on spatial and temporal variation in distribution pattern of cell wall polymers in the fibres of tension wood. Immunolabelling has emerged as a precise tool for understanding the distribution pattern of cell wall polymers due to their high specificity as compared to the conventional histochemical methods. *Leucaena leucocephala* is an evergreen, tropical species, thrives well in wide variety of climates and has considerable commercial importance due to their suitability for production of paper and pulp. Our previous study revealed the anatomical features and biochemical composition of cell wall polymers in tension wood and opposite wood of *L. leucocephala* (Pramod et al. 2013). In the present study, the distribution of two cell wall polymers, namely β-(1-4)-D-galactan and xylans, which are believed to have important interaction with lignin during fibre wall development, has been investigated in tension wood and opposite wood.

Materials and Methods

Plant Materials

Two 5-year-old trees of *L. leucocephala* growing in the premises of Department of Biosciences, Sardar Patel University, Gujarat (India), were harvested in April 2009. Two wood discs having a girth of 25 cm were extracted from the main trunk at approximately 2 m height from the ground level of each tree. The portion of the wood identified as tension wood (TW) due to its eccentric growth on the upper side of the disc was separated from the opposite wood (OW) on the lower side of the discs.

Sample Processing for LR White Embedding

Stem samples after trimming to 2 × 5 mm size pieces were fixed in a mixture of 0.1% glutaraldehyde and 4% paraformaldehyde in 50 mM sodium cacodylate buffer for 4 h at room temperature. After washing in buffer, tissues were dehydrated in graded series of ethanol (30–95%, 15 min each, pure ethanol × 3, each for 20 min.). Following infiltration with a mixture of LR white and ethanol (V/V) 1:3, 1:1, 3:1 and pure resin (4 days in each solution), the tissues were dropped into gelatin capsules and capsules were filled with fresh LR white and the capped capsules were left in oven at 60 °C for polymerization (2 days). During infiltration, tissue samples were kept under vacuum and rotator during day and night, respectively.

Light Microscopy

Semi-thin sections (1–2 μm thick) were taken from the LR white-embedded samples by using ultramicrotome (RMC Powertome X, USA) with a diamond knife. The sections were stained with 0.05% toluidine blue O (Berlyn and Mikshe 1976). Observation and imaging were recorded using a Leica DM200 fluorescence microscope with a Canon digital camera (DM 150).

Immunofluorescence Microscopy

Immunolabelling was carried out according to the method described by Kim et al. (2010). Transverse sections of 1-μm thickness were prepared from LR white-embedded blocks and mounted on formvar-coated slides and treated with

50 mM glycine/phosphate-buffered saline (PBS) solution for 15 min. Sections were washed with PBS buffer and suspended in blocking buffer (PBS) containing 3% skim milk for 30 min at room temperature. Sections were incubated in monoclonal antibodies (Plant Probes, UK; 1:100 dilution in PBS buffer) specific for β(1-4)-D-galactans (LM-5) and xylans (LM-10, LM-11) for 2 days at 4 °C. After washing in three changes in PBS buffer for 10 min each, sections were incubated with anti-mouse IgG Alexa Fluor 568 (Invitrogen, USA; 1:1000 dilutions in PBS buffer) for 2 h at 35 °C. After washing in PBS buffer for 3 times (10 min each) and mounted in Fluoroshield (Sigma, Germany) on a clean glass slide, sections were examined under a fluorescence microscope (Ziess, Axio Observor Z1, Germany) fitted with a filter combination of 570-nm exciter and a 603-nm emitter. Control sections were incubated without primary antibody treatment.

Results

Structure of Tension Wood and Normal Wood Fibres

The light microscopic observation of semi-thin sections stained with toluidine blue 'O' revealed that the tension wood fibres are characterized by a moderately thick, non-lignified tertiary wall (gelatinous layer) which replaces major part of inner secondary wall layer found in the normal wood fibres (Fig. 1a, b).

Immunofluorescence Localization of β-(1-4)-D-Galactan (LM-5) and Xylans (LM-10 and LM-11) in Normal and Tension Wood Fibres

LM-5 labelling of normal wood fibres showed weak labelling from both middle lamellae and secondary wall regions of cell wall, indicating the relatively less β-1,3-galactans or masking effect of other polymers binding to them (Fig. 1c). Less substituted xylans labelled with LM-10 revealed strong fluorescence signals from the secondary walls, while weak labelling was evident from cell corners and compound middle lamellae (Fig. 1d). Highly substituted xylans (LM-11) also showed more or less similar pattern of labelling as shown by LM-10; however, the intensity of labelling was stronger at the corner region of secondary wall (Fig. 1e).

The relatively high concentration of β-1,3-galactans in the gelatinous layer was evident from strong fluorescence signals of the inner tertiary wall labelled with LM-5 (Fig. 1f). The compound middle lamella and lignified secondary wall region showed weak labelling (Fig. 1f). LM-10 labelling of tension wood fibres revealed strong signals from lignified secondary wall region, while weak labelling was observed from the gelatinous layer and compound middle lamellae regions

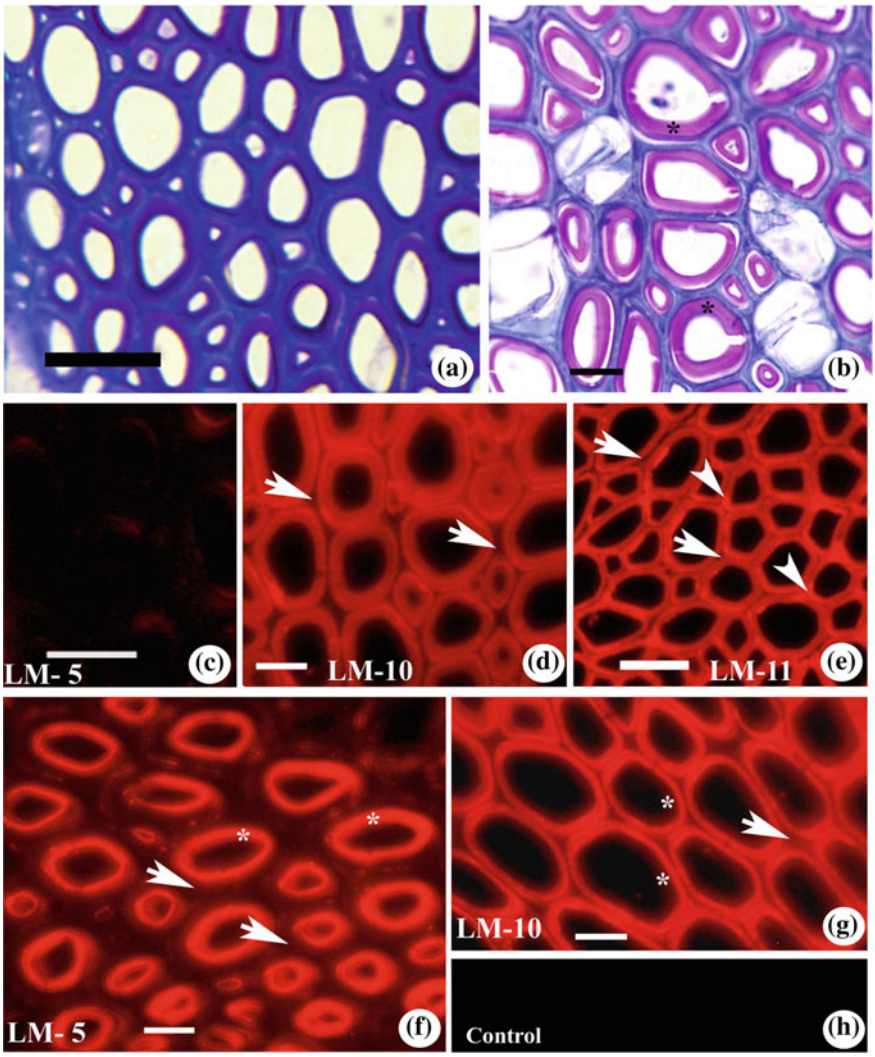

Fig. 1 Light (**a**, **b**) and immunofluorescence images from the normal (**a**, **c**–**e**) and tension wood (**b**, **f**–**h**) fibres in *L. leucocephala.* **a** Normal wood fibres showing thick secondary wall. **b** Gelatinous fibres showing thick, non-lignified, gelatinous layer (asterisks). **c** Weak labelling of normal fibres for β-1,3-galactans. **d** LM-10 labelling showing distribution of less substituted xylans in the secondary wall. *Arrows* indicate the weak labelling in the compound middle lamellae region. **e** Weak labelling from the compound middle lamellae region with LM-11 (*arrows*). *Arrowheads* indicate the strong labelling from the corner region of secondary wall. **f** Detection of labelling of β-1,3-galactans from the gelatinous layer (*asterisk*). *Arrows* indicate the absence of fluorescence from the lignified secondary wall and compound middle lamellae region. **g** Absence of LM-10 labelling from the gelatinous layer. *Arrow* indicates the compound middle lamellae showing weak labelling. Note intense labelling in the lignified secondary wall region of G-fibres. **h** Control section omitted with primary antibody. *Scale bar* **a** = 50 μm; **b**–**g** = 25 μm

(Fig. 1g). Control sections omitted with primary antibody treatment showed absence of fluorescence signals (Fig. 1h).

Discussion

The chemistry of gelatinous layer is considered to be of special interest due to its non-lignified nature in majority of plants. The reduction in total lignin content in tension wood can be due to the purely cellulosic thick gelatinous layer which replaces the inner lignified wall layers in G-fibres, which is reported to be a common feature in tension wood (Pilate et al. 2004). High α-cellulose has been reported in the tension wood of many species (Fujii et al. 1982; Baba et al. 1996), and it is mainly attributed to the presence of non-lignified, cellulosic gelatinous layer in the tension wood fibres. Formation of non-lignified gelatinous layer is typical to tension wood fibres found in the reaction xylem of majority of angiosperms. On the other hand, there is also evidence that angiosperms exhibit reaction xylem fibres lacking the G-layer (Kucera and Philipson 1977). In our previous study, we noticed the occurrence of thick gelatinous layer in the tension wood fibres of *L. leucocephala* (Pramod et al. 2013). The interaction behaviour of polysaccharide constituents with lignin have great scientific value to understand the biology of cell wall strengthening during evolution of higher plants. The present study showed the distribution pattern of two cell wall polymers (β-1,3-galactans and xylans) in the tension and normal wood fibres of *L. leucocephala*. They are the constituents of hemicelluloses and pectins in plant cell wall.

Previous studies using immunolocalization technique also suggest a close relationship between galactan and increased lignification in softwood tracheids (Schmitt et al. 2006; Altaner et al. 2010; Mast et al. 2009; Donaldson and Knox 2012). Recent studies have also shown that lignification is controlled by other polysaccharides such as manans and xylans (Kim et al. 2010, 2012) through chemical bonding with lignin to form lignin–carbohydrate complexes (Tenkanen et al. 1999; Barakat et al. 2007). The present study shows more amount of β-1,4-galactans in the non-lignified G-layer. The distribution pattern of β-1,4-galactans in the TW fibres of poplar was restricted to the interface between the G-layer and adjacent secondary wall, suggesting that it may play an important role in cross-linking the G-layer and secondary wall of G-fibres (Arend 2008). On the contrary, our results showed uniform distribution of β-1,4-galactans throughout the G-layer of fibres in tension wood fibres of *Leucaena.* Similar pattern of labelling was also reported in the gelatinous fibres in the *Acacia* roots (Pramod et al. 2014). This suggest the possibility of interspecific variation in spatial and temporal distribution of β-1,4-galactans in the G-fibres of trees growing in tropical and temperate conditions.

β-1,4-galactans are closely associated with initiation and extension of lignification as suggested by Donaldson and Knox (2012). However, our results showed a weak labelling from highly lignified compound middle lamellae region of both

normal and tension wood fibres. The weak labelling of β-1,4-galactans in the lignified secondary wall regions of normal and tension wood fibres suggests the possibility of either a weak interaction between β-1,4-galactans and lignin or masking effect of lignin on labelling. However, more studies using delignification experiments are required to confirm this hypothesis.

Xylan distribution in TW fibres of *Leucaena* was mainly confined to the secondary walls, while no labelling was found in the G-layer, indicating a different chemical composition of tertiary wall compared to secondary wall. Kim and Daniel (2012) also noticed a similar pattern of xylan distribution with LM-10 labelling in TW fibres of poplar and suggested that secondary wall of TW may not be much affected by tension stress even though xylan is the major hemicelluloses in hard wood cell walls. These authors have also noticed a weak labelling with LM-11 from gelatinous layer indicating the presence of highly substituted xylans in the tertiary wall and suggested that xylans may not be completely absent in gelatinous layer. The lignified secondary wall regions of normal fibres also showed strong labelling with both LM-10 and LM-11. In the mature fibres of poplar, LM-10 labelling showed stronger xylan (ls ACG Xs) localization in outer SW than in inner layer, while LM-11 revealed uniform distribution of hs ACG Xs in the whole SW, indicating heterogeneous composition of xylans in the fibre wall (Kim et al. 2012). In *Leucaena*, on the contrary, LM-11 labelling was intense from the outer part of SW layer, especially corner region, while LM-10 labelling was uniform throughout SW. This suggests that interspecific variation can exist in distribution pattern of xylans in the same cell type of trees growing in temperate and tropical climates. According to Awano et al. (2002), this heterogeneity in xylan distribution could be associated with intussusceptional or appositional mode of deposition of less substituted and highly substituted xylans.

In conclusion, the present study revealed the distribution pattern of β-1,3 galactans and xylans in the normal and tension wood fibres of *L. leucocephala*. In addition to the generalized concept that the G-layer in tension wood fibre is rich in cellulose, our results showed that constitution of pectins and xylans is also different compared to the secondary wall of normal wood fibres. Our results also suggest significant difference in spatial and temporal distribution pattern of these cell wall polymers. This distribution pattern could have a relationship with variation in lignification pattern between secondary and tertiary (G-layer) wall layers of fibres.

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Comparative Wood Anatomy of Four *Artocarpus* Species of North East India with Reference to Their Identification

M.K. Singh, C.L. Sharma and M. Sharma

Abstract The present study was carried out in four *Artocarpus* species namely *A. chaplasha*, *A. heterophyllus*, *A. lakoocha* and *A. nitidus* collected from the forests of Assam and Mizoram, NE India. The main objective of study was to evaluate variation in anatomical features among species both qualitatively and quantitatively. All selected species had diffuse-porous wood with indistinct rings, vessels solitary or in radial multiple of 2–3, simple perforation plate, intervessel pits alternate, laticifers in the form of black streaks among fibres and rays, vessel ray pits similar to intervessel pits, vasicentric and lozenge aliform parenchyma. In addition, presence of ray flecks and latex ducts in rays of *A. chaplasha,* gelatinous fibres in *A. chaplasha* and *A. nitidus*, sheath cells in *A. lakoocha* and *A. nitidus*, axial parenchyma confluent and winged aliform in *A. heterophyllus* were observed. Tissue proportions among species showed maximum percentage of fibres in *A. nitidus*, vessel and rays in *A. heterophyllus* and parenchyma in *A. nitidus*, respectively. Maximum vessel frequency was observed in *A. chaplasha*, while maximum rays were recorded in *A. nitidus* and *A. lakoocha*. Quantitative characters showed statistically non-significant differences within species, while these characters exhibited significant differences among species. Correlation for most of the anatomical characteristics was too weak to be significant, but the correlation of specific gravity was positively significant with ray width and negatively significant with fibre diameter.

Keywords *Artocarpus* · Anatomical characteristics · Diffuse-porous · Axial parenchyma · Tissue proportion

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Introduction

The family Moraceae comprises of 37 genera and 1050 species (Berg et al. 2006) and has mostly trees or shrubs. It is widely distributed in tropical and sub-tropical regions of the world and a few in temperate regions (Raturi et al. 2001). It consists of five tribes namely Moreae, Artocarpeae, Dorstenieae, Castilleae and Ficeae (Berg 2001). Of these, Artocarpeae also known as breadfruit tribe, is characterised by inflorescence architecture (simple spike to complex globose heads), reduction of stamen number, peltate interfloral bracts, vitreous silica and straight filaments. *Artocarpus*, *Hullettia*, *Paratocarpus*, *Prainea*, *Batocarpus* and *Clarisia* are important genera of this tribe (Datwyler and Weiblen 2004; Clement and Weiblen 2009). *Artocarpus* J.R. Forst. is the largest genus in the tribe and third largest genus in the family Moraceae after *Ficus* and *Dorstenia*. It has about 45 species which are distributed in Indo-Malayan region of South-East Asia and about 7–8 species are known to occur in India (Raturi et al. 2001).

A limited information is available on anatomical studies of *Artocarpus* (Gamble 1922; Purkayastha 1996). Topper and Koek Noorman (1980) reported presence of laticifers in ray tissue and fibre tissue in number of *Artocarpus* species, while Anoop et al. (2011) carried out study on variation in bark thickness, heartwood content, fibre morphology, vessel and ray morphology in *A. heterophyllus* Lam. and *A. hirsutus* to understand radial variation in wood anatomical properties within trees. Raturi et al. (2001) described the gross structure of *A. chama*, *A. gomezianus*, *A. heterophyllus*, *A. hirsutus* and *A. lakoocha* collected from other parts of India, Bangladesh and Myanmar. There is no report on anatomy of *A. nitidus* in the available literature. The present study is an attempt to describe the wood anatomy of four species of *Artocarpus* of NE India with an aim to evaluate variation in anatomical characteristics within and among species both qualitatively and quantitatively.

Materials and Methods

The samples of *A. chaplasha* Roxb., *A. heterophyllus* Lam., *A. lakoocha* Roxb. and *A. nitidus* Trec. were collected from forests of Assam and Mizoram as listed in Table 1. Eight trees with straight bole and uniform crown were selected for each species, and wood samples of size 5 cm^3 were taken at breast height. Each wooden block was cut into 2 cm^3 size and fixed in FAA for 24–48 h, after which they were preserved in 50% alcohol. Cross, radial longitudinal and tangential longitudinal sections were cut with the help of a sliding microtome. Permanent slides were prepared by following standard procedure. Small radial slivers of different *Artocarpus* species were treated with Franklin's solution for maceration to measure fibre and vessel length. Temporary slides were prepared by using 50% glycerol, and a random sample of 50 fibres and 50 vessels were measured from each sample with

Table 1 List of selected *Artocarpus* species

Species name	Average height (m)	Average diameter at breast height (cm)	Coordinates	Locality
A. chaplasha	14	43	27° 23′N, 95° 36′E 24° 01′N, 92° 54′E	Digboi reserve, Assam, Darlawn, Mizoram
A. heterophyllus	13	33	27° 21′N, 95° 50′E 24° 01′N, 92° 54′E	Kothabasti reserve, Assam, Darlawn, Mizoram
A. lakoocha	14	39	27° 36′N, 94° 35′E 24° 01′N, 92° 54′E	Poba reserve, Assam, Darlawn, Mizoram
A. nitidus	15	36	24° 01′N, 92° 54′E	Dampui, Mizoram

the help of an ocular micrometre at 40×. Twenty-five counts were taken for parameters like vessel diameter, fibre diameter, fibre wall thickness, ray height, and ray width, and 10 fields were selected on cross section to determine tissue proportion, vessel frequency and number of rays per mm. The data was analysed by software SPSS 16 and Microsoft excel 2007. The terminology and measurements were taken according to IAWA Committee (1989). Wood specific gravity was determined by water displacement method.

Results and Discussion

Growth ring boundaries indistinct with diffuse porous wood. Vessels circular or oval in outline, solitary or in radial multiple of 2–3, simple perforation plate, intervessel pits alternate and vessel ray pits similar to intervessel pits in size and shape (Fig. 1a–e). Quantitative anatomical features are summarised in Table 2. Mean vessel length, vessel diameter and vessel frequency vary from 284.4 ± 82.61 µm (*A. nitidus*) to 350.5 ± 102.75 µm (*A. chaplasha*), 213 ± 76.80 µm (*A. chaplasha*) to 277.92 ± 93.82 µm (*A. nitidus*). Range of vessel frequency is 4–5 per mm^2 (Table 2). Vessel percentage varies from 12% (*A. nitidus*) to 26% (*A. heterophyllus*) (Fig. 2).

Fibres are square or polygonal in outline and constitute the ground tissue. Mean fibre length, fibre diameter and fibre wall thickness range from 1193.73 ± 326.71 µm (*A. heterophyllus*) to 1425.0 ± 338.70 µm (*A. lakoocha*), 165.57 ± 70.11 µm (*A. nitidus*) to 209.3 ± 59.92 µm (*A. chaplasha*) and 3.21 ± 2.34 µm (*A. heterophyllus*) to 4.90 ± 2.43 µm (*A. nitidus*) respectively. Septate fibres present except in *A. heterophyllus*. Fibre percentage varies from 44% (*A. chaplasha*) to 50% (*A. lakoocha*) (Fig. 2). Laticifers in the form of black streaks present among fibres in *A. heterophyllus* and *A. nitidus* (Fig. 1n).

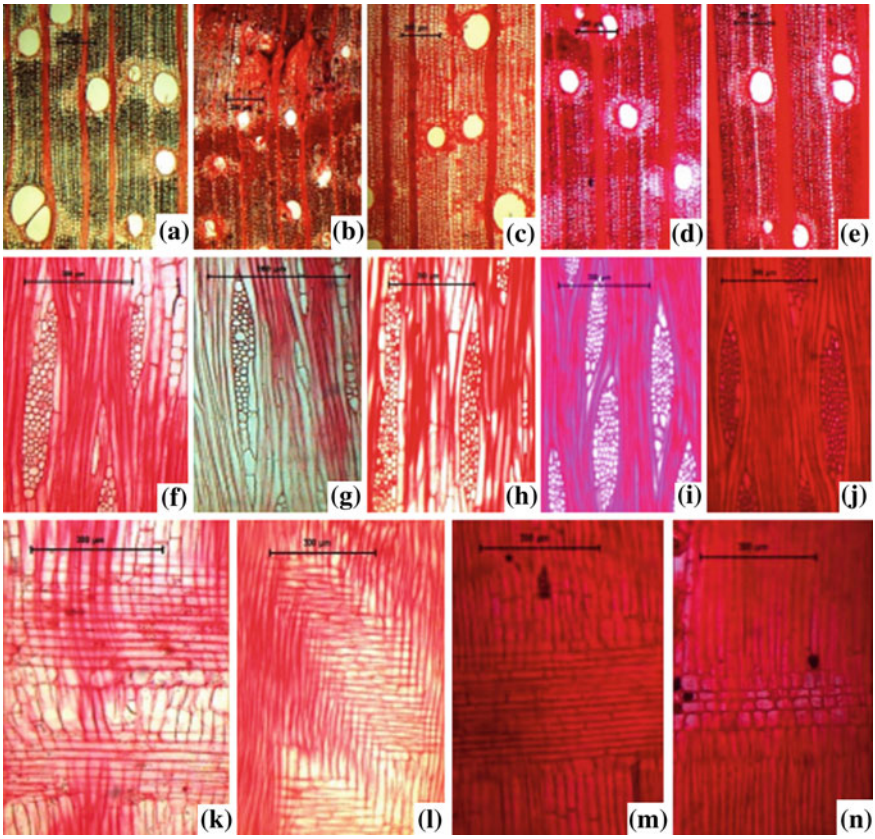

Fig. 1 **a–e** Cross sections: diffuse-porous wood; vessels mostly solitary and in radial multiple of 2 in *A. chaplasha* (**a**) and *A. nitidus* (**e**); lozenge aliform and confluent parenchyma in *A. chaplasha* (**a**, **b**); lozenge aliform and vasicentric parenchyma in *A. heterophyllus* (**c**); lozenge aliform parenchyma in *A. lakoocha* and *A. nitidus* (**d**, **e**); ray flecks in *A. chaplasha* (**b**). **f–j** Tangential longitudinal sections: Biseriate, multiseriate rays and radial latex ducts in *A. chaplasha* (**f**, **g**); multiseriate rays and parenchyma strand in *A. heterophyllus* (**h**); multiseriate rays and sheath cells in *A. lakoocha* and *A. nitidus* (**i**, **j**). **k–n** Radial longitudinal sections: heterocellular rays comprising of body ray cells procumbent with marginal rows of square/upright cells in *A. chaplasha* (**k**), *A. lakoocha* and *A. nitidus* (**m**, **n**); homocellular rays comprising of procumbent cells in *A. heterophyllus* (**l**); laticifers in the form of dark streaks present among fibres in *A. lakoocha* (**m**) and in square/upright cells in *A. nitidus* (**n**)

Lozenge aliform parenchyma is present in all selected species. In addition, vasicentric parenchyma in *A. lakoocha* and *A. heterophyllus* and confluent parenchyma in *A. chaplasha* and *A. lakoocha* are observed (Fig. 1a, d). Parenchyma strands are 2–4 celled. Parenchyma percentage varies from 13% (*A. chaplasha*) to 21% (*A. lakoocha*) (Fig. 2).

Rays are uniseriate, biseriate and multiseriate in all species (Fig. 1f–j). Rays are heterocellular and composed of procumbent body cells with mostly two rows of

Table 2 Dimensions of xylem elements in *Artocarpus* species

Features	*A. chaplasha* range (µm) (Mean ± SD)	*A. heterophyllus* range (µm) (Mean ± SD)	*A. lakoocha* range (µm) (Mean ± SD)	*A. nitidus* range (µm) (Mean ± SD)
Vessel length	128.2–641 (350.5 ± 102.7)	128.2–615.3 (318.9 ± 92.0)	153.8–666.6 (345.1 ± 102.7)	128.2–512.8 (284.4 ± 82.6)
Vessel diameter	78.1–448.0 (213 ± 76.8)	46.8–797.1 (215.3 ± 163.9)	78.1–396 (213.7 ± 66.5)	151.0–609.5 (277.7 ± 93.8)
Vessel frequency	2–21 (5.3 ± 3.6)	2–16 (4.7 ± 2.5)	2–12 (4.0 ± 1.8)	2–7 (4.2 ± 1.3)
Intervessel pits	5.2–18.2 (9.8 ± 2.3)	5.2–18.2 (10.0 ± 2.5)	5.2–20.8 (9.9 ± 2.5)	5.2 ± 13 (8.5 ± 1.8)
Fibre length	538.4–2487.1 (1390.7 ± 342.)	461.5–2051.2 (1193.7 ± 326.7)	589.7–2538 (1425 ± 338.7)	641–2564 (1321.3 ± 347.5)
Fibre diameter	105.2–368.3 (209.3 ± 59.9)	105.2–394.6 (204.9 ± 184.1)	52.6–394.7 (186.9 ± 68.3)	52.6–420.9 (165.5 ± 70.1)
Fibre wall thickness	1.3–10.4 (3.8 ± 2.0)	1.3–11.7 (3.2 ± 2.3)	1.3–11.7 (4.5 ± 2.2)	1.3–11.7 (4.9 ± 2.4)
Ray height	166.7–104.2 (496.8 ± 171.7)	270.9–1000.3 (570.1 ± 152.4)	156.3–1407 (583.4 ± 204)	197.9–937.8 (599.0 ± 144.0)
Ray width	31.2–114.6 (58.8 ± 14.0)	41.6–135.4 (67.4 ± 15.5)	20.8–114.6 (60.6 ± 19.6)	41.6–93.7 (70.9 ± 13.2)

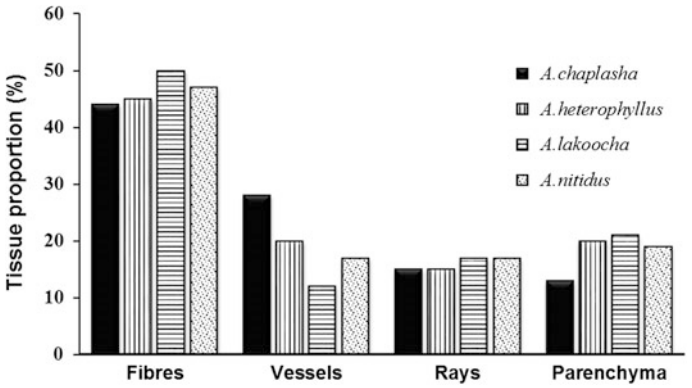

Fig. 2 Tissue proportion of selected species of *Artocarpus*

upright/square marginal cells. Homocellular rays are recorded in few samples of *A. heterophyllus* (Fig. 2k–n). Ray height and ray width vary from 496.83 ± 171.75 µm to 58.87 ± 14.04 µm (*A. chaplasha*) to 599.08 ± 144.02 µm and 70.99 ± 13.23 µm (*A. nitidus*) respectively. Rays per mm are 4 (*A. chaplasha*) to 6 (*A. heterophyllus*). Rays percentage is recorded 15% in *A. chaplasha* and *A.*

heterophyllus, while *A. lakoocha* and *A. nitidus* show 17%. Ray flecks and radial latex ducts in rays of *A. chaplasha* (Fig. 1b, g), sheath cells in rays of *A. lakoocha* and *A. nitidus* (Fig. 1j) are recorded. Also laticifers in the form of black streaks are recorded in square/upright cells of *A. lakoocha*.

The present study shows that the wood of all selected species have common qualitative characters like indistinct growth rings, diffuse-porous wood, solitary vessel, simple perforation plate, vessel ray pitting, heterocellular ray and lozenge aliform parenchyma. These features are in agreement with the findings of Raturi et al. (2001) and Purkayastha (1996). Since these features are present in all species, therefore, these can be considered as characteristics of genus *Artocarpus*. Despite these, other features like presence of vasicentric parenchyma in *A. lakoocha* and *A. heterophyllus*, confluent parenchyma in *A. chaplasha* and *A. lakoocha*, absence of septate fibres in *A. heterophyllus*, radial latex ducts and ray flecks in *A. chaplasha*, sheath cells in *A. lakoocha* and *A. nitidus* have been reported first time and can be

Table 3 Analysis of variance for selected parameters among selected species of *Artocarpus*

Source	Dependent variable	Type III sum of squares	df	Mean square	F
Species	Fibre length	4.203E7	3	1.401E7	38.197**
	Fibre wall thickness	25.924	3	8.641	15.056**
	Fibre diameter	31,912.233	3	10,637.411	31.169**
	Ray height	6,815,036.196	3	2,271,678.732	34.411**
	Ray width	7.218	3	2.406	5.443**
	Vessel length	898,130.850	3	299,376.950	36.579**
	Vessel diameter	8731.198	3	2910.399	40.126**
Replication	Fibre length	5,390,032.479	7	77,0004.640	2.099^{ns}
	Fibre wall thickness	3.186	7	0.455	0.793^{ns}
	Fibre diameter	5132.835	7	733.262	2.149^{ns}
	Ray height	1,004,294.230	7	143,470.604	2.173^{ns}
	Ray width	1.011	7	0.144	0.327^{ns}
	Vessel length	115,648.175	7	16,521.168	2.019^{ns}
	Vessel diameter	685.082	7	97.869	1.349^{ns}
Error	Fibre length	7,702,659.011	21	366,793.286	
	Fibre wall thickness	12.053	21	0.574	
	Fibre diameter	7166.906	21	341.281	
	Ray height	1,386,355.631	21	66,016.935	
	Ray width	9.282	21	0.442	
	Vessel length	171,872.492	21	8184.404	
	Vessel diameter	1523.143	21	72.531	

ns Non-significant

**Significant at $P < 0.01$ level

used to differentiate these four species. Ray flecks were observed in few samples of *A. chaplasha*. The formation of ray flecks may be attributed to localised injury of cambium (Carlquist 2001) and was also reported by Purkayastha (1996) in the same species. Topper and Koek-Noorman (1980) reported radial latex ducts in *A. lakoocha* and *A. nitidus*. On the other hand, the laticifers in the form of dark streaks among fibres in *A. heterophyllus* and *A. nitidus* and radial latex ducts only in *A. chaplasha* are reported in the present study.

Analysis of variance was carried out in quantitative anatomical characters to see the variation within and among species. The results, presented in Table 3, indicate statistically non-significant differences within species and statistically significant differences among species. It indicates that selected wood element dimensions do not have any variation within species. But *Artocarpus* species can be differentiated on the basis of significant quantitative variation in wood element dimensions.

Correlation among different dimensions of wood elements and their relationship with specific gravity is presented in Table 4. The relationship among most of the parameters was too weak to be significant. Fibre length exhibited positive and significant correlation with fibre diameter and vessel length. Specific gravity was positively and significantly correlated with ray width, but negatively and significantly correlated with fibre diameter. It indicates that most of the anatomical parameters do not influence each other, but some fibre characteristics affect specific gravity. The present results corroborate the findings of other workers (Pande et al. 2005; Sharma et al. 2011a, b, 2014; Singh et al. 2013) in other hardwood species.

Table 4 Pearson correlation among parameters of wood elements among selected species of *Artocarpus*

Parameters	FL	FD	FWT	VL	VD	RH	RW	SG
Fibre length (FL)	1	0.514*	0.306	0.516*	0.104	−0.204	−0.487*	−0.460
Fibre diameter (FD)		1	0.434	0.383	−0.154	0.135	−0.252	−0.629**
Fibre wall thickness (FWT)			1	0.155	0.570*	0.284	0.113	0.030
Vessel length (VL)				1	−0.201	−0.095	−0.443	−0.315
Vessel diameter (VD)					1	0.192	0.078	0.214
Ray height (RH)						1	0.094	0.128
Ray width (RW)							1	0.508*
Specific gravity (SG)								1

*Significant at 0.05 level
**Significant at the 0.01 level

Key for identification of *Artocarpus* species is given below:

1. Heterocellular rays and septate fibre present————————2
   Heterocellular rays and septate fibre absent————————*A. heterophyllus*
2. Laticifers in the form of dark streaks among fibres present————————*A. nitidus*
   Laticifers in the form of dark streaks among fibres absent————————3
3. Sheath cells present, both ray flecks and radial latex duct absent————————*A. lakoocha*
   Sheath cells present, both ray flecks and radial latex duct present————————*A. chaplasha.*

Conclusions

All the *Artocarpus* species have common features like diffuse-porous wood, vessel solitary or in radial multiple of 2–3, simple perforation plate, alternate intervessel pits, vessel ray pits similar to intervessel pits in shape and size, heterocellular ray and lozenge aliform parenchyma. Septate fibres are present in all selected species except *A. heterophyllus*. Ray flecks and radial latex ducts are present only in *A. chaplasha*, while, in other species, laticifers in the form of black streaks among fibres (*A. heterophyllus* and *A. nitidus*) and in upright/square cells of rays (*A. lakoocha*) are present. Quantitative anatomical characters like fibre length, fibre diameter, fibre wall thickness, vessel length, vessel diameter, ray height, ray width show statistically non-significant variation within species and statistically significant variation among species. Wood specific gravity is positively and significantly correlated with ray width. Identification key is prepared on the basis of qualitative anatomical features for separation of species.

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Wood Anatomy of Some Members of Family Fagaceae from North-East India

M. Sharma, W. Shylla and C.L. Sharma

Abstract Wood anatomical characteristics of *Castanopsis kurzii*, *Lithocarpus fenestrata*, *Quercus griffithii, Quercus lamellosa* and *Quercus spicata* were studied to determine intergeneric and intrageneric similarities and dissimilarities. Vasicentric tracheids, apotracheal diffuse, diffuse-in-aggregate axial parenchyma were the common characteristics among genera. *Lithocarpus* differed from *Quercus* and *Castanopsis* by presence of exclusively solitary vessels with diffuse porous wood and indistinct growth rings, long vessels with helical thickenings restricted to their tails, scalariform perforation plate, scalariform and opposite intervessel pits, vessel-ray pits with much reduced borders to apparently simple and scalariform, maximum vessel frequency (32/mm^2) and vessel percentage (41%), heterocellular and biseriate rays. *Castanopsis* was highly similar to *Quercus* except the absence of aggregate rays. All three selected species of *Quercus* were closely related and were characterized by the presence of simple perforation plate, opposite intervessel pits, vessel-ray pits with much reduced border pits to apparently simple and vertical (palisade), gelatinous fibres and aggregate rays. However, *Q. griffithii* could be separated from *Q. lamellosa* and *Q. spicata* by diagonally arranged vessels, homocellular rays and chambered crystalliferous axial parenchyma. *Q. spicata* differed from other species by the absence of gelatinous fibres and the presence of axial parenchyma in the form of bands alternating with fibres.

Keywords Fagaceae · Vasicentric tracheids · Gelatinous fibres · Aggregate rays · Tissue proportion

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Introduction

Fagaceae is one of the most important families comprising of trees and shrubs and is an important source of timber, cork, tannins and nuts. It includes 9 genera and about 1000 species of evergreen or deciduous trees or shrubs which are confined in temperate seasonally dry regions of Northern hemisphere with a centre of diversity found in tropical South-East Asia (Manos et al. 2001). In India, it is represented by four genera namely *Castanea* Miller, *Castanopsis* (D. Don) Spach., *Lithocarpus* Blume., and *Quercus* L. Of these, *Castanea* is a native of Southern Europe and is cultivated for its fruits in Shillong, Meghalaya. There are about 6 species of *Castanopsis*, 12 species of *Lithocarpus* and 20 species of *Quercus* in India and species of *Castanopsis* and *Lithocarpus* are reported to occur in Eastern Himalayas (Haridasan and Rao 1987; Purkayastha 1996).

A perusal of the literature reveals that wood anatomical description of members of family Fagaceae is scattered. Gamble (1922) described gross anatomy of 16 species of *Quercus* and 3 species of *Castanopsis*. He placed *Lithocarpus* as sub-genus under tribe Quericineae. Pearson and Brown (1932) studied gross features and minute anatomy of six *Quercus* species and two *Castanopsis* species of India. They recorded the presence of broad rays in Castanopsis. Metcalfe and Chalk (1950) studied wood anatomy of *Quercus* and *Castanopsis* with other genera of Fagaceae, while Shimaji (1962) studied ontogeny trend in ray development of this family. Rao et al. (1991) reported that broad rays may or may not be present in *Castanopsis* and *Lithocarpus* species but are regularly present in *Quercus semiserrata*. Noshiro and Sasaki (2011) identified species of *Quercus* and *Lithocarpus* by size, frequency of vessels and ray structure.

Though a number of members of family Fagaceae occur in North-East India, but a limited information is available on the anatomy of members of this family. Sharma et al. (2011a, b) studied wood anatomy of few species of *Quercus* and *Castanopsis* of Meghalaya in detail to see interspecies variation in anatomical characteristics. The present study is an attempt to study wood anatomical characteristics of some members of Fagaceae in detail with the aim of determining intergeneric and intrageneric similarities and dissimilarities.

Materials and Methods

Wood samples of *Castanopsis kurzii* (Hance) S.N. Biswas, *Lithocarpus fenestrata* (Roxb.) Rheder, *Quercus griffithii* Hook. & Thom., *Quercus lamellosa* Sm. in Rees and *Quercus spicata* Smith were collected from forests of Arunachal Pradesh, Nagaland and Meghalaya (Table 1). The wooden blocks of 5 cm^3 were taken at breast height from mature straight trees with well-developed crown. The samples were further cut in 2 cm^3 size and fixed in FAA for 24–48 h and were then preserved in 50% alcohol. Sections were cut in three planes namely cross section

Table 1 List of selected species of family Fagaceae

S. no.	Species	Latitude (°N)	Longitude (°E)	Locality
1.	*C. kurzii*	25° 28′	92° 13′	Mukhla, Meghalaya
2.	*L. fenestrata*	25° 28′	91° 21′	Nongsing, Meghalaya
3.	*Q. griffithii*	25° 30′	92° 10′	Mukhla, Meghalaya
4.	*Q. lamellosa*	27° 06′	92° 16′	Shergaon, Arunachal
5.	*Q. spicata*	25° 30′	92° 10′	Mukhla, Meghalaya
		25° 56′	94° 14′	Tseminya, Nagaland

(C. S.), tangential longitudinal section (T. L. S.) and radial longitudinal section (R. L. S.) with the help of a sliding microtome. Permanent slides were prepared by staining as per standard procedure. Anatomical characteristics on different sections were photographed with the help of Leica DFC camera attached to the Leitz Laborlux research microscope. Thin slivers of wood from radial sides of each block were taken in test tubes and macerated with Franklin's solution by keeping them in oven at 60 °C for 24 h. Temporary slides were made by using 50% glycerol for measuring fibre length and vessel length. For each tree, a random sample of 50 fibres and 50 vessels were measured. Ten fields were selected for vessel frequency and tissue proportion, while 30 counts were taken for fibre diameter, fibre wall thickness, ray height, ray width and vessel diameter. The anatomical features of selected species were studied according to list of microscopic features for hardwood identification of IAWA Committee (1989). Statistical analysis was carried out using MS Excel 7.

Results and Discussion

Anatomical structure (cross sections and tangential longitudinal sections) of different species is shown in Figs. 1 and 2. The quantitative anatomical characters of selected species are present in Table 2.

Growth Rings—Distinct in *C. lanceofolia, Q. griffithii* and *Q. spicata*, usually marked by radially flattened thick-walled fibres and small sized vessels. Indistinct in *L. fenestrata* and *Q. lamellosa* (Fig. 1a–h).

Vessels—Semi-ring porous to ring porous in *C. kurzii, Q. griffithii*, *Q. spicata* and diffuse porous in *L. fenestrata* and *Q. lamellosa*. Vessels are circular to oval in outline, solitary (*L. fenestrata*), radially arranged (*Q. lamellosa*, *Q. spicata*, *C. kurzii*) and diagonal, dendritic in *Q. griffithii* (Fig. 1a–h). Perforation plates simple except in *L. fenestrata*. In *L. fenestrata* only, vessels have long tails with spiral thickenings (Fig. 2e). Intervessel pits opposite (*Q. griffithii*, *Q. lamellosa*), alternate (*Q. spicata* and *C. kurzii*) (Fig. 2l) and scalariform (*L. fenestrata*). Vessel-ray pits with much reduced borders to apparently simple, pits horizontal (scalariform, gash like) in *L. fenestrata* (Fig. 2d) to vertical (palisade) in all selected species of *Quercus* and *C. kurzii*) (Fig. 2k). Tyloses present in *C. kurzii.* Mean vessel element

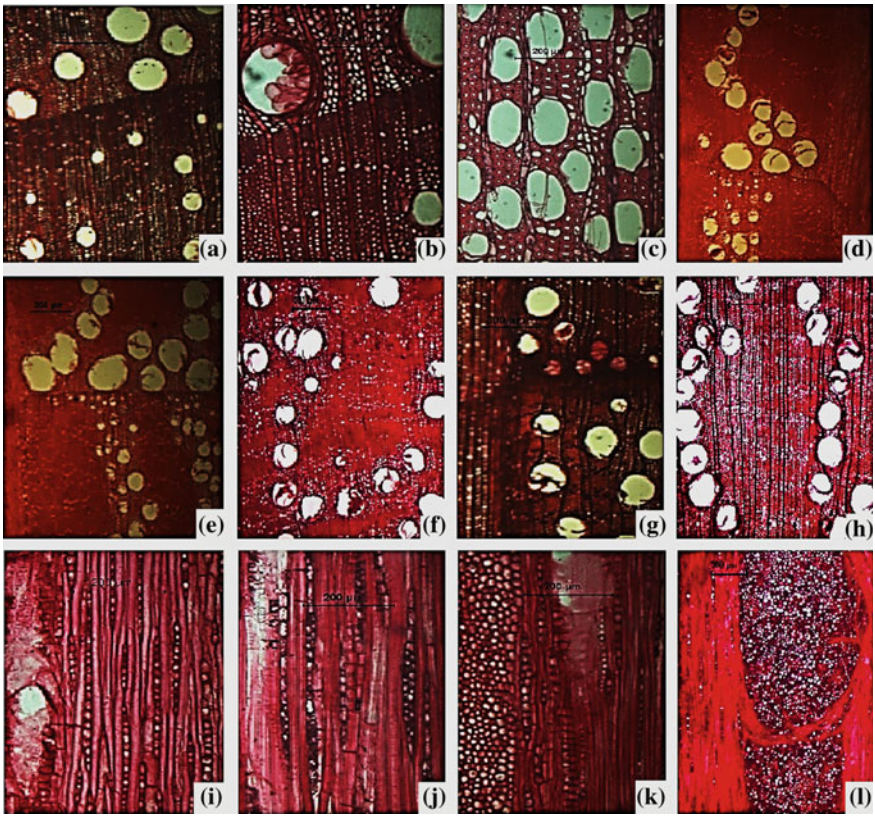

Fig. 1 a–h Cross sections of woods. Ring porous wood; tyloses present; diffuse and diffuse-in-aggregate apotracheal parenchyma (**a**, **b**) (*Castanopsis kurzii*). Diffuse porous wood; solitary vessels with angular outline; diffuse and diffuse-in-aggregate apotracheal parenchyma (**c**) (*Lithocarpus fenestrata*). Ring porous wood; vessels in dendritic pattern (**d**, **e**) (*Quercus griffithii*). Diffuse porous wood; vessels in diagonal and radial pattern; gelatinous fibres present; diffuse and diffuse-in-aggregate apotracheal parenchyma (**f**) (*Quercus lamellosa*). Ring porous wood; solitary vessels in radial pattern; tyloses present; scanty paratracheal parenchyma and parenchyma bands alternating with fibres (**g**, **h**) (*Quercus spicata*). **i–l** Tangential longitudinal sections of woods. Uniseriate rays; parenchyma strand (**i**) (*Castanopsis kurzii*). Uniseriate and biseriate rays; prismatic crystals in axial parenchyma (**j**) (*Lithocarpus fenestrata*). Uniseriate and aggregate rays (**k**, **l**) (*Quercus griffithii* and *Quercus lamellosa)*

length and tangential diameter of vessel vary from 569.2 ± 21.6 µm (*C. kurzii*) to 1736.5 ± 327.4 µm (*L. fenestrata*) and 103.8 ± 17.1 µm (*L. fenestrata*) to 197.5 ± 2.46 µm (*Q. lamellosa*). Vessel frequency (vessel/mm^2) varies from 7 (*Q. lamellosa*) to 32 (*L. fenestrata*). Vessel percentage 17% (*Q. lamellosa*) to 41% (*L. fenestrata*) (Fig. 3). Tyloses present in *Q. spicata* and *C. kurzii* (Fig. 1b, g).

Fibres—Fibres constituting the ground tissue are polygonal or rectangular in all species (Fig. 1a–h). Mean fibre length varies from 1229.5 ± 91.6 µm (*C. kurzii*) to

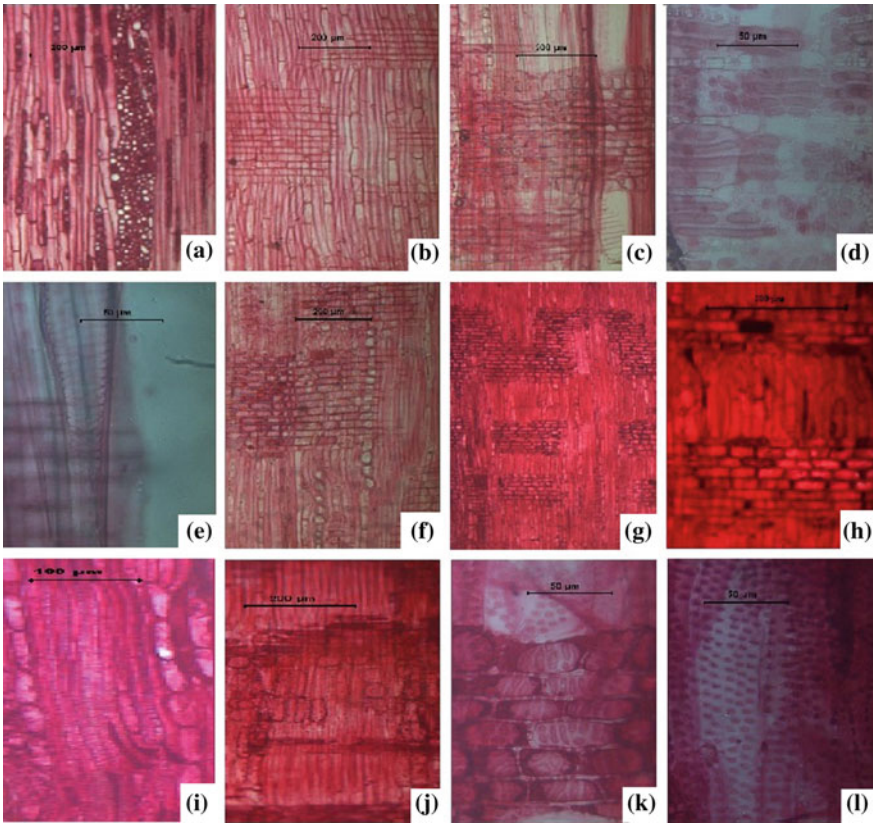

Fig. 2 **a** Tangential longitudinal section of wood. Uniseriate, aggregate rays and parenchyma strand (**a**) (*Quercus spicata*). **b–l** Radial longitudinal sections of woods. Homocellular ray composed of procumbent cells (**b**) (*Castanopsis kurzii*). Heterocellular ray composed of procumbent cells in the body and marginal rows of square/upright cells; scalariform perforation plate (**c**); vessel-ray pits much reduced border to apparently simple, pits scalariform (**d**); spiral thickenings in vessel (**e**) (*Lithocarpus fenestrata*). Homocellular ray composed of procumbent cells; crystals in parenchyma (**f**) (*Quercus griffithii*). Homocellular ray composed of procumbent cells; vasicentric tracheids (**g–i**) (*Quercus lamellosa*). Heterocellular ray composed of procumbent cells in the body and marginal rows of square/upright cells (**j**); vessel-ray pits much reduced border to apparently simple, pits vertical/palisade (**k**); intervessel pits alternate (**l**) (*Quercus spicata*)

2457.4 ± 325.9 µm (*L. fenestrata*). Mean fibre diameter and mean fibre wall thickness are 19.7 ± 3.1 µm (*Q. griffithii*) to 41.5 ± 6.9 µm (*L. fenestrata*) and 4.4 ± 1.6 µm (*C. kurzii*) to 14.1 ± 3.0 µm (*L. fenestrata*) (Table 2). Gelatinous fibres present in *L. fenestrata*, *Q. griffithii* and *Q. lamellosa*. Fibre percentage varies from 35% (*L. fenestrata* and *Q. spicata*) to 48% (*Q. lamellosa*) (Fig. 3).

Vasicentric tracheids present in all selected species (Fig. 2i). Mean length and diameter of vasicentric tracheid vary from 924.4 ± 147.2 µm (*Q. spicata*) to

Table 2 Microscopic features of selected species of family Fagaceae

Species	VL (μm)	VD (μm)	RH (μm)	RW (μm)	FL (μm)	FD (μm)	FLD (μm)	FWT (μm)	VTL (μm)
C. kurzii	368.31–789.3 (569.2 ± 21.6)	72.9–208.4 (142.9 ± 8.8)	312.6–864.9 (486.4 ± 140.6)	20.8–31.3 (21.7 ± 2.9)	1078.7–1447.1 (1229.5 ± 91.6)	18.2–36.4 (22.5 ± 4.0)	10.4–18.2 (13.8 ± 2.8)	2.6–10.4 (4.4 ± 1.6)	552.5–1210.4 (1024.3 ± 134.7)
L. fenestrata	1315.5–2078.5 (1736.5 ± 327.4)	72.9–145.9 (103.8 ± 17.1)	197.9–687.7 (366.4 ± 132.8)	20.8–31.3 (27.1 ± 5.2)	1894–3051.9 (2457.4 ± 325.9)	26.1–52.0 (41.5 ± 6.9)	7.8–20.8 (13.3 ± 3.9)	7.3–19.5 (14.1 ± 3.0)	2108.2–2889.2 (1945.4 ± 254.8)
Q. griffithii	473.6–789.3 (576.2 ± 75.2)	83.4–250.1 (144.6 ± 29.1)	208.4–573.1 (366.7 ± 86.1)	20.8–31.3 (22.1 ± 3.5)	1157.6–1657.5 (1417.2 ± 147.7)	15.6–26.0 (19.7 ± 3.1)	5.2–13.0 (6.9 ± 2.1)	3.9–9.1 (6.5 ± 1.3)	631.0–1315.0 (886.6 ± 165.6)
Q. lamellosa	487.3–893.3 (698.9 ± 99.9)	157.1–240.8 (197.5 ± 24.6)	213.4–1120.0 (408.9 ± 178.3)	26.1–80.1 (48.0 ± 16.3)	1297.3–2162.2 (1762.7 ± 216.9)	15.6–31.2 (22.5 ± 4.0)	5.2–13 (8.9 ± 2.4)	2.6–10.4 (6.8 ± 1.6)	1270.3–1756.8 (1572.9 ± 176.8)
Q. spicata	594.6–1216.21 (816.2 ± 170.5)	114.6–187.6 (157.9 ± 22.2)	156.3–395.9 (273 ± 56.9)	20.8–41.7 (22.9 ± 5.2)	1105.0–1525.9 (1310.2 ± 112.3)	18.2–31.2 (23.5 ± 3.6)	10.4–20.8 (12.3 ± 2.5)	2.5–7.8 (5.7 ± 1.3)	657.0–1183.9 (924.4 ± 147.2)

VL vessel length; *FD* fibre diameter; *VD* vessel diameter; *FLD* fibre lumen diameter; *RH* ray height; *FWT* fibre wall thickness; *RW* ray width; *VTL* vasicentric tracheid length; *FL* fibre length

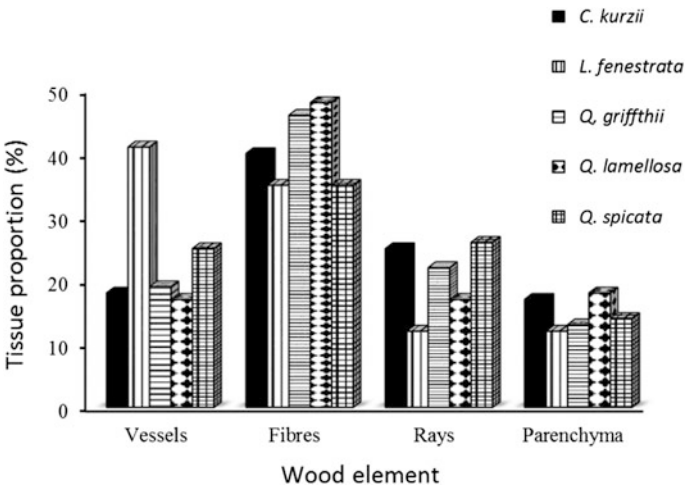

Fig. 3 Tissue proportion of selected species of family Fagaceae

1945.4 ± 254.8 µm (*L. fenestrata*) and 21.4 ± 2.9 µm (*Q. lamellosa*) to 29.1 ± 5.0 µm (*C. kurzii*) (Table 2).

Axial parenchyma—All species show apotracheal diffuse to diffuse-in-aggregate parenchyma (Fig. 1a–h). *Q. spicata* also show scanty paratracheal parenchyma and bands of parenchyma alternating with fibres (Fig. 1h). Parenchyma strands are usually 4–8 celled. Parenchyma percentage varies from 12% (*L. fenestrata*) to 18% (*Q. lamellosa*) (Fig. 3).

Rays—All three *Quercus* species have uniseriate and aggregate rays (Figs. 1k–l and 2a). *C. kurzii* has only uniseriate rays while both uni- and biseriate rays are present in *L. fenestrata* (Fig. 1i–j). Average height and width of uniseriate rays range from 366.4 ± 132.8 µm (*L. fenestrata*) to 486.4 ± 140.6 µm (*C. kurzii*) and 21.7 ± 2.9 µm (*C. kurzii*) to 48.0 ± 16.3 µm (*Q. lamellosa*) (Table 2). Rays both homocelluar and heterocellular. Homocellular rays comprise of either procumbent cells or square/upright cells. Heterocellular rays comprise of procumbent body cells and marginal rows of square/upright cells (Fig. 2c). Rays per mm. range from 7 (*Q. lamellosa*) to 12 (*C. kurzii*). Ray percentage varies from 12% (*L. fenestrata*) to 26% (*Q. spicata*) (Fig. 3).

Crystals—Prismatic crystals in chambered axial parenchyma cells present in *Q. griffthii* and *L. fenestrata* (Fig. 2c, f).

According to Gamble (1922), the selected *Quercus* species belong to three subgenera. *Q. griffthii* of subgenus Lepidobalanus is deciduous tree, while *Q. lamellosa* of subgenus Cyclobalanopsis and *Q. spicata* of subgenus Pasania are evergreen trees. The selected *Quercus* species have both ring porous wood with distinct growth rings (*Q. griffthii*) and diffuse porous wood with indistinct growth rings (*Q. lamellosa*) and distinct growth rings (*Q. spicata*). The available literature reveals that deciduous oak trees have ring porous wood and evergreen tree have

diffuse porous. The present study is in agreement with the findings of Das (1990) and Raturi et al. (2001). All the selected *Quercus* species have common features like simple perforation plates, intervessel pits opposite and vessel-ray pits with much reduced borders to apparently simple and vertical (palisade), uniseriate and aggregate rays. However, *Quercus* species differ from each other in the absence of gelatinous fibres and the presence of paratracheal parenchyma (*Q. spicata*), less vessel frequency and rays/mm (*Q. lamellosa*). Most of the features of *C. kurzii* are similar to *Quercus* species except aggregate rays. It differs from *L. fenestrata* in vessel characteristics and the presence of biseriate rays.

The presence of aggregate rays in *Lithocarpus* species has been reported by number of workers (Rao et al. 1991; Joshi 2004; Noshiro and Sasaki 2011). However, in the present study, aggregate rays are absent in selected species. Some features like vessels with helical thickenings in tail, scalariform perforation plates, scalariform intervessel pits, maximum vessel frequency and biseriate rays are reported only in *L. fenestrata* and these may be used to differentiate it from *Quercus* species and *C. kurzii*. The anatomical features of *C. kurzii* are similar to other *Castanopsis* species of Meghalaya (Sharma et al. 2011a). The present study reveals that different genera of family Fagaceae can be recognized by their wood anatomical characters.

Conclusions

The members of family Fagaceae have both distinct and indistinct rings with ring-porous, diffuse-porous and semi-ring-porous woods. Vasicentric tracheids, diffuse and diffuse-in-aggregate axial parenchyma are the common characters present in all genera. *Quercus* species are characterized by aggregate rays. Long vessels with helical thickenings in their tails, scalariform perforation plates, scalariform intervessel pits, maximum vessel frequency (32/mm^2) and vessel percentage (41%) are present only in *L. fenestrata*. *C. kurzii* shows the absence of both aggregate and biseriate rays. Crystals in chambered axial parenchyma are present in *Q. griffithii* and *L. fenestrata*.

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Anatomical Characterisation and In Vitro Laboratory Decay Test of Different Woods Decayed by *Xylaria hypoxylon*

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Abstract Different species of *Xylaria* are often reported as an endophyte in different groups of plants starting from liverworts to angiosperms. In the present study, *Xylaria hypoxylon* isolated from branch stubs of living trees was utilised for in vitro decay test to investigate the pattern of cell wall alterations in sound wood blocks of *Azadirachta indica*, *Leucaena leucocephala*, and *Tectona grandis.* Naturally infected as well as in vitro decayed wood showed cavities and erosion troughs at the lumen surface. In laboratory decay test, vertical invasion of mycelia occurred through the lateral wall pits of the vessels and vessel-associated parenchyma while ray cells enabled radial movement of mycelia. At the end of 60 days, the progression of degradation in the fibre cell wall was evident by the formation of transverse boreholes in the cell walls, erosion troughs at the lumen surface, and larger cavities in the S_2 layer of secondary wall. The erosion channels were angular to round (i.e. V-shaped or U-shaped). Fungus was also tested for polyphenol oxidase (Bavandemm's test) production by on-plate assay and was found to be positive. Confocal microscopy revealed delignification pattern during degradation of cell wall of different cell types in all the three woody species. The SEM analysis of degraded wood showed the ultrastructural changes in the cell wall particularly penetration of hyphae through the S_2 layer of the cell walls forming tunnels through it.

Keywords *Azadirachta indica* · *Leucaena leucocephala* · *Tectona grandis* · Soft rot · Wood degradation · *Xylaria hypoxylon*

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Introduction

Fungus is one of the indispensible constituents of forest ecosystem, which plays an important role in salvaging of the carbon stored in the form of different complex organic materials (Fazio et al. 2010; Sanghvi et al. 2013). Wood degradation is a part of carbon recycling process under natural condition, but it also causes extensive economic loss by destructing (degrading) the quality of timber, furniture, ancient sculptures, and many other wood products. The process of wood degradation is categorised into three major classes on the basis of removal of wood cell wall components, viz., white rot, brown rot, and soft rot (Liese 1970; Blanchette 1991, 2000; Eaton and Hale 1993; Worall et al. 1997; Schwarze 2007; Koyani et al. 2010; Hickman et al. 2011). Beside economic loss to human fraternity, it also acts as a symbiont with algae and as an endophyte to almost all groups of plants and helps them in various ways. Endophytes help the plants as growth promoters, increase in disease resistance, tolerance to environmental stress, and help in nutrient recycling by producing different secondary metabolites/by-products (Petrin and Petrini 1985; Rogers 2000; Sturz et al. 2000; Davis et al. 2003; Oses et al. 2008; Tenguria et al. 2011).

Fungi from various groups are reported as endophytes of many plants including higher plants to lower cryptogams including liverworts and algae (Arnold et al. 2000; Sturz et al. 2000; Davis et al. 2003; Tenguria et al. 2011) to almost all the vascular plants (Oses et al. 2008). Most of these endophytic fungi belong to ascomycetes (Davis et al. 2003) from which many of them are members of the Xylariaceae. Some members of this family are also said to be symbiotic, in which fungal partner produces fungal toxin that provided protection from herbivory while host plant provides nutrition to the fungal partner (Davis et al. 2003). However, one of the existing hypotheses is that Xylariaceae endophytes are silent coloniser, which later on degrade wood cell wall when the plant becomes weak (Petrin and Petrini 1985; Davis et al. 2003; Oses et al. 2008; Tenguria et al. 2011). These endophytes use this strategy as an advantage over hostile saprobes to have a right over the cell wall substrate before other saprobe attacks (Carroll 1995). Therefore, Hoff et al. (2004) recommended further studies on the role of xylem-inhabiting endophytic fungi in wood biodegradation under natural condition.

It may be the fact that some of the xylariaceous members may be true endophytes though there are no documented evidences for any benefits of Xylariaceae to living host plants (Rogers 2000; Davis et al. 2003). In contrast, others may be silent invaders and pretending as endophytes. Available information indicates that nutrient and limited oxygen supply may be associated with the non-pathogenic behaviour of the fungus when existing in host tissue (Sieber 2007; Oses et al. 2008). *Tectona* falls under durability class I and is highly resistant to decay (ASTM 1981; Bhat et al. 2005); therefore, it is possible that teak trees do not express any disease symptoms, but if inoculated in healthy dry wood blocks, *Xylaria hypoxylon* may act as a wood degrader. Hence, in the present study, fruiting bodies of *X. hypoxylon* growing on the branch stubs of living *T. grandis* trees were used for in vitro decay

test to investigate whether it alters the wall components of wood cells of *Tectona grandis*, *Leucaena leucocephala*, and *Azadirachta indica.*

The main aim of the present investigation is to study whether *X. hypoxylon* is an endophyte or acts as a silent wood degrader. If it is saprobe, what are the structural alterations induced in cell wall components in response to wood-degrading enzymes and pattern of wood degradation.

Materials and Methods

Sample collection: Infected wood samples were collected from the branch stubs showing fruiting bodies of *Xylaria hypoxylon* from the *T. grandis* growing in Girnar Hills (Junagadh) and Pavagadh forest, Gujarat State (India). Wood samples measuring about 60 × 60 mm in length and width, and deep enough to include infected and healthy wood along with fruiting bodies were excised from the branch stubs with the help of chisel and hammer. Some of the samples were immediately fixed in formalin–acetic acid–alcohol (Berlyn and Miksche 1976) for histological study. Rest of the samples and fruiting bodies were packed separately in sterile polyethylene bags for the isolation of fungus.

Isolation and identification of fungus: Infected wood blocks packed in sterile polyethylene bags were cut into small pieces (approximately 15–20 mm in length and 8–10 mm in width) and washed thoroughly in running tap water. Wood pieces and fruiting bodies were surface-sterilised by 0.1% $HgCl_2$ and washed with sterile distilled water. Thereafter, fruiting bodies were cut into small pieces with the help of sterile surgical blade. Subsequently, both wood and fruiting bodies were treated with 70% alcohol for 4–5 s and inoculated on MEA media containing 2.5% agar. Pure cultures were established by serial transfer and stored at 4 °C. Fungal DNA was extracted as described by Möller et al. (1992) by crushing approximately 20 mg of mycelia and aseptically opened fruiting bodies. Obtained DNA was amplified with thermocycler (Applied Biosystem Pvt. Ltd), and the product was sent to Gujarat State Biotechnology Mission (GSBTM), Gandhinagar, for sequencing. On the basis of sequence, the fungus was identified as *Xylaria hypoxylon.*

In vitro decay test and characterisation of wood decay pattern: Sound wood blocks of *T. grandis*, *L. leucocephala*, and *A. indica* were cut into 20 × 20 × 20 mm cubic blocks. The blocks were soaked in water for 24 h to obtain optimum moisture content to expedite fungal growth. These wood blocks were autoclaved at 120 °C for 30 min. After surface sterilisation with 70% alcohol, these blocks were kept on the autoclaved petri dish containing MEA media. Subsequently, these petri plates with wood blocks were inoculated in triplicates with one-week-old active culture of *X. hypoxylon* and incubated at 27 ± 1 °C and 75–80% relative humidity. One set of petri dish without fungal inoculation was maintained as control. Prior to fungal inoculation, some of the wood blocks were marked and same wood blocks were weighed prior to and after (oven-dried for 48 h at 60 °C) fungal inoculation.

Per cent weight loss was determined as % weight loss = $[(W_O–W_D)/W_O] \times 100$, where W_O and W_D are oven-dried weight of wood block before and after fungal incubation.

Incubated wood blocks were harvested at 30, 60, 90, and 120 days of incubation. After harvesting, wood blocks were cleaned with brush to remove the mycelia on wood surface and fixed in FAA as described above. Both naturally infected and treated wood blocks were trimmed into cubic blocks of 10 × 10 × 20 mm. Transverse, tangential, and radial sections of 12–15 μm thick were cut using sliding microtome and stained with Safranin-Astra blue combination (Srebotnik and Messener 1994). Sections were dehydrated through ethanol xylene series and mounted in DPX. Observations and microphotographs were obtained with Leica DME 2000 (Germany) research microscope.

Enzyme test: Ligninolytic enzyme activity was performed by Bavandemms's test and ABTS on-plate assay. Five-mm agar plug of one-week-old culture was inoculated in the centre of petri plate containing 2.5% malt extract and agar (MEA) solid media. To confirm the production of polyphenol oxidase, MEA media was supplemented with 1% tanic acid while 2 μM ABTS was added to it to confirm the production of laccase through green or greenish blue colour under and around the fungal colony (Abdel-Raheem 1997). For cellulase enzyme, carboxymethyl cellulose (CMC) was supplemented in MEA media and cellulolytic activity was visualised by flooding the petri plate with Congo red (Teather and Wood 1982). Petri plates were incubated at 28 °C. After one week of inoculation, enzyme activity was qualitatively evaluated by observing the colour change.

Results

Enzyme test: On-plate decolourisation assay for the extracellular enzyme production by *X. hypoxylon* showed positive response for the production of ligninolytic enzymes and cellulases. It gave positive reaction for the activity of peroxidases while weak activity was noticed for cellulases. In the Bavandemm's test (for polyphenol oxidase), browning of the medium initiated after the 4th day of its incubation and the plates became partially brown within 9 days of incubation (Fig. 1a) while complete browning was observed on 12th day. MEA media supplemented with carboxymethyl cellulose when flooded with Congo red after 9 and 12 days of inoculation showed weak zone of clearance.

Fungal colonisation and structural changes in the wood: The anatomical studies of sap wood blocks of three hard wood species (*L. leucocephala*, *A. indica*, and *T. grandis*) inoculated with *X. hypoxylon* revealed the structural changes during colonisation of mycelium and pattern of degradation of cell wall. The colonisation of mycelia occurred mainly through ray cells. The radial walls showed locally degraded area through which penetration hyphae migrate to adjacent axial elements like parenchyma and fibres (Fig. 1b). The boreholes are narrower than the fungal hyphae present within the lumen (Fig. 1b). Parenchyma cells showed localised

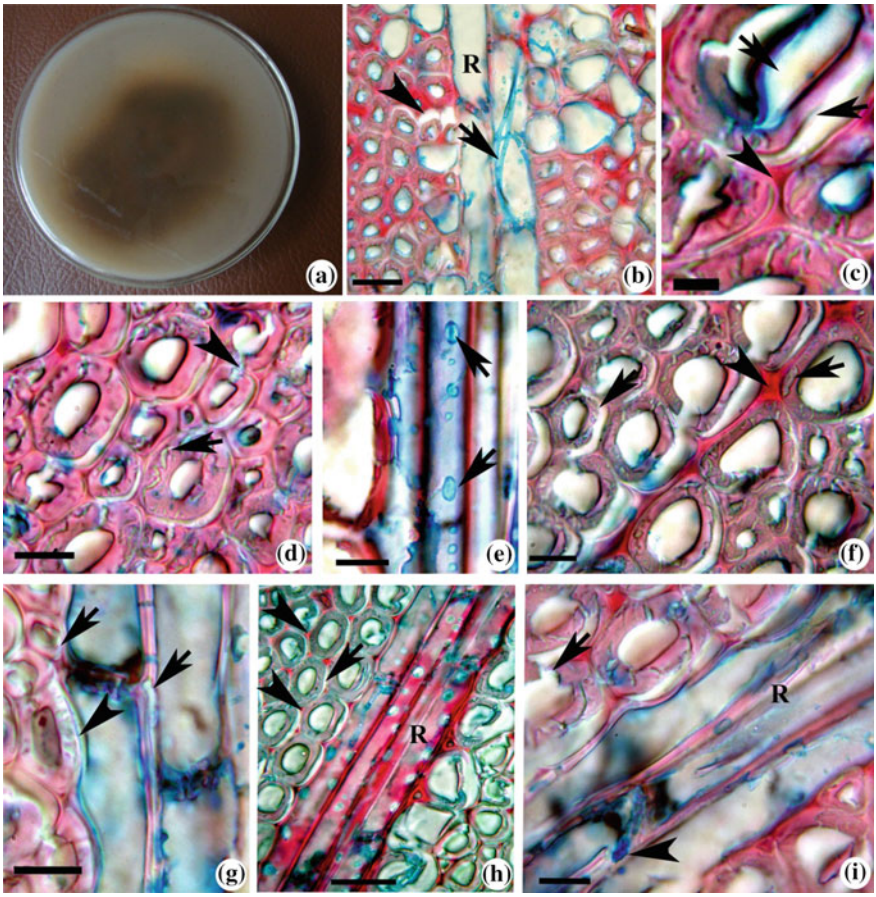
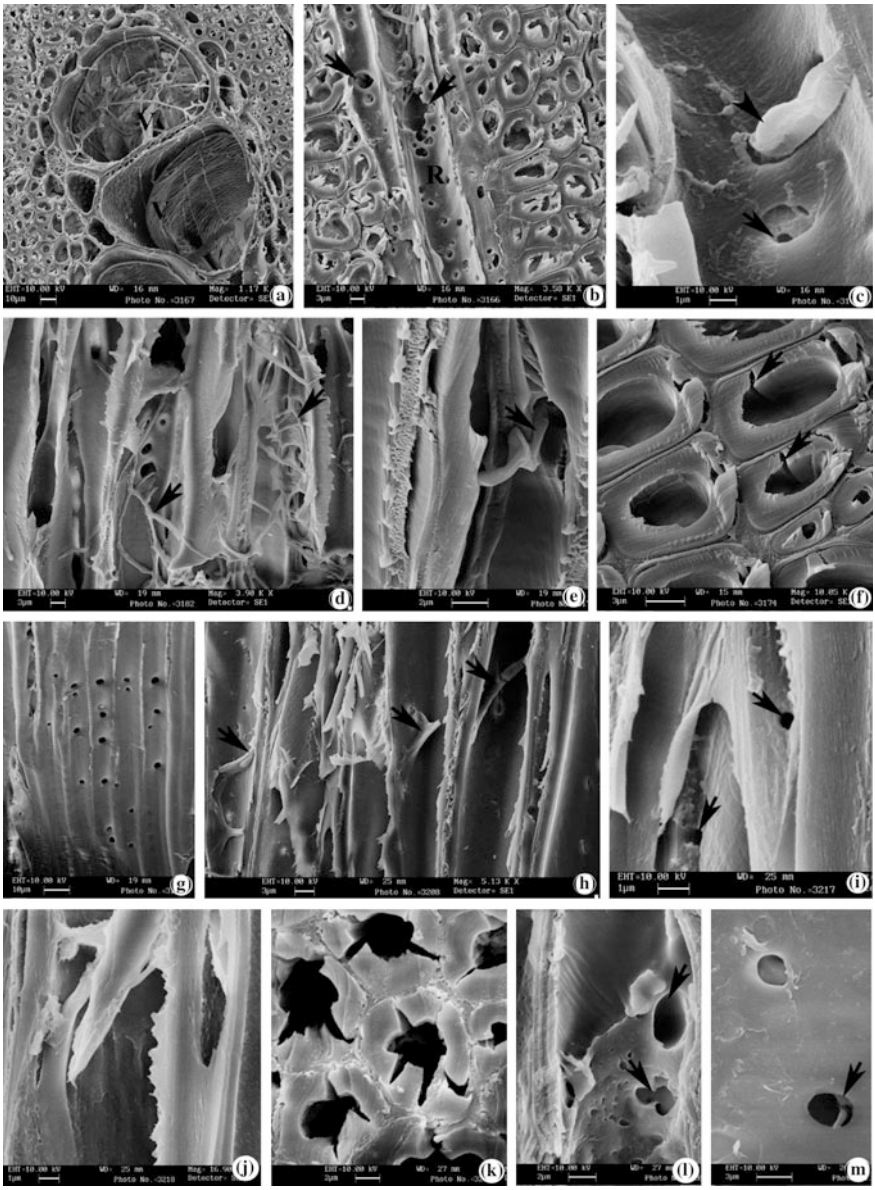


Fig. 1 Photomicrograph of on-plate assay for polyphenolic oxidase activity (**a**) and light micrographs from the transverse sections of sap wood of *L. leucocephala* (**b**–**d**), *A. indica* (**e**–**g**), and *T. grandis* (**h**–**i**) infected with *X. hypoxylon*. **a** Bavendamm test showing polyphenolic oxidase activity by *X. hypoxylon.* **b** Fungal hyphae (*arrow*) migrating through rays (R). *Arrowhead* indicates the separation of fibre wall adjacent to rays. Note the small mycelia within the lumen and inner surface of cell wall of parenchyma cells. **c** Fibre wall showing separation from transition zone between S1 and S2 layer and within S2 layer (*arrows*). *Arrowhead* indicates the intact cell corner and middle lamellae. **d** Lateral progression of cell wall degradation within S2 layer (*arrow*). *Arrowhead* indicates localised degradation of cell wall and middle lamellae. **e** Ray cells showing large boreholes produced through pit erosion (*arrows*). **f** Fibres showing separation of cells at transition area between S1 and S2 layers and progressive degradation towards the inner wall layers (*arrows*). *Arrowhead* indicates the degradation resistance from middle lamellae region of fibres. **g** Ray cells showing degradation of compound middle lamellae region (*arrows*) and penetration of hyphae through the radially extending hole (*arrowhead*). Note the middle lamellae degradation confined only to rays while the adjacent fibres showing cell separation at S1 layer region. **h** Teak wood showing degradation-resistant compound middle lamellae region of fibres (*arrowheads*). *Arrow* indicate the separation of fibres wall at transition region of S1 and S2 layers. Note the extensive erosion of pits in the rays (R) resulting large boreholes in radial wall. **i** Fibre wall showing cell separation followed by localised degradation of wall polymers across the cell wall (*arrow*). *Arrowhead* indicates the hyphae penetrating through degraded radial wall of ray cell (R). *Scale bar* **b**, **h** = 50 μm; **c** = 10 μm; **d**–**i** = 25 μm

Fig. 2 Scanning electron micrographs from the sapwood of *A. indica* (**a**–**g**), *L. leucocephala* (**h**–**i**), and *T. grandis* (**k**–**m**) infected with *X. hypoxylon*. *Note TS* transverse section; *RLS* radial longitudinal section. **a** Colonisation of mycelia within vessel lumen (V). Note the migration of mycelia into adjacent paratracheal parenchyma cells through pits (TS). **b** Ray cell showing large cavities (*arrow*) produced through merging of eroded pit regions (TS). **c** Erosion of pit membrane (*arrow*) and penetration of hyphae through pit in the ray cell (*arrowhead*) (TS). **d** Colonisation of mycelia (*arrowhead*) and spreading of hyphae in parenchyma cells and fibres (RLS). **e** Fungal hyphae (*arrow*) passing through the borehole in the fibre cell wall (RLS). **f** Fibre showing separation at transition area between S1 and S2 layer and 'L'-shaped bending from lumen side of the secondary wall (*arrows*) (TS). **g** Fibre wall showing series of conical-shaped bore holes (RLS). **h** Fungal hyphae within the fibre lumen (*arrows*) (RLS). **i** Bore holes (*arrows*) produced near the tip region of fibres (RLS). **j** Simultaneous degradation resulting in large bore holes in the radial and longitudinal walls of fibres during advancement of decay (RLS). **k** Teak wood fibres showing localised degradation from cell lumen towards middle lamellae (TS). **l** Fibre wall showing cavities (*arrows*) produced in the inner lumen during 'L' bending pattern (RLS). **m** Penetration hyphae within the bore hole (*arrow*) in the secondary wall of fibre (RLS) ►

degradation of cell wall and middle lamellae during penetration of hyphae into fibres (Fig. 1b). Following the entry of hyphae into fibres, separation of cells is also observed (Fig. 1b). Two types of cell separation are noticed in the fibre of all the wood species. In the first pattern, cell separation occurs at transition zone between S_1 and S_2 layer and spreads laterally. Sometimes the inward entry of hyphae is observed for short distance followed by its lateral movement results in separation within S_2 layer (Fig. 1c, f, h). The second pattern involves the localised degradation of secondary wall and compound middle lamellae between fibres (Fig. 1d, f, i). Boreholes having different sizes are formed in response to the progression of pit erosion which is evident in the ray cells (Fig. 1e, h). In *A. indica* wood, fibres show separation near the S_1 layer and degradation occurs within the S_2 layer resulting in minute cracks perpendicular to the orientation of middle lamellae (Fig. 1f). The ray cells show delignification of the middle lamellae, and fungal hyphae are observed within the channels (Fig. 1g, i). In contrast, fibres are not separated through the compound middle lamellae by *X. hypoxylon* in all the three wood species (Fig. 1c, f, h). After separation, degradation of fibre walls in teak progresses from S_3 layer towards outer secondary wall layers and results in localised and minute cracks across the cell wall (Fig. 1i).

Scanning electron microscopy (***SEM***): SEM analysis revealed the structural alterations in the cell walls during colonisation of *X. hypoxylon* and subsequent alternation in different cell types in the wood of *L. leucocephala*, *A. indica*, and *T. grandis*. Abundant mass of hyphal colonised within the vessel lumen (Fig. 2a). The penetration hyphae migrate to the adjacent paratracheal parenchyma cell through pit by eroding it (Fig. 2b). Ray cells show formation of boreholes by degrading the pits and merging of adjacent holes that result in large cavities in the cell wall (Fig. 2b). Erosion of pit membrane and subsequent penetration of hyphae through the simple pits is observed in ray cells (Fig. 2c). Longitudinal sections show extensive branching of hyphae within fibre lumens and parenchyma and migrate into adjacent cells through boreholes (Fig. 2d, e). Separation of cells near transition zone between S_1 and S_2 layers and 'L' bending in the fibre wall is observed in transverse



sections (Fig. 2f). In *Leucaena* wood, hyphae found within the cell lumen and boreholes often appeared near the tip region of the fibres (Fig. 2h, i). During the advanced stages of decay, simultaneous decay of cell wall polymers results in extensively degraded, large void areas in both radial and tangential wall of fibres

(Fig. 2j). In contrast, teak wood shows typical 'L' bending in the secondary wall due to localised simultaneous degradation, starting from the cell lumen towards middle lamella (Fig. 2k, l).

Discussion

In all the three hardwood species, vessels and ray cells were colonised with abundant mass of hyphae from early stages of infection, indicating these cells may act as radial and axial channels during initial stages of fungal invasion within woody tissue. The axial alignment of xylem vessels and the radial arrangement of xylem ray parenchyma make easy access into the wood and allow widespread distribution of hyphae within the secondary xylem (Schwarze et al. 2004). SEM studies revealed that the entry of fungal hyphae occurs mainly through pit erosion and enters either into cell lumen or moves laterally through boreholes. According to Schwarze (2007), access to adjacent xylem cells occurs via pit apertures or direct penetration through the cell wall. Present study also shows that multiple branching of hyphae forms penetration hyphae inside the cell lumen which pass through the pits and produce erosion channels leading to the extensive growth of fungi.

During degradation process, *X. polymorpha* induced majority of structural alternations which are characteristic to soft rot decay type I. The major changes include cavity formation in the S_2 layer of secondary wall, cavities originate by L-branching of lateral penetration hyphae which traverse the cell wall through conical-shaped boreholes, the diameter of the boreholes was narrower than the hyphae found in the lumen, and these features are specific to soft rot decay type I (Anagnost 1998). The SEM analysis also confirms these features especially chain-like formation of cavities and its widening leading to coalescing into large voids.

The separation of cells was noticed in all the wood species studied. Cell separation by dissolution of middle lamellae is a characteristics feature of selective mode of white rot decay (Anagnost 1998). However, *X. hypoxylon* caused separation of cells from transition zone between S_1 and S_3 layers while compound middle lamellae remain intact. Ruel et al. (1981) reported a similar pattern of degradation in spruce wood by *P. chrysosporium* in which an inward attack of the cell originated at the pit membrane and degradation occurred across the transition between S_1 and S_2 layers. In the present study, the local degradation of cell wall and middle lamellae resulting in minute cracks between adjacent fibres was also noticed. *T. versicolor* caused degradation of fibre walls through localised degradation along with typical generalised thinning during simultaneous white rot decay in the heart wood of *Liquidambar orientalis* (Yilgor et al. 2013). Therefore, pattern of cell separation and localised degradation of cell wall during degradation by *X. hypoxylon* shows similarities with white rot mode of decay. Biochemical studies on wood decay by xylarious ascomycetes suggested some similarities to both soft rot and white rot decay (Worall et al. 1997; Nilsson et al. 1989; Anagnost 1998). The

soft rot mechanism may involve an enzyme system that is sensitive to the spatial arrangement of cell wall components such as nature of cellulose crystalline lattice structure, arrangement of microfibrils, different types of lignin and hemicellulose which may lead to difference in the soft rot attack on hardwood and softwood. Our current understanding on decay pattern by *X. hypoxylon* is based on information available from softwood species (Pine) and only one hardwood species, birch wood (Anagnost 1998). Therefore, we assume that variation observed in the decay pattern of three tropical hardwood species by *X. hypoxylon* might be related to their difference in chemistry and cell wall structure compared to those in temperate climates.

Although vessel lumen was invaded with fungal mycelia, the migration of fungus to adjacent element occurred only through pits. The vessel wall and compound middle lamellae of fibres in the sap wood of *T. grandis, L. leucocephala,* and *A. indica* showed resistance against degradation during the attack by *X. hypoxylon,* whereas the secondary wall of fibres became susceptible to fungal enzymes and underwent extensive degradation. Generally, members of Xylariaceae shows relatively poor lignolytic ability, and this is confined to the decomposition of syringyl lignin (Nilsson et al. 1989). In hardwood xylem, the vessel wall and compound middle lamellae of fibres are rich in guaiacyl monomeric units while secondary walls of fibre are rich in syringyl lignin (Grunward et al. 2002; Pramod and Rao 2012; Pramod et al. 2013). Therefore, the variation in lignin monomeric composition might be resulted in degradation resistance of vessel wall and middle lamellae while degradability of fibre walls in the hardwood species might be related to their lignin chemistry. On the contrary, the middle lamellae of ray cells in the *A. indica* showed delignification, suggesting their cell wall chemistry may have different chemical composition compared to other hardwood species.

Conclusions

The light and scanning electron microscopic analysis of the three hard wood species inoculated with *X. hypoxylon* indicates that it is an efficient wood degrader and it deteriorates wood cell wall by making bore holes on the cell wall, tunnelling through the S_2 layer of the fibre wall, by forming erosion channels and defibration, which is characteristic of the soft rot decay type I. However, the pattern of separation of cells at transition zone of S_1 and S_2 layers and producing minute cracks in the fibre wall showed structural similarities to those induced by white rot fungi. This result suggests the *X. hypoxylon* shares the characteristics of soft rot and white rot during degradation of three tropical hardwood species. Positive test for Bavandemm's test also indicates that it produces polyphenol oxidase, which could be responsible for the separation of fibres. Mycelial invasion occurs through the vessel lumen, and increase in pit diameter of ray parenchyma and vessel elements indicates mycelial movement is facilitated by the cell wall pits and by forming additional bore holes.

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Growth Ring Structure and Specific Gravity Variation in Juvenile and Mature Wood of Natural-Grown Teak (*Tectona grandis* L.f.)

Satish Kumar Sinha, R. Vijendra Rao, T.S. Rathore and H.P. Borgaonkar

Abstract The radial variation of specific gravity and growth ring structures was studied in juvenile and mature wood of *Tectona grandis* L. grown naturally in dry (Chandrapur) and moist (Thane) deciduous forests of Maharashtra, India. Five trees each from age group of 48–66-year from Chandrapur and 120–154-year-old trees from Thane were selected. The study revealed that the mean specific gravity of growth rings in juvenile wood was more than the mature wood from both the sites. The annual growth in juvenile and mature period was higher in dry site than moist site. Ring width of juvenile and mature wood was 4.45 and 2.30 mm with the latewood content of 85.70 and 72.97%, respectively, in dry site. The patterns of radial variation of ring width, latewood content and specific gravity demonstrated inconsistency in juvenile to mature wood in both the sites. The specific gravity of all the five trees from both sites showed a poor association between ring width and latewood content in juvenile wood; however, it was positive and strong in most of the samples from mature wood. Considering mean values, there was a positive strong association between ring width and specific gravity in both juvenile and mature wood of both the sites. However, the latewood content did not show strong association with specific gravity. The overall result showed that ring width, specific gravity and latewood content vary among individuals within and between two sites. It is also important that individual tree variation needs to be studied while breeding for higher specific gravity rather than mean variation of all the trees in specific site.

Keywords Juvenile wood · Latewood content · Mature wood · Ring width · Specific gravity · Teak

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Introduction

Variation in wood properties is a discrete disadvantage to the user who encounters problem in developing efficient methods for manufacture of variable wood products because it lowers the efficiency of use of wood as a raw material. Generally, variability within a single tree is more than among trees growing on the same site or between trees growing on different sites. The variation in wood properties may be due to environmental differences, genetic differences and their interactions (Zobel and Van Buijtenen 1989; Zobel and Jett 1995).

Specific gravity is generally considered to be one of the most useful properties in assessing the wood quality. It largely determines the value and utility of wood and overshadows the importance of other wood properties. Specific gravity (or wood density) is, in fact, not a single wood property but is determined by several wood properties such as latewood content, wall thickness, cell size, the amount of ray cells, the size and amount of vessel elements. Specific gravity does not vary evenly along the stem radius. Its variation is related to the growth ring structure in both juvenile and mature wood zone of ring-porous hardwoods (Vavrcik et al. 2010). Each growth ring consists of lighter earlywood and darker latewood. The earlywood zone of ring-porous hardwoods has a relatively high proportion of vessels, and the width of earlywood remains fairly constant from year to year; the latewood content, with fewer vessels, increases as ring width increases. This results in higher specific gravity of latewood and explains the reason for increase in specific gravity of wood with increasing latewood content (Zobel and van Buijtenen 1989).

Teak (*Tectona grandis* L.f.) is a ring-porous hardwood species renowned for its high wood quality in solid wood industries. Old natural-grown teak has been traditionally utilized in boat building, ship construction, wall panelling and fine woodworking furniture (Ladrach 2009). The natural distribution of teak is primarily confined to the peninsular region of India below 24°N latitude, and it is recognized centre for its genetic multiplicity and variability (Tewari 1992).

Review showed that there are contradictory findings considering the relationship between growth ring structures (ring width and latewood content) and specific gravity (or wood density) in teak from tropical regions (Bhat and Indira 1997; Varghese et al. 2000; Moya 2002; Jayawardana and Amarasekera 2009; Nocetti 2011; Sinha et al. 2014). The dry deciduous forests of Chandrapur and moist deciduous forests of Thane are renowned for natural-grown teak in Maharashtra state of peninsular India. In the present study, the pith to periphery variation of growth ring structures (ring width and latewood content) and specific gravity in the juvenile and mature wood and its interrelationship was compared using old teak trees located at Chandrapur and Thane of Maharashtra.

Materials and Methods

Sampling

The trees, mostly mature and over-mature grew naturally in dry deciduous forests of Chandrapur (19°57′ N; 79°21′ E) and moist deciduous forests of Thane (19°12′ N; 73°02′ E), were used in the study. From each site, five cross-sectional discs of teak trees of different age-groups were collected in November, 2006 at Mamla forest range of Chandrapur (coded C1–C5) and November, 2004 at Shirshad forest range of Thane (coded T1–T5) of Maharashtra (Table 1). Thus, total ten discs were collected for analysis either from the base of the logged trees or from the left over stumps (20–30 cm above ground) during felling period from Forest Development Corporation of Maharashtra, India.

The cross-sectional area of ten discs was polished with sand paper to expose the growth ring structures of wood for microscopic analysis. The age of trees was estimated by counting annual growth ring in the discs (Fig. 1c, d). The age used to differentiate the boundary between juvenile and mature wood of teak was estimated around 20 years based on the literature studies (Bhat et al. 2001; Kumar et al. 2002).

A radial strip of 1.5 cm width obtained from each disc having all growth rings from pith to periphery and was analysed in the laboratory.

Annual Ring Width, Latewood Proportion and Specific Gravity

Each annual ring width in the radial strip was measured to the nearest 0.01 mm under a Leica stereo-zoom microscope with the help of LAS live measurement

Table 1 Information of locality factors and general wood features of teak discs collected from Chandrapur and Thane of Maharashtra

Factor	Dry site (Chandrapur)	Moist site (Thane)
Forest type	Tropical dry deciduous forest	Tropical moist deciduous forest
Mean annual rainfall (mm)[a]	1250	2500
Mean temperature range (°C)[a]	20.4–35.5	24.4–30.2
Soil type	Sandy-loam soil	Red laterite well-drained soil
Age group (years)	48–66	120–154
Mean diameter range (cm)	34.7–38.2	40.4–47.7
Mean heart wood (%)	76.56	85.91

[a]Mean annual rainfall and temperature range from 1901 to 2000
Source India Meteorological Department (IMD), Pune

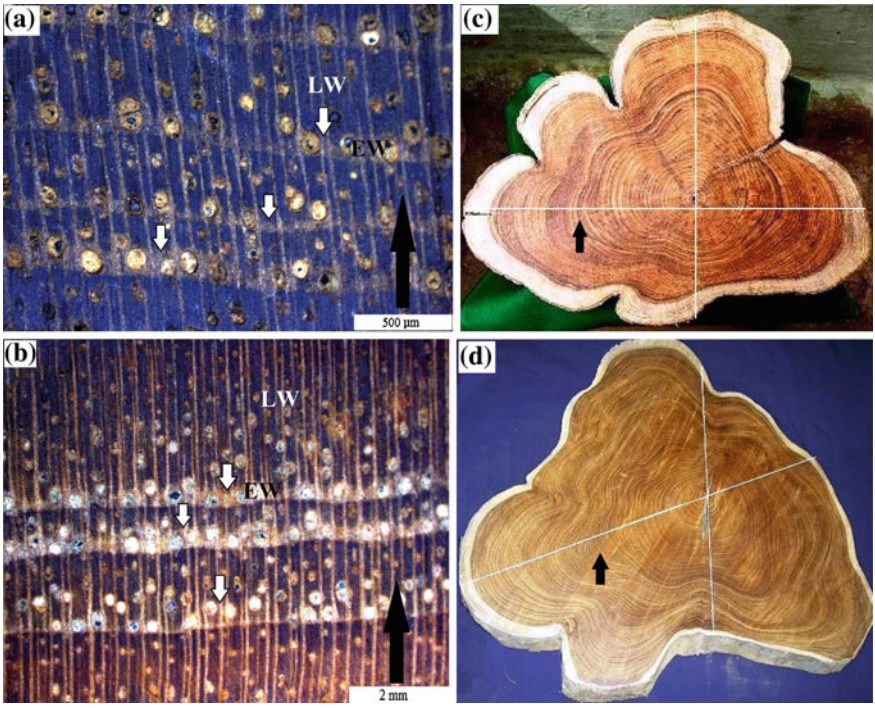

Fig. 1 Cross sections of a polished *Tectona grandis* wood from Chandrapur (*Top row*) and Thane (*Bottom row*). **a** and **b** Microscopic pictures showing gradual transition between earlywood (EW) and latewood (LW) of annual rings; *white arrows* indicate the arbitrary delineation between EW and LW; *black arrow* shows growth direction. **c** and **d** Macroscopic photographs of wood cross sections showing discrete annual growth rings; the *black arrow* indicates the border between juvenile and mature wood

software. Generally, there is a gradual transition between earlywood and latewood in growth rings of teak. While measuring ring width, latewood width was also computed in all the radial strips after making an arbitrary delineation between earlywood and latewood, by classifying the earlywood with wide vessels, initial parenchyma and thin-walled fibres and latewood with narrow vessels and highly lignified thick-walled fibres (Fig. 1a, b). Latewood proportion was calculated as latewood width divided by annual ring width multiplied by 100.

The radial strips were then cut tangentially to separate each annual ring into smaller blocks for the measurement of specific gravity. During measurement, two or more than two narrow rings were combined in few blocks of the mature wood zone of the strip, where these rings were too small to separate and measure individually. The basic specific gravity of each block was then determined by oven dry weight divided by weight of displaced water by green volume of the block. The same value of specific gravity was set for each narrow ring equal to the specific gravity of

individual block with the assumption that the ring width of each narrow ring in the block was more or less same.

Data Analysis

A simple correlation analysis was carried out using MS-Excel in order to find out the linear relationships between growth ring structures (ring width and latewood content) and specific gravity. The radial variation of growth ring structures and specific gravity was compared in juvenile and mature wood of trees growing between two sites using t test to confirm significant differences, if any.

Results and Discussion

Growth Ring Structures and Specific Gravity

The comparison of mean values of growth ring structures (ring width and latewood content) and specific gravity in juvenile and mature wood of each tree from two sites are presented in Table 2. The average ring width of wood in former and latter was 4.45 and 2.30 mm in dry site, while 4.03 mm and 1.19 in moist site. The variation among individual trees for annual ring width was more in juvenile wood of moist site than dry site, and it varied from 1.75 to 7.52 mm. However, the variation was less in mature wood of trees at both the sites.

The latewood content in juvenile and mature wood was 85.70 and 72.97% of the annual ring width, respectively, at dry site, whereas it was 76.36% in juvenile wood and 59.41% in mature wood of trees at moist site. The mean specific gravity of growth rings in juvenile wood was more than the mature wood at both the sites. It varied from 0.607 to 0.655 in juvenile wood and 0.539–0.605 in mature wood of all individuals at dry site. However, the mean specific gravity of growth rings at moist site varied from 0.602 to 0.702 in juvenile wood and 0.562–0.665 in mature wood.

The teak discs C1 and T2 showed a very high specific gravity in both juvenile and mature wood from dry site of Chandrapur and moist site of Thane, respectively. The wider annual rings of teak at both the sites exhibited significantly more latewood proportion than narrow rings, whereas earlywood proportion remained relatively constant (Fig. 1a, b). These results are in conformity with the findings of Bhat (1998), who reported that juvenile wood of teak showed wide growth rings with high proportion of latewood and high specific gravity (or wood density) as compared to mature wood in 65-year-old trees at three locations of diverse environmental conditions in Kerala, India.

Table 2 Comparison of mean values of ring width, latewood proportion and specific gravity in juvenile and mature wood of *Tectona grandis* from Chandrapur (C1–C5) and Thane (T1–T5) (Values presented are mean ± standard deviation)

Tree	Wood	No. of rings (N)	Ring width (mm)	*t* value	Latewood %	*t* value	Specific gravity	*t* value
C1	Juvenile	20	4.94 ± 2.32	3.95[c]	85.97 ± 6.04	6.18[c]	0.655 ± 0.034	3.81[c]
	Mature	46	2.67 ± 1.46		66.98 ± 12.94		0.605 ± 0.053	
C2	Juvenile	20	4.34 ± 2.04	4.01[c]	79.04 ± 9.23	6.38[c]	0.607 ± 0.027	5.83[c]
	Mature	46	2.31 ± 1.23		62.38 ± 9.77		0.543 ± 0.045	
C3	Juvenile	20	3.86 ± 2.39	4.11[c]	89.14 ± 10.80	3.76[c]	0.619 ± 0.034	3.68[c]
	Mature	34	1.54 ± 0.82		66.68 ± 11.98		0.569 ± 0.053	
C4	Juvenile	20	4.64 ± 1.36	6.63[c]	80.08 ± 8.03	3.47[b]	0.621 ± 0.037	3.80[c]
	Mature	28	2.29 ± 1.05		71.97 ± 7.64		0.560 ± 0.105	
C5	Juvenile	20	4.49 ± 1.47	3.72[c]	85.16 ± 5.24	3.95[c]	0.628 ± 0.034	4.15[c]
	Mature	33	2.91 ± 1.46		75.67 ± 9.73		0.539 ± 0.091	
Overall Mean	**Juvenile**	**20**	**4.45 ± 1.08**	**8.01**[c]	**85.97 ± 9.02**	**7.76**[c]	**0.626 ± 0.016**	6.25[c]
	Mature	**66**	**2.30 ± 0.93**		**72.97 ± 9.73**		**0.567 ± 0.040**	
T1	Juvenile	20	3.41 ± 0.88	14.03[c]	78.67 ± 7.41	6.41[c]	0.602 ± 0.031	2.49[a]
	Mature	134	1.02 ± 0.68		58.59 ± 13.61		0.562 ± 0.070	
T2	Juvenile	20	1.75 ± 1.20	2.19[a]	71.01 ± 9.14	3.19[b]	0.702 ± 0.055	2.51[a]
	Mature	128	1.14 ± 0.82		59.16 ± 16.13		0.665 ± 0.061	
T3	Juvenile	20	7.52 ± 2.06	12.57[c]	71.96 ± 7.80	4.57[c]	0.693 ± 0.018	5.53[c]
	Mature	114	1.50 ± 1.23		56.17 ± 14.99		0.624 ± 0.054	
T4	Juvenile	20	3.99 ± 1.32	9.06[c]	81.29 ± 6.54	6.73[c]	0.685 ± 0.023	6.08[c]
	Mature	110	1.05 ± 0.76		58.65 ± 14.65		0.595 ± 0.065	
T5	Juvenile	20	3.48 ± 1.34	6.22[c]	78.89 ± 5.59	5.24[c]	0.645 ± 0.029	2.58[a]
	Mature	100	1.51 ± 0.91		64.46 ± 11.95		0.620 ± 0.065	
Overall Mean	**Juvenile**	**20**	**4.03 ± 0.72**	**20.29**[c]	**76.36 ± 4.10**	**9.17**[c]	**0.665 ± 0.018**	7.33[c]
	Mature	**134**	**1.19 ± 0.56**		**59.41 ± 8.29**		**0.606 ± 0.035**	

[a]significant at 5% level
[b]significant at 1% level
[c]significant at 0.1% level

Radial Variation of Specific Gravity and Growth Ring Features

Pith to periphery variations of specific gravity and growth ring features viz. ring width and latewood proportion among five individual trees from Chandrapur (C1–C5) and Thane (T1–T5), is shown in Fig. 2. Ring width of all individual trees illustrated almost a similar radial variation from pith outwards (Fig. 2e, f). Ring width showed a variation or increasing trend in the beginning up to 20 years and then it declined in the middle. Finally, it remained relatively constant towards the bark. It signifies that ring width during juvenile period of tree growth is instable and after maturation of the cambium it becomes stable. A similar pattern was examined in more or less all individuals, and it confirmed that this attribute may be intrinsic. Many authors have reported that initial growth of teak is fast up to 20 or 25 years, afterwards its growth slows down which corresponds to the juvenile period of wood (Priya and Bhat 1998; Bhat et al. 2001; Kumar et al. 2002; Sousa et al. 2012).

Latewood proportion in annual ring of all individual trees at dry and moist sites of Maharashtra also exhibited almost a similar radial patterns of variation like ring width (Fig. 2c, d). Wide annual rings showed the high percentage of latewood, whereas the percentage of latewood was low in narrow rings. Bhat (1998) studied

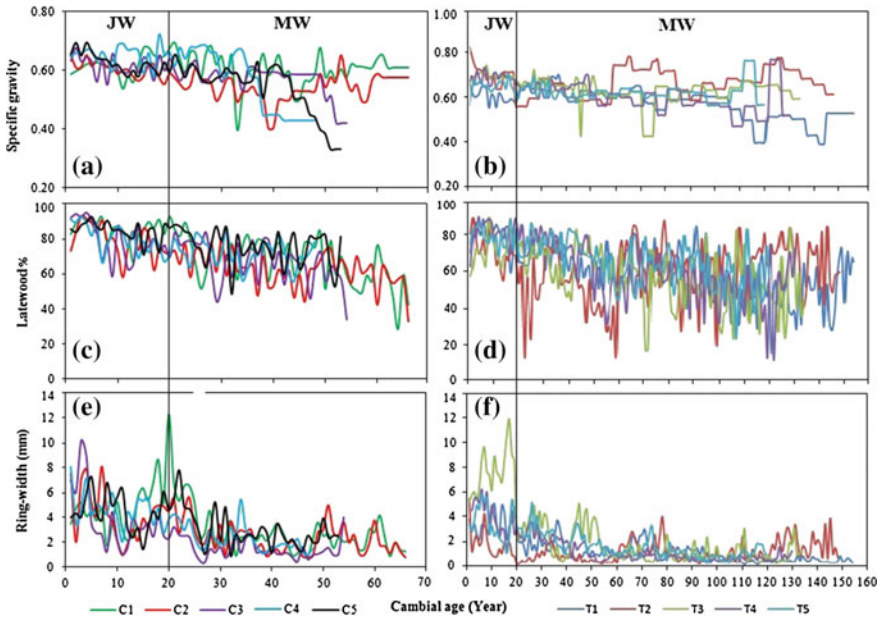

Fig. 2 Pith to periphery variation of specific gravity, latewood content and annual ring width in juvenile (JW) and mature wood (MW) of five teak trees from dry (**a**, **c** and **e**) and moist (**b**, **d** and **f**) sites of Maharashtra

on 7, 13, 20, 40 and 147 years teak trees from Kerala and revealed that the mean latewood content was 92, 90, 48, 44 and 70% of the mean ring width, respectively.

Pith to periphery variations of specific gravity for the five trees investigated from dry and moist sites is presented in Fig. 2a, b. In teak discs C3, C4, C5 and T1, the specific gravity decreased slightly in first few rings from pith outwards and then increased towards middle portion and finally dropped off towards the bark. In disc samples C2 and T2, the specific gravity declined rapidly up to 30 years and then increased up to 60 years afterwards, it remained relatively constant and at last, it decreased towards the bark.

In teak discs C1, T3, T4 and T5, the specific gravity showed an increasing trend up to 10 years, afterwards a little decreased and then it increased up to 30 years and finally remained relatively constant towards the periphery.

Correlation Between Ring Width and Specific Gravity

The relationship between mean ring width and mean specific gravity of five trees from pith to periphery of Chandrapur (C1–C5) and Thane (T1–T5) sites is shown in Fig. 3c, d. The mean ring width and specific gravity values gradually increased in juvenile wood while, it decreased rapidly up to 50 years in mature wood afterwards increased up to 60 years and finally, it remained relatively constant towards the bark. Correlation coefficients between ring width and specific gravity of five trees and their mean values in juvenile and mature wood from two sites are presented in Table 3. It is evident from t test that the correlation between ring width and specific

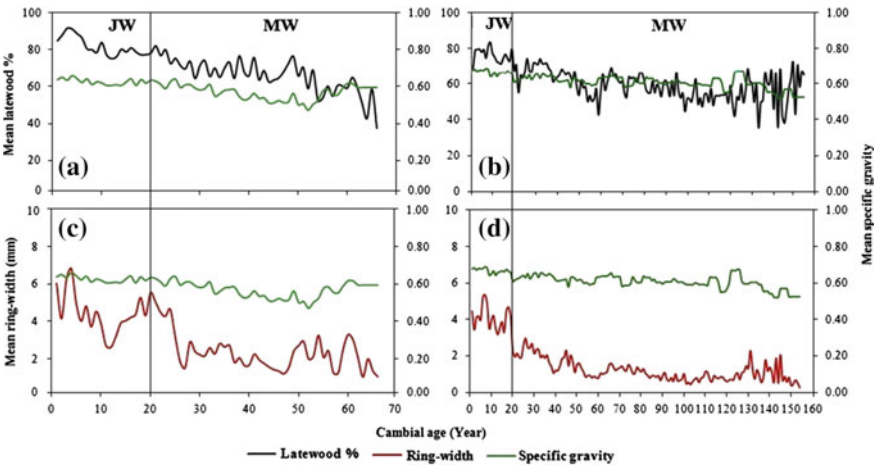

Fig. 3 Pith to periphery variation of mean specific gravity with latewood content and ring width in mature wood (MW) and juvenile wood (JW) of teak from Chandrapur (**a** and **c**) and Thane (**b** and **d**) of Maharashtra

Table 3 Correlation coefficients between growth ring structures (ring width and latewood content) and specific gravity in juvenile and mature wood of five disc samples of teak trees and their mean value each from Chandrapur (C1–C5) and Thane (T1–T5)

Trees	Wood	LW% versus SG	RW versus SG	Trees	Wood	LW% versus SG	RW versus SG
C1	Juvenile	0.496^{a}	0.643^{b}	T1	Juvenile	0.127^{ns}	0.067^{ns}
	Mature	0.259^{ns}	0.571^{c}		Mature	0.523^{c}	0.680^{c}
C2	Juvenile	0.305^{ns}	0.530^{a}	T2	Juvenile	0.714^{c}	0.636^{b}
	Mature	0.219^{ns}	0.366^{a}		Mature	0.517^{c}	0.543^{c}
C3	Juvenile	0.014^{ns}	0.535^{a}	T3	Juvenile	0.438^{a}	0.401^{ns}
	Mature	0.480^{b}	0.079^{ns}		Mature	0.251^{a}	0.370^{c}
C4	Juvenile	0.035^{ns}	0.153^{ns}	T4	Juvenile	0.301^{ns}	-0.057^{ns}
	Mature	0.030^{ns}	0.608^{c}		Mature	0.326^{c}	0.432^{c}
C5	Juvenile	0.389^{ns}	0.333^{ns}	T5	Juvenile	0.407^{ns}	0.043^{ns}
	Mature	0.190^{ns}	0.277^{ns}		Mature	0.008^{ns}	0.083^{ns}
Mean	Juvenile	0.644^{b}	0.742^{c}	Mean	Juvenile	0.372^{ns}	0.747^{c}
	Mature	0.081^{ns}	0.438^{b}		Mature	0.212^{a}	0.383^{c}

RW, ring width; LW%, latewood content; SG, specific gravity; ns, non-significant
[a]significant at 5% level
[b]significant at 1% level
[c]significant at 0.1% level

gravity was non-significant in juvenile wood of teak discs bearing number C4, C5, T1, T3, T4 and T5 except C1, C2, C3 and T2. However, there was a significant positive correlation at 1 and 0.1% level for the mean value of all five trees in juvenile and mature wood from both the sites. A significant positive correlation was also found at 5 and 0.1% level in mature wood of all individual trees, except C3, C5 and T5.

The relationship between ring width and specific gravity has been the subject of debate in the scientific studies. In some cases, few authors accounted for a significant positive correlation between ring width and specific gravity (Bhat and Indira 1997; Sinha et al. 2014), whereas several authors have reported a negative or non-significant correlation between these traits (Varghese et al. 2000; Moya 2002; Jayawardana and Amarasekera 2009; Nocetti 2011).

Correlation Between Latewood Content and Specific Gravity

The associationship between mean latewood content and mean specific gravity of five teak trees from pith to periphery of Chandrapur (C1–C5) and Thane (T1–T5) is shown in Fig. 3. The association of radial variation between latewood content and specific gravity was comparable to that of association observed between ring width and specific gravity. Correlation coefficients between latewood content and specific

gravity of five trees and their mean values in juvenile and mature wood from both the sites are presented in Table 3. It is evident from t test that the correlation between latewood proportion and specific gravity in juvenile wood of teak discs bearing number C2, C3, C4, C5, T1, T4 and T5 was not significant. Conversely, the correlation was significant at 0.1% level in T2 and 5% level in C1 and T3, respectively.

It is found that there was a significant positive correlation between latewood content and specific gravity in mature wood of all five trees at moist site, except T5, which was non-significant while, there was no significant correlation between these traits in trees studied from dry site except C3 which showed a significant positive correlation at 1% level. However, the mean value of all trees at dry site showed a positive correlation at 1% significant level in juvenile wood and it showed non-significant correlation in mature wood. At moist site, the correlation was non-significant in juvenile wood, while it was positively correlated in mature wood at 5% significant level. These results are in conformity with the findings of Zobel and van Buijtenen (1989) who reported that in wide growth rings, the proportion of latewood with small vessels increases and vice versa which may influence the specific gravity variations in juvenile and mature wood of a ring-porous hardwood.

It is revealed from the current investigation that the specific gravity of all individual trees showed a poor associationship with ring width and latewood proportion in juvenile wood, whereas a strong associationship was observed in the mature wood of most of the individual trees at both the sites. However, there was a strong relationship between mean ring width and mean specific gravity in juvenile and mature wood of all studied trees, whereas the latewood content did not show strong relationship with specific gravity.

Conclusion

The mean values of ring width, latewood proportion and specific gravity were high in the juvenile wood of trees at both dry and moist sites. In spite of more latewood proportion and wider growth ring width of *Tectona grandis* at dry site, the higher specific gravity is found at moist site. It shows that specific gravity, in addition to latewood proportion and ring width, also depends on the quality of latewood. Pith to periphery variations of ring width, latewood content and specific gravity is more intrinsic in the juvenile wood than mature wood because of cambial ageing. Consequently, it is suggested to consider the variations of individual tree while selection for high specific gravity during tree breeding programme rather than mean variation of all the trees at specific site.

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Variability for Heartwood Content in Three Commercially Important Tree Species of Peninsular India—*Hardwickia binata, Pterocarpus santalinus* and *Santalum album*

A.N. Arun Kumar, Geeta Joshi and S. Manikandan

Abstract In most of the tree improvement programs of highly valued wood tree species, generally, height and diameter are the two important factors considered while selecting superior genotypes. However, it is now being realized and accepted that it is essential to include various wood traits while selecting superior genotypes for breeding strategies. We are of the opinion that it is necessary to also include the heartwood content as another important trait because of its commercial value. As a prelude to consider heartwood content as one of the selection traits for tree improvement programs, it becomes imperative to document the variation in this trait in a given population. Information about heartwood formation, progression, and genetics is being studied globally, and preliminary indications are that it is genetically controlled, but the role of environmental factors is also equally important. In this paper, heartwood content variation in known aged plantation and its relationship with tree girth in three important indigenous highly valued tree species—*Hardwickia binata* (Anjan), *Pterocarpus santalinus* (Red Sanders) and *Santalum album* (Indian Sandalwood)—is discussed. Significant variation was observed in young-aged plantations in all the three species.

Keywords Heartwood · Variation · *Hardwickia binata* · *Pterocarpus santalinus* · *Santalum album*

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Introduction

Documenting variability is an important aspect before initiating any strategies in tree improvement work. As a first step, growth traits such as tree diameter, height, and clear bole height are considered. These are supported with some morphological traits such as leaf, fruit, and seed. Generally, the most common practice while selecting superior genotypes was restricting to these morphological traits. However, it is now being realized to essentially incorporate wood traits during selection process. Specific gravity has been considered as an important wood-quality trait that is considered to be heritable and having impact both on wood and on pulp quality. However, in some of those tree species having commercial value for timber applications, the heartwood component of the tree can influence its utilization as it forms an important characteristic of internal stem structure (Knapic and Pereira 2005). Therefore, it becomes necessary to document the variation in heartwood content. However, an important rider while considering heartwood as a trait for selection process is the tree age as heartwood can be significantly affected by the age.

The International Association of Wood Anatomists (IAWA) has defined heartwood as "the inner layers of the wood, which, in the growing tree, have ceased to contain living cells and in which the reserve materials (e.g., starch) have been removed or converted into heartwood substance" (IAWA 1964). In essence, heartwood is the dead part of the tree and is located in the older part of the stem, as an inner core that approximately follows the stem profile. It is physiologically inactive, without water-conducting properties, and contains a large amount of resinous and phenolic extractives with biotic resistance.

The living part of the tree is known as sapwood and is defined by IAWA (1964) as "the portion of the wood that in the living tree contains living cells and reserve materials." Sapwood has high moisture content, containing reserve material such as starch, and plays an important role in the transpiration stream of the tree (Taylor et al. 2002). It primarily conducts water from the roots to the crown (Gartner 1995), and there is a strong correlation between amount of foliage and sapwood in a tree (Berthier et al. 2001; Dean and Long 1986; Whitehead et al. 1984). The heartwood and the sapwood are distinctly different based on features such as color, natural durability, and suitability for chemical treatment (Panshin and de Zeeuw 1980; Haygreen and Bowyer 1989).

Relatively, most of the work on heartwood and its variation has been extensively carried out in temperate species and conifers. However, some of the tropical species that have been studied are Acacias and Eucalypts, primarily for paper pulp where heartwood is considered to be a deterrent factor due to color and extractive contents in the heartwood. In the case of tree species, even though the presence of heartwood has high technological relevance, the physiological aspects of it are still not clearly understood.

The present study aims at documenting heartwood variation within a known aged population in the case of three important tropical species—*H. binata*, *P. santalinus* and *S. album*, which are popular tree species in India.

Hardwickia binata (Family: Fabaceae) commonly known as Anjan is a moderate- to large-sized deciduous, gregarious tree and is native to tropical Southeast Asia. The tree attains height up to 36 m and girth of 4 m having a clean cylindrical bole of 12–15 m. *H. binata* is monotypic tree genera (Garad et al., 2015), widely distributed in the dry deciduous forests of Peninsular India and North India, excluding Northeast India (Irwin and Narasimhan 2011). *H. binata* is a typical species of dry and hot climate characterized by long period of drought, scanty to moderate rainfall, and intense heat during the summer season. Within its natural range, the minimum and maximum temperature may vary from 10 to 47 °C and the rainfall may vary from 250 to 1500 mm (Anon 1983). The wood is extremely hard and heavy, fairly durable and moderately strong, and highly resistant to decay and classified as class I timber. The heartwood is dark reddish brown streaked with purple and is clearly differentiated from sapwood which is pale white in color. It is one of the timbers accepted by Indian Railways for sleepers even without treatment. The wood has high calorific value (4900 k cal/kg) and is used for charcoal making. The leaves are classed as good fodder, and the tree is popular now in agroforestry.

Pterocarpus santalinus (Family: Fabaceae) is a deciduous tree commercially known as Red Sanders. As an endemic tree of India, its natural distribution being restricted to tropical dry deciduous forests of Andhra Pradesh being localized and predominantly confined to Cuddapah landscape (Raju et al. 1999). In its natural habitat, the tree girth varies from 90 to 160 cm and height reaching up to 15 m with dense round crown. Red Sanders grows in harsh conditions as is evident by the minimum and maximum temperature varying from 11 to 46 °C and the rainfall being as low as 100–1000 mm. The heartwood color is red to almost purplish black and the wood is strong, hard, and heavy with dried wood-specific gravity ranging from 0.80 to 1.20. Wenbin and Xiufang (2013) reported that in China Red Sanders wood is valued as gold and costs around US$ 150,000/m^3.

Santalum album (Family: Santalaceae) is an evergreen tree commercially known as Indian Sandalwood is known world over for its highly valued heartwood from which fragrant essential oil is distilled. The oil has earned a sobriquet as “queen of essential oil.” Sandalwood is naturally distributed from 30°N to 40°S, from Indonesia in the east to Juan Fernandez Islands (Chile) in the west and from Hawaiian Archipelago in the north to New Zealand in the south (Srinivasan et al. 1992). Natural population of sandalwood in India had been predominantly found in southern part of Karnataka and northern part of Tamil Nadu. It has also been found in the states of Kerala and Andhra Pradesh. Isolated populations have been reported in various states such as Bihar, Gujarat, Haryana, Maharashtra, Madhya Pradesh, Orissa, Punjab, Rajasthan, Uttar Pradesh, West Bengal, Himachal Pradesh and Assam which might have been introduced.

Materials and Methods

To document the heartwood variation in *H. binata, P. santalinus* and *S. album*, plantations were selected preferably of 20 years so as to capture the maximum variation. In the case of *H. binata*, a 20-year-old plantation established by Karnataka Forest Department in Madapaura, Bagalkote Range, Bagalkote Division was selected. For *P. santalinus* also a 20-year-old plantation established by Karnataka Forest Department in Jharakbande A Block, Research Range Office, Bengaluru, was considered. In the case of *S. album,* a 20-year-old germplasm bank of sandalwood established by the Institute of Wood Science and Technology at Gottipura Research Station, Hoskote, ~30 kms from Bengaluru, was selected.

Sample plots were randomly laid out in *H. binata* and *P. santalinus* plantation, and all the trees within the sample plot were considered for recording the field data as well as collection of core samples. The number of sample plots varied depending on the plantation area and tree availability; however, care was taken such that observation was recorded from a minimum of 100 trees. In the case of *S. album*, three ramets from each accession were selected, and from 37 accessions, a total 111 ramets were considered for the study. During the collection of core samples in *H. binata,* it was found that in trees of girth size <40 cm, heartwood formation had not been initiated. Therefore, for the present study, trees greater than 40 cm were generally considered for collecting the core samples.

Tree girth was measured at breast height using a measuring tape and expressed in centimeter. To estimate the bark, sapwood, and heartwood thickness, core samples were drawn using increment borer (Haglof). Two core samples from each tree were drawn at breast height at right angles to each other in order to obtain correct length of heartwood, sapwood, and bark thickness. By converting the tree girth into radius using the formula $2\pi r$ (where $\pi = 3.1415$ and r = radial length), the measurement of heartwood radius was calculated. Similarly, bark and sapwood thickness was also calculated.

Results

The average girth of *H. binata* trees ($n = 100$) selected for the study was 58.87 cm and the girth ranged from 43 to 86 cm. It is evident from Table 1 that there is a considerable variability in the case of radial heartwood thickness (CV = 48.68%). The highest heartwood content was 68.09%, and the lowest heartwood content of 6.35% was found in a tree of 52 cm girth. Among the trees from which core samples were collected ($n = 100$), heartwood had not formed in five trees of girth sizes, 43, 48, 49, 53, and 58, respectively. There was a strong positive correlation

Table 1 Summary of statistics on girth, bark, sapwood, and heartwood thickness (%) for *H. binata* trees of 20 years old in Madapura plantation ($n = 95$[a])

Parameters	Girth (cm)	Bark thickness (%)	Sapwood thickness (%)	Heartwood thickness (%)
Range	43–86	6.18–20.56	21.10–89.76	6.35–68.09
Mean	58.87	10.26	53.70	36.02
Standard deviation	2.68	2.63	16.80	17.54
CV (%)	15.82	25.64	31.29	48.68

[a]Five trees which did not have heartwood have been excluded

Table 2 Correlation between girth and wood traits in 20-year-old trees of *H. binata*

	Girth	Bark thickness	Sapwood thickness	Heartwood thickness
Girth	1			
Bark thickness	−0.63[a]	1		
Sapwood thickness	−0.45[a]	0.39[a]	1	
Heartwood thickness	0.74[a]	−0.57[a]	−0.98[a]	1

[a]Significant at $p < 0.01$

Table 3 Summary of statistics on girth, bark, sapwood, and heartwood thickness (%) for *P. santalinus* trees of 20 years old in Jharakbande plantation ($n = 98$[a])

Parameters	Girth (cm)	Bark thickness (%)	Sapwood thickness (%)	Heartwood thickness (%)
Range	32–79	11.26–33.79	22.86–78.08	3.36–65.71
Mean	49.84	18.67	45.68	35.66
Standard deviation	9.76	4.06	12.35	12.54
CV (%)	19.58	21.74	27.01	35.16

[a]32 trees which did not have heartwood have been excluded

between girth and heartwood thickness suggesting that higher the tree girth, the more would be the heartwood thickness (Table 2).

In the case of *P. santalinus*, the average girth was 49.84 cm and the heartwood thickness varied from 3.36 to 65.71% with a CV value of 35.16% (Table 3). Out of the 130 trees selected for the study, in 98 trees heartwood had formed. Out of the 32 trees in which heartwood had not formed, 12 trees were within the girth class of 30–40 cm, 17 trees were in 40–50 cm girth class, and three trees in 50–60 cm girth class. There was a strong positive relationship between girth and heartwood thickness (Table 4).

Considerable variation was observed in *S. album* for girth and wood traits. Heartwood thickness had maximum coefficient of variation value (33.63%) and it

Table 4 Correlation between girth and wood traits in 20-year-old trees of *P. santalinus*

	Girth	Bark thickness	Sapwood thickness	Heartwood thickness
Girth	1			
Bark thickness	−0.41[a]	1		
Sapwood thickness	−0.43[a]	−0.12	1	
Heartwood thickness	0.56[a]	−0.21	−0.95[a]	1

[a]Significant at $p < 0.01$

Table 5 Summary of statistics on girth, bark, sapwood, and heartwood thickness (%) for *S. album* trees of 20 years old in Hoskote germplasm bank ($n = 96$[a])

Parameters	Girth (cm)	Bark thickness (%)	Sapwood thickness (%)	Heartwood thickness (%)
Range	26–56	5.32–13.96	22.44–81.95	12.75–78.93
Mean	36.97	5.41	54.06	40.58
Standard deviation	6.34	1.60	13.14	13.64
CV (%)	17.14	29.57	24.30	33.63

[a]15 trees which did not have heartwood have been excluded

Table 6 Correlation between girth and wood traits in 20-year-old trees of *S. album*

	Girth	Bark thickness	Sapwood thickness	Heartwood thickness
Girth	1			
Bark thickness	−0.08	1		
Sapwood thickness	0.46[a]	−0.19	1	
Heartwood thickness	0.63[a]	−0.06	−0.38[a]	1

[a]Significant at $p < 0.01$

varied from 12.75 to 78.93% (Table 5). Out of 111 trees, heartwood had not formed in 15 trees in sandalwood. There was a strong positive linear relationship between girth and heartwood ($r = 0.64$) (Table 6).

Discussion

Heartwood is one of the important components in determining wood quality in most of the commercially important tree species. Panshin and Zeeuw (1980) reports that those species that are less efficient in utilizing food products through photosynthesis have the tendency to initiate the early formation of heartwood while it is the other

way in the case of species which are efficiently utilizing the photosynthates as food. However, formation of heartwood and sapwood especially in hardwoods varies with species, location, and season, and little is known about the variation in heartwood and the factors associated with it especially in most of the tropical tree species except for teak and eucalypts. Three commercially important tree species of India, *H. binata*, *P. santalinus* and *S. album,* were studied for understanding the variation especially in growth and heartwood. Age plays a significant role in heartwood formation. For better comparison, in the present study, age of the trees were kept constant which was twenty years in all the three species. There was considerable variability in girth among all the three species, and the CV values were >15%.

Among the three species, bark thickness was the highest in the case of *P. santalinus* but the CV value was less compared to the other two species. CV values for bark thickness varied between 21.74 and 29.57%. Heartwood thickness was maximum in sandalwood (78.93%) compared to *P. santalinus* (65.71) and *H. binata* (68.09%). Heartwood formation starts much earlier in the case of sandalwood and it is reported to start by the end of seventh or eighth year. However, not much information is available for the other two species. But the CV value was more or less similar in the case of sandalwood (33.63%) and Red Sanders (35.16%). Though sandalwood grows well in adjoining area of Bengaluru, being an endemic species, growth of Red Sanders in Bengaluru seems to be encouraging considering the extent of heartwood formation. However, maximum variability in heartwood thickness was recorded in the case of *H. binata* (CV = 48.68%) which may be attributed to the stress condition in which it was growing as the area is prone to high temperature with scanty rainfall. Heartwood and sapwood formations of living trees, especially in hardwoods, vary with species, site, parts of the tree, and season and water distribution inside the stem. Björklund (1999) while identifying heartwood-rich stands of *Pinus sylvestris* found that heartwood content varied considerably between individual trees and between stands. It was found that the variation between trees within the same diameter at breast height (DBH) class, growing in the same stand, was higher than the variation between stands. Wide range of variability was observed for heartwood content in different half-sib progenies of *Eucalyptus tereticornis* (Kumar and Dhillon 2014). The heartwood content increases with tree age and various authors found evidence that, after a certain initiation age, heartwood is formed at a constant annual ring rate especially in the case of temperate species (Hazenberg and Yang 1991; Wilkes 1991; Sellin 1994; Björklund 1999; Gjerdrum 2003). In the case of tropical tree species *Tectona grandis*, Moya et al. (2014) reported that some of the factors that play an important role in heartwood properties of trees when grown as fast growth plantations are tree age, longitudinal variation, geographic location, environmental conditions and silvicultural activities.

Tree girth and diameter have always been considered as an important trait in plantation management. While selecting genotypes for superior traits it would always be easy if tree girth can be considered as one of the criteria. Therefore, relationship between tree girth and sapwood/heartwood contents if any would facilitate not only in identifying superior genotypes, but also in applying suitable

management practices so that tree girth is enhanced. In the present study, there was positive linear relationship between tree girth and heartwood in *H. binata* ($r = 0.74$), *P. santalinus* ($r = 0.56$), and *S. album* ($r = 0.63$). Strong positive genetic and phenotypic correlations of heartwood diameter were found with stem DBH and with heartwood proportion in *Eucalyptus globulus* (Miranda et al. 2009, 2014). Positive variations in tree growth and heartwood dimensions have been reported in *Larix decidua* (Leibundgut 1983), *E. globulus* (Gominho and Pereira 2000), while stem diameter was a good predictor of heartwood diameter in *Pinus pinaster* (Pinto et al. 2004; Knapic and Pereira 2005) and (Climent et al. 2003), suggesting that larger trees have higher heartwood diameter. Nocetti et al. (2010) found significant differences between clones of *Prunus avium* for growth and heartwood traits which indicate that they are moderately genetic controlled. Similarly, moderate to high genetic control has been reported in black walnut (Rink 1987; Woeste 2002). Björklund (1999) was of the opinion that to obtain stems richer in heartwood, genetical breeding plays an important role than through silviculture and site selection. The variation in heartwood and sapwood content is essential in understanding within tree wood trait variability especially for breeding purposes. But, at the same time, the clear correlation between girth and heartwood paves way for early selection of superior genotypes.

Conclusions

The present study clearly demonstrates that considerable variability occurs in heartwood content at the age of 20 years in trees of *H. binata, P. santalinus* and *S. album*. As age plays a significant role in heartwood formation, selection of genotypes based on heartwood at young age would be apt as the variability would be at maximum and option for better selection would be higher. A positive linear relationship between girth and heartwood content was found in all the three species which would also assist in quicker selection process.

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Relations Between Growth Traits and Wood Parameters of *Tectona grandis* L.f. in Even-Aged Plantations in Tamil Nadu

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Abstract In the present study, the relation of stand growth parameters with the basic wood properties of the *Tectona grandis* L.f. in southern and western zone of Tamil Nadu was studied. The results revealed highly significant correlation between diameter and area of heartwood in southern zone ($r = 0.86$) and western zone ($r = 0.91$). There was no significant correlation between wood density and diametrical growth in both the zones. There was significant positive correlation between tree height and heartwood area. The result indicates that faster growth is associated with higher heartwood content and lower sapwood proportion disproving the general notion that faster-grown trees will always have greater sapwood content. This affirms that the heartwood production in the teak plantations is predictable from diameter measurement.

Keywords *Tectona grandis* · Heartwood · Sapwood · Diameter · Height · Age · Correlation

Introduction

Teak (*Tectona grandis* L.f.), a tropical timber species, is native to Indo-Malayan region. This multipurpose timber has favourable strength properties besides having resistance to termite and fungal attack by the presence of polyphenols. It has been described as one of the most durable timbers of the world. Teak is highly useful timber, which cannot be eclipsed by any other timber. Traditional use of teak poles for electricity transmission and timber for railway sleepers are a time-tested

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testimony of its suitability for outdoor uses. The persistent demand and continued shortfall of its availability make it one of the dearest species in the tropics. The natural teak forests of India are confined to peninsular India below 24°N latitude (Seth and Khan 1958). The most suitable soil for teak is deep and well-drained alluvium, with an optimum pH range of 6.5–8.0 and relatively high content of calcium and phosphorous (Seth and Yadav 1959). Teak grows from sea level to an altitude of 1200 m with precipitation range of 800–2500 mm. On a global basis, the total area under teak plantation extends to 5.79 million ha, of which 42.9% is in India (ITTO 2009).

In teak, the amount and distribution of heartwood and sapwood is an important determinant of wood value (Wiemann et al. 2002), and there is an increasing interest in heartwood yield of plantations (Bhat 1999; Climent et al. 2002). Of all the wood properties, density is the most significant in determining the end use and wood density is strongly correlated with strength properties of wood (Zobel and van Buijtenen 1989). In the present study, the relation of stand growth parameters with the basic wood properties of the *T. grandis* in southern and western zone of Tamil Nadu was studied.

Materials and Methods

Study Sites

Based on rainfall, temperature, soil type and other ecological conditions, seven agro-climatic zones have been identified in the state of Tamil Nadu in India (Anon 1993). Out of seven zones, the present study was carried out in two agro-climatic zones, viz. southern and western zones of Tamil Nadu which lie in Tirunelveli and Coimbatore forest divisions. Twenty sample plots of size 20 × 20 m were laid out in each age group, and all the trees were measured for girth at breast height (GBH). The whole girth range was divided into five different girth classes. Various measurements such as total height, basal girth, mid girth were recorded from the felled sample trees. Wood samples were collected from the sample trees and studied for wood properties such as density, heartwood (HW) and sapwood (SW) content. Density was estimated by adopting weight/volume method. Heartwood and sapwood thicknesses were measured, and HW/SW percentages were determined by area method (Bhat 1999). Pearsons's correlation, regression and other statistical analyses were performed using SPSS statistical software. Analysis of variance (ANOVA) was performed to test significant differences in the mean values of various parameters (Panse and Sukhatme 1985).

Results and Discussion

Southern Zone

Descriptive statistics on heartwood and sapwood area, percentage of heartwood and sapwood, and density for the 40 trees sampled in southern zone of Tamil Nadu, India, are summarized in Table 1. The heartwood area at breast height of sample trees ranged from 76.9 to 1157.5 cm^2, having the mean value of 449.7 cm^2. The sapwood area ranged from 8.0 cm^2 to 226.8 cm^2 with a mean value of 100.4 cm^2. The heartwood percentage ranged from 66.7 to 95.3, whereas the sapwood content varied from 4.7 to 33.3% of total disc area. By considering all the plantations (age ranging from 25 to 47 years) studied in this zone, the mean percentage values for heartwood and sapwood were 81.6 and 18.4, respectively. The sapwood percentage was found to have greater variation with coefficient of variation of 37% than the heartwood (CV = 9%). With regard to air-dry wood density, the minimum and maximum values recorded are 0.65 and 0.83 g/cc, respectively. The mean wood density was 0.73 g/cc. Variability in wood density was found to be lesser (CV = 9.4%) as compared to other wood traits (heartwood area, sapwood area and sapwood percentage).

Western Zone

Table 2 presents descriptive statistics on various parameters for the 35 trees sampled in western zone. While considering all the plantations studied, the heartwood area ranged from 50.2 to 776.5 cm^2 (mean value of 346 cm^2) and sapwood area ranged from 60.9 to 283.3 cm^2 (mean value of 121.8 cm^2). The percentage of heartwood ranged from 45.2 to 90.8. The mean percentage values for heartwood and sapwood were 71.5 and 28.5, respectively, with age ranging from 20 to 34 years. It was observed that sapwood percentage exhibited greater variation (CV = 33%) than the heartwood (CV = 14%). The wood density ranged from 0.65

Table 1 Descriptive statistics on properties of wood in teak plantations of southern zone

Characteristics	DBH (cm)	HW area (Sq cm)	SW area (Sq cm)	HW (%)	SW (%)	Density (g/cc)
Mean	25.93	449.7	100.4	81.6	18.4	0.73
Standard error	0.96	38.80	9.50	1.10	1.10	0.009
Standard deviation	6.07	245.0	59.8	7.1	7.1	0.059
CV %	23.40	54.5	59.9	8.8	37.2	9.4
Minimum	14.65	76.9	8.0	66.7	4.7	0.65
Maximum	42.65	1157.5	226.8	95.3	33.3	0.83

Table 2 Descriptive statistics on properties of wood in teak plantations of western zone

Characteristics	DBH (cm)	HW area (Sq cm)	SW area (Sq cm)	HW (%)	SW (%)	Density (g/cc)
Mean	24.04	346.0	121.8	71.5	28.5	0.72
Standard error	0.91	29.98	9.13	1.63	1.63	0.006
Standard deviation	5.38	177.4	54.0	9.7	9.7	0.038
CV %	22.38	51.3	44.3	13.7	33.1	5.2
Minimum	12.5	50.2	60.9	45.2	9.2	0.65
Maximum	34.1	776.5	283.3	90.8	54.8	0.79

to 0.79 g/cc with a mean value of 0.72 g/cc. Similar to southern zone, variability in wood density was lesser compared to other parameters in western zone.

Between the zones, heartwood proportion was higher in trees of southern zone (82% of total disc area) than in western zone (72% of total disc area). However, there was no significant difference in the mean wood density (0.01).

Relations Between Growth and Wood Parameters

Diameter and Wood Properties

In southern zone, dbh exhibited highly significant positive correlation with heartwood area ($r = 0.864$) and sapwood area ($r = 0.717$) and non-significant correlation with wood density ($r = 0.26$) (Table 3), whereas in western zone, the correlation was found to be highly significant between dbh and heartwood area ($r = 0.91$) (Table 4). The correlation between dbh and area of sapwood was also observed to be significant, but with moderate *r*-value of 0.64. However, there was significant correlation between wood density and dbh ($r = 0.48$).

Tree Height and Wood Properties

In southern zone, the correlation analysis revealed statistically significant positive relation of height with heartwood area ($r = 0.60$) and a moderate correlation with sapwood area ($r = 0.49$). On the contrary, there existed non-significant correlation between wood density and height ($r = 0.17$) (Table 3). The observations on axial variation revealed that the heartwood area gradually decreased from bottom (587–642 cm^2) to top (13–48 cm^2) height of the trees, while heartwood percentage ranged from 74 to 88 at the base and from 22 to 43% in the top of the trees. Sapwood percentage gradually increased up to 3/4th of tree height (ranging from 30 to 78%). The observations also revealed highly significant variations in wood density at

Table 3 Correlation coefficient (r) for various growth parameters and mean heartwood and sapwood area in teak plantations of southern zone

Parameters	Age (yr)	DBH (cm)	Height (m)	Sapwood area (cm^2)	Heartwood area (cm^2)
Age (yr)	1				
DBH (cm)	0.173	1			
Height (m)	0.143	0.754*	1		
Sapwood area (cm^2)	0.188	0.717*	0.493*	1	
Heartwood area (cm^2)	0.203	0.864*	0.599*	0.371	1
Wood density (g/cc)	0.051	0.259	0.171	0.071	0.279

*Correlation is significant at the 0.001 level

Table 4 Correlation coefficient (r) for various growth parameters and mean heartwood and sapwood area in teak plantations of western zone

	Age (yr)	DBH (cm)	Height (m)	Sapwood area (cm^2)	Heartwood area (cm^2)
Age (yr)	1				
DBH (cm)	0.490*	1			
Height (m)	0.172	0.651*	1		
Sapwood area (cm^2)	0.154	0.631*	0.040	1	
Heartwood area (cm^2)	0.437*	0.913*	0.580*	0.078	1
Wood density (g/cc)	0.511*	0.476*	0.043	0.206	0.494

*Significant at p value of 0.05

different heights of the trees. In general, wood density decreased from base (ranging from 0.66 to 0.71 g/cc at base) to the top (ranging from 0.58 to 0.62 g/cc at top). The trend in wood density at different heights indicated a slight decrease up to 1/4th of the tree height from base and thereafter gradual increase up to 3/4th height and subsequently a sharp decline at the top 1/4th height of the trees (Fig. 1).

In western zone, the results of correlation analysis showed a statistically significant relation of height with heartwood area ($r = 0.58$), but a non-significant correlation with sapwood area ($r = 0.04$). Likewise, there existed non-significant correlation between wood density and height ($r = 0.043$) (Table 4). The observations on axial variation revealed that area of heartwood gradually decreased from bottom (225–446 cm^2) to top (10–63 cm^2) of the trees. Correspondingly, heartwood content ranged from 76 to 79% at the base and from 22 to 39% at the top of the trees. SW% gradually increased up to 3/4th of tree height (varying from 22 to 32%) and rapidly increased at the top 1/4th height (varying from 40 to 79%). With reference to axial variation, wood density decreased from base (0.71–0.74 g/cc) to the top (0.64–0.66 g/cc). In this zone, there was a slight decrease in wood density

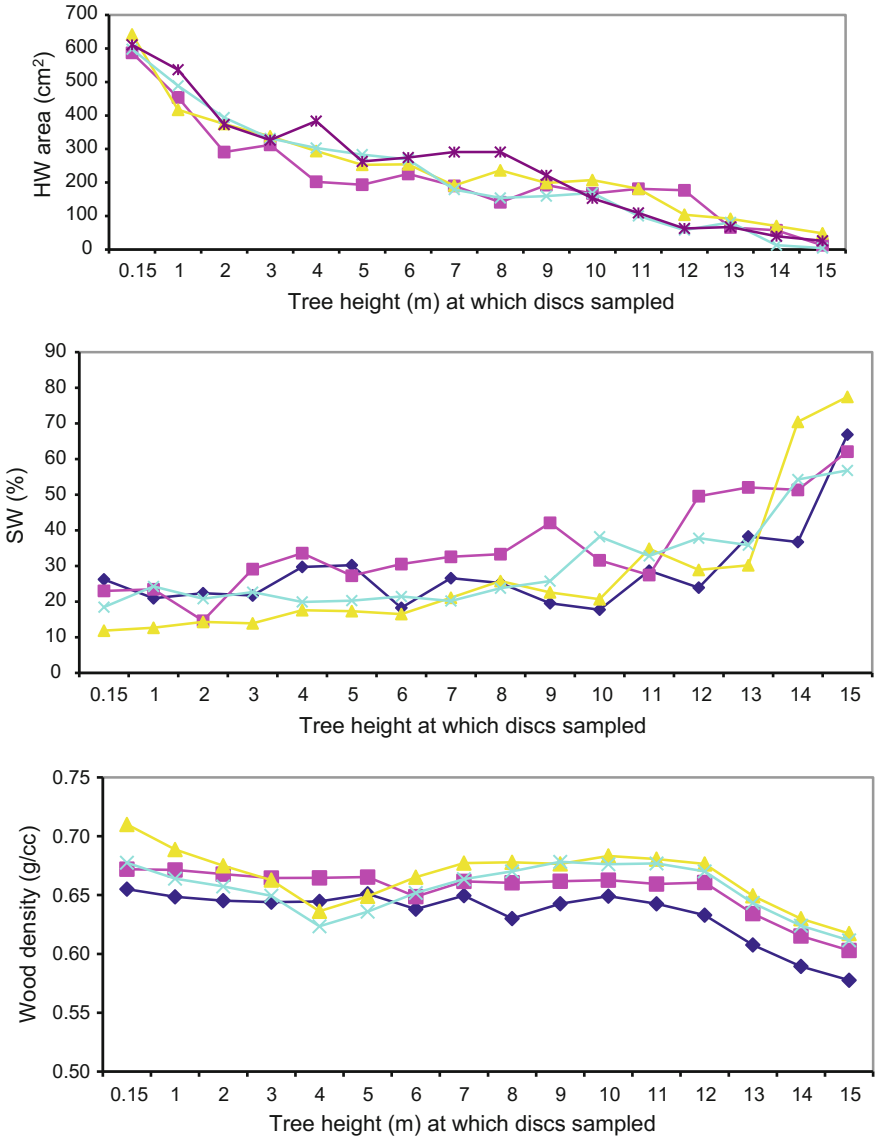

Fig. 1 Axial variation in heartwood area (cm^2), sapwood (%) and wood density (g/cc) at different heights of four representative trees in southern zone

from base up to 3/4th height followed by sharp decrease at the top 1/4th height of the trees (Fig. 2).

Within a tree, the axial variation in wood density was statistically significant in both southern and western zones (Tables 5 and 6).

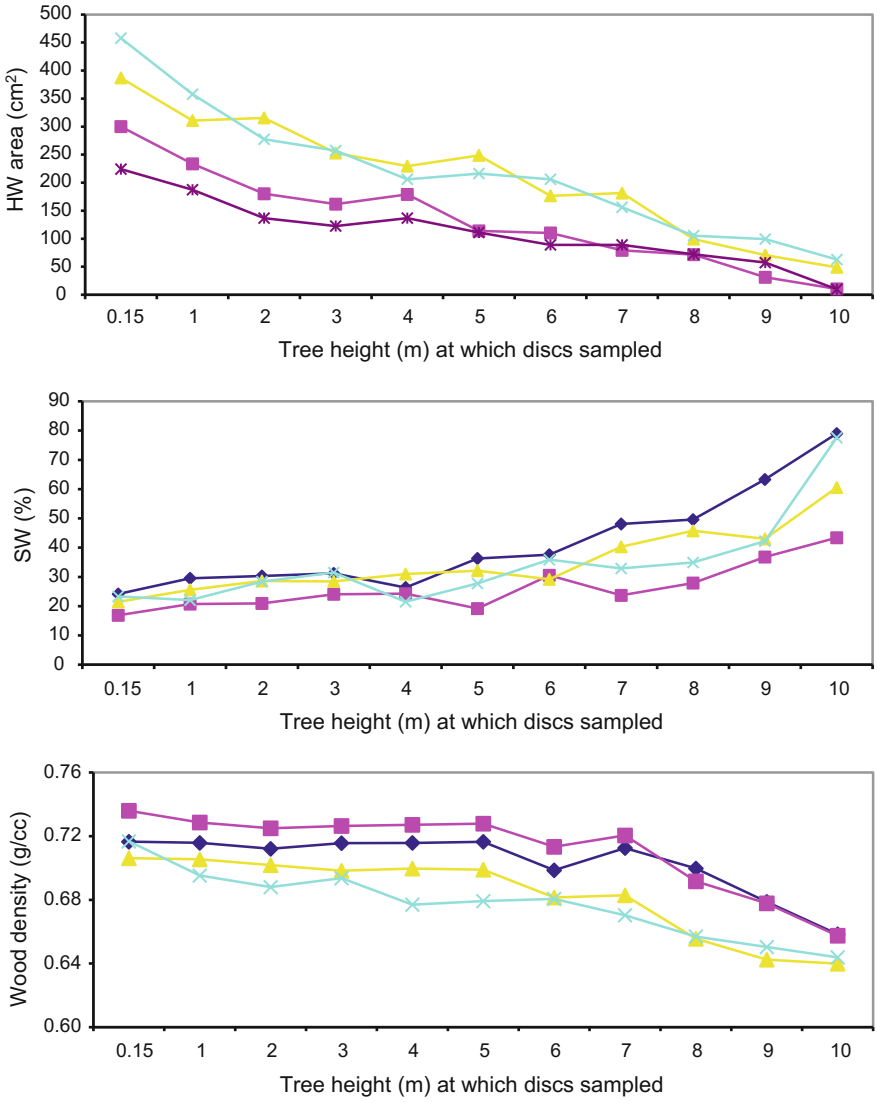

Fig. 2 Axial variation in heartwood area (cm^2), sapwood (%) and wood density (g/cc) at different heights of four representative trees in western zone

Age and Wood Properties

The age of plantation in southern zone ranged from 25 to 47 falling in eight age groups, and the correlation was made between age and area of sapwood and heartwood. It is noted that the correlation was non-significant and the observed

Table 5 *F*-value from ANOVA test for wood density in teak plantations at different heights of samples trees in southern zone

Wood traits	Source of variation	df	Sum of squares	Mean square	*F*-value	Statistical significance
Density	Within tree	15	0.0247	0.0016	15.8	Significant at the 0.01 level
	Between trees	3	0.0084	0.0028	26.7	
	Error	45	0.0047	0.0001		
	Total	63	0.0378			

Table 6 *F*-value from ANOVA test for wood density in teak plantations at different heights of samples trees in western zone

Wood traits	Source of variation	df	Sum of squares	Mean square	*F*-value	Statistical significance
Density	Within tree	10	0.0189	0.0019	27.4	Significant at the 0.01 level
	Between trees	3	0.0095	0.0032	46.1	
	Error	30	0.0021	0.0001		
	Total	43	0.0305			

r-values for heartwood and sapwood area were 0.203 and 0.188, respectively (Table 3). There was a non-significant correlation between age and density as well in southern zone ($r = 0.05$).

The age of plantations in western zone ranged from 21 to 34, falling in seven age groups. A significant correlation was observed between age and heartwood area ($r = 0.44$), whereas it was non-significant between age and sapwood area ($r = 0.15$) (Table 4).

However, ANOVA indicated statistically significant differences among different age groups with reference to heartwood area and wood density in both the zones (Tables 7 and 8). This significant variation observed among plantations may be brought out by the locality factors rather than age as such.

Table 7 *F*-value from ANOVA test for wood density in teak plantations of different age groups in southern zone

Wood traits	Source of variation	df	Sum of squares	Mean square	*F*-value	Statistical significance
Heartwood area	Age of the plantations	7	1,402,093	200,299	6.83	0.00005
	Error	32	939,119	29,347		Significant at the 0.01 level
	Total	39	2,341,213			
Density	Age of the plantations	7	0.075	0.011	3.14	0.0121
	Error	32	0.109	0.003		Significant at the 0.01 level

Table 8 *F*-value from ANOVA test for wood density in teak plantations of different age groups in western zone

Wood traits	Source of variation	df	Sum of squares	Mean square	*F*-value	Statistical significance
Heartwood area	Age of the plantations	6	386,858	64,476	2.64	0.037
	Error	28	682,800	24,386		Significant at the 0.01 level
	Total	34	1,069,658			
Density	Age of the plantations	6	0.038	0.0063	16.00	0.0000
	Error	28	0.011	0.0004		Significant at the 0.01 level
	Total	34	0.049			

The mean values observed with reference to heartwood in the present study are comparable with that reported for teak by Bhat et al. (1985) that is 76.6% at 51–52 years. The present results are also in tune with that reported by Bhat (2001) who recorded 58–65% of heartwood at age between 13 and 21 years and 85–88% at age between 55 and 65 years in teak plantations from Nilambur, Kerala. While comparing the wood traits observed in southern zone with that in western zone, it is found that on an average basis, the heartwood produced was in greater proportion in trees of southern zone (82%) than in western zone (72%). For the purpose of comparison, plantations of similar ages in these two zones were selected and the overall mean values were worked out. While comparing heartwood percentage in teak plantations of two zones studied at comparable ages (from 30 to 35 years), mean heartwood percentage for southern zone (83%) was greater than that of western zone (76%). This difference could be attributed to the eco-climatic variation between the zones.

Observation on variation among trees in wood traits in the present study is well within the range reported by Bhat (2001) who recorded CV range of 6.5–13.5% for specific gravity in teak. This result is also in consonance with Bhat et al. (1990) who observed CV values ranging from 6.6 to 8.9% for wood density in *Eucalyptus grandis* plantation. Varghese et al. (2000) also reported low CV (6.2%) for wood density in samples tested across nine plantations of teak in various locations from peninsular India.

With reference to correlation and regression relations of growth traits with wood properties, Todorovski (1966) reported that stem diameter was one of the first variables used to predict heartwood diameter in *Pinus sylvestris* and *Pinus nigra*. Ihara (1972) also observed highly significant correlation between heartwood diameter and stem diameter for *Cryptomeria japonica*. Bhat (1995) also observed statistically significant positive correlation between heartwood and breast height diameter in teak plantation and inferred that faster growth is associated with higher heartwood content and lower sapwood proportion disproving the general notion that faster-grown timber always has higher sapwood content. Mukerji and Bhattacharya (1963) reported that the correlation between the rate of diametrical

growth (in terms of number of rings per 2.5 cm) and specific gravity is not statistically significant in teak.

With regard to relations between height and wood traits, Nicholls and Matheson (1980) also reported that there were significant differences in heartwood between 7-year-old trees of *Eucalyptus obliqua* and those showing better height growth which have a greater proportion of heartwood. Bhat et al. (1990) observed that wood density is not significantly related to height in *Eucalyptus grandis* when the age of the plantations was between three and seven years.

Axial variation in wood density was studied in selected trees in both zones. The observations revealed that there exist highly significant variations in wood density at different heights of trees. In general, wood density decreased from base to the top in both southern and western zones. Sekhar and Negi (1966) studied variation of strength properties along the length of stem in teak and they found that strength properties were higher at the bottom. However, the differences in wood density in actual values were relatively small. On average, wood density was 0.7 at the base and 0.63 at the top of trees.

Conclusions

The present study confirms a significant correlation between tree diameter and heartwood content and affirms that the heartwood production in the teak plantations is predictable from diameter measurement. The study also corroborates with earlier studies that increased diametrical growth owing to genetics or environmental factors will not bring change in heartwood production. On the other hand, the proportion of sapwood production is influenced by site factors. Specific gravity remains unaltered in teak after the reported wood maturation age of 20 years and beyond.

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Variation in Heartwood Formation and Wood Density in Plantation-Grown Red Sanders (*Pterocarpus santalinus*)

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Abstract *Pterocarpus santalinus* L. f. (Red Sanders) is narrowly endemic to the Seshachalam, Veligonda, Lankamala and Palakonda hill ranges in Andhra Pradesh. The wood and wood products of Red Sanders continue to be in high demand and are traded internationally in large volumes that find use in the musical instruments, furniture, handicrafts, cosmetics, medicine and food industry. Over exploitation without commensurate replenishment of natural stands and illegal logging has posed a severe threat to the very existence of this precious timber species and classified as globally threatened in the IUCN Red List. Good-quality heartwood of Red Sanders is illegally traded and fetches very high price in the global market. As harvest of heartwood from natural population may not be sustainable, any future plan of harvest of Red Sanders wood and export should be from cultivated sources. About 5000 ha of plantations of Red Sanders exists in various states in South India. Little information is available about the quality and quantity of heartwood formation in plantations compared to natural populations. In this backdrop, the current study was conducted to evaluate the variation in heartwood, sapwood and bark content, and wood density of plantation-grown Red Sanders trees of various age classes located in various places. The core wood samples from various locations were collected using increment borer based on standard sampling procedure. The heartwood, sapwood and bark content were measured as a percentage of the cross-sectional area at breast height. Wood density was determined using core wood samples taken at breast height of the tree. The variation in heartwood content and wood density of Red Sanders were found to be influenced by the age and size of the trees.

Keywords Red sanders · Plantation · Heartwood content · Wood density

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Introduction

Pterocarpus santalinus L. f. (Red Sanders) is a narrowly endemic species naturally distributed over an area of about 0.4 million ha mainly in the hilly tracts in Chittoor, Cuddappah, Nellore, Prakasam and Kurnool districts of Andhra Pradesh (IFGTB 2011). The species occurs in 'Dry Red Sanders-Bearing Forest' which is classified as '5A/C2 Forest Type' (Champion and Seth 1968). Red Sanders is highly valued for its heavy, dark claret-red heartwood which yields 16% of red coloring matter to santalin, a natural dye used to color liquors, pharmaceutical preparations, food stuffs, etc. (Ramakrishna 1962; Anuradha and Pullaiah 1998). It is renowned for its characteristic timber of exquisite color, beauty and superlative technical qualities and ranks among the finest luxury wood in the world which is highly useful for making high-value musical instruments, cabinets, ornamental veneers, toys, dolls and was used in incense.

The Red Sanders is under severe threat in its native range, owing to its endemism to small area, poor regeneration and overexploitation due to illegal felling. Despite a ban on felling and sale, good-quality Red Sanders are illegally traded since they fetch very high price in the global market. Based on the grain type, the Red sanders trees may be divided into two types in commerce, viz. wavy-grained and straight-grained which occur mixed in the forest or plantations (Lohidas and Dayanand 1984; Dayanand and Lohidas 1988). The wavy-grained variants are preferred in the global markets and fetch higher price compared to normal-grained types. State Forest Departments of Tamil Nadu, Kerala and Andhra Pradesh have raised Red Sanders plantations in several locations other than its natural ranges during last 50 years. A few number of tree growers and farmers are also cultivating this species in farm land in Andhra Pradesh, Tamil Nadu, Orissa and Karnataka and in other states, and about 3000 ha of Red Sanders plantations currently exists (IFGTB 2011).

Though growth rate is reported to be faster under plantations raised in rich soils compared to natural population (ICFRE 1992), not much information is available regarding growth rate of the species according to location, climate and soil outside its natural range. The heartwood is the main economic part of the tree in Red Sanders. Information regarding extent of formation of heartwood and quality of heartwood in Red Sanders plantations compared to its natural range is also not available. In this backdrop, the current study was conducted to evaluate the variation in heartwood, sapwood and bark content, and wood density of plantation-grown Red Sanders trees of various age classes grown in various locations across southern India.

Materials and Methods

Reconnaissance survey was carried out to find various age class plantations across various regions in Tamil Nadu, Andhra Pradesh, Karnataka and Kerala states in southern India. Various age class plantations available were visited for collection of

growth as well as wood data during 2011–2014. Totally, 27 plantations were selected from 9 forest divisions (Table 1). Sampling of the selected plantations was done based on the total area of the plantation with at least 3% sampling intensity. Sample plots of size either 50 m × 50 m or 20 m × 20 m (in small size plantations) dimensions were laid. Quantitative and qualitative growth data of trees, such as diameter at breast height (DBH), total tree height, clear bole height, branching habit, stem form, health, phenology and wood grain pattern, were collected in all the sample plots laid. Red Sanders trees possess characteristic thick dead outer bark (dead corky tissue) that is blackish brown in color resembling the skin of crocodile. The cracks are divided into rectangular plates by deep vertical and horizontal

Table 1 Details of Red Sanders plantations sampled and climatic factors of their locations

District/state	Plantations	Age (years)	Min temp (°C)	Max temp (°C)	Rainfall (mm)	Dry months
Tiruvannamalai, Tamil Nadu	Vallimalai	28	22.6	32.7	1022	7
	Allikuttai	32	23.1	33.1	1071	7
	Arani	39	23.1	33.2	1031	7
	Kasthambadi	49	23.1	33.2	1063	7
	Thaniyar	94	21.6	31.8	1007	7
Vellore, Tamil Nadu	Banavaram-1	23	23.3	33.3	988	7
	Banavaram-2	24	23.3	33.3	988	7
	Banavaram-3	28	23.3	33.3	988	7
	Rettapettai	32	23.0	33.1	973	7
	Kalpudur	40	22.8	32.9	961	7
	Nelakanrayanpet	41	23.1	33.2	967	7
	Kalmelkuppam	54	23.0	33.1	973	7
	Ammoor	56	23.0	33.1	973	7
Tiruvallur, Tamil Nadu	Nayapakkam	31	23.7	33.5	1102	6
	Pullarampakam	36	23.8	33.5	1099	6
	Avajipetai	37	23.8	33.5	1101	6
	Nemalur	37	23.9	33.6	1112	7
	Malanthur	41	23.8	33.5	1101	6
	Vengal	42	23.8	33.5	1101	6
	Mylapore	47	23.8	33.5	1101	6
Andhra Pradesh	Tirupathi	30	22.1	32.2	870	7
Kadapa, Andhra Pradesh	Kodur-1	67	23.2	33.3	840	9
	Kodur-2	78	23.2	33.3	840	9
Tumkur, Karnataka	Ankapura	15	19.1	30.1	575	10
Bengaluru, KA	Jarakabande	26	18.1	29.4	774	9
Thrissur, Kerala	Palappilly	31	23.6	31.5	2950	4
Ernakulam, Kerala	Kodanad	31	23.7	31.5	3230	4

fissures. The inner bark lies next to outer bark. To study the variation in heartwood formation, wood samples were collected from these plantations using increment borer by laying 10 m × 10 m subplots within all the sample plots and the samples were taken back in airtight container to the laboratory at the Institute of Forest Genetics and Tree Breeding (IFGTB), Coimbatore. Heartwood length, sap wood length, bark thickness and wood density were analyzed from these wood samples, and the difference in heartwood content and wood density among various plantations was studied. The heartwood, sapwood and bark content were measured as a percentage of the cross-sectional area at breast height. The core wood samples were dried and weighed, and the wood density was calculated as the ratio of oven-dried weights to green volume. Various climatic data like rainfall, minimum and maximum temperatures pertaining to locations of plantations were obtained through DIVA-GIS to compare these site factors with tree growth and wood parameters measured.

Results and Discussion

The age of surveyed plantations varied from 15 to 94 years across various locations. Details of plantations in which sample plots were laid out for testing heartwood formation, growth data of sampled trees and proportion of sampled trees with heartwood formation are provided in Table 2. The growth data and wood data collected from sampled trees were also grouped according to the age classes (Table 3). Out of 27 plantations, only five plantations were more than 50 years of age. The majority of the plantations surveyed occurred in the age classes of 21–30, 31–40 and 41–50 years. Number of trees sampled in 27 plantations (sample plot size 10 M × 10 M) varied from 2 to 108 based on the size of the plantation area and also stocking of trees. Some of the locations like Kalpudur, Rettapettai and Ammoor in Tiruvannamalai had large areas of plantation and good stocking; therefore, the number of sample plots was increased. Generally, the Red Sanders trees attain medium height of 8–10 meters in plantations as well as in natural locations. However, in some of the high rainfall locations like Palappilly and Kodanad in Kerala, the average tree height reached 16–18 m at 31 years age. In some of the very old plantations like Thaniyar in Vellore Division (94 years age) and Kodur in Andhra Pradesh (78 years), the average tree height was high (Table 2). The average girth at breast height (GBH) varied from 52.0 (in 15-year-old plantation) to 160.63 cm (in 94-year-old Thaniyar plantation).

The growth data collected in sample plots of 50 m × 50 m size (data not shown here) revealed that the GBH slightly increased linearly with the age. However, the average GBH did not always linearly increase with the age because of location effects in some of the plantations. For instance, in some plantations of 21- to 30-year girth class, the GBH was higher than that in 31–40 girth class plantations. In case of Kodur plantations situated at Andhra Pradesh which was above 60 year old, trees had lower girth than those in few of the 28-year-old plantations.

Table 2 Details of sample plots laid for testing heartwood formation, growth data of sampled trees and proportion of sampled trees with heartwood formation

Location	Plantation	Age (years)	No. of sample plots	No. of trees sampled	Average GBH (cm)	GBH range (cm)	Average height (m)	Height range (m)	Percent sampled trees with heartwood formation
Tiruvannamalai Tamil Nadu	Vallimalai	28	3	17	103.5	76–136	10.86	10–12	57.14
	Allikuttai	32	3	9	67.94	48–85	10.67	6–13.8	100.00
	Arani	39	4	29	55.96	21–118	11.52	6–16.5	79.17
	Kasthambadi	49	3	11	106.11	84–137	11.49	9–14	100.00
	Thaniyar	94	3	7	160.63	116–200	16.96	10–23.5	100.00
Vellore Tamil Nadu	Banavaram-1	23	3	11	100.00	70.0–122.0	10.9	8.0–14	100.00
	Banavaram-2	24	3	13	95.46	72–134	13.79	10.0–16	100.00
	Banavaram-3	28	3	8	85.25	70–122	10.56	9.0–12	100.00
	Rettapettai	32	13	66	67.22	40–98	9.64	7–12.8	100.00
	Kalpudur	40	15	108	55.64	35–81	10.61	6.5–13.5	96.30
	Nelakanrayanpet	41	4	19	61.28	34–96	8.56	4.0–12	100.00
	Kalmelkuppam	54	6	19	110.79	94–146	10.96	6.5–14.5	100.00
	Ammoor	56	6	36	115.29	64–188	12.19	8.5–16	100.00
Tiruvallur Tamil Nadu	Nayapakkam	31	3	9	52.38	37–78	9.59	5.5–15	100.00
	Pullarampakam	36	3	7	69.79	57–82	10.76	4.5–15	100.00
	Avajipetai	37	3	8	61.16	37–74	10.13	4–15	100.00
	Nemalur	37	3	8	61.44	38–92	9.8	4.5–13.8	75.00
	Malanthur	41	3	9	114.00	91–148	9.38	5–14	100.00
	Vengal	42	3	9	114.00	94–130	10.30	6–14.6	100.00

(continued)

Table 2 (continued)

Location	Plantation	Age (years)	No. of sample plots	No. of trees sampled	Average GBH (cm)	GBH range (cm)	Average height (m)	Height range (m)	Percent sampled trees with heartwood formation
	Mylapore	47	3	13	87.92	46–130	10.71	7–13.5	100.00
Andhra Pradesh	Tirupathi	30	3	11	68.05	13–84	9.81	4.5–15	53.85
	Kodur-1	67	1	4	76.78	57–95	11.44	7–16	100.00
	Kodur-2	78	1	5	91.94	61–115	19.89	7–25	100.00
Karnataka	Ankapura	15	1	3	52.00	19–57	12.1	6–15.5	66.67
	Jarakabande	26	1	2	59.75	39–78	9	7–13	100.00
Kerala	Palappilly	31	1	4	73.50	53–110	16.53	6–27	100.00
	Kodanad	31	1	4	96.25	60–132	18.47	11.5–26	100.00

Table 3 Age class-wise mean DBH, bark content, sap wood, heartwood content and wood density in sampled trees in Red Sander plantations

Age class (in years)	No. of plantations	Av. DBH (cm)			Av. Bark content (%)			Av. Sapwood content (%)			Av. Heartwood content (%)			Av. Wood density		
		Mean	Std. Dev	Range	Mean	Std. Dev	Range	Mean	Std. Dev	Range	Mean	Std. Dev	Range	Mean	Std. Dev	Range
<20	1	16.56	–	–	49.76	–	–	48.72	–	–	1.52	–	–	0.868	–	–
21–30	6	27.18	5.70	19.03–32.96	33.71	6.78	27.18–46.53	34.35	10.27	24.91–50.06	31.94	12.56	17.41–47.90	0.865	0.070	0.776–0.961
31–40	10	21.06	4.01	16.68–30.65	41.11	8.10	27.11–50.0	35.06	2.49	31.30–38.29	23.83	8.11	15.69–40.87	0.888	0.095	0.760–1.03
41–50	5	30.79	7.15	19.52–36.51	33.70	6.66	28.29–44.03	29.13	3.99	24.71–33.68	37.18	10.07	23.13–45.53	0.841	0.088	0.753–0.970
51–60	2	36.00	1.02	35.28–36.72	30.67	1.15	29.86–31.49	28.33	3.58	25.80–30.86	49.49	7.27	44.35–54.63	0.880	0.009	0.874–0.886
>61	3	34.96	14.23	24.45–51.16	25.94	2.79	22.76–27.98	24.55	4.64	19.56–28.53	49.51	7.18	44.20–57.68	0.940	0.064	0.871–0.996

Therefore, it is evident that location factors and probably density of tress in plantations (stocking) had effect on GBH.

The proportion of sampled trees with heartwood is also given in Table 2. In Tiruvannamalai Forest Division, Vallimalai (28 years) and Arani (39 years), 57.14 and 79.12% of trees sampled had heartwood, respectively. In Vellore Division, in Kalpudur (40 years age) 96.30% trees sampled had heartwood. In rest of the plantations, all the sampled trees had heartwood. In Tiruvallur Division, in Nemallur (37 years) plantation only 75% trees had heartwood. Out of 2 plantations visited in Karnataka, in one plantation at Anakupura, Tumkur (15 years), 66.67% trees sampled had heartwood. The heartwood development was found in 15-year-old trees, indicating that heartwood in Red Sanders trees is initiated before 15 years of age. High tree-to-tree variation within plantations was found for height, girth and heartwood content.

The average bark, sapwood and heartwood area expressed as percent of total cross-sectional area of stem at breast height in the sampled trees across age classes is provided in Table 3. The mean bark proportion was higher in lower girth class plantations, i.e., below 40-year plantations as compared to plantations above 60 years old. Heartwood proportion ranged between 1.52% in 15-year-old plantation to 57.68% in 94-year-old plantation. Sapwood proportion decreased with the age. The above 60-year-aged plantations had an average of 24.55% sapwood. Generally, trees with higher DBH had high heartwood content even when they occurred in lower age classes. Average wood density ranged from 0.841 to 0.940 across different age classes (Table 3). Considerable variation was also observed in heartwood content and wood density within trees based on age, growth and location. Heartwood proportion increased slightly with increasing age, while sapwood gradually decreased.

Location-wise and plantation-wise bark, sapwood and heartwood proportions and wood density are given in Table 4. Considerable variation in wood density in sampled trees across plantation was observed. Average wood density was relatively lower (0.760 and 0.790, respectively) in Kodanad and Palappilly plantations which are located in high rainfall areas of Kerala. However, some of the plantations located in drier areas of Tamil Nadu, for example, Malanthur (0.753), Vengal (0.787), Vallimalai (0.776) and Mylapore (0.805) had relatively lower wood density. The Nayapakkam (1.03) and Pullarampakkam (1.01) had higher average wood density which was above 1.0. Very high variations were observed among trees within same plantations for wood density, heartwood, sapwood and also bark contents. The average bark and sapwood proportions were higher in lower age class plantations and also in trees with lower girth. The heartwood content was generally high in higher aged plantations. However, all three plantations in Banavaram which were aged 23–28 years characteristically had higher heartwood proportions (36.93–47.90%). Two 31-year-old Palappilly and Kodanad plantations also had higher heartwood proportions (34.11–40.87%). This was mainly due to higher average DBH of trees. These plantations had higher DBH than many of the older plantations.

Table 4 Variation in bark, sapwood, heartwood content and density with location of Red Sanders

Location	Plantation	Age (years)	Avg. DBH (cm)	Av. Bark content (%)	Av. Sap Wood (%)	Av. Heart Wood (%)	Heartwood % range	Av. Basic density	Basic density range (min–max)
Tiruvannamalai Tamil Nadu	Vallimalai	28	32.96 (1.35)	30.78 (1.14)	43.70 (2.86)	25.53 (2.71)	7.95–39.80	0.776 (0.024)	0.632–0.951
	Allikuttai	32	21.64 (1.43)	40.90 (3.14)	36.64 (2.83)	22.46 (4.90)	3.5–43.19	0.862 (0.015)	0.767–0.902
	Arani	39	17.82 (1.40)	50.00 (2.89)	34.31 (1.85)	15.69 (2.97)	0.00–51.91	0.864 (0.015)	0.751–1.071
	Kasthambadi	49	33.79 (1.90)	28.39 (1.43)	28.23 (1.25)	43.28 (2.23)	31.17–52.73	0.888 (0.024)	0.755–1.003
	Thaniyar	94	51.16 (3.69)	22.76 (1.67)	19.56 (2.45)	57.68 (3.70)	38.91–68.92	0.871 (0.035)	0.747–1.006
Vellore Tamil Nadu	Banavaram-1	28	31.85 (1.45)	27.18 (1.88)	24.91 (2.73)	47.90 (3.79)	24.25–69.60	0.839 (0.029)	0.611–0.983
	Banavaram-2	24	30.4 (1.53)	30.31 (1.53)	26.44 (1.73)	43.26 (2.95)	28.19–70.28	0.832 (0.022)	0.692–0.954
	Banavaram-3	23	27.15 (1.27)	34.95 (1.48)	28.12 (2.65)	36.93 (2.94)	25.81–50.43	0.846 (0.030)	0.754–1.027
	Rettapettai	32	21.41 (0.57)	37.24 (1.04)	37.21 (0.76)	25.56 (1.28)	1.22–46.04	0.802 (0.009)	0.660–1.097
	Kalpudur	40	17.72 (0.31)	43.68 (0.71)	38.29 (0.74)	18.03 (0.92)	0.01–44.53	0.842 (0.006)	0.667–1.116
	Nelakanrayanpet	41	19.52 (1.16)	44.03 (1.48)	32.84 (1.50)	23.13 (2.12)	2.39–40.84	0.970 (0012)	0.816–1.037
	Kalmelkuppam	54	35.28 (1.07)	31.49 (1.19)	30.86 (2.48)	37.65 (2.81)	9.31–53.78	0.874 (0.024)	0.650–1.025

(continued)

Table 4 (continued)

Location	Plantation	Age (years)	Avg. DBH (cm)	Av. Bark content (%)	Av. Sap Wood (%)	Av. Heart Wood (%)	Heartwood % range	Av. Basic density	Basic density range (min–max)
	Ammoor	56	36.72 (1.31)	29.86 (1.050)	25.80 (1.25)	44.35 (1.62)	24.06–62.97	0.886 (0.012)	0.786–1.054
Tiruvallur Tamil Nadu	Nayapakkam	31	16.68 (1.39)	48.47 (3.95)	35.75 (1.13)	15.79 (4.59)	0.004–40.26	1.030 (0.03)	0.930–1.200
	Pullarampakam	36	22.22 (1.12)	49.88 (2.61)	31.30 (3.32)	18.82 (3.92)	10.83–34.54	1.010 (0.03)	0.880–1.160
	Avajipetai	37	19.48(1.43)	45.41 (3.77)	32.02 (1.94)	22.57 (4.16)	1.86–33.93	0.960 (0.03)	0.820–1.060
	Nemalur	37	19.57 (2.24)	39.16 (5.07)	36.42 (3.26)	24.42 (7.21)	0.01–53.07	0.953 (0.014)	0.897–1.005
	Malanthur	41	36.31 (6.27)	28.29 (2.38)	26.18 (1.43)	45.53 (1.84)	36.97–54.15	0.753 (0.019)	0.622–0.799
	Vengal	42	36.31 (4.76)	31.31 (2.40)	24.71 (2.63)	43.98 (4.05)	22.72–64.02	0.787 (0.035)	0.638–0.969
	Mylapore	47	28 (1.95)	36.46 (2.79)	33.68 (1.62)	29.86 (3.34)	4.62–46.01	0.805 (0.028)	0.693–0.969
Andhra Pradesh	Tirupathi	30	21.67 (0.96)	32.53 (1.56)	50.06 (1.63)	17.41 (2.40)	0.28–32.76	0.935 (0.02)	0.779–1.048
	Kodur-1	67	24.45 (2.46)	27.07 (1.05)	28.73 (2.43)	44.20 (3.42)	35.71–51.72	0.952 (0.05)	0.790–1.027
	Kodur-2	78	29.28 (3.16)	27.98 (0.84)	25.36 (2.09)	46.66 (1.72)	42.97–51.7	0.996 (0.03)	0.936–1.11
Karnataka	Ankapura	15	16.56 (2.12)	49.76 (4.5)	47.38(3.33)	0.70 ((1.19)	0.0–3.86	0.868 (0.013)	0.850–0.893

(continued)

Table 4 (continued)

Location	Plantation	Age (years)	Avg. DBH (cm)	Av. Bark content (%)	Av. Sap Wood (%)	Av. Heart Wood (%)	Heartwood % range	Av. Basic density	Basic density range (min–max)
	Jarakabande	26	19.03 (2.79)	46.53 (8.08)	32.88 (6.38)	20.59 (14.46)	6.13–35.05	0.961 (0.038)	0.922–0.999
Kerala	Palappilly	31	23.41 (4.02)	34.11 (4.17)	36.66(2.2)	34.11 (4.17)	23.08–41.3	0.79 (0.03)	0.718–0.849
	Kodanad	31	30.65 (5.10)	27.11 (4.53)	32.02(2.40)	40.87 (4.28)	29.29–48.87	0.760 (0.04)	0.683–0.848

Relationship Among Growth, Wood Characters and Location Factors of Plantations

Pearson's correlation studies for growth and wood characters in Red Sander plantations sampled are given in Table 5. Tree age had significantly high positive correlation with (DBH) ($r = 0.572$) and heartwood proportion ($r = 0.602$) and significant negative correlation with sapwood and bark proportions ($r = -0.574$ and -0.469). DBH was positively correlated with heartwood (0.837), and wood density had moderate negative correlation with rainfall ($r = -0.393$) and DBH ($r = -0.406$) and was positively correlated with bark proportions ($r = 0.449$).

Perez et al. (2004) studied the heartwood content in several age class plantations of *Bombacopsis quinata*, a high-valued timber species of Costa Rica. No clear differences in heartwood content could be observed between trees of similar age from dry and wet zones. Heartwood content increased with age of the trees. Arun Kumar (2011) studied growth and heartwood content variability in two Red Sanders plantations of age 20 and 45 years in Karnataka and concluded that considerable variability exists for girth and heartwood content in the younger aged plantation. It was found that heartwood formation had not been initiated in trees less than 30-cm girth, and there was a strong relationship between girth and heartwood in both the plantations. Heartwood initiation was observed in 70% of the trees in 20-year-old plantation and 97% of the trees in the case of 45-year-old plantation.

To summarize the results, it was observed that in all the plantations there was high variability among trees within plantations for growth characteristics, bark, sapwood and heartwood content. There were no clear-cut variations in heartwood content and growth of Red Sander trees across locations, indicating climatic factors

Table 5 Correlations among growth and wood characters in Red Sander plantations sampled

	Age (years)	Rainfall (mm)	Diameter at breast height (DBH)	Bark proportion (%)	Heartwood proportion (%)	Sapwood proportion (%)
Age (years)	1					
DBH	0.572*	0.067 ns	1.000			
Bark proportion (%)	−0.469*	−0.276 ns	−0.777*	1.000		
Heartwood proportion (%)	0.602*	0.181 ns	0.837*	−0.884*	1.000	
Sapwood proportion (%)	−0.574*	−0.013 ns	−0.650*	0.473*	−0.831*	1.000
Basic wood density	0.202 ns	−0.393*	−0.406*	0.449*	−0.299 ns	0.028 ns

*P <0.01

may have less influence on the heartwood formation and content in Red Sanders trees. DBH had highly significant positive relationship with heartwood content ($r = 0.837$) irrespective of age. The general belief is that faster the tree grows, lesser will be the heartwood content. However, the results in the present study indicated that the higher growth rate actually resulted in higher content of heartwood. Although plantations located in high rainfall areas of Kerala had lower wood densities, it was not very clear that there were differences in wood density among trees of similar age from dry and wet zones because some of the plantations located in drier areas of Tamil Nadu also had similar wood densities. It is also contrary to general belief that the Red Sanders trees grown in wet zones possess lower wood density compared to those grown in dry areas. The results indicated that the Red Sanders trees can be successfully grown in varied eco-geographic and climatic conditions outside its natural ranges which are entirely different from those present in its natural locations. The tree-to-tree variability for growth and heartwood content within a plantation indicates that there is scope for selection and genetic improvement for these characteristics. The heartwood proportion analysis across age classes of plantations indicated that Red Sanders trees can safely be harvested for moderate amount of heartwood in 40- to 50-years rotation age in plantation conditions. Generally, the trees planted singly or in rows along the boundaries attain good girth at lower age. The heartwood content can be increased, and thereby, rotation age can be reduced, provided higher growth rate is achieved in plantations. Too high growth rate may also have adverse impact on wood density because wood density had moderate negative correlation with DBH. Genetic improvement in Red Sanders needs to be initiated for high heartwood content, higher wood density and higher growth rate to reduce the rotation age and also to increase the productivity.

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Identification and Characterization of Tension Wood in *Acacia auriculiformis* A. Cunn. ex Benth. and *Acacia mangium* Willd. Grown in Kerala

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Abstract The present study was carried out to characterize the tension wood and normal wood in *Acacia auriculiformis* and *Acacia mangium* and to analyse their variation in fibre and vessel morphology. The fibre morphological characters included fibre length and diameter, fibre lumen diameter and fibre wall thickness, and those of vessels included vessel diameter and frequency. Wood blocks of 1 cm^3 were obtained from the base billets of felled Acacia trees, and microtome sections were prepared. In order to distinguish tension wood and normal wood, sections were stained using toluidine blue-'O'. In order to obtain fibre dimensions, the maceration was carried out with Jeffry's fluid. The results revealed that the anatomical properties, viz., fibre morphology and vessel morphology, were found to vary between tension wood and normal wood. The fibre morphology of both the species followed the similar pattern in which magnitude of all the fibre properties was higher in tension wood than in normal wood. Vessel morphology of both the species followed the similar trend; however, frequency and diameter of the vessels were much lesser in tension wood than in normal wood. The present study distinctly proved the occurrence of tension wood in *Acacia auriculiformis* and *Acacia mangium*. In order to reduce the occurrence of tension wood or to prevent its occurrence, better silvicultural and management practices could be adopted, such as closer spacing, and early and proper pruning.

Keywords Fibre morphology · Vessel morphology · Tension wood · *Acacia auriculiformis* · *Acacia mangium*

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Introduction

Acacia mangium and *Acacia auriculiformis* are two tropical species of the genus *Acacia* which are having an ability to grow in a wide range of soils and poor growing conditions (Grieve and Hellmuth 1970). These are being introduced to degraded tropical and subtropical regions to establish forest communities (Norisada et al. 2005; Peng et al. 2005; McNamara et al. 2006; Yang et al. 2009). In the Indian context, these two Australian acacias are being planted on a large scale, especially in the southern states viz. Kerala, Karnataka and Tamil Nadu. They are widely used for timber, fuel wood and soil improvement purposes (Turnbull 1986; Shanavas and Kumar 2006). *A. auriculiformis* is reported to be one of the most promising tree species for plantation forestry and reforestation (Pinyopusarerk et al. 1993) and *A. mangium* for paper and pulp manufacture in the tropical environment. In angiosperms, when trees have to restore their verticality after some accidental leaning (or partial uprooting), they will produce a rather wide sector of reaction wood known as tension wood on the upper side (Badia et al. 2006). Due to the usual higher growth rate in the reaction wood sector, the total volume of reaction wood in a log can be significant (Constant et al. 2003; Timell 1986). Tension wood is one of the major defects on the plantation-grown timber species that adversely affects the quality of timber, and the occurrence of tension wood restricts its versatile utilization for various applications. Since both *A. auriculiformis* and *A. mangium* are grown in plantations under short rotation, the chances of occurrence of tension wood due to growth stresses are high. Hence, the present investigation was formulated to characterize the tension wood and normal wood (non-tension wood) in *A. auriculiformis* and *A. mangium* and to analyse their variation in fibre and vessel morphology using histochemical staining and Image Analysis techniques.

Materials and Methods

The wood specimens used in the present study were collected from 9-year-old *A. auriculiformis* and *A. mangium* trees which were planted in the instructional farm of College of Forestry, Kerala Agricultural University, at Vellanikkara, Thrissur (10° 32′N latitude, 76°16′E longitude), Kerala, India, established in August 1996 as part of the provenance trial. The trees were planted at a spacing of 3 m × 3 m. Wood discs of 6 in thickness were cut out from the base of the felled logs, and blocks of 1 cm^3 were obtained from the discs. From this block, specimens of about 1 cm^2 cross section were traced out for sectioning using a wood microtome (Leica SM2000R). These specimens were boiled in water in order to soften them for easy sectioning. Sections (T.S) of 10–15 μm thickness were cut out from the specimens using a wood microtome. The histochemical staining of tension wood fibres was carried out using toluidine blue-'O' (General purpose; pH < 4.5) for distinguishing

tension wood (non-lignified) from normal wood (lignified). The stain was prepared by dissolving 5 gm citric acid, 6 gm sodium citrate, 5 gm toluidine blue-'O' in 100 ml distilled water. After staining, the sections were thoroughly washed with water to remove excess stain and then passed through the TBA/xylene series. This series, composed of five solutions of TBA (tertiary butyl alcohol) and xylene, is graded like pure TBA, TBA/xylene in 3:1, 1:1, 1:3 and pure xylene. The specimens were mounted in DPX to make permanent slides and observed under microscope, and the tension wood fibres (S_3G layer) were coloured as violet/rose partially lignified as light blue and normal wood (lignified) as blue (Plates 1 and 2).

For extraction of wood fibres, radial chips or wood shavings were taken from wood blocks. They were boiled with the macerating fluid (Jeffrey's solution) in order to separate individual fibre, and the dimensions of fibres were recorded after staining in toluidine blue-'O'. Anatomical features of the normal wood zone and tension wood zone like fibre length, fibre diameter, fibre lumen diameter, fibre wall thickness, vessel diameter and vessel frequency were recorded. Paired t test was conducted on the data obtained, using the statistical software SPSS 16.

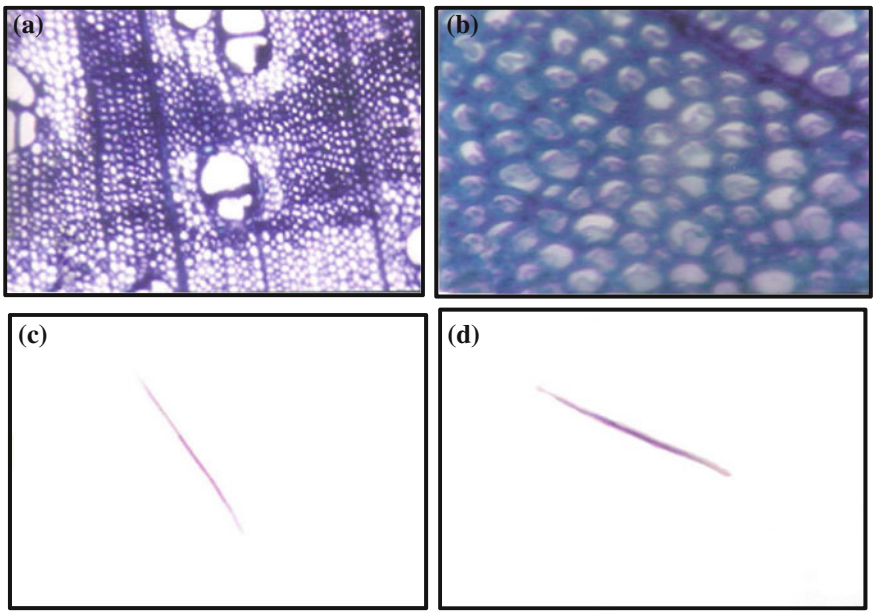

Plate 1 Transverse sections and individual fibres showing difference in tension wood and normal woods in *A. auriculiformis*. **a** T.S (×100) showing compact tension wood zone. **b** T.S (×100) showing gelatinous fibres. **c** Individual fibre from tension wood zone. **d** Individual fibres from normal wood zone

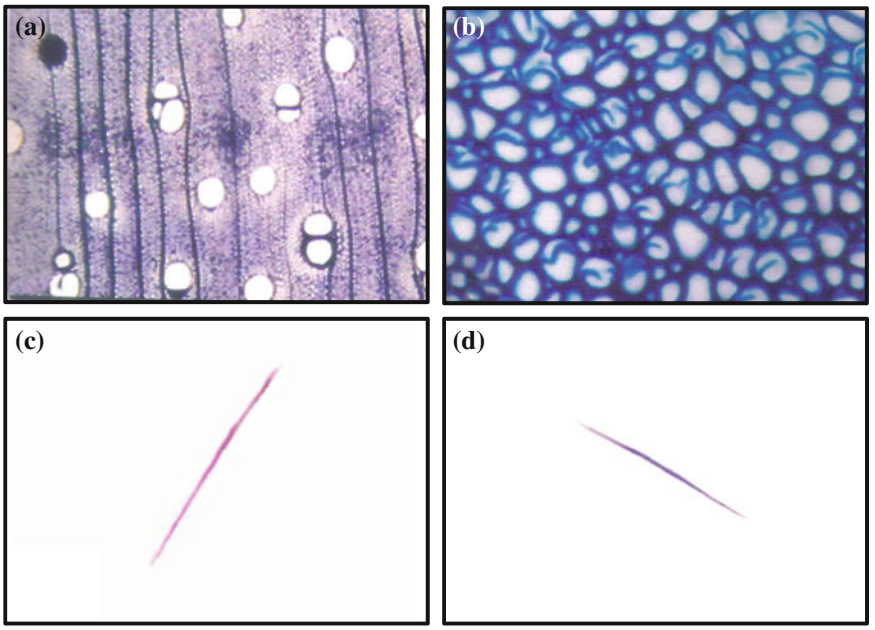

Plate 2 Transverse sections and individual fibres showing difference in tension wood and normal wood in *A. mangium*. **a** T.S (×100) showing diffuse tension wood zone. **b** T.S (×100) showing gelatinous fibres. **c** Individual fibre from tension wood zone. **d** Individual fibres from normal wood zone

Results and Discussion

Variation in fibre and vessel morphological characters of tension wood and normal wood (non-tension wood) of *A. auriculiformis* and *A. mangium* is given in Table 1. All the fibre morphological characters such as fibre length, fibre width, fibre lumen width and fibre wall thickness and vessel morphological characters such as vessel diameter and vessel frequency showed significant variation (at 1% level) between both tension wood and normal wood in both species. All the fibre morphological characters were higher in tension wood as compared to normal wood. Vessel morphological characters were found to be higher in normal wood. According to Zobel and Van Buijtenen (1989), fibres in tension wood are much longer than in normal wood. The variation in vessel morphology of both types of wood in both species is in line with earlier studies in which the number and size of vessels were much lesser in tension wood than in the normal wood. (Chow 1946; Kaeiser and Boyce 1965; Jourez et al. 2001). Similar to the present study, a comparison between tension wood and normal wood of *Hevea braziliensis* showed a higher fibre length, fibre cell wall thickness and vessel diameter in tension wood, whereas fibre lumen width, fibre diameter and vessel frequency were higher in normal wood (Jusoh et al. 2005).

Table 1 Variation in fibre and vessel morphological parameters between tension wood and normal wood in *Acacia auriculiformis* and *A. mangium*

Parameters (µm)	Tension wood	Normal wood	*t* value
A. auriculiformis			
Fibre length	1903.32 (137.64)	1458.70 (140.92)	25.59**
Fibre width	37.77 (2.31)	29.72 (3.13)	20.18**
Fibre lumen width	30.59 (2.38)	23.87 (3.26)	15.95**
Fibre wall thickness	3.59 (0.25)	2.93 (0.34)	14.59**
Vessel diameter	191.69 (29.55)	225.38 (37.14)	7.25**
Vessel frequency	245.04 (32.65)	309.84 (45.36)	16.54**
A. mangium			
Fibre length	2062.40 (76.76)	1546.29 (80.68)	51.12**
Fibre width	37.15 (3.47)	30.92 (3.25)	12.62**
Fibre lumen width	30.15 (3.59)	25.05 (3.43)	9.80**
Fibre wall thickness	3.50 (0.30)	2.94 (0.32)	12.78**
Vessel diameter	210.98 (22.07)	250.98 (26.40)	11.19**
Vessel frequency	166.77 (25.78)	214.85 (32.06)	25.67**

Value in parenthesis is standard error
*significant at 5% level
**significant at 1% level

A comparison of fibre and vessel morphological parameters of tension wood between the two species showed significant variation in fibre length, fibre wall thickness, vessel diameter and vessel frequency (Table 2) which did not follow any particular pattern. According to Raghu (2002), the occurrence of tension wood in rubber wood is due to its large and heavy crown. Similar to this observation, the presence of tension wood in both species might be due to their highly branching nature from a very low height itself, which makes the crown heavier and susceptible to growth stresses. Difference in growth stresses between species may be the reason for varying fibre and vessel parameters. Variation in fibre and vessel morphological characters of tension wood between *A. auriculiformis* and *A. mangium* is given in Table 2. All the parameters except fibre length and vessel diameter were higher for *A. auriculiformis*.

Intra-specific (between trees) variation in fibre and vessel morphology of tension wood of *A. auriculiformis* and *A. mangium* was also studied. In *A. auriculiformis*, all the characters except fibre wall thickness and vessel diameter showed a significant variation between trees, while fibre length and vessel frequency differences were only significant in the case of *A. mangium.* The mean values of each tree for the two species are given in Table 3. Generally, the proportion of tension wood varies from tree to tree (Lim et al. 2003). A study by Lim and Ani (1999) in rubber wood showed that tension wood properties would vary with respect to age and height (axial variation) of the tree. Hence, the between-tree variation in both species might be due to factors like height, girth.

Table 2 Inter-specific variation in fibre and vessel morphology of tension wood in *Acacia auriculiformis* and *A. mangium*

Parameters (μm)	*A. auriculiformis*	*A. mangium*	*t* value
Fibre length	1903.32 (137.64)	2062.40 (76.76)	10.30**
Fibre width	37.77 (2.31)	37.15 (3.47)	1.51ns
Fibre lumen width	30.59 (2.38)	30.15 (3.59)	1.05ns
Fibre wall thickness	3.59 (0.25)	3.50 (0.30)	2.17*
Vessel diameter	191.69 (29.55)	210.98 (22.07)	5.47**
Vessel frequency	245.04 (32.65)	166.77 (25.78)	21.43**

Value in parenthesis is standard error
*significant at 5% level
**significant at 1% level
ns Non-significant

Table 3 Intra-specific variation in fibre and vessel morphology of tension wood between trees in *A. auriculiformis* and *A. mangium*

Parameter (μm)	Tree 1	Tree 2	*t* value
A. auriculiformis			
Fibre length	1934.31 (115.77)	1872.33 (151.37)	2.07*
Fibre width	38.42 (2.41)	37.13 (2.04)	2.85**
Fibre lumen width	31.22 (2.57)	29.97 (2.02)	2.68**
Fibre wall thickness	3.60 (0.27)	3.58 (0.23)	0.36 ns
Vessel diameter	188.88 (26.32)	194.50 (32.48)	0.86 ns
Vessel frequency	210.78 (20.78)	212.35 (23.50)	9.38**
A. mangium			
Fibre length	2081.27 (79.84)	2043.53 (69.32)	2.57*
Fibre width	37.33 (3.55)	36.97 (3.41)	0.51 ns
Fibre lumen width	30.30 (3.68)	30.00 (3.53)	0.40 ns
Fibre wall thickness	3.52 (0.33)	3.49 (0.27)	0.48 ns
Vessel diameter	210.78 (20.78)	212.35 (23.50)	0.32 ns
Vessel frequency	185.24 (24.68)	152.51 (17.35)	8.34**

Value in parenthesis is standard error
*significant at 5% level
**significant at 1% level
ns Non-significant

From the results of the study, it can be concluded that vessel and fibre morphology varied between tension wood and normal wood in both species. Fibre properties of both species followed similar pattern in which all properties were higher in magnitude than in normal wood. The number and size of the vessels were much lesser in tension wood than in normal wood. The present study distinctly proved the occurrence of tension wood in both the acacia species. In order to reduce the occurrence of tension wood or to prevent its occurrence, better silvicultural and management practices can be adopted, such as closer spacing and early and proper pruning.

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Basic Density and Fibre Morphological Characteristics of Selected Pulpwood Species of Kerala

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Abstract The present study focuses on variation in basic density and fibre morphology of important pulpwood species of Kerala, viz. *Acacia auriculiformis, Acacia mangium, Bambusa bambos, Bambusa vulgaris, Erythrina indica, Eucalyptus grandis, Eucalyptus tereticornis* and *Macaranga peltata*. Fibre morphological characters such as fibre length, fibre diameter, fibre lumen diameter, fibre wall thickness and fibre ratios such as fibre length-to-diameter ratio and shape factors which are important in pulp and paper making were determined in these species. Wood samples of selected trees were collected from various saw mills and pulp mills of the state. The samples collected were converted to blocks of size 3 cm × 3 cm × 3 cm and for each species, 11 blocks were prepared and ten were used to estimate the basic density and the remaining for maceration studies. Results indicated that basic density and fibre morphology and fibre ratios varied significantly among the species studied, and all the species were suitable for pulp and paper making. *Bambusa bambos* recorded the highest Runkel ratio and shape factor. The two species *M. peltata* and *E. indica* which are common in the state but under-utilised for pulp and paper making had fibre morphological parameters similar to that of other hardwood species and hence offer good potential for pulp and paper making.

Keywords Fibre morphology · Basic density · Runkel ratio · Shape factor

Introduction

As there is an ever-increasing demand for pulp and paper, the raw materials used for commercial pulp and paper making have changed greatly over the years. The changes in raw materials are still continuing, but these newer materials are to be

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adequately evaluated prior to utilisation so as to efficiently use these fibre resources. Many indigenous as well as exotic trees are being tried in Kerala for pulp and paper making. The exotic Acacias such as *Acacia auriculiformis* and *Acacia mangium* and exotic Eucalyptus species have wide adaptability and higher rates of growth in a wide range of sites. *Bambusa bambos* is the common thorny bamboos species distributed all over India and commonly found in the homesteads of southern India. *Bambusa vulgaris*, the ornamental bamboo species, is also very common in our homesteads. *Erythrina indica* is a native of the eastern coast of India and is very common in our home gardens as a supporting tree for pepper. *Macaranga peltata* is a medium-sized pioneer tree species, native to India. The potential of these species for producing pulp and paper of sufficient quality is a matter of investigation. Wood properties exert an influence on pulp and paper-making properties (Dadswell et al. 1961; Dinwoodie 1965). Barefoot et al. (1964) and Wangaard (1962) demonstrated strong influence of certain fibre dimensions on pulp and paper properties. Studies on fibre morphological variation in commonly used pulpwood species of the state is needed for the better utilisation of the raw material for pulp and paper making. With this background, the present investigation was framed out to study the variation in basic density and fibre morphology of eight pulpwood species with particular reference to their suitability for pulping.

Materials and Methods

The present investigation was conducted in the department of Wood Science, College of Forestry, Kerala Agricultural University, Thrissur, Kerala, to find out the variability in basic density and fibre morphological properties of eight pulpwood species, viz. *A. auriculiformis* A. Cunn. ex Benth., *A. mangium* Willd, *B. bambos* Voss, *B. vulgaris* Schrad ex. Wendl, *E. indica* Lamk, *Eucalyptus grandis* Hill ex Maind, *Eucalyptus tereticornis* Sm and *M. peltata* (Roxb) M.A. The wood samples were collected from different saw mills and pulp mills of the state of which majority of the samples were collected from the Hindustan News Print Limited factory at Velloor, Kottayam. The samples were converted to blocks of size 3 cm × 3 cm × 3 cm. For each species, 11 blocks were prepared, out of which ten blocks were used to measure basic density and the remaining blocks were used for studies on fibre morphology through maceration. The basic density of the pulpwood species was determined using the formula,

$$\textit{Basic density} = \textit{Oven dry weight of the sample} / \textit{volume of the sample in green condition}.$$

Maceration of wood samples was carried out following Schultz method (30% nitric acid and a pinch of potassium chlorate). The macerated samples were stained using saffranin and mounted on temporary slides using glycerine as the mountant. From the macerated fibres, fibre length (FL), fibre diameter (FD), fibre wall

thickness (FWT) and fibre lumen diameter (FLD) for each of the species were measured using the Image Analyzer (Labomed-Digi 2). Different derived ratios such as the Runkel ratio, shape factor and FL/FD ratio which are important in pulp and paper manufacturing were derived from the data obtained (Uju and Uewoxe 1997; Yanez-Espinosa et al. 2004). The data were subjected to one-way analysis of variance, and the treatment means were compared with lsd (least significant difference).

Results and Discussion

Among the physical properties, basic density is a key wood property that has a major effect on the yield and quality of both fibrous and solid wood products (Haslett and Young 1990). Analysis of variance revealed highly significant difference in basic density among the pulp wood species. Results indicated that the highest basic density was recorded in *E. tereticornis* (0.61), and the lowest value was recorded in *E. indica* (0.24) (Fig. 1).

The selection of a suitable tree species for the pulp and paper industry depends on the specific gravity, yield of wood and on the anatomical characteristics of fibres (Dinwoodie 1966; Wright and Sluis 1992; Rudie et a1. 1994; Brolin et a1. 1995). The influence of density extends from transport costs and chipping properties to digester capacity, pulp yield per unit mass of wood and paper quality (Balodis 1981). According to de Guth (1960), wood density can be correlated with strength properties of wood, pulp yield and pulp quality. It can also be used as a predictor of yield and quality of pulp and paper products (Dadswell and Wardrop 1959a, b; Barefoot et al. 1970). Ikemori et al. (1986) had stated that basic density which was

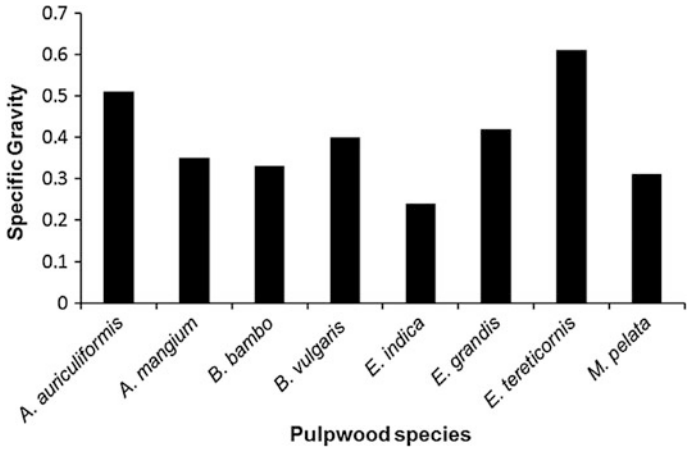

Fig. 1 Variation in specific gravity (standard) of selected pulpwood species

in the range of 480–570 kg m^{-3} was ideal for paper and pulp making, whereas Chittenden and Palmer (1990) opined that the species with basic density ≤ 0.60 is suitable for pulp and paper making. The results obtained in this study have shown that with the exception of *E. tereticornis*, all the others showed suitability of raw material for paper and pulp where the required basic density is met with. Wood density is the strongest predictor for handsheet properties (duPlooy 1980; Malan et al. 1994). It is related negatively to tensile, burst and tear indices. Denser wood produces bulkier, stiffer and more porous sheets, while low-density wood results in smoother, denser sheets, with higher tensile strength. Denser wood also corresponds with increase in light scattering coefficient, opacity (Cown and Kibblewhite 1982) and paper surface roughness (Scurfield 1976).

Values with the same superscript within a column do not vary significantly. Values in parenthesis correspond to standard deviation.

Among the fibre dimensions, with the exception of fibre wall thickness, all parameters varied significantly ($p = 0.01$) between species. The longest fibre length (2906 µm) was recorded in *B. bambos,* followed by *B. vulgaris* (2592 µm) and *E. indica* (2394 µm). The lowest fibre length (1193 µm) was recorded in *E. grandis* (Table 1). Wimmer et al. (2002) had suggested that higher the fibre length, better the pulp and paper-making qualities. With respect to fibre diameter, highest values were recorded in *E. indica* (31.3 µm) followed by *A. mangium* (29.9 µm), and the lowest fibre diameter was observed in *B. vulgaris* (15.6 µm). The fibre wall thickness was highest in *A. auriculiformis* (6.9 µm) followed by *E. tereticornis* (6.8 µm), and the lowest values were recorded for *B. vulgaris* (5.6 µm). The fibre lumen diameter was the highest in *E. indica* (20.8 µm) and was followed by *M. peltata* (19.5 µm), and the lowest value was recorded in *B. vulgaris* (5.7 µm).

The fibre dimensions are said to vary with species, and the dimensions observed for the two acacia species in the present study was within the range observed by Mohan Varghese et al. (1999).The study showed that acacias have higher fibre dimensions compared to two eucalyptus species. High values of fibre length will give better tensile strength to paper (Clark 1962) and also affect Runkel and FL/FD ratios which has direct influence on paper properties. The fibre dimensions obtained

Table 1 Variation in fibre morphology and derived ratios of selected pulpwood species

Species	Fibre dimensions (µm)				Shape factor	FL/FD
	FL	FD	FWT	FLD		
Acacia auriculiformis	1450^{e} (201)	28.2^{b} (5.5)	6.9 (1.5)	17.4^{cd} (4.5)	0.45^{c} (0.14)	53.4^{cd} (12.8)
Acacia mangium	1436^{e} (215)	29.9^{ab} (7.2)	6.4 (1.7)	18.8^{bc} (6.3)	0.44^{c} (0.13)	50.7^{cd} (14.1)
Bambusa bambos	2906^{a} (386)	17.8^{e} (2.5)	6.8 (1.7)	6.3^{f} (1.3)	0.77^{a} (0.01)	166.7^{a} (35.5)
Bambusa vulgaris	2592^{b} (317)	15.6^{f} (3.9)	5.6 (1.5)	5.7^{f} (1.8)	0.76^{a} (0.01)	177.0^{a} (52.2)
Erythrina indica	2394^{c} (294)	31.3^{a} (8.5)	6.1 (1.0)	20.8^{a} (8.1)	0.41^{c} (0.13)	82.8^{b} (26.2)
Eucalyptus grandis	1193^{f} (181)	25.8^{c} (4.5)	5.9 (1.3)	15.9^{d} (3.9)	0.45^{c} (0.10)	47.9^{d} (12.8)
Eucalyptus tereticornis	1248^{f} (271)	21.0^{d} (3.4)	6.8 (1.3)	11.8^{e} (2.9)	0.52^{b} (0.01)	61.1^{c} (17.7)
Macaranga peltata	1644^{d} (295)	28.7^{b} (4.6)	5.8 (0.8)	19.5^{ab} (4.5)	0.37^{d} (0.13)	58.8^{cd} (14.4)

Note: The values with same superscript within a row are not significantly different (p=0.01)

for bamboos in the present study falls within the range recorded by Singh et al. (1976, 1988). The values obtained in the present study showed that two bamboo species had higher value for fibre length in comparison with all other species selected for the study which confirm the superiority of bamboos for pulp and paper making. Both the bamboo species had higher fibre diameter and lower lumen width than rest of the species which indicates that the paper made out of the two species have superior tearing strength. Rao et al. (2002) reported the fibre characteristics of Eucalyptus as fibre length (848–938 µm), fibre diameter (14.5–16.9 µm), fibre lumen diameter (8–9.1 µm) and fibre wall thickness (6–8 µm). The values reported in the present study were also within this range. The results also showed that eucalyptus species had least values of fibre length, lower fibre diameter and fibre lumen diameter in comparison with acacias. However, fibre diameter had higher values in comparison with bamboos. The study provides the first information on the two fast growing indigenous species *E. indica* and *M. peltata,* having good fibre dimension values. The fibre lumen diameters of these species were very high compared to other species. This may result in some difficulty in pulping. But these two species have sufficient scope for producing better quality paper after suitable treatments. All the other parameters conform to the range of typical hardwoods usually used for pulping.

The Runkel ratio which is obtained by dividing double wall thickness with fibre lumen diameter is an important parameter, which is often taken into consideration in paper and pulp studies. It is clear from Fig. 2 that *B. bambos* had maximum value (2.23) while *M. peltata* (0.63) had minimum. Shape factor was also determined, and the fibres with lower values of shape factor will give better strength paper. *B. bambos* recorded the maximum value (0.77), while *M. peltata* had the minimum value (0.37) (Table 1). Higher the fibre length/fibre diameter ratio, greater will be the expected fibre flexibility which in turn is expected to give better tensile and tear

Fig. 2 Variation in Runkel ratio of selected plywood species

property to paper. The table showed that *B. vulgaris* had the maximum value (177) while *E. grandis* recorded the minimum value (47.9).

Fibre morphological ratios have great influence on paper properties. To produce pulp of reasonable quality, the approximate limit of Runkel ratio ranges from 0.25 to 1.5 (Singh et al. 1991) for a species, whereas Dadswell and Wardrop (1959a, b) and Okereke (1962) suggested it to be less than 1. The data obtained in the present investigation show significant variation with regard to this ratio for all species except the two bamboo species which was only less than 1.5. All the species obtained lower values for shape factor. So we can conclude that all these species can produce paper of better strength. Greater the length and width ratio of the fibre, higher will be the fibre flexibility and better the chance of obtaining paper with superior tear and tensile properties (Anon 2000). All the species covered in the present investigation recorded higher fibre dimension ratios. Overall, it can be concluded that all the species in the present study can produce pulp and paper of reasonable quality.

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Estimation of Leaf Area–Wood Density Traits Relationship in Tropical Dry Evergreen Forests of Southern Coromandel Coast, Peninsular India

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Abstract This study estimates the existing eco-physiological traits relationship between leaf area (LA) and wood density (WD) in tropical dry evergreen forests (TDEFs) of southern Coromandel Coast, peninsular India. Leaf area and wood density of 56 tree species belonged to 49 genera and 28 families were measured to understand the relationship between the traits. Leaf area and wood density were estimated with standardised protocols. On average, each species had 3400 ± 3040 mm^2 LA and 0.74 ± 0.16 g cm^{-3} WD. On average, each site had 2.44% of total soil organic carbon, 0.11% of nitrogen and 15.7 μg g^{-1} of phosphorus. The ratios of carbon–nitrogen varied from 16.2 to 32.6 (mean = 22.4 ± 4.92) and of nitrogen–phosphorus ranged from 12.7 to 19.6 (mean = 70.5 ± 15.1). The relationship between leaf area and wood density was negative and significant ($r^2 = 0.34$, $n = 56$, $P < 0.005$; $t_{54} = 9.07$, $P < 0.001$). There was no significant difference recorded between mean LA and WD of evergreens and deciduous species. Oligotrophic habitat, semi-aridness and longer dry periods are few of the possible reasons behind the profuse occurrence of small-leaved, dense wooded evergreens in tropical dry evergreen forests.

Keywords Dry forest · Leaf trait · Semi-arid tropics · Soil nutrition

Introduction

One of the recent trends in plant ecological studies is to find plant traits capable of expressing differences in ecological behaviour among species (Garnier et al. 2001). Plant traits represent specific functional adaptations to diverse biotic and abiotic factors, and they may act as valuable indicators of the response of species to various environmental circumstances (Milla and Reich 2011). Classifying species into clus-

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ters based on their functional characteristics rather than on conventional methods has promoted intensive research to find out various plant functional traits. Currently, creation of a large database for plant functional traits (PFTs) gets high priority in the research agenda of plant ecology since it helps to understand and predict the distribution of species in present and future environments (Westoby 1998).

Soil fertility and environmental factors largely decide quantitative measure of plant functional traits. The status of soil fertility was found to play an important role in the formation of plant functional traits. Ordonez et al. (2009) realised the significance of soil factors and recommended researchers to report soil information in plant trait studies. Information on soil nutrients, especially N and P, is important, as these nutrients are often found to be the lead variables in determination of soil fertility (Pritchett and Fisher 1987). Concentrations of C, N and P are important that affect the fertility of soils, thereby influencing the growth and development of individuals, physiognomy and species compositions of vegetation. Likewise, the ratios of carbon–nitrogen (C/N) and nitrogen–phosphorus (N/P) are important factors that influence nutrition cycle in forest. The C/N ratio is recognised as an indicator of site quality in tropical forests (Yamakura and Sahunalu 1997). There are several complex factors that affect the C/N and N/P ratios in the soil. Factors such as topography and species composition of the vegetation (Mackay and Band 1997; Fisher and Binkley 2000), temperature and soil acidity (Nordström-Högberg 2004), forest stand type, age and soil properties (Cote et al. 2000), site productivity and climate (Khanna et al. 2001), forest floor materials and its rate of decay, atmospheric N deposition and forest history determine the C/N ratio of the soil (Lovett et al. 1999; Kudish 2000; Weathers et al. 2000).

Leaf size is one of the most plastic functional traits of plants. Leaf size varies among genera, within genus, among species, between individuals of species, and even in the same individual at different stages of ontogeny (sapling and adult tree) or on different parts of the same tree (e.g. sun exposed versus shaded leaves) (Givnish 1984, 1987; Malhado et al. 2009). Givnish (1987) reported that the leaf size of a species can vary along gradients of rainfall, humidity, soil fertility, irradiance, elevation, aridity and even with age of the tree. Several reasons are given for changes in leaf size. According to Givnish (1984, 1987), leaf sizes tend to decline in habitats with high light, low nutrients or low moisture availability; other factors include increasing elevation (Cordell et al. 1998), mean annual rainfall and humidity and lower soil fertility (Schimper 1903; Fonseca et al. 2000). It is noteworthy that good numbers of palaeobotanical studies utilise fossil leaf size to estimate past rainfall (Wilf et al. 1998).

Wood density (WD) is of great ecological and phylogenetic importance, and it is closely associated with several factors such as growth and survival rates of woody plants (Mcculloh et al. 2011; Muller-Landau 2004), hydraulic properties such as water storage capacity, degree of resistance to xylem cavitation (Bucci et al. 2004; Santiago et al. 2004), drought-induced embolism (Pratt et al. 2007) and level of resistance of stems to pathogen attack and to mechanical damage (Turner 2001). Scores of available literatures recorded a negative relationship between wood density and leaf area/size (Ackerly et al. 2002; Cavender-Bares et al. 2004; Rossetto

and Kooyman 2005; Cornwell 2006; Kraft et al. 2008; Wright et al. 2006, 2007; Ackerly and Cornwell 2007; Malhado et al. 2009) and unanimously, suggesting that this relationship between traits may be quite generally true. To our knowledge, no studies have been undertaken to find out the traits relationship, if any, between WD and LA in tropical dry evergreen forests (TDEFs) of Coromandel Coast, peninsular India. Earlier, Mani and Parthasarathy (2007) published their findings on WD of certain trees of TDEFs from Coromandel Coast. Thus, the primary objective of this study was to find out whether the broadly reported LA–WD traits relationship is exist in TDEF or not?

Study Site

This study was conducted at 10 TDEF sites, each two of five districts, namely Cuddalore, Nagapattinam, Pudukottai, Thiruvarur and Villupuram, in Tamil Nadu state of the Indian subcontinent. The mean annual rainfall is 1079, 1174, 1033, 1091 and 1311 mm in the nearest towns, namely Cuddalore, Nagapattinam, Pudukottai, Thiruvarur and Villupuram, respectively. Mean annual maximum and minimum temperatures are 33.64 and 22.75 °C in Cuddalore, 32.00 and 24.6 °C in Nagapattinam, 33.40 and 25.40 °C in Pudukottai, 36.90 and 29.80 °C in Thiruvarur and 34.40 and 24.10 °C in Villupuram. The hottest month is May (temperature goes up to 45 °C). Study area experiences a typical maritime tropical climate with three–six dry months in a year. The rainfall regime is dissymmetric, receives maximum rainfall during north-east monsoon (October–December) and very little, inconsistent during south-west monsoon (June–September). Study area receives most proportion of rainfall during monsoon (90.14–95.74%) and very little on dry season (4.46–9.86%; Table 1).

Soil type of the eastern coastal region of India is known as new coastal alluvium; it supports characteristic vegetation. Soil is mainly quartzitic or calcareous sands with low silt and clay fraction content. The depth of the soil is variable, and the water table is remarkably high. The nutrient status is typically low, especially with regard to nitrogen and phosphorus. Soil type of the study area is coastal alluvium except Cuddalore where the soil is ferralitic red.

Table 1 Geographical coordinates of study area wherein the LA and WD data were collected

District	Geographical coordinates (Altitude amsl m)	Rainfall	
		Rainfall at monsoon period	Rainfall at dry season (no. of dry months)
Cuddalore	11° 43′N, 79° 49′E (5–7)	94.87%	5.13% (5–6)
Nagapattinam	10° 10′N, 79° 15′E (4–6)	90.14%	9.86% (5)
Pudukottai	10° 23′N, 78° 52′E (3–6)	90.84%	9.16% (5)
Thiruvarur	10° 20′N, 79° 15′E (11–13)	92.11%	7.89% (5)
Villupuram	11° 93′N, 79° 48′E (7–10)	95.74%	4.26% (5–6)

The terrestrial forest type on the Coromandel Coast of peninsular India is recognised as tropical dry evergreen forest (TDEF) (Champion and Seth 1968). Short-stature, three-layered forest and a sparse and patchy ground flora are the characteristic features of TDEFs. In TDEFs, buttresses are rare, *cauliflory* is uncommon, herbaceous vascular epiphytes are very rare and large-vertebrate seed dispersers are absent. Largely, human activities (forest clearance, hunting, habitat loss, etc.) destroyed the lives of large vertebrates (Blanchflower 2005). Compared to tropical wet forests, they receive less annual rainfall (<1400 mm). To date, 86 TDEF patches are reported from southern Coromandel Coast of peninsular India (Udayakumar and Parthasarathy 2010). At present, TDEFs are highly fragmented and invariably protected as 'sacred groves' (SGs), except two reserve forests at Marakkanam (Villupuram district) and Point Calimere (Nagapattinam district). TDEFs are regarded as repository of considerable number of endemic and threatened plant species (Parthasarathy et al. 2008).

Materials and Methods

Estimation of Soil Carbon, Nitrogen and Phosphorus

To estimate soil, C, N and P samples (soil cores) were collected from five different places in each site, each 10 cm deep. Collected samples of each study site were pooled together and analysed to estimate the amount of total soil C, N and P. Soil analyses were carried out in Environmental Science laboratory at Central Leather Research Institute, Chennai. Total soil C was determined using Walkley and Black method (1934), P assessed according to the protocol outlined by Fonseca et al. (2000), whereas N was estimated by improved Kjeldahl method (Bradstreet 1965).

Leaf Area

Leaf area and wood density of 56 tree species representing 49 genera and 28 families were measured to reveal the relationship between WD and LA in TDEFs (Table 2). Leaf area is the one-sided area of a fresh leaf, and it is considered as one of the important leaf traits. For leaf area estimation, whole twig sections that had fully expanded and hardened leaves were collected two hours after sunrise during January 2012 from ten TDEF sites (Cornelissen et al. 2003). Samples wrapped with moist paper were kept in plastic bags and sealed to maintain the leaves in water-saturated condition. The leaves of deciduous species were re-hydrated by placing the cut end of the stem in deionised water in the dark for 6 h. Leaves were removed from the whole twig section just prior to the leaf area measurement. Each leaf was gently rubbed to dry before measurement. Leaf area of 40 leaves (leaflets

in case of compound-leaved plants) was measured. For most of the species, samples were collected from two individuals in each site. If species not found in any of the study site(s) or diseased, then more than two samples were collected from site(s) (Table 2). Leaf area is measured with the help of Adobe Photoshop CS4 and HP Scanjet 2400 flatbed scanner.

Wood Density

Tree species recorded in more than 10 TDEF sites (total sites reported = 96) by Parthasarathy and Udayakumar (2010) and Udayakumar (2011) were selected for the determination of WD. To estimate WD value of selected trees, wood core samples (three cores per individual) were collected from five individuals per species. Sampled trees were ≥ 5 cm diameter at breast height (dbh) (range 5–100 cm). Wood cores were taken in the height of 100–150 cm from the ground. The length of cores was roughly equal to half the diameter of the trees. The resulting holes were filled with synthetic resin to avoid infestation by pathogens. Wood cores were kept in plastic bags and sealed until returned to the laboratory; further, the samples were then cut into small cylinders. Firmly attached bark (if any) or equivalent phloem tissue is integral part of the functioning stem; therefore, these were included in stem density estimation (Cornelissen et al. 2003). The length and volume of the cylindrical cores were calculated from the length and inner diameter of the increment wood corer. Fresh weight of the samples was estimated; then, the wood cores were kept in hot air oven at 105 °C for 48 h to bring them to constant weight.

Data Analyses

Pearson's coefficient of correlation was employed to predict the relationship between soil N concentration and evergreen–deciduous ratio (E/D), soil P and E/D, C/N and E/D, N/P and E/D, WD and LA. Data were log-transformed prior to the correlation analysis. *F* test was applied to test the significance of means, while *t* test was used to verify the significance of obtained correlation coefficient value (*r*).

Results

Soil Nutrition and Physiognomy

On average, each site had 2.44% of total organic C, 0.11% of N and 15.7 μg g^{-1} of P in its soil. Total soil organic carbon of the study sites ranged from 1.95%

Table 2 Species, family, physiognomy and leaf types of tree species

Species	Family	Physiognomy[a]	Leaf type[b]	Sample collection[c] (number of samples)	Leaf area (mm^2) (Mean[d] ± S.D.; n = 40)	Wood density (g cm^{-3}) (Mean ± S.D.; n = 12)
Acacia leucophloea	Mimosaceae	Deci.	Com.	C(4), P(4), T(2)	65 ± 2	0.87 ± 0.02
Aglaia elaeagnoidea	Meliaceae	Ever.	Com.	C(2), N(2), P(2), T(2), V (2)	85 ± 3.0	0.92 ± 0.03
Alangium salviifolium	Alangiaceae	Deci.	Sim.	C(2), N(2), P(2), T(2), V (2)	3450 ± 15	0.75 ± 0.01
Albizia amara	Mimosaceae	Deci.	Com.	C(4), P(4), V(2)	6513 ± 35	0.63 ± 0.02
Albizia lebbeck	Mimosaceae	Deci.	Com.	C(2), N(2), P(2), T(2), V (2)	386 ± 6	0.88 ± 0.02
Allophylus serratus	Sapindaceae	Ever.	Com.	C(2), N(2), P(2), T(2), V (2)	9765 ± 17	0.51 ± 0.03
Atalantia monophylla	Rutaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	688 ± 6	0.91 ± 0.02
Azadirachta indica	Meliaceae	Deci.	Sim.	C(2), N(2), P(2), T(2), V (2)	835 ± 11	0.88 ± 0.04
Benkara malabarica	Rubiaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	587 ± 9	0.81 ± 0.02
Breynia vitis–idaea	Euphorbiaceae	Deci.	Com.	C(2), N(2), P(2), T(2), V (2)	297 ± 4	0.83 ± 0.01
Canthium coromandelicum	Rubiaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	655 ± 7	0.77 ± 0.02
Canthium dicoccum	Rubiaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	6489 ± 23	0.76 ± 0.03

(continued)

Table 2 (continued)

Species	Family	Physiognomy[a]	Leaf type[b]	Sample collection[c] (number of samples)	Leaf area (mm^2) (Mean[d] ± S.D.; n = 40)	Wood density (g cm^{-3}) (Mean ± S.D.; n = 12)
Casearia elliptica	Flacourtiaceae	Ever.	Sim.	C(4), P(2), V(4)	6324 ± 57	0.59 ± 0.02
Cassia fistula	Caesalpiniaceae	Deci.	Com.	C(2), N(2), P(2), T(2), V (2)	4130 ± 32	0.67 ± 0.03
Cassia roxburghii	Caesalpiniaceae	Ever.	Com.	C(4), N(2), P(2), T(2)	189 ± 3	0.87 ± 0.02
Catunaregam spinosa	Rubiaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	378 ± 6	0.56 ± 0.02
Chionanthus zeylanicus	Oleaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	1326 ± 19	0.79 ± 0.01
Chloroxylon swietenia	Flindersiaceae	Ever.	Com.	C(4), P(4), V(2)	152 ± 5	0.89 ± 0.02
Clausena dendata	Rutaceae	Ever.	Com.	C(4), P(4), V(2)	317 ± 14	0.78 ± 0.03
Commiphora caudata	Burseraceae	Deci.	Com.	C(4), N(2), P(2), T(2)	287 ± 11	0.42 ± 0.01
Cordia pubescens	Boraginaceae	Deci.	Sim.	C(2), N(2), P(2), T(2), V (2)	5387 ± 62	0.61 ± 0.02
Crateva magna	Capparidaceae	Deci.	Com.	C(2), N(2), P(2), T(2), V (2)	6587 ± 81	0.47 ± 0.03
Diospyros ebenum	Ebenaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	4526 ± 26	0.72 ± 0.02
Diospyros ferrea	Ebenaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	155 ± 15	0.92 ± 0.03

(continued)

Table 2 (continued)

Species	Family	Physiognomy[a]	Leaf type[b]	Sample collection[c] (number of samples)	Leaf area (mm^2) (Mean[d] ± S.D.; n = 40)	Wood density (g cm^{-3}) (Mean ± S.D.; n = 12)
Diospyros montana	Ebenaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	3421 ± 59	0.69 ± 0.02
Dodonaea angustifolia	Sapindaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	453 ± 14	0.77 ± 0.02
Drypetes sepiaria	Euphorbiaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	1430 ± 43	0.98 ± 0.02
Ficus benghalensis	Moraceae	Deci.	Sim.	C(2), N(2), P(2), T(2), V (2)	7832 ± 102	0.52 ± 0.02
Ficus microcarpa	Moraceae	Ever.	Sim.	C(4), P(4), T(2)	3453 ± 86	0.79 ± 0.03
Ficus religiosa	Moraceae	Deci.	Sim.	C(4), N(4), P(2)	6789 ± 112	0.57 ± 0.02
Flacourtia indica	Flacourtiaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	317 ± 19	0.79 ± 0.01
Garcinia spicata	Clusiaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	7977 ± 98	0.69 ± 0.02
Glycosmis mauritiana	Rutaceae	Ever.	Com.	C(2), N(2), P(2), T(2), V (2)	1029 ± 54	0.86 ± 0.01
Gmelina asiatica	Verbenaceae	Deci.	Com.	C(2), N(2), P(2), T(2), V (2)	162 ± 19	0.76 ± 0.01
Holoptelia integrifolia	Ulmaceae	Ever.	Sim.	C(4), P(4), V(2)	6512 ± 119	0.72 ± 0.02
Ixora pavetta	Rubiaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	7422 ± 103	0.62 ± 0.01

(continued)

Table 2 (continued)

Species	Family	Physiognomy[a]	Leaf type[b]	Sample collection[c] (number of samples)	Leaf area (mm^2) (Mean[d] ± S.D.; n = 40)	Wood density (g cm^{-3}) (Mean ± S.D.; n = 12)
Lannea coromandelica	Anacardiaceae	Deci.	Com.	C(2), N(2), P(2), T(2), V (2)	6743 ± 115	0.41 ± 0.01
Lepisanthes tetraphylla	Sapindaceae	Ever.	Com.	C(2), N(2), P(2), T(2), V (2)	7012 ± 92	0.69 ± 0.02
Manilkara hexandra	Sapotaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	6855 ± 86	0.87 ± 0.01
Maytenus emarginata	Celastracaee	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	847 ± 35	0.91 ± 0.02
Memecylon umbellatum	Melastomataceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	1571 ± 19	0.87 ± 0.03
Morinda coreia	Rubiaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	6314 ± 92	0.46 ± 0.02
Ochna squarrosa	Ochnaceae	Deci.	Sim.	C(2), N(2), P(2), T(2), V (2)	5865 ± 67	0.59 ± 0.01
Pamburus missionis	Rutaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	1575 ± 35	0.86 ± 0.02
Pavetta indica	Rubiaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	7427 ± 81	0.62 ± 0.02
Pleiospermium alatum	Rutaceae	Ever.	Com.	C(4), P (2), V(4)	368 ± 16	0.89 ± 0.02
Pongamia pinnata	Papilionaceae	Deci.	Com.	C(2), N(2), P(2), T(2), V (2)	6872 ± 121	0.77 ± 0.01
Prosopis juliflora	Mimosaceae	Deci.	Com.	C(2), N(2), P(2), T(2), V (2)	189 ± 11	0.89 ± 0.02

(continued)

Table 2 (continued)

Species	Family	Physiognomy[a]	Leaf type[b]	Sample collection[c] (number of samples)	Leaf area (mm^2) (Mean[d] ± S.D.; n = 40)	Wood density (g cm^{-3}) (Mean ± S.D.; n = 12)
Pterospermum canescens	Sterculiaceae	Deci.	Sim.	C(2), N(2), P(2), T(2), V (2)	2322 ± 56	0.51 ± 0.03
Sapindus emarginatus	Sapindaceae	Ever.	Com.	C(2), N(2), P(2), T(2), V (2)	7420 ± 137	0.62 ± 0.02
Streblus asper	Moraceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	485 ± 32	0.92 ± 0.02
Strychnos nux–vomica	Loganiaceae	Deci.	Sim.	C(4), P(4), V(2)	5812 ± 142	0.81 ± 0.02
Syzygium cumini	Myrtaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	3243 ± 59	0.76 ± 0.02
Tamarindus indica	Caesalpiniaceae	Ever.	Com.	C(2), N(2), P(2), T(2), V (2)	125 ± 12	1.05 ± 0.03
Tarenna asiatica	Rubiaceae	Ever.	Sim.	C(2), N(2), P(2), T(2), V (2)	6057 ± 165	0.72 ± 0.02
Tricalysia sphaerocarpa	Rubiaceae	Ever.	Sim.	C(8), V(2)	6953 ± 142	0.77 ± 0.03
				Mean	3400 ± 3040	0.74 ± 0.16

[a]Physiognomy: Deci.—Deciduous, Ever.—Evergreen
[b]Leaf type: Com.—Compound, Sim.—Simple
[c]Sample collection (Leaves): C—Cuddalore, N—Nagapattinam, P—Pudukottai, T—Thiruvarur, V—Villupuram
[d]Mean—mean value calculated with all collected samples together

(Cuddalore) to 3.2% (Thiruvarur). Among sites, Thiruvarur had maximum amount of soil N (0.135%) followed by Pudukottai (0.13%) and Villupuram (0.12%). As to the quantity of soil phosphorus, Thiruvarur stood first with a content of 17.7 μg g^{-1}, whereas Cuddalore had least amount 14.1 μg g^{-1}. Overall, Thiruvarur had rich source of soil nutrients, while Cuddalore had poor among sites sampled. The ratios of carbon–nitrogen varied from 16.2 to 32.6 (mean = 22.4 ± 4.92) and of nitrogen–phosphorus ranged from 12.7 to 19.6 (mean = 70.5 ± 15.1, Table 3).

An increase in soil C, N and P decreased the proportion of evergreens in study area. The correlations involving soil C and E/D (the ratio of evergreen–deciduous) ($r^2 = 0.42$, $n = 10$, $P < 0.01$); soil N and E/D ($r^2 = 0.38$, $n = 10$, $P < 0.01$); and soil P and E/D ($r^2 = 0.61$, $n = 10$, $P < 0.01$) were negative and significant. Like soil nutrients, the ratio of carbon–nitrogen also had negative, significant correlation ($r^2 = 0.54$, $n = 10$, $P < 0.01$) with E/D. The t test confirmed the relationships among soil nutrients and E/D (C and E/D, $t_8 = 4.16$, $P < 0.005$; N and E/D, $t_8 = 3.58$, $P < 0.005$; P and E/D, $t_8 = 4.56$, $P < 0.005$; C/N and E/D, $t_8 = 4.16$, $P < 0.005$; N/P and E/D, $t_8 = 3.06$, $P < 0.01$).

Leaf Area and Wood Density

Majority of the species (66%) are evergreen and simple-leaved (62.5%, Table 2). LA and WD of species varied from 65 ± 5 (± SD) mm^2 (*Albizia amara*) to 9765 ± 155 mm^2 (*Sapindus emarginatus*), and 0.42 ± 0.01 g cm^{-3} (*Commiphora caudata*) to 1.05 ± 0.02 g cm^{-3} (*Tamarindus indica*), respectively. On average, each species had 3400 ± 3040 mm^2 LA and 0.74 ± 0.16 g cm^{-3} WD. Average leaf area and wood density of deciduous trees were 3535 ± 3067 mm^2 and 0.68 ± 0.17 g cm^{-3}, whereas evergreens had 3331 ± 2906 mm^2 and 0.76 ± 0.13 g cm^{-3}, respectively (Table 2). There was no significant difference between mean values of WD (F test; $F_{(36, 18)} = 0.99$; $P > 0.05$; n.s.) and LA (F test; $F_{(36, 18)} = 0.58$; $P > 0.05$; n.s.) of evergreens and deciduous. Mean LA and WD of compound-leaved individuals were 2649 ± 3309 mm^2 and 0.75 ± 0.18 g cm^{-3}, while simple-leaved individuals were 3851 ± 2820 mm^2 and 0.74 ± 0.13 g cm^{-3}. We did not find significant difference between mean values of LA (F test; $F_{(34, 20)} = 0.72$; $P > 0.05$; n.s.) and WD (F test; $F_{(34, 20)} = 0.58$; $P > 0.05$; n.s.) of compound- and simple-leaved species. Statistically no significant difference prevailed between mean values of LA (F test; $F_{(33, 22)} = 1.06$; $P > 0.05$; n.s.) of small- (<1000 mm^2) and large-leaved species (>1000 m^2) in this study. The correlation between leaf area and wood density of studied species was negative and significant ($r^2 = 0.34$, $P < 0.005$; $n = 56$; $t_{54} = 9.07$, $P < 0.001$).

Table 3 Soil nutrient status of ten TDEF forest sites in southern Coromandel Coast of peninsular India

Site	C mg g^{-1} Mean ± S.D.	N mg g^{-1} Mean ± S.D.	P μg g^{-1} Mean ± S.D.	C:N	N:P	E:D[a]
Site 1 (Cuddalore)	18.6 ± 0.12	0.57 ± 0.01	12.7 ± 0.1	32.6	44.9	20.3
Site 2 (Cuddalore)	20.4 ± 0.21	1.20 ± 0.02	15.5 ± 0.1	17.0	77.4	16.3
Site 1 (Nagapattinam)	14.6 ± 0.11	0.55 ± 0.01	13.6 ± 0.1	26.6	40.44	15.5
Site 2 (Nagapattinam)	27.7 ± 0.17	1.23 ± 0.02	16.2 ± 0.2	22.5	75.92	14.2
Site 1 (Pudukottai)	20.3 ± 0.31	1.25 ± 0.02	14.7 ± 0.3	16.2	85.0	18.6
Site 2 (Pudukottai)	32.6 ± 0.29	1.42 ± 0.01	18.4 ± 0.2	22.9	77.2	14.2
Site 1 (Thiruvarur)	41.1 ± 0.17	1.58 ± 0.04	19.6 ± 0.2	26.0	80.6	13.1
Site 2 (Thiruvarur)	22.8 ± 0.22	1.12 ± 0.03	15.9 ± 0.3	20.3	70.4	14.6
Site 1 (Villupuram)	21.6 ± 0.19	1.08 ± 0.01	14.1 ± 0.2	20.0	76.6	16.3
Site 2 (Villupuram)	24.5 ± 0.26	1.22 ± 0.02	16.0 ± 0.2	20.1	76.3	14.5
Mean	24.4 ± 7.67	1.12 ± 0.33	15.7 ± 2.11	22.4 ± 4.92	70.5 ± 15.1	15.8 ± 2.2

[a]E:D—ratio of evergreen to deciduous species density in forest site ha^{-1}

Data source (Udayakumar and Parthasarathy 2010)

Discussion

Soil Nutrition, Leaf Area and Wood Density

Results of the present study on the relationship between soil nutrition and leaf area concur with earlier reports by Fonseca et al. (2000) and Wright et al. (2004); they observed smaller mean leaf area in plants growing in drier environment. It was consistently observed that poor nutrient soils especially less N and P favoured smaller leaves (Schimper 1903; Givnish 1984; Cunningham et al. 1999; Fonseca et al. 2000; Ackerly et al. 2002). Our findings (smaller-leaved plants on soil poor with N and P) are in agreement with earlier reports by Schimper (1903), Ackerly et al. (2002) and hence support the well-established positive relationship between soil nutrition and leaf size in forests around the world. Mean temperature of the study area, i.e. ca. 34 °C, could also be an important factor for smaller leaf sizes in TDEFs. It has been shown that the small effective leaf size was found to be associated with higher temperature and drier environments (Givnish and Vermeij 1976; Givnish 1984). According to Gates (1965), smaller leaf size is generally advantageous in places with lower rainfall under a combination of high solar radiation and low water availability. By enhancing convective heat loss, smaller leaves maintain favourable leaf temperatures and higher photosynthetic water-use efficiency. In contrast to this, larger leaves under such drier environmental conditions develop thicker boundary layers of still air, thereby enhancing leaf temperature unfavourably to a higher level (above air temperature). Due to this over-heat, the rate of photosynthesis gets reduced to a lower level than that of respiration; this could be a disadvantage under water-limiting, stronger radiation environments (Gates 1965; Givnish 1984). Soil water availability is one of the limiting factors at dry sites where plants adaptively close their stomata, this phenomenon may lead the large leaves to realise potentially damaging leaf temperatures and hence dry sites favour small leaves (Givnish and Vermeij 1976). In addition, smaller-leaved species had an advantage that they may suffer less herbivory due to its shorter leaf expansion duration (Gates 1965).

Findings of the present study as TDEFs as oligotrophic habitat having very poor P and N concentration are in conformity with that of Visalakshi (1994). Soil deficient of nutrients as found in our study areas could have contributed to small leaf sizes. Similar observations were reported earlier by Fonseca et al. (2000). Lower soil nutrition (especially P) was found to be associated with smaller leaf size. According to Givnish, production of photosynthetic enzymes is limited in trees growing in oligotrophic soil, and because of this, they benefit less under temperature-related increases in photosynthetic metabolism; under such conditions, poor nutrient soil favours the evolution of smaller leaves.

Higher WD observed in plants growing in TDEF sites by us could be due to high temperature and poor nutrient soil. Occurrence of high WD in plants from low nutrition, arid and semi-arid sites was reported earlier by Ackerly (2004), ter steege et al. (2006), and Swenson and Enquist (2007). High WD species under high

temperature and low nutrient soil is of great importance in arid or semi-arid environments. It has been shown that high WD species tended to be more resistant to xylem cavitation and drought-induced embolism (Bucci et al. 2004; Santiago et al. 2004). Tissue density is reported to be tightly linked with both modulus of elasticity and fracture toughness of stems and leaves (Bucci et al. 2004; Muller-Landau 2004), and it seems to be important in the survival and longevity of both seedling and mature tree. Subsequently, high WD species are frequently positively associated with slow-growing, late-successional species. Generally, evergreens (slow-growing species) tend to have higher wood density than fast-growing pioneer species (Muller-Landau 2004; Poorter et al. 2010). Most probably, this is the reason why evergreens are dominating TDEFs both in terms of species richness and in terms of density. For example, out of 8000 surveyed individuals (10 ha of TDEFs) ~87% are evergreens (Udayakumar 2011).

Average C/N ratio (22.4 ± 4.97) obtained in this study is in agreement with earlier reports, and it is very well within the mean value of tropical forests. Aitkenhead and McDowell (2000) reported a mean value of 24.96 C/N ratios for tropical forests. An increase in soil C, N, P and ratios of C/N and N/P increase the density of deciduous trees in study area. Scores of literature showed a positive association between soil nutrient and physiognomy (Schimper 1903; Givnish 1984; Cunningham et al. 1999; Fonseca et al. 2000; Ackerly et al. 2002). Vegetation composition (E/D, 13.1–20.3, mean = 15.8 ± 2.2) may influence the C/N ratios in study sites. MacKay and Band (1997), Cote et al. (2000), Fischer and Binkley (2000) recognised vegetation composition as a determining factor in C/N ratio of soils. It has been reported that generally leaf litter of evergreen tree has a higher C/N ratio than deciduous leaf litter; leaves add a lower amount of nitrogen to the soil and make soils as acidic media. Soil acidity and lower nitrogen content favour the growth of more evergreens and lower the persistence of deciduous trees. Yamakura and Sahunalu (1997) found a negative relationship between soil nutrients (C, N) and evergreens and a positive relationship with deciduous species in three south-east Asian forests. In oligotrophic habitats, the decomposition of litter is slow and accumulates thickly, and the opposite is true with eutrophic environments (Swift et al. 1979). In addition, slow decay of litter decreases the mobilisation of nutrients (Yamakura and Sahunalu 1997). Low nutrient loss rate of evergreens also has been reported for their dominance on nutrient-poor environments (Aerts 1995). According to Aerts (2002), longer tissue longevity, low nutrient concentrations and possible existence of a positive feedback between evergreens and poor nutrient availability provide a selective advantage to evergreens under nutrient-poor environments. Notably, the ratio of nitrogen–phosphorus has been adopted to identify thresholds of nutrient limitation (Aerts and Chapin 2000; Gusewell and Koerselman 2002). The ratios of N/P are very high (range 44.9–85.0; mean 70.5 ± 15.1) in this study, indicating that soil P is one of the limiting factors (possibly) in TDEFs. However, the present study concentrated on limited soil depth (10 cm); studies on soil nutrient concentrations up to 100 cm would reveal the impacts of soil P in TDEFs.

Physiognomy and Leaf Types

The phenomenon of predominant occurrence of evergreen leaf habit on poor nutrient soil is in conformity with the predictions of Givnish (1984), who argued that plants with evergreen leaves are common in habitats with nutrient-poor soils. Deciduous leaf habit is less common when compared to evergreens. As species with persistent simple leaves are common when compared to plants with compound leaves, TDEFs surveyed in the present study retain ever-greenness throughout the year. Previously, Givnish (1978, 1984) reported the predominant occurrence of compound-leaved individuals in arid and semi-arid environments and at low elevation–deciduous forest ecosystem. Although the TDEFs of our study occur in low elevation, semi-arid drier environment, with a combination of poor nutrient soil, water scarcity, high temperature and dissymmetric rainfall regime, they thrive well as unique evergreen forests with a predominance of plants with simple leaves, evergreen leaf habit.

Relationship Between Leaf Area and Wood Density

The results of our study carried out are similar to those of earlier authors who conducted research pertaining to LA and WD across a range of vegetation types and soil characteristics. They reported a negative relationship between LA and WD and opined that this traits relationship may be quite generally true. Ackerly (2004) reported a negative relationship between LA and WD (0.33–0.80 g cm^{-3}) in 20 chaparral species, California; Cavender–Bares et al. (2004) found a negative link between WD and LS in 17 Floridian oak species; Diaz et al. (2004) researched on 12 standardised traits with floras of four countries (Argentina, England, Iran and Spain) and estimated a negative relationship between LA and WD; Pickup et al. (2005) recorded a negative relationship ($r^2 = 0.44$) between LA and WD, when researched on 70 species in two contrasting sites (high, low rainfall and high, low nutrient) in Australia. Likewise, Rossetto and Kooyman (2005) reported a significant negative relationship between LS and WD when performed correlation with 90 species in Australia; Malhado et al. (2009) reported a significant negative relationship between LA and WD in Amazon forest with large set of species (2788) (leaf size varied from nanophyll to macrophyll); Wright et al. (2007) worked on six ecological traits across seven neotropical forests (WD range 0.10–1.11 g cm^{-3}; LS range 2.6–257000 mm^2) and observed a strongest and most consistent negative relationship between LA and WD. In addition, Ackerly and Cornwell (2007) found a negative relationship (LS–WD within plots = −0.76; among community = −0.83) between LA and WD with 54 native species in California, USA, their study includes range of species, from riparian deciduous woodland to sclerophyll chaparral shrubland; Kooyman et al. (2001) reported a significantly higher negative relationship between LS and WD ($r = -0.75$).

Although LS–WD relationship is frequently reported from range of vegetation types and environmental gradients, the mechanism underlying the association has not yet been studied well (Wright et al. 2006). A growing body of literature stated that the lower wood specific gravity of stem is linked with higher stem hydraulic conductance per unit sap wood area (Ks), which possibly support larger leaves; however, this hypothesis still needs to be broadly tested and proved (Ackerly 2004; Bucci et al. 2004; Santiago et al. 2004; Wright et al. 2007). Further, Ackerly (2004) and Cavender–Bares et al. (2004) put forwarded that within the habitat, species with higher Ks can possibly able to transport more water and thus deploy a larger total leaf area per stem. Westoby (1998) stated that for survival and ecological opportunities, a species is highly depending on other species of their community; thus, within site, the community produce diverse leaf sizes. Ecologists and eco-physiologists should travel a long way to decipher the biological secrets hidden in the life-supporting green natural world.

Conclusion

This study sheds light on existing relationship between two important plant functional traits, namely leaf area and wood density, in an understudied, endangered tropical forest ecosystem of the Indian subcontinent. Results of our study are in line with those of earlier reports by different investigators who have researched on range of vegetation types, woody life forms, altitudinal and latitudinal, rainfall, temperature, and soil nutrient gradients. However, this study concentrated only on trees in TDEF; similar studies of this kind on other woody life forms, namely liana and shrub, are necessary to predict the overall relationship between LA and WD. Findings of this study will be of immense value in afforestation programs in southern Coromandel Coast region of peninsular India. If trees of LA (range = 65–9765 mm^2) and WD (range = 0.41–1.05 g cm^{-3}) planted on Coromandel Coast region, they thrive well and deliver ecosystem services as fullest because species characteristic of eutrophic conditions cannot perform well under poor nutrient soils (Pywell 2003). Poor nutrient soil and higher temperature may be attributed to higher wood density and smaller leaf size in our study area. Our results on relationship between two traits were already broadly reported by studies from around the world except India. The researchers recognised that lesser rainfall, poor nutrient soil and higher temperature are linked with smaller LA and higher WD. Studies on other important ecological traits outlined by Cornelissen et al. (2003) and testing the well-established ecological traits relationship, e.g. specific leaf area–leaf dry matter content, specific leaf area–leaf lifespan (LL), WD–LL and WD–growth rate, are need of the hour to learn about the real eco-physiological nature of TDEFs. Testing the Corner's rule with TDEF species will be of great eco-physiological importance. Eventually, more studies on plant functional traits of TDEFs could pave a path to understand how the existing species form their niches in nutrition-scarce TDEFs.

Predominant occurrence of evergreens in TDEFs shows its climatic–climax plant succession stage and old-growth status.

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Variations on Pulping Properties of *Gmelina arborea* Roxb. Grown in Different Geographical Regions of Tamil Nadu, India

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Abstract *Gmelina arborea* (Family: Verbenaceae) is considered as a useful multi-purpose species. The wood of *Gmelina* is used as raw material for cellulose, and it has received attention as a source of good quality pulp, medium density fiberboard and plywood because of its beautiful white color and stronger fiber. The aim of this study was to determine the fiber dimensions of xylem elements to check their pulping quality. A total of 21 trees were selected randomly at different altitudes (37–1387 m) in Tamil Nadu. The results revealed that the fiber length and fiber diameter ranged from 1113.16 ± 89.05 to 1804.97 ± 144.40 μm and 24.08 ± 1.93 to 53.19 ± 4.25 μm with an average of 1473.01 ± 194.26 μm and 36.09 ± 6.39 μm, respectively. High lumen diameter (39.36 ± 3.14 μm) and fiber wall thickness (6.92 ± 0.55 μm) were observed in TESHG-2. Slenderness ratio (SL) is one of the important derived indices to determine tearing property of pulp. The highest SL ratio was recorded in TTPJG (67.91) followed by TPDKG (55.70). Out of 21 trees, four trees such as TTPJG, TCBSG-7(Siruvani), TESHG-8 (Sathyamangalam) and TCBSG-24 (Siruvani) had appreciable SL ratio, Runkle's ratio and fiber length. Therefore, these trees may be used as a source for selective breeding especially for pulp and paper industries.

Keywords *G. arborea* · CPTs · Fiber length · Altitude · Pulp and paper

Introduction

Gmelina arborea is a fast growing timber-yielding species originated from South and Southeast Asian countries such as India, Pakistan, Sri Lanka and Myanmar. It was latter introduced to Bangladesh, Thailand, Southern China, Vietnam, Indonesia

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and Philippines (Jensen 1995). *G. arborea* has received good attention as a source of quality pulp, medium density fiberboard and plywood due to its beautiful white color and stronger fiber than other fast growing tree species like Acacias and *Paraseranthes* spp. (Zobel and Sprague 1998; Wahyudi et al. 1999, 2000). Though *G. arborea* was considered as a potential high-grade timber for making furniture and constructions, little work has been done on the timber quality (Dvorak 2004). Gmelina has high pulping properties while compared with other hard wood species. (Lamb 1968; Palmer and Gibbs 1974; Doat 1976). During the formation of vascular cambium, xylem elements are affected by many factors like site, ecological conditions, management and age of trees growing in plantations (Zobel and van Buijtenen 1989). The wood strength, value and utility of wood species are certainly determined by the principal element of the fiber (Akachukwu 2001).

The paper industries desire to utilize wood with long fiber for pulp and paper production (Panshin and de Zeeuw 1980; Jorge et al. 2000). Fiber length and derived values are important for quantifying pulp and paper quality in which the former can influence the tearing index (Biermann 1996; Bowyer et al. 2003; Smook 1997) while the later can be used to assess the suitability of species (Ogbonnaya et al. 1997; Ververis et al. 2004). Fiber dimensions and their ratios provide greater information about the quality of pulping properties (Ademiluyi and Okeke 1979). Though the fiber characteristics of *G. arborea* are considered as prime sources for pulpwood industries, the studies on these aspects with relation to different geographical regions are still lacking. Hence, the present investigation had been carried out to compare the fiber dimensions of *G. arborea* grown in different geographical regions of Tamil Nadu, India, and to identify the best fiber-producing trees for increasing pulp and paper quality.

Materials and Methods

Study Area

A total of 21 trees were selected from different geographical regions of Tamil Nadu, India, based on growth superiority. The sampled trees lie between the latitude of 10°18′–11°48′ N° and longitude of 10°18′–11°48′ N°. The altitude at which the trees selected ranged between 37 and 1387 m (Table 1).

Sample Collection and Measurement

The core wood samples were collected from each of the selected tree at breast height (1.37 m) using increment borer. These samples were cut into small pieces with razor blade and placed in test tubes containing Hydrogen peroxide (H_2O_2),

Table 1 Details of geographical location of selected trees of *Gmelina arborea*

S. No	Location name	Code	Latitude(N°)	Longitude (E°)	Altitude (m)
1	Pudukkottai	TPDKG	10°18′	79°02′	75
2	Tiruchirappalli	TTPJG	10°46′	78°41′	107
3	Bargur	TESBG	11°45′	77°33′	992
4	Keelapalaiyur	TAKPG	11°28′	79°25′	37
5	Yercuad	TSYCG-1	11°46′	78°11′	1070
6	Yercuad	TSYCG-2	11°47′	78°12′	1387
7	Yercuad	TSYCG-3	11°47′	78°12′	1382
8	Yercuad	TSYCG-4	11°48′	78°12′	1375
9	Yercuad	TSYCG-7	11°48′	78°12′	1415
10	Anaikatti	TCAKG-1	11°03′	76°46′	956
11	Anaikatti	TCAKG-2	11°03′	76°46′	971
12	Siruvani	TCBSG-7	10°56′	76°43′	483
13	Siruvani	TCBSG-16	10°57′	76°41′	446
14	Siruvani	TCBSG-24	10°57′	76°40′	560
15	Siruvani	TCBSG-30	10°57′	76°42′	521
16	Siruvani	TCBSG-49	10°57′	76°41′	515
17	Sathyamangalam	TESHG-2	11°40′	77°10′	1180
18	Sathyamangalam	TESHG-8	11°36′	77°07′	1174
19	Sathyamangalam	TESHG-10	11°36′	77°06′	1137
20	Sathyamangalam	TESHG-14	11°36′	77°06′	1202
21	Sathyamangalam	TESHG-15	11°36′	77°04′	1215

water and glacial acetic acid in the ratio of 1:4:5 followed by maceration (Peterson et al. 2008). About 30 fibers were randomly picked from each sample for studying morphological characteristics of fiber dimensions such as fiber length, fiber width, lumen diameter and cell wall thickness. Fiber length and other fiber dimensions were measured using the procedure prescribed by Sharma et al. (2013). Fiber-derived values such as Runkle's ratio, slenderness ratio and flexibility coefficient were calculated using standard procedures (Ververis et al. 2004; Silitonga et al. 1972). All the fiber dimensions were photographed by Leica DM RBE RS232G camera attached microscope and measured using image analyzer (Fig. 1).

Statistical Analysis

The data were analyzed by applying Software Package (SPSS ver. 20, IBM Corporation 1989, 2011, US). One-way ANOVA Post hoc multiple comparisons was made using LSD Duncan's test for each fiber dimensions of *G. arborea* in order to reveal the differences on wood properties between samples.

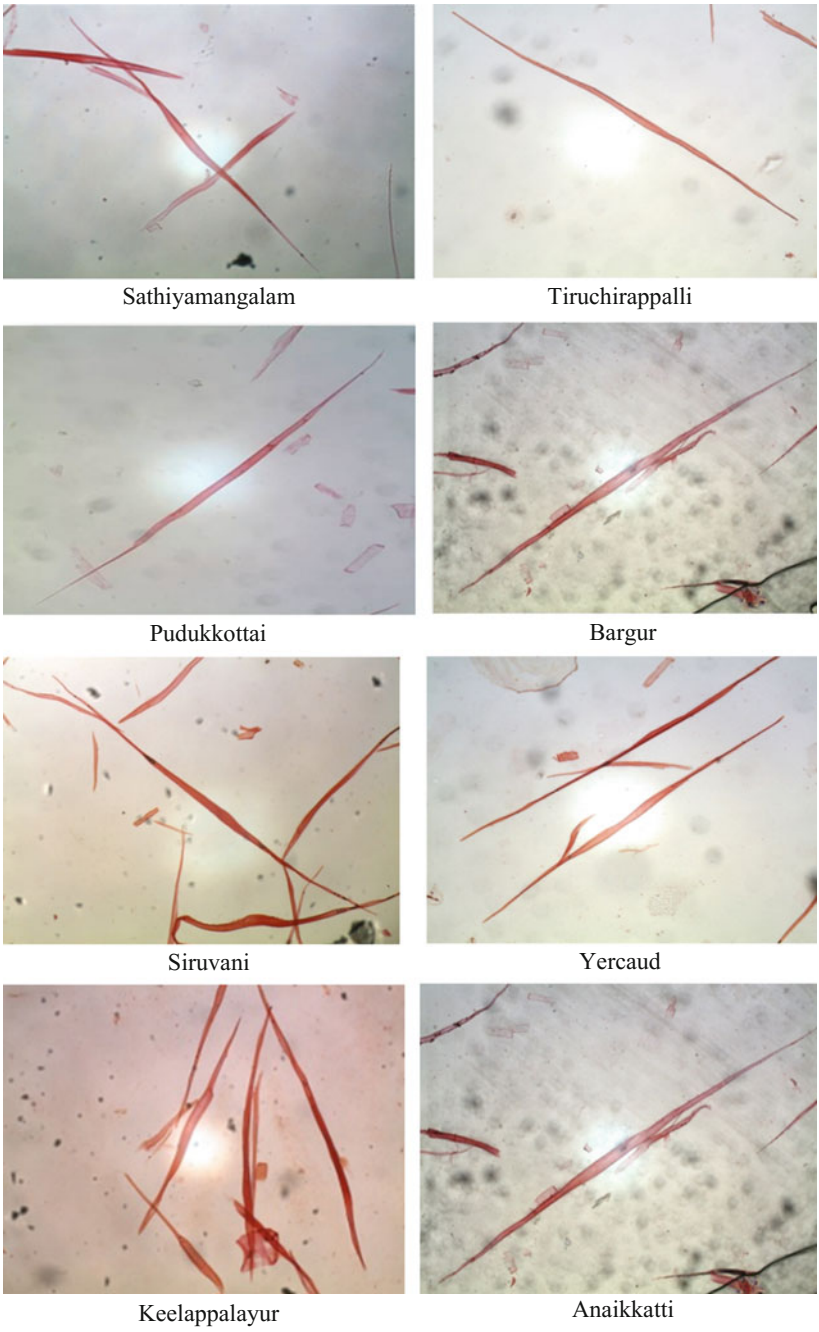

Fig. 1 Fiber images from Selected CPTs of *Gmelina arborea* in Tamil Nadu

Result and Discussion

Fiber Dimensions

It has been well established that fiber length fiber diameter and lumen diameter are genetically controlled (Wheeler et al. 1965; Zhang and Jiang 1998). The fiber dimensions such as fiber length, fiber diameter, lumen diameter and cell wall thickness observed for all 21 trees of *G. arborea* grown in different geographical region of Tamil Nadu are shown in Table 2. The results showed a significant difference was observed on the trees selected from different geographical locations. The fiber characteristics and lumen size are important parameters for pulping quality in which the later affect rigidity and paper strength. The results revealed that the fiber length and fiber diameter ranged from 1113.16 ± 89.05 (TESHG-15 of Sathyamangalam) to 1804.97 ± 144.40 µm (TCBSG-24 of Siruvani) and 24.08 ± 1.93 µm (TTPJG of Tiruchirappalli) to

Table 2 Fiber dimensions in xylem elements of selected *G. arborea* trees

S. No	Locations	Fiber length (µm)	Fiber diameter (µm)	Lumen diameter (µm)	Fiber cell wall thickness (µm)
1	TPDKG	1636.57 ± 130.93	29.67 ± 2.37	20.97 ± 1.68	4.35 ± 0.35
2	TTPJG	1433.26 ± 114.66	24.08 ± 1.93	15.58 ± 1.25	4.25 ± 0.34
3	TESBG	1359.82 ± 108.79	33.05 ± 2.64	22.78 ± 1.82	5.1 ± 40.41
4	TAKPG	1413.70 ± 113.10	34.60 ± 2.77	19.54 ± 1.56	7.53 ± 0.60
5	TSYCG-1	1459.70 ± 116.78	32.87 ± 2.63	21.92 ± 1.75	5.48 ± 0.44
6	TSYCG-2	1459.60 ± 116.77	42.31 ± 3.38	30.71 ± 2.46	5.80 ± 0.46
7	TSYCG-3	1436.65 ± 114.93	36.78 ± 2.94	25.33 ± 2.03	5.72 ± 0.46
8	TSYCG-4	1388.12 ± 111.05	38.91 ± 3.11	27.02 ± 2.16	5.94 ± 0.48
9	TSYCG-7	1279.79 ± 102.38	32.74 ± 2.62	22.99 ± 1.84	4.87 ± 0.39
10	TCAKG-1	1637.36 ± 130.99	30.80 ± 2.46	21.71 ± 1.74	4.54 ± 0.36
11	TCAKG-2	1403.79 ± 112.30	37.05 ± 2.96	26.50 ± 2.12	5.27 ± 0.42
12	TCBSG-7	1633.60 ± 130.69	35.37 ± 2.83	27.27 ± 2.18	4.05 ± 0.32
13	TCBSG-16	1776.82 ± 142.15	36.15 ± 2.89	25.11 ± 2.01	5.52 ± 0.44
14	TCBSG-24	1804.97 ± 144.40	43.54 ± 3.48	32.01 ± 2.56	5.77 ± 0.46
15	TCBSG-30	1607.47 ± 128.60	41.04 ± 3.28	30.14 ± 2.41	5.45 ± 0.44
16	TCBSG-49	1395.03 ± 111.60	36.70 ± 2.94	25.93 ± 2.07	5.38 ± 0.43
17	TESHG-2	1385.89 ± 110.87	53.19 ± 4.26	39.36 ± 3.15	6.92 ± 0.55
18	TESHG-8	1461.34 ± 116.91	38.16 ± 3.05	29.37 ± 2.35	4.40 ± 0.35
19	TESHG-10	1604.60 ± 128.37	38.46 ± 3.08	27.24 ± 2.18	5.61 ± 0.45
20	TESHG-14	1242.01 ± 99.36	30.50 ± 2.44	20.81 ± 1.66	4.85 ± 0.39
21	TESHG-15	1113.16 ± 89.05	32.02 ± 2.56	22.09 ± 1.77	4.97 ± 0.40
	Mean ± SD	1473.01 ± 194.26	36.09 ± 6.40	25.45 ± 5.38	5.32 ± 0.91
	CD Value	22.54	0.79	0.68	0.11
	F value	6.218	12.723	18.694	11.579
	p value	0.000	0.000	0.000	0.000

53.19 ± 4.25 μm (TESHG-2 of Siruvani) with an average of 1473.01 ± 194.26 and 36.09 ± 6.39 μm, respectively. Bowyer et al. (2003) opined that the long fiber is used to produce high tear strength of pulp or paper. Recently, Sharma et al. (2013) reported that fiber length and fiber diameter of *G. arborea* in Arunachal Pradesh, India, ranged from 1131.33 to 2131.33 μm and 28.06 to 52.00 μm with an average mean of 1478.97 and 37.96 μm, respectively. Similarly, the observed length of fiber in the study is almost equal (or some times better) to those reported for various hard wood species. For instance, the fiber length of 800–1650 μm for *Melia azedarach* (Abdul Wasim 2007), 1243–1900 μm for *Casuarina equsitifolia* clones (Warrier et al. 2015), 812–1147 μm for *Eucalyptus grandis* (Bhat et al. 1988) are in agreement with the data obtained in this study. The lumen diameter and cell wall thickness ranged from 15.58 ± 1.25 μm (TTPJG in Tiruchirappalli) to 39.36 ± 3.15 μm (TESHG-2 of Siruvani) and 4.05 ± 3.15 μm (TESHG-7 of Siruvani) to 7.53 ± 3.15 μm (TAKPG of Keelapalaiyur), respectively, with an average of 25.45 ± 5.38 and 5.32 ± 0.91 μm. Emerhi (2012) had reported that thicker cell wall reduces the paper quality. All the individuals included in the study registered a less value for lumen diameter and cell wall thickness, thereby making it more favorable for the production of the quality paper. Sharma et al. (2013) had also reported that these two traits were in the range of 18.20–44.20 and 2.60–11.70 μm, respectively. Interestingly, more than 40% of the trees in this study exhibited cell wall thickness less than 5 μm.

Fiber-Derived Indices

Runkle's ratio is considered as one of the important traits for pulp and paper production. Many authors have stated that Runkle's ratio with less than 1 is good for paper making and if the value is more than 1, it is very difficult to collapse and form bulkier paper with less bonded area (Ohshima et al. 2005; Dutt et al. 2009; Jang and Seth 1998; Kpikpi 1992). The Runkle's ratio of all the 21 trees showed less than 1 and varied from 0.30 (TCBSG-7, TESHG-8) to 0.78 (TAKPG) indicating that all the trees can be utilized for pulp and paper making (Table 3). Slenderness ratio (SL) is another important derived index to determine tearing property of pulp and paper making which is determined from fiber length and fiber diameter. Trees with maximum SL ratio tend to produce good quality pulp and paper with high tearing resistance. The results revealed that the SL ratio was high in TTPJG of Tiruchirappalli (67.91) followed by TPDKG of Pudukkottai (55.71). Xu et al. (2006) suggested that SL ratio of fiber dimensions more than 33 is considered for good pulp and paper production. The current investigation all the Gmelina trees had SL ratio of more than 33 except for TESHG-2 in Sathyamangalam (25.27). Flexibility coefficient determines the degree of fiber bonding in paper sheet which is derived from lumen diameter and fiber diameter. Earlier, Smook (1997) had reported a high flexibility coefficient value ranging from 50 to 75 for both hard and softwood species. The flexibility coefficient of Gmelina selected at Siruvani (TCBSG-7) and Sathiyamangalam (TESHG-8) was 77.10 and 76.97, respectively, implying that they are rich in elastic and elastic fiber.

Table 3 Fiber-derived indices of selected trees of *G. Arborea*

S. No.	Location	Runkel's ratio	Slenderness ratio	Flexibility coefficient
1	TPDKG	0.42 ± 0.04	55.71 ± 4.46	70.66 ± 5.65
2	TTPJG	0.56 ± 0.05	67.91 ± 5.34	64.71 ± 5.18
3	TESBG	0.42 ± 0.04	38.57 ± 3.08	69.48 ± 5.55
4	TAKPG	0.78 ± 0.06	39.12 ± 3.13	56.47 ± 52
5	TSYCG-1	0.50 ± 0.04	44.42 ± 3.55	66.67 ± 5.34
6	TSYCG-2	0.38 ± 0.03	34.50 ± 2.76	72.58 ± 5.81
7	TSYCG-3	0.45 ± 0.04	43.02 ± 3.44	68.87 ± 5.51
8	TSYCG-4	0.44 ± 0.04	35.54 ± 2.84	69.46 ± 5.56
9	TSYCG-7	0.42 ± 0.04	42.46 ± 3.40	70.23 ± 5.62
10	TCAKG-1	0.42 ± 0.03	40.23 ± 3.22	70.49 ± 5.64
11	TCAKG-2	0.41 ± 0.04	46.61 ± 3.73	71.54 ± 5.72
12	TCBSG-7	0.30 ± 0.03	39.26 ± 3.14	77.10 ± 5.17
13	TCBSG-16	0.44 ± 0.04	46.69 ± 3.73	69.46 ± 5.56
14	TCBSG-24	0.36 ± 0.03	41.26 ± 3.30	73.51 ± 5.88
15	TCBSG-30	0.36 ± 0.03	44.06 ± 3.52	73.43 ± 5.88
16	TCBSG-49	0.42 ± 0.04	41.78 ± 3.34	71.07 ± 5.5.69
17	TESHG-2	0.35 ± 0.03	25.27 ± 2.02	73.99 ± 5.92
18	TESHG-8	0.30 ± 0.02	36.72 ± 2.94	76.97 ± 5.16
19	TESHG-10	0.41 ± 0.03	38.57 ± 3.08	70.83 ± 5.67
20	TESHG-14	0.47 ± 0.04	54.09 ± 4.33	68.22 ± 5.46
21	TESHG-15	0.45 ± 0.04	34.09 ± 2.72	68.99 ± 5.46
	Mean	0.43 ± 0.10	42.37 ± 2.72	70.23 ± 5.52
	CD value	0.01	1.17	0.59
	F value	23.693	19.659	1.789
	p value	0.000	0.000	0.056

Conclusion

The present investigation provides a new insight in selecting trees from natural population that hold potential pulp wood properties. The statistical analysis revealed that though the fiber dimensions and its derived indices for all trees in the present study were found to be good for pulp and paper making due to its ample fiber length (>1000 μm) and Runkle's ratio (<1). Nevertheless, only four trees namely TTPJG (Tiruchirappalli), TCBSG-7(Siruvani), TESHG-8 (Sathyamangalam) and TCBSG-24 (Siruvani) exhibited appreciable SL ratio, Runkle's ratio and fiber length. Therefore, it is suggested that these four trees are suitable and can be selected with top priority for pulp and paper making. These individuals can serve as a base genetic resources in initiation of species improvement programme in future course of research for producing good quality fibers in *G. arborea*.

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Biochemical Characterization of Wood Lignin of *Hevea brasiliensis*

S. Pramod, C.P. Reghu and K.S. Rao

Abstract Lignins are phenolic polymers of the plant cell wall and functionally associated with mechanical support, sap conduction, defense mechanisms, strengthening to plant tissue and imperviousness to biodegradation. The biosynthesis of lignin and polyphenol derivatives in living trees, especially in fast growing woody species is known to improve the quality and durability of timber. The biochemical characterization of wood lignin of two elite clones of *Hevea brasiliensis* (rubberwood), viz. RRII 105, RRII 414 and one wild germplasm accession of *Hevea* viz. AC 4830 was carried out through Klason lignin analysis, FTIR spectroscopy, Thioacidolysis and GC-MS analysis. FTIR spectroscopy revealed that most of the peaks in the cell wall Residue (CWR) represent major cell wall polysaccharides such as cellulose, hemicelluloses and pectins. When the Klason lignin was subjected to FTIR analysis, the absorbance in the polysaccharide region from 1200 to 900 cm^{-1} was strongly diminished compared to that of CWR. The peaks at 1627, 1541, 1505, 1461, 1322, 1285 and 1010 cm^{-1} corresponding to Syringyl (S) and Guaiacyl (G) lignin monomers became more prominent. Thioacidolysis/GC-MS analysis revealed significant variation in the S/G ratio between different clones of *Hevea brasiliensis*. Corresponding to the variation in total lignin content in three clones, significant variation in S/G ratio was also noticed. RRII 105 and AC 4830 were characterized by high amount of S-lignin in its monomeric composition.

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Introduction

Lignins are the second most abundant group of biopolymers of the plant cell wall after cellulose (Roth et al. 1997; Boudet 2000) and functionally associated with mechanical support, sap conduction, defence mechanisms, strengthening of plant tissue and its imperviousness to biodegradation (Gierlinger et al. 2004). Lignification is a regulated and dynamic process subject to modulations during normal development and response to different environmental stresses. Enzymatic and genetic engineering studies on lignins carried out by various researchers earlier revealed a specific route to the synthesis of lignin precursors in the cytoplasm, which is translocated to the cell wall for polymerization (Boudet 2000; Gierlinger et al. 2004) and the regulation and transport or polymerization affects the quantity of lignin produced.

Hevea brasiliensis (Euphorbiacea), a perennial tropical hardwood tree species, indigenous to Amazon Valley of South America, were introduced to India in the later half of eighteenth century (Teoh et al. 2011) and commercially cultivated for natural rubber. Generally the economic life span of rubber trees pertaining to natural rubber production is 25–30 years, after which the timber, which is popularly known as rubber wood, is being widely utilized as an eco-friendly potential source of timber for various industrial applications. The depletion of tropical forests leading to a shortage of timber resulted into the special attention towards rubber wood as an alternative source of timber (Naji et al. 2011). Rubber wood is considered to be suitable for the manufacture of various stable wood products such as doors, windows, particle board, plywood, furnitures, floor tiles, moldings etc. after proper processing (Gnanaharan et al. 2002). The estimated total availability of rubber wood (Rubber Grower's Companion 2014) was 2.30 million m^3 during 2012–2013 of which stem wood accounted for 1.38 million m^3 and the consumption pattern was dominated by plywood (45%), followed by packing case sector (40%), treated wood sector (10%), safety matches and others (5%).

The utility of wood for any commercial purposes is correlated with structure and chemical constituents. The chemical composition of rubber wood includes lignin (20%), holocellulose (70%), α-cellulose (40%), hemicellulose (20%), extractives (6–10%) and 0.8–2.3% ash (Zhao et al. 2008). Among these components lignin plays significant role in determining the suitability of wood for timber related products. From a functional point of view, lignin impart rigidity to cell walls, facilitate water transport, and impede the degradation of wall polysaccharides, thus acting as a major line of defence against pathogens, insects, and other herbivores. Lignin is a phenolic polymer with hydrophobic properties that improves rigidity and impermeability of xylem cells (Douglas 1996; Mellerowicz et al. 2001) and

protecting cellulose fibres from chemical and biological degradation (Grabber et al. 1998).

In dicotyledonous woody species like *H. brasiliensis*, lignin consists of two monomeric units, namely Guaiacyl (G) and Syringyl (S) lignin units. The total lignin content and its monomeric composition are important determinants of wood properties. Heterogeneous nature of lignin content and composition within and between wood species has already been reported earlier (Pramod 2012). Reghu et al. (2007) reported that the localization of cinnamyl alcohol dehydrogenase (CAD) and quantification of lignin in the juvenile growth phase of *H. brasiliensis* as an early selection parameter for wood quality. However, information on the structural composition and characterization of lignin monomeric units in rubber wood is meagre. Therefore, the present study was aimed to investigate the quantification and characterization of wood lignin of two elite clones and one wild germplasm of *H. brasiliensis.*

Materials and Methods

Two popular clones of *H. brasiliensis* (rubberwood) namely RRII 105, RRII 414 and one wild *Hevea* Germplasm accession viz. AC 4830 from the germplasm source bush nursery of Rubber Research Institute of India, Kottayam, Kerala, India were selected for the study. Wood samples were collected from three plants per clone at the age of four years for lignin analysis. Fresh wood samples collected were frozen in liquid nitrogen for 1 h. and subsequently freeze dried for 72 h at −80 °C. The frozen wood tissue was separated by removing the pith and bark portion and pulverized in a ball mill and pass through the 180 μm sieve. The grounded wood of the wood was then sequentially extracted with distilled water, ethyl alcohol, alcohol: toluene (1:1) and acetone (Merck, Germany) in Soxhlet extractor and dried to get extractive free xylem residue (EXR).

FTIR Spectroscopy

The Cell wall residues (CWR) and Klason lignin (KL) were used for FTIR analysis. KBr pellets for IR spectroscopy were prepared using macrotechnique (13 mm pellet; Ca. 1.5 mg sample with 350 mg KBr). The spectra were recorded with the FTIR spectrometer with TGS detector (Perkin Elmer, Spectrum GS, USA) at resolution of 4 cm^{-1} for 32 scans in the range from 600 to 4000 cm^{-1}; background spectra of clear window was recorded prior to acquisition of sample spectra. The spectrum of the background was subtracted from spectra of the sample before conversion into absorbance units. For each sample, three different sub samples were analyzed and averaged to obtain mean spectrum per individual sample.

Determination of Lignin Content

Lignin analysis was carried out on dry extract free cell wall residue grounded to pass through a 180 μm sieve before exhaustive solvent extraction toluene: ethanol (2:1;v/v), ethanol and water). The lignin content in the wood was determined by Klason lignin method (Dence 1992). Acid soluble lignin content was determined by measuring the UV absorption at 205 nm using extinction coefficients of $110 \times 1\ g^{-1}\ cm^{-1}$ of H_2SO_4 hydroxylate.

Thioacidolysis

Thioacidolysis was carried out according to Lapierre et al. (1995). The reagent was prepared by introducing 2.5 ml of BF_3 etherate (Aldrich) and 10 ml of ethanethiol EtSh (Aldrich) into a 100 ml flask and adjusting the final volume to 100 ml with dioxane. A mixture of the sample (12 mg) and 12 ml of reagent were put in tube fitted with Teflon lined screw cap. Thioacidolysis was performed at 100 °C in an oil bath for 4 h with an occasional shaking. The cooled reaction mixture and the washings with water were combined with and the mixture was poured over 1 ml Dichloromethane (Fluka, Germany) including internal standard (0.50 mg tetracosane from Sigma, Germany). After adjusting the pH of the aqueous phase to pH 3–4 with 0.4 M sodium carbonate aqueous solution, the aqueous phase was then extracted thrice with dichloromethane (20 ml). The combined organic extracts were dried over Na_2SO_4 and the solvent was evaporated under reduced pressure at 40 °C in a rotary evaporator. The residue was re-dissolved in dichloromethane (1 ml). The thioacidolysis products (7 μl) were sylated with 50 μl of N, O-bis (trimethylsilyl) trifluroacetamide (BSTFA, Sigma, Germany) and 5 μl of pyridine (Sigma, Germany) in a 200 μl GC vial with Teflon lined screw cap and kept in room temperature for overnight. The sylated products were separated by Gas chromatography using silicon based capillary column (30 m × 0.25 m × 250 μm) and each peak was identified by GC/MS. The temperature program of the GC increased at a rate of 5 °C/min from 100 to 280 °C and then final temperature was maintained for 60 min.

Results and Discussion

FTIR Analysis of Cell Wall Residue (CWR) and Klason Lignin (KL)

FTIR analysis of cell wall residue (CWR) of rubberwood showed prominent peaks in the fingerprint region of 1800–600 cm^{-1} (Fig. 1). The peaks were numbered and

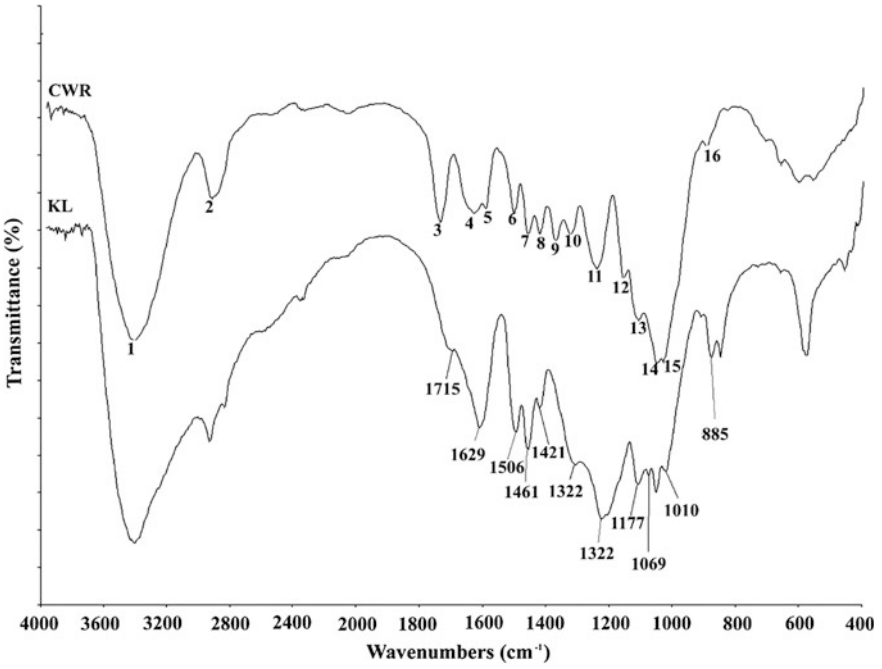

Fig. 1 FTIR spectra of grounded wood (CWR) and Klason lignin (KL) from the wood of *H. brasilienis*

assigned to chemical compounds according to the published literature as shown in Table 1. Most of the peaks in the CWR represent major cell wall polysaccharides such as cellulose (peaks 9, 12, and 14), hemicelluloses and pectins (peaks 3, 8, 13, and 14).

When the Klason lignin was subjected to FTIR analysis, the absorbance in the polysaccharide region from 1200 to 900 cm^{-1} was strongly diminished compared to that of CWR (Fig. 1). The peaks 4, 5, 6, 7, 10, 11 and 15 (1629, 1506, 1461, 1322 and 1010 cm^{-1}) corresponding to S and G lignin types became more prominent in Klason lignin. The peak 3 in the FTIR spectra of CWR at 1738 cm^{-1} corresponding to unconjugated carbonyl and carboxyl group in xylan (Akerholm and Salmen 2001; Chen et al. 2010) were shifted to position 1715 cm^{-1}. The peak at 1715 cm^{-1} can be formed due to conjugated acids and unconjugated carbon groups in lignin (Xu et al. 2011) and guaiacyl ring breathening in G lignin (Rana et al. 2009) respectively.

We assume that this shift could be due to the cross linkage between xylan and G-lignin. The shift in wavenumber can be associated with the inductive effect of substituents in the aromatic ring system of lignin (Rana et al. 2009). On the other hand, the peak numbers 6, 7 and 10 (1505–1510, 1459–1461 and 1322–1329 cm^{-1}) belongs to G and GS type lignin did not show any peak shift in FTIR spectra of

Table 1 Band assignments of cell wall residue (CWR) and Klason lignin (KL) from wood of *H. brasiliensis*

No.	Wave number (cm^{-1})		Band assignment	References
	CWR	KL		
1	3410	–	O–H Stretching vibration	Faix (1991)
2	2906	–	C–H stretching vibration	Faix (1991)
3	1738	1715	C=O Stretching vibration	Faix (1991) and Harington et al. 1964
4	1639	1629	C=O stretching vibration in lignin	Harington et al. 1964
5	1599	1599	Aromatic skelton vibration plus C=O stretch; S > G; G condensed > G etherified	Faix (1991)
6	1510	1506	Aromatic skelton vibration, Benzene ring stretch in lignin(G > S)	Faix (1991)
7	1461	1461	C–H Deformation vibration	Faix (1991) and Harington et al. 1964
8	1425	1421	CH_2 scissor vibration and CH_3 bending vibration	Faix (1991) and Harington et al. 1964
9	1375	–	CH_3 bending	Harington et al. 1964
10	1329	1322	S-ring + G-ring condensed in lignin	Faix (1991)
11	1247	1284	CH3 COOR acetic ester (Upward direction)	Rana et al. 2009
12	1161	1177	C–O–C antisymmetric bridge stretching vibration	Faix (1991) and Harington et al. 1964
13	1111	–	O–H association band	Harington et al. 1964
14	1056	1069	C=O stretching	Harington et al. 1964
15	1035	1010	aromatic C–H in plane deformation G > S plus C=O stretch	Faix (1991)
16	897	885	Anomeric carbon group frequency	Harington et al. 1964

CWR and KL indicating that they may represent pure lignin peaks without the interference from any substituent on its aromatic ring.

The chemical assignments of peak at 1629 cm^{-1} is very complex, it has been attributed to many noncellulosic polysaccharides such as pectin, xylan, galactan, arabinogalactan (Marga et al. 2003) and aromatic skeleton vibration in highly conjugated C=C bond of lignin (Harrington et al. 1964). In the present study, this peak also showed a shift in its position in the spectra of CWR and KL indicating the complex lignin-hemicellulose interaction. Many of the prominent peaks in the FTIR spectra of CWR specific to cellulosic polysaccharides i.e., peaks 9, 12 and 14 (at 1375, 1161 and 1056 cm^{-1}) was diminished in the spectra of the Klason lignin of the same samples. This shows that the cellulosic polysaccharides have significantly removed from the cell wall residue during acid hydrolysis. The peak 13 (at 1111 cm^{-1}) in the CWR belong to xyloglucan was also removed in the spectra of

Table 2 Klason lignin content, guaiacyl and syringyl lignin yield and S/G ratio of lignin from three clones of *H. brasiliensis*

Clone	Klason lignin (%)	Guaiacyl lignin	Syringyl lignin	S/G rtio
RRII 105	21 ± 0.8	431.57 ± 24	1125.96 ± 22	2.61 ± 0.2
RRII 414	16.0 ± 1	529.47 ± 28	648.89 ± 25	1.22 ± 0.3
AC 4830	27.43 ± 1.5	525.98 ± 18	1120.47 ± 32	2.13 ± 0.05

G, S, S + G: Yields of the thioethylated guaiacyl (G), Syringyl (S) and total (S + G) expressed as μmoles per gram of lignin. Mean values from the two trees × two samples per each tree are given in table

Klason lignin. On the other hand, the peaks 3 and 14 (at 1721–1738 and 1056–1069 cm^{-1}) corresponding to hemicelluloses was observed in the spectra of both CWR and KL. This may be due to their cross linkage with lignin resulting in resistance to acid treatment.

Lignin Content

For the determination of lignin content by Klason method, cell wall residue was subjected to acid hydrolysis and acid insoluble lignin was estimated using Klason method and data are given in Table 2. Lignin content was high in the wild accession, AC4830 (27.43%) compared to the popular clones RRII 105 (21%) and RRII 414 (16%). The latex yield is the primary concern for the development of new clones of *H. brasiliensis.* The elite clone RRII 414 has reported to have relatively high latex yield compared to RRII 105 (Saraswathyamma et al. 2006) whereas the wild *Hevea* accessions, in general are very poor latex yielders (Reghu et al. 2012). Present study showed less klason lignin content in the high latex yielding variety RRII 414 followed by RRII105 while a significant increase in lignin content was evident in the wild accession AC4830. This indicates the possible inverse relationship between total lignin content and latex yielding potential of different varieties of rubber plants.

Lignin Monomeric Composition

Thioacidolysis is an acid catalysed reaction which involves the solvolysis in dioxane/ethanethiol (9/1) and 0.2 M boron trifluride etherate resulting in the depolymerization of lignin. The target structures are mainly β-aryl ether structure

which is the most typical structures in native lignin specifically give rise to thioethylated H, G and S monomers with a high reaction yield (Lapierre 1993). The main aromatic monomers recovered from the thioacidolysis of triplicated samples of rubberwood lignin were identified after sylation by GC mass spectroscopy and quantified by GC. The major peaks resulting from erythro/threo forms of guaiacyl and syringyl monomeric units were used for the calculation of concentration of these monomers in rubberwood lignin. The GC response factors of the main syringyl and guaiacyl trithioethyl C_6C_3 diastereomers defined as the ratio of relative concentration to relative area, are identical or equal to 1.50 using tetracosane as an internal standard (Lapierre 1993).

The results of the gas chromatographic separation of thioacidoltytic monomers from lignin of three varieties *of H. brasiliensis* are displayed in Fig. 2. The S/G ratios in the wood lignins evaluated using the sum of the G and S types of thioethylated monomers are listed in Table 2. The S/G ratio was higher in the RRII 105 (2.61) followed by AC4830 (2.13) while least S/G ratio was observed in RRII 414 (1.22). Although this variation in S/G ratio is related to reduction in total lignin content, it is noteworthy that a higher yield of guaiacyl lignin units (529.47 μm/g) compared to that of RRII 105 (431.67 μm/g) and AC 4830 (525.98 μm/g). Guaiacyl lignin units are more condensed form of monomers compared to syringyl units and provides more mechanical strength during conductive function of the wood (Xu et al. 2006). Therefore, an increase in proportion of guaiacyl units could be a mechanism to balance the mechanical properties when total lignin has reduced in RRII 414. Interestingly, RRII 105 showed maximum yield of syringyl units compared to AC4830 in spite of its less lignin content compared to the later. The lignocellulosic materials with high syringyl units have potential applications in many paper and pulp industry, bioethanol production etc., due to their easy degradability using chemical/biological delignification processes (Boerjan et al. 2003). The content and chemical structure of wood components particularly lignin content and its composition in terms of its guaiacyl (G) and syringyl (S) moieties are important parameters in paper and pulp industry in terms of delignification rates, chemical consumption and pulp yield (Rencoret et al. 2007). The higher reactivity of S lignin compared to G lignin in alkaline system is known (Tsutsumi et al. 1995) and therefore the lignin S/G ratio in hard woods affect pulping efficiency. This is mainly because of highly branched structure of guaiacyl lignin due to rapid polymerization and coupling by mainly condensed bonds in contrast to the syringyl lignin formed by extensive coupling of less condensed β-O-4 linkages (Boerjan et al. 2003). Therefore, further detailed studies on content and composition of cell wall polysaccharides in different varieties of *H. brasiliensis* is required to confirm the suitability of the wood for such industrial applications.

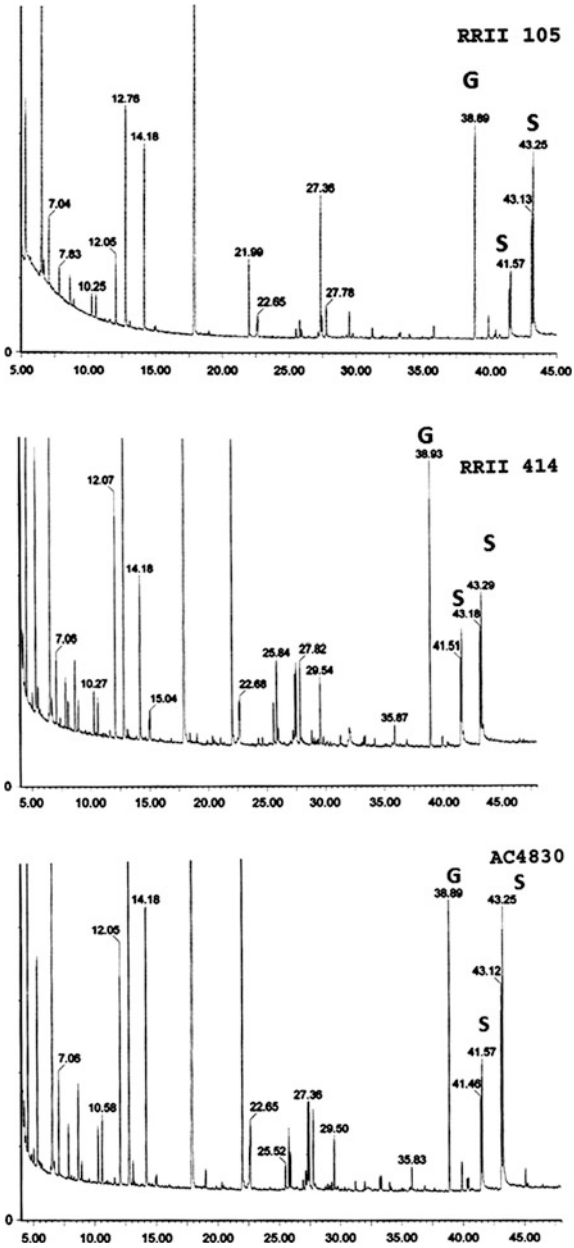

Fig. 2 Gas chromatographs of TMS thioacidolysis products recovered from wood lignin of *Hevea brasiliensis*. G: Guaiacyl lignin, S: Syringyl lignin

Conclusions

Present study revealed the variation in quantity and monomeric composition of lignins in elite clones and wild accessions of *H. brasilienisis*. IR spectroscopy, Klason lignin analysis and Thioacidolysis/GC-MS analysis revealed the cell wall polymers in rubberwood both qualitatively and quantitatively. This study indicate the occurrence of significant variation in the klason lignin content and S/G ratio between the wild (AC 4830) and two high latex yielding clones (RRII 105 and RRII 414) of *H. brasiliensis*.

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Part II
Wood Protection

Protection of Wood: A Global Perspective on the Future

Jeffrey J. Morrell

Abstract The current state of wood protection is briefly reviewed, and then the issues that are currently affecting preservative treatments are summarized. The strategies for addressing these issues are discussed in relation to the role of wood as a renewable building material. The potential for addressing biological attack, ultraviolet light degradation and dimensional stability in a single product are discussed in relation to the need to produce a longer-lasting material that retains the environmental attributes of wood.

Keywords Wood deterioration · Wood protection · Preservatives · Barriers · Wood modification

Introduction

Wood and wood-based materials are among our most important renewable materials with many desirable properties, but susceptibility to damage by combinations of sunlight exposure (primarily ultraviolet light), repeated wetting/drying and biological degradation remains as major negative attribute. The agents of deterioration can combine to markedly shorten the useful lives of many wood-based products. Shorter service lives diminish the value of wood as a renewable resource while placing additional pressure on our forests.

While estimates of total global losses to degradation are scarce, Boyce (1961) long ago suggested that 10% of the timber harvested in the USA was used to replace wood that had failed prematurely in service due to biodeterioration. Extended globally, the UN Food and Agricultural Organization (FAO 2006) estimated global timber harvests to be 3 billion m^3 per year, with 60% of this production being used for products and the remainder for fuel. The 10% of harvest

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figure would translate into 180 million m^3 of wood that could be conserved by controlling degradation losses. This does not account for other squandered resources associated with energy consumption during harvesting and processing as well as installation, environmental impacts, and economic effects of the added harvesting. Clearly, limiting degradation can have sizable impacts on both economies and quality of life. While it would be virtually impossible to completely eliminate this loss, it is readily apparent that wood must be used more efficiently and protected more fully if it is to reassume a leading role as a critical structural material. Preservative treatments already contribute to improved wood conservation through extended service life, but there are opportunities for improvement. An important aspect of this effort must be the continued development of effective strategies for protecting wood against physical agents such as UV light, wetting as well as biological attack.

Protecting wood from all of these agents is certainly not new, but the methods used for protection have come under increasing scrutiny from a skeptical public that questions the use of chemicals for all purposes. For almost two centuries, we have depended on heavy-duty preservatives such as creosote, pentachlorophenol or heavy metal combinations for wood protection, but public pressures have encouraged substitutions in many applications. Changes have not been uniform globally, and examining the various strategies and patterns of change may help us to take a more holistic approach to wood protection. In this paper, we will review the general trends in wood protection in North America with references to activities taking place elsewhere. For the purposes of this review, we will concentrate on long-term protection of exterior exposed solid wood products, thereby avoiding the limited market for whole-structure treatments and treated composites. While we recognize that naturally durable wood species have a role to play, they will not be discussed here and we will restrict ourselves to initial wood treatments excluding those used strictly to limit fungal mold and stain attack.

Current State of Affairs

Although wood protection is a global need, the vast majority of treated wood is used in temperate climates and the bulk is used in North America (Vlosky and Shupe 2006). This market constitutes approximately 60% of the total global market for treated wood. It is unfortunate that the areas with the most critical needs for wood protection tend to employ these technologies, but the higher costs of treatment largely limit their use to developed nations. There remains a critical need for low-cost wood protection for developing countries in tropical regions where deterioration rates are more severe.

The North American markets have long been dominated by the so-called heavy-duty wood preservatives. Industrially, creosote, pentachlorophenol and heavy metal-based systems remain the dominant preservatives for industrial applications. While there have been challenges to the continued use of these

chemicals, the producers have generated the required data to demonstrate that these systems can be safely used with minimal environmental impacts. The US Environmental Protection Agency and Canada's Pesticide Management Regulatory Agency have both reviewed chemicals under their jurisdictions and continue to allow industrial uses (note: there are some differences in chemicals allowed between the two countries). In general, industrial uses of chemicals have been judged on their technical merits and very few chemicals are banned outright, although they may be restricted to specific uses. At the same time, some alternatives for industrial wood protection have emerged, including copper naphthenate and alkaline copper compounds. However, users, who are, by nature, conservative in adopting new systems without long-term data, have been slow to adopt these systems.

On the residential side, the market was long dominated by chromated copper arsenate (CCA), but the 2004 decision by the manufacturers to withdraw the use of CCA for residential applications created opportunities for new systems. Much has happened in the intervening decade. The first CCA alternative was alkaline copper quaternary, closely followed by alkaline copper azole. These systems both depend upon copper as the primary biocide with smaller amounts of a carbon-based biocide to protect against copper-tolerant organisms. Alkaline copper systems have been touted as more environmentally friendly because they lack arsenic or hexavalent chromium. However, they also contain much higher levels of copper than CCA and this can pose issues with regard to metal migration from the treated product. The high pH of these systems also creates the potential for corrosion of unprotected steel connections, necessitating the requirement for either hot-dip galvanized or stainless steel hardware. Despite their different handling characteristics, the use of these systems rapidly grew and they dominated the residential markets until the recent introduction of micronized copper systems. Micronized systems use finely ground copper suspensions in place of solubilized copper, along with either a triazole or quaternary ammonium co-biocide (McIntyre and Freeman 2008). Micronized systems are widely used to treat southern pine, which is highly permeable and easily treated; however, these systems are not suitable for more difficult to impregnate species, making them less suitable for treatment of most Canadian wood species as well as woods from the western USA. The shift to micronized systems has not been without debate because of concerns about the lack of long-term performance data and the lack of standardization by the American Wood Protection Association (AWPA); however, they appear to be performing well when properly applied.

The primary suppliers of wood preservative systems have also been working to develop metal-free alternatives (Morris 2002). These systems can incorporate mixtures of triazoles, carbamates, quaternary ammonium compounds and various insecticides. While they appear to be working well for non-soil contact applications, they are not yet suitable for direct ground contact. As we will discuss later, the potential for replacing metal-based preservative with these organics has largely been muted by their inability to perform well in soil contact. Interestingly, some producers of these colorless products have had to add colorants including small amounts of copper because the public expects treated wood to be colored.

At the same time, the North American market has seen the emergence of alternative systems including various wood extracts, silanes, and a host of other systems that claim to provide non-biocidal protection. Unfortunately, there are very little publically available data to support these claims. There have also been attempts to introduce acetylated wood and heat-treated wood into the market, but these products have not achieved substantial market acceptance, primarily because of higher cost.

Europe has seen the emergence of a host of alternative protection methods including acetylation, thermal modification and furfurylation. Ironically, both acetylation and thermal modification have roots in North American research dating back to the 1950s. The situation in Europe is a bit different owing to a very different regulatory structure and a public willingness to pay more for wood products coupled with a lower risk of decay in many parts of the continent. This has fostered a willingness to look more closely at alternatives and a seeming willingness to accept some level of reduced performance. This has allowed the development of products with shorter expected service lives. This approach recognizes the tendency of wood users to more often remove wood products from service for changes in appearance rather than any loss in biological performance. However, this approach does have a negative side in that shorter service lives mean that wood products will not perform as well in life cycle analyses. The outcome of shorter service life can be negative when the tree required to replace the product takes longer to grow than the resulting product service life.

Europe has been the center of developments in dimensional stabilization, heat treatment, silanes, and barriers or coatings (Hill 2006). All of these processes invariably produce materials that are more costly, but these costs do not appear to be a barrier to market entry, perhaps because alternative (non-wood) materials also have higher costs.

Future Concerns

In order to more fully understand where the use of treated wood is headed, we need to understand why changes are necessary.

There is no doubt that society has a strong desire for the use of less toxic chemicals for all purposes and wood protection is no exception. At the same time, there is increasing public concern about the potential for migration of preservatives into the surrounding environment. Virtually all of the currently used wood preservatives have some degree of water solubility. In addition, these molecules tend to have a much greater effect in aquatic environments because nontarget organisms are literally bathed in the chemical. Concerns about preservative migration have led some regulatory bodies to severely restrict the use of treated wood (Brooks 2011a, b; WWPI 2012).

Another factor affecting the use of treated wood is disposal. The rules regarding disposal vary widely across the globe. In the USA, the first recommendation for

treated wood that has reached the end of its useful life is to reuse it in a similar application. For example, a utility pole might become a parking barrier or a railway sleeper might become a landscape timber. Ultimately, the wood will no longer be useful in any application. In most of North America, treated wood can be disposed of in lined municipal solid waste facilities (landfills) provided that it meets certain criteria. Virtually all wood treated with oil-borne preservatives meets these requirements, and there is an exemption for water-based systems such as CCA. There is no shortage of landfill capacity in many parts of North America, and this has made it difficult to develop alternative disposal options. Most industrial-treated wood is given away or reused, while most residential-treated wood appears to be placed into landfills.

Despite the lack of a major incentive to avoid landfilling, some options are emerging. Wood treated with oil-based materials contains almost 20% by weight of oil and represents a valuable energy source. At present, creosoted railway sleepers are burned for energy production, but poles and other products are more difficult to process because of the presence of penta, which has more restrictive combustion permitting requirements. As a result, little penta-treated wood is currently burned, but could be a useful bioenergy resource. The other issue related to disposal is the presence of heavy metal-treated wood in waste streams that are destined for combustion. The final hurdle to developing alternative methods for resulting or recycling treated wood is the cost of collecting a widely dispersed material with differing degrees of treatment (Smith et al. 2002). Given the current costs of collecting a widely dispersed material, landfilling seems the most viable option in North America, but disposal represents a key lingering issue among wood users.

New Approaches

As with any industry, technologies related to preservative-treated wood must continue to advance or alternative materials will be substituted. There are a number of opportunities involving new chemistries, treatment methods, non-biocidal treatments and coatings.

New Chemistries: The process of developing a new wood preservative can vary from as little as 5–10 or more years. This includes developing toxicological as well as performance data. In general, it is not economical to develop a chemical solely for wood protection. Many agricultural pesticides have been adapted for wood use as evidenced by the use of triazoles for wood protection. While chemicals are often developed without close public scrutiny until they are released, the time periods required for establishing efficacy of wood protectants generally result in gradual emergence of chemicals for increasingly more aggressive environments (Cabrera and Morrell 2012; Pernak et al. 2004; Schultz and Nicholas 2006; Schultz et al. 2004; Zabielska-Metjuk et al. 2004). One disconcerting observation for the development of new wood preservatives is the relative paucity of new chemicals entering major markets over the past few years. The exception has been micronized

copper, which has only been commercially available for a few years but now dominates the residential market in the eastern USA (Preston et al. 2008; Cookson et al. 2008; McIntyre and Freeman 2008; Larkin et al. 2008). This system, however, is still dependent on heavy metals and could be viewed as a modification more than a completely new development. The lack of a pool of readily available alternative treatments suggests the need for further development of new chemicals and could be an opportunity for the company that can create the ideal system.

The other area that continues to receive research interest is the potential for using natural products extracts for wood protection (Kawamura et al. 2011; Kondo and Imamura 1986; Li et al. 2008; Schultz and Nicholas 2000). Researchers have long sought to use heartwood extractives as potential wood preservatives; however, the approach has two problems. Extractives removed from highly durable woods are rarely effective when introduced into less durable species. This may reflect that inability to achieve the same microdistribution that was present in the original wood, as well as the tendency for these chemicals to be water soluble and therefore susceptible to leaching. A more important problem is that many naturally durable species are already in short supply, making it difficult to justify cutting more wood for production of natural preservatives. Extraction of by-products such as sawdust may be possible, but this material contains a mixture of non-durable sapwood and heartwood and may therefore produce lower yields. It may be more useful to employ these by-products for the production of durable composites, provided that the materials are compatible with resins.

An alternative to the use of heartwood extracts might be the use of foliar extracts or materials from other organisms (Li et al. 2008). Many plants have evolved to produce foliage that contains an array of compounds designed to discourage attack by bacteria, fungi and insects. Foliage may be an especially attractive source of biologically active compounds because it can be repeatedly harvested without cutting the tree, or alternatively, it could be collected at the same time the tree is harvested for wood. A number of recent studies suggest that foliage extracts exhibited activity against a variety of fungi and insects, although none of the extracts appears to have the broad-spectrum toxicity necessary to function in a natural environment. It may be possible to combine extracts to produce a more effective cocktail of natural products. At the same time, it is important to remember that natural products extracts are, potentially, just as toxic to nontarget organisms as synthetic pesticides. As these compounds are explored, it will be essential that they be tested accordingly to ensure that we do not inadvertently introduce more toxic molecules into the system.

Another interesting natural products approach has been the use of chitosans for wood protection (Maoz and Morrell 2004; Eikenes et al. 2005). These compounds are derived from shrimp-farming operations and are available in large quantities. Modified chitosans have been shown to be effective against a variety of fungi, although their effectiveness against insects remains untested. Nevertheless, they offer the potential for producing antimicrobial compounds from what is largely a waste product. These examples highlight the potential for developing alternative systems from waste streams produced by other processes.

The search for lower toxicity systems for protecting wood against the diverse array of wood-degrading agents will be essential for retaining the viability of wood as renewable construction material in adverse environments.

Non-biocidal Treatments: The protection of wood without biocides has long been a goal of many wood users. The use of glycol to bulk wood and the development of dimensional stabilizers such as acetic anhydride show that wood can be made less susceptible to the water uptake that creates conditions conducive to biological attack (Hill 2006). However, these approaches have drawbacks that include the need to impregnate with large volumes of expensive reactants, lingering odors and textural changes. These systems also appear to be limited to use on a restricted number of highly permeable wood species. Alternatively, heat treatments can be used to modify the hemicelluloses in the wood to render the wood less susceptible to fungal attack (Esteves et al. 2007, 2011; Jamsa and Viitaniemi 1998; Kamdem et al. 2002; Tjeerdsma et al. 1998; Vidrine et al. 2007). However, this process is not completely protective and can reduce wood properties.

Despite their limitations, dimensional stabilization strategies do have some applications. Wood modification clearly limits water uptake, and this reduces the risk of fungal decay; however, the process does not appear to alter susceptibility to surface molds or UV degradation (DeVetter et al. 2010a, b; Donath et al. 2004; Dubey et al. 2012; Lande et al. 2004; Mai and Militz 2004; Metsa-Kartelainen and Viitanen 2012; Pfeffer et al. 2012; Weigel et al. 2012). Thus, there remains a need for non-biocidal treatments that are more broadly effective against abiotic and biotic agents of deterioration.

New Treatment Practices: The wood treatment processes employed to impregnate the majority of treated wood used globally date to the middle part of the nineteenth century. The seeming lack of progress in this aspect of wood protection stems, in part, from the limited ability to overcome the inherent impermeability of heartwood and the overall effectiveness of existing treatment processes. In essence, good performance of properly treated materials has limited interest in investing in entirely new treatment technologies. Despite the overall acceptance of existing processes, there is considerable opportunity for both improving the quality of treatment and placing the chemical in the wood in such a way that it is less likely to migrate outward once in service.

Reducing the risk of preservative migration has become a major concern in some regions, notably where treated wood is used in close proximity to riparian zones. While there is no doubt that some chemical will migrate from treated wood, the goal is to ensure that the levels remain below those capable of inducing a negative environmental effect. Models have been developed that use migration rates for a given volume of treated wood coupled with information about specific waterway conditions such as pH or water current speed to predict total releases over time (Brooks 2011b). These predictions can then be compared to known minimum effects levels for various organisms. At the same time, treatment practices have been modified to reduce the risk of over-treatment, remove surface deposits of chemical, reduce the risk of bleeding in service and, where ever possible, ensure that preservatives have been immobilized or reacted with the wood. These best

management practices are required in many localities across North America (WWPI 2012).

At the same time, there is still a need for new treatment processes that result in more effective preservative penetration. While much of the coniferous wood treated globally has thick bands of easily treated sapwood, there are many species that resist impregnation. Developing methods for effectively treating these woods would help improve performance, thereby reducing the need to harvest additional trees. Modifications to existing liquid treatments, with the possible exception of dual treatments involving an initial boron treatment with a diffusion period, following by subsequent over-treatment with a heavy-duty wood preservative are limited by the inherent impermeability of the resource. The further development of supercritical fluid treatment processes offers the potential for overcoming the inherent refractory nature of many major wood species (Kjellow and Hendriksen 2009; Morrell et al. 1997). This process is currently commercially used in Denmark and has been explored elsewhere, but the high costs of entry in terms of equipment have largely limited development. Ultimately, SCF impregnation will emerge as a viable technology as we move to carbon-based systems and employ more wood-based composites.

There is a need for continued development of other novel systems for impregnating wood and for limiting the ability of the treatment to migrate outward once installed.

Coatings: While we have developed preservative systems capable of protecting wood against biological degradation for 50 years or more, most treated wood ultimately fails because its appearance declines to the point where the user will no longer accept it. This remains a major problem for wood in exterior applications.

Coatings can reduce damage caused by ultraviolet light as it strikes the wood and also reduce the ability of the wood to sorb water, thereby reducing the wetting and drying that leads to warping, twisting and checking.

UV degradation of lignin on the wood surface, coupled with subsequent removal of other wood components, markedly reduces wood appearance (Feist 1990; Hon and Chang 1984; Schauwecker et al. 2009). While opaque coatings can reduce this damage, most wood users want to see the natural grain and color of the wood. Transparent or semi-transparent coatings can provide some protection, but this protection generally declines within 1–2 years of exposure. Developing effective treatments that can be impregnated into wood to provide long-term UV protection remains a major challenge. Iron oxide pigments, titanium dioxide or hindered amine light stabilizers are just a few of the many possible surface protectants that have shown some promise, but most are rapidly inactivated by sunlight (Schauwecker et al. 2009; Schmalzl and Evans 2003; Rowell and Banks 1985). Water repellency is often produced through the inclusion of various waxes or silicates in the treating solution (Levi et al. 1970; Lesar and Humar 2011; Sun et al. 2010). These treatments can reduce the rate of water uptake, but add cost to the system and only slow water uptake.

Ultimately, however, wood protection must be considered in a more holistic fashion. Biological performance is important, but so is resistance to water and UV

light. The material must not only remain structurally sound, it must look sound as well. If it does not, the wood is replaced prematurely. It is also important to alter the premise that wood has to be the less expensive alternative. Homeowners have shown a willingness to pay 2–3 times more for wood/plastic composites that promise infinite service life with no maintenance. These materials have their own issues, but they highlight the potential for upgrading wood materials to reach a higher market.

Material specifiers are increasingly comparing the environmental attributes of materials to make specifying decisions. One of the most important emerging tools for these comparisons is life cycle analysis (LCA). The LCA examines all of the inputs required to produce a product including energy and water along with the environmental impacts. There is no correct answer regarding a given material. LCAs allow users to compare the impacts of different materials that can be used for the same application. Wood, by virtue of its renewability, low manufacturing impacts and ability to sequester carbon, should have a major advantage in these comparisons. However, service life plays an important role in these comparisons. Premature removal of wood sharply increases the overall life cycle impact. Thus, factors such as weathering and wood instability must be considered in performance because they often lead to premature wood replacement.

As a result, biological protectants, water repellents and coatings must all be considered as an integral part of a wood protection system that ensures long-term performance. Another performance component is the original wood. Some species are inherently prone to warping and checking. While it is unlikely that species will be replaced, it may be possible to selectively sort lumber for treatment. For example, dimensional changes tend to be greatest in the tangential direction in most wood species (flat-sawn wood). Selecting materials that are vertically sawn would result in a lower tendency to shrink and swell. Careful material selection would reduce the tendency of treated wood to check and deform in service.

None of these approaches is without some cost; however, it is also important to avoid the view of wood as the cheapest material. In North America, treated wood is typically the least expensive decking material, followed by naturally durable heartwoods and finally by wood/plastic composites (WPCs). Surveys show that consumers perceive these products in terms of increasing quality in the same order. Purchasers have clearly shown a willingness to pay a premium for products that they perceive to be more durable and less maintenance intensive. At the same time, extensive advertising has convinced them that WPCs are greener. Wood-based materials, however, should have more favorable LCAs provided that they are properly treated, and consumers have demonstrated their willingness to pay for materials they perceive to combine greenness, durability and low maintenance. There appears to be niche for the development of a durable, more dimensionally stable wood product.

Barriers: Preservative treatment is ultimately a barrier that precludes entry by wood-degrading organisms, but there have been recent efforts to develop physical barriers to protect wood. The first successful products originated in South Africa in response to early failures of creosoted utility poles, and these products have spread

across the globe (Baecker and Behr 1995; Behr and Baecker 1994; Behr et al. 1997). In some cases, they encapsulate untreated wood, but generally, they involve coating of preservative-treated wood. Barriers reduce contact between soil and wood, thereby diminishing the risk of fungal decay and insect attack. They also reduce the potential for preservative migration from wood into the surrounding environment. Barriers clearly reduce the risk of environmental contamination, but they may also have a side benefit. Since less chemical will migrate from the wood and soil is not in direct contact, the barrier may allow the use of lower preservative loadings to produce equivalent protection. Barriers can be simple polyethylene barriers or heavy plastic sleeves applied by shrink wrapping. Other systems spray polyurea on the wood surface to provide a flexible coating whose thickness is based upon the environment to which the wood is exposed. Several barriers systems are currently standardized by the American Wood Protection Association (AWPA 2012). These systems add cost, and users must clearly determine if the added expense is worthwhile, but they help address the issues related to biocide mobility.

New Opportunities

Wood has a long history of use in a variety of applications, and preservative treatments have played a major role in the extension of useful life, but there are still other opportunities for growth in the use of treated wood. Among these applications are wood used as solid packing material in global trade, wood used in mass timber structures and a higher-end decking product.

Wood pallets seem to be everywhere, and most people assume that they have always been used, but palletized shipping only dates back to the Second World War. Pallets make shipping easier and fast, but the lower-quality wood used in these pallets and other solid wood packing materials can harbor insects and fungi (Morrell 1995). These organisms can be inadvertently introduced into new environments during shipping. Nearly all countries require that solid wood packing materials used in global trade be subjected to some type of mitigation treatment. The two most commonly applied treatments are heating to 56 °C for 30 min or fumigation with methyl bromide (FAO 2002). These treatments are not verifiable, nor do they prevent reinvasion. Preservative treatment may provide a more verifiable method for limiting the risk of pest introduction that also provides long-term protection against reinvasion. Preliminary tests of solid wood packing material infested with the new house borer (*Arhopalus productus*) suggested that beetles were not killed by treatment with ACQ, borates or an organic preservative mixture, but also never completed their life cycle (Schauwecker and Morrell 2008). Clearly, much additional work needs to be completed before preservative treatment is approved as a mitigation tool, but the volumes of wood used in this area are well worth the effort.

Mass timber structures are seeing increasing use in more temperate climates as a part of efforts to compete with concrete and steel in the high-rise building market. Cross-laminated timber is one of the primary products used in this area. While this

material has a number of advantages over alternative materials, it will ultimately need some types of protection against biological degradation. This protection need not entail heavy-duty wood preservation, but the fact that all buildings eventually leak means that these structures will experience water intrusion that creates conditions suitable for fungal attack. Some types of treatment will be needed to ensure performance. These appears to be a hesitancy to use traditional wood preservatives in this application, but alternatives such as thermal modification may find some use creating new markets for durable wood.

The most promising potential new market for treated wood is decking. Treated wood long dominated this market; however, wood/plastic composites (WPCs) have continued to erode market share. Declining market share has been less noticeable because the overall decking market has also grown, masking the change. Wood decks have generally been perceived as of lower quality than either WPC or naturally durable decks; however, there is also a general desire to use wood in decks. There is an opportunity to create wood decking products that are both durable and able to remain visually attractive for a longer period of time. Consumers have already shown their willingness to pay more than two times the cost of a treated wood deck for a WPC deck. There is clearly an opportunity to create a better decking product that is cost competitive with WPC products but incorporates features that make it more durable. These features might include a carbon-based wood preservative, selection of materials that are more stable (i.e., vertical grain), and application of UV stabilizers to the wood. The resulting product would not compete with traditional lower cost wood decking, but rather with the higher-end products.

Conclusions

Wood remains one of our most important renewable building materials. Continued use of this material under adverse conditions will require renewed interest in developing technologies that resist biological and physical damage. Some of these technologies are already available, but remain too costly. Other approaches are under exploration. Effectively protecting wood against biological and physical damage without depending on broad-spectrum pesticides must remain a goal if wood is to retain its rightful place in a green society.

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Conveyor Belt Pressure Impregnation of Wood

Peter Vinden, Grigori Torgovnikov and Anil K. Sethy

Abstract The *Unitreat* or *Conveyor belt* treatment process utilizes residual vacuum trapped in the wood to improve preservative distribution, once the treated commodity has been removed from the treatment plant (Vinden 2003). Unlike traditional industrial wood treatment technologies, (Bethel, Lowry and Rueping processes) limited quantities of preservative are metered into the wood rather than using traditional methods of treating to refusal or low final rates of flow. This paper explores a range of treatment options that benefit from this technology. Highlights of the technology include: (a) very short and low pressure treatment periods, whilst maintaining full sapwood penetration (b) absence of any final vacuum with no preservative dripping or wood sugar contamination of preservative. (c) hot treatments with very rapid preservative fixation and no sludge formation (i.e. reaction between CCA preservatives and wood sugars in the parent solution). Industrial applications of the *Unitreat* technology include: (a) treatment of pressure steamed green pine round-wood with copper-chrome-arsenic preservatives, (b) conveyor belt processing using microwave technology, (c) treatment of framing timber with water-based boron preservatives, (d) vapour phase treatment with boron preservatives, (e) chemical modification, (f) antisapstain chemical impregnation. Large-scale microwave conditioning substitutes high-pressure steaming and provides conveyor belt treatment processes whereby trees are converted into poles or railway sleepers that are ready for use within minutes rather than days or weeks. Most importantly, treatment with microwave technology extends the number of wood species that can be preservative treated by rendering the wood more permeable. This is achieved by micro-incising the wood during microwave processing.

Keywords Wood preservation · *Unitreat* process · Conveyor belt treatment · Microwave modification of wood

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Introduction

In New Zealand and Australia, large-scale preservative treatment of radiata pine posts and poles is undertaken following pressure steam conditioning at 127 C. Steaming times vary depending on post or pole diameter. Traditionally, the steamed wood is then either treated by the Alternating Pressure Method (APM) within 24 h, once the round-wood has cooled (Vinden and McQuire 1978; Viden 1987), or the "Q" process where the logs are left to air dry under cover for 7–21 days to allow further moisture loss (typically 300 L/m^3) and moisture redistribution (McQuire 1974). The success of treatment arises from the rupturing of soft unlignified uni-seriate ray tissue that is prevalent in pine species (Plate 1). This provides radial penetration pathways for rapid penetration of liquids rather than depending on longitudinal penetration pathways through vessels.

Disadvantages of APM treatment arise from wood sap mixing with the parent preservative. This often results in sludge formation. A further disadvantage of pressure steaming is the loss of strength associated with long steaming times. Typical strength losses can include an 18% loss in modulus of elasticity (MOE) and up to 25% loss in modulus of rupture (MOR). Strength loss has not been an issue, however, and from a commercial perspective, simply requires acknowledgement in any engineering design calculations. The strength losses may only be relevant in timber or round-wood used above Fibre Saturation Point (FSP) since anecdotal

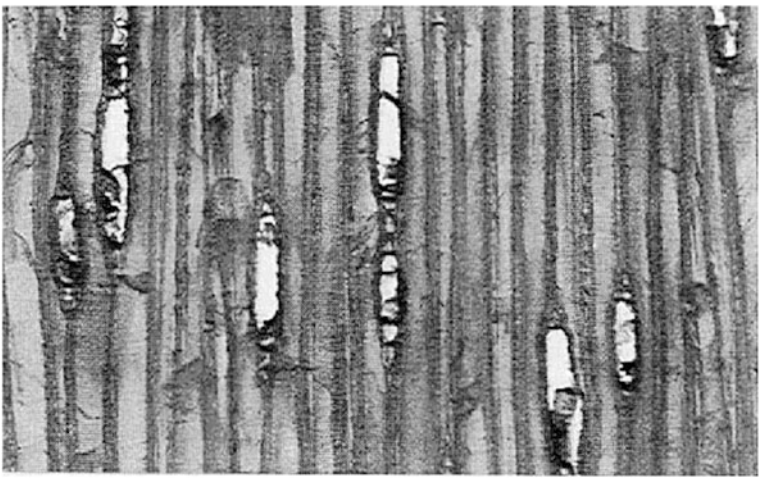

Plate 1 Longitudinal/tangential section of Radiata pine. Soft uni-seriate ray tissue destroyed following pressure steam release

evidence indicates that the wood regains much of its original strength once the wood has been dried below FSP (Collins and Vinden 1987).

Heat Stability of Copper Chrome Arsenic (CCA)

Commercial experience indicates that CCA precipitates when heated. However, virgin CCA (free of wood sugars) is stable to high temperatures (Vinden and Cobham 1995). A mixture of wood sugars and CCA preservative will sludge as a result of hexavalent chrome (Cr^6) converting to trivalent Chrome (Cr^3). This reaction results in the precipitation of CCA, one of the basic mechanisms resulting in CCA fixation in wood. However, any wood sugar contamination of CCA, for example from kickback operations, will also result in the reactions continuing in the store tank. This gives rise to irreversible sludge formation that often appears as a green slime on the surfaces of posts, poles and sawn timber. However, if the CCA is kept free of wood sugars, the preservative will remain heat stable.

A patent developed by Vinden and Cobham (1995) "*Hot CCA Treatment*" describes a process whereby heated (CCA) preservative is pressure impregnated into wood. Whilst under pressure, compressed air is introduced into the treatment cylinder and the treatment solution removed from the treatment vessel whilst retaining the pressure in the treatment vessel with air. When pressure is released at the end of the pressure cycle, any air present in the wood tends to expand and kick back residual preservative on or near the surface of the wood. Preservative and a mixture of wood sap (rich in sugars) will exude from the wood surface and drip and collect in the bottom of the treatment cylinder. This process is aided by the application of a vacuum after treatment to further expand residual air in the wood and maximize kickback. When the vacuum is released any residual chemical on the wood surface should in theory be pulled back into the wood. The method is effective in reducing preservative dripping after treatment, but some dripping still occurs. The technique also usually extends treatment time by at least 30 min.

The Vinden/Cobham invention outlined above describes a separation of kick-back solution from the parent solution. The parent solution is largely free of wood sugars, whereas the kickback solution is full of wood sugars and needs to be treated before being returned to the store tank. However, treatment of the kickback solution for example by ozonation or the addition of acids to prevent sludging is expensive and technically complex.

The *Unitreat* process (Vinden 2003) overcomes the complexity associated with segregating kickback solution and treating the sludge by providing schedules that not only prevent sludge formation but also does away with the need to segregate the kickback preservative from the parent solution. The technique also provides a means of cleaning up contaminated preservative in the store tank (i.e. preservatives contaminated with wood sugars). The process modifies some important aspects of "normal" commercial wood treatment practices. The difference is that preservative is applied so that there is no kickback of preservative from the treated wood. If there

is no kickback, then wood sugars do not leach from the wood into the parent preservative solution, and there is no need to segregate the parent solution from the kickback solution. There is also no need to conduct a final vacuum.

Thus there is no need to empty the treatment cylinder whilst maintaining air pressure in the treatment cylinder. In fact the authors have discovered that maintaining the pressure using air during emptying is disadvantageous. This arises because the air penetrates the wood so that when pressure is released at the end of treatment, there is expansion of air in the wood leading to excessive dripping of preservative when the timber is placed on the drip pad. With the *Unitreat* process, there is no dripping after preservative treatment. This aspect is very important when considering the environmental performance of preservative treatment systems.

The normal process of preservative impregnation requires pressure to be applied until effectively no more preservative can be forced into the wood, i.e. the wood is saturated with preservative and has reached a condition known as "refusal". (It should be noted that some wood species are refractory and cannot be impregnated to any great depth). *Unitreat* applies a partial vacuum to the wood and limited preservative pressure impregnated or metered into the wood to achieve the desired preservative loading required for the particular commodity. Very often this can be achieved within seconds. Treatment is then terminated, and the vessel emptied as quickly as possible.

The use of miniature pressure transducers indicates that after evacuation and once preservative moves into wood under pressure that some areas of the wood remain under vacuum (Cobham and Vinden 1995). The net result is that there is still sufficient vacuum in the wood to continue to pull the envelope of preservative into the wood to achieve total treatment of the permeable sapwood. In addition, there is no preservative dripping and no kickback of contaminated preservative into the treatment cylinder. These features are unique and overcome the deficiencies of existing "Hot CCA treatment". The *Unitreat* process goes further and provides a more economic treatment compared to the existing traditional wood treatment technologies such as Bethel, Lowry and Rueping treatment, because there is no loss of preservative and the treatments are very rapid. The wood treatment solutions and treated wood become very clean and free of sludge. There is no environmental contamination with preservative waste, and the absence of contamination in the parent solution from wood sugars ensures that there is no build up of minute sludge particles in the preservative that might tend to block ray tissue and pit membranes and therefore flow of preservative into the wood during treatment.

Following experimental work in 1999 and 2000, the use of the Unitreat process was tested at Auspine in 2002, a commercial treatment facility located at Kalangadoo, South Australia. The net results following commercial trials indicated that a significant acceleration in treatment could be achieved using the process with no preservative dripping after treatment and rapid fixation immediately following treatment (within 4 h of treatment). Longer-term tests indicated that the storage solution became cleaner with no sludge formation.

Treatment of Pressure Steamed Round-Wood with Copper-Chrome-Arsenic Preservatives

Commercial trials conducted at Kalangadoo tested the feasibility of steam evacuation to meet adequate moisture loss in round-wood prior to preservative impregnation. Normally, the Pressure Steam Conditioning/Bethell ('Q') treatment method is applied to steam conditioned wood which is cool and has been left for at least 7 days to achieve moisture losses of 300 L/m^3 from the green condition and moisture redistribution (McQuire 1974; Vinden 1985). The objectives of the Kalangadoo trials were to:

1. determine whether adequate moisture loss could be achieved by steam evacuation immediately after steaming to undertake Bethel treatment rather than APM treatment. Two schedules were examined and compared. In the first schedule, steam evacuation in the treatment vessel was conducted one hour after steaming had been completed. In the second schedule, there was no delay between steaming and then placing the steamed material in the treatment vessel and continuing moisture evaporation from the timber under vacuum. The vacuums were nominally −85 kPa. However, a consistently high standard of vacuum was achieved (−92 kPa). In all cases, the vacuum was maintained for 45 min. The results of these trials indicated that a consistent standard of preservative distribution could be achieved irrespective of whether the vacuum was applied within 5 min of steam conditioning or an hour after steam conditioning.
2. test the consistency of preservative treatment and preservative fixation. It was anticipated that application of the *Unitreat* process to steam conditioned and evacuated round-wood would provide treated charges completely free of any kickback or preservative dripping and effectively completely fixed preservative soon after treatment. In the trials described below, the fixation of freshly steamed timber is compared with cold timber.

Preservative Treatment Regime

Commodity:

Species: Radiata pine,
On average 85 poles in each pack
Age-thinned material (age 7 years)
Length, 1.79 m. Average Diameter, 115 mm

Preservative composition:

CrO_3 = 47.8%
CuO = 22.9%

As_2O_5 = 29.3%
Preservative solution strength of CCA was 26.3 g/L.

Schedule:

Vacuum to −85 kPa minimum for 45 min
Flood treatment cylinder using vacuum
Pressurise to 370 kPa
Hold at pressure for 5 s
Slow release pressure (ramp-down time 10 min)
Pump cylinder empty.
Preservative Net Uptake of CCA was 240 L/m^3.
Ambient atmospheric temperature = 13 °C

Steady state temperature of treatment cylinders:

Before treatment = 25.4 °C
After treatment = 26.8 °C

CCA Fixation Time Using Simulated Rainfall

Simulated rainfall as described by Walley et al. (1996a, b) was used to determine the effective fixation of CCA after treatment. The volume of water spray was calculated based on average weekly rainfall and the total calculated curved surface areas of poles exposed to the spray. Four "cold"-treated packs were used as controls. Spraying was conducted 1, 4, 9 and 24 h after treatment and the collected run-off analysed for chromium by titration using the methodology outlined in *AS/NZS 1605:2000 (2nd Edition)*. Preservative distribution was determined by spot testing increment cores using rubeanic acid and ammonia—*AS/NZS 1605:2000 (2nd Edition)*.

Results

The state of the poles, for both the pre-heated and cold packs, immediately after treatment was "touch-dry" and completely free of any preservative dripping, i.e. no leachate solution was observed.

Preservative distribution obtained by spot testing increment cores indicated complete penetration of all sapwood in all samples. Thus, a high standard of preservative (CCA) treatment can be achieved in radiata pine round-wood following steam evacuation prior to pressure impregnation. The recommended evacuation time after steaming to remove and redistribute moisture in the wood is

45 min at −85 kPa. Preservative flooding can continue immediately after steam evacuation.

Simulated rainfall leaching trials indicated that the concentration of chromium (Cr) in the leachate solution decreases with time. There was a higher concentration of Cr collected in the solution leachate when sprayed with water after an hour, in comparison with traditional cold treatments. The concentrations were 62.3 and 44.5 mg/L, respectively. However, after 4 h the solutions collected from the heated packs exhibited no Cr content. Solutions collected from the cold pack continued to exhibit concentrations of Cr, but in decreasing amounts up to the 24 h reading.

Accelerated fixation is achieved through a combination of heating the timber (by steam conditioning) and heating the preservative solution. The ambient temperature at Kalangadoo was 13 °C, whereas the steady state treatment solution temperature was 25 °C. The trials indicated a major improvement in fixation by having hot wood. The slightly better initial result for cold wood may have arisen from a higher air pressure in hot wood immediately after treatment compared to the cold wood. However, within 4 h after treatment, the amount of Cr available for leaching is effectively zero. One is led to the conclusion that the steam conditioned timber needs to be held under cover for 4 h prior to sale. However, further work could fine-tune this holding period. There is also an option of heating the CCA solution to a higher temperature, for example, 30 °C.

A delay of up to one hour between steaming and evacuation has no impact on the quality of treatment. Longer delays may be possible without compromising the standard of treatment. However, it is recommended that evacuation is undertaken as soon as practicable after steaming and that any delay should not exceed one hour until work is completed to determine the influence of longer delays.

The *Unitreat* process provides drip free charges of wood immediately after treatment. This is an important contributing factor to the high level of fixation of CCA in the wood immediately after treatment and effectively controls the potential for preservative leaching into the environment. The *Unitreat* process reduces any potential for preservative kickback during treatment. This is also an essential element in the success of the treatment because the quantity of wood sugars being kick-backed into the parent preservative is effectively zero. This facilitates the use of heated CCA without the formation of sludge. From an environmental perspective, this represents a major advance in the environmental application of CCA preservatives. The absence of sludge formation minimizes the production of toxic waste that needs disposal and provides cleaner-treated products. This was evident in the series of trials undertaken at Kalangadoo. The quality of finish (absence of sludge and fines on the surfaces of treated wood) was dramatically reduced on completion of the trials. The recommended schedules for use at Kalangadoo include a treatment pressure of 375 kPa with pressure treatment being terminated, once this working pressure has been held for 2 min. No final vacuum is applied. This schedule has been customized for the commodities (round-wood) and treatment plants at Kalangadoo. Different commodities may require schedule variations to optimize treatment.

Treatment of half or quarter rounds were less effective because of the difficulty of penetrating heartwood in the tangential direction. Radiata pine is refractory when penetration pathways are limited to tangential surfaces (Vinden 1986).

Conveyor Belt Processing Using Micro-Wave Technology

The advantages of using micro-wave heating to substitute pressure steaming arises from the inherent ability of microwaves to penetrate the total cross section of the timber or round-wood and reach the required temperature in minutes or seconds. High temperatures (for example 150 °C) can be achieved throughout the cross section in time periods shorter than two minutes rather than applying pressure steaming times of for example of 5–12 h (depending on the diameter of the log). Such long pressure steaming periods result in hydrolysis of cellulose and inevitable strength loss. Intrinsic strength loss of cellulose can be lower using microwave processing because of the very short heating periods.

High temperatures are possible during MW processing because pressure builds up in the log faster than the steam pressure can dissipate. This results in the same physical modification of wood i.e., blow-out of the soft uni-seriate ray tissue in pine species as is achieved using high-pressure steaming (Plate 1). However, unlike pressure steaming, the effectiveness of microwave modification of wood structure can be extended to the sapwood and heartwood of other wood species, for example, Douglas fir or eucalypts. In fact any wood species can be structurally modified with microwaves and rendered permeable for any application of liquids, even pulping liquids. A further advantage of microwave processing involves the loss of wood moisture on-line during microwave heating, so that treatments may employ "Unitreat" schedules rather than APM.

Extensive applications of microwave technology have been demonstrated using 60, 100 and 300 kW microwave pilot plant. Vinden et al. (1999), Vinden and Torgovnikov (1999, 2000) and Torgovnikov and Vinden (2000). The plants, for example Plate 2, employ special microwave applicators to concentrate or focus microwave energy to where it is needed within the wood cross section to provide macro- and micro-modification of the wood structure.

The *Unitreat* process was developed originally for metering resins into *Torgvin* for *Vintorg* production (sawn timber modified with microwaves and impregnated with wood resins that are then compressed and cured to provide a structural product).

Unitreat was designed to provide conveyor belt processing in-line with micro-wave modification. However, it was soon discovered that the technology could be used in-line to provide impregnation of any liquid (water-borne, oil organic solvent or gases). At its simplest the *Unitreat* process can be applied to green, freshly microwaved eucalypt thinnings. A limited soaking period (for example 15 min) metres the required volume of creosote into the post. Gradual cooling of the post creates a partial vacuum in the post that facilitates total cross-sectional penetration

Plate 2 Continuous 300 kW pilot microwave processing of poles

of the post in both sapwood and heartwood. An interesting observation relates to subsequent drying of treated posts. There is no drastic shrinkage or distortion of posts that have been conditioned by microwaves, unlike the controls that undergo extensive check formation (Plate 3).

Treatment of Framing Timber with Boron-Based Preservatives

The *Unitreat* process has been used commercially to great effect in New Zealand where there is a need to provide total sapwood treatment with water-based preservatives with minimum preservative uptake, minimal swelling of the timber and no subsequent drying operations. Whilst treatment specifications for framing timber using boron preservatives require full sapwood treatment, there is no heartwood treatment requirement. A purpose built pilot plant ("Conveyor Belt Plant") was designed and built by the Cooperative Research Centre Wood Innovation in 2002 (Plate 4). The plant was partially funded by the Australian Forest and Wood Products Research and Development Corporation (now FWPA).

Plate 3 Effect of microwave conditioning on the treatability of eucalypt heartwood. Microwave-treated samples *left*. Controls (*a*) *right*

Unique features included filling and emptying times of 2–3 s each and full treatment cylinder evacuation (−85 kPa) within 10–15 s. Thus, the plant is capable of dipping times of less than 10 s (faster than conventional dipping) and total vacuum/pressure treatment times of less than 30 s. Whilst every attempt was made to replicate such short treatment times in a large automated conveyor belt-driven commercial plant treating 4 packets of framing timber at a time, such a large commercial application required slightly longer treatment times, of the order of 2–4 min. Nonetheless, it will be appreciated that depending on the volumes of timber requiring treatment in a conveyor belt application, treatment times of substantially less than 30 s can be achieved when treatments are fully integrated into manufacturing rather than as separated batch processes.

An overriding objective in designing the *Unitreat* plant is to provide minimal within- and between-treatment variation in the cores of treated samples, for example, the middle ninth of the treated cross section. Whilst there have been efforts to duplicate the *Unitreat* process, for example by applying double vacuum schedules, such treatments have been largely unsuccessful because of large core preservative retention variability (Nasheri et al. 2001, 2006). The *Unitreat* process metres the required preservative retention (in this instance 30 L/m^3). With preservatives like *Boracol* (Glycol borate diluted with water) and permeable wood species like radiata pine, this is achieved in just a few seconds when vacuum is released and

Plate 4 *Unitreat* pilot plant located at the University of Melbourne, Creswick

air pressure applied to achieve the desired uptake as quickly as possible. Thus, the moisture content of pine timber that has been high temperature dried and conditioned to 12% moisture content will not exceed fibre saturation point after treatment on completion of treatment (Plate 5).

Mapanda (1998) investigated the effect of vacuum (ranging from −10 to −85 kPa.) and pressure levels (ranging from 35 to 700 kPa.) on preservative uptake in radiata pine sapwood and demonstrated that preservative uptakes normally exceed 100 L/m^3 when treatments continue to refusal. A standard evacuation time of 30 min was used prior to pressure impregnation; one hour of pressure was selected as effectively treating to refusal and then a final vacuum (−85 kPa) and evacuation time of 30 min was imposed for all charges to maximize kickback.

When pressure impregnation is terminated prematurely (the basis of the *Unitreat* process) preservative uptake is restricted and can be manipulated to any desired loading of preservative, i.e. preservative is metered into the timber. The *Unitreat* process uses the residual vacuum in the wood instead of final vacuum to remove surface or excess preservative to prevent dripping when the charge of timber is removed from the treatment cylinder. A final vacuum becomes largely redundant. Timber is "touch-dry" on removal from the plant without the need for a final vacuum.

Plate 5 Framing timber spot tested for boron preservative. There is full sapwood penetration (*red colour*) and incomplete heartwood penetration (*yellow*)

Mapanda (1998) utilized borax/boric acid as the test preservative but added glycerol to the formulation. This may have improved the penetrating properties of the preservative for example by reducing the surface tension of the preservative.

The permeability of radiata pine sapwood is very high. Continuing evacuation for 30 min prior to pressure impregnation is largely redundant when the objective is to impregnate sapwood. Thus, the *Unitreat* process usually employs zero holding time once the requisite vacuum has been achieved. Thus, the evacuation period continues for just a few seconds. Similarly, time on pressure is usually zero since pressure impregnation continues until the required quantity of preservative is metered into the wood. Once again this is achieved within seconds. Within-charge retention variability is minimized by ensuring rapid filling and emptying of the treatment cylinder. This also ensures accurate metering of preservative into the wood. Thus, total treatment times for radiata pine sapwood can be reduced to seconds whilst maintaining very high standards of treatment.

Vapour Phase Treatment with Boron Preservatives and Chemical Modification of Wood

Wood Conditioning

Conveyor belt microwave conditioning can be applied towards the end of any wood drying process to achieve rapid and accurate final wood moisture content conditioning. This is desirable for many subsequent wood manufacturing activities such as furniture manufacturing where such conditioning minimizes subsequent wood movement and the loss of joint integrity. Microwave conditioning also achieves

moisture levelling in the cross section of the timber and stress relaxation. This is desirable for treatment processes that involve acetylation (chemical modification of wood) and vapour-phase treatments of wood for example the treatment of wood with trimethyl borate (TMB).

Vapour Phase Treatments

TMB is moisture sensitive leading to its conversion to solid boric acid and alcohol. This is the basis of vapour phase treatment of wood (Vinden et al. 1988, 1990, 1991). The wood has to be below 6% moisture content to ensure that total boron penetration can be achieved in the vapour phase. Conveyor belt microwave conditioning of timber for vapour boron treatment (VBT) is ideal because of its accuracy and apparent absence of stress build up in timber as timber is microwaved to 6% mc. TMB can be applied as a liquid for example by momentary immersion. However, such a process is best undertaken under controlled conditions. The *Unitreat* plant is ideal for this purpose since:

- flooding and emptying of the treatment plant can be achieved in seconds;
- a very even coating of preservative can be achieved on individual sticks of timber i.e. there is low between piece variability in preservative uptake;
- consistent schedules using the *Unitreat* process ensures that there is little variability between charges;
- there is no human contact with the volatile liquid;
- contamination of the parent preservative solution (TMB) with wood sap is avoided because of the rapidity of the treatment process. Any moisture contamination of TMB will result in the hydrolysis of the preservative to solid boric acid;
- The *Unitreat* plant can potentially be used for further subsequent treatments to the timber after TMB treatment, for example the application of surface coatings.

The advantages of VBT become apparent when considering the treatment of refractory species that are susceptible to insect attack. Substantially, greater penetration of preservative is possible in the vapour phase relative to liquid applications.

Light Organic Solvent Treatment (LOSP)

The *Unitreat* plant is suitable for applying light organic solvent preservatives. Processing times are very rapid; preservative is metered into the timber, and within- and between-charge retentions are potentially very low. Since residual vacuum can be retained in the timber following preservative application, there is no need for a subsequent vacuum to recover excess preservative.

Chemical Modification

Acetylation was once a very time-consuming operation. However, the application of microwave technology to heat and condition timber to optimum moisture contents for example 6% mc, together with the application of novel catalysts provides acetylation times of minutes rather than hours (Sethy 2011). Catalysts can be metered into the wood using the *Unitreat* process prior to sequential treatment with acetic anhydride. Research undertaken by Sethy (2011) has modelled the process utilizing both microwave conditioning and the use of novel catalysts. There may also be a role for microwave removal of bi-products from the acetylation process, for example the removal of acetic acid.

Anti-Sap-Stain Chemical Impregnation

The shipping of radiata pine logs benefits from debarking and the application of antisapstain treatments to reduce stain developing during transport. Clearly, the effectiveness of such treatment depends on rapid removal of logs from forests and processing to effect protection. A number of techniques have been tested to provide such treatments. These include the use of dipping, electrostatic spraying to provide lower volume use of prophylactics, foams etc. Clearly, overriding considerations are to provide targeted treatments that eliminate contamination of the environment and OH&S issues and maximize the effective surface coverage of the commodities. The *Unitreat* plant has potential for large-scale protection of logs whereby treatments can be applied very rapidly (more rapidly than dipping) where chemical can be deposited into the surfaces of the log by vacuum impregnation and at the same time eliminate subsequent dripping. In some circumstances, surface application of microwaves or pressure steaming may have a role in sterilizing the surfaces of the debarked logs prior to applying the *Unitreat* process.

Conclusions

The "*Unitreat* process" facilitates the following advantages over conventional wood treatments:

- Very fast treatment of framing timber (faster than momentary immersion) with water-borne preservatives in which total sapwood treatment can be achieved in 1–2 min with uptakes of approximately 30L/m^3 with limited swelling and no necessity for re-drying the timber.
- Treatments completely free of any preservative dripping after treatment.
- Hot treatments with CCA to accommodate fast fixation of copper-chrome-arsenic-type preservatives, during and after treatment.

- Retention of high-quality preservative solution in the store tank with no sludge formation or preservative contamination
- Treatment with preservatives that are normally sensitive to wood moisture, e.g. organic boron compounds.
- Rapid dipping of timber with no subsequent dripping or environmental contamination or worker exposure to toxic chemicals.
- Plant design to facilitate self-contained spillage of chemicals and integration into manufacturing operations.

In combination with microwave structural modification of the wood structure, the *Unitreat* process can provide:

- Conveyor belt, "on-line" and automated treatment of round-wood, sleepers and structural timber and joinery, with light organic solvent, oil-based, water-based preservatives and resins.
- Chemical modification of timber, for example acetylation, in which timber can be conveyor belt conditioned with microwaves to optimum moisture contents for treatment (for example 6% moisture content), apply *Unitreat* schedules to provide sequential treatments of catalysts and then chemical modification agents and then accelerated reaction conditions using microwave heating and potential waste product removal (for example acetic acid) using microwave technology, the whole process taking minutes rather than hours.
- Sterilization of debarked round-wood prior to *Unitreat* application of anti-sap-stain chemicals.
- Improve the permeability of refractory wood species or heartwood that can then be treated using the *Unitreat* process.

The benefits of the *Unitreat* process arise primarily where there is a permeable sapwood treatment requirement. In some instances where higher hazard class treatments require preservative penetration of refractory heartwood, similar standards of preservative distribution can be obtained using the *Unitreat* as achieved using traditional treatments. Experimentation is needed to fine-tune schedules to meet heartwood penetration requirements.

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Natural Resistance of Imported Timbers Against Termites and Fungi in Indian Condition—A Comparison

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Abstract Timber continues to be the most vividly used structural commodity even during the twenty-first century despite the advent of many modern constructional materials. Although India is blessed with 4000 woody plant species, the country is timber deficient. In order to bridge the gap between supplies and demand, timber from different countries is being imported. But the performance of such timbers under various service conditions in different Indian environments is not known. Since information on these lines is desirable to put any timber to its best end-use, a study was performed to understand resistance of these timbers against termites and fungi in Indian condition. A total of twenty timbers that originated from different countries together with Indian grown *Hevea brasiliensis* were subjected to field trails against termites at six agro-eco-zones of India as per Indian standards (BIS—4833: 1993 and BIS—401: 1982) and laboratory tests against fungi as per BIS—4873: 2008. The trial followed by data analysis showed that performance of many of the tropical imported timbers is reasonably good, whereas performance of temperate timbers was rather poor. Results of the study were compared to see whether there is any deference in the performance of same timber species in field against termites and in laboratory against fungi. The analysis revealed that there is no difference in the resistance class of imported woods against fungi and termites and findings are discussed in this communication.

Keywords Imported timber · Resistance · Bio-deteriorating agents

Introduction

India is a timber-deficient country, despite being bestowed with over 4000 woody species, and the gap between supply and demand is burgeoning (Shashidhar and Agarwal 2006). The demand for the industrial wood has been progressively increasing

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during the last half century. 4741 million m^3 of growing stock in the Indian forest with an annual increment of 87.62 million m^3 was unable to meet the demand, and the lower productivity of Indian forest (0.7 m^3/ha/year) as against the global productivity of 2.1 m^3/ha/year (Mahapatra et al. 2011) is also posing problem. Considering the scenario import policy of wood and wood product in India is liberalized with a view to support local need and facilitates conservation of forest and environment. In India, many timber species are being imported from different countries such as Malaysia, Indonesia, South Africa, Australia, Nigeria. Among them, certain species of wood are known to comprise inherent quality to resist the termites and other insects. But before putting timber into use, it is necessary to know the behaviour of wood species under different environmental conditions as durability varies according to the geographic location (Harris 1961). The degree of deterioration of wood is dependent on conditions such as soil, rainfall, altitude, temperature and environmental conditions under which the timber is put to use (Rao 1982). Further, it is a well-known fact that heartwood of certain species exhibits considerable resistance to bio-deteriorating agents. Therefore, knowledge of natural resistance of wood is of great importance for making rational and judicious use of timbers. The knowledge on the durability of timber shall enable the selection of right timber for right purpose (Sen-Sarma et al. 1975). With this view in mind, a study was undertaken to evaluate the natural durability of imported wood under Indian conditions against major bio-deteriorating agents, termites and fungi. The results of such studies were compared to appraise the difference in performance of same wood species against two deteriorating agents.

Materials and Methods

Based on the information obtained from plant quarantine offices of India, timber depots and Indian timber market, twenty commercially available imported timber species which are in demand were selected for the study. The timber merchants were contacted and two-cubic-feet heartwood of each imported timber species was procured and wood stakes for the study were prepared. For the experimental purpose, selected logs consisting of pure heartwood, avoiding sapwood and pith, were prepared. Care was taken to ensure that the stakes selected came from different logs, to avoid pseudo-replications, and they were free from large knots, stains, moulds, decay or other defects.

All 20 timber species imported from different countries were evaluated along with Indian rubber wood for comparison. Resistance rating of wood against termites was done in field as per the Lebow et al. (2006). For this purpose, wood stakes of 30.0 $\times$ 3.8 $\times$ 3.8 cm were prepared. The stakes were dried at 80 °C to attain constant weight, numbered and then implanted in the soil at termite test yard of the Institute at Nallal following a completely randomized design. Ten replicates were made for each wood species and all these were half-buried in rows and a distance of one foot was maintained between the stakes. The surrounding soil was firmly

pressed around the stakes to ensure good contact with the soil. Observations were made at an interval of three months for two years.

Laboratory assessment against fungi was performed as per Indian Standard (BIS 4873, 2008) and Bakshi (1967). To perform the experiment test, blocks of size 1.9 × 1.9 × 1.9 cm with long axis parallel to the grain of the wood are prepared from imported wood stakes. These test blocks (six matched blocks) were numbered and kept in the oven at 102 ± 3 °C for 48 h and weighed. The weighed blocks were then placed in a desiccator. Fungal cultures were established with a nutrient medium consisting of agar (1.3%) and malt (2%) and were inoculated with the test white rot fungi (*Tyromyces versicolor* and *Polyporus sanguineus*) and brown rot fungi (*Polyporus meliae* and *Oligoporus placenta*), respectively. After getting pure fungal growth, air-dried weighed test blocks were inoculated in aseptic condition with respective fungal cultures and then incubated for the next 16 weeks. Three replications (two blocks in one bottle) were made for each wood species. After incubation test blocks were removed, fungal mat removed and oven-dried before obtaining final weight. Fungal resistance was classified based on their weight loss into four groups viz., highly resistant/durable (weight loss <10%), resistant (weight loss 11–24%), moderately resistant (weight loss 25–44%) and non-resistant class (weight loss 45% above). Durability classes were further assigned as durability class I (highly resistant/durable), durability class II (resistant), durability class III (moderately resistant) and perishable or not at all durable (non-resistant class). Wood resistance classes based on the field testing against termites and laboratory testing against fungi were compared to see whether there is a difference in their performances against termites and fungi.

$$\text{Weight loss } (\%) = \frac{(\text{Initial weight} - \text{Final weight})}{\text{Initial weight}} \times 100$$

Results and Discussion

Resistance is a critical determinant of life span of tree species. Many heartwood species were known for their resistance against degradation (Harris 1961). Our results on resistance of imported wood are similar to the results obtained by many other researchers (Oshima 1919; Bavendumm 1955; Walcott 1957; Sandermann and Dietrichs 1957; Behr et al. 1972; Sen Sarma et al. 1975; Badawi et al. 1985; Mohd Dahlan and Tam 1985; Ling 1996; Grace et al. 1998; Wong et al. 2005; Nzokou et al. 2005; Rapp and Augusta 2006; Evans et al. 2008). Based on results, we can tell that there is a similarity in the resistance class of most of the imported timbers against termites and fungi in Indian condition (Table 1). The tested timbers were categorized into three groups: (1) susceptible—*Acer pseudoplatanus* from Belgium and France, *Fagus grandifolia* from France, *Fagus sylvatica* from Belgium and France, *Fraxinus angustifolia* from France, *Fraxinus excelsior* from

Table 1 Comparison of resistance of imported timbers against biodegrading agents

Sl. No.	Trade name	Origin	Scientific name	Resistance class	
				Termite	Fungi
1	Maple	Belgium	*Acer pseudoplatanus* L.	III	III
2	Maple	France	*Acer pseudoplatanus* L.	III	IV
3	Kapoor	Malaysia	*Dryobalanops aromatica* C.F. Gaertin	I	I
4	White Beech	France	*Fagus grandifolia* Ehrh.	III	III/IV
5	Beech	Belgium	*Fagus sylvatica* L.	III	IV
6	S Beech	France	*Fagus sylvatica* L.	III	III
7	Ash	France	*Fraxinus angustifolia* Vahl.	III	III
8	Ash	Belgium	*Fraxinus excelsior* Quctnon Flynn	III	IV
9	Rubber	India	*Hevea brasiliensis* Muell. Arg.	III	III
10	Narra	Africa	*Pterocarpus soyauxii* Taub.	I	I
11	Padouk	Cameroon	*Pterocarpus soyauxii* Taub.	I	I
12	European Oak	France	*Quercus robur* L.	II	I
13	Balau	Indonesia	*Shorea laevis* Ridl.	I	I
14	Maranti	Malaysia	*Shorea marcoptera* Dyer.	I	I
15	Sal	Malaysia	*Shorea robusta* Gaerth. F.	I	I
16	Myanmar Teak	Myanmar	*Tectona grandis* L.f.	I	I
17	African Teak	Ivory coast	*Tectona grandis* L.f.	I	I
18	African Teak	Ghana	*Tectona grandis* L.f.	I	I
19	African Teak	Tanzania	*Tectona grandis* L.f.	I	I
20	Australian Teak	Indonesia	*Tectona grandis* L.f.	I	I
21	Pyinkado	Myanmar	*Xylia dolabriformis* Benth.	I	I

Belgium; (2) highly resistant—*Dryobalanops aromatica* from Malaysia, *Pterocarpus soyauxii* from S. Africa and Cameroon, *Shorea laevis* and *Shorea marcoptera* from Malaysia, *Shorea robusta* from Indonesia, *Tectona grandis* from Myanmar, Ivory coast, Ghana, Tanzania and Indonesia and *Xylia dolabriformis* from Myanmar; (3) moderately resistant—*Quercus robur* from France. The study also revealed that timbers from temperate countries are more susceptible to bio-deteriorating agents than tropical timbers (Fig. 1).

Studies have revealed that same timber species sometimes fall into different durability classes against different kinds of biological agents, and in such cases, study of factors responsible for these differential performances becomes most interesting. Prior analyses have showed that such inconsistent performance may be due to a combination of endogenous and exogenous factors (Meyer 2012). Our comparison indicated a similarity in the resistance of imported timbers against termites (field trial) and fungi (laboratory trail) and revealed that in this particular set of timbers rather than the exogenous factors, endogenous factors are playing major role. Endogenous factors such as wood density, chemical composition and concentration of extractives were found high in the case of highly durable timbers

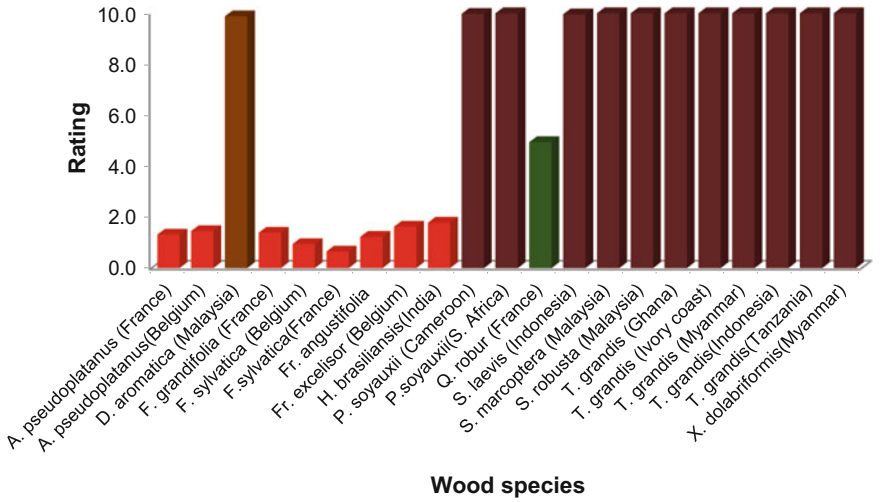

Fig. 1 Cumulative rating of wood species based on resistance against termite attack

which are collectively known to confer higher resistance against any kind of wood-degrading agents (Da Costa and Osborne 1967; Behr et al. 1972; Bultman and Southwell 1976; Abreu and Silva 2000).

Many earlier studies have shown the influence of chemical components of wood on its resistance property (Moore 1979). Chemical constituents such as cellulose, lignin and the total phenolic content of wood showed the influence on the rate of degradation, and it was found that the higher the cellulose content, the higher the susceptibility to termite attack, whereas higher the lignin (Wainhouse et al. 1990; Eaton and Hale 1993; Francis and Schwarze 2007) and extractives content (Oshima 1919; Bavendamm 1955; Walcott 1957; Sandermann and Dietrichs 1957; Sen-sarma et al. 1975; Gupta and Sen-sarma 1978), the higher the resistance of wood species. Extractives confer resistance against both termites (Whittakar and Fenny 1971; Bultman and Southwell 1976) and fungi (Hawley et al. 1924; Carter et al. 1978; Hillis 1987; Chang et al. 1999; Santana et al. 2010). Several studies have clearly demonstrated that removal of extractives makes durable wood lose its natural resistance and render it more susceptible to deterioration and degradation (Taylor et al. 2006; Oliveira et al. 2010).

Conclusion

There is a similarity in the resistance class of most of the imported timbers against termites and fungi in Indian condition. Accordingly, the susceptible timbers are *A. pseudoplatanus, F. grandifolia, F. sylvatica, F. angustifolia, F. excelsior;* highly

resistant timbers are *D. aromatica, P. soyauxii, S. laevis and S. marcoptera, S. robusta T. grandis* and *X. dolabriformis*; and *Q. robur* is a moderately resistant timber. The study also revealed that timbers from temperate countries are more susceptible to bio-deteriorating agents than tropical timbers. There is an urgent need to provide better guidance to users on proper end uses of imported timber as Indian market is widely opened to these timbers. Guidance provided will be of great help towards the judicious and proper use of timber in the cost-effective manner. Better understanding of the natural durability of imported woods under Indian environmental condition is much needed, and hence a long-time study has been underway to know the durability class of imported timbers as per bureau of Indian standards.

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Enhancing Photostability of Wood Coatings Using Titanium Dioxide Nanoparticles

Kavyashree Srinivas and Krishna K. Pandey

Abstract Protection from UV radiation is important for the performance of wood substrate and the coating that protects it. Ultraviolet light causes degradation of both wood substrate and coating polymers. Additives (UV stabilizers) are generally added into the coating to reduce the adverse influence of UV radiations under exterior applications. The performance of coating depends upon the amount and type of UV absorbers used in the coating. In this study, the effectiveness of titanium dioxide (TiO_2) nanoparticles for protection of wood surfaces and polyurethane (PU) against UV light was studied. TiO_2 nanoparticles were chemically functionalized with an organic alkoxy silane (3-GlycidoxyPropylTriMethoxySilane), and modified nanoparticles were dispersed in PU coating. Rubberwood surfaces were coated with nanoparticles dispersed PU coatings and were exposed to UV light in an accelerated weathering tester for 500 h. Colour changes occurred due to UV light exposure were analysed at regular intervals of time. Dispersion of TiO_2 nanoparticles in PU coatings restricted the photo-yellowing of wood surfaces.

Keywords Accelerated weathering · Nanoparticles · Photo-degradation · Rubberwood · Titanium dioxide · UV resistance

Introduction

Wood is one of the versatile materials for several applications including building, construction and furniture. It has favourable mass/strength ratio, low thermal conductance, easy to process and great aesthetic value. However, there are some inherent drawbacks in wood which hinder its use in certain applications. These drawbacks include dimensional instability with change in moisture content, low resistance of some of the species against fungi and insect attack and photo-degradation of wood polymers when exposed to outdoor. Photo-degradation

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of wood polymers is one of the major problems when used in outdoor conditions. On exposure to light, wood changes its colour and turns yellow to brown (Feist and Hon 1984; Hon 2001; Williams 2005; Evans 2013). Wood polymers absorb ultraviolet light present in solar radiation and undergo photochemical degradation. Among wood polymers, lignin is most susceptible to photo-degradation. Degradation of wood polymers by UV light results in discoloration and eventually causing fractures and cracking. Polymer coatings exposed to UV light also degrade and lose mechanical strength. Therefore, photo-protection of wood and wood coatings is necessary for outdoor applications.

One of the most widely used methods of UV protection of materials is the dispersion of transparent UV-absorbing molecules (UVA) into coating. UVA absorb the UV radiation and dissipate the absorbed energy harmlessly before reaching the substrate. Inorganic materials, such as metal oxide films or particles, or organic molecules, such as benzophenone derivatives, can be effectively used to absorb or scatter light. Titania (TiO_2), which is commonly used in sunscreens, is an example of an inorganic UV-protector. The effectiveness of nanometal oxides to improve the performance of exterior coatings and wood underneath coatings has been studied in the recent past (Allen et al. 2002; Aloui et al. 2007; Cristea et al. 2010; Saha et al. 2011; Forsthuber et al. 2013).

In the present work, the efficacy of TiO_2 nanoparticles for enhancing the photostability of rubberwood surfaces has been studied. Nanoparticles of TiO_2 were chemically functionalized with an organic alkoxy silane (3-Glycidoxy-PropylTriMethoxySilane). Modified nanoparticles were dispersed in isopropanol and polyurethane coating and were spray coated on to surfaces of rubberwood. Coated wood surfaces were exposed to UVA-340 fluorescent light source for 500 h. Colour changes due to UV light exposure were regularly monitored using spectrocolorimeter.

Materials and Methods

Wood Specimens and Other Materials

Rubberwood (*Hevea brasiliensis*) logs were purchased from a local sawmill, seasoned and converted to specimens of size 150 × 75 × 5 mm (length × width × thickness). Titanium (IV) oxide nanoparticles (particle size ~21 nm) were purchased from Sigma-Aldrich. 3-GlycidoxyPropylTriMethoxySilane (GPTMS) was purchased from Gelest Inc. and two pack 2 K polyurethane coating (without any additives) was procured from Asian Paints, Mumbai. All other chemicals used in the study were of AR grade.

Surface Modification of Nanoparticles and Dispersion of Nanoparticles in Wood Coatings

In order to obtain homogenous distribution of nanoparticles and avoid agglomeration, the nanoparticles (NP) were modified with GPTMS. Amount of GPTMS used for modification was based on molar ratio of nanoparticles and silane (NP:GPTMS —1:0.05). Initially, nanoparticles were dispersed in isopropanol solution using ultrasonication for 20 min, and then, silane solution was added slowly dropwise and sonicated for 30 min in an ice bath. Silane-modified nanoparticles were separated using ultracentrifugation and washed with ethanol to remove unreacted silanes. Modified nanoparticles were oven-dried.

Different concentrations (0.5, 1.0, and 2.0% w/v) of silane-modified TiO_2 nanoparticles were added to isopropanol or polyurethane coating and subjected to homogenization for 20 min at 10,000 rpm (IKA T25 digital ULTRA-TURRAX). Wood surfaces were coated with two coats of homogenized solution of nanoparticles using sprayer and dried at room temperature.

UV–Visible Absorption Spectroscopy and Scanning Electron Microscopy

UV–Vis spectra of surface modified nanoparticles in powder form were measured using UV–visible spectrophotometer (Ocean Optics HR 4000 UV–Vis spectrophotometer, UV–Vis-NIR light source, DT-MINI-2-GS, Jaz detector). The distribution of nanoparticles in polyurethane was examined using high-resolution scanning electron microscope (Gemini Ultra 55, with ESB detector at 5.0 kV) at Centre for Nano Science and Engineering, Indian Institute of Science, Bengaluru.

Evaluation of Photostability of Wood Surfaces Coated with Nanoparticle

Photostability of TiO_2 coated wood was assessed by exposing coated wood specimens to UVA-340 fluorescent light source in a QUV accelerated weathering tester (Q-Lab, USA) at an irradiance of 0.68 W/m^2 and chamber temperature of 60 °C. The samples were removed from the weathering tester at an interval of 50 h and were analysed for colour and chemical changes.

Changes in colour of wood surface due to light irradiation were measured using CIELAB system in a Hunterlab-Labscan XE model colorimeter. Values of lightness (L^*), redness (a^*) and yellowness (b^*) were measured on each samples before and after UV irradiation, and change in colour co-ordinates due to exposure ΔL^*, Δa^*

and Δb^* values was calculated. The total colour change, ΔE^*, as a function of the exposure time was calculated using following Eq. 1,

$$\Delta E^* = \left[(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\right]^{1/2} \quad (1)$$

Measurements were taken from three replicates for each treatment, and colour was measured from six different locations in a sample and average values were calculated.

Results and Discussion

UV–Visible Spectra

TiO_2 nanoparticles were modified using GPTMS. Modified nanoparticles were characterized using UV–visible absorption spectroscopy. UV–visible absorption spectrum of silane-modified nanoparticles is shown in Fig. 1. Spectrum showed a broad and strong absorption in UV region.

Scanning Electron Microscopy

In order to know the distribution of nanoparticles in PU coatings, thin films were prepared and scanning electron micrographs (SEM) were recorded. Figure 2 shows the SEM image of unmodified nanoparticles dispersed in PU coating. Severe

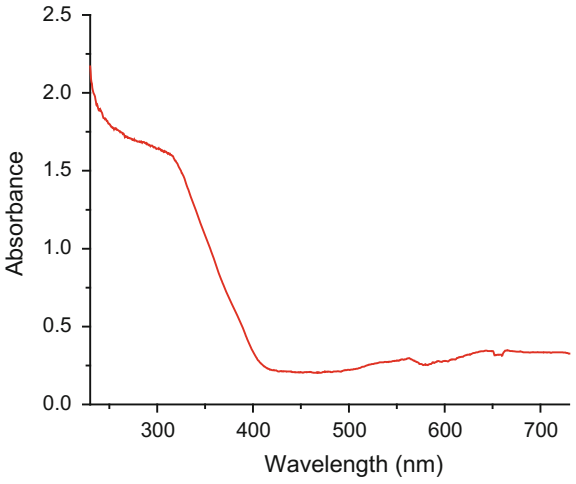


Fig. 1 UV–visible absorption spectrum of GPTMS modified TiO_2 nanoparticles

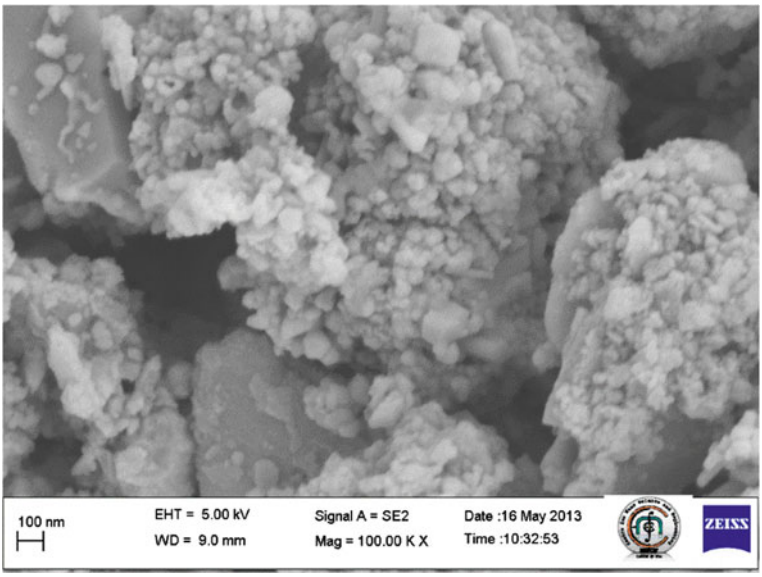


Fig. 2 SEM images of unmodified TiO_2 nanoparticle dispersed in PU coating

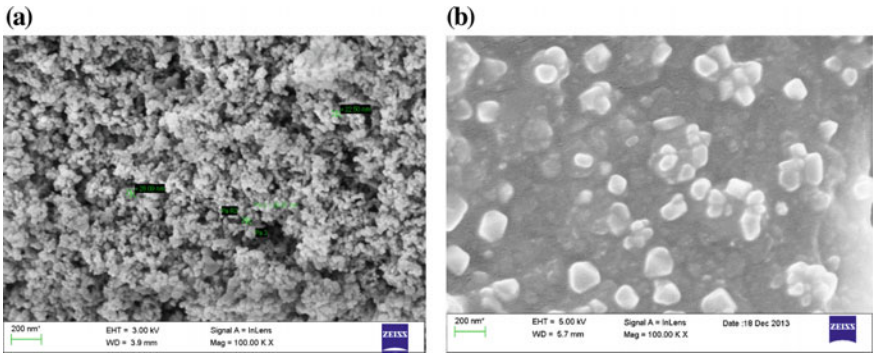


Fig. 3 SEM images of **a** modified nanoparticles powder and **b** nanoparticles modified with GPTMS dispersed in PU coatings

agglomeration was observed in unmodified nanoparticles dispersed in PU coating. SEM image of TiO_2 particles modified with GPTMS is shown in Fig. 3a. SEM images of modified nanoparticles dispersed in PU coatings are shown in Fig. 3b. Effectiveness of surface modification in de-agglomeration and uniform dispersion of nanoparticles in coating material can be clearly seen. A uniform dispersion of nanoparticles was obtained by surface modification.

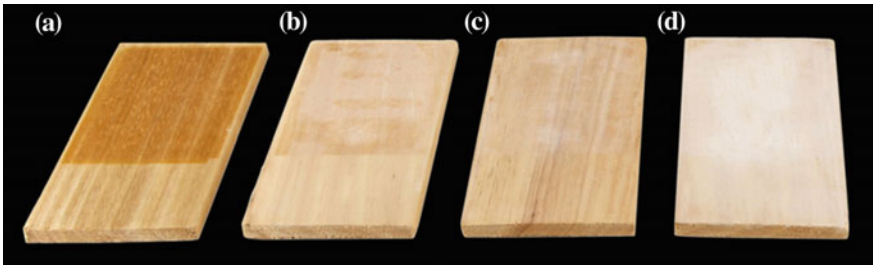


Fig. 4 Colour changes after 500 h of UV exposure of **a** uncoated and **b** coated with 0.5% TiO_2, **c** coated with 1.0% TiO_2 and **d** coated with 2% TiO_2

Photostability of Wood Coated with Nanoparticles

A set of rubberwood specimens was coated with different concentrations of modified nanoparticles dispersed in isopropanol. Coated wood and uncoated wood were exposed to UV light in accelerated weathering tester. The colour changes in uncoated and wood surfaces coated with nanoparticles after 500-h exposure are shown in Fig. 4. Uncoated wood exposed to UV light showed rapid colour changes (photo-yellowing) within short exposure durations. The colour darkening was suppressed by coating with TiO_2. Wood coated with nanoparticle loading of 2% was highly effective in arresting light induced colour changes. The colour changes were further quantified by measuring CIE colour parameters using spectrocolorimeter.

Colour of wood becomes light after coating with nanoparticles, which is indicated by increase in L^* values and decrease in a^* and b^* parameters. Effect of UV light irradiation on L^* and b^* values of wood coated with different concentrations of TiO_2 is shown in Fig. 5. In uncoated wood, L^* values decreased with exposure time, the rate of change was high during initial exposure time. Decrease in L^* value due to UV irradiation is indicative of darkening of wood as a result of photo-degradation of wood polymers. The decrease in L^* value decreased in wood coated with nanoparticles. The changes in L^* values were very small even after 500 h of UV exposure in wood coated with 2% of TiO_2. This indicates effectiveness of nanoparticles in restricting light induced darkening of wood surfaces particularly at higher concentrations.

UV light irradiation on uncoated wood resulted in increase in b^* values (Fig. 5). The corresponding changes in wood coated with nanoparticles of TiO_2 were less. Contrary to uncoated wood, the values of b^* decreased in wood coated with nanomaterial, particularly at higher concentration of nanoparticles. Changes in Δb^* values were very small and approached to negative values in case of wood coated with 2% TiO_2. This indicates effectiveness of TiO_2 coating in arresting photo-yellowing of wood surfaces at 2% nanoparticle concentrations.

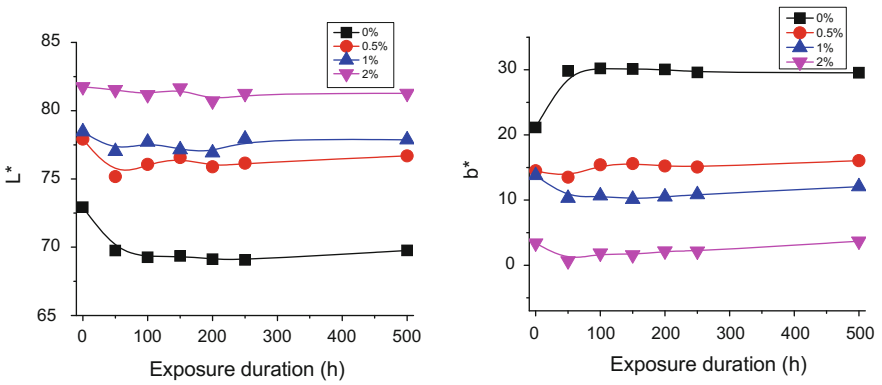


Fig. 5 Effect of UV irradiation on L^* and b^* values of wood coated with different concentrations of TiO_2 nanoparticle

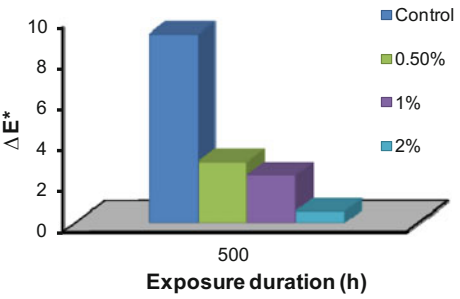


Fig. 6 Effect of UV irradiation on ΔE^* values of unmodified and wood coated with different concentrations of TiO_2 nanoparticle after 500 h irradiation

The overall colour changes (ΔE^*) in wood coated with TiO_2 after 500 h exposure are presented in Fig. 6. ΔE^* values were maximum in uncoated wood. Wood coated with 2% nanoparticle showed very less change in ΔE^* values.

Photostability of Wood Coated with Nanoparticles Dispersed in Polyurethane

Wood samples were coated with different concentration of nanoparticles (0.5–2%) dispersed in PU coating. Coated specimens were subjected to UV light irradiation, and performance of nanoparticle dispersed PU coating on wood surfaces was compared with PU coating without nanoparticles.

Wood coated with PU alone (without nanoparticle) exhibited severe colour changes, which increased with irradiation time. This indicates that PU coating without any UV stabilizer gets degraded rapidly upon UV light irradiation. Incorporation of nanoparticle in PU restricted colour changes. Figure 7 illustrates

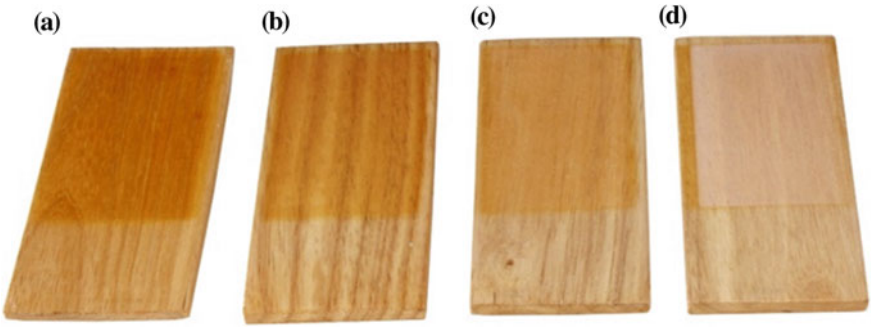
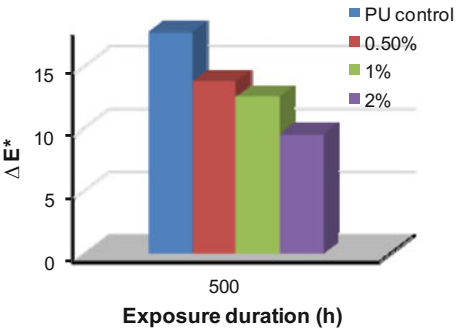


Fig. 7 Colour changes after 500 h of UV exposure in rubberwood coated with TiO_2 dispersed PU: TiO_2 concentrations are: **a** 0%, **b** 0.5%, **c** 1% and **d** 2%

Fig. 8 Total colour change (ΔE^*) of wood coated with different concentration of TiO_2 nanoparticles exposed to UV light for 500 h



the colour changes in wood coated with different concentrations of nanoparticles embedded in PU coatings after 500 h of UV irradiation. Photo-yellowing of wood surfaces was suppressed with incorporation of TiO_2 in the coating. The overall colour change ΔE^* of wood coated with TiO_2 is shown in Fig. 8. The ΔE^* value after 500 h of UV exposure in PU coating without and with 2% concentrations of TiO_2 was 17.7 and 9.5, respectively.

Conclusions

The efficacy of titanium dioxide nanoparticles for photo-stabilization of rubberwood surfaces was studied. TiO_2 nanoparticles were functionalized with an organic alkoxy silane (3-GlycidoxyPropylTriMethoxySilane) to achieve uniform dispersion of nanometal oxide. Wood surfaces were coated with nanoparticles dispersed in isopropanol and polyurethane coating and exposed to UVA-340 light source in an accelerated weathering tester up to 500 h. Dispersion of nanoparticles in coatings restricted the colour changes due to photo-degradation of wood polymers.

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Screening of Oils of *Pongamia pinnata* Linn., *Jatropha curcas* Linn. and *Simarouba glauca* D.C. for Developing Eco-Friendly Wood Preservatives

D. Venmalar

Abstract Efficacy of pure and copper-incorporated oils of *Pongamia pinnata* Linn. *Jatropha curcas* Linn. and *Simarouba glauca* D.C. as eco-friendly wood preservatives against fungi and termites was studied in the laboratory and field conditions, respectively. *Hevea brasiliensis* (rubber wood), a highly perishable wood, was treated with these oils by pressure methods. Pure oils show 'Moderately resistant class III', and copperised oils show 'Resistant class II' against fungus. Incorporation of a very low concentration of copper into these oils resulted in a good protection against fungus and termites. *P. pinnata* oil was found to be highly resistant to termites as the destruction was very less compared to *J. curcas* and *S. glauca*. The results show that biocidal properties of oils can be significantly increased by incorporating toxic copper ions. Results indicate the potential of these oils as a viable less toxic eco-friendly wood preservative and an alternative to conventional wood preservatives.

Keywords Eco-friendly wood preservative · *Pongamia pinnata* Linn. *Jatropha curcas* Linn. · *Simarouba glauca* D.C.

Introduction

Wood has remained one of the most renewable natural resources available to mankind for broad variety of purposes. The major role of wood is providing material for our buildings and many others products. The ever-increasing gap between demand and supply of wood is a matter of serious concern. Wood is to be protected with environmental-friendly, thoroughly biodegradable substances, so that the treated wood is fit for the purpose, has a safe life cycle and has eventual

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disposal. Wood being lignocellulosic material is liable to degradation due to termite and microbial agencies causing significant loses. The most effective way to control/check the deteriorating organism is through wood preservation. Wood preservation enhances the service life of timber several times.

From time immemorial, attempts were made by many workers to impart durability by treating wood with natural and synthetic chemicals (Purushotham 1970). The conventional wood preservatives copper chrome arsenic (CCA), copper chrome boric (CCB), although found to be very effective against wood-destroying organisms, are said to cause environmental pollution, and a few of them are hazardous to animals and human beings (Fisher 1968; Thompson 1971; Onuorah 2000). Over the past few decades, there has been substantial global awareness to develop eco-friendly wood preservatives and those that do not cause any ill effect on the health of mammals (Onuorah 2000). There is a continuous search for methods of bio-control for wood preservation. The current trend is therefore to seek alternatives to synthetic chemicals with attention focused on the use of natural products of plant origin, which are not only effective, but are also biodegradable.

Green plants acts as a reservoir for inexhaustible source of innocuous fungicide/pesticides, such as cashew nut shell liquid (CNSL), neem oil, Pongamia oil which are mammalian non-toxic and easily biodegradable than synthetic chemicals. These compounds as such are not effective wood preservatives as they have very low biocidal properties, but when they are incorporated with small concentrations of inorganic metal ions such as copper, zinc and titanium, they may serve as good wood preservatives. Copper-incorporated CNSL and neem oil have been found to be very effective wood preservatives and give significant protection against termite and fungi (Venmalar and Nagaveni 2005). Some of the non-edible seeds such as *Pongamia pinnata*, *Jatropha curcas* and *Simarouba glauca* D.C. oils have insecticidal and fungicidal properties due to the presence of phyto-chemicals such as sterols, alkaloids, phenolic groups and tannins (Anon 1948). Though these oils have insecticidal and fungicidal properties, they were not fully tapped and are underutilised as wood preservatives.

In this work, potential of these oils for the preparation of eco-friendly wood preservatives with or without incorporating copper has been examined. The efficacy of these wood preservatives against decay fungi in the laboratory and insects in the field conditions has been evaluated.

Materials and Methods

Wood Specimens

Two different size test specimens were prepared from defect-free air-dried rubber wood (*Hevea brasiliensis*). Test specimens of dimension 305 × 38.1 × 38.1 mm^3 were prepared for field test for evaluation of termite resistance. Specimens of

20 mm^3 were prepared for evaluation of fungal resistance of preservative-treated wood.

Preparation of Preservatives

Fresh and cold pressed oils of *Pongamia pinnata* Linn., *Jatropha curcas* Linn. were procured from M/S Triagni Private Ltd, Bengaluru, and *Simarouba glauca* D.C. oil was procured from Shreemathi Renuka Foundation, Gadag Taluk, Karnataka. The oils were diluted to 50% W/W concentration in toluene. Incorporation of copper metals into the oils was carried out as per the published procedure (Sharma et al. 1964; Venmalar and Nagaveni 2005). The copper content in the prepared oil was estimated as per IS 2753 part I (Anon 1991). The absorption of Cu after 32 h refluxing was 2.64, 1.90 and 1.73% for Pongamia, Jatropha and Simarouba oils, respectively (Table 1). The pure oils and the copperised oils were used as wood preservatives, and their efficacy was studied against insects and decay fungi.

Treatment for Test Specimens

Wood specimens were treated with pure oil and copperised oil preservatives by adopting full cell pressure treatment process in a vertical vacuum pressure treatment plant. A set of 10 matched replicates were used in each case. The treatment schedule adopted was as follows: the initial vacuum of 50 cm Hg was applied for 15 min followed by an air pressure of 3 kg/cm^2 applied through a compressor for 60 min and a final vacuum of 50 cm Hg for 5 min (15′/3 kg cm^2/60′/5′) applied to remove the excess solution from the surface of the specimens. After treatment, the specimens were weighed individually and kept for air-drying in shade for facilitating proper fixation of preservative within the specimens. The amount of preservative absorbed by each of the test specimen was estimated using following Eq. 1 (Purushotham 1970)

$$\text{Absorption} = (G * C/V) * 10\,\text{kg/m}^3 \quad (1)$$

Table 1 Percentage of copper sulphate incorporated in the oils at four different reaction periods

Time in hours	8	16	32	64
Pongamia pinnata	4.9 (1.70)	7.07 (2.46)	7.6 (2.64)	11.2 (3.89)
Jatropha curcas	2.53 (0.88)	4.43 (1.54)	5.39 (1.90)	11.8 (4.10)
Simarouba glauca	–	3.02 (1.05)	5.01 (1.73)	5.12 (1.74)

Value in parentheses is % of copper ion

where $G = (W_2 - W_1)$ is amount in grams of preservative solution absorbed by the specimen, W_1 is the initial weight of the wood specimen before treatment, W_2 is the final weight of the specimen after treatment, C is the concentration of the preservative in 100 g of solution used for treating wood specimen and V is the volume of wood specimen (cm^3).

Evaluation of Termite Resistance of Preservative-Treated Wood

Untreated and treated specimens were installed following completely randomised design, in the IWST test yard at Nallal, 40 km from Bengaluru, as per Indian Standard (No. 4833-1968) (Fig. 1). Termite activity and percentage of damage to the test specimens were recorded at the intervals of every three months by visual observation. Specimens were removed from underground and cleaned off mud, and damage was visually ascertained by visual rating. Rating for no attack specimen, i.e. sound wood, was 0 whereas completely destroyed by white ants was rated 10. Specimens were reinstalled in their respective positions after each inspection. The knife test or sound test was carried out when necessary to determine the extent of decay or destruction due to fungal or termite attack. Observation was made till the specimen was destroyed completely. Percentage of deterioration and increase in durability were calculated with reference to the control specimens.

Fig. 1 Exposure of preservative specimens in the test yard

Fungal Resistance of Treated Wood

Wood specimens of size 20 mm × 20 mm × 20 mm, ten matched replicates for each case, were treated with preservatives by pressure method. The absorption of the specimen was calculated individually. The fungal resistance of the treated blocks were tested as per Indian Standard No. 4873 (Anon 1968a, b, c). The treated specimens were exposed to two white rot *Pycnoporus sanguineus* (PS) and *Trametes versicolor* (TV) and two brown rot *Oligoporus placenta* (OP) and *Polyporus meliae* (PM) fungi in the laboratory along with the untreated controls. After 16 weeks, test blocks were withdrawn from the cultured bottles. These blocks were carefully brushed off the fungal mats, dried in warm air, again conditioned and weighed to obtain constant weight (final weight). Percentage of weight loss was calculated for each block after fungal exposure using Eq. 2.

$$\text{Weight Loss } (\%) = [100(W_3 - W_4)/W_3] \tag{2}$$

where W_3 is weight of the wood specimen before exposing to fungus and W_4 is weight of the wood specimen after exposing to fungus.

Results and Discussion

Figure 2 shows the mean absorption values of all the three preservatives in pressure-treated rubber wood. The absorption of copperised oils was higher than pure oils.

The decay resistance of rubber wood specimens treated with three pure oils preservatives and copper-incorporated preservatives against fungi is shown in Fig. 3. The mean weight loss in control wood was 46.5 and 58.6 for white rot and

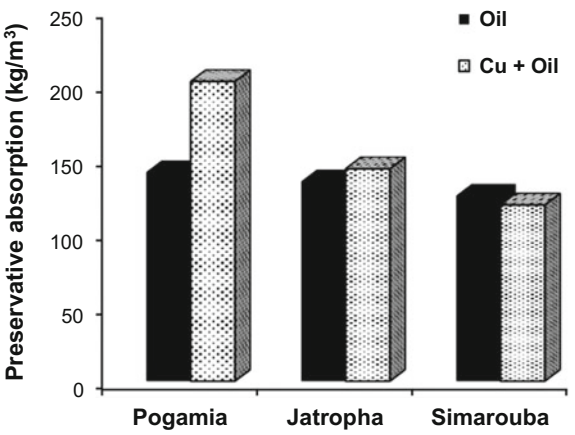


Fig. 2 Mean absorption of preservatives

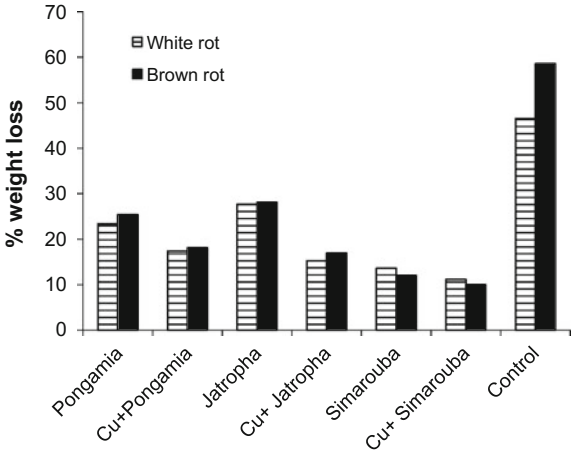


Fig. 3 Mean weight loss of rubber wood exposed to fungi

brown rot exposed wood, respectively. The effectiveness of oil based preservative against fungal degradation can be clearly seen. Fungal degradation was significantly inhibited in oil-treated wood. The weight loss in Pongamia oil-treated wood was 23.3 and 25.4%, respectively, for white rot and brown rot exposed wood. Similarly, Jatropha and Simarouba oils provided good resistance against white and brown rots. Simarouba oil was most effective among three oils.

The effectiveness of oil preservatives can be significantly enhanced by incorporation of copper into oil (Fig. 3). Copper is a well-known fungicide, and hence, weight loss was lower in copper-incorporated oils as compared to pure oils. Comparing the three preservatives, both pure oils preservatives and copper-incorporated preservatives, the mean weight loss was least in Simarouba oil followed by Pongamia and Jatropha oil. Analysis of data against fungus copperised oils show 'Resistant class II' and pure oils show 'Moderately resistant class III' (Bakshi 1967). The untreated rubber wood is classified as non-resistant class.

Termite Resistance of Treated Wood

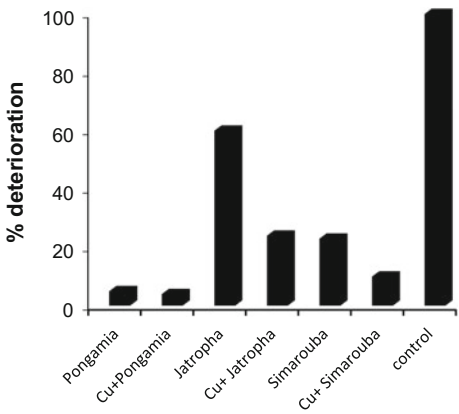
The condition of untreated and preservative-treated wood specimens after 36 months of exposure in test yard is shown in Fig. 4. Figure 5 shows the comparison of the efficacy of the rubber wood specimens treated with these three preservatives, pure and incorporated with copper after 36 months of field exposure. Up to 10% deterioration was seen in specimens with pure and copperised Pongamia, and in copperised Simarouba oil preservatives specimens. Less than 25% deterioration was observed in specimens pressure-treated with pure Simarouba oil and copperised Jatropha oil preservatives.

Based on the performance of the specimens in the test yard after 36 months of field exposure the efficiency of preservatives can be rated as copperised Pongamia is > pure Pongamia > copperised Simarouba > copperised Jatropha > pure

Untreated Treated

Fig. 4 Condition of untreated and preservative-treated specimens after 36 months of exposure in test yard

Fig. 5 Mean % of deterioration of rubber wood exposed to termites in field for 36 months



Simarouba > pure Jatropha. An average increase in life due to treatment as compared to untreated specimens was minimum 4–5 times more in Pongamia, Jatropha and Simarouba oils preservatives and 7–8 times more in copper-incorporated preservatives of these oils. Impregnation of pure oils into rubber wood helps in protection against termites to some extent, but copper-incorporated oils helped in improving the durability significantly.

Conclusions

Rubber wood without any treatment is highly susceptible to fungi and termites and deteriorates within 12 months when put into use. Treatment of rubber wood with copperised *Pongamia pinnata*, *Jatropha curcas* and *Simarouba glauca* oils offered

very good protection against decay fungi and termites. Pure oil-treated wood show 'Moderately resistant class III' and copperised oils show 'Resistant class II' against decay fungi. Incorporation of very low concentration of copper with these oils enhances decay resistance significantly. It can be concluded that treatment with pure oils increases the life of untreated specimens to 4–5 times and copperised oils increases the life 7–8 times. Impregnation of oils into rubber wood helps in protection against termites to some extent, but incorporating copper helped in improving the durability significantly higher. The results indicate that all the three oils *Pongamia pinnata*, *Jatropha curcas* and *Simarouba glauca* can be used as eco-friendly wood preservatives. Compared to all the three oils, the *Pongamia pinnata* oil, both pure and copper-incorporated, gave complete protection against the wood-deteriorating agents.

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Fungal Inhibition in Wood Treated with *Lantana camara* L. Extract

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Abstract Wood is a sustainable, strong, economical and renewable organic material. However, under certain conditions of exposure it deteriorates rapidly due to a combination of biological, chemical and physical processes. Wood decomposition by fungal micro-organisms has adverse effects and is a major source of losses to wood. Therefore, the present investigation has been carried out to ensure protection from the fungal decay and to enhance the durability of wood. Although treatment with synthetic chemical preservatives is an effective method to induce biological resistance in wood but it causes environmental pollution. Thus the use of plant based environmental-friendly formulations as biopreservatives is emphasized. *Lantana camara* L. belonging to family Verbenaceae, known worldwide as an obnoxious weed, possesses antifungal properties. The wood samples of three species viz., *Pinus roxburghii* Sargent, *Celtis australis* L., and *Bombax ceiba* L. were dip treated for 72 h with methanol extract of *Lantana camara* L. leaves at 0.25, 0.5, 1, 1.5 and 2% (w/v) concentrations. These treated samples were assessed for fungal inhibition. The results revealed significant inhibition in the fungal growth in wood treated with 1.5 and 2% concentrations of *Lantana camara* L. extract.

Keywords Environmental friendly preservative · Fungal inhibition · *Lantana camara* L. · Wood

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Introduction

Wood is a three-dimensional biopolymer composite composed of an interconnected network of cellulose, hemicellulose and lignin with minor amounts of extractives and inorganics. The main problems associated with wood under outdoor use are its dimensional instability due to moisture sorption and its decay by micro-organisms. Wood being ligno-cellulolytic material is liable to degradation due to microbial agencies and termites causing significant losses. Wood is degraded biologically because organisms recognize the polysaccharide polymers in the cell wall (e.g., cellulose and hemicelluloses) and have very specific enzyme systems capable of hydrolyzing these polymers into digestible units. Chemical modification of cell wall polymers is one of the effective methods to induce biological resistance in wood (Rowell 2005). The conventional wood preservatives such as chromated copper arsenate (CCA), creosote consisting of various polycyclic aromatic hydrocarbons, chlorophenols, pentachlorophenol etc., although found to be very effective against wood destroying organisms but are said to cause environmental pollution (Onuorah 2000). Therefore, much emphasis is now paid towards the development of toxic plant extract based ecofriendly formulations. The use of these biological means have greater advantages over the use of chemicals for protection against degradation because these are less toxic to users and are also environment friendly. Therefore, these can offer substantial advantages for wood protection, providing decay resistance against fungi at low cost and low mammalian toxicity. *Lantana camara* L. is known worldwide as an obnoxious weed. It also known to possess antifungal properties. In this work we report the efficacy of *L. camara* extract against a wood decay fungus.

Materials and Methods

The wood samples of size 5 ± 0.25 cm $\times$ 2.5 ± 0.15 cm $\times$ 2.5 ± 0.15 cm (longitudinal $\times$ radial $\times$ tangential) were prepared from air-dried woods of *Pinus roxburghii* Sargent, *Celtis australis* L., and *Bombax ceiba* L.

Preparation of Extract and Treatment

The tender twigs of *Lantana camara* L. along with leaves were collected, shade dried and grounded into fine powder. The ratio of twigs vs leaves was 1:4. The powdered material was extracted with methanol in Soxhlet apparatus on a pre-heated water bath. After complete extraction, methanol was completely distilled off from the extract and the residue was vacuum dried. Stock solution was prepared by dissolving the vacuum dried extract in 5% methanol. From the prepared stock solution, different concentrations of 0.25, 0.50, 1.00, 1.50 and 2.00% (w/v) were

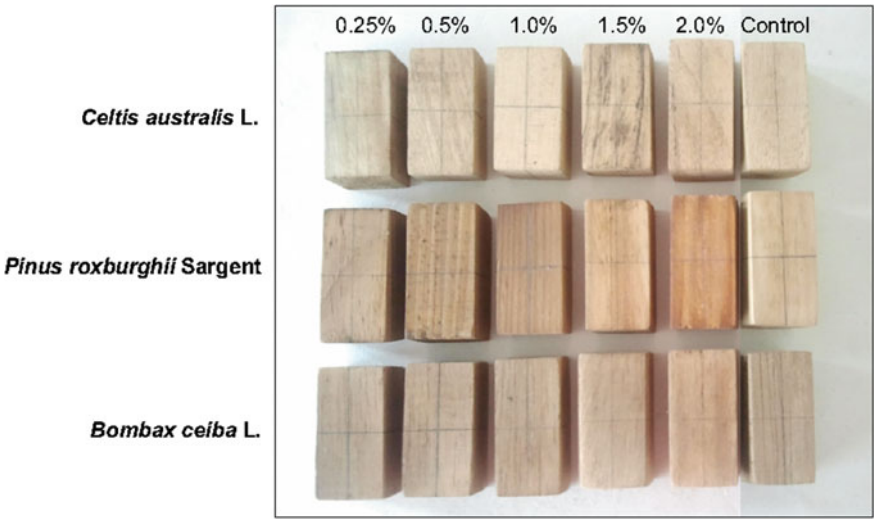

Fig. 1 *Lantana camara* extract treated wood

prepared for dip treatment. The control was taken as 5% methanol solution in distilled water.

The wood specimens of *Pinus roxburghii* Sargent, *Celtis australis* L., and *Bombax ceiba* L. of size 5 cm × 2.5 cm × 2.5 cm (longitudinal × radial × tangential) were dried at 105 ± 2 °C to constant weights and dipped in different concentrations of *Lantana camara* L. extract solution for 72 h. Three replicates for every treatment in each species were taken and seven samples were used in each replication. The treated samples were then dried at 105 ± 2 °C to constant weights. Preservative treated specimens of all the species are shown in Fig. 1. The difference in weight of the samples after and before treatment was taken as the weight gain in the wood sample as given by Rowell and Ellis (1978).

$$W = \frac{W_a - W_b}{W_b} \times 100$$

where

W_a Weight of oven-dried sample after treatment
W_b Weight of oven-dried sample before treatment.

Isolation and Identification of Fungus

A commonly occurring wood rotting fungus was isolated from the sporophore of *Polyporus* sp. growing on the dead wood on routine malt extract-agar medium. The

isolated fungus was transferred to the agar slants and purified by hyphal tip method. The purified fungal culture was again transferred to petri plates containing malt agar medium and kept for incubation at 25 ± 1 °C for routine work. The identification of the isolated fungus was confirmed with the help of standard literature and appropriate standard keys (Waterhouse 1963, 1970; Ho 1982) as *Polyporus* sp. The isolated fungus was grown on malt extract agar medium. For the growth of fungus malt agar solid media was prepared directly by adding 2% malt and 2% agar in 1000 ml distilled water. About 100 ml of the medium was poured in glass jars of 500 ml capacity and autoclaved at 121 °C and 15 lb pressure per square inch (psi) for 20 min. The glass jars with solidified medium were inoculated with the fungus culture bits (5 mm) cut from 10 days old vigorously growing culture and the inoculated glass jars were then incubated at 25 ± 1 °C in a BOD incubator for 15 days. The treated wood samples were then placed in the middle of the jar on the actively growing fungal colony for 21 days and incubated at 25 ± 1 °C again. For Fungal Colonization (%) the following scale was used:

Numerical ratings	Description
0	0% area cover
1	1–25% area cover
2	26–50% area cover
3	51–75% area cover
4	>76% area cover

Per cent fungus colonization index (C) was calculated according to standard method given by McKinney (1923):

$$C = \frac{\text{Sum of all disease ratings}}{\text{Total number of ratings} \times \text{Maximum disease grade}} \times 100$$

Per cent fungus growth inhibition (Vincent 1947) was calculated by using following formula:

$$I = \frac{C - T}{C} \times 100$$

where

I Per cent fungus growth inhibition
C Per cent fungus colonization in control wood
T Per cent fungus colonization in treated wood.

Results and Discussion

The variation in weight gain of treated and untreated wood for different wood species and treatments was found to be significant at 5% level of significance (Table 1). Among different wood species, the maximum preservative uptake (0.539%) was recorded in *Bombax ceiba* L. (S_3) and the lowest uptake (0.411%) was observed in *Pinus roxburghii* Sargent. (S_2). For the treatments at different concentrations, the highest weight gain of 0.843% was observed at T_5 (2% concentration) and the lowest weight gain of −0.640% in control. Preservative uptake generally increases with the increase in concentration. The values for control were found to be negative which indicates that the water soluble extractives may have leached out from untreated samples. The interactions between species and treatments were also found to be significant with maximum weight gain (1.323%) in *Bombax ceiba* L. (S_3) at T_5 (2% concentration) and minimum (−0.828%) in *Bombax ceiba* L. (S_3) in control (T_6). From the present findings it can be observed that the treated samples have gained weight as compared to the untreated samples because of absorption of the extract. Similar findings have been reported by Sotannde et al. (2011) where the wood samples soaked in leaf extract showed the highest absorption. There is an increase in per cent weight of the samples with the increase in concentration of plant extract. Jusoh et al. (2012) have also reported similar results that increase in concentration of preservatives from 0.1 to 1% resulted in increased retention in all wood species.

The wood is a degradable substance and susceptible to the attack of various insects and fungi. The main aim of this study was to test the efficacy of *Lantana camara* L. extract as a bio-preservative. The preservative treated wood were exposed to fungal degradation. The fungal colonization on untreated and treated wood is shown in Figs. 2 and 3, respectively. Fungus growth index and fungus growth inhibition, for all the species and treatments, has been presented in Table 2.

Table 1 Per cent variation in oven dry weight of treated and untreated wood (%)

Treatment (T)	Species (S)			
	(S_1) *Celtis australis* L.	(S_2) *Pinus roxburghii* Sargent	(S_3) *Bombax ceiba* L.	Mean
T_1 (0.25%)	0.619	0.522	0.271	0.471
T_2 (0.50%)	0.836	0.542	0.569	0.649
T_3 (1.00%)	0.847	0.594	0.783	0.741
T_4 (1.50%)	0.833	0.573	1.115	0.840
T_5 (2.00%)	0.654	0.552	1.323	0.843
T_6 (Control)	−0.777	−0.315	−0.828	−0.640
Mean	0.502	0.411	0.539	

$CD_{0.05}$
Species (S): 0.08
Treatments (T): 0.11
S × T: 0.20

C. australis L. *P. roxburghii* Sargent *B. ceiba* L.

Fig. 2 Fungus colonization in untreated wood

C. australis L. *P. roxburghii* Sargent *B. ceiba* L.

Fig. 3 Fungus colonization in wood treated with *L. camara* extract (2% concentration)

The maximum fungus colonization (100%) was recorded in control for all the species and also for *Celtis australis* L. (S_1) at T_1 (0.25% concentration) and *Pinus roxburghii* Sargent. (S_2) at T_1 (0.25% concentration) and T_2 (0.50% concentration) and the minimum fungus colonization (41.67%) has been found at 1.50% (T_4) and 2.00% (T_5) concentrations in *Bombax ceiba* L. (S_3). Therefore, highest fungus growth inhibition (58.33%) was evident in Bombax ceiba L. at 1.5 and 2% concentrations and the lowest (i.e. no fungal growth inhibition) in control for all the species and also for Celtis australis L. at 0.25% concentration and for Pinus roxburghii Sargent at 0.25% and 0.5% concentrations. This indicates antifungal activity of extracts of *Lantana camara*. Similar findings has been reported by Kabir and Alam (2007) using 3% concentration of leaf extract in acetone. The results are in contrast with Tripathi et al. (2009) where the ethanolic extract up to 0.01%

Table 2 Effect of *Lantana camara* L. leaf extract on fungus colonization on treated and untreated wood samples

Treatments	Fungus Growth Index (%)			Fungus Growth Inhibition (%)		
	(S_1) *Celtis australis* L.	(S_2) *Pinus roxburghii* Sargent	(S_3) *Bombax ceiba* L.	(S_1) *Celtis australis* L.	(S_2) *Pinus roxburghii* Sargent	(S_3) *Bombax ceiba* L.
T_1 (0.25%)	100.00	100.00	91.67	0.00	0.00	8.33
T_2 (0.50%)	91.67	100.00	75.00	8.33	0.00	25.00
T_3 (1.00%)	75.00	83.33	66.67	25.00	16.67	33.33
T_4 (1.50%)	58.33	75.00	41.67	41.67	25.00	58.33
T_5 (2.00%)	58.33	75.00	41.67	41.67	25.00	58.33
T_6 (Control)	100.00	100.00	100.00	–	–	–

concentration level recorded no growth of *Tremetes versicolor* (white rot) and *Oligoporus placenta* (brown rot) as compared to 100% observed in control plates. Thus, the leaf extract of *L. camara* L. has been found inhibitory against *Polyporus* sp. and could protect the treated wood samples at the test concentrations of 1.5 and 2% in case of *Bombax ceiba* L. It was effective at concentration $\geq 1.5\%$ in *C. australis* L. and at concentration $\geq 1\%$ in case of *P. roxburghii* Sargent. Earlier plant extracts have also been reported to inhibit the growth of many fungi *in vitro* as such or on that treated wood samples (Kumar 2004).

Conclusion

The leaf extract of *Lantana camara* L. has been found inhibitory against *Polyporus* sp. and could protect the treated wood samples at the concentrations of 1.5 and 2%. Fungal colonization has decreased with the increase in *Lantana camara* L. extract concentration. However, further studies are needed for the exploitation of plant extracts for eco-friendly preservation and treatment of the wood at commercial scale.

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Powderpost Beetle Menace in Wooden Handicraft Industries and Their Management

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Abstract In India, the handicraft industry is considered the second largest industry next to agriculture. Apart from lack of supply of conventional wood raw material, insect pest problems are an added threat to these wooden handicraft industries. Many of the wooden handicraft products are inferior in quality due to use of untreated raw material, which is susceptible to powderpost beetle damage. Infestation by these powderpost beetle usually starts in the raw material and may continue in the finished product or after being passed on to the consumer. Effective management measures are a prerequisite for this industry to deliver quality wooden handicraft products. This paper deals with the powderpost beetle menace experienced by the wooden handicraft industries in Karnataka, India. The remedial measures for the effective management of powderpost beetle are also suggested.

Keywords Alternate timbers · Borers · Handicraft · Pests · Powderpost beetle

Introduction

Biological degradation or insect attack takes place right from the time the tree is felled and continues till its conversion to structural material/finished products and further beyond during its service life by the consumer (Purushotham 1975). There is an ecological sequence of attack by different borers depending primarily upon the moisture gradient and other related factors in the wood. There are different kinds of insects belonging to different orders which could be classified as insect borers of wood. The Coleoptera has been observed as the most important order containing insect borers, causing damage to timber. These insect borers are found on trees, logs

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and in timber containing varying moisture content and even in dry timber (Mathur 1961). The damage continues after making of the finished product, or in a wooden structure of a building. Many finished wood products like handicraft items, packing cases, veneers, plywoods, blockboards, brush handles, bobbins, photo frames are heavily infested by insect borers. Timber infested by insect borers is usually neglected by the manufacturer or consumer until the damage reaches such a state that the finished product requires to be discarded incurring heavy loss of revenue.

The susceptibility of different timber species varies with different species of insect borer, and the magnitude of damage depends mainly on parameters such as climate and locality. Starch in wood is the most preferred component by these insect borers.

In India, the handicraft industry is considered the second largest industry next to agriculture and its existence is known since the beginning of civilization and is represented as an important social, economic and cultural activity. Wood is one of the most prominent materials used by the handicraft industry in India. Lack of availability of conventionally used timber species has resulted in use of alternative timber species. Apart from lack of supply of wood raw material, insect pest problems are an added threat to the wooden handicraft industries. Many of the handicraft products are inferior in quality due to the attack by insects. This affects their commercial value both in the national and international markets. Therefore, in this study a survey was carried out to collect the information about various timber species and different beetle pests involved in the biodegradation of these timber species utilized by the handicraft industries in Karnataka, India.

Materials and Methods

Systematic surveys were carried out to collect information, using a formatted questionnaire from different handicraft industries, handicraft villages, individual artisan houses, handicraft showrooms, handicraft exhibitions, handicraft godowns at Chennapatna, Mysore and Bengaluru in Karnataka, India. Detailed discussions regarding the different timbers used, insect-related problems, method of storage, etc., were held with the artisans and proprietors of the industries, and the information was documented. Infested raw wood material and finished handicraft products containing insect borer infestation were collected and labeled. The infested wood materials were brought and caged in the laboratory using zinc cages for the emergence of adult beetles. The emerged adult beetles were set and preserved for identification. The emerged beetles were mounted, set and dried in the hot air oven and later transferred to teak display boxes. Adult beetles were identified using a zoom stereoscopic microscope and by using keys.

Results and Discussion

Surveys conducted in the handicraft industries indicate that the artisans are facing severe shortage of the conventional timbers used for handicraft products. Low availability and high prices of the raw material have forced the artisans to use alternate timbers, many being very susceptible to insect borer attack.

A detailed list of the alternate and conventional timber species used in handicraft industries is shown in Table 1, and the different insect borers collected from these handicraft timber species is shown in Table 2.

Table 1 Alternate and conventional timber species used in handicraft industries in Karnataka, India

	Timber species	Common/trade name	Handicraft products
1	*Acacia arabica*	Gobbadi, Karijali	Toy-making, turnery
2	*Acacia auriculaeformis*	Bengal Jali	Turnery
3	*Adina cordifolia*	Yethiga, Heddi, Haldu	Inlay work
4	*Artocarpus integrifolia*	Halasu, Jackfruit tree	Inlay work
5	*Alstonia scholaris*	Maddale, Satin wood	Turnery
6	*Azadirachta indica*	Bevu, Neem wood	Wood carving
7	*Dalbergia latifolia*	Beete, Shisham, Rose wood	Wood carving, turnery
8	*Diospyros ebenum*	Karimara, Bale, Ebony wood	Inlay work
9	*Eriodendron anfractuosum*	Pania	Inlay work
10	*Eucalyptus hybrid*	Hybrid Neelgiri	Turnery
11	*Ficus infectoria*	Basan, Karibasari	Turnery
12	*Ficus religiosa*	Arali	Inlay work
13	*Grevillea robusta*	Silver oak	Wood carving
14	*Hevea brasiliensis*	Rubber wood	Wood carving, turnery
15	*Leucaena leucocephala*	Subabul, White Lead tree	Turnery
16	*Mangifera indica*	Mavu, Mango wood	Wood carving
17	*Michelia champaka*	Sampige, Champa	Inlay work
18	*Pithecolobium dulce*	Dakhani babal, Vilayati Imli	Wood carving
19	*Pithecolobium saman*	Rain tree	Wood carving
20	*Pterocarpus dalbergiodes*	Rakta Chandan	Inlay work
21	*Pterocarpus marsupium*	Honne, Bijasal	Inlay work
22	*Santalum album*	Srigandha, Chandana, Sandalwood	Wood carving
23	*Syzygium cuminii*	Nerale, Jambul, Jamun	Inlay work
24	*Tamarindus indica*	Hunase, Imli	Inlay work
25	*Tectona grandis*	Saguvani, Tega, Teak	Wood carving, turnery
26	*Terminalia arjuna*	Neeramathi, Holemathi	Inlay work
27	*Terminalia chebula*	Alale, Gall nut, Harda	Inlay work
28	*Vitex leucoxylon*	Hole-lakki, Sankani	Inlay work
29	*Wrightia tinctoria*	Hale, Kadumurkha	Turnery

Table 2 Insect borers collected from the above timber species used in handicraft industries

S. No.	Timber species	Insect pests collected
1	*Acacia arabica*	*Lyctus africanus* Lesne *(Lyctidae)* *Sinoxylon anale* Lesne *(Bostrychidae)*
2	*Acacia auriculaeformis*	*L. africanus* Lesne *(Lyctidae)* *Minthea rugicollis* Walker *(Lyctidae)*
3	*Adina cordifolia*	*L. africanus* Lesne *(Lyctidae)* *H. aequalis* Waterhouse *(Bostrychidae)* *S. anale* Lesne *(Bostrychidae)* *Sinoxylon conigerum* Gerst *(Bostrychidae)* *Platypus solidus* Walker *(Platypodidae)* *Xyleborus* sp. *(Scolytidae)*
4	*Alstonia scolaris*	*Heterobostrychus aequalis* Waterhouse *(Bostrychidae)* *L. africanus* Lesne *(Lyctidae)* *M. rugicollis* Walker *(Bostrychidae)* *S. anale* Lesne *(Bostrychidae)* *Xylothrips flavipes* Illiger *(Bostrychidae)*
5	*Artocarpus integrifolia*	*Xyleborus similis* Ferr. *(Scolytidae)* *Cossonus divisus* Mischl. (*Curculionidae)* *Sintor* sp. *(Anthribidae)*
6	*Azadirachta indica*	*S. anale* Lesne *(Bostrychidae)* *Crossotarsus saudersi* Chapuis *(Platypodidae)*
7	*Dalbergia latifolia*	*S. anale* Lesne *(Bostrychidae)* *M. rugicollis* Walker *(Lyctidae)* *Xyleborus interjectus* Bland. *(Scolytidae)*
8	*Diospyros ebeum*	–
9	*Eriodendron anfractuosum*	*S. anale* Lesne *(Bostrychidae)* *Dinoderus* sp. *(Bostrychidae)* *X. flavipes* Illiger *(Bostrychidae)* *P. solidus* Walker *(Platypodidae)* *X. interjectus* Bland. *(Scolytidae)*
10	*Eucalyptus hybrid*	*S. anale* Lesne *(Bostrychidae)*
11	*Ficus lacor (Ficus infectoria)*	*L. africanus* Lesne *(Bostrychidae)* *Platypus* sp. *(Platypodidae)*
12	*Ficus religiosa*	–
13	*Grevillea robusta*	*L. africanus* Lesne *(Bostrychidae)* *S. anale* Lesne *(Bostrychidae)* *Xyleborus* sp. *(Scolytidae)*
14	*Hevea brasiliensis*	*L. africanus* Lesne *(Lyctidae)* *M.rugicollis* Lesne *(Lyctidae)* *S. anale* Lesne *(Bostrychidae)* *Sinoxylon atratum* Lesne (*Bostrychidae*) *S. conigerum* Gerst *(Bostrychidae)* *H. aequalis* Waterhouse (*Bostrychidae)* *P. solidus* Walker *(Platypodidae)* *Stromatium barbatum* Fabricius *(Cerambycidae)* *Phaenomerus sundevalli* Boh *(Curculionidae)*

(continued)

Table 2 (continued)

S. No.	Timber species	Insect pests collected
		Xyleborus sp.*n.r similes (Scolytidae)*
15	*Leucaena leucocephala*	*H. aequalis* Waterhouse *(Bostrychidae)* *S. anale* Lesne *(Bostrychidae)* *S. atratum* Lesne *(Bostrychidae)* *Xyleborus* sp. *(Scolytidae)*
16	*Mangifera indica*	*S. anale* Lesne *(Bostrychidae)* *H. aequalis* Waterhouse *(Bostrychidae)* *L. africanus* Lesne *(Lyctidae)* *Platypus solidus* Walker *(Platypodidae)* *Xyleborus* sp. *(Scolytidae)*
17	*Michelia champaka*	*L. africanus* Lesne *(Lyctidae)*
18	*Pithecolibium dulce*	*L. africanus* Lesne *(Lyctidae)*
19	*Pithecolibium saman*	*L. africanus* Lesne *(Lyctidae)*
20	*Pterocarpous indicus*	*L. africanus* Lesne *(Lyctidae)* *S.anale* Lesne *(Bostrychidae)*
21	*Ptercarpous marsupium*	*L. africanus* Lesne *(Lyctidae)* *S. anale* Lesne *(Bostrychidae)*
22	*Santalum album*	*L. africanus* Lesne *(Lyctidae)* *S. anale* Lesne *(Lyctidae)* *S. atratum* Lesne *(Bostrychidae)*
23	*Syzygium cuminii*	*S. anale* Lesne *(Bostrychidae)* *P. solidus* Walker *(Platypodidae)* *Xyleborus* sp. *(Scolytidae)*
24	*Tamarindus indica*	–
25	*Tectona grandis*	*L. africanus* Lesne (*Lyctidae)* *S. anale* Lesne *(Bostrychidae)* *Platypus* sp. *(Platypodidae)*
26	*Terminalia arjuna*	*L. africanus* Lesne *(Lyctidae)* *S. anale* Lesne *(Bostrychidae)* *S. atratum* Lesne *(Bostrychidae)*
27	*Terminalia chebula*	*S. anale* Lesne *(Bostrychidae)* *Xyleborus* sp. *(Scolytidae)*
28	*Vitex leucoxylon*	–
29	*Wrightia tinctoria*	*L. africanus-Lesne (Bostrychidae)* *Xyleborus* sp. *(Scolytidae)*

Freshly felled logs having high moisture content were attacked by *Platypodidae* containing the pinhole borers and *Scolytidae* containing the shothole borers, bark beetles and ambrosia beetles. When the felled logs further dries and gets seasoned, the next major group of insect borers or beetles which attack and damage timber in storage were the powderpost beetle (Bostrychidae and Lyctidae) (Remadevi 2002). These two major families, under the Order Coleoptera (1) Family: *Bostrychidae* comprise the cylindrical powderpost beetle and (2) Family: *Lyctidae* comprise the flattened powderpost beetle. The reasons for attack by the two families of insects

were totally dependent on two conditions, namely the moisture content of the timber logs, i.e., when moisture is reduced to about 30–50%, and the other condition for the attack of these beetles is sapwood containing sufficient starch for the development of the larvae, and it is only under this condition that adult beetles bore inside the sapwood to lay her eggs. Bostrychids being polyphagous, thrive well in different timber depots containing different timber species. The quantity of starch in the sapwood is dependent on the timber species and the season of the year when it was felled.

The family Lyctidae is a major insect pest attacking dry timber. It is also dependent on starch for its nutrition and thrives well in sapwood with a moisture content of 10–15%; wood less than 10% moisture content is not attacked. However, higher moisture is favored for larval development (Beeson 1941). Only timbers having minimum average vessel diameter of about 60 μ are likely to be attacked by Lyctids. The reason for the above condition is that the female Lyctid beetle lays her eggs only by introducing its ovipositor measuring from 50 to 90 μ into the wood vessel of the timber. Few eggs are also laid in the lumen of wood vessels exposed by the mandibular action of the adult beetles. These tasting marks are referred by Parkin (1936). Many handicraft timbers were found susceptible to *Lyctus africanus*-Lesne. This Lyctid is the most common species which is widely found distributed throughout India. Since the emergence of Lyctids was observed throughout the year, its damage could be encountered throughout the year in the handicraft industry.

Wood Carving or Statue Making

It was noticed that wood carvings made out of alternate timbers like *Hevea brasiliensis, Mangifera indica, Grevillea robusta, Azadiractha indica, Pithecolubium dulce, Pithecolubium saman* suffered damage due to either Platypodid, Bostrychid or Lyctid infestations. Wood carvings made of conventional timber such as *Dalbergia latifolia* also suffered damage due to *Minthea rugicollis* attack when sapwood was included in these carved images. Stained Platypodid holes were also observed in the sapwood of *Dalbergia latifolia* (Table 2).

For a carved image, usually the half girth of a timber log is used for carving. The inner half girth is used as the back of the carved image and the curved outer half or circumference of the bole is used as the portion for carving. This results in more sapwood being used on the carved portion, and many wood vessels were exposed for Lyctid attack. The rough surface or unpolished carved product and the delay in sanding and polishing of the carved material make it further susceptible to Lyctid and Bostrychid attack. The godowns containing insect borer infested finished handicraft products were found to be a breeding ground for both Lyctid and Bostrychid beetles infestations. Timely detection and isolation of borer infested material and preservative treatment are essential to save these finished handicraft products and from future destruction and spread of these timber pests to other

uninfected finished handicraft products. Polishing handicraft products prior to insect attack made the product immune to fresh attack and could prevent seasonal reabsorption of moisture required for larval development. Use of linseed oil for polishing was observed to give a higher protection against moisture and attained moisture curbing properties (Badoni et al. 1990). *Hevea brasiliensis* timber, which is found suitable for handicraft products (Rao et al. 1993), was found to be highly susceptible to insect attack. *Sinoxylon anale*-Lesne was observed to cause severe economic loss (Gnanaharan et al. 1983).

Inlay/Relief or Patchwork

Traditionally, the inlay work or relief work or patch work was done, using veneers of different timber species of different colors pasted on to solid wood base, namely *Dalbergia latifolia*. Presently, this same inlay work or relief work using different timber species of different colors is fabricated using untreated one-sided teak plywood or block board as the base material. This in return has led to the infestation of insect borers mainly *Lyctus africanus*-Lesne. The different timbers used in the patch work were also found susceptible to different insect borers (refer Table 2).

Wood-Turning or Toy-Making

Wood-turning or toy-making industries were found using lathe machines for turning toys and other handicraft products, and the wood material used was mainly *Wrightia tinctoria* (Dudi) *Alstonia scholaris* (Hale), and *Adina cordifolia* (Haldu). *W. tinctoria* as a raw material with bark was found infested with Platypodid beetles *Platypus s*p., Scolytid beetles *Xyleborus* sp and Lyctid beetles *Lyctus africanus*-Lesne. *A. scholaris* (Hale) was found to be susceptible to *Sinoxylon sp,* and *Heterobostrychus aequalis*-Waterhouse. Another timber used for turning in the toy industry, *A. cordifolia* (Haldu) as a raw material was found susceptible to Bostrychid beetles, namely *H. aequalis*-Waterhouse, *Sinoxylon anale*-Lesne, *Sinoxylon conigerum*-Gerst, Platypodid beetles namely *Platypus solidus*-Walker and Scolytid beetles namely *Xyleborus* sp. Alternate timbers such as *Acacia auriculaeformis, Eucalytptus camaldulensis, Eucalyptus tereticornis, Maeopsis eminii and Swietenia mahogany* are also recommended as alternate for turning purposes (Kumar et al. 1995). *A. auriculaeformis* was found to be attacked by *L. africanus*-Lesne *and Minthea rugicollis*-Walker. The sapwood of *Eucalyptus camaldulensis, Eucalyptus tereticornis and Leucaena leucocephala (Subabul)* used for pulp, paper production and other purposes was found to be destroyed in varying degrees by powderpost beetle (Bostrychids), namely *Sinoxylon* spp. and *Heterobostrychus aequalis.*

Alternate timbers used in the wood-turning or toy-making industry were *Eucalyptus hybrid* and *Ficus lacor*. Raw wood material, *Eucalyptus hybrid* was found prone to the attack of Bostrychid beetles *Sinoxylon anale*-Lesne (Bostrychidae). *Ficus lacor* used for making rocking horses was found susceptible to Lyctid beetles, namely *Lyctus africanus*-Lesne (Lyctidae) and Platypodid beetles, namely *Platypus* sp. (refer Table 2). *Dalbergia latifolia* sapwood was found infected with *Xyleborus interjectus*-Bland. (Scolytidae) in the wood-carving industry at Mysore. Studies have proved that influence of pinholes caused by these ambrosia definitely weaken the strength of timber (Rajput and Gupta 1980; Rajput et al. 1990) and are rendered useless for decorative purposes. However, after turning the wood material with lathes, it was smoothened with sand paper and lacquer dyes of different colors were immediately applied on the turned wood material over the lathe machines itself and this finished product was rendered immune to the above insect borers.

Timber/Finished Product Protection Practices

In the handicraft sector, the only precaution taken by the artisans was that they procured raw wood material only in the rainy season, assuming that insect borer population or activity was low in this period. Some treated their finished handicraft only after it was found attacked by beetle pests, by immersing the finished handicraft product in caustic soda and later in cashew nut liquid preparation. Since a lot of revenue is gained by foreign exchange in the handicraft industry, it is envisaged that quality handicraft products are expected by the consumer.

Management of Timber Insects in the Wood Handicraft Industries

Preventive Measures

- Procurement of raw material preferably during winter or after rainy season and shifted at the earliest from felling site to place of fabrication/factory site.
- Debarking timber soon after arrival to prevent bark beetles.
- Low temperature kiln drying of timber.
- Stringent hygienic conditions to be maintained before and after procurement at factory site.
- Consumption of raw material on first come first basis.
- Continuous inspection of raw material and finished product in godowns for any insect infestation.

Remedial Measures

- Sterilization by heat treatment kilns at 120–150 °F for 2–3 h or dipping in boiling water for 10 min.
- Fumigation in an airtight enclosure for 48 h using fumigants such as methyl bromide, sulfuryl fluoride, carbon disulfide, formaldehyde at 40 ml per 28 m^3 of space. Aluminum phosphide 4 g/m^3 for indoors for 24 h.
- Insecticidal treatment: Protection of logs using aqueous solution of chlorpyrifos (0.5–1%) using sprayers or impregnation of wood using 1% chlorpyrifos or permethrin or bifenthrin for finished products or structural materials.

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Effect of Thermal Modification on Physical Properties of *Bambusa nutans*

Kiran Ghadge and Krishna K. Pandey

Abstract Thermal modification is a promising eco-friendly technique for improving water repellency and decay resistance of wood-based products. In this work, specimens of *Bambusa nutans* were thermally modified in a vacuum oven between 195 and 225 °C for different durations. The effect of heat treatment on physical properties (colour, mass and density) and dimensional stability was investigated. Heat treatment resulted in uniform dark colour, reduced density and improved dimensional stability of bamboo. Anti-swelling efficiency increased with severity of modification.

Keywords *Bambusa nutans* · Colour · Dimensional stability · Thermal modification

Introduction

Bamboo is a fascinating bio-resource extensively used as a construction material and for making furniture and other artefacts. It is one of the most abundant and renewable resources. However, susceptibility of bamboo to fungi and insects limits its usage. Other major drawbacks of furniture and artefacts made of bamboo are its poor aesthetic look due to its light colour and colour variability. Improvement in the colour and uniform colouration of the raw material will add value to the end product. Although artificial colouration can be achieved by using synthetic dyes, the coating is superficial and may need frequent refinishing.

One of the artificial ways of improving the colour of wood- and/or bamboo-based products is through thermal treatment. The technique mainly involves subjecting the raw material to higher temperature for certain period of time under inert environment

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without use of any chemicals. The thermal modification imparts several important chemical and physical changes to the material. The colour of raw material changes to an attractive darker colour that penetrates the entire thickness of the piece unlike synthetic dye which is only confined to the surface. Thermal modification is an environmental friendly method and alternative to pressure-treated wood because there is no chemical treatment involved in the process. Thermal modification has been found to increase moisture resistance, dimensional stability and durability of wood (Esteves and Pereira 2009; Viitaniemi et al. 1997). Effect of thermal modification on the colour and chemical changes of heat-treated softwoods and hardwoods has been extensively studied (Esteves et al. 2008; Esteves and Pereira 2009; Viitaniemi et al. 1997; Tjeerdsma et al. 1998; Militz 2002; González-Peña et al. 2009; Bekhta and Niemz 2003; Nuopponen et al. 2005; Kocaefe et al. 2008; Tuong and Li 2010; Srinivas and Pandey 2012). However, studies on thermal modification of bamboo are meagre (Nguyen et al. 2012; Bremer et al. 2013).

In this paper, preliminary results on some of the physical properties of thermally modified *Bambusa nutans* are presented. Specimens of *B. nutans*, with or without epidermis, were heat treated in the temperature range 195–225 °C in a vacuum oven, and some of the properties (colour changes, mass loss, density and anti-swelling efficiency) were evaluated.

Materials and Methods

Material

Bambusa nutans culms of 8–10 years old and 20–30 ft height were harvested from the Forest Research Institute (FRI), Dehradun. Another set of culms of about 2 years old and 20 ft height were collected from the nursery of Institute of Wood Science and Technology (IWST), Bengaluru. The culms after harvesting were cut into ring shapes at an interval of 4 cms excluding the nodes. One set of culms (collected from FRI) were sanded to remove the epidermis, whereas another set (collected from IWST) were left without sanding. These culms were then further cut into two equally sized pieces, upper half and lower half. The specimens were dried at room temperature and were then transferred to a hot air oven at 65 °C for 2 days and finally at 103 ± 2 °C till constant weight was attained.

Heat Treatment

The samples were thermally modified in a vacuum oven at 190–225 °C for 3 and 6 h, and their mass loss, density, colour and chemical changes evaluated. Six samples each with and without epidermis were thermally modified at different

temperatures. Then, the samples were allowed to cool and their oven-dried weights were measured.

Evaluation of Mass Loss

The mass loss due to thermal modification was calculated. The mass loss was calculated according to Eq. 1,

$$\text{Mass Loss}\,(\%) = [(m_o - m_t)/m_o] * 100 \tag{1}$$

where m_o and m_t are the mass of the oven-dried samples before and after thermal modification, respectively.

Colour Changes

The colour was measured using a Spectrocolorimeter (Hunterlab-Labscan XE model) according to the CIELAB colour space, which is characterized by lightness (L^*), redness (a^*) and yellowness (b^*). Three samples from each set were selected for determining the colour. For every sample, 6 measurements were taken at different places and average value was calculated. Colour co-ordinates, i.e. L^*, a^* and b^*, were measured on each samples before and after heat treatment, and change in these parameters due to heat treatment, i.e. ΔL^*, Δa^* and Δb^*, was calculated. The colour difference ΔE^* was calculated according to the following Eq. 2,

$$\Delta E^* = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \tag{2}$$

Estimation of Density

The density of untreated and thermally modified bamboo specimens was calculated using Eq. 3,

$$\text{Density}\left(\text{g/cm}^3\right) = m/V \tag{3}$$

where m is mass and V is the volume.

Evaluation of Dimensional Stability

The degree of dimensional stability was determined by calculating the volumetric swelling coefficient (S) and anti-swelling efficiency (ASE) by repeated water soaking method (Rowell and Ellis 1978). Samples were submersed in distilled water and evacuated in a vacuum desiccator. The volumetric swelling coefficient was calculated according to the following Eq. 4,

$$S\,(\%) = [(V_2 - V_1)/V_1] * 100 \tag{4}$$

where V_1 = volume of bamboo sample before wetting; V_2 = volume of bamboo sample after wetting in water. The anti-swelling efficiency was calculated using Eq. 5,

$$\mathrm{ASE}\,(\%) = [(S_u - S_m)/S_u] * 100 \tag{5}$$

where S_u = swelling coefficient of untreated specimen; S_m = swelling coefficient of thermally modified specimen.

Results and Discussion

The various physical properties such as colour, mass loss, density, dimensional stability showed a remarkable change in bamboo due to thermal modification.

Colour Changes

Colour of bamboo became darker upon thermal modification. Thermally modified bamboo showed a uniform colour throughout (Fig. 1). The values of total colour change (ΔE^*) increased with increase in temperature and exposure duration. The effect of temperature and exposure duration for specimens of *B. nutans* without epidermis is shown in Fig. 2.

Mass Loss

Thermal modification resulted in a remarkable change in the weight of the bamboo. Bamboo, like wood, consists of cellulose, hemicelluloses and lignin which differ in their thermal stability. Mass loss of bamboo differed according to the modification conditions. Mass loss increased with increase in temperature and duration (Fig. 3). The mass loss after 6-h heat exposure of the bamboo samples with epidermis at

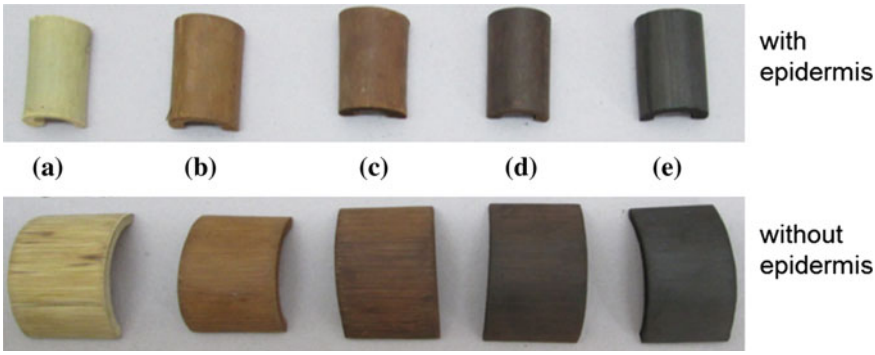

Fig. 1 Colour changes in *B. nutans* after 3-h heat treatment at different temperatures: **a** control, **b** 195 °C, **c** 205 °C, **d** 215 °C and **e** 225 °C

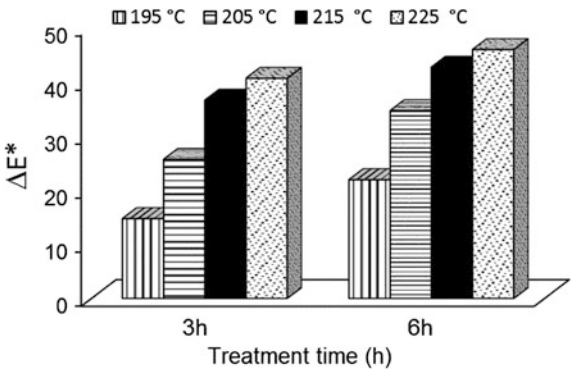

Fig. 2 Effect of temperature and heat treatment time on total colour changes (ΔE^*) of *B. nutans* without epidermis

195 °C was 4.4%, whereas at 225 °C it was 17%. Similarly, the mass loss of bamboo without epidermis after 6-h heat treatment was 3.83 and 14.76% at 195 and 225 °C, respectively.

Density

Density of thermally modified bamboo was determined by measuring the weight and volume of the samples. Thermally modified specimens have lower density as compared to the unmodified specimens. Density of the bamboo decreased with increase in temperature and duration of the modification (Fig. 4). The strength loss is related to the density decrease factor, and hence, reduction in density can be an indicator of strength loss in bamboo due to heat treatment.

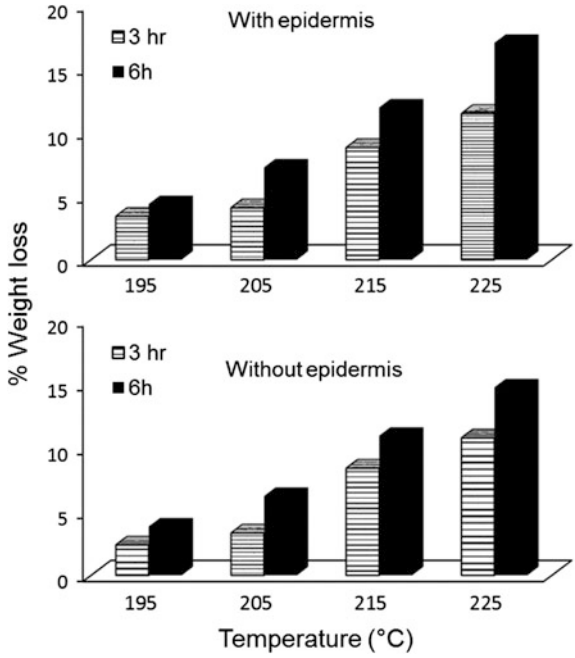

Fig. 3 Effect of heat treatment on weight loss of *B. nutan* with and without epidermis

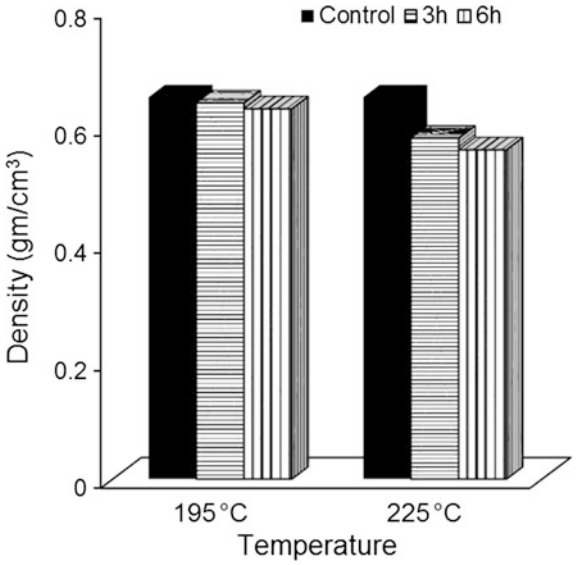

Fig. 4 Effect of thermal modification on density of *B. nutans* without epidermis

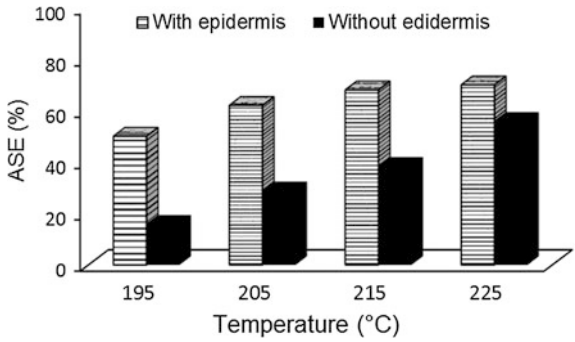

Fig. 5 Anti-swelling efficiency (ASE) of *B. nutans* heat treated for 6 h at different temperatures

Dimensional Stability

The dimensional stability of heat-treated bamboo was evaluated by determining anti-swelling efficiency (ASE) by water soaking method (Rowell and Ellis 1978). Volumetric swelling coefficient of heat-treated bamboo decreased considerably. ASE value increased with severity of treatment (Fig. 5). The improvement in dimensional stability of heat-treated bamboo is due to the chemical changes of cell wall polymers of bamboo, particularly hemicelluloses. Treatment at high temperatures modifies and degrades the hemicelluloses content of wood (Esteves and Pereira (2009). As hemicelluloses are highly hygroscopic, degradation of hemicelluloses reduces the hygroscopicity of bamboo, thus increasing its dimensional stability. The dimensional stability of bamboo increased with increase in temperature. The dimensional stability of specimens with epidermis was greater than that of samples without epidermis (Fig. 5).

FTIR Spectra

A variety of processes occur when lignocellulosic material undergoes heat treatment. Hemicelluloses are degraded to a higher extent than any the other cell wall components. This reduction takes place due to the presence of acetyl group. Thermal modification affects cellulose to a lesser extent than hemicelluloses because of its semi-crystalline nature. The proportion of lignin is generally increased in case of heat treatment (Esteves and Pereira 2009). FTIR spectroscopy was used to monitor chemical changes in heat-treated bamboo.

FTIR spectra of thermally modified bamboo are shown in Fig. 6. Decrease in the peaks at 1730, 1370 and 1237 cm^{-1} indicates degradation of carbohydrates present in bamboo. The decrease in the C=O carbonyl band at 1730 cm^{-1} and C–O absorption peak at 1237 cm^{-1} shows degradation of hemicelluloses. Increase in the peaks at 1598 cm^{-1} indicates increase in lignin proportion. However, a slight decrease at 1505 cm^{-1} at highest temperature (225 °C) shows that lignin has been partially degraded at very high temperature.

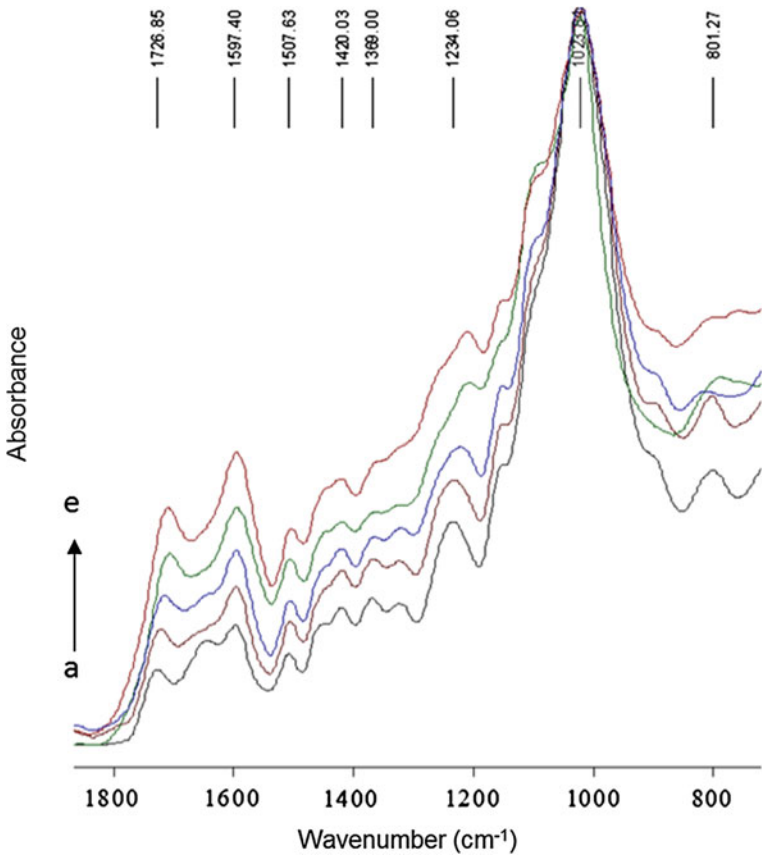

Fig. 6 FTIR spectra of thermally modified bamboo without epidermis after 3-h exposure at different temperatures (*a*) control, (*b*) 195 °C, (*c*) 205 °C, (*d*) 215 °C and (*e*) 224 °C

Conclusions

Thermal modification of bamboo resulted in changes in the properties of *B. nutans*. Severity of changes was dependent on the modification conditions (temperature and duration of modification). Temperature had more influence than duration. The colour of the bamboo turned from green to dark brown as modification intensity increased. The mass loss increased with increase in temperature and duration. The density of bamboo decreased with increasing temperature and treatment time which indicates negative influence of heat treatment on mechanical properties. The thermally modified bamboo showed good dimensional stability as compared to unmodified bamboo. ASE values increased with severity of modification.

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Part III
Wood-Based Composites

Micromechanics of Cellulose Fibres and Their Composites

Ramesh Babu Adusumalli, Karthik Chethan Venkateshan and Wolfgang Gindl-Altmutter

Abstract Cellulose fibres such as flax, hemp, viscose and lyocell were studied with respect to their use as reinforcing agents in composites. Initially, these fibres are subjected to single fibre tensile tests, and their adhesion with polypropylene and epoxy matrices was determined by application of a microbond technique. Unidirectional epoxy composites with fibre rovings and short fibre–epoxy composites with needle punched nonwovens were manufactured by means of compression moulding. Composites were subjected to mechanical vibrations, bending and tensile tests. Interfacial adhesion was also studied at the macro-level by means of double-notch shear test and scanning electron microscopy. Lyocell fibres performed equally well in comparison with natural cellulose fibres when the dimensional variability was taken into consideration, but less well than Glass fibres at both micro- and macro-levels. The low yield strength and high failure strain observed in the stress–strain diagram of lyocell and lyocell–epoxy composites can be the critical parameter in finding new applications for these biodegradable composites.

Keywords Cellulose fibre composites · Single fibre testing · Microbond test · Interfacial shear strength · Interlaminar shear strength · Damping

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Introduction

Fibre reinforced plastics (FRP) were developed in 1950 to improve the stiffness of monolithic plastics and as a potential alternative to metals where high specific strength and modulus are required (Chawla 1998a; Netravali and Chabba 2003; Kelly et al. 2000). These "advanced" plastic composites are used in aerospace industry and to make automotive parts, building materials and recently in special sporting goods as shown in Fig. 1. In 95% of the cases, glass fibres are used as reinforcing agents in FRPs (Mohanty et al. 2005). Due to the difficulties in the recycling and re-use of these glass fibre composites (Baillie 2004) and also motivated by European regulations on composite waste management such as "End-of-life vehicles regulation (ELV)" and "European Composite Recycling Concept", new composites called biocomposites were emerged in the late 1980s in which glass fibres were replaced by renewable natural fibres (Marsh 2003; Mohanty et al. 2000).

The advantages of natural fibres such as flax, hemp, ramie and others include low density, low cost, ease of processing, low energy consumption, CO_2 neutrality and biodegradability (Lampke 2001; Kessler et al. 1998; Nickel and Riedel 2003; Bledzki and Gassan 1999; Hanselka and Herrmann 1999). Since the introduction of flax/sisal fibre mat embedded in epoxy resin matrix in making door panels of the Mercedes-Benz E-Class in 1995 (Schuh 1999), natural fibres were commercialised (see Fig. 1). Subsequently, flax/PP composites have also been used in automotive interior components (Specht et al. 2002). However, natural fibres also have a number of disadvantages with respect to their use in composites (Lützkendorf et al. 2000; Gindl and Keckes 2006). The most important disadvantage is the considerable variation in their mechanical properties as shown in Fig. 2. Depending on the soil and climatic conditions, fibre diameter varies and also changes in retting conditions creates weak spots along the fibre length, which could be the reason for large variation in mechanical properties of natural fibres. Furthermore, the high mechanical properties often quoted for these materials are not representative for longer fibre bundles used in the manufacturing of composite materials (Wallenberger and Weston 2004).

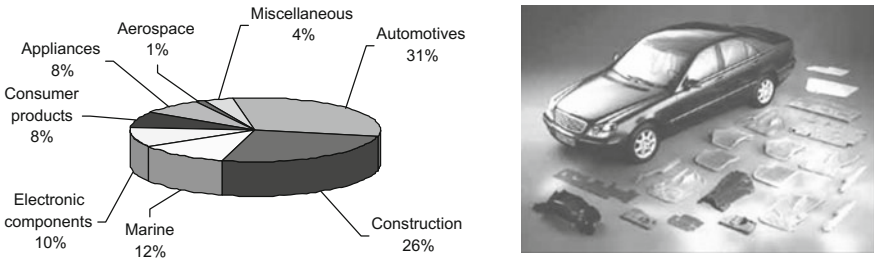


Fig. 1 *Left* FRPs used in 2002 (Mohanty et al. 2005); *right* natural cellulose fibre reinforced parts as interior components for the car (Wallenberger and Weston 2004)

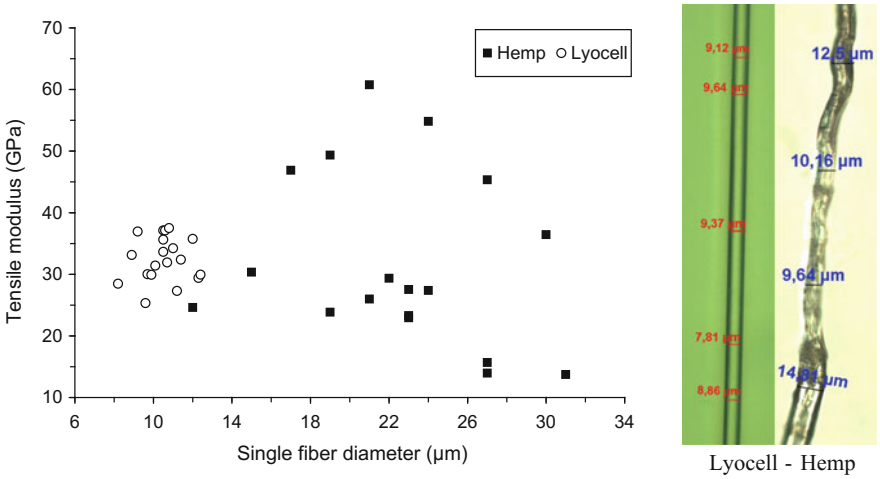


Fig. 2 *Left* variation in tensile modulus and fibre diameter of natural cellulose fibre (hemp) in comparison with regenerated cellulose fibre (lyocell). *Right* dimensional variability of lyocell and hemp

Here, regenerated cellulose fibres may be more competitive to glass fibres than natural fibres. Regenerated cellulose or man-made cellulose is made by dissolving high grade pulp through viscose process (cellulose xanthogenate) and, more recently, by direct dissolution in NMMO/water (lyocell process) (Woodings 2001). The latter technology produces fibres of superior properties (Fig. 2) and is environmentally friendly (Wallenberger and Weston 2004). Currently regenerated cellulose fibres are used mainly in textiles, tire cords and nonwoven products (Woodings 2001). A number of groups have studied composites with tire cord reinforcement (Fink et al. 2001; Ganster et al. 2006) and with lyocell reinforcement (Seavey et al. 2001; Franko et al. 2001; Lützkendrof et al. 2000). Currently, the focus is on applications where high toughness is required.

Micromechanics of these fibres and their adhesion with polymer matrices have not been rigorously studied until now. With the aim of assessing their suitability for polymer reinforcement, these fibres were subjected to single fibre tensile testing (Chawla 1998b; Gindl and Keckes 2006) to determine strength, stiffness and elongation. To avoid the slippage in tensile testing, the paper frame set-up, which is also used to test single glass fibres, was adopted. Microbond technique (Miller et al. 1987; Pitkethly et al. 1993) was used to quantify the interfacial shear strength exist between cellulose fibres and polymer matrices. Until now, scanning electron microscopy (SEM) fractographs as quality parameter (Lampke 2001) and interlaminar shear strength (ILSS) as quantity parameter (Chawla 1998a) were used to understand the fibre–matrix adhesion in lyocell composites. In the present work, all three techniques were compared, and conclusions were drawn. Finally, model composites with two kinds of preforms (fibre rovings and nonwovens) were developed and subjected to mechanical testing such as bending, tensile and

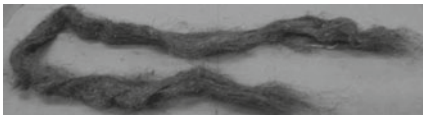
double-notch shear tests. Since these fibres are well suited for toughness applications, damping measurements (Buksnowitz et al. 2007) were also taken on unidirectional (UD) epoxy composites. Hybrid nonwovens consisting of lyocell and natural fibre were used as reinforcements to produce short fibre reinforced composites. Due to the inherent quality differences among natural fibres, both low-quality (flax) and high-quality (hemp, ramie) natural cellulose fibres were selected for comparison. From micro- to macro-level tests, cellulose fibres were compared with glass fibres.

Materials and Methods

Different kinds of cellulose fibres and glass fibres were used for mechanical testing and to manufacture composites. Two different kinds of regenerated cellulose fibres, namely viscose and lyocell, were obtained from Lenzing AG, Austria (Fig. 3). Fibres varied according to their length (staple and filament) and their diameter (9–34 μm). Tire cord viscose filaments were received from Cordenka GmbH, Obernburg, Germany. Flax staple fibres for nonwovens (10–40 μm diameter; 38 mm long) and flax rovings for UD composites (Fig. 3) were received from Holstein Flachs GmbH, Mielsdorf- and Hanf-Faser-Fabrik, Uckermark-Germany, respectively. Rovings of hemp and ramie were supplied by Lotteraner, Vienna, Austria. Rovings of E-glass (2400 tex) were obtained from R&G Faserverbundwerkstoffe GmbH, Waldenbuch, Germany. Nonwoven mats with 100% lyocell, 100% flax, 50% lyocell—50% flax, 75% lyocell—25% flax and 75% flax—25% lyocell were manufactured at Lenzing AG.

Polypropylene (PP) sheets were bought from Goodfellow Cambridge Limited, England, and the melting points of PP were in the range of 180 °C. Epoxy matrix LF, hardener LF1 (pot life of 40 min), active diluent (EPD BD) and degasser were obtained from R&G Faserverbundwerkstoffe GmbH, Waldenbuch, Germany. Two different coupling agents namely maleic anhydride (MAH-63210, Fluka) and polypropylene-*graft*-maleic anhydride (PP-MAH, 426512-250 G, Aldrich) were used to improve the adhesion between cellulose fibres and PP matrix. Accessories needed for the production of epoxy composites such as film release agent PVA, release spray, priming wax, brushes, nonwoven rollers, Teflon rollers, metal disk rollers and solvents for cleaning epoxy residues were obtained from R&G Faserverbundwerkstoffe GmbH.

Lyocell filament and staple fibres



Flax tow (uncleaned)

Fig. 3 Fibres used for single fibre testing and to manufacture composites

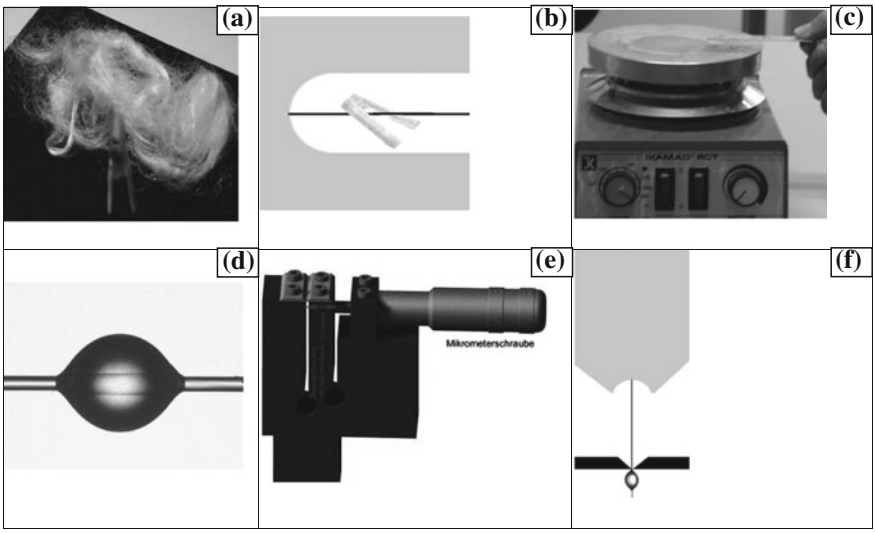


Fig. 4 Steps involved in microbond specimen preparation. **a** Cellulose fibres, **b** thermoplastic trouser lying on single fibre, **c** melting the trouser on heating plate to form the droplet, **d** cured microdroplet, **e** specially developed microvise having inbuilt micrometre to adjust the distance between the knifes, and **f** schematic of specimen prior to microbond test

To make microbond test specimens, fibres were placed on black velvet cloth and tweezers were used to separate the single fibres. Uncrimped single fibres were fixed between two paper frames using "UHU" glue similar to ASTM D 3379-75 (Gindl and Keckes 2006). Using microscope and razor blade, small trousers of thermoplastic films were sectioned and carefully placed on single fibres. Thereafter, the whole specimen was kept above a laboratory heating plate briefly to bond the polymer to the fibre as shown in Fig. 4. Afterwards, the whole specimen was kept in an oven at the melting temperature of the thermoplastic film. In the case of thermosets, a thin metallic rod was used to place the resin on the fibre and left for curing. After curing, the "drop-on-fibre" system was clamped to the upper jaw of the tensile testing machine. Special care was taken to adjust the droplet below the microvise with the help of micrometre as shown in Fig. 4, so that the fibre only passed through the microvise, and not the droplet.

Methods

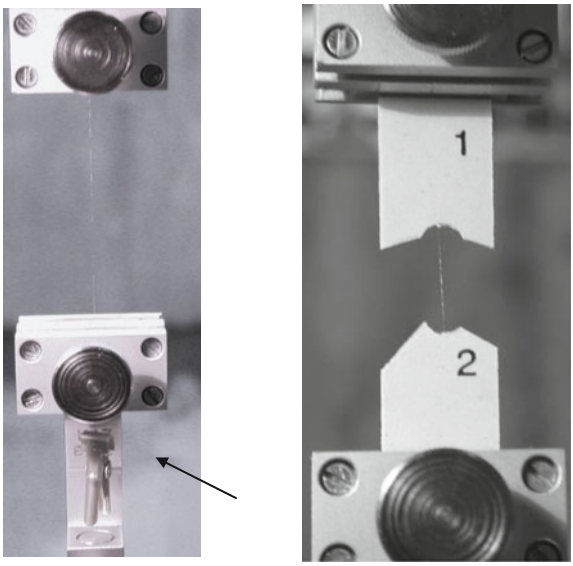
Single Fibre Diameter and Perimeter

It was important to know the diameter and perimeter of the fibre cross section, since these parameters influenced the fibre mechanical properties and fibre adhesion with

the matrix polymer. To obtain diameter and perimeter, fibres were embedded in epoxy resin and cured overnight at 60 °C. By using SiC abrasive paper, the specimens were polished and 1-μm thick fibre cross sections were made by means of an ultramicrotome equipped with a diamond knife. Gentian violet was used to stain the fibre cross sections, and the images were captured in a light microscope equipped with a CCD camera. The fibre diameter was determined from these images by means of image analysis software which fits the fibre cross section with an ellipsis. Since microscopic images were readily available, perimeter and cross-sectional areas were also measured.

Mechanical Testing of Fibres

A full description of mechanical testing is provided elsewhere (Adusumalli et al. 2006a, b). Depending on fibre modulus, two types of single fibre tensile tests are generally carried out. Low-modulus fibres (e.g. textile viscose) are usually tested by direct gripping, and high-modulus fibres (e.g. carbon) are tested by using a carrier like paper frames as shown in Fig. 5 (BISFA 2004; Chawla 1998b; Daniel and Ishai 1994). Details of both tests are presented in Table 1. Since the present investigation involves cellulose and glass (high-modulus) fibres, the paper frame set-up was adopted as discussed in Adusumalli et al. (2006a). A universal testing machine (Zwick/Roell) equipped with a 50 N load cell was used in the paper frame set-up. Specially ordered rubber jaws were used for excellent gripping. At first, any crimps



Direct gripping Paper frame set-up

Fig. 5 Single fibre testing set-up by direct gripping (*left*) and paper frame set-up (*right*). *Arrow* indicates pretension weight, which is used to remove the crimp before testing in direct gripping

Table 1 Two different methods used for single fibre tensile testing

Parameter	Paper frame set-up	Direct gripping
Test standard	ASTM D 3379-75[a] or tabbing technique of ASTM D 3822	ASTM D 3822[b] (gripping the fibre between the jaws)
Test speed	1 mm/min	2 mm/min (E-modulus); 10 mm/min (strength and elongation)
Gauge length	16 mm	10 mm (E-modulus); 20 mm (strength and elongation)
E-modulus	Linear regression	Between 0.2 and 0.5% strain
Pretension weights	-variable-	60 mg/dtex
Climatic conditions	23 °C; 50–65% relative humidity	21 °C; 65% relative humidity

[a]ASTM D 3379-75: standard test method for tensile strength and E-modulus of high-modulus single-filament materials
[b]ASTM D 3822: standard test method for tensile properties of single textile fibres

in the fibre were removed manually, and the fibre was fixed to the paper frames as shown in Fig. 5. Tensile tests were carried out until failure at a cross head displacement rate of 1 mm/min. To obtain a representative set of results, more than 50 single fibres of each type were tested. Tensile strength and failure strains were calculated from the respective maxima in the recorded stress–strain graph. In both tests (paper frame set-up and direct gripping), elongation at break was measured using indirect strain measurement (cross head displacement).

Microbond Testing

The reader is referred to Adusumalli et al. (2006b, 2010a) for detailed understanding. There are several methods available to quantify the interfacial adhesion in composite materials (Drzal et al. 2000; Chawla 1998a; Zhandarov and Mäder 2005). Microbond technique was widely used for measuring the interfacial shear strength (IFSS) between single fibres and polymer matrices. In this test, only a very small amount of the matrix was used in the form of a droplet deposited on the fibre as shown in Fig. 4. Afterwards, this "drop-on-fibre system" was subjected to tensile testing until the fibre was pulled-out of the droplet. Force–displacement curves were recorded and apparent shear strength values was calculated using Eq. (1):

$$\tau_{app} = \frac{F}{\pi d l} \tag{1}$$

where τ_{app} is the interfacial shear strength; F is the maximum load prior to debonding of the fibre; d is the fibre diameter; l is the fibre embedded length.

In microbond technique, the nature of the recorded force curves during the test allows one to distinguish between shear debonding, fibre breakage and slippage of the droplet. It was important to prepare the specimens with the lowest embedded length; otherwise fibre breakage occurs, as opposed to debonding. This problem was more pronounced for low tensile strength fibres such as lyocell and viscose fibres. On average, 8 values were obtained from each fibre–matrix combination. Thorough monitoring of the debonding process with video improved the test efficiency (efficiency defined as test quality and test speed) in our study.

Composite Manufacturing

This technique was used to prepare both unidirectional (UD) composites and nonwoven composites in which epoxy was used as matrix polymer. Since fibre rovings and nonwovens were involved in the present study, compression moulding was chosen as the single manufacturing technique. For UD composites, a special steel mould was constructed to align the fibres before pressing (Fig. 6b2). Fibre

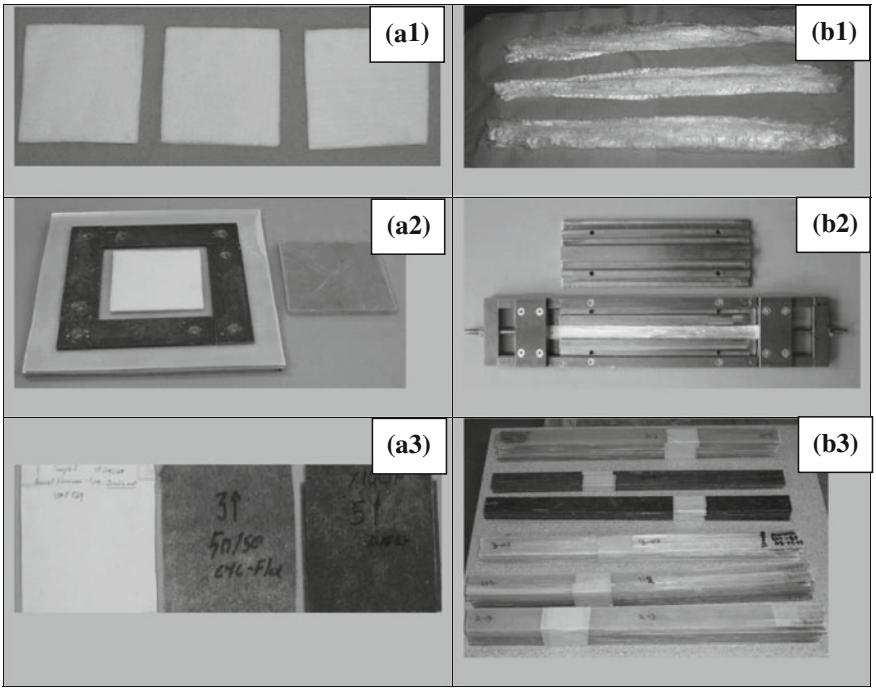


Fig. 6 Different steps involved in composite manufacturing. *Left* Nonwoven epoxy composite; *right* unidirectional epoxy composites). **a1** lyocell nonwoven mats; **b1** lyocell fibre rovings; **a2** and **b2** preforms placed on moulds; **a3** and **b3** composite specimens after machining

bundle rovings were placed in the mould, and ends were fixed in such a way that fibres were aligned parallel. Epoxy resin and hardener were mixed in 100:40 ratios (weight). To decrease the resin viscosity, 5% (volume) diluent was added and the whole mix was preheated to 50 °C. Fibre rovings were slowly impregnated with the resin, and parallel metal disk rollers were used for de-airing and to compact the roving fabric. Thereafter, composites were pressed at 80 °C and 18 bar pressure for two hours. Final composite dimensions were 300 × 20 × 2 mm^3 in which a fibre content of 55% (volume) or 67% (weight) was maintained. Priming wax as first layer and film release agent PVA as second layer were applied on steel moulds after each and every demoulding. Before the fresh coat of PVA, moulds were mechanically cleaned.

Nonwoven epoxy composites were prepared using a special aluminium mould (Fig. 6a2). From the needle punched direction, 10 × 10 × 4 mm^3 sized nonwoven mats were cut and impregnated with the epoxy resin which was used earlier to make UD composites. Nonwoven rollers with short bristles were used for better impregnation. De-airing and optimisation of the resin content were carried out using teflon rollers. Final thickness of the composite was 0.4 mm, and fibre content was 67% (weight). Composites were pressed at 80 °C and 7 bar pressure for one hour. Release spray was applied on upper and lower moulds before each cycle to ease the demoulding process. Butyl acetate was used as cleaning agent for tools used in manufacturing epoxy composites.

Mechanical Testing of Composites

UD composites were subjected to static bending, static tensile, shear and damping measurements. In the case of hybrid nonwoven composites, only tensile tests were performed. In the damping (logarithmic decrement—Λ) experiment, specimens were clamped on one end. The other free end was subjected to mechanical vibrations, and the resulting amplitude–time signals were captured by a laser device as shown in Fig. 7b. Damping or damping capacity was measured according to Eq. (2):

$$\Lambda = \ln \frac{x_q}{x_{q+1}} \tag{2}$$

where Λ is the logarithmic decrement; x_q is the amplitude q; x_{q+1} is the amplitude $-q + 1$ (directly following x_q).

Similarly, damping can also be measured using dynamic mechanical thermal analysis, where the ratio of the loss modulus to storage modulus is defined as damping (loss tangent—tan δ), and the relationship between the logarithmic decrement and the loss tangent is $\Lambda = 2\pi \tan \delta/2$ (Buksnowitz et al. 2007). Both damping and tensile modulus were measured on full-length specimens; afterwards, specimens were machined (145 × 20 × 2 mm^3) for three point bending, shear and

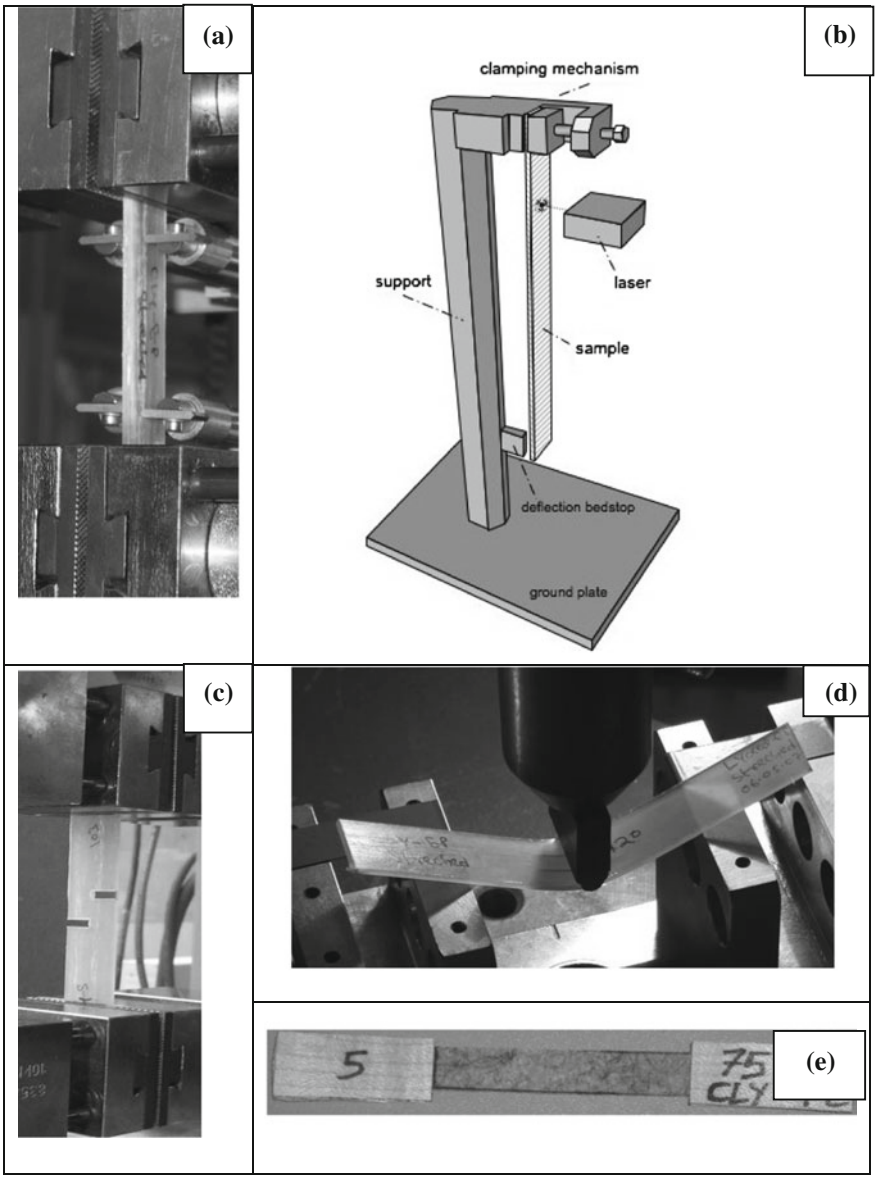


Fig. 7 Mechanical testing of composites (**a** measuring tensile parameters using mechanical extensometers; **b** longitudinal damping measurement; **c** double-notch shear test; **d** three point bending test; **e** specimen for tensile testing)

tensile tests (Fig. 7) using a fine band saw. Span length was 70 mm for bending test. For the shear test, 2 mm width notches were made with the distance of 10 mm (Fig. 7c). The distance between the notches and the specimen thicknesses were

Table 2 Different standards used for composite testing

Composite type	Standards	References
Tensile testing of unidirectional composites	ASTM D 3039/D 3039 M-93	Herakovich (1998)
Three point bending test of unidirectional composite	ASTM D 790	Arib et al. (2006)
Double-notch shear test of unidirectional composite	ASTM D 3846-85	Ünal and Bansal (2000)
Damping measurement of unidirectional composites	DIN ISO 6721-1 and DIN 6721-3	Buksnowitz et al. (2007)
Tensile strength and modulus of nonwoven composites	DIN EN ISO 527	Wielage and Leonhardt (2003)

used as input parameters to calculate the ILSS. Composite tabs were used as grips for tensile measurements. The cross head speed was kept at 1 mm/min (tensile and shear) and 40 mm/min (bending). All mechanical tests were carried out similar to the standards described in Table 2. On average, 8 specimens were tested for each mechanical test.

Nonwoven composites were subjected to tensile testing with a cross head speed of 1 mm/min. Specimens for tensile testing were prepared using special wood tabs consisting thermoplastic adhesives as shown in Fig. 7e. Specimens were prepared in such a way that the needle punched direction corresponded to the tensile direction. Mechanical extensometers were used for elongation measurements. Tensile modulus was obtained by linear regression, and strength was measured from the stress–strain diagram.

Results and Discussion

Single Fibre Properties

Microscopic images of regenerated cellulose fibres and natural cellulose fibres are shown in Fig. 8. The images emphasise their different cross-sectional geometry with respect to the cylindrical geometry of glass fibre (not shown). Flax and hemp, being cellulosic bast fibres, showed polyhedral cross sections consisting of a thick cell wall and a central cavity, the cell lumen as presented in the literature (Charlet et al. 2007). Circular appearance of lyocell fibres resembled glass fibres. Inhomogeneous cross sections and formation of fibre bundles characteristic for natural fibres were clearly shown in Fig. 8. Climate, retting degree and fibre damaging due to scutching and carding are some of the reasons for such variability (Mohanty et al. 2005). Fibre diameters were measured by fitting the fibre cross section with an ellipsis. The significant heterogeneity found in natural fibre cross

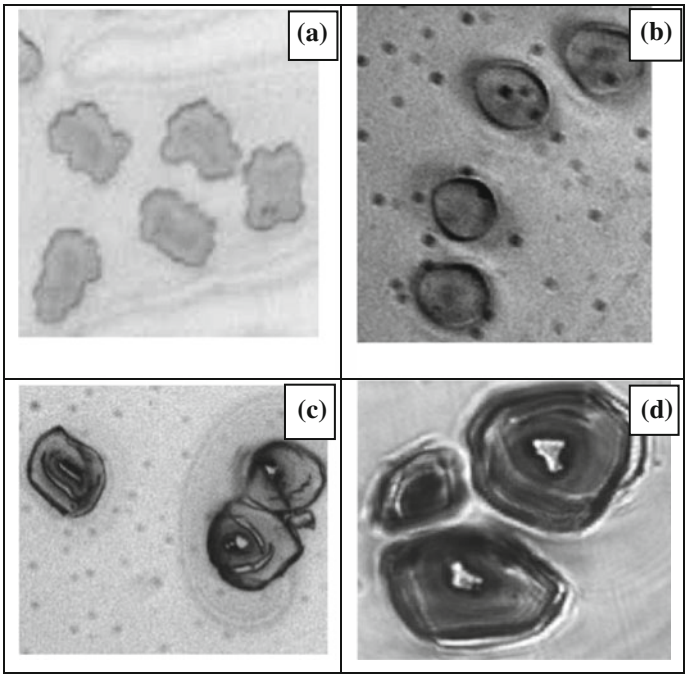


Fig. 8 Single fibre cross sections of viscose (**a**), lyocell (**b**), flax (**c**) and hemp (**d**). Here viscose fibre is shown for comparison. Epoxy is used as embedding medium

sections was also observed in their diameters which lead to inhomogeneity in their mechanical properties as shown in Fig. 2. Single fibre tensile properties measured using paper frame set-up and direct gripping are compared in Fig. 9 for regenerated cellulose fibre. Regarding tensile strength, a good agreement between paper frame set-up and direct gripping was achieved. On average, slightly higher tensile strengths were measured using direct gripping compared to paper frame set-up. It is apparent that elastic moduli measured by direct gripping were systematically lower than values measured by paper frame set-up. The reason for this significant difference may be found in the method of strain measurement and specimen preparation. Substantial systematic differences were also found between two methods in failure strain measurements. Since paper frame set-up measurements were validated with video extensometry (Adusumalli et al. 2006a) and found to be accurate, it seems that direct gripping overestimates failure strain thereby underestimating the E-modulus. This could be due to the fibre slippage in direct gripping.

Since regenerated cellulose fibres are crimped (stuffer box), specimen preparation for mechanical testing was always an important step. In direct gripping, crimps were removed before the tensile test using specially developed pretension weights. But in the paper frame set-up, crimp was removed manually before fixing the fibre to the paper frame. It is likely that the manual crimp removal can induce unwanted prestretching in regenerated cellulose fibres. This prestretching could also account

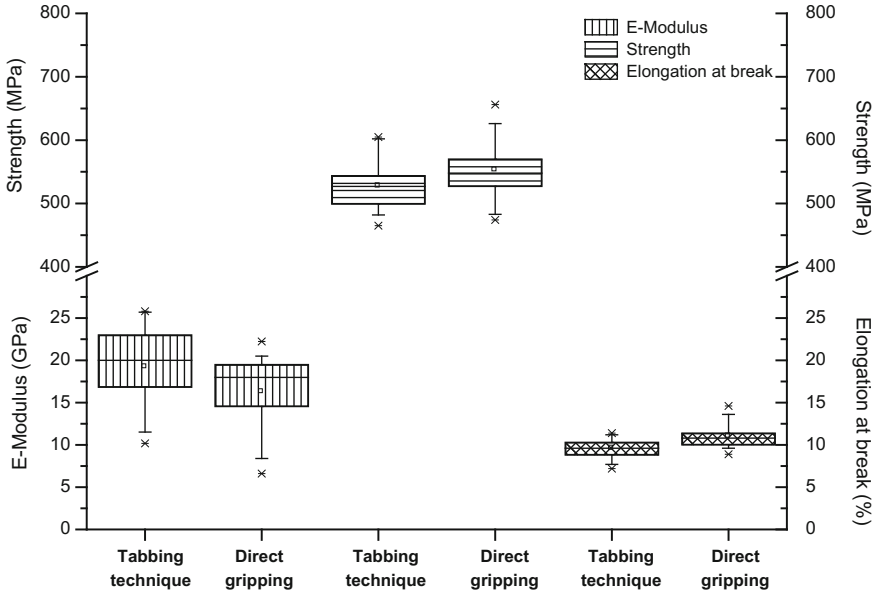


Fig. 9 Comparison of single fibre tensile properties obtained from two different methods, direct gripping and paper frame set-up (tabbing technique) for a regenerated cellulose fibre

for the observed high E-modulus and low elongation for the paper frame set-up shown in Fig. 9. Thus, care should be taken with low-modulus (textile) fibres while using paper frame set-up, which was originally developed for single fibre testing of high-modulus fibres (Chawla 1998b).

In the present work, the paper frame set-up was successfully applied to measure the tensile properties of glass, flax, hemp and series of regenerated cellulose fibres. The obtained results were compared with the literature data and found accurate (Lützkendorf et al. 2000; Wielage and Leonhardt 2003). All single fibre specific tensile properties of fibres are displayed in Fig. 10. Single lyocell fibres revealed better strength and stiffness values compared to standard viscose fibres, but their strength values were slightly lower than rayon tire cord filaments. Glass is superior to all cellulose fibres even when its high density is taken into account. Elementary flax fibre, as discussed earlier, revealed better properties than lyocell, but variability is very high as shown in Fig. 10. This could be due to the higher degree of polymerisation (DP) of cellulose polymer characteristic of native cellulose exists in flax. Both DP and alignment are seems to be low for regenerated cellulose fibres (Woodings 2001).

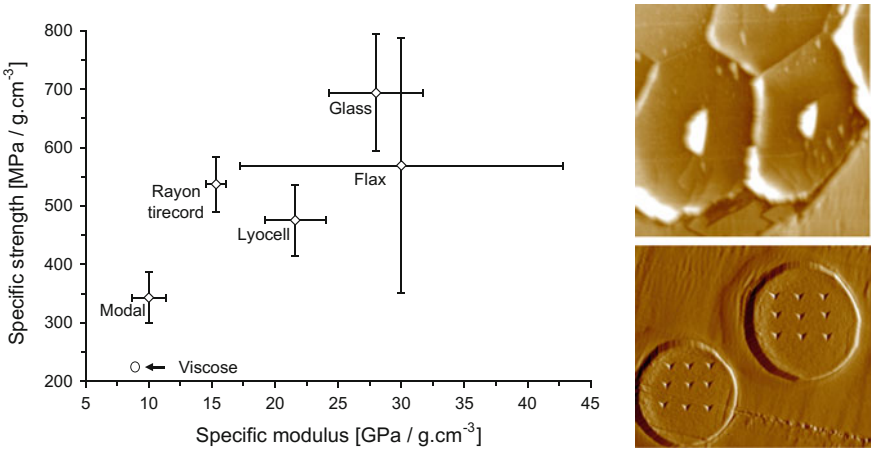


Fig. 10 *Left* specific properties of single fibres obtained from paper frame set-up. *Right-top* flax fibre bundle having lumen at centres. *Right bottom* two lyocell fibres having no lumen (ignore the small nanoindentation imprints visible on the surface)

Microbond Technique

Microbond technique was employed to measure the shear strength in single fibre composites. Shear strengths between cellulose fibres and two different polymer matrices namely PP and epoxy are summarised in Table 3. Direct comparison was made between glass, natural cellulose fibre (ramie) and regenerated cellulose fibre (lyocell). The Ramie fibre showed higher IFSS than lyocell presumably due to the higher surface roughness. In Adusumalli et al. (2010a), this study was extended to fibre modification with maleic anhydride (MAH) and its influence on adhesion with PP matrix. It is found that a chemical modification (marination) of man-made cellulose fibre with 2.5 wt% MAH resulted in a twofold increase in IFSS with a PP matrix, with no reduction in fibre mechanical properties (Fig. 11). This is attributed to the following two reasons: i) the chemical coupling of MAH with a certain amount of lyocell resulting in a lyocell-MAH phase and ii) the remaining free MAH which was adsorbed on the lyocell surface providing reactive sites to form MAH *grafted* PP phase. The consequence of this being the increased compatibility and interfacial affinity between the lyocell-MAH phase and MAH *grafted* PP phase. Similarly higher value of IFSS was obtained for lyocell modified with MAH-PP coupling agent as shown in Table 3 and this is attributed to increased interfacial compatibility. This increase in IFSS was also confirmed by the change in force–displacement plots and change in the slope of the relationship between the debonding force vs embedded length. In Adusumalli et al. (2010a), the adhesion of fibres with PP and epoxy was compared. Thermoplastics revealed low IFSS values in the range of 3–9 MPa whereas thermosets revealed good adhesion with cellulose fibres, with IFSS values ranging from 15 to 20 MPa.

Table 3 Compilation of IFSS results obtained from microbond technique for fibre–matrix combinations

Fibre/matrix	Interfacial shear strength (MPa)	
	PP	Epoxy
Lyocell	5.3 ± 1.0	14.8 ± 1.3
Lyocell-MAH-PP	8.1 ± 0.8	14.0 ± 2.5
Natural cellulose fibre	5.9 ± 1.1	21.2 ± 3.9
E-glass	6.7 ± 2.0	36.5 ± 2.9

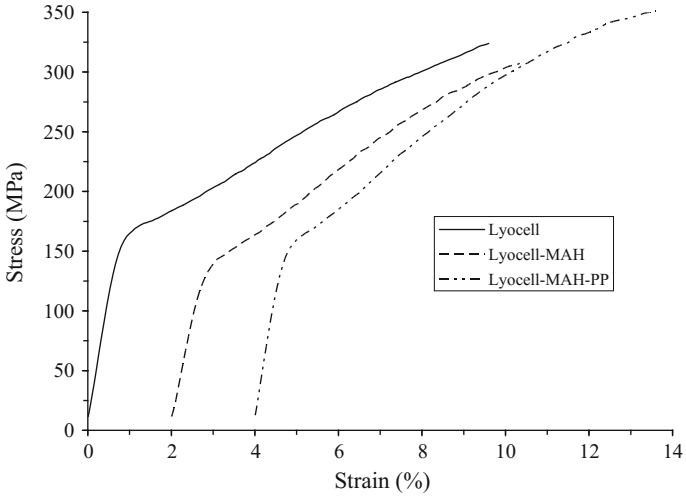


Fig. 11 Stress–strain diagram of modified and unmodified lyocell fibres (diameter of 30 μm) obtained from single fibre tensile testing. For clarity, curves are offset by 2% strain

In Adusumalli et al. (2010b), the applicability of microbond technique for regenerated cellulose fibres was discussed. Due to the low tensile strength of cellulose fibres compared to glass, the IFSS values were underestimated for lyocell and modal. This is due to the overlapping of the breaking force and the debonding force. To avoid such overlapping, drops with very low embedded length are needed, which are difficult to test using the present equipment. So a second approach has been followed to recognise the debonding force which is not overlapping with the breaking force. Contact angle of "drop-on-fibre system" were compared with the IFSS values in Adusumalli et al. (2010b). Since it is obvious that drop with low contact angle reveals high IFSS, an exponential relation was found between IFSS and contact angle for tested fibre–matrix combinations. The relation between IFSS and contact angle together with force–displacement plot was used to find the accurate debonding forces in microbond test.

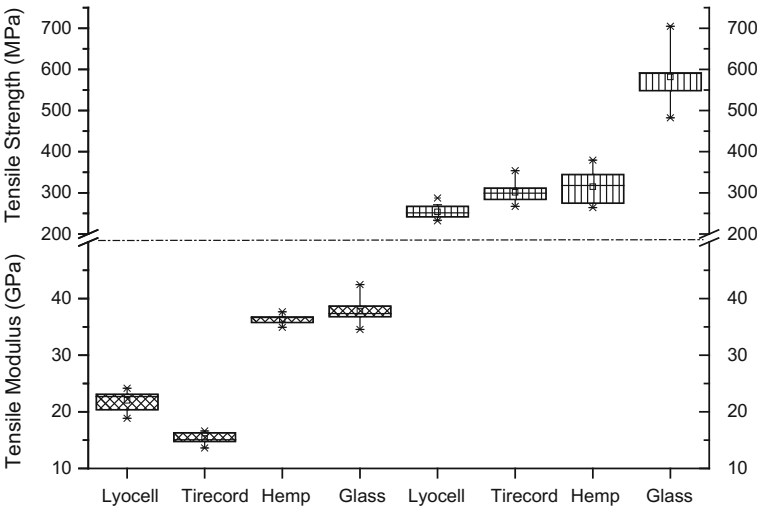


Fig. 12 Tensile strength (*top*) tensile modulus (*bottom*) of unidirectional epoxy composites. Tirecord refers to another variety of viscose fibre

Mechanical Properties of Composites

Tensile results of unidirectional (UD) composites are presented in Fig. 12. Regarding tensile modulus of UD composites, lyocell surpasses the tire cord, but tire cord composites were superior to lyocell composites in yield strength and ultimate tensile strength. Hemp composites had a 60% higher modulus than man-made cellulose composites and even surpassed the glass composites on weight basis. However, it is important to note that the hemp was a high-quality fibre tow with low impurities. The yield strength of lyocell–epoxy is 100 MPa, which is equivalent to 1/3 of the yield strength of hemp–epoxy, but the elongation at break of man-made cellulose composite is 200% higher than hemp composites. Bi-phasic (elastic and plastic) behaviour was observed in all tested composites except hemp–epoxy. The reason could be the low fibre length and the low elongation at break (due to the presence of lignin) of hemp compared to other fibres.

For SEM analysis, samples were sputter coated with Au/Pd for 60 s and analysed in a Hitachi S-4000 SEM with an acceleration voltage of 5 keV. SEM fractographs of tensile specimens were displayed in Fig. 13. Fibre pull-out in fracture surfaces is considered as one of the adhesion parameters in composite materials. The adhesion between fibre and matrix seems to be very good in lyocell–epoxy, because of its low fibre pull-out on the fracture surface (simultaneous fracture of fibre and matrix). In hemp–epoxy, surface of the fracture was uneven with fibre pull-out; however, the bonding was strong because polymer residues were visible on the pulled-out fibres (not shown). In addition, a clear fracture of fibres together with the surrounding polymer was also observed indicating the strong adhesion within the hemp fibre bundlles (Fig. 13).

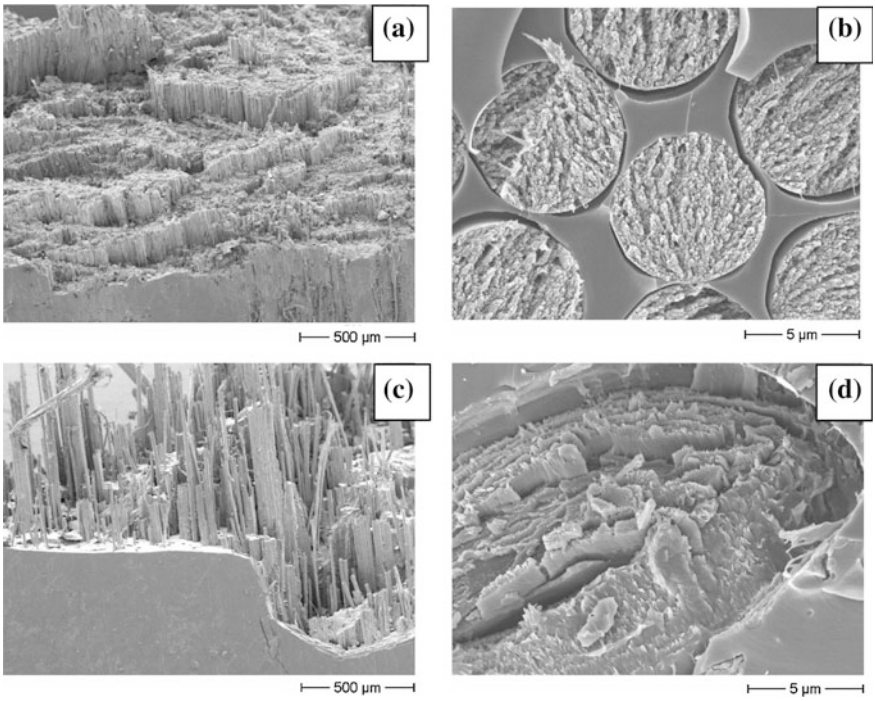


Fig. 13 SEM fractographs of lyocell (**a**, **b**) and hemp (**c**, **d**) unidirectional epoxy composites

The results of the double-notch shear test presented in Fig. 14 are quite matching with the macro-level adhesion studied by SEM fractographs. ILSS of 37 MPa was measured for lyocell–epoxy and glass–epoxy. Since glass fibres are surface coated with sizing agent, the high ILSS is inevitable. But the similar ILSS measured for unsized lyocell tow and low fibre pull-out observed in the SEM fractographs of lyocell specimens, together indicate a strong bonding between fibre and matrix. The low ILSS of hemp–epoxy compared to lyocell–epoxy supports the severe fibre pull-out observed in SEM fractographs of hemp specimens. A direct comparison between microlevel adhesion (IFSS) and macro-level adhesion (ILSS) is also presented in Fig. 14. A strong correlation was observed between micro- and macro-values except for lyocell–epoxy. It is likely that IFSS values were underestimated for lyocell–epoxy due to the tendency of the lyocell fibres exhibiting necking in the vicinity of the droplet.

The three point bending results of UD composites are presented in Figs. 15 and 16. The bending properties of low-quality flax composites were slightly lower than lyocell–epoxy, which were lower than high-quality hemp composites. Although the average properties of hemp fibre composites are excellent, variability in its mechanical properties is high, thus composite design will need to address this problem. In contrast, lyocell composites revealed low variability in their mechanical

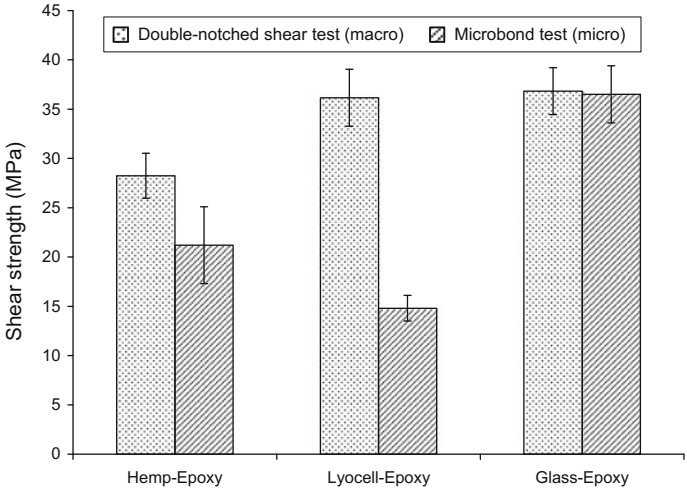


Fig. 14 Shear strength of single fibre composite and unidirectional composite

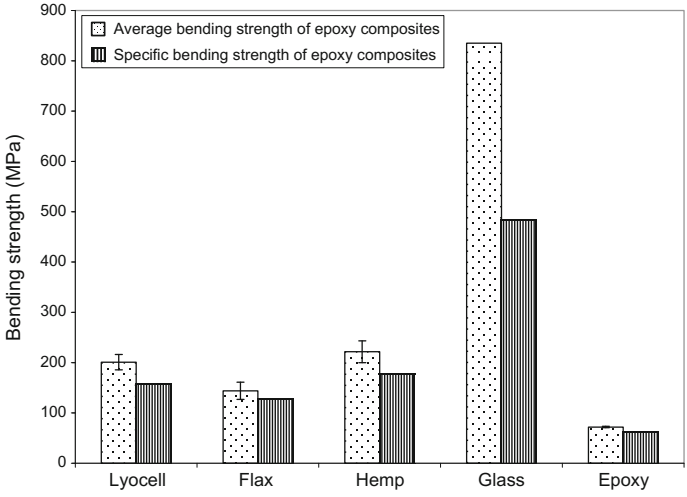


Fig. 15 Three-point flexural strength of unidirectional epoxy composites

properties (bending and tensile) which is an obvious advantage for regenerated cellulose fibre composites compare to natural cellulose composites.

When flexural properties of cellulose-based composites are compared to glass composites, the difference in density between the two composites should be taken into account. While glass composites had a density of 1.73 g cm^{-3}, the density of cellulose composites was typically only 1.25 g cm^{-3}. Figures 15 and 16 show the result of flexural properties on weight basis for the composites tested in our study.

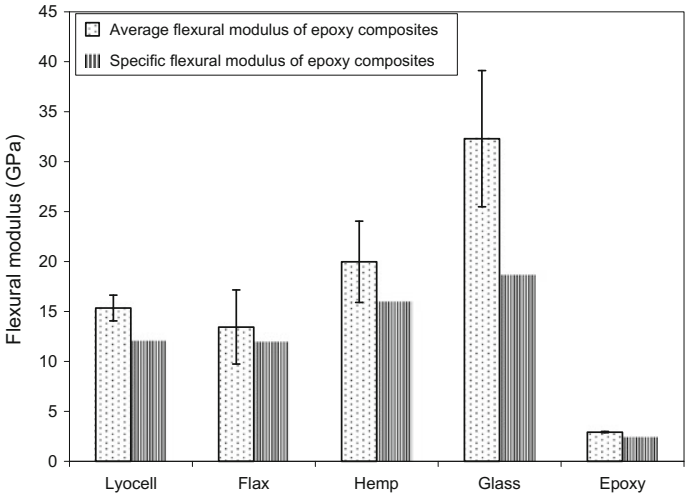


Fig. 16 Three-point flexural modulus of unidirectional epoxy

The specific flexural strength of glass composites was three times higher than that of cellulose composites, and the specific flexural modulus of glass composites was 1.5 times higher than that of cellulose fibre composites. The elongation at maximum force was very high for lyocell–epoxy (5%) followed by glass–epoxy (3.5%), hemp–epoxy (3%) and flax–epoxy (2%).

To understand the reduction in flexural properties of flax–epoxy, tensile specimens were studied under SEM. Despite strong bonding between flax and epoxy (Fig. 17, low fibre pull-out and fill up of lumen with the polymer), it revealed lower flexural properties than hemp–epoxy. Uncleaned flax tow (high content of shives and dust) caused quite a number of air bubbles and foreign substances in the composite as shown in Fig. 17 (white arrows). The formation of such air bubbles could be the reason for the observed reduction in flexural properties of flax–epoxy.

No such air bubbles were found in hemp composites, since cleaned hemp tow (high quality) was used. But the matrix penetration into the lumen of thick flax fibres as shown in Fig. 17 (right) could explain the reason for the high IFSS values and low contact angles observed in the microbond test.

Since fibres are randomly oriented, the tensile strength and modulus of composite reinforced with nonwoven mats were half the values observed for UD composites (Fig. 18). Lyocell–epoxy composites revealed 20% lower tensile strength and E-modulus than flax–epoxy. The positive effect of hybrid reinforcement was partially observed in tensile results. An increase in tensile modulus was observed with respect to an increase of the flax content in the lyocell nonwoven mat. This could be due to the excellent modulus of flax fibres. Similar results were published by Lützekdorf et al. (2000). They reported slightly higher values than the results presented in Fig. 18 for the same matrix.

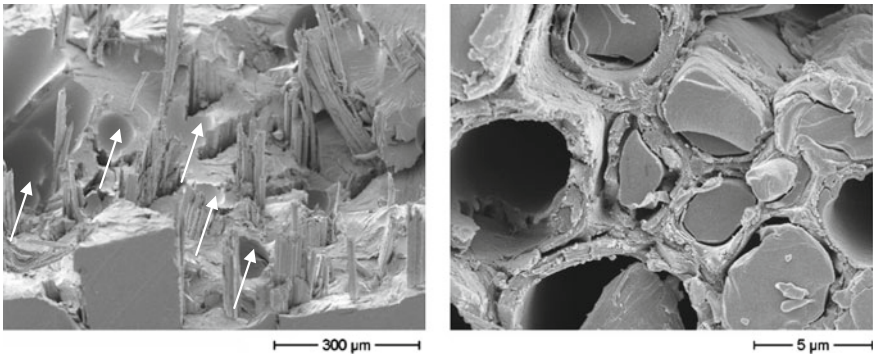


Fig. 17 SEM fractographs of flax–epoxy tensile specimens. The *arrows* in *left* image indicates regions of poor bonding due to the presence of foreign substances and air bubbles

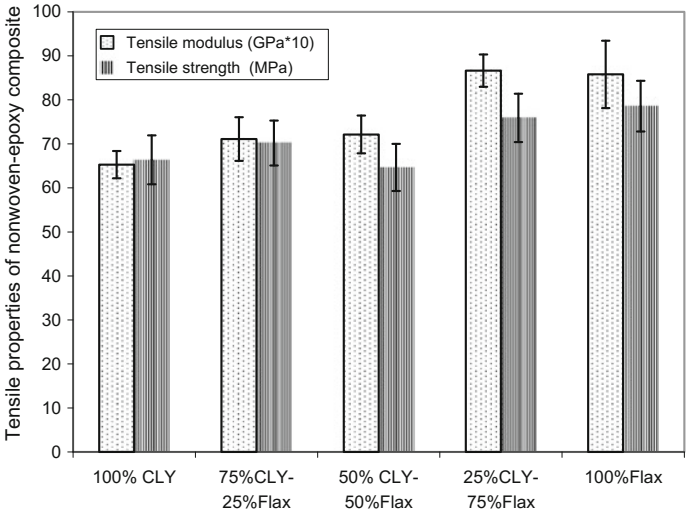


Fig. 18 Tensile properties of short fibre reinforced epoxy composites (CLY = Lyocell)

Damping results of UD epoxy composites are displayed in Fig. 19. Since moderate interfacial bonding and presence of amorphous phase in lyocell corresponds to superior impact properties and increased damping. Hence, the obtained higher damping value of 0.067 for lyocell–epoxy was not surprising. The low damping value of glass–epoxy supports the high IFSS value measured from microbond technique. It can be concluded that the energy dissipation was very high in cellulose–epoxy composites due to the low modulus of cellulose fibres and their composites compared to their glass counterparts. Among cellulose fibres, lyocell composites revealed little higher damping values than hemp composites again due

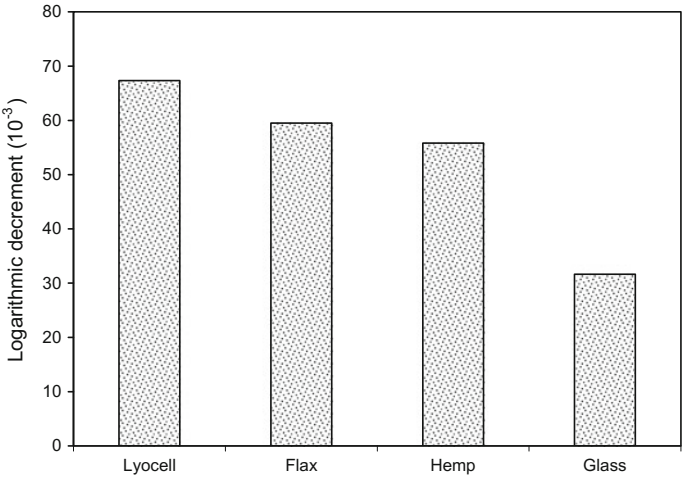


Fig. 19 Mechanical damping values of unidirectional epoxy composites

to the low IFSS value and high elongation at break of lyocell composites than hemp composites. These results indicate that lyocell composites have good structural damping properties and impact behaviour which could be used to make components in sound proof construction materials, transportation and machinery industries.

Conclusions

- Single fibre tensile tests were conducted on regenerated cellulose, natural cellulose and glass fibres by employing both methods of direct gripping and paper frame set-up.
- Microbond tests were conducted on regenerated cellulose fibre, natural cellulose and glass fibre composites with both PP and epoxy matrices in order to determine interfacial shear strength (IFSS). Cellulose fibres revealed good adhesion with thermoset matrices, but with thermoplastics maleic anhydride modification is necessary for the better adhesion.
- Tensile test, double-notch shear test, flexural tests and mechanical vibration tests were conducted on cellulose and glass fibre composites. The tensile strength of regenerated cellulose fibre composites must be improved by a factor of 2 in order to attain glass fibre composite values.
- Regenerated cellulose fibres (Lyocell) composites are ideally suitable for semi-structural applications (e.g. automobile components) due to properties such as moderate specific modulus, moderate interfacial shear strength, high damping capacity and high elongation at break compared to glass composites.

Acknowledgements This research would not have been possible without the financial support provided by the Austrian government, the provinces of Lower Austria, Upper Austria and Carinthia, as well as by the Lenzing AG. I would also like to express my sincere appreciation and gratitude to Prof. Herbert Sixta and Dr. Hedda Weber, Dr. Thomas Roeder for their consistent support and encouragement.

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Adhesives of Bio-origin for Wood Composites

D. Sujatha, S.K. Nath and B.S. Mamatha

Abstract Phenol formaldehyde (PF) resin has remained as an absolute resin for making waterproof exterior-grade plywood since the discovery of its use as wood adhesive. Phenol is synthesized from petroleum and natural gasderived chemicals. Hence the prices of the phenolic resin are directly dependent on the fluctuation of the petroleum prices. Materials which can replace the phenol either partially or wholly in the phenolic resin would have a significant role on the cost. The renewable materials should match the reactivity, applicability and the bonding performance of the PF resin. In a bid to become more environmentally sustainable, Indian Plywood Industries Research and Training Institute (IPIRTI) has developed adhesives using biomaterials of natural origin which are alternatives to commonly used petroleum-based wood adhesives. Considering that adequate supply of formaldehyde will be available in future, the only concern for the resin manufacturer is the long-term regular supply of phenol at a reasonable price. Some of the biomaterials resemble phenol in their molecular architecture units and they can undergo all the chemical reactions as similar to phenol in cross-linking. These biomaterials are available on a renewable basis and address the issues on the scarcity of phenol or the petroleum resource. Natural materials, such as cashew nut shell liquid, tannin, lignin, defatted soya, have been tried as a substitute for phenol in the development of PF resin for panel products. In this paper, the results on work carried out by Indian Plywood Industries Research and Training Institute (IPIRTI), Bengaluru, on the development of phenolic resin adhesives by substituting phenol with materials of bio-origin have been summarized. These materials are made to partially replace phenol in phenolic resin of exterior grade and have been used to manufacture boil water-resistant-grade plywood. These are comparatively cheaper, indigenously available from natural renewable resource and also help in minimizing the pollution. It has been observed that 30–40% replacement of phenol in phenolic resin by these biomaterials can yield satisfactory bond quality as per relevant specification. Due to the natural origin of the materials used, the cost of the raw

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material is cheaper which in turn helps in cost reduction of the phenolic resin adhesive by 20–30%.

Keywords Panel products · Phenol formaldehyde · Renewable · Wood

Introduction

The main types of synthetic resin adhesives required for the manufacture of panel materials are urea-formaldehyde (UF) and phenol formaldehyde (PF). The panels produced from UF resins are generally used for interior purposes and that from PF resin are used for exterior purpose. The main raw materials for UF and PF resin are urea, phenol and formaldehyde. Long-term supply of phenol is less assured since the raw material used for the manufacture of PF resin is increasingly in demand for other uses. Increase in price and doubts about future supply of the above materials and environmental concerns due to use of these synthetic materials have revived interest in the search for biomaterial-based substitutes for partial or total replacement of synthetic resins used in adhesive manufacture.

Research & Development of adhesive based on renewable resource has always been the desire for adhesive self-sufficiency. The additional factor that creates interest in new adhesives is a desire of the panel industry to decrease its dependence on petroleum-based adhesives especially phenolic, the price of which keeps fluctuating in increasing order day by day.

Phenol which is the basic raw material for PF resin is derived from benzene, a building block derived from petroleum. Some of the biomaterials resemble phenol in their molecular architecture units and they can undergo all the chemical reactions as similar to phenol in cross-linking. These biomaterials are available on a renewable basis. The scarcity and the fluctuating price of phenol have revived interest in the search for biomaterial-based substitutes for partial or total replacement of synthetic resins used in adhesive manufacture.

During the manufacturing of paper, the paper and pulp mills discard the slurry containing lignin, viz., black liquor. As the presence of lignin in paper manufacturing intervenes with the quality of the paper, the lignin is removed out from the mixture of cellulose, hemicelluloses during pulping. The black liquor is discharged as waste in the paper and pulp mills and leads to serious problems related to the discharge of residual liquids. From these discarded waste of the slurry, lignins and their derivatives can be isolated and can be used for many end-use applications. The main sources of lignin derivatives are black liquors from pulping process.

The major source of industrial lignin is pulp and paper industry. During pulping, the lignin macromolecules are degraded and modified. In sulphite process, degraded lignin fragments called ligno-sulphate are of rather high molecular weight but kept in solution by sulphonic acid groups introduced during pulping. During alkaline pulping, lignin is extensively modified and cleavage of alkyl aryl ether linkages in lignin takes place resulting in the formation of phenolic group rendering the lignin

fragments water soluble. Lignin can be recovered from spent pulping liquor simply by lowering the pH of the liquor.

Recently, lignin fragment with high number of reactive sites in C9 unit has been obtained from industrial pulping of sugarcane bagasse. The resulting by-product lignin contains a high number of approximately 0.7 unsubstituted 3 and 5 positions on phenolic C9 unit. The value is about twice as high as that of other industrial lignin.

Substitution of phenol in PF wood adhesive is certainly the most widely explored avenue of lignin utilization. Lignin–PF formulations have been used in manufacturing particle board, plywood and fibre board. Under certain conditions, up to 40% of phenol can be replaced by ligno-sulphate or black liquor for the manufacture of phenolic resins without deteriorating bond properties.

Wattle tannin is water soluble, renewable from an 8-year tree rotation resource. It requires less cross-linking agent. They have a molecular weight range of 300–3000 with increased glue-line brittleness. Comparing with pine tannin, wattle tannin is slower reacting tannins. Mostly preferred are slower reacting wattle tannins as it will be easy to control the reaction according to industrial situations.

Reactivity of tannin when extended in phenol formaldehyde resin is high. This helps in one way to use the high moisture content veneers for bonding higher-grade panels. When high moisture content veneers are coated with tannin extended phenol formaldehyde resin adhesive, the high moisture present in the veneers tends to decrease, due to the fast curing action of the tannin extended resin adhesive. This in turn helps to maintain the glue-coated veneer moisture content level to that of the conventional PF resin-coated veneers, thereby giving good bond quality.

Soya-based glues were popular in the early twentieth century and worked well in plywood panels, as long as they were kept dry. The poor water-resistant property of the soya-based adhesives limited their use to internal applications. These adhesives were limited in their industrial applications, due to their cost and poor wet bond performances. The poor bond durability of soya adhesives is due to limited amount of cross-linking in final polymer in resin. Soya bean is available commercially as defatted soy flour (SF), soy protein isolate (SPI) and soy protein concentrate (SPC).

Cashew nut shell liquid (CNSL) is one of the sources of naturally occurring phenols. Anacardic acid and cardol are the main constituents of CNSL. The approximate ratio of these constituents is 90:10. Cardanol is the product obtained by distillation of commercial CNSL. Cardanol is a mixture of monohydroxy phenol with a linear side chain in the meta-position. The side chain contains on an average two double.

Research on wattle tannin-based adhesives started in the 1950s with the initial studies conducted by Dalton (1950, 1953). Subsequent work by Plomley (1959, 1966) has reported that wattle-bark tannins are suitable raw materials for plywood and particleboard adhesives production. However, tannin use has been limited due to its high reactivity and viscosity which results in quick curing mechanism and leads to a short pot life of the adhesive. Lack of intermolecular cross-linking causes weak adhesive bonds (Pizzi 1978). Alkaline or acid hydrolysis of the high

molecular weight gums and use of simple aromatic molecules as hydrogen bond breakers will decrease the viscosity of a tannin solution.

Forss and Fuhramann (1976) have reported that high molecular weight lignin derivative from spent sulphite liquor or black liquor could be used as a substitute for phenol in the manufacture of PF resin by copolymerization replacing 40% of the resin in the glue mix without undue effect on wood failure or durability. Dolenko and Shields (1980) have used methylolation of Kraft lignin (MKL) with suitable phenolic resin adhesive and succeeded in replacement of 70–80% of the petroleum-derived PF resin component with lignin. Investigations have also been reported on the use of 100% ligno-sulphonate-based adhesives especially for particle board (Shen 1974) and wafer board (Shen 1977; Shen and Calve 1980). Shiaishi and Kishi (1986) have investigated the possibility of dissolving the whole wood substance in phenol. It has been reported in their work that phenolation occurs with an indication that phenol bonds to lignin and then produce resol-type resins suitable to bond southern plywood. In their research, the phenolation is carried out by wood meal liquification in phenol in the presence of an acid. However, the results on bonding properties have not been indicated. Studies by Narayanamurti et al. (1962) on the substitution of phenol with lignin-rich materials and acid hydrolysed wood which is enriched in lignin have resulted in replacement of 30–60% of phenol in PF resin adhesives. Lignin precipitated from black liquor (Jain et al. 1965), a rich and readily available source of lignin for developing resins has shown that phenol could be replaced up to 60%. George et al. (1977) have reported that sodium lignin sulphonates, derived from Ca sulphite liquor, could be used as an extender or substitute for phenolic resin to the extent of 20% to bond plywood when hot pressed at a temperature of 140 °C. Physical–technological properties of the plywood bonded with these resin adhesives were found to be fully equivalent to those of plywood bonded with phenolic resin adhesives.

Jain et al. (1965) have discussed the use of lignin precipitated from black liquor. Lignin-based PF resins were prepared by Khan et al. (2004) using different agricultural wastes. It was found that 50 wt% of phenol can be substituted by lignin to give modified lignin phenol formaldehyde resin with better bonding strength compared to control PF resin. Thermal studies revealed a lower curing temperature for LPF resin than PF resin. However, thermal stability was found lower than control PF resin. The early soya-based adhesives were made by extending the protein with amino resins (Sarkar et al. 1985). During the early twentieth century, the soya-based glues were popular and were adopted for making plywood panels. The panels made yielded good bond quality only when kept in dry condition. However, when immersed in water, the panels indicated poor water-resistant property. Due to poor water-resistant property of the soya-based adhesives, the application was limited to internal applications (Lambuth 2003).

Soya-based adhesives have been investigated extensively (Wescott et al. 2006; Frihart et al. 2007; Allen et al. 2010). Sarkar et al. (1985) and Zoolagud et al. (1994) have reported that the dispersion of soya flour in liquor ammonia or sodium hydroxide for extending UF resin yields better adhesive property. Zoolagud et al. (1994) have reported that extending PF resin within 10% de-oiled soya flour using less percentage

of sodium bisulphite as dispersion agent has yielded good bond quality. More than 10% extension resulted in poor bond quality.

Phenol Lignin Formaldehyde Resin (Lignin Slurry/Black Liquor)

The lignin in the industrial black liquor obtained after the digestion of wood was fractionated by membrane separations and was used in development of phenolic resins by partial replacement of phenol.

Alkali–lignin as obtained from the paper mill was diluted to 20% solid content by adding requisite quantity of water. No alternation in the composition of the black liquor was made. Ultra-filtration equipment containing ceramic filter tubes was used for fractionation of the lignin in black liquor. By ultra-filtration employing ceramic-based membranes having molecular weight cut-off 0–5000, 5000–10,000, 10,000–20,000 and >20,000, fractions of different molecular weight of black liquor were obtained. Various fractions obtained in aqueous solution contained about 20% of solid. These slurries were neither concentrated nor diluted but used as such as phenol replacement.

Materials of different fractions obtained after ultra-filtration were made to replace phenol partially in phenol formaldehyde resin and have been used to manufacture boil water-resistant plywood. The phenol content in the lignin in the slurry was determined, and the formaldehyde requirement for polymerization was calculated. 20–30% replacement of phenol by black liquor of molecular weights 5000–10,000 and 10,000–15,000 in phenol formaldehyde resin has yielded good adhesive properties. Accordingly, formulations for lignin–phenol–formaldehyde were made and cooking of the resin was carried by condensing the reaction mixture at 82–85 °C for a period of 90–100 min and consequently the flow time of the resin in hot condition is maintained to achieve 14.5–15.5 s in IS 39444 B4 flow cup. The cost reduction in phenolic resin adhesive after co-condensing with lignin was in the range of 20–30%.

Phenol Lignin Powder Formaldehyde Resin Adhesive

Lignin-based specialty chemical, Protobind 1075 supplied by ALM, was used as partial substitute of phenol in phenol formaldehyde resin suitable for plywood manufacture.

Lignin extracted from plant is in passive form and requires to be activated for polymeric reaction. Under the present reaction procedure, this was done in two stages. Initially phenol and lignin are digested in the presence of aqueous caustic solution till a uniform mixture is formed. This may lead to activate both phenol and

lignin in the presence of sodium hydroxide. After addition of formaldehyde, the methylene donor for linking phenol and lignin, the temperature of the mixture is raised to 90 ± 2 °C. This process helps all reacting molecule to be sufficiently active to react.

Actual reaction is carried out under controlled condition of temperature around 80–82 °C to avoid very fast reaction leading to sudden formation of bulky molecule and fast viscosity build up. Ratio of lignin + phenol to formaldehyde has been optimized. Lignin being already a bulky polymeric molecule will require less formaldehyde compared to phenol. 30% replacement of phenol by lignin (weight basis) in phenol formaldehyde resin 1:2 molar ratio (phenol:formaldehyde basis) was found to be ideal. Sodium hydroxide which acts as catalyst for the reaction need to be judiciously added to carry out controlled reaction and ultimate curing of the adhesive during plywood manufacture. Conventional PF resin made in single stage uses 6–8% caustic on the weight of phenol. For preparation of Protobind–phenol–formaldehyde (PPF) resin, it was found that only 6% caustic on the combined weight of phenol + lignin is sufficient. Higher percentage of caustic leads to faster reaction and shorter shelf life of the resulting PPF resin.

20% replacement of phenol by Protobind 1075 in PF resin has been found to yield sufficiently good quality resin to manufacture BWR- and BWP-grade plywood conforming to relevant IS specifications.

Tannin Extended Phenol Formaldehyde Resin

Tannins are extracted products found almost universally in plant tissues in small or large quantity. These are most abundant and widely distributed phenol polymer found in higher plants in the wood, bark, leaves and fruits. Tannins consist of a highly complex mixture of polyphenolic compounds soluble in water. These are divided into two groups, i.e. hydrolysable and condensed tannins. The former are esters of either gallic acid, hexahydroxydiphenic acid or similar substances with glucose or other saccharides. While condensed tannins are polymers of flavonols, a large majority of them being derived through the polymerization of flavon-3-ols and flavon 3,4 diols. Condensed tannins are characterized by low solubility but high reactivity with formaldehyde in comparison with hydrolysable tannins. In India, in addition to tree barks there are other sources of tannins such as tamarind seed testa, tea waste, areca dust and chogaru from arecanut industry.

The age of bark, condition of preparation of extract and influence of resin factors like viscosity, hardener, pH, filler content and keeping qualities need detailed investigations for commercial utilization of tannin formaldehyde adhesive.

Mimosa wattle (tannin) was used for extending PF resin for manufacture of plywood using veneers having moisture content greater than 6%. Tannin has been used as extender in phenol formaldehyde resin adhesive. Three different percentage of solid tannin (15, 20, 25) on PF resin were used in this study. The moisture content of veneers taken was at 6–10% and 12–16%. Tannin slurry solutions of

tannin:water = 1:2 and 1:3, respectively were used. 20% of this slurry on the total weight of PF resin (50% solids) was added. Characteristics of tannin extended PF resin adhesives (flow time, pH and solid content) were studied. Plywood was made with same species and thickness as in the case of PLF resin. The panels made were tested for BWP grade as per IS: 848-2006 "Specification for synthetic resin adhesives (phenolic and amino plastics) for plywood".

The panels bonded with 6–10% and 12–16% m.c. of veneers showed excellent results with both 20 and 25% extension by tannin in phenolic resin with slurry in ratio tannin:water = 1:2 and 1:3. Tannin extended PF resin adhesive is cheaper by 25–32% when compared to conventional PF resin. As the adhesive has the ability to bond high moisture content veneers of 10–16%, there will be energy saving, thereby reducing the total cost of the product further more.

Soya-Based Phenolic Resins

The protein in soya flour contains many reactive side chain amino acid groups (25–30% of total amino acids) that have the ability to react with phenolic resin. This reactive nature provides soya flour adhesive systems with the ability to form thermoset networks with a suitable cross-linking agent. In addition to the protein fraction of soya flour reacting with phenolic cross-linking agent, the carbohydrates fraction also contributes to additional durability through copolymerization. Due to the presence of high percentage of carbohydrates, soya flour requires more complex cross-linking techniques. Cross-linking these carbohydrates results in the much improved water resistance of the soya-based adhesives.

The soya protein treated with caustic increases the solubility of soya protein by increasing the net charge of protein. Hence the denaturing of protein plays an important role. The soya flour was denatured with alkali to alter the protein for good bonding and to break the covalent bonds (hydrolysed). After stipulated time, the viscosity of the alkaline-denatured soya flour mixture rapidly decreases. This is due to hydrolysis of the soya flour and the excessive breakdown of the of the protein structures which are considered to be of importance for the formation of both strong adhesive and cohesive bond. Thus, a balance of denaturing and retention of some secondary/tertiary/quaternary structure is optimized to improve the bond quality of the adhesive. Hence denaturing of soya flour has been optimized with minimal hydrolysis.

Soya chunks was procured and grounded. The flour was ground such that 90–100 weight percentage (wt%) passed through a 100-mesh screen. Soya flour was denatured by reacting with caustic solution. The denatured soya protein was modified and stabilized by adding formaldehyde and phenol. Under certain conditions, up to 30–40% of phenol can be replaced by soya without deteriorating bond properties. To attain an acceptable final product, it is required that soya reacts with formaldehyde and phenol in a high degree.

Phenol Cardanol Formaldehyde Resin

Cashew nut shell liquid (CNSL) is one of the sources of naturally occurring phenols. Anacardic acid and cardol are the main constituents of CNSL. The approximate ratio of these constituents is 90:10. Cardanol is the product obtained by distillation of commercial CNSL. Cardanol is a mixture of monohydroxy phenol with a linear side chain in the meta-position. The side chain contains on an average two double.

Reaction of Cardanol with formaldehyde During Polymerization

Cardanol can undergo all the reaction of phenolic nucleus. Cardanol can react with formaldehyde to give resins requiring organic solvents for formulation as adhesives. However, cardanol and phenol in nearly equal proportion by weight when reacted with formaldehyde using an alkali catalyst gave water dispersible resins. Adhesive formulations are developed using cardanol as a partial replacement for phenol in PF resin adhesives suitable to make BWR- and BWP-grade plywood.

Reaction of phenol cardanol mixture with formaldehyde was carried out in two stages, namely novolak resin stage and resol resin stage. Novolac resins are normally prepared by the interaction of a molar excess of phenolics with formaldehyde commonly under acidic conditions. Novolacs are mixture of long chain polymers in which the phenolic nuclei are connected by methylene bridges. Novolac resin formed in the first stage was subsequently dispersed in aqueous alkali and further reacted with formaldehyde to convert it into resol resin. Phenol cardanol formaldehyde resin thus formed was highly soluble in water.

Known proportion of phenol was replaced with 20% cardanol in the preparation of two stage alkali–alkali catalysed phenol cardanol formaldehyde resin.

In the second stage of condensation, control over the reaction is critical and must be carefully maintained both for the uniformity of the product and avoidance of too much cross-linkage which would otherwise result in gelling of the resin in the kettle.

Storage life of the PCF resin was found to depend mainly on the storage temperature and was about 45 days at about 25 °C. Higher the room temperature, lesser is the shelf life of the resin. Also the shelf life of the resin is dependent on the initial flow time of the resin, higher the flow time, lesser is the storage life of the resin.

Conclusions

This paper is being published based on the various research work carried out by IPIRTI on the “Development of phenolic resin adhesives by substituting phenol with materials of bio-origin”.

The conclusion drawn from the research is that about 20–25% of biomaterial obtained from natural renewable source, viz., mimosa wattle extract tannin powder can be used to extend phenol formaldehyde resin. 20–25% of biomaterial, viz., tannin powder with 1:2 and 1:3 slurry concentration of tannin solution can be extended with conventional 50% solid content PF resin. The resin developed also yield good bond quality adhesive for the manufacture of boiling water-resistant-grade plywood (BWR and BWP) conforming to relevant BIS specification; in addition, energy saving during production of panel products is also achieved.

It can be further concluded that 20–25% of black liquor having molecular range 8000–15,000 and 0–15,000 can be partially replaced for phenol in PF resin adhesive for the manufacture of BWP-grade plywood. Also cardanol and lignin powder of 20–25% can be substituted for phenol in PF resin for the manufacture of higher-grade panels which confirms to relevant specification for BWR-/BWP-grade plywood.

Presently on the investigations carried on laboratory scale for replacing phenol by soya, it has been observed that phenol can be replaced by about 20–30% of soya in the manufacture of phenol soya formaldehyde resin confirming to boiling water-resistant grade as per IS 848: 2006—specification for synthetic resin adhesives for plywood (phenolic and amino plastics).

The panels bonded using the phenolic adhesives partially substituted by renewable materials of bio-origin emits less formaldehyde. The replacement of 20–25% of phenol by biomaterial in the resin/adhesive system will contribute to 37–40% on the actual phenol formaldehyde resin being commercially used. There will be a saving of 6–8 tonnes of phenol.

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Experimental and Numerical Modeling of Hemp–Polyester Composites

D.S. Chethan, G.S. Venkatesh, Gunti Ranga Srinivas and C.S. Vinod Kumar

Abstract In this study, hemp–polyester composites were prepared with two volume fraction of hemp fibers and characterization for tensile properties. The mechanical characterization provided the quantitative insights into the elasto-plastic behavior of hemp–polyester composites. Young's modulus, yield strength, ultimate tensile strength and failure strain were obtained by testing the specimens in a uniaxial testing machine. Tensile strength and modulus of the composites were superior in the composites with higher volume fraction of hemp. The numerical modeling was then performed to simulate the constitutive behavior through out nonlinear range of hemp–polyester composite and study the effect of volume fraction of fibers/matrix on mechanical properties. Finite element model calibration was done to establish the validity of the modeling procedures and accuracy of prediction by comparing the numerical results with experimentally determined results. Understanding the elasto-plastic behavior through experimental and numerical simulations is considered to be a more viable tool for analyzing products made of hemp–polyester composites for possible use in automotive and other industrial applications in advanced simulations.

Keywords Composites · Hemp · Hemp–polyester composites · Finite element analysis · Numerical modeling

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Introduction

Hemp is a commonly used term for high growing varieties of cannabis plant. The hemp is refined into products such as hemp seed foods, hemp oil, wax, rope, cloth, pulp, paper, fuel and composite materials in which the material is reinforced with matrix materials in order to improve mechanical, physical, thermal properties. Relatively high strength and modulus of hemp fibers make them a preferred natural fiber as a reinforcement in both thermoplastic and thermoset composite materials. In recent years, the use of hemp fiber-reinforced composites has increased exponentially for various applications.

A number of studies have been carried out on hemp-reinforced composites, and these composites have been recommended for number of applications. Misnon et al. (2015) presented a detailed characterization of the woven hemp fabric composites. They recommended the use of fabric instead of yarn to enhance the reinforcement handling during composites fabrications. Habib et al. (2011) performed a study on hemp-based biopolymer composites to understand the effect of interfacial damage on the mechanical properties. Dhakal et al. (2007) reported a significant improvement in load-bearing capability and impact energy absorption of the composite following the introduction of hemp fiber as the reinforcement.

Hautala et al. (2004) reported flexural strength of hemp fiber strips–epoxy resin composites comparable to that of traditional plywood and found suitable for floor and furniture applications based on its appearance, properties and workability. The flexural strength of the composites at 50–60% fiber weight faction was determined at 140 MPa and flexural modulus at 6 GPa. Hemp–polyester composites have been reported to be comparable in properties with glass fiber-reinforced polyester composites. Yuanjian and Issac (2007) found that, with similar fiber weight fractions, the hemp and glass-reinforced materials exhibited similar static tensile properties and fatigue lifetimes. Although the slightly steeper S–N (the S–N diagram plots nominal stress amplitude "S" versus number of cycles to failure "N") curve of the hemp-based material indicated a higher rate of reduction in fatigue strength with increasing cycles, it remained above the S–N curve for the glass-based material, showing that it was able to withstand slightly higher cyclic stress levels for equivalent numbers of cycles. Ku et al. (2011) in their case study reviewed the tensile properties of natural fiber-reinforced polymer composites. They found that tensile properties of natural fiber reinforce polymers (both thermoplastics and thermosets) are mainly influenced by the interfacial adhesion between the matrix and the fibers. However, the Young's modulus of the natural fiber-reinforced polymer composites increases with increasing fiber loading.

There are a variety of phenomena that involve large-scale plastic deformation, geometric and contact nonlinearities. Modeling material nonlinearity requires additional information on material behavior. For example, for ductile metals, yield condition and failure criterion have to be defined and flow rule is tacitly assumed. The most common yield criterion for metals is the Von Misses surface in a multi-dimensional stress space; the yield strength obtained from a uniaxial tensile

test is required to define this surface. Venkatesh et al. (2012) used the finite element modeling tool in their work for predicting the behavior of composite materials and arriving at desirable filler contents for maximizing mechanical performance. They obtained quantitative information on the effect of reinforcing polypropylene with various proportions of nanoclay through experiments. Micromechanical finite element analysis combined with Monte Carlo simulation has been carried out to establish the validity of the modeling procedure and accuracy of prediction by comparing against experimentally determined stiffness module of nanocomposite. Macromechanical modeling was done to capture the nonlinear stress–strain behavior including failure observed in experiments as this is deemed a more viable tool for analyzing products made of nanocomposite. Eichhorn and Young (2004) showed that the microdeformation of single hemp fibers can be monitored by following the peak shift of the 1095 cm^{-1} Raman band with respect to strain and stress. This relationship was then used to monitor the deformation micromechanics of strained single hemp fibers with a microdroplet of epoxy resin attached along the gauge length. It was shown that it is possible to map the stress along the fiber both outside and inside the droplet. The profile of the stress distribution indicated the buildup of shear stress at the interface. The stress distribution within the droplet can be explained in terms of the effect of surface tension and contraction of the matrix around the fiber.

In the present work, quasi-static tensile tests were conducted for composites developed with hemp fiber reinforcement in polyester resin matrix to obtain the tensile strength and modulus of elasticity. Numerical modeling was carried out and validated with the experimental data.

Experimental Studies

Materials

Hemp is the shiny vegetable fiber. Hemp fabric was procured from Compact Buying Services, Sector-35, Faridabad, Haryana, India, having a density of 1400 kg/m^3 and was used as reinforcement in hemp–polyester composites. The isostatic polyester resin, having a density of 1280 kg/m^3, cobalt naphthenate accelerator and methyl ethyl ketone peroxide (MEKP) catalyst were procured from Swathi chemicals, Bengaluru, India.

Preparation of Composites

Hemp–polyester composites were fabricated by simple hand layup technique using compression-molding machine. Hemp cloth was prepared to the dimensions of the

Table 1 Details of hemp fiber used in hemp–polyester composites

Composite	Diameter of hemp fiber (mm)	Weight of hemp cloth (g)	Total weight of composites (g)
Hemp polyester (3 ply)	0.3	77.5	199.83
Hemp polyester (6 ply)	0.3	155	381.55

size 250 mm × 250 mm for 3- and 6-ply composites, and cloth was weighed on an electronic weighing machine to get different weight fractions. Table 1 shows the details of the hemp cloth used for preparation of composites. The working surfaces were treated with mold releasing agent polyvinyl acetate (PVA), and Mylar sheet was used to get glossy finish to laminates. The matrix material was prepared by using general purpose polyester resin, accelerator and catalyst in the ratio (1:0.15:0.15). Each layer was impregnated with the mixture up to completion of 3 ply and 6 ply of different volume fractions approximately.

Prepared sample was placed on the bottom plate in hydraulic press, and the uniform pressure was applied. The extra resin flows out, and uniform thickness was achieved by placing metallic spacers of specified thickness at the four corners of the bottom plate. The sample of each composite was cured under a load of 25 kg by applying pressure for about 4 to 6 h and thereafter removed from the mold. The composite sheets were post-cured in the air for another 6 h after removing from the mold (Fig. 1). Since the composites were prepared with 3-ply and 6-ply configuration, they were expected to have different volume fraction of hemp fibers with respect to resin material due to significant differences in the densities of fiber and matrix. The volume fraction of hemp fiber in the composite was calculated for both the composite compositions.

Fig. 1 Hemp–polyester composite laminate

Preparation of Specimens

The test specimens of hemp–polyester composites were cut from the laminates to the required dimensions as per the ASTM standard D3039. Five specimens each were cut from same laminates for tensile tests (Fig. 2) in warp and weft directions. Specimens were cut carefully by giving sufficient allowances for finishing and avoiding any defect centers at the edges of the test specimens.

Tensile Test

Tensile tests were carried out using a computer-controlled Shimadzu make 10 KN universal testing machine (Autograph AG-IS 10 KN). The crosshead speed during the test was 1 mm/min. All measurements were taken at five replicates, and the values were averaged.

Finite Element Modeling

Finite element modeling was performed to describe the strain–strain behavior of the composite material with different proportion of hemp fibers. The numerical simulation was carried out with the aid of the ANSYS R14.5 package. ANSYS model represented in Fig. 3 was subjected to uniaxial tensile load as in a standard tensile test. A bilinear material model was used by defining yield strength, tangent modulus, etc. Table 2 shows the inputs provided for the simulation. These input values were obtained from the mechanical testing of the composite material. PLANE 42 elements were used for meshing in the finite element modeling. Load is applied at one end, whereas other end is fixed with all degrees of freedom (Fig. 3). The model-generated values were compared with corresponding experimental values.

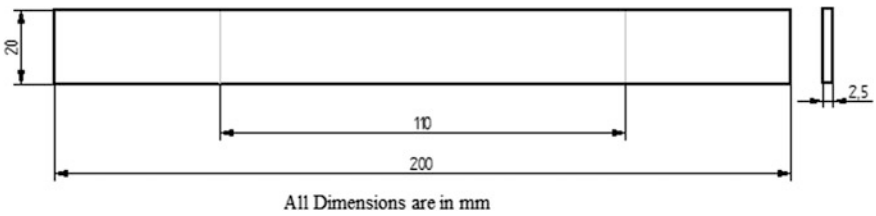

Fig. 2 Tensile specimen of hemp–polyester composites as per the ASTM standard D3039

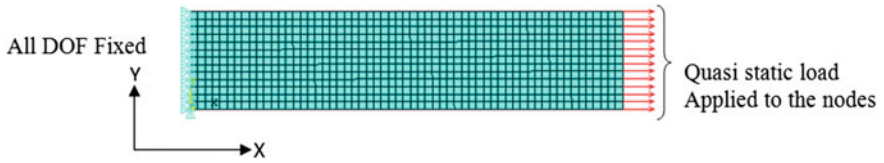

Fig. 3 Finite element model of tensile test specimen

Table 2 Material properties used in finite element modeling

Material properties	Magnitude
Young's modulus (GPa)	1.7
Poisons ratio	0.3
Yield stress (MPa)	15
Yield strain	0.01
Failure stress (MPa)	35
Failure strain	0.085
Tangent modulus (MPa)	266.6

Results and Discussion

Volume Fraction of Hemp–Polyester Composites

Volume fraction of hemp–polyester composites was calculated using Eq. (1) (Messiry 2013):

$$V_f = \frac{\rho_m w_f}{\rho_m w_f + \rho_f w_m} \tag{1}$$

where V_f is the fiber volume fraction, ρ_f is the density of fiber, w_f is the weight of the fiber, w_m is the weight of the matrix, and ρ_m is the density of the matrix.

In case of 3-ply composites, the weight of hemp cloth was 77.5 g and the weight of the polyester resin was 122.33 g, whereas in 6-ply composites, hemp fiber weight was 155 g as against the 226.55 g of resin weight. Correspondingly, the hemp fiber volume fraction for 3-ply laminate with 1.2-mm-thick composite was calculated as:

$$V_f = \frac{(1.28 \times 77.5)}{((1.28 \times 77.5) + (1.40 \times 122.33))} = 36.67\%$$

The fiber volume fraction for 6-ply laminate with 2.5-mm-thick composite was:

$$V_f = \frac{(1.28 \times 155)}{((1.28 \times 155) + (1.40 \times 226.55))} = 38.48\% \quad (1)$$

It was evident that the fiber volume fraction was about 2% higher in 6-ply composites than 3-ply composites.

Tensile Testing

Tensile properties for volume fraction of 36.67 and 38.48% of fiber reinforcement were obtained by conducting uniaxial tensile tests. The experimental results consist of stress–strain behavior of the material under tensile load, tensile strength and modulus data. Table 3 shows the Young's modulus and tensile strength in warp and weft direction for volume fraction of 36.67 and 38.48% of fiber reinforcement. There were slight differences in tensile strength in warp and weft directions in both composites. Tensile strength for a volume fraction of 36.67% was found to be 34.8 and 33.5 MPa, respectively, in warp and weft directions, and Young's modulus was found to be 1.7 GPa irrespective of the direction of measurement. For the volume fraction of 38.48%, the tensile strength was found to be 37.8 and 38.2 MPa in warp and weft directions, respectively, and the Young's modulus was found to be 2.0 GPa. The results indicate that with 2% increase in volume fraction of the fiber in the composite, tensile strength increased by 9% in the warp direction and 14% in the weft direction, whereas increase in the modulus was found to be approximately 18% in both the warp and weft directions. The increase in strength with increasing fiber content can be attributed to the superior properties of hemp fibers as compared to matrix material and efficient load transfer mechanism from matrix to the fibers.

Stress–strain plots of hemp–polyester composites for 3-ply and 6-ply composites in the warp and weft directions are shown in Fig. 4a–d. Bilinear behavior is observed in both the cases. It was observed that the stress–strain curve was linear up to 10 MPa stress level in 3-ply composite, whereas the linear range extended up to

Table 3 Mechanical properties of hemp–polyester composites

Samples	Volume fractions (%)	Young's modulus (GPa)	Tensile strength (MPa)
Sample 1 warp	36.67	1.7	34.8
Sample 1 weft	36.67	1.7	33.5
Sample 2 warp	38.48	2.0	37.8
Sample 2 weft	38.48	2.0	38.2

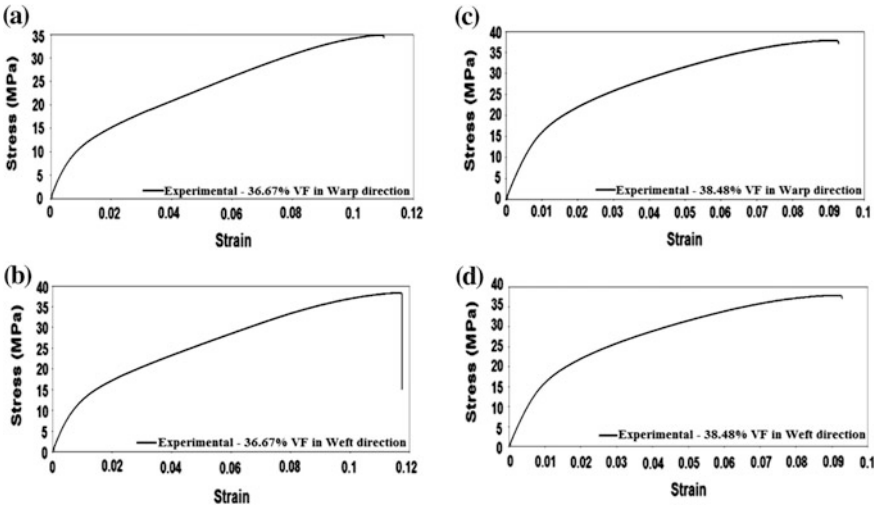

Fig. 4 Experimentally observed stress–strain curves for **a** 3-ply composite in *warp* direction, **b** 3-ply composite in *weft* direction **c** 6-ply composite in *warp* direction and **d** 6-ply composite in *weft* direction)

nearly 15 MPa in case of 6-ply composite. The failure strain for composite with 36.67% volume fractions of hemp fiber was slightly higher (0.11–0.12) as compared to composite with 38.48% (0.09–0.1).

Finite Element Modeling

The experimental results obtained from the tensile test were used for comparing the results obtained from numerical simulations. The comparison of stress–strain plots in warp and weft directions for 36.67% volume fractions and 38.48% volume fractions from the ANSYS simulation and the experimental results are shown in Fig. 5a–d. The results obtained from the numerical simulations were in good congruence with experimental results in the elastic region, whereas in the plastic region simulations predicted lower stress values when compared to the experimental results.

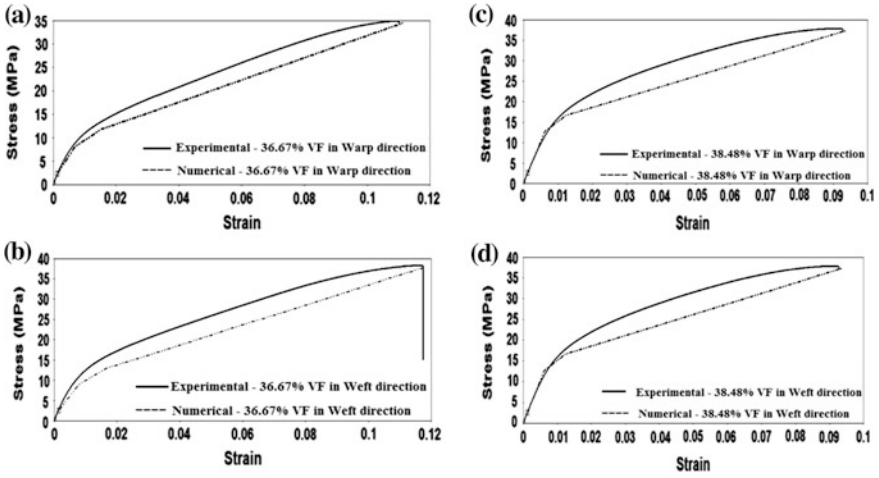

Fig. 5 Comparison of model-generated stress–strain curve with experimentally observed behavior for **a** 3-ply composites in *warp* direction, **b** 3-ply composites in *weft* direction, **c** 6-ply composites in *warp* direction, and **d** 6-ply composites in *weft* direction

Conclusions

In this study, preparation and characterization of hemp–polyester composites with different volume fraction were successfully carried out. Quasi-static tensile tests were carried out to know mechanical properties such as tensile strength and modulus in warp and weft directions for prepared composites with different volume fractions of reinforcement. There was no significant difference in properties found in warp and weft directions. It was observed that the increase in volume fractions of hemp fiber reinforcement resulted in increase in tensile strength and modulus. The results from simulations of tensile tests predicted stress–strain behavior which almost matched with the behavior observed in experiments.

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Lantana Fiber-Filled Polypropylene Composite

Amey Kale, N. Raghu, Shakti S. Chauhan and Pankaj Aggarwal

Abstract *Lantana camara* is a small perennial shrub species, native to central and South America. The species has established itself in agricultural fields as well as in forest areas of India and has become dominant weed crowding out native species and reducing bio-diversity. So far, Lantana has very limited commercial uses like furniture making and as a source of energy. In order to get rid of its invasion to forest and agricultural areas, it has to be utilized at the industrial scale. Using lantana stems as the source of lignocellulosic fiber in thermoplastic composites is one the potential applications. In this study, Lantana flour-filled polypropylene composites were prepared. Lantana stems were cut, chipped into smaller size, dried and pulverized to get the fine powder. Lantana flour was compounded with polypropylene in a twin-screw extruder using maleic anhydride-grafted polypropylene as the coupling agent. Composites were prepared at varying fiber loading from 10 to 50%, and mechanical properties were investigated. The mechanical tests indicated that the tensile and flexural strength properties increased with the increase in the fiber loading percentage in case of coupled composites. The presence of bark in lantana flour had no significant influence on the strength properties of composites. The properties of lantana-filled composites were found to be comparable with wood and bamboo fiber-filled composites. The study clearly demonstrates the potential utility of lantana stems for wood polymer composites.

Keywords Composite · Lantana · Polypropylene · WPC · Weed

Introduction

Bio-based fibers extracted from biomass are extensively being used in thermoplastic and thermoset composites, over glass fibers, talc and mica because their properties like light weight, low density, low cost, non-abrasive to processing equipments,

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good thermal properties, enhanced energy recovery renewability and most importantly biodegradability (Karnani et al. 1997). High specific strength, availability and cheap cost make natural fibers attractive as reinforcing material. The reinforcement effect of natural fibers in thermoplastic polymers depends on the interfacial adhesion between the two materials. Surface properties at the interface largely influence the mechanical properties of such composites. Natural fibers being hygroscopic, its compatibility with non-polar polymeric matrix such as polypropylene and polyethylene is a major concern in the development and application of these composite materials. Due to differences in surface energies and material densities, natural fiber tends to segregate and agglomerate during compounding, and extrusion lead to poor dispersion of fibers in matrix.

To overcome the compatibility issues and to improve the mechanical properties of such composites, coupling agents or compatibilizers are added during blending of fibers with polymer matrix. Over forty coupling agents with different functional groups have been used in wood fiber-filled thermoplastic composites (Lu et al. 2000). Coupling agents based on isocyanate (Karmarkar et al. 2007), silanes (Xie et al. 2010), anhydride (Kim et al. 2007; Dányádi et al. 2007) and acrylates (Parcella et al. 2010) groups have been investigated for their efficacy. Among the studied coupling agents, maleic anhydride-grafted polypropylene (MAPP) is one of the most often used coupling agents.

Different types of natural fibers such as wood flour, pulp fiber, bamboo, hemp, sisal, jute, coir and kneaf have been used as the reinforcement in such composites using different coupling agents, and an extensive review about the suitability of such fibers is given by Mohammad et al. (2015). In addition, agro-based residues such as rice husk and wheat straw have also been used as the filler material in thermoplastic composites. Widening the spectrum of natural fiber for their suitability in natural fiber-filled composites is always desired to design and develop the composite material with the desired properties.

Lantana camara is a flowering ornamental plant of the family Verbenaceae. The species was introduced to India in the early 18th century, mainly as an ornamental plant (Deo 2010). Over a period of time, it has spread to a large part of our country and has become a dominant invasive plant species in many forest areas, fallow lands and even on agricultural lands. The stem of *L. camara* is thin and the wood is very tough and durable (Reddy 2013; Deo 2010). In an effort to utilize lantana and add an economic value to a weed, the stems are used in making cheap furniture, utility articles and as a fuel.

The present study deals with exploring the potential of using *L. camara* stems as a source of natural fiber in making natural fiber-filled polypropylene composite, thereby expanding the choice of natural fibers as well as effectively utilizing this invasive weed species. Lantana stem fiber and polypropylene composites were prepared at varying proportions using MAPP as the coupling agent and also without any coupling agent. The composites were evaluated for mechanical properties. Inclusion of bark in the lantana during the processing of composite was also investigated.

Materials and Methodology

Lantana stems were extracted from the IWST field station located at about 40 km from Bengaluru, debarked and dried. The debarked stems were chipped and pulverized into lantana flour. The flour was sieved to −85 + 100 standard BSS mesh size (particle size 150–180 μm). The sieved flour was oven-dried in oven at 103 ± 2 °C.

Repol H200 MG (Reliance make) polypropylene with melt flow index of 20 g/10 min at 190 °C and 2.16 kg load was used as the polymer. Fusabond (P 613, Dupont make) maleic anhydride-grafted polypropylene (MAPP) was used as the coupling agent. Commercial-grade anti-oxidants and wax were used as received.

Lantana–polypropylene composites with 10, 20, 30, 40 and 50% (on weight percent basis) were prepared. The dried lantana flour, PP, MAPP (2.5% of the combined weight of wood and PP) and additives (1% of the total weight) were mixed in a specific proportion in a high-speed mixer for about 2 min. The mixture was fed into a 28-mm co-rotating twin-screw extruder. Extrusion temperature was set from 160 to 175 °C in different zones of the extruder. The screw RPM was set to 100, and the total residence time of the mixture in the extruder was about 2 min. The extruded composite strands were passed through water bath for cooling and then palletized to 3-mm-long pallets using a palletizer. The pallets were dried in hot-air oven at 80 ± 2 °C for a minimum of 24 h to get moisture-free material for injection molding. The composites were also prepared without the addition of coupling agent in the formulation.

The dried lantana–PP granules were injection-molded into standard test specimens using L&T Demag make 60 ton injection molding machine. The temperature of different zones was set to 160 °C (near hopper zone)—180 °C (Nozzle). The injection pressure was 80 bar, injection speed was 80 mm/s and the mold cooling time was 45 s. The injection-molded test specimens were stored at room temperature for at least 48 h before mechanical testing (Fig. 1).

Mechanical properties, i.e., tensile, flexural and impact strengths of the composite material, were evaluated as per the ASTM standard methods. Tensile test was conducted in accordance with ASTM D638. Tensile test specimens were 160 mm long, 13 mm wide and 3.2 mm thick. The testing was carried out at 50 mm/m strain rate. Flexural strength was measured as per ASTM D790 where the specimens were 127 mm long, 12.60 mm wide and 6.60 mm thick. The strain rate for flexural testing was 2.8 mm/m. Tensile and flexural testing was carried out on a Shimadzu make 10 KN universal testing machine (model AG 10 KNIS MS). Five replicates were tested for each composition. Test specimens used for flexural testing were also used for impact strength. Un-notched Izod impact strengths were measured according to ASTM D256 on Ceast make pendulum impact tester (5.4 J hammer). Ten replicates for composition were tested for impact strength.

Fig. 1 Lantana–PP composite pallets and test specimens

Results and Discussion

The strengths and modulus of the composites prepared with and without coupling agent at different proportion of lantana and polypropylene are shown in Figs. 1, 2, 3, 4 and 5. The tensile strength of the coupled composites remained nearly constant up to 30% lantana content and exhibited marginal improvement at 50% fiber. The composites without coupling agent exhibited continuous decline in tensile strength with the increasing fiber content. At 50% lantana, the tensile strength of the coupled composites was nearly 35% higher than uncoupled composites.

The flexural strength of the composites exhibited continuous increase with increasing lantana in MAPP-coupled composites. At 10% lantana content, flexural strength was 48.64 MPa that increased to 56.32 MPa at 50% lantana. The strength decreased from 48.31 to 42.15 MPa in composites without coupling agent. The improved tensile and flexural strengths of the coupled composite indicate the superior interfacial adhesion between the lantana fiber and polypropylene in the presence of MAPP. The results suggest that with MAPP, lantana fibers are acting as a reinforcing element in PP matrix and effective load transfer is taking place from matrix to fiber.

The improvement in tensile and flexural strengths in natural fiber-filled polypropylene composites, when MAPP was used as the coupling agent, has been reported by other researchers. Felix and Gatenholm (1991) observed a linear increase in tensile strength with fiber content when MAPP-treated fibers were used instead of untreated fibers. About 21% increase in tensile strength was reported for

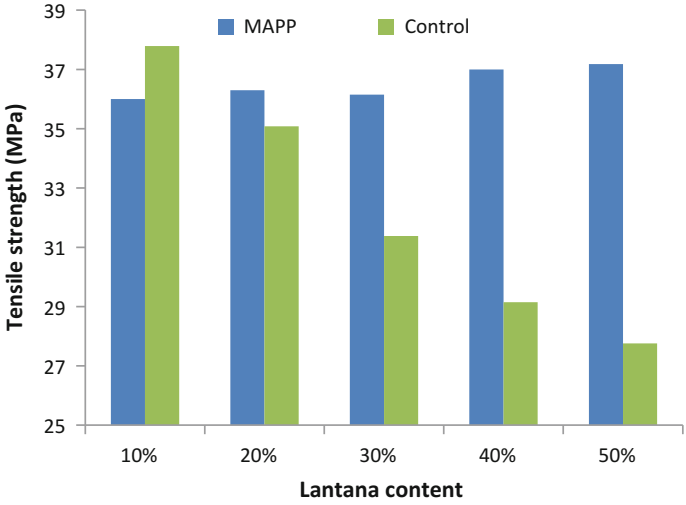

Fig. 2 Tensile strength of lantana–PP composites at varying filler level

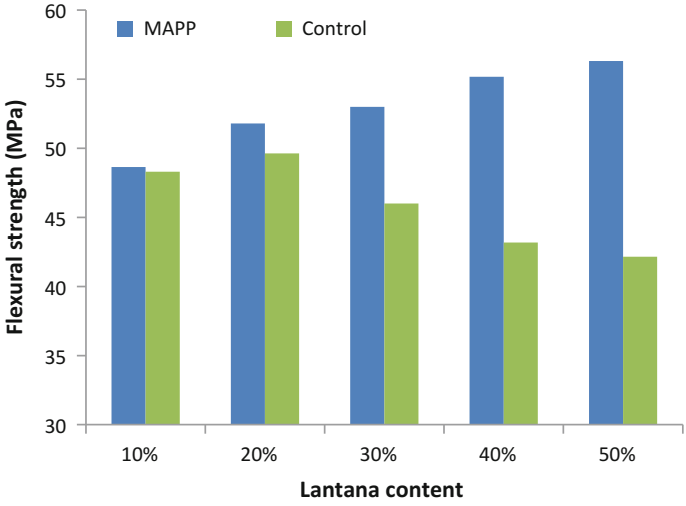

Fig. 3 Flexural strength of lantana–PP composites at varying filler level

50% wood flour and 50% polypropylene composites with MAPP as a compatibilizer (Myers et al. 1993). Similarly, 27% increase in tensile strength of composite prepared with 40% wood–fiber and 3% MAPP was reported by Stark and Rowlands (2003). The decline in strength properties with fiber content in uncoupled composites indicates that there is a little stress transfer from the polymer matrix to the lantana fibers, and the fibers are acting as filler rather than reinforcing element.

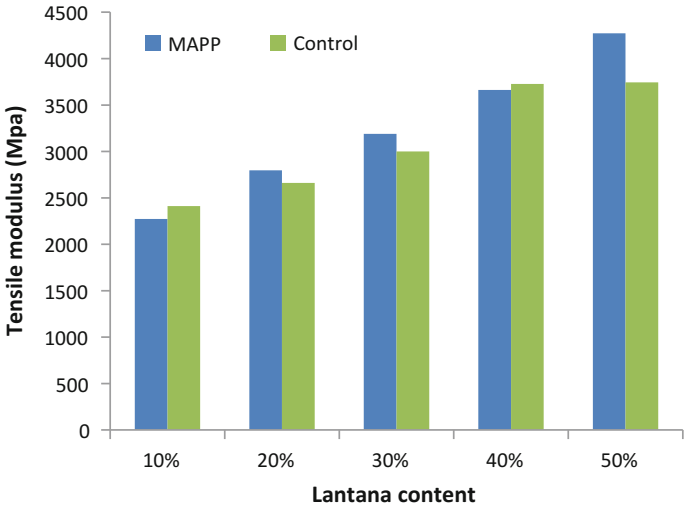

Fig. 4 Tensile modulus of lantana–PP composites at varying filler level

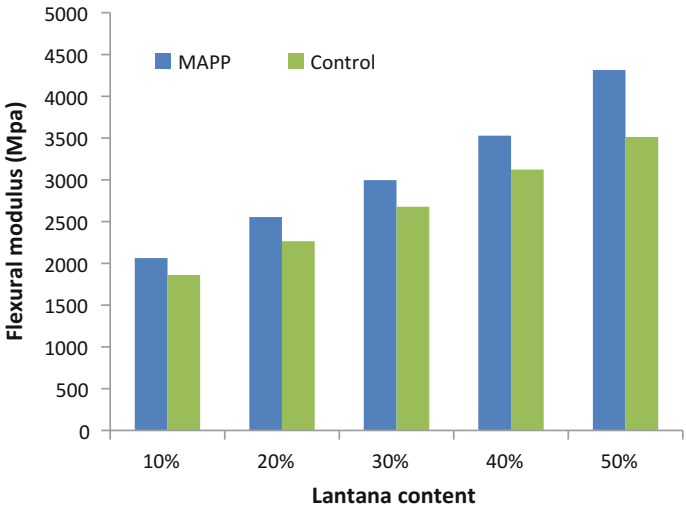

Fig. 5 Flexural modulus of lantana–PP composites at varying filler level

Both tensile and flexural modulus increased with the filler content of composites both with and without coupling agent (Figs. 3 and 4). There was nearly two-fold increase in the modulus values of the composites when the lantana content was increased from 10 to 50%. As compared to virgin PP, the modulus at 50% lantana was nearly three times (1.5 GPa for virgin PP and ~4.5 GPa at 50% lantana) The increase in modulus is attributed to the increasing proportion of high-modulus

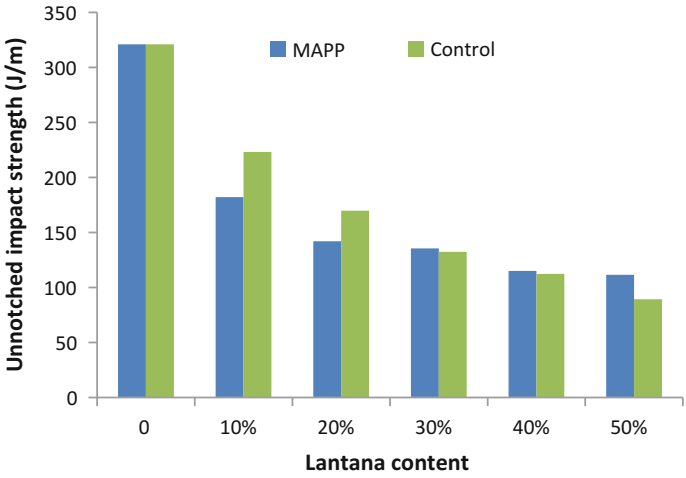

Fig. 6 Un-notched impact strength of lantana–PP composites at varying filler level

lantana in the low-modulus matrix material. The modulus of composite depends on the modulus of the individual component of the composite, volume fraction of fiber and matrix, fiber orientation and fiber shape factors (Chauhan et al. 2006). However, the difference in modulus of coupled and uncoupled composites tends to increase with the increasing lantana content. At 50% lantana, the tensile modulus and flexural modulus of coupled composites were 14 and 23% higher, respectively, than uncoupled composites. The increased difference in modulus could be attributed to the formation of a trans-crystalline layer at the interface in coupled composites (Harper and Walcott 2004).

The un-notched impact strength declined with increasing lantana content in both coupled and uncoupled composites (Fig. 6). The virgin PP exhibited impact strength of 321 J/m and declined to 182 and 223 J/m at 10% lantana in coupled and uncoupled composite, respectively. There was no significant difference in the impact strengths of coupled and uncoupled composites at fiber loading of more than 30%. The reduction in impact strength of polypropylene with the addition of filler or fiber is a well-known phenomenon as the fiber in the matrix polymer provides point for crack initiation and propagation. In addition, stiffening of polymer chains due to strong bonding between fiber and polymer in coupled composites leads to abrupt fracture and therefore decline in impact strength (Karmarkar et al. 2007).

Effect of Bark

In case of wood fiber-filled polymer composites, bark and other leafy materials are undesirable as they are not fibrous. However, removing bark in lantana stems can be very challenging and enormously energy intensive, particularly after little drying

Table 1 Properties of the composites with and without bark

S. no.	Formulation	Tensile strength (MPa)	Tensile modulus (GPa)	Flexural strength (MPa)	Flexural modulus (GPa)
1	Without bark	38.20 (0.51)	2.49 (0.28)	51.19 (0.89)	2.66 (0.31)
2	With bark	38.41 (0.68)	2.39 (0.33)	50.71 (0.65)	2.33 (0.87)
3	Virgin PP	37.71 (0.07)	1.48 (0.16)	38.83 (0.36)	1.37 (0.10)

Value in parenthesis is standard deviation

of stems during storage due to their smaller dimensions and limited diametral growth. In practice, it is not feasible to completely remove the bark for preparation of composite material. To study the effect of lantana bark on the properties of the composites, lantana stems with and without bark were pulverized and sieved. The flour was oven-died and used for composite preparation. The composites were prepared at 40% lantana content with coupling agent as per the procedure described earlier. The mechanical properties of the composites are given in Table 1.

It was observed that bark has no significant influence on the properties of the composites. Though, the color of the composite with bark was slightly darker which can be attributed to thermal degradation of bark during compounding at 180 °C. Since bark thickness and bark proportion (by weight) is very small as compared to woody material in lantana stems, composites can be prepared with bark thus avoiding energy and time-consuming process of bark removal.

The results of this study clearly demonstrate that lantana stems can be effectively used as a reinforcing natural fiber in thermoplastic composites. The strength properties of such composites are at par with the other natural fibers. The use of lantana for such composites would add an economic value to this abundantly available "weed," species.

Conclusions

The woody stems of *L. camara* were found to be suitable for natural fiber-filled polypropylene composites. The composites were prepared with lantana flour and polypropylene at varying proportions with MAPP coupling agent. The tensile and flexural strengths of the composites increased with increasing lantana content indicating the effective wetting of lantana particles by matrix polymer during compounding. The elastic modulus as well as impact strength was less affected by the presence of coupling agent in the composite. The modulus increased while impact strength decreased with increasing lantana content in the composites. The presence of bark in the lantana flour did not make any significant effect on the strength properties of the composite.

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Development of Fire Retardant Wood Composite Using Amino Resin

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Abstract Research on fire retardant wood and wood-based composites is being carried out worldwide for over a decade. Although many kinds of fire retardants for wood and wood-based composites have been studied, the focus is still mainly on compounds or mixtures containing phosphorus, nitrogen and boron, which can be used as aqueous solution. In this paper, fire retardant properties of particle board treated with boron and phosphorus chemicals are presented. Two methods were employed for the manufacture of fire retardant particle board using melamine urea formaldehyde resin either by treating the particles or by adding chemicals in the glue. The flame retardancy of the composites was studied by using limiting oxygen index test (LOI). The boards made were tested for flammability and rate of burning tests as per IS:5509. Physical and mechanical properties of the board were evaluated as per the standard IS:3087. It has been observed that the addition of flame retardant chemicals increased the LOI values. Samples were found showing time duration more than the required in the fire retardant test. The study indicates that the addition of fire retardant chemicals up to maximum of 2% on the weight of the particles in either glue composition or particle treatment would give excellent fire retardant properties without deteriorating the physical and mechanical properties of the boards depending on the chemicals used. Particle treatment method has shown better fire resistance and mechanical properties than the chemical incorporated in the glue.

Keywords Fire retardant · Particle board · Wood composite · Limiting oxygen index

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Introduction

Different types of particle board are manufactured according to their usages in the situations such as moisture-resistant particle board (for outdoor use), thin particle board (for furniture industry), high-density particle board (for flooring) and fire retardant treated particle board (for fire protection purposes) (Kamal et al. 2009). Research on fire retardant particle board is gaining importance. The basic raw material for the manufacture of particleboard is wood which is flammable. Hence, the wood particles and/or the resulting particleboard needs to be treated using fire retardant chemicals.

The primary objective of any fire-resistant is to maintain the structural integrity during a fire for a sufficient period so that all the occupants may safely evacuate, firemen may extinguish the fire, and the loss of property may be minimized (Kamal et al. 2009). The requirements for fire retardancy of wood composite have expanded from an initial focus on fire retardancy to include the factors such as smoke inhibition, environmental impact and economic aspects.

Thermal degradation of composite treated or untreated with fire retardants showed the loss of absorbed water, the formation of volatile products and the char. The overall pyrolysis of wood particleboard was affected by the type and amount of fire retardant (Karastergiou and Philippou 2000). A study by LeVan and Winandy (1990) has shown the reduction of strength in both the untreated and fire retardant-treated woods at elevated temperatures. However, the acid dehydration mechanism that helps in reducing flame spread also leads to premature strength loss in treated wood in some environments (Lebow and Winandy 1999a, b).

Fire retardant systems are generally based on phosphorus, aluminium and magnesium hydrates, and borates. The addition of borate-based buffers to fire retardant chemicals was found to significantly mitigate thermal degradation (Winandy 1997a, b). The fire retardant chemicals most used for wood products contain phosphorus (LeVan and Winandy 1990). Addition of zinc borate decreased the mechanical and physical properties of flakeboards with the increase in borates (Laks and Palardy 1999). On the other hand, Tsunoda et al. (2002) found no significant loss in mechanical and physical properties in medium-density fibreboard treated with zinc borate at retentions of up to 1.5% boric acid equivalent.

As boron compounds have neutral pH and less impact on mechanical properties compared to some other flame retardant chemicals, they are often considered a good flame retardant. Boron-based products are effectively used as a smoke suppressant, either alone or in conjunction with other fire retardant chemicals. Melamine, which basically contains nitrogen, and its combination with phosphorus are also used. Mixtures of borax and boric acid have been used as a preservative in wood. Borax and boric acid have low melting points and form glassy films on exposure to high temperature. The borax inhibits surface flame spread but promotes smouldering. Whereas boric acid reduces smouldering and glowing combustion, however has little effect on flame spread. Therefore, the combination of chemicals has some advantages (LeVan and Tran 1990). Boric acid suppresses both the dehydration

reaction during the early storage of pyrolysis and the free radical-induced degradation reaction during thermal decomposition (LeVan and Tran 1990).

Fire tests of different loading levels of borax boric acid add-on by weight for southern pine suggest that minimum loading levels of 7.5% add-on (3 lb/ft^3) (48 kg/m^3) of borax–boric acid are required to meet the ASTM E84 Class I for flame retardant (LeVan and Tran 1990). A study on surface roughness of plywood treated with fire retardant chemicals like borax, boric acid, monoammonium phosphate (MAP), diammonium phosphate (DAP) revealed that surface quality of the panels reduced with the increasing concentration above 6% (Ayrilmis et al. 2006). Fire retardant chemicals can be added either to the particles/fibres/veneers or to the adhesives or as coating in wood composites. When adding fire retardant chemicals in adhesives or with particles, it is necessary to assess the compatibility of particular type of fire retardant in a particular resin system without compromising the properties of the boards. Hence, a study using boron, phosphorus and nitrogen as fire retardant chemicals has been taken up. The purpose of the research was to determine the physical, mechanical and fire resistance properties of particle board using amino resin.

Materials and Methods

Particles of poplar wood were used for the manufacture of fire retardant particle board. Melamine urea formaldehyde resin (MUF), was used for the study.

Fire Retardant Chemicals

The fire retardants included in this investigation are as shown in Table 1:

The fire retardant treatments selected for these experiments include a number of common inorganic treatments which have proven effective for wood, and were

Table 1 Percentage of composition in the fire retardants used

Chemicals	Composition	Percentage
Borax	B_2O_3 Na_2O	36 16
Boric acid	B_2O_3	56
Monoammonium phosphate	P_2O_5 N	56 12
Diammonium phosphate	P_2O_5 N NH_3	43 25 21
Zinc Borate	B_2O_3	25

considered economically practical for the manufacture of particle board. Two methods of application of fire retardant chemical were studied.

Method-1: solutions of water soluble fire retardant chemicals (10% W/V) were made and then applied to the wood particles of 10–15% moisture content by spraying method. The particles after thoroughly mixing with chemicals were left for drying at ambient temperature. The particles were again dried in an industrial oven at 50 °C to achieve desired moisture content of 3–4% and kept ready for the manufacture of particle board.

Method-2: mixing of fire retardant chemicals in the glue, followed by mixing glue to the particles prior to drying to a moisture content of 3–4%.

Composition of Fire Retardant Chemicals Used for the Study

1. Borax, boric acid (PB)
2. Mono- and Diammonium phosphate (PN)
3. Zinc borate (ZB)

ZB and PB compositions were used for method-2 application process. PN composition was used for method-1 application process.

Manufacture of MUF Resin

Formalin of weight 160 kg (37% concentration) was charged into resin kettle, the pH of the formalin was adjusted to 8.5–9.0, and stirring was started. pH of the resin was adjusted with cooled sodium hydroxide solution. 40 kg of melamine and 40 kg of urea were added to the Resin kettle. The kettle was heated to a temperature of 85 °C and was maintained at the temperature 85 ± 2 °C until the water tolerance of 1:4 is achieved and then cooled to room temperature. After complete cooling, the resin was discharged and stored in air-tight container at room temperature. Shelf life of this resin when stored at 25 ± 2 °C was found to be 2–3 weeks.

For the manufacture of three layered particle board, the slenderness ratio (length/thickness) was maintained around 160–180 for face particle and around 50–80 for core particles. The resin prepared was added with other ingredients as given in Table 2 (adhesive formulation), and then the resultant glue is blended with the particles for panel preparation. Resin was admixed with wax emulsion, fire retardant chemicals and hardener using stirrer at fixed RPM of 100 for about 15 min for thorough dispersion.

Table 3 gives the formulation of boards named AC1–AC5. These boards were manufactured without and with selected fire retardant chemicals incorporated in the resin. AC1 is the particle board manufactured using pure resin as controlled board, and AC2, AC3 are the boards made using borax–boric acid composition and zinc

Table 2 Composition of adhesive

	Method-1		Method-2	
	Face	Core	Face	Core
Particles	Treated	Treated	Untreated	Untreated
Resin (%) on particles	12	8	12	8
Fire retardant chemicals	PN	PN	ZB/PB	ZB/PB
Chemicals (%) on particles	2	2	2	2
Wax (%) on resin	1	1	1	1
pH of the glue	6.5–7	6.5–7	6.5–7	6.5–7

Table 3 Formulations of boards

Sl. no.	AC1	AC2	AC3	AC4	AC5
Particles	UT	UT	UT	T	T
Resin	MUF	MUF	MUF	MUF	MUF
Chemical in glue		PB	ZB		ZB

T—treated particles; UT—untreated particles

borate composition, respectively, using untreated particles, whereas the AC4 board is made using treated particles. AC5 board is made with treated particles using zinc borate in the glue.

Manufacturing Process

Particles were dried to a moisture content of 3–4% before blending with the resin. The details of the boards formulated are given in Table 3. The particles were blended with 12% resin for face and 8% resin for core on dry solid basis. The glue-blended particles were placed into a mat forming box with base dimensions of 330 mm × 330 mm. Prepressing and compression of the particles were done by pressing a matching wooden plate on the mat in the forming box by applying manual pressure. Supporting rods to control the thickness to 12 mm were placed on either end of the assembly. The assembly was then loaded into a hot press of size 350 mm × 350 mm wherein temperature of the platens was maintained at 155–160 °C for particle board. Pressure of 25 kg/cm^2 for compression cycle and pressure of 14 kg/cm^2 for curing cycle with requisite curing time for respective resin systems were employed. The boards were kept for stabilization for about 24–48 h to attain equilibrium moisture content and then trimmed. The trimmed boards were further dimensioned to required sizes and subjected for testing as per relevant specifications.

Method Followed for Testing of Fire Retardant Particle Board

Fire retardant of particle board was tested as per IS: 1734-2003 (specification for testing fire retardant plywood). Inflammability, flame penetration and rate of burning were evaluated.

Inflammability: the time taken for the second ignition should not be less than 30 min.

Flame penetration: the time taken for flame penetration should not be less than 10 min for every 6 mm thickness.

Rate of burning: the time taken to lose weight from 30 to 70% should not be less than 20 min.

Limiting oxygen index: the limiting oxygen index (LOI) is used to measure the minimum oxygen concentration required to support combustion. The LOI test was done as per ASTM D 2863. In the test, the sample was held vertically in the transparent chimney, where the flow of oxygen and nitrogen was controlled. The test was repeated under various concentrations of oxygen and nitrogen to determine the minimum concentration of oxygen needed for burning the sample in 3 min or 5 cm.

Results and Discussion

Effect of fire retardant chemicals on the physical, mechanical and fire properties of poplar particle board was studied. Two methods were employed for the treatment of particle board, viz., particle treatment and by adding chemicals to the resin. For particle treatment, diammonium phosphate (DAP) and MAP were preferred as the solubility of these chemicals was better than the borax and boric acid solutions. On comparing the solubility of MAP and DAP chemicals of 10% w/v, the solubility of DAP with water was better than MAP and the pH of both MAP and DAP was found to be 9 and 4, respectively. A combination of DAP and MAP together 10% (W/V) was used for the study so as to maintain the pH required while mixing with the particles and subsequently with the glue. As the glue used in our study is MUF resin which is a pH-sensitive resin, the concentration of mixture of MAP and DAP has been optimized to get the desired pH of not less than 7.0.

For the glue composition borax, boric acid and zinc borate were the chemicals used for the study. Using the above two methods, fire retardant particle boards of 300 mm × 300 mm × 12 mm were made. Triplicate boards of each composition (Table 3) were made named AC1–AC5 using MUF resin. Board AC1 was the board made using only MUF resin as controlled board. MOE and MOR values of all the treated boards slightly decreased when compared with the values of control boards but were within the limit as per IS: 3087 as shown in Table 4. Incorporation of fire retardant chemicals generally caused the reduction in mechanical properties.

Thickness swelling and water absorption values of treated panels were increased compared to those of control boards. Internal bond strength values of treated panels significantly reduced but did not deteriorate by the fire retardant chemicals. This may be due to the usage of melamine urea formaldehyde resin. Melamine is a stable compound in its pure form, and the high cross-linking density of resin has shown good bonding at 2% of fire retardant chemicals.

Out of all the composition boards made, AC5 failed to comply with thickness swelling property as per IS: 3087. Board AC5 was made using fire retardant chemicals in both the particle treatment and glue. Due to much of acidity in the boards, it attracted more water towards itself and failed to satisfy thickness swelling properties. However, the water absorption property was higher than the controlled board, but values were within the limit as prescribed by the IS: standard.

The highest fire properties as per IS: 5509 have been shown by the board AC5. Figure 1 indicates that the boards AC2–AC4 have shown excellent fire retardancy than the controlled board without deteriorating the physical and mechanical properties of the board. This is due to the H^+ ion concentration of the usage of MAP, DAP, B_2O_3, which changes the pyrolysis of wood forming more percentage of char. The result of rate of burning indicates very clearly that the phosphate produces more char than the borax boric acid and zinc borate. Hence, the increase in the percentage of char has increased the fire retardant capacity of the composite. The flammability test has also shown the same trend indicating treatment of particles has shown better fire resistance properties than the other method.

Out of AC2, AC3, AC4 boards which have satisfied the properties of IS: 3087 and IS: 5509, AC4 boards have shown excellent physical, mechanical and fire

Table 4 Physical and mechanical properties of fire retardant treated amino resin particle board

Sl. no.	Properties	Prescribed value in IS 3087-2005 Grade-II	AC1	AC2	AC3	AC4	AC5
1.	Density (kg/m^3)	500–900	835	813	822	818	810
3.	Water absorption (%) (a) After 2 h of soaking (b) After 24 h of soaking	Max 40 Max 80	15.72 26.39	35.8 44.4	27.39 35.07	15.598 27.19	35.3 48.47
4.	Thickness swelling due to general absorption, % (after 2 h soaking)	Max 12	7.28	11.06	9.08	7.41	13.4
5.	Modulus of rupture (N/mm^2)	Min 11	23.12	20.68	21.96	21.15	17.86
6.	Modulus of elasticity (N/mm^2)	Min 2000	3927	3182	3613	3161	2891
7.	Tensile strength perpendicular to surface (internal bond strength) N/mm^2	Min 0.3	1.056	0.59	0.676	0.63	0.44

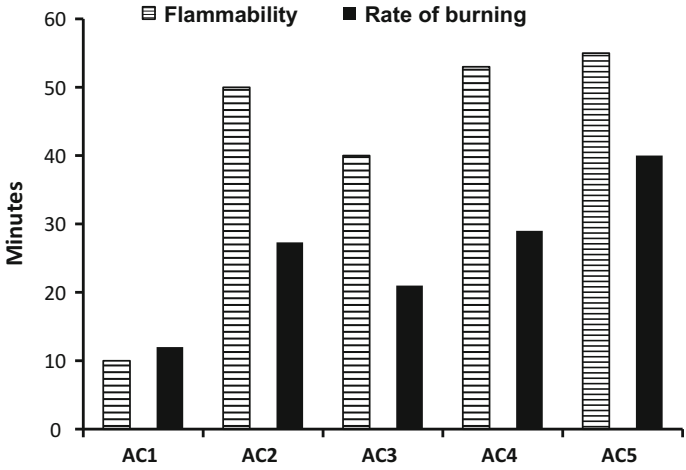

Fig. 1 Flammability and rate of burning of different particles boards tested for fire retardancy as per IS: 5509

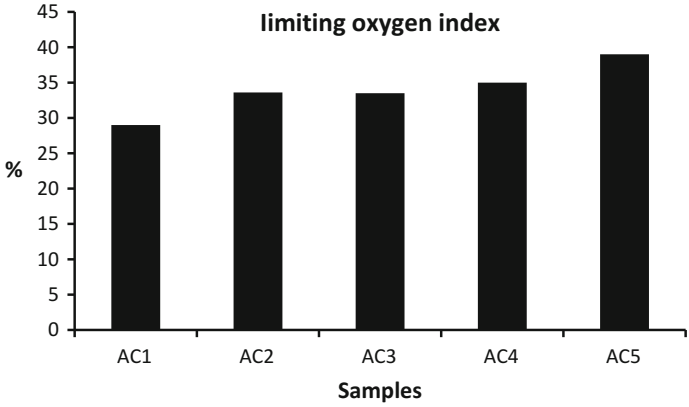

Fig. 2 Limiting oxygen index of the fire retardant particle board

retardancy. The particles after treatment with chemicals were dried at a lower temperature of 50 °C, so as to avoid the escape of nitrogen from MAP or DAP as the presence of nitrogen is necessary for the retention of phosphate group which in turn enhances the fire retardant properties of the board.

The LOI values of the particle boards are given in Fig. 2. As expected, the addition of fire retardant chemical into the particles increased the LOI values, an indication of increased flame resistance. The LOI values increased from to 29–38% with the incorporation of 2% of fire retardant chemicals on the particles. Highest LOI values were observed for the sample AC5 followed by AC4. It can be

observed that particle treatment method gives the increased fire retardancy than the glue treatment. This revealed that higher oxygen content was required to initiate and sustain combustion of the samples after the addition of flame retardant into particle board.

Conclusions

It can be concluded that the addition of fire retardant chemicals up to maximum of 2% on the weight of the particles in either glue composition or particle treatment would give excellent fire retardant properties without deteriorating the physical and mechanical properties of the boards depending on the chemicals used and method applied. Particle treatment method has shown better fire and mechanical properties than the chemical incorporated in the glue. The research findings are based on the laboratory-scale studies.

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Study on Utilization of Plantation-Grown Timber Species *Grevillea robusta* (Silver Oak) for Medium-Density Fibre Board

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Abstract There is an increase in production of industrial wood from agro/farm forest due to ban on sourcing of timber from forest areas for industrial production. Use of plantation-grown species for different panel productions poses different challenges in processing due to inherent defects present in the plantation species. There is a lack of information on the suitability of fibre from many plantation timbers for the manufacture of alternative panel products. Hence, a study to determine the suitability of plantation timber species *Grevillea robusta* (silver oak) as a raw material for medium-density fibre (MDF) board was taken up. Refining parameters of 0.3-mm disc gap and 6-bar pressure were found suitable for the production of fibres to manufacture MDF from silver oak species. The fibres were characterized to study the length and width using a progress capture camera with image analysis software. Test panels of thickness 12 mm were made of urea–formaldehyde resin. Physical and mechanical properties of the board were evaluated according to Indian standards IS 12406-2003. The result conforms to the IS specifications and indicates that MDF panels can be made from *G. robusta.*

Keywords MDF · Plantation species · Silver oak · Fibre · Refining parameters · Disc gap

Introduction

Since 1980s, the Government of India has promoted plantations under different agroforestry and social forestry plantation schemes as well as investment in industrial plantations. The plantation area in India is 32.57 m ha, which accounts for 17% of the global forest plantation, and is the second largest in the world after

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China. It also has the largest share in the global plantation of teak (44%) (MoEF 2000). The most prominent plantation species are eucalyptus, poplar, acacia, silver oak and rubber wood. Currently, silver oak species is used for plywood as it gives good peeled veneers. However, parts of lops and tops or parts of branches will be the source of fibre for the manufacturing of medium-density fibre (MDF) boards.

MDF is a non-structural wood-based panel that is composed of wood fibres bonded together with a resin under heat and pressure. MDF is manufactured in a wide variety of thicknesses from 2 to 100 mm and a very wide range of panel sizes. The density of these panels lies in the range of 500–900 kg/m^3.

Production of this product has increased drastically due to its ability to be produced in moulded form, as well as in straight-edged flat panels, for a host of industrial markets. MDF is used extensively in factory-assembled and ready-to-assemble furniture, as well as in cabinets, underlayment, drawer fronts, moulding and countertops. Finishes and overlays can be used to provide a grain pattern typical of lumber, and many wood finishing components such as door edgings, decorative trim, frames, and cornices are being made from MDF. Moreover, MDF is replacing thin plywood and wet-process hardboard in the production of moulded and flush door skins. New MDF products include generic and proprietary panels. One example is a refined board in which fine fibres are distributed throughout the board to facilitate deep routing and machining. In some countries, panels are being made from many different hardwood and softwood species as well as from non-wood-based lignocellulosics raw materials such as bagasse and cotton stalks.

MDF can be made from small-dimension logs/branches or from agro and forest residue. Conversion ratio from wooden chips to fibre varies from 80 to 85%. MDF possesses certain improved physical–mechanical properties similar to solid wood. In most of the end uses, MDF can replace solid wood and plywood because of its improved qualities. Due to user-friendly nature of the product, the demand for MDF is increasing in India. Since there is a huge gap between domestic production and demand, substantial quantity of MDF is imported into India from Malaysia, China and other countries. Looking into demand and better utilization of available raw material, there is urgent need for growth of MDF industry in India.

The industry cannot depend for long on tropical hard wood which cannot be sustainable source forever. There is a vast source of natural fibre like timber of plantation origin and agro and forest residue. Even residue generated in other wood and panel processing industries can be a source of fibre for MDF. But to utilise a new fibre material, research is to be carried out to evaluate adequate technology to convert into fibre, develop suitable binder and modify, if necessary, the process parameter for manufacturing MDF as per standard norms. Research is also required for product upgradation of existing product, to launch new variety in the product and also to bring solution to various technology problems faced in the process. This paper presents the manufacture of MDF panels from *Grevillea robusta* (silver oak) wood fibres and testing of their suitability as per the Indian standards.

Materials and Methods

G. robusta (silver oak) wood, procured from Coorg, Karnataka, was used to make the fibres for the manufacture of MDF board. The process steps for the production of MDF are described in more detail as follows:

Chipping—wood was chipped into square particles with sides of approximately 25 and 5 mm thick using a chipper. The chips were screened to remove oversized (>50 mm). The so-formed chips were stored in silos.

Soaking—the chips were soaked in water for about 24 h before passing it through a defibrator. This was performed to increase the moisture content of the chips to 100% as the increase in moisture content in the material resulted in the yield of good quality fibre. Chip soaking was found to be a necessary step in order to (a) remove the soil, bark and other abrasive contaminants and (b) to increase the durability of refiner discs. The surface wetting caused by soaking was bound to improve steaming and refining.

Steaming—the chips were steam-heated at 6-bar pressure, which created an internal temperature of 160–165 °C. The size of the digester was 25 m^3; hence, the retention time of the chips was kept 7 min for the study. It was observed that increase in retention time of the chips in the digester affected fibre colour and quality.

Refining—refining is a process to convert wood chips, flakes and shavings into fibres. Andritz-make refiner was used for defibrillation (Figs. 1 and 2). Refiner has two discs with one rotating at about 3000 rpm. Refiner plates with profile on surface dams and closed periphery were used for the refining process. Wood chips were fed between two discs through a round opening in the middle of the stationary disc. The surfaces of the discs have a series of raised profiles having coarse pattern in the centre and fine towards periphery. Centrifugal forces pushed the wood chips across the radius of the disc and were gradually broken down into its constituent fibres and fibre bundles. The adjustable gap between the two discs regulates the

Fig. 1 Refiner

Fig. 2 Refiner trials on silver oak

Table 1 Refining parameters/defibrillation process

Particulars	B1	B2	B3
Soaking time	Nil	24 h	24 h
Moisture content of chips (%)	26–28	50–60	50–60
Pre-heating pressure (kg/cm^2)	6	6	6
Pre-heating temperature (°C)	160–165	160–165	160–165
Pre-heating time (min)	7	7	7
Disc clearance (mm)	0.3	0.2	0.3

refiner energy consumption (150–400 kWh/ton fibre) and fibre quality. The blow valve attached to the discharge opening is operated to collect fibres from the refiner housing. The refining parameters are given in Table 1.

The wet fibres which had a moistures content of about 100–150% were collected in the tub and were spread on bamboo mat for open-air drying for about 24–48 h. Subsequently, drying was done in an industrial oven at controlled temperatures to achieve a moisture content of 4–6%. Moisture content of the fibres was measured using oven-dry methods. The dried fibres were characterized to study the length and width using a progress capture camera with image analysis software. Dried fibres were passed through ASTM sieve 18 and 45 to find the percentage of particular fibre length in the three batches of fibres.

Urea–Formaldehyde Resin

Conventional urea–formaldehyde resin was used for the preparation of MDF. For the preparation of resin, higher molar ratio of urea/formaldehyde was used at initial stage [mole ratio 1:1.7 or weight ratio of urea:formaldehyde = 1:2.3]. The properties of resin and adhesive formulation are listed in Tables 2 and 3, respectively.

The raw materials indicated in Table 4 were added through the feeder of a specially designed rotary drum blender shown in Fig. 3. The resin admixed with other ingredients like wax emulsion or hardener required (as given in Table 3) was taken in a spray gun (Fig. 4), and the resin spraying was done continuously with continuous feed of silver oak fibres into the rotary drum blender as shown in Fig. 5. The resin spraying was done up to the specified levels. The motor speed was maintained 80 rpm initially and 100–120 rpm for 3–4 min at the final stages.

Table 2 Properties of resin

Sl. no.	Particulars	Results
1.	Flow time of resin in B4 flow cup	20–22 s
2.	Water tolerance	1:3
3.	Solid content	50%
4.	Gelation time	62 s
5.	Shelf life	One and half months

Table 3 Adhesive formulation

Sl. no.	Particulars	Quantity
1.	Liquid UF resin	1000 g
2.	Wax emulsion 1%	10 g
3.	Scavenger 2.5%	25 g
4.	Liquor ammonia	10 ml
5.	Hardener (ammonium chloride) Water (mixed with hardener)	4 g 10 g

Table 4 Formulation of boards

Particulars	K	L	M	N	P
Refining process	B1	B2	B3	B3	B3
Fibre (gm) with dust <0.35 mm	900 19%	900 21%	900 10%	900 10%	900 Nil
Resin (%)	10	10	10	11	10

Fig. 3 Rotary drum blender for mixing

Fig. 4 Resin in spray gun

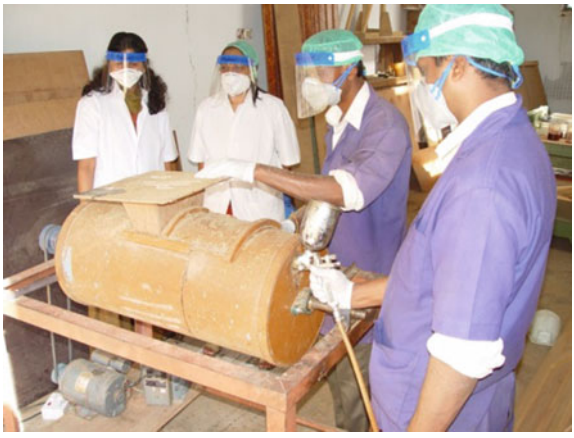

Fig. 5 Spraying of resin to the fibres

Fibre Mat Forming and Pre-pressing

The glue blended fibres were placed into a mat-forming box with base dimensions of 330 mm × 330 mm (Fig. 6). Aluminium/SS plates spread with releasing agents were placed on either sides of the fibre mat furnish. Pre-pressing and compression of the fibres were done by pressing a matching wooden plate on the fibre mat in the forming box by applying pressure manually. The pre-pressed furnish is shown in Fig. 7.

Fig. 6 Mat-forming box

Fig. 7 Prepressed furnish ready to load into press

Hot Pressing

The assembly was then loaded into a hot press of size 350 mm × 350 mm wherein temperature of the platens was maintained at 175–180 °C. Supporting rods to control the thickness to 12 mm were placed on either ends of the assembly. Pressure of 24 kg/cm^2 for compression cycle and 12 kg/cm^2 for curing cycle with requisite curing time for about 12 min was employed. Initially, the pressure is given higher so as to create a high surface density of the fibre board. The core density was then formed by reducing the applied pressure to 12 kg/cm^2. After stipulated period, the pressure was brought down to zero for few seconds to release the generated steam and gases from the fibre boards before opening the press, and then, the press was completely opened to download the boards. The boards were kept for stabilization for about 24–48 h to attain equilibrium moisture content and then trimmed. The trimmed boards were further dimensioned to required sizes and subjected to testing as per relevant specifications.

Results and Discussion

Medium-density fibre boards from silver oak of size 0.3 m × 0.3 m × 12 mm were prepared using urea–formaldehyde resin. Three batches of refining process were studied for the manufacture of MDF board. Characteristics of the fibres from each batches of refining were evaluated. The characteristics of refined fibre are given in Table 5.

Five sets of boards were manufactured from the three batches of fibre produced from refiner. The physical and mechanical properties of the MDF boards determined as per IS 12406-2003: Specification for General Purpose Medium-Density Fibre Board are presented in Table 6. Before testing, the specimens were conditioned for 24–48 h at 65 ± 5% relative humidity and a temperature of 27 ± 2 °C.

Three batches of refining process were studied, namely B1, B2 and B3. The parameters followed are listed in Table 1. B1 and B2 processes were followed to study the effect of soaking and B2 and B3 processes to study the effect of disc gap

Table 5 Characteristics of refined fibre

Particulars	B1	B2	B3
Moisture content of fibres (%)	75–80	115–120	115–120
Avg. fibre length, mm	1.504	1.67	1.89
Avg. fibre width, μm	31	28.6	25.3
Aspect ratio	48.52	58.39	74.7
Dust < 0.35 mm	18.8%	20.6%	9.8%
>1.0 mm	62.44%	36.22%	63.4%
=1.0 mm	15.2%	35.8%	23.2%

Table 6 Physical and mechanical properties of silver oak MDF in comparison with IS 12406 Specification for General Purpose MDF

Sl. no.	Properties	Prescribed value as per IS 12406-(2005) grade 2	K	L	M	N	P
			B1	B2	B3	B3	B3
1.	Density (kg/m^3)	600–900	878	874	898	858	856
2.	Moisture content (%)	5–10	5.25	5.50	5.20	6.20	5.80
3.	Water absorption (%) (a) After 2 h of soaking (b) After 24 h of soaking	Max. 9 Max. 30	6.50 16.0	7.40 17.8	6.20 13.8	4.82 11.95	5.40 13.4
4.	Swelling due to general absorption (%) (after 24-h soaking) (a) Thickness (b) Width	Max. 7 Max. 0.4	2.10 0.23	4.10 0.22	3.26 0.20	3.18 0.20	3.45 0.20
5.	Swelling due to surface absorption (after 2-h soaking), %	Max. 5	1.48	1.86	1.72	1.68	1.70
6.	Modulus of rupture (N/mm^2) (a) Average (b) Min. individual	Min. 28 Min. 25	44.33 33.49	41.87 33.22	56.7 50.2	50.6 45.8	45.03 41.05
7.	Modulus of elasticity (N/mm^2) (a) Average (b) Min. individual	Min. 2800 Min. 2500	3763 3579	4341 3530	4515 3666	4515 3666	4518 4264
8.	Tensile strength perpendicular to surface (internal bond strength) N/mm^2	Min. 0.8	0.40	0.40	0.60	0.85	0.80
9.	Screw withdrawal strength (N) (a) Face (b) Edge	Min. 1500 Min. 1250	2470 1640	3775 2110	4081 2475	4310 2440	3840 2510

on the fibre characteristics, amount of fibre bundles or fibre dust produced. Boards K, L, M, N and P were manufactured using urea–formaldehyde resin of E1 emission. K and L boards were manufactured using B1 and B2 batches of fibres, whereas M, N P were manufactured using B3 refining process.

Soaking of chips has great influence on amount of dust produced as given in Table 5. B1 process (without soaking of chips) has produced 19% dust as compared with B3 process (with soaking of chips) which produced 10% dust for the same disc gap of 0.3 mm and retention time of 7 min at pressure of 6 bar. The presence of more quantity of dust has significantly affected the mechanical properties of the board as given from Table 6. Modulus of rupture (MoR), modulus of elasticity (MoE) and internal bond (IB) strength of MDF from B3 process were significantly higher than that from B1 process.

The disc gap also plays a vital role in the quality of fibres according to Maloney (1993). Table 5 clearly shows how the percentage of dust decreases and fibre length increases when disc gap was increased from 0.2 to 0.3 mm for fibres from boards

B2 and B3, respectively. Higher dust formation in B2 process is attributed to the strong mechanical action/higher refining intensity due to reduction in the disc gap. Water absorption and thickness swelling were higher, whereas MoR, MoE, IB and screw withdrawal strengths were lower for B2 fibre board as compared to B3 fibre board.

Sundholn (1999) stated that softening of the fibres is very important factor in order to preserve fibre length and also to develop high-quality fibre with better bonding ability. Refining at low defibrator housing pressures or temperatures is not sufficient to soften the lignin components for smooth defibrillation of wood. Wood requires at least a temperature of 160 °C to generate optimal fibre quality. Hence, fibre softening was done in the digester at pressure of 6 kg/cm^2 at a glass transition temperature of lignin at a temperature of 160 °C.

According to Suchsland and Woodson (1991), the fibre length distribution and the strength of the fibre type influence the properties of MDF. Smaller fibre lengths and increased amount of dust and fine particles result in inferior quality of MDF. It is well-known fact that fibre aspect ratio influences almost all the properties of the MDF. Table 5 shows that the average length of the Silver oak wood fibres was approximately 1.50–1.89 mm and the average fibre width was in the range of 25–31 μm for B1, B2 and B3 methods of refining. From the three batches of refining process, it was found that B3 refining process gave satisfactory results with respect to fibre quality and properties of MDF except tensile strength.

Table 5 shows that B3 has shown higher average fibre length than B1 and B2 processes. Further, the boards (M) made from B3 fibres have shown higher MOR values as high percentage of longer fibres resulted in improvement in fibre-to-fibre bond. This is consistent with the results observed by Aisyah et al. (2012) and Nourbaksh and Ashori (2009) that one of the most important parameters controlling the mechanical properties in composites is having long fibres with high aspect ratio. Hence, boards N and P were manufactured using fibres from B3 refining process to improve the tensile strength of the boards. K, L, M and P boards were manufactured using resin content of 10%, whereas board N was manufactured using 11% resin on the oven-dry weight of the fibre. All the boards showed values above the prescribed value for MoR and MoE, whereas boards with N and P formulations only exhibited internal bond strength as per the standards.

For the manufacture of board M, resin content of 10% was used. Table 6 shows that they exhibited much poorer IB strength. However, when 1% resin content increased in the board N, tensile strength also increased. This decrease in the internal bond strength is attributed to the presence of 10% dust in the B3 process. It is well known that increasing the resin content increases the properties of the boards but also enhances the formaldehyde emission and cost of the product. Hence, the dust (0.35 mm) from the fibres was removed using ASTM-35 sieve, and the board was manufactured using 10% resin content which influenced the improvement in the fibre-to-fibre bond. This is given in P sample in Table 6 showing an improved tensile strength.

Conclusion

The study shows that pre-soaking of chips and disc gap has great influence on control of amount of dust produced besides on the strength properties of the panels. Eleven percentage resin content is needed for the manufacture of board with 10% fine dust fibres. However, when dust fine fibres are removed, 10% resin content is sufficient for the manufacture of the board. From the results, it can be concluded that MDF on a laboratory scale can be manufactured with plantation-grown *G. robusta* (silver oak) species to meet the requirement of IS 12406-2003 "Specification for MDF for General Purpose". Additionally, pilot-scale and full production trials must be conducted to confirm our laboratory results. These research results are promising and indicate that plantation-grown *G. robusta* (silver oak) can successfully be utilized for the manufacture of MDF.

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Suitability of Mixed Species of Bamboo (*Bambusa polymorpha* and *Bambusa tulda*) for Medium-Density Particle Board

Amit Ashok Mahadik, Anil Negi and Amey Pravin Awale

Abstract Bamboo is a versatile and important building material particularly in Asian countries. It has a higher compressive strength than wood, brick or concrete and a tensile strength that rivals steel. Particleboard offers a mean to utilize the wide verities of forest and industrial wood waste. In the present study, suitability of mixed particles of two bamboo species for the preparation of medium-density particle board has been assessed. Properties of medium-density particle board prepared using mixed dried particles of *Bambusa polymorpha* and *Bambusa tulda* at three different ratio of particle, i.e., 30:70, 50:50, 70:30, two different resin content, i.e., 10 and 12% and two different pressures, i.e., 14 and 17.5 kg/cm^2 at 150 °C have been evaluated as per Indian Standard Specification IS: 2380-1977. Suitable particle board can be prepared by mixing particles of *B. tulda* and *B. polymorpha* in the ratio of 30:70 with 12% resin content at 14 kg/cm^2 specific pressure for 15 min. The particle board meets most of the requirements of medium-density particle board as per the Indian Standard IS 3087-1985 except water absorption and swelling due to surface absorption, which may be controlled by adding wax emulsion at time of glue mixing.

Keywords Particleboard · Resin · *Bambusa polymorpha* · *Bambusa tulda*

Introduction

Bamboo is one of the fastest-growing plants in the world due to a unique rhizome-dependent system. It is a versatile raw material as a food source and is a suitable building material. Bamboo has a higher compressive strength than wood, brick or concrete and a tensile strength that rivals steel. The forest area, over which

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bamboos occur in India, on a conservative estimate, is 9.57 million hectares, which constitutes about 12.8% of the total area under forests (Bahadur and Verma 1980). Bamboo offers good potential for processing it into composites as a wood substitute.

Particleboard offers a mean to utilize as much as the forest and industrial wood waste as possible because it is so tolerant of wood quality and a wide variety of species. The technical feasibility for making single-layer experimental particleboard panels from bamboo waste (*Dendrocalamus asper* Backer) by converting bamboo into strip was evaluated by Laemlaksakul (2010). Bamboo particleboards made from acetylated particles of *Phyllostachys bambusoides* have great dimensional stability (Rowell and Norimoto 1988). Particles of Bamboo (*Gigantochloa scortechinii*) are suitable for the manufacture of urea formaldehyde particleboards. With more resin available at higher resin content, more bonding sites are made available, thus improving the strength properties and increased their dimensional stability significantly (Kasim et al. 2001). Similar observations on the strength properties–resin contents relationship were also reported by Chen et al. (1991).

The strength and dimensional properties of particleboard are directly influenced by board density (Moslemi 1974). This is particularly true since higher density is usually associated with higher strength properties. The increase in strength properties could be probably associated with higher compaction ratio at higher density. Other researchers (Chen et al. 1991; Chew et al. 1992) also reported similar findings.

In the present study, the suitability of medium-density particle board from mixed particles of *Bambusa polymorpha* and *Bambusa tulda* at different ratio of particles, resin and pressures has been evaluated.

Materials and Method

Culms of *B. tulda* and *B. polymorpha* were collected from Range Office of Forest Research Institute, Dehradun. Phenol formaldehyde resin with 35% solid was used as binding agent. Boards were prepared using 10 and 12% phenol formaldehyde resin at two specific pressures, i.e., 14 and 17.5 kg/cm^2 for 15 min at 150 °C temperature.

Air-dried *B. tulda* and *B. polymorpha* particles were screened to remove the dust from the particles. The accepted particles were dried in an oven at 60 °C to achieve moisture content of 4–6%. Five hundred grams of dried particles was taken for making particleboards. Phenol formaldehyde resin was applied at a resin content of 10 and 12% resin solids based on oven-dry weight of wood particles. After blending, the particles were spread evenly into 30 × 30 cm wooden box placed on

metal caul plate. Paraffin wax was applied onto the caul plate to prevent the panel from sticking to the plate during hot pressing. The mat formed was initially pre-pressed manually to consolidate the thickness. After pre-pressing for 5 min, wooden frame was taken out. The mat was then hot pressed in hot press at an elevated temperature of 150 °C at two different specific pressures, i.e., 14 and 17.5 kg/cm^2. The mat was hot pressed for 15 min.

After hot pressing, particleboards were conditioned for 3–4 days at ambient room temperature and humidity prior to evaluation of properties. The physical and mechanical properties of the particleboard were evaluated based on the Indian Standard Specification IS: 2380 (Anon 1977).

Results and Discussion

The values of various physical and mechanical properties of particle board prepared from mixed particles of *B. tulda* and *B. polymorpha* in different ratio with 10 and 12% resin content at 14 and 17.5 kg/cm^2 specific pressure are given in the Table 1. The values are calculated as per IS: 2380-1977. The maximum density of particle board 0.84 g/cm^3 was observed in the boards prepared from *B. tulda* and *B. polymorpha* particles in the ratio of 50:50 at 17.5 kg/cm^2 specific pressure with 12% resin content. Moisture content of the boards prepared from *B. tulda* and *B. polymorpha* particles in different ratio and at different pressures with 10 and 12% resin content varies from 1.97 to 4.21%. Water absorption after 2 and 24 h was higher in all the boards. Minimum 39.3 and 53.4% water absorption after 2 and 24 h soaking was observed in the particle board prepared from the particles of *B. tulda* and *B. polymorpha* with the ratio of 70:30 and 50:50, respectively, at 17.5 kg/cm^2 specific pressure with 12% resin content. Water absorption after 2 and 24 h properties does not meet the requirement of Indian Standard. Swelling due to surface absorption also does not meet the requirement of Indian Standard. It varies from 10.01 to 21.32% in different particle boards.

Modulus of rupture (MOR) of particle boards prepared by mixing the particles of *B. tulda* and *B. polymorpha* in different ratio meets the requirement of Indian Standard IS: 3087 (Anon 1985). MOR values of particle board vary from 12.34 to 33.76 N/mm^2, and maximum value, i.e., 33.76 N/mm^2, was observed in the board prepared by mixing particles of *B. tulda* and *B. polymorpha* for the ratio of 30:70 at 17.5 kg/cm^2 specific pressure with 12% resin content. Out of the 20 different types of particle board prepared from *B. tulda* and *B. polymorpha* particles, only in six types of boards the tensile strength perpendicular to the grain meets the IS requirements. The maximum internal bond of 0.87 N/mm^2 was observed in the board prepared from *B. tulda* and *B. polymorpha* particles (ratio 30:70) at 17.5 kg/cm^2 specific pressure with 12% resin content.

Table 1 Physical and mechanical properties of particle boards from mixed species (*Bambusa tulda*: *Bambusa polymorpha*) prepared at different specific pressures with 10 and 12% resin content

Board specification ratio (*B. tulda: B. polymorpha)*	Applied pressure (kg/cm^2)	Resin (%)	Moisture content (%)	Density gm/cm^3	Water absorption (%)		Thickness swelling due to general absorption (%) 2 h	Thickness Swelling due to surface absorption (%) 2 h	MOR N/mm^2	Tensile strength (internal bonding) N/mm^2
					2 h	24 h				
30:70	14	10	2.2	0.72	66.4	76.9	18.29	14.44	24.06	0.5
		12	2.6	0.69	55.9	60.5	15.56	15.64	27.23	0.86
	17.5	10	2.02	0.74	60.3	70.4	17.19	16.64	27.12	0.83
		12	2.25	0.79	43.9	59	13.16	8.2	33.76	0.87
50:50	14	10	2.6	0.66	66	74.3	13.28	10.01	31.82	0.53
		12	2.6	0.67	51.2	60	16.94	21.32	27.4	0.56
	17.5	10	2.05	0.75	40	56.3	16.97	15.42	21.8	0.71
		12	2.5	0.84	41.6	53.4	15.81	15.3	29.13	0.82
70:30	14	10	1.97	0.67	91.7	105	13.87	14.85	16.37	0.75
		12	3.21	0.62	68.4	78.4	17.04	16.43	18.89	0.82
	17.5	10	2.29	0.69	57.9	67.5	18.69	13.57	20.57	0.56
		12	2.45	0.77	39.3	56	14.39	10.16	22.52	0.59
B. Tulda	14	10	3.48	0.45	118	118	21.6	18.07	12.94	0.14
		12	4.21	0.5	78.3	81.1	13.36	14.54	13.25	0.27
	17.5	10	3.05	0.7	92.7	91.2	17.28	15.76	16.08	0.36
		12	2.29	0.72	60.9	70.1	15	11.77	19.31	0.5
B. Poly	14	10	3.45	0.5	90.7	95.7	15.27	18.61	12.34	0.27
		12	3.03	0.75	62.2	72.7	15.77	13.03	13.1	0.81
	17.5	10	3.28	0.5	88.4	99.5	14.59	12.84	19.47	0.42
		12	3.44	0.69	64.5	71.1	12.74	14.32	23.08	0.68

Conclusions

The suitable particle board can be prepared by mixing particles of *B. tulda* and *B. polymorpha* in the ratio of 30:70 with 12% resin content at 14 kg/cm^2 specific pressure for 15 min which meets most of the requirements of medium-density particle board as per the Indian Standard IS 3087-1985. However, water absorption and swelling due to surface absorption does not meet IS requirement, which may be controlled by adding wax emulsion at time of glue mixing.

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Part IV
Wood Utilization Pattern

A Comparative Assessment of Autoclave and Microwave-Assisted Peroxometal Complex in Delignification of Wood Biomass for Enhanced Sugar Production

Pradeep Verma and Venkatesh Chaturvedi

Abstract For production of biofuels from woody biomass, an initial pretreatment step is required for removal of lignin prior to enzymatic saccharification. In the present study, ameliorating effects of peroxometal complexes on delignification of beech wood have been studied using external (autoclave) heating and microwave irradiation. The results clearly show that ammonium molybdate, when transformed to peroxometal complex by hydrogen peroxide (H_2O_2), exhibits potent delignification property. The beech wood gave sugar yield of 69 and 41.8% after microwave irradiation and autoclave heating, respectively, under optimized conditions. The results indicate that maximum sugar yield depends upon delignification of biomass as lignin inhibits conversion of cellulose into sugars. It can be concluded that excellent delignifying capability of the H_2O_2-activated ammonium molybdate system can be achieved through microwave radiation.

Keywords Biofuels · Peroxometal complex · Delignification · Autoclave · Microwave irradiation

Introduction

The intensive utilization of fossil fuel has lead to many serious consequences like environmental pollution and long-term economic and national security concerns. Biofuels produced from biomass can act as a convenient alternative to fossils fuels (Araque et al. 2008). Biofuels are environmental friendly, emit fewer amounts of greenhouse gases and can also be used in pure form or mixed with gasoline (Sanchez and Cardona 2008).

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Various methods have been developed to produce bioethanol (Demirbas 2005). But for large-scale production, the method should be economically viable so that biofuels become competitive with fossils fuels. Currently, bioethanol is mainly produced from sugarcane or cereals starch, which is quite expensive (Mosier et al. 2005). Lignocellulosic biomass (LCB) is also a potential substrate for biofuels (Itziar et al. 2012). The major components of LCB are cellulose, hemicelluloses and lignin (Lee 1997; Sun and Cheng 2002; Sindhu et al. 2015). Architecture of wood consists of systematically arranged cellulose molecules, filled with hemicellulose and lignin (Chum and Overend 2001). Conversion of LCB to ethanol involves pretreatment for the opening of the crystalline structure of cellulose by breaking down of lignin, conversion of cellulose to glucose by hydrolysis with a combination of enzymes and finally fermentation of glucose to form ethanol (Wyman 2003; Mosier et al. 2005; Alvira et al. 2010). All the three steps are important. However, the pretreatment remains very crucial as it makes up for the one-third of the total cost of ethanol production (Mcaloon et al. 2002). The pretreatment alters the lignocellulosic structure and increases the rate of enzymatic hydrolysis of the cellulose (Kim and Dale 2004).

Currently, various pretreatment methods are in practice, e.g., ammonia fiber/freeze explosion (Eggeman 2001), lime method (based on calcium and sodium hydroxide) (William and Holtzapple 2000), alkali pretreatment, steam explosion (Ballesteros et al. 2006), solvolysis (Itoh et al. 2003), dilute acid pretreatment using sulfuric acid or SO_2 (Guo et al. 2008) and various biological pretreatment strategies (Sindhu et al. 2015). The main drawback of all these processes is high cost, making these processes unacceptable for industrial applications.

Another promising approach is the addition of an activator or catalyst in the presence of hydrogen peroxide, which can degrade lignin and alter the framework of LCB. Various approaches have been used to overcome the low ability of peroxide to degrade lignin selectively. One of them is the addition of metallic catalyst directly to peroxide step, which by interaction with the peroxide would generate more potent oxidants *in situ*, allowing improved delignification with negligible or no carbohydrate degradation. Eckert (1982) proposed delignification of kraft pulp with hydrogen peroxide catalyzed by transition metals. Kubelka et al. (1992) demonstrated the ability of heptamolybdate metal oxide in the presence of peroxide in acidic conditions to delignify kraft pulp similarly, Agnemo (2002) suggested that the delignification property of peroxide is reinforced by molybdate in slightly acidic condition.

Microwave irradiation causes localized heating which lead to disruption of complex architecture of LCB and thus an easy access to cellulose and hemicellulose for enzymatic hydrolysis. (Sarkar et al. 2012). Verma et al. (2011) proposed microwave pretreatment of woody biomass in the presence of hydrogen peroxide (H_2O_2) with different ammonium salts and discussed the role of peroxometal complex on lignin removal.

In the present study, we have used ammonium molybdate, hydrogen peroxide and water as solvent in pretreatment process using autoclave and microwave system. The effect of the pretreatment time, concentration of ammonium molybdate and

hydrogen peroxide on pulp yield were evaluated using external heating (autoclave) and microwave irradiation. In addition, the pulp fraction obtained from pretreatment process was hydrolyzed with enzyme for saccharification to evaluate the sugar yield.

Materials and Methods

Samples

Beech (*Fagus crenata*) wood powder (approximately mesh size 30–42 μm) was used in the study. The wood powder was air-dried, it contained approximately 7–8% moisture content and was stored under dry conditions. Ammonium heptamolybdate and hydrogen peroxide were purchased from Wako Pure chemical industries Ltd., Japan, and Meicelase cellulase was purchased from Meiji Seika Co., Ltd Japan.

Optimization of Various Ammonium Molybdate Concentrations for Pretreatment of Biomass

Beech wood powder (2.0 g), an aqueous solution of ammonium molybdate (concentration range 1–45 mM), and 0.88 mM hydrogen peroxide were placed in 200-ml Erlenmeyer flasks. The total volume of solution was 20 ml. The reaction was carried out at 80 °C for 5 h. After the completion of reaction, the mixture was cooled and filtered. The residual wood powder was thoroughly washed with distilled water thrice to obtain the pulp fraction.

Pretreatment of Beech Wood Powder Using Microwave Irradiation and Process Optimization

The microwave pretreatment was carried out using Biotage microwave initiator 2.0 operating at 2450 MHz. One gram of beech wood powder was taken in a 20 ml of microwave reaction vials, and ammonium heptamolybdate was added to final concentration of 0.25, 0.5, 0.75 and 1 mM. The wood powder was presoaked in solution in a vial sealed with aluminum caps using especially designed septa supplied by the manufacture. The vials were kept in the microwave racks. The program was set up in chemistry mode (prestirring—30 s, FHT on, cooling off, power 400 μW and maximum absorption value—normal mode, magnetic stir speed —900 rpm, temperature −140 °C for 30 min and the pressure limit—maximum 20 bars). The program was executed using auto-sampler mode. The pulp fraction or pulp yield was calculated using the following equation:

$$\text{Pulp yield} = \left[\left(W_i - W_f\right)/W_i\right] \times 100$$

where W_i and W_f are weight of substrate before and after pretreatment, respectively.

Effect of Microwave Pretreatment Temperature, Pretreatment Time and Hydrogen Peroxide Concentrations on Beech Wood Powder

One gram of beech wood powder was treated with 1 mM of ammonium molybdate and 0.88 mM of hydrogen peroxide at different pretreatment temperature ranging from 80 to 180 °C for 30-min pretreatment time. In addition, the samples were treated at 160 °C for 3, 6 and 9 min pretreatment time to evaluate the efficiency of pretreatment temperature and time. Further, wood powder was treated with ammonium molybdate (1 mM) and different concentrations of hydrogen peroxide (0.22, 0.44, 0.66 and 0.88 mM respectively) at 140 °C for 30-min pretreatment time to study the effect of hydrogen peroxide concentration on sugar yield with respect to wood.

Pulp Saccharification

The obtained pulp fraction was used for enzymatic hydrolysis as per the following procedure. 0.5 ml of 1.0 M sodium acetate buffer (pH 4.5), 9.2 ml of distilled water and 0.1 ml of 2% aqueous sodium azide solution and 0.04 g (8FPU) of Meicelase (Meiji Seika Co., LTD., *Trichoderma viride*, 224 FPU g^{-1}; β—glucosidase activity, 264 IU g^{-1}) were added for a total volume of 10 ml (substrate concentration of 2%) in 50 ml of screw cap conical tube The tubes were then incubated on water bath shaker (45 °C, 140 rpm) for 48 h. After enzymatic hydrolysis, 1 ml sample was collected from each tube, placed in 1.5-ml Eppendorf tube and centrifuged at 5000 rpm for 5 min. The appropriate dilutions were made to estimate sugar yield using Somogyi–Nelson assay (Somogyi 1952).

Results and Discussion

Saccharification of Beech Wood Powder Treated in the Flasks

Commercial cellulase Meicelase preparation was used for the saccharification of the pulp obtained from beech wood powder pretreated with ammonium heptamolybdate peroxide system in the flask. At lower ammonium molybdate concentration of 1 mM and 5 mM, approximately 75–90% of the pulp yield was obtained, whereas

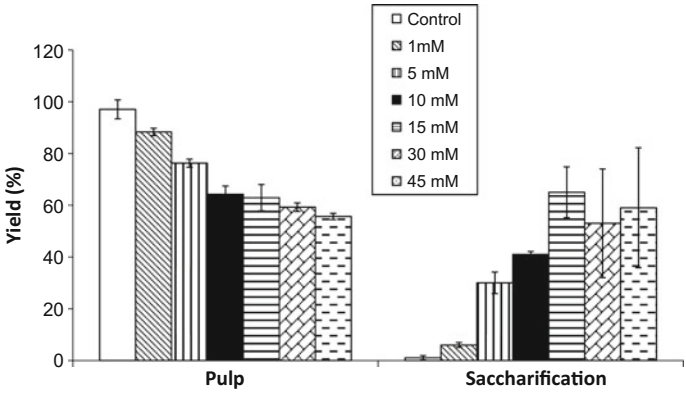

Fig. 1 Pulp and saccharification yield of beech wood powder (4% substrate concentration) pretreated with 1, 5, 10, 15, 30 and 45 mM of ammonium molybdate and 0.88 mM hydrogen peroxide in 200 ml of Erlenmeyer flasks at 80 °C for 5 h

only 6% and 30% of sugar yield with respect to wood were obtained, respectively (Fig. 1). The sugar yield was 64% at 15 mM of molybdate concentration. Further increase in the catalyst concentration did not improve the sugar yield.

Saccharification of the Autoclaved Pretreated Beech Wood Powder

Beech wood powder was pretreated with different concentrations of ammonium molybdate at varying pretreatment temperature and pretreatment time using autoclave. The results show a significant difference in sugar yield obtained from the pulp fractions for the different pretreatment conditions, i.e., ammonium molybdate concentrations, pretreatment temperature and time.

In the present study, maximum sugar yield was 62.7% using 15 mM molybdate concentrations at 100 °C for 90 min pretreatment time. Further increase in pretreatment temperature and reduced pretreatment time did not improve the saccharification yield. However, saccharification yield from pulp was improved at reduced molybdate concentration (2 mM) from 6 to 40% using autoclave at 140 °C for 60-min pretreatment time as compared to the flask experiment, which reduces the pretreatment time from 5 h to 1 h. Further, sugar yield was 5.1% at 140 °C for 60 min in the absence of catalyst with only peroxide, while sugar yield was 6.6% in the presence of catalyst without the peroxide. These results show that the addition of catalyst in the presence of hydrogen peroxide significantly improved the catalytic efficiency of peroxide. Eckert (1982) has also reported that improved delignification of kraft pulp was obtained in the presence of hydrogen peroxide and transition metals.

Saccharification of the Microwave-Pretreated Beech Wood Powder

Effect of microwave pretreatment of beech wood powder was evaluated in terms of low catalyst concentration. Figure 2a shows pretreatment of beech wood powder with different ammonium molybdate concentration. The sugar yield (obtained from pulp fraction per wood) was increased with increasing ammonium molybdate concentrations, while in the case of pulp yields, significant effects were not observed. The highest sugar yield of 65 and 51% (obtained from pulp fraction per wood) was achieved using 1 mM ammonium molybdate concentrations with microwave irradiation and 15 mM ammonium molybdate with autoclave pretreatment, respectively (Fig. 2b). Results indicate that the 1 mM of ammonium molybdate for beech wood is sufficient to make cellulose separate from lignin with minimum loss of cellulose to achieve high sugar yield.

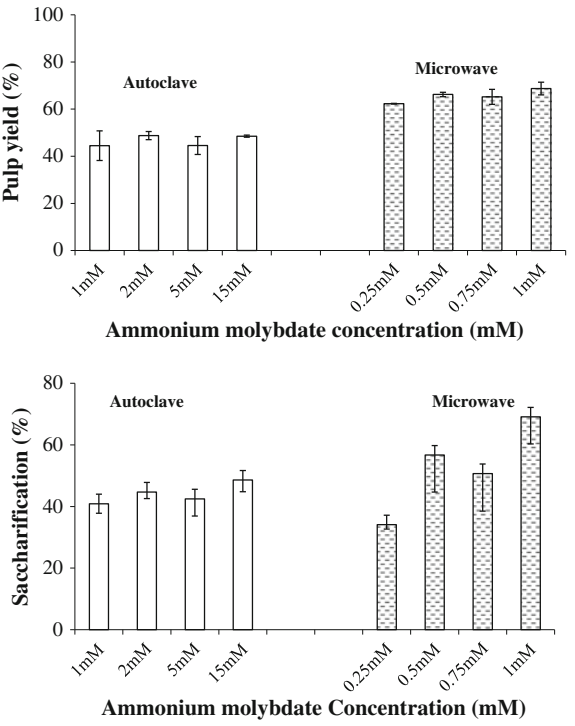

Fig. 2 Comparison of autoclave and microwave pretreatment on pulp yield and saccharification yield of beech wood powder (2% substrate concentration) at different ammonium molybdate concentrations (0.25, 0.5, 0.75, 1 mM) and 0.88 mM hydrogen peroxide in a reaction mixture using autoclave and microwave at 160 °C for 1 h and 9 min, respectively

Comparison of Autoclave and Microwave-Assisted Pretreated Beech Powder Using Ammonium Molybdate Hydrogen Peroxide System

These results show that the beech wood powder shows higher pulp yield after microwave as compared to autoclave pretreatment (Fig. 2a). The saccharification per wood yield is 48–69% after autoclave and microwave pretreatment, respectively (Fig. 2b). The results clearly show that higher sugar yield was obtained with microwave pretreatment at lower ammonium molybdate catalyst (1 mm) concentration as compared to autoclave process. In addition, it reduced pretreatment time from 1 h to 9 min as compared to flasks and autoclave pretreatments; microwave pretreatment drastically reduced the catalyst concentrations and pretreatment time.

The use of microwave pretreatment shows significant advantages in terms of efficiency as well as reduced energy consumptions which minimizes the overall cost of the process. Increased efficiency of microwave pretreatment process may be due to distribution of uniform heat in the wood particle because of continuous stirring of the samples.

Conclusions

Ammonium molybdate salt in combination of hydrogen peroxide shows great potential for the pretreatment of beech wood. The degradation of cellulose was avoided by performing pretreatment at neutral pH. The microwave-assisted pretreatment of beech wood in the presence of very low ammonium molybdate concentration with hydrogen peroxide and simultaneous fermentation enabled to achieve high amount of sugar yield. Microwave-assisted pretreatment gave further advantages with respect to efficiency and cost-effectiveness as compared to autoclave. Higher sugar yield was achieved even at low enzyme concentration and short fermentation time as compared to conventional processes used in the industries. Results indicate that microwave-assisted ammonium heptamolybdate with hydrogen peroxide is promising and offers good prospects for inexpensive, environmental friendly bioethanol production.

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Yield Evaluation of Oyster Mushroom on Dust Waste of Some Common Timber Species

C. Sneha and Minnu Tomy

Abstract An investigation on the feasibility of utilizing sawdust of locally available timber species as potential substrates for cultivation of oyster mushrooms was carried out. Oyster mushroom (*Pleurotus* spp.) was selected for the study because it is the most suitable species for utilization of lignocellulosic wastes. Its conversion rate, i.e., mushroom production from the substrate is highest, and the rate of growth is very fast. Sawdust of the most commonly available five timber species was used as substrates which include *Swietenia macrophylla*, *Tectona grandis*, *Xylia xylocarpa*, *Terminalia bellirica* and *Melicope lunu-ankenda.* A control treatment was maintained using mixture of sawdust of all these five species in equal proportions. All these six substrates were well sterilized, and spawn was inoculated and allowed to incubate under suitable temperature and relative humidity. Mycelial growth and pinhead formation were fastest in *Xylia* and slowest in *Terminalia.* In *Melicope*, no pinhead formation occurred even though there was good mycelial growth. Mushroom yield was maximum in control and minimum in *Melicope*.

Keywords Oyster mushroom · Dust waste · Lignocellulose · Spawn

Introduction

The increasing expansion of agro-industrial activity has led to the accumulation of large quantity of lignocellulosic residues all over the world. Zhang (2008) estimated that the global production of lignocellulosic biomass is more than 200×10^9 tons per annum. Cultivation of edible mushroom is one of the most economically viable processes for the bioconversion of lignocellulosic wastes (Bano et al. 1993; Cohen et al. 2002) which otherwise may cause serious environmental and health hazards. It offers an opportunity to utilize renewable resources for the production of protein-rich food (Philippousis et al. 2007). Lignocellulosic compounds are difficult

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to handle and dispose due to their chemical structure and decomposition properties. Edible mushroom or fungi possess approximate enzymatic mechanism for transformation of complex organic macromolecules into simple compounds.

Various lignocellulosic substrates such as sawdust, wood chips, corn stalk, cotton stalk, rice straw, waste hulls and other agricultural wastes can be used for mushroom cultivation (Oei 1996; Dundar et al. 2009). Digestion of cellulose produces glucose and cellobiose, while digestion of hemicellulose produces mostly xylose and other sugars, such as glucuronic acid and galacturonic acid as secondary products (Jefferies 1990; Clarke 1997). Since many sugars are released which are converted into sources of carbon when lignocellulosic substrates are digested, lignocellulosic substrates therefore are good substrate for mushroom cultivation (Keller 1993). Sawdust is the best as a substrate for its physical properties such as low apparent-specific gravity, high porosity, high water retention, moderate water drainage, high bacteria tolerance and biodegradability at an acceptable rate (Kitsui and Terazawa 1999).

Mushrooms are fleshy, spore-bearing reproductive structures of fungi. They are good sources of sugars, fibre, proteins and minerals (Aletor 1995). Among edible mushroom fungi, oyster mushroom (*Pleurotus* species) has received considerable attention for their nutritional value, medicinal properties and biodegradation abilities (Gregori et al. 2007; Israilides et al. 2008). It can be cultivated in various types of lignocellulosic materials such as straw, sawdust, rice hull, but the production efficiency was maximum in sawdust (Hami 1990). The objective of this study was to evaluate production of oyster mushroom on sawdust of locally available timber species.

Materials and Methods

Sawdust of most commonly available five timber species such as *Tectona grandis*, *Swietenia macrophylla*, *Xylia xylocarpa*, *Melicope lunu-ankenda*, *Terminalia bellirica* was used for mushroom production. A mixture of sawdust of all the five species was taken as control. Time taken for completing the spawn run (mycelial growth), date of appearance of first pinhead, etc., were recorded. Upon maturity, mushroom was harvested and weighed separately, and the weights were recorded. Total mushroom yield (five flushes) was expressed as accumulated fresh weight of mushrooms and as the accumulated biological efficiency (BE), i.e., accumulated fresh weight of mushrooms expressed as percentage dry weight of initial substrate. The days required for the completion of spawn running in the substrate bag were recorded. Days for pinhead formation, fruit body (flush) formation and for harvest were recorded.

Experimental Design

In the experiment, randomized block design with six treatments and five replications was used.

Statistical Analysis

Data obtained were subjected to statistical analysis using SPSS (v 20) software. The analysis used included Pearson's correlation, ANOVA and Duncan's multiple range tests.

Results and Discussion

Spawn run: Mycelial growth was the maximum in *Xylia* when compared to all other treatments (Fig. 1). In *Xylia*, spawn run competed within 6 days, while it was 7, 8, 9 and 10 days, respectively, for the treatments Mixture, *Swietenia*, *Melicope* and *Tectona*. Spawn run was slowest in *Terminalia*. During the vegetative phase of mushroom life cycle, mycelium grows through the substrate, biodegrades its components and supports the formation of fruiting bodies. Speed of spawn run denotes how easily the fungal hyphae establish in the medium for absorption of nutrients and water. From the study, it was observed that *Xylia* sawdust supports a faster growth of mycelial growth, whereas it was slowest in *Terminalia bellirica.*

Pinhead formation: The time taken to appear first pinhead in each treatment is illustrated in Fig. 1. Pinhead formation was fastest in *Xylia* which took 8 days. Regarding the other treatments, some took 14 days (*Swietenia*, *Tectona* and Mixture), whereas in *Terminalia* pinhead formation started only after 21 days. No pinhead formation taken place in *Melicope* till the end of the study period. Pinhead

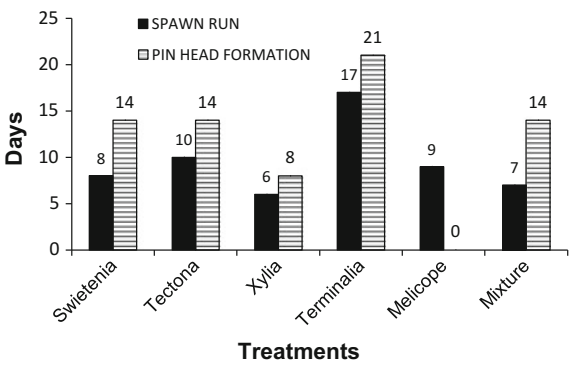

Fig. 1 Days taken for full hyphae formation and pinhead formation of oyster mushroom under different treatments

formation suggests the ability of fungi to produce fruiting body or reproductive structure which is happening only after full hyphae formation. Cellulose and hemicellulose (the main sources of carbohydrates) are often incrusted with lignin, which forms a physical seal around these two components. The proportions of the three structural components along with nitrogen content of residues affect mycelium growth, mushroom quality and crop yield (Shah et al. 2004; Philippoussis et al. 2011).

Yield: In the case of total yield, mixture showed significant increase when compared to all other treatments (Table 1). Mixture produced maximum yield of 35.78 ± 1.87 g. Among other treatments, *Xylia* and *Melicope* showed significant reduction in mushroom yield and these two were on par (11.85 ± 3.66 and 0.0 g, respectively). Similarly there was no significant variation in mushroom yield between *Swietenia*, *Tectona* and *Terminalia* in the present study (28.12 ± 1.71, 19.49 ± 2.25 and 21.93 ± 2.13 g, respectively).

Biological efficiency (BE): The term denotes to the accumulated fresh weight of mushrooms expressed as percentage dry weight of initial substrate. Among the six treatments, biological efficiency was highest in Mixture (Fig. 2). In all other treatments, BE was below 20. In *Swietenia*, *Tectona* and *Terminalia*, the percentage of BE was 18.7, 13.0 and 14.6, respectively. In *Xylia*, the percentage of BE was only 7.9, whereas in *Melicope*, it was found to be zero.

Table 1 Total yield of oyster mushroom under different treatments

Treatments	Mean yield ± SMe (g)
Swietenia	28.12 ± 1.71^{b}
Tectona	19.49 ± 2.25^{b}
Xylia	11.85 ± 3.66^{c}
Terminalia	21.93 ± 2.13^{b}
Melicope	0.00^{c}
Mixture	$\pm 1.87^{a}$

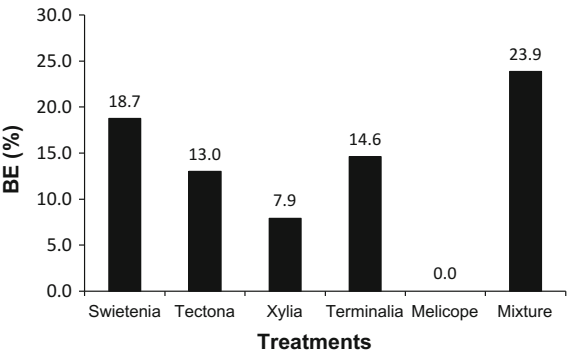

Fig. 2 Biological efficiency of oyster mushroom under different treatments

Conclusion

The present study revealed the possibility of using sawdust as an effective substrate for oyster mushroom cultivation. A major part of the plant biomass which is otherwise wasted can potentially be converted into protein-rich food material through mushroom cultivation.

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Study on Fuel Properties of Important Biomass Briquetting Feedstocks in India

Ritesh Kumar, M. Srinivasa Rao, R. Ezhumalai and R. Tailor

Abstract Briquetting of biomass presents a possible avenue for the large scale and sustainable utilization of waste biomass resources for energy production. Biomass feedstocks in their original form are difficult to use because of their high moisture content, irregular shape and sizes, and low bulk density. Densification of biomass materials, known as briquetting, not only improves the energy efficiency of biomass resources but also facilitates easy transportation, handling and storage. In this paper, we have studied fuel properties (calorific value, ash, volatile and fixed carbon content) of important biomass residues such as bamboo dust, coffee husk, groundnut shell, rice husk and saw dust. These lignocellulosic biomasses are commercially utilized for biomass briquetting in India. The result of the study suggests significant variations in fuel properties of biomasses. Ash content, volatile matter content and fixed carbon content varied significantly between 0.6–20, 61–73 and 13–20%, respectively. Calorific value also varied significantly (14–20 MJ/kg) among different biomass. Fuel properties of agriculture residues were found to be different from those of forest residues. The variation in fuel properties has been attributed to difference in chemical and elemental characteristics of biomasses.

Keywords Ash · Bamboo · Coffee husk · Groundnut shell · Rice husk · Saw dust

Introduction

Biomass fuels are promising and cost-effective alternative renewable energy source. Presently, biomass contributes around 14% of total world's energy supply, second only to fossil fuels (Asif and Muneer 2007). Biomass fuels are termed as carbon neutral fuel as they assimilate CO_2 during plant growth and emit the same during combustion. With the introduction of new technologies, biomass feedstocks are

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increasingly adopted for heat and power production in countries like India (MNRE 2015a). A surplus biomass of about 120–150 million metric tones per annum is available in India which may generate 18,000 MW of power (MNRE 2015b). Increasing demand of heat and energy from renewable resources, electricity problems, increased power tariffs and environmental concerns has further increased the demand of biomass energy. However, in their original form biomass feedstocks are difficult to use, because of high moisture content, irregular shape and sizes, and low bulk density. Such characteristic makes the transportation, storage and utilization process difficult. Biomass briquetting, which is a process of densification of loose biomass material and production of compact solid composites, has therefore been employed for overcoming these problems. The demand of briquettes is many times higher than its supply (Shekhar 2011).

Biomass can be burned directly or it can be converted into solid, liquid and gaseous fuels through thermochemical and biochemical conversion processes (Peter 2002a). A wide variety of biomass materials like wood waste, saw dust, bagasse, rice husk, straw, cotton stalk, coconut shells, soya husk, de-oiled cakes, coffee waste, jute wastes and groundnut shells are used for power generation in India either in its raw form or as briquettes. However, the quality of fuel depends upon the basic fuel characteristics of raw material (Peter 2002b). Fuel value is a function of chemical and elemental composition of biomass (Peter 2002b). Calorific value, ash content and moisture content are important fuel parameter which directly affects the quality of fuel (Kumar et al. 2010; Peter 2002b). In this paper, fuel properties (calorific value, ash, volatile and fixed carbon content) of important biomass residues such as bamboo waste, coffee husk, groundnut shell, rice husk and saw dust have been investigated. These biomasses were selected based on the feedback from the briquetting industries about their preference of raw material as they are commercially used for briquette manufacturing.

Methodology

Biomass residues which are commercially used for briquette making were selected for this study. Around three kilogram of sample was collected from farm waste dump. Samples were oven dried to constant weight at 80 °C. The oven-dried samples were pulverized to fine powder using a Wiley mill. The pulverized material was then placed in a sieves shaker, to pass through 425-μm-mesh sieve but retained on 250-μm-mesh sieve. The powder, thus, obtained was again oven dried and used for further analysis. One-gram biomass powder was pelleted, oven dried to constant weight at 80 °C and burned in an oxygen bomb calorimeter (LECO AC-350) for determining calorific value. The moisture content, ash content and volatile matter content were determined according to ASTM D5142, using a proximate analyzer (LECO TGA-701). The ultimate parameters (carbon, hydrogen, nitrogen) were determined using elemental analyzer (vario EL cube). Fixed carbon content (FCC) was estimated using Eq. 1.

$$\text{FCC } (\%) = [100 - (\% \text{ Moisture} + \% \text{ Ash} + \% \text{ Volatile matter})] \quad (1)$$

In order to overcome the experimental and instrumental errors, experiments were repeated four times and the average values were obtained. The data were analyzed using statistical software SPSS Ver 8.0. One-way analysis of variance was performed to see the significant differences for each fuel characteristics among different biomass.

Result and Discussion

The quality of briquettes depends upon the fuel characteristics of biomass feedstock. The maximum useful energy that can theoretically be extracted from a biomass is its chemical energy. The structural components of biomass are basically chemical–elemental bonds between carbon, hydrogen and oxygen. When the chemical bonds between the carbon, hydrogen and oxygen molecules are broken, the energy is released, known as bioenergy (Peter 2002b). Fuel properties results of bamboo dust, coffee husk, groundnut shell, rice husk and saw dust are shown in Table 1. Most biomass materials have moisture content <10%, suggesting they can be used for combustion purpose. High moisture content (>15–20%) is not desirable as it affects the energy value of a fuel (Senelwa and Sims 1999).

The quality of a fuel is affected by amount of ash present in a feedstock. The biomass having low ash is considered better feedstock (Tortosa-Marisa et al. 2007). The ash content affects both the handling and processing costs of the overall biomass energy conversion cost (Peter 2002b). Results presented in Table 1 show significant variation in ash content (0.6–20%) of biomass used for briquetting. Low ash content was recorded in saw dust (0.6%) and bamboo biomass (2.5%). High calorific value and low ash content make wood and bamboo suitable for energy production (Kumar and Chandrashekar 2014). The results of this study are similar

Table 1 Fuel characteristics of biomass feedstocks used for briquetting

Biomass feedstock	Ultimate analysis (%)		Proximate analysis (%)				CV (MJ/kg)
	C	H	MC	Ash	VMC	FCC	
Bamboo flour	46.7^{d}	5.9^{c}	6.5^{b}	2.5^{b}	73.2^{c}	17.6^{b}	18.9 ± 0.1^{c}
Coffee husk	41.3^{c}	6.3^{d}	5.0^{a}	12.5^{d}	65.6^{b}	16.9^{b}	18.0 ± 0.1^{b}
Groundnut shell	36.2^{b}	5.6^{b}	7.1^{c}	8.9^{c}	66.6^{b}	18.4^{b}	18.4 ± 0.6^{bc}
Rice husk	32.0^{a}	5.4^{a}	6.1^{b}	20.0^{e}	61.0^{a}	12.9^{a}	14.1 ± 0.1^{a}
Saw dust	47.3^{e}	5.9^{c}	6.7^{b}	0.6^{a}	72.6^{c}	20.5^{c}	20.1 ± 0.3^{d}

MC moisture content; *VMC* volatile matter content; *FCC* fixed carbon content; *CV* calorific value; *C* carbon; *H* hydrogen

Means followed by same letter within each column do not differ significantly (Turkey test) at 5% level

to earlier reported results on various wood and bamboo species (Kataki and Konwrer, 2001; Kumar and Chandrashekar 2014; Peter 2002b). In case of wood species, the ash content, volatile matter content and fixed carbon content are reported to vary between 0.5–3, 60–76 and 17–25%, respectively (Peter 2002b; Senelwa and Sims 1999). Similarly in bamboo biomass, the ash content, volatile matter content and fixed carbon content were found in the range of 1.4–3.0, 77.2–80.7 and 17.6–21.1%, respectively (Kumar and Chandrashekar 2014). The calorific value of coffee husk, groundnut shell and rice husk was found to be 18.0, 18.4 and 14.1 MJ/kg, respectively. The low calorific value of rice husk can be attributed to its high ash content (20%). The ash content in coffee husk and groundnut shell is found to be 12 and 9%, respectively. The high calorific value in coffee husk and groundnut shell (18 MJ/kg) can be attributed to their high fixed carbon content, i.e., 17 and 18%, respectively. Among agriculture residues, both coffee husk and groundnut husk are found as suitable feedstock, mainly because of their high calorific value and high fixed carbon content. The cellulose, hemicellulose and lignin constitute the major portion of both agriculture and forest biomass waste. The main chemical elements in biomass (apart from associated mineral matter) are C, O, H, N and S. The results of elemental analysis, presented in Table 1, indicate variation in the elemental composition of biomasses. Among different biomass studied, higher elemental carbon was found in saw dust and bamboo powder (47%) and the lowest carbon was found in rice husk (33%). In a biomass fuel, higher proportion of carbon–carbon bond is desirable, whereas higher proportion of carbon–oxygen and carbon–hydrogen bonds reduces the energy value of fuel (Peter 2002b).

Conclusions

Quality of briquettes depends upon the fuel properties (calorific value and ash content) of biomass. The results indicate that with high calorific value and fixed carbon content, groundnut shell and coffee husk are the promising agriculture feedstocks for thermochemical energy conversion. Groundnut shell and coffee husk waste have calorific value around 18.0 MJ/kg, which is more compared to rice husk (14.7 MJ/kg). The occurrence of high ash content in rice husk (20%) makes it less desirable for direct combustion. High calorific value, low ash content and high volatile matter content make saw dust and bamboo biomass suitable for energy production through pyrolysis and/or gasification.

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Study on Chemical, Elemental and Gasification Characteristics of *Lantana camara* Wood

R. Ezhumalai and Ritesh Kumar

Abstract *Lantana camara* is an invasive weed species, which is widely available in different agro-climatic zones of India and is expected to play a major role in future bioenergy schemes. In this study, fuel properties (basic density, calorific value, ash content, volatile matter content and fixed carbon content) along with elemental and gasification characteristics of *L. camara* stems have been investigated. Calorific value of *L. camara* was found to be 19.5 MJ/kg, whereas ash content, volatile matter content and fixed carbon content were 1, 80 and 19%, respectively. Gasification of *L. camara* was carried out using a 1 KWh gasifier, and composition of producer gas was evaluated. Based on experimental results, *L. camara* wood was found to be a suitable feedstock for energy conversion through gasification.

Keywords Ash content · Biomass · Calorific value · Gasification · *Lantana camara*

Introduction

Biomass is the most common form of renewable energy sources. The potential of biomass has been well recognized to meet the domestic and industrial energy requirements of India (Ravindranath and Hall 1996). Biomass fuels are cost-effective and suitable alternatives for fossil fuel. The environmental benefits of biomass fuel are now being recognized and valued. In addition to these benefits, biomass fuels also provide social benefits, such as the creation of additional employment, especially in rural areas (Ravindranath and Hall 1996). Among different biomasses, wood has received most attention because of its long and continuing precedent as a fuel. Primary solid biomass accounts for almost 10% of the

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world's total energy production (Sims et al. 2007). The percentage for developing countries is much higher, ranging from 13.5% of total energy production in Latin America to 19% in Asia and 26.2% in Africa (Stupak et al. 2011). Since there is limited supply of fuelwood from the forest, it is important to find out alternative available biomass resources for meeting energy requirement. *Lantana camara* is a scrambling aromatic shrub and is described as one of the worst weeds in the world (Bhagwat et al. 2012). In India, *L. camara* is distributed from Central Himalayas in the north and in the southernmost part of India (Kannan et al. 2013). It is widely available and dominating weed species which is reported as potential biomass source. One of the ways to make effective use of this weeds are to utilize it as a source of energy (Kumar et al. 2009; Kumar and Chandrashekar 2013; Prasad et al. 2001; Senelwa and Sims 1999).

Wood can be directly burned as firewood, or it can be converted into high-energy-content fuel through thermochemical and biochemical energy conversion techniques (Peter 2002a). Among different energy conversion methods, biomass gasification is one of the most efficient techniques. Biomass gasification technology has been effectively utilized by using locally available bioresources such as wood waste and agricultural residues (Dasappa et al. 2004). In gasification process, the dried biomass is converted into gaseous fuel by thermochemical conversion which is further purified and used in engine (Dasappa et al. 2003). Gasification involves partial combustion of biomass under controlled air supply, leading to the generation of producer gas constituting of combustible gases H_2 (20%), CO (20%), CH_4 (1–2%) and rest inert (Dasappa and Paul 2001). The energy value of producer gas is about 5.0 MJ/m^3 (Dasappa and Paul 2001).

The choice of energy conversion process is significantly affected by the type and quality of biomass feedstock (Peter 2002b). Therefore, knowledge on physical, chemical and elemental nature of the feedstock before its utilization is vital. In this paper, fuel properties (basic density, calorific value, ash, volatile and fixed carbon content) of *L. camara* have been presented. The chemical, elemental, gasification properties of *L. camara* were also investigated. The results presented here have the implication toward efficient utilizing *L. camara* for energy purpose.

Methodology

The test sample of *L. camara* was procured from nearby forest areas of Bengaluru, India. Samples were oven-dried to constant weight at 80 °C. The oven-dried samples were then chipped, hammer-milled and powdered to pass −40 + 60 mesh and used for fuel properties and chemical analysis. For gasification, samples were cut into small pieces and dried up to 11–12% moisture content. Basic densities (g/cm^3) of the samples (d) were determined using Eq. 1.

$$d = W_{\mathrm{OD}}/V_g \tag{1}$$

where W_{OD} is the oven dry mass of biomass and V_g is the green volume of biomass. Volume of freshly cut water-saturated samples was determined by mercury displacement method (Kumar et al. 2009). The powdered samples were pelleted, oven-dried to constant weight at 80 °C and burned in an oxygen bomb calorimeter (LECO AC-350) to estimate the calorific value. Ash and volatile matter were determined according to ASTM D5142, using a proximate analyzer (LECO TGA-701). The elemental parameters (carbon and hydrogen) were determined using a CHN analyzer (LECO—CHN-2000). The fixed carbon content (FCC) of the sample was estimated using following equation:

$$\mathrm{FCC}\ (\%) = [100-(\%\ \mathrm{Ash\ dry} + \%\ \mathrm{Volatile\ dry})] \tag{2}$$

In order to overcome the experimental and instrumental errors, experiments were repeated four times and the average values were obtained.

Gasification of *L. camara* was carried out by using 1 Kwh downdraft gasifier (M/S Ankur Scientific Energy Technologies Pvt. Ltd., Baroda, India). The gasifier consists of one wet scrubber, saw dust filter, fabric filter and blower. A known quantity (3 kg) of oven-dried wood samples was used for gasification. Blower was used to keep entire system in partial vacuum condition. The temperature in oxidation zone was maintained at 950–1100 °C, while the feed rate varied from 1 to 1.18 kg/hr. Composition of gas was measured continuously through online sampling from the gas stream which was directly connected to the gas chromatography (GC) (BG-1000, Mayura Analytical Pvt. Ltd., Bengaluru, India) instrument. The gas chromatography instrument was equipped with Hayesep A column (80–100 mesh, 3 m) and molecular sieve column with thermal conductivity detector (TCD) and flame ionization detector (FID). The proportions of combustible gases, i.e., carbon monoxide (CO), hydrogen (H_2) and methane (CH_4) gases were analyzed. In order to overcome the experimental and instrumental errors, experiments on gasification were repeated seven times and the average values were obtained.

Result and Discussion

Fuel Properties of Biomass

The basic density, calorific value and the data obtained from proximate analysis of *L. camara* are summarized in Table 1. The biomass having higher density is preferred as fuel because of their high energy content per unit volume and their slow burning property (Peter 2002b). The basic density of *L. camara* wood was found to be 0.55 gm/cm^3.

Table 1 Result on fuel properties analysis of *L. camara* wood

Species	BD (gm/cm^3)	Ash (wt%)	VMC (wt%)	FCC (wt%)	CV (MJ/kg)
L. camara	0.55 (±0.07)	1.2 (±0.1)	80.0 (±0.08)	19.2 (±0.02)	19.51 (±0.04)

Values in parentheses are standard deviation
BD basic density; *VMC* volatile matter content; *FCC* fixed carbon content; *CV* calorific value

Table 2 Ultimate analysis of *L. camara* wood

Species	Carbon (%)	Hydrogen (%)	Nitrogen (%)	Sulfur (%)
L. camara	46.9 (±0.01)	6.42 (±0.02)	1.04 (±0.01)	0.13 (±0.00)

The fuel quality is also affected by the amount of ash present in the biomass. The higher amount of ash in biomass makes it less desirable as fuel (Kataki and Konwrer 2001). The amount of ash content in *L. camara* wood was found to be 1.2%, which is similar to many wood species (Kumar et al. 2011). High volatile matter content meagerly contributes in heating value of a solid fuel. However, for energy production through pyrolysis and gasification, the use of feedstock with high volatile content is preferred (Mangut et al. 2006). The volatile matter content and fixed carbon content in *L. camara* wood biomass were found to be 80 and 19%, respectively (Table 1). The quality of fuel is known by the amount of heat (energy) generated from a unit mass of fuel (MJ/kg). The calorific value is one of the important parameters for differentiating one fuel from others. The calorific value of biomass is dependent on its chemical composition, i.e., cellulose, hemicellulose, lignin, extractives and ash-forming minerals (Demirbas 2001; Peter 2002b). The calorific value of *L. camara* was found to be 19.5 MJ/kg. The calorific value of *L. camara* is comparable to many wood species (Kataki and Konwrer 2001; Kumar et al. 2011).

The ultimate analysis gives the chemical composition of a fuel. The chemical analysis usually includes the carbon, hydrogen, oxygen, nitrogen, sulfur and ash content of dry fuel on a weight percentage basis. The result on elemental analysis of *L. camara* is presented in Table 2.

The amount of ultimate carbon and hydrogen in *L. camara* was found to be 46.9 and 6.42%, respectively. The H/C ratio calculated from the values given in Table 2 for *L. camara* wood was around 0.14. In a biomass fuel, higher proportion carbon–carbon bond is more desirable, whereas higher proportion of carbon–oxygen and carbon–hydrogen bonds reduces the energy value of fuel (Peter 2002b). Lower amount of nitrogen (1%) and sulfur (0.1%) in *L. camara* wood is favorable from environmental point of view.

Gasification Properties of L. Camara *Wood*

Biomass gasification is a chemical process that converts solid biomass fuel into useful convenient gaseous fuels by thermochemical reactions. During gasification, energy from biomass is released in the form of combustible gases, i.e., CO, CH_4 and H_2 (Dasappa et al. 2003). The process of gasification in a downdraft gasifier can be divided into different sub-processes. The first step involves exothermic reactions of oxygen in air with the pyrolysis gas under fuel-rich conditions (Dasappa et al. 2004). The second step involves the endothermic reaction involving gases such as CO_2 and H_2O with hot char, leading to product gases, namely CO, H_2 and CH_4 (Bhavanam and Sastry 2011; Dasappa et al. 2004). In the present study, a small-scale (1 KWh) downdraft gasifier was used for experiments. The TCD and FID gas chromatograms are shown in Figs. 1 and 2. The composition of producer gas (CO, H_2 and CH_4 content) was analyzed, and the results are given in Table 3. The retention time of CO and CH_4 was found to be 0.933 and 1.252 min, respectively. The retention time for H_2 was around 0.707 min (Fig. 2). The composition of CO, CH_4 and H_2 was found to be 9, 2.9 and 14.5%, respectively (Table 3). Typically, producer gas contains 15–30% CO, 10–20% H_2, 2–4% CH_4, 5–15% CO_2, 6–8% H_2O and the remainder N_2 (Sheth and Babu 2009). The lower amount of CO, CH_4 and H_2 found in this study can be attributed to the size of gasifier. The gas composition from gasifier varies from startup time until the temperature of the reaction zones in the gasifier stabilizes to their steady-state values. There after gas composition attains a stabilization. The composition of gas is also affected by the temperature of the reaction zone (Bhavanam and Sastry 2011; Dasappa et al. 2004; Sheth and Babu 2009).

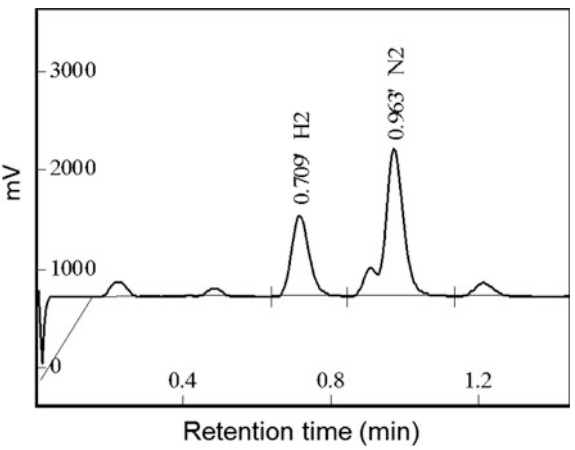

Fig. 1 TCD chromatogram profile of producer gas

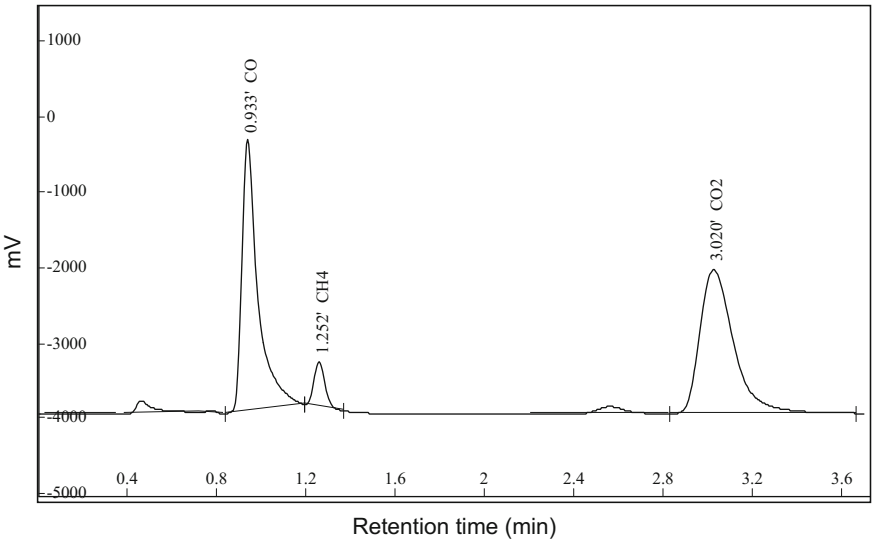

Fig. 2 FID chromatogram profile of producer gas

Table 3 Properties of producer gas from *L. camara* wood using small gasifier

S. No	Type of gas	Retention time (min)	Quantity (%)
1	Carbon monoxide	0.933	9.0
2	Methane	1.252	2.9
3	Hydrogen	0.707	14.5

Conclusions

High calorific value (19.5 MJ/kg) and low ash content (1%) were found in *L. camara* wood biomass. The presence of high volatile matter content (80%) and fixed carbon content (19%) makes this biomass suitable for gasification. Gasification of *L. camara* under small-scale (1 KWh) gasifier shows gas composition of 9% CO, 2.9% CH_4 and 14.5% H_2.

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Search for Future Fuels—Pathway Points to a 'Boring' Process

L.N. Santhakumaran

Abstract The article compiles information on the efforts so far attempted for the production of clean fuels from potentially vast and diverse sources, like agricultural produce, algae, and also from the enormous quantities of lignocellulose wastes (woody material, crop residue, etc.). Among the several feedstocks, investigated upon in different laboratories as alternative sources for biofuels, are oils from soybean, palm, rapeseed, coconut, sunflower, peanut, cottonseed, linseed and rice bran; seeds from jetropha, neem, poon, rubber, mahua and karanj; and also maize, tapioca, castor oil, fish oil, animal fat, tallow, oleaginous fungi, microalgae and aquatic weeds. Among these, investigations on jetropha have now progressed well to the extent of commercial production. Utilization of these materials, however, is bereft with one or the other limitations, especially the non-availability of an efficient cost-effective method for their conversion into fuel in place of the presently employed thermal, physical, chemical and biochemical methods. It is suggested that microalgae, water hyacinth (with all their advantages including the manifold higher yield) and waste biomass have the potential to help us to overcome the energy crisis, for which added inputs in biotechnological research, aimed at developing a cheap, technically feasible and economically competitive methodology, holds the key. In this context, the possibility of utilizing the powerful cellulose-digesting enzymes of the marine wood-borer, *Limnoria*, to effectively break down any lignocellulose materials into simpler glucose, deserves special attention. Utilization of raw materials, mentioned above, involving an innovative process for their conversion into glucose and ethanol, based on the efficient cellulase enzyme system of

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Limnoria, can be our best option for future energy security. Research in this direction, initiated in UK, has been summarized.

Keywords Biofuel · Microalgae · Aquatic weeds · Lignocellulose · Marine wood-borer · *Limnoria* · Cellulase

Introduction

The purpose of this article is to pinpoint the potential of developing novel technology for the production of biodiesel from waste wood resources, including all types of non-edible cellulosic materials, using methodology, which may appear as a strange aberration from the existing ones. The subject assumes all the more importance in the present day context of global warming and climate change, impacted by several anthropogenic activities, including excessive burning of petroleum fuels. The search, therefore, for a renewable, clean, environmentally acceptable green alternative for the soon-to-be exhaustible fossil fuel, through innovative approaches utilizing hitherto unexploited feedstock, is on all over the world with added impetus.

What are the existing options before us, while driving this thrust towards new biofuel production technologies? In nature, it took several millions of years to convert various biomasses, including woody materials and algae through different processes, such as compression and heating, into crude petroleum oil, which we refine and use as fuel. Since exploitation and excessive use of these exhaustible natural petro-fuels have almost reached a point of no return, leaving at the same time its deleterious effects on the environment, our immediate priority is to hopefully produce the next generation of clean energy as alternatives *within a short period,* so as to reverse the otherwise aggravating climatic catastrophe. There are several methodologies available through physical, catalytic, biological and biotechnological processes to produce clean fuels utilizing the enormous quantities of waste biomass (unwanted woody materials including wood shavings from logging and sawmill, and discards from lawn and tree maintenance activities, non-edible plant residues such as wheat stems, corn stalks and leaves, municipal solid wastes), aquatic weeds and algae. But each technology is bereft with one or the other limitations, and there is an urgent need to develop a nearly perfect, cost-effective method, so as to ensure sufficient supplies of clean energy for the future—one of society's daunting challenges. This is possible only with an increased commitment for sustained investigations on the subject. The present scenario is that the commercial production of advanced second-generation biofuels is still far in the future.

Feedstock and Technology Available for Biofuel Production

Among the agricultural produce, investigated upon as alternative feedstock for biofuels are oils from soybean, palm, rapeseed, coconut, sunflower, peanut, cottonseed, linseed and maize. In addition, at least another 22 potential alternatives, which *inter alia* include neem (*Azadiracta indica*), poon (*Calophyllum inophyllum*), rubber (*Hevea brasiliensis*), tapioca (*Manihot utilissima*), mahua (*Madhuca indica*), karanj (*Pongamia pinnata*) and the now popular jatropha (*Jatropha curcas*), have also been studied in detail (including compatibility with present day engines) and results are available in several publications (Razon 2009). Considerable research has already been completed on these, but a viable methodology has not been evolved yet, questioning their practical application. Variation in oil content, availability and collection of seeds and its steady supply in large sustainable quantities are all unfavourable factors against producing second-generation biofuel from these feedstocks. Similarly, production of biodiesel from tapioca, which is a staple food for poor people, will also be ethically unsound, unless ambitious large-scale farming using high-yielding varieties is employed to produce surplus quantities. In fact, search for alternative biofuel sources is so aggressively pursued that preliminary screening of several herbs, shrubs and trees is being undertaken in various laboratories, subjecting them to initial physico-chemical tests.

While feasibility studies on most of the above alternative feedstock are still in a very preliminary stage, investigations on jatropha have remarkably progressed to the extent of production in a limited commercial scale. In fact, biofuel development in India centres mainly around the cultivation and processing of jatropha seeds, which contain 40% oil. However, despite the tremendous possibility offered by jatropha, its rational utilization is subject to expansion of its cultivation, aimed at higher consistent yields per unit area through development of improved planting stock. Because, only 500 litres of biofuel can be manufactured from 1 ha of jatropha plantation, that too, if the yield of seeds is at least to the extent of 3 tonnes. This is too meagre, when compared to that of microalgae. Again, jatropha takes two to three years for commercial yield, and the production of seeds is not continuous, so as to ensure a steady supply. Hence, it is reported that jatropha seeds yield little hope for India's oil dreams (Orange 2009). However, other long-term environmental benefits that can be derived from extensive plantation of jatropha are indeed favourable factors, while assessing its preference as a profitable resource for biofuel. Nevertheless, it is still argued that biofuel production from the above-mentioned feedstock has the following general disadvantages: (1) attempt to boost feedstock production by expansion of cultivation will clash with land use for various agricultural crops; (2) genetic modification and biotechnological investigations to introduce improved planting stock are to be augmented. Acceptability of genetically modified crop is again another sensitive issue; (3) although change in the designs of existing engines is not required when biodiesel is used to blend up to 20%, its exclusive use warrants modification in diesel engine design; (4) while use of raw vegetable oils combines cost benefit with environmental advantages,

necessity for engine modifications makes its use impractical. At the same time, conversion of vegetable oils to biodiesel to overcome the problem of engine compatibility is prohibitively expensive; (5) most importantly, the ethics behind using food for human consumption as fuel are of grave concern; (6) the inadequacy of raw material. It is reported that conversion to biodiesel of all vegetable oils, produced globally in a year, can meet only 18% of the demand for fuel for the transportation industry alone (Razon 2009). Even if we are to utilize the millions of tonnes of lignocellulosic agricultural wastes, available every year as untapped resources, the cost of its conversion into fermentable sugars virtually defeats the purpose, because it involves more difficult, yet to be perfected, processes.

Utilization of algae as an alternative feedstock for the production of biodiesel is being attempted by scientists in several laboratories all over the world for quite some time now, but a viable methodology has still eluded them or yet to be improved upon (Gouveia and Oliveira 2009). As early as 2008, the Fisheries College and Research Institute, Tuticorin, India, had succeeded in extracting biofuel from marine algae using 'transesterification' method (conversion of an organic acid ester into another ester of that acid) involving catalysed chemical reaction on oil from microalgae (Anonymous 2008a, b). They have reportedly perfected the protocol in this connection, perhaps a pioneering step indeed. Similarly, in 2010, the Central Salt and Marine Chemicals Research Institute, Bhavnagar, initiated work on preparation of biodiesel from marine algae, and within two years, they not only successfully developed technology to extract biodiesel from algae, but also conducted the test-drive a Chevrolet Tavera on 30-03-2012, using but 10% of the new fuel (named 'B-100 Microalgae Biofuel') in petrol. However, the cost of production was prohibitively high (www.dnaindia.com/india/report-marine-algae-yield-precious-iodiesel-1677059). It is reported that a US Patent has been granted for this technology and some manufacturers would be testing and evaluating their most advanced and futuristic automobile engines on this new alternative fuel. However, several major problems encountered in this process, such as (i) selection of the most viable algal species for extraction, (ii) techniques for mass production of algae for continuous supply of feedstock in huge quantities, are still to be overcome, and therefore, the new biofuel is expected to hit the market in only five to ten years. Moreover, the technology has to be made economically feasible and streamlined so as to reduce the cost of production (Sachan 2012). According to Chisti (2007, 2008) and Gouveia and Oliveira (2009), the yield of oil from microalgae, with 70% oil by weight in its biomass and cultured in 1 ha, is sufficient to produce 136,900 l of biodiesel in a year. Another estimate is that 7.5 billion gallons of biodiesel can be produced from 200,000 ha (780 km^2) of desert land (Zhang and Hu 2011). In fact, some of the microalgae, like *Botryococcus braunii*, have amount of hydrocarbon up to 85% of its dry weight. Thus, algae, grown in an area equal to half of the State of Mexico, would be enough to meet the energy requirements of the entire USA, or oil extracted from algae cultured in about 1% of India's total land area can render the country self-sufficient in liquid fuel, ensuring energy security and several other economic benefits. Advantages of microalgae as a feedstock for biofuel are: (1) its remarkable ability to mass multiply (increased biomass yield per hectare) assuring a

steady supply of algae within two to three days and, thereafter, it can be harvested every day; (2) it does not compete for land with other crops; (3) it is carbon neutral (quantity of CO_2 absorbed by the algae during photosynthesis is equivalent to the amount of CO_2 produced while burning, thereby maintaining CO_2 equilibrium); (4) it utilizes freely available light energy, converting it into a natural biomass oil product equivalent to crude oil; (5) the product is free of sulphur; (6) most importantly, the fuel from algae can be directly used in cars, airplanes, etc., without any additional modification to the existing designs of the engines; and (7) residual de-oiled cakes are excellent source of high value protein that can be supplemented as a cattle feed. At the same time, their disadvantages/constraints are: (1) need further careful studies for standardization and optimization of mass culture technique by developing ideal protocol for factors, such as nutrients, pH, salinity, temperature, light intensity, in the growing ponds, and for subsequent harvesting and extraction; (2) improvements to algal biology through genetic and metabolic engineering are to be taken up; (3) maintenance of large-scale monoculture in special photobioreactor and prevention of bacterial infection add up to operational costs; (4) use of energy-intensive fertilizer; (5) separation of water from algal cells involves high-energy inputs; and (6) solvents used for retrieval of oils from the algal biomass lead to a limited degree of pollution. Nevertheless, the very fact that the quantity of biodiesel produced from microalgae can be 10–20 times higher than the yield from seeds and/or vegetable oils renders the microalgae feedstock the best option as a source for biodiesel, provided a viable methodology is developed for the production.

Another important feedstock, available in abundance, is the widely distributed aquatic weed, water hyacinth (*Eichhornia crassipes*)—a noxious plant known for its prolific biomass production. Dense matting of this fast-spreading weed makes the ecosystem less fertile by preventing penetration of light, depleting oxygen and nutrients leading to anaerobic conditions. Obstruction to navigation and irrigation systems and to fishing activities is other harmful effects caused by this weed, in addition to providing ideal breeding ground to vectors of human and animal diseases. The prodigious fecundity of the weed renders its eradication by physical, chemical and biological means virtually impossible, despite the enormous amount of money spent annually all over the world in its control. Hence, water hyacinth is subjected to extensive research for its potential for economic utilization in areas, such as food and energy source, mushroom cultivation, phytoremediation of heavy metals, biogas and biofuel production, manure, medicine and several other applications (Pawar and Haram 2011). Naturally, the prospects of producing biofuel from water hyacinth are also being pursued in several laboratories in various countries. In addition to all the advantages it shares with microalgae as an ideal bioenergy crop, water hyacinth has the following unique benefits too: (1) its prodigious fecundity and amazing biomass production rate ensuring continuous availability of raw materials in nature without any additional efforts; (2) its high cellulose and low lignin content—a favourable combination for an efficient biofuel feedstock (Bhattacharya and Kumar 2010); (3) access to easy harvesting and requirement of minimum preconversion treatments; (4) unlike microalgae,

development of an elaborate protocol for its mass production under controlled conditions is not required; (5) no additional area is necessary for cultivation; (6) economic utilization of a material, hitherto considered a menace and harmful, as clean energy with all its associated socio-economic and environmental benefits; and (7) efficient eradication of an invasive plant of extreme nuisance value. However, investigations are still on to improve the pretreatment methods with a view to developing cost-effective techniques for fuel production (Sierra et al. 2008). Although some degree of success has been achieved for conversion of water hyacinth to liquid ethanol by acid hydrolysis and yeast fermentation (Chartchalerm and Tanawut 2007; Pillai et al. 2009), search for methods to significantly increase ethanol yield is still continued so as to reduce cost of production.

To sum up, it may be mentioned that, although endowed with several desirable qualities, such as renewable resources, biodegradability, superior lubrication and, above all, environmental acceptability being non-toxic and with minimal emissions of greenhouse gases, considerable research and technology development are still involved in conversion and effective utilization of various biodiesel feedstock as alternative energy, retaining the characteristics and requirements of fuel properties. Excessive use of fossil fuel and the resultant irreversible changes to the already over-exploited and stressed environment make it imperative that we should further accelerate our activities to unravel potential resources and methodology for the production of alternative harmless energy. This requires enormous research inputs. Results so far achieved are encouraging, and several of the alternative feedstock, discussed above, especially the marine algae, water hyacinth and agricultural wastes, have the potential to overcome the energy crisis. Though the resources are diverse and abundant, the development of still cheaper, technically feasible and economically competitive methodology for their conversion to biofuels actually holds the key for its success.

Concept of a Viable Technology for Biofuel Production, Based on Cellulose-Digesting Ability of Marine Wood-Borers

At this juncture, a novel research project, that is being undertaken in UK to elucidate the cellulose-digesting mechanism of marine wood-borers and to utilize the results on the action of their powerful enzymes in effectively breaking down any trash wood into simpler glucose to be subsequently further fermented to ethanol, deserves special attention. It is indeed an innovative approach, pursuit of which may appear bizarre, but certainly holds great possibility to develop much cheaper procedure to produce fuel from waste wood or any such cellulosic materials, thereby fundamentally changing the economics of producing alternative biofuel. The concept is briefly described below.

Marine wood-borers (shipworms, piddocks, pill bugs and gribbles) are present in almost everywhere in oceans and estuaries, causing extensive damage to underwater timber structures including fishing craft. They are ruthless in their ability to destroy any type of timber species and are, therefore, economically a very important assemblage of organisms for any country having an extensive coastline, using large quantities of timber to provide infrastructure facilities for an ever-expanding culture and capture fisheries. The borers bring about damage to timber with remarkable rapidity, and even teak and such timbers, accredited for their high natural durability under terrestrial conditions, cannot defy decay in marine borer infested localities, even for a period of 6–8 months (Santhakumaran 1997). During this short time, the timber will be riddled by innumerable individuals, resulting in its complete internal destruction, impairing the very strength of the wooden structures (Figs. 1 and 2). *Significantly enough, some members of these pests (shipworms and gribbles) are endowed with enzyme systems capable of breaking down complex wood constituents (celluloses, hemicelluloses and lignin) and utilizing them as food*. While source of enzymes for this unique function in shipworms is still a debated issue, it has been conclusively proved that gribbles (Genus *Limnoria*) themselves produce exceptionally powerful wood-digesting enzymes in their gut (King, et al. 2010).

Generally, most of the lignophagous organisms can exploit wood for sustenance, if only the wood is already degraded to a readily digestible form by fungi and

Fig. 1 Keel and side planks of a teakwood boat, showing severe destruction by marine wood-borers. Note the intensity of infestation. (Locality: Panaji, Goa)

Fig. 2 Severe surface destruction of wooden test panels by *Limnoria* within eighteen months. (Locality: Mumbai Harbour)

bacteria or, as in the case of termites, with the help of cellulase-producing symbiotic microorganisms in their digestive tract. On the other hand, *Limnoria* gut is sterile, devoid of any resident microorganisms, but still, the borer has the unique ability to break down complex wood ingredients. It is really a remarkable accomplishment because, though wood is an excellent source of energy, its constituents (cellulose, hemicelluloses and lignin) are apparently resistant to enzymatic action. Glucose in cellulose is in the form of crystalline microfibrils and are again attached to hemicelluloses, making it inaccessible to intercept by enzymes. On the other hand, hemicelluloses harbour a variety of sugars rendering them a complicated system to be converted into a single product (say ethanol). Further, these two constituents are well protected by lignin—a sturdy complex mass of polymers, cross-linked and strongly bonded to each other and are not easy to be broken down by chemicals. The complex process of converting lignocellulosic materials into biofuel, therefore, involves pretreatment with heat and powerful chemicals to release the cellulose-hemicelluloses mass from lignin for subsequent enzymatic action to liberate glucose for further fermentation to ethanol.

Under these circumstances, how best can we adopt this ability of *Limnoria* to our advantage? In their search for new and efficient enzymes for lignocellulose saccharification to reduce costs of liquid biofuel manufacture, scientists of the University of Portsmouth and University of York in UK are probing the potential of digestive enzymes of *Limnoria* to process and produce biofuel from *non-food* plant biomass. The discovery and isolation, by their collaborators in Institute of Biomedical and Biomolecular Sciences (IBBS), of a 'cDNA encoding ab-1,4-endoglucanase (called cellulase) in *Limnoria* open up the opportunity of using this organism as a model for investigating the animal-wood breakdown mechanism in the absence of microbial enzymes'. According to them, this unusual nature of lignocelluloses digestion in *Limnoria* holds great potential for gaining new insights in cheap methodology to break down wood cellulose into energy-rich sugars that can be fermented into alcohol-based fuels for the development of second-generation fuels, a procedure much closer in its nature to current industrial systems (a 'gribble-like Processing Plant'). The transcriptome of the *Limnoria* gut has been determined by them by deep sequencing, and an entire suite of hydrolytic enzymes, involving about 60 genes, have also been discovered and patented. The borer species, they have worked with is *Limnoria quadripunctata* Holthuis (King et al. 2010).

In this context, it is appropriate to examine the possibility of undertaking similar studies in India, employing *Limnoria* species available along the coast. So far, nine species of *Limnoria* and about 40 species of shipworms have been reported from Indian waters (Santhakumaran 2005). Of these, *Limnoria platycauda* Menzies is well established along Karwar coast and indigenous *Limnoria indica* Kampf and Becker (Fig. 3) along Mumbai, Tuticorin, Chennai and Visakhapatnam coasts in India. The borers can be collected using wooden test blocks, cultured under laboratory conditions and subjected to further biochemical/biomolecular investigations mentioned above.

It may be concluded that formulation and active pursuit of a Project, in the above pattern and goal, may turn out to be quite rewarding and is crucial in our hunt for alternative biofuel, in the wake of the already fast depleting natural energy resources. Development of an efficient cellulose-ethanol technology, with controlled production cost, supplemented by sources of feedstock mentioned above, particularly microalgae, water hyacinth and lignocellulosic wastes, can collectively support us in our hunt for alternative fuel. A combination of microalgae or water hyacinth (or even agricultural wastes) as resources and a revolutionary process based on the powerful Limnoria cellulase enzyme system to break it down to glucose and ethanol can be our best option to implement this Policy. However, considering the limitations of microalgae as raw material, enumerated elsewhere in this article, water hyacinth holds better prospects as a potential feedstock. Such a methodology, if successfully developed, can also be effective on any type of woody and plant materials including macroalgae, thereby eliminating the elaborate inputs in labour and techniques involved in microalgae culture on a mass scale, and would certainly turn out to be a boon for our future energy security. However, as mentioned by Anderson (2011), '*the biggest hurdle faced by these second-generation*

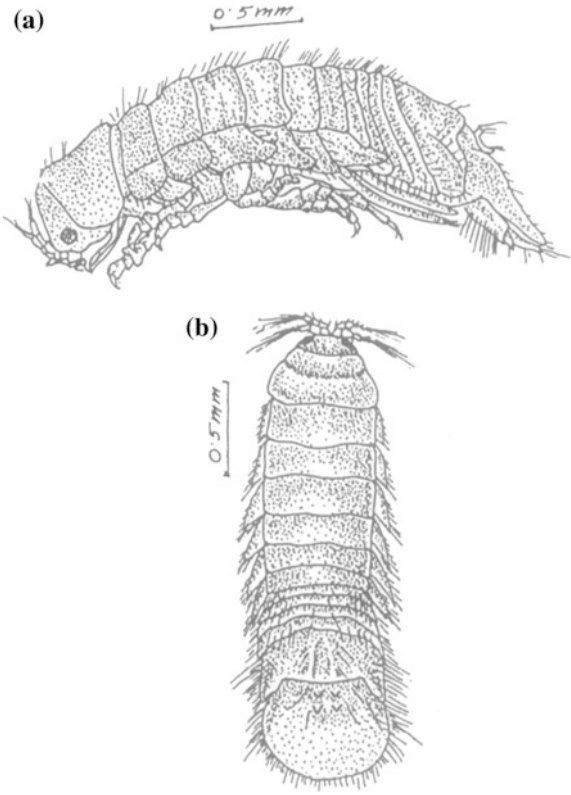

Fig. 3 *Limnoria indica*. **a** Lateral view; **b** dorsal view

biofuel technologies is not a dearth of clever science. It is the lack of brave investors willing to take a gamble on a new technology…'. The thrill and excitement in scientific research lie in treading uncharted paths of uncertain outcomes and, in the end, getting rewarded for successfully unravelling any extraordinary approaches for human welfare and security.

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Wood Use in India—Readying for that Elusive Renaissance?

K. Satyanarayana Rao

Abstract Promoting the forest product sector in general and Wood Forest Product (WFP) sector in particular in India is a highly complex mission burdened with conflicting interests. The WFP industry needs not only to balance the demand–supply chain, a daunting task by itself considering the immense pressures and raw material shortages, but also to develop efficient tools and mechanisms required for its sustainable development. While new opportunities are emerging for this sector due to the current global revival of interest in recognizing/rediscovering the values of wood, especially the environmental advantages inherent in its production and usage, the changing scenario is also throwing up fresh challenges that have to be met. This paper highlights the main issues that need to be immediately addressed at different levels that includes concerns regarding assured raw material supplies from sustainable sources (labelled and certified wood) at affordable prices, poor utilization of available technologies resulting in widespread unscientific utilization, wastage of resources and leakage of R&D and extension efforts and the lack of reliable scientific data to validate the environmental advantages of usage of many harvested wood products. Enunciation of a 'National Wood Use Policy' that aims at balancing 'market' and 'non-market' values of wood use and promoting an enabling, environmentally sound operational regime at all levels is advocated. It is argued that such a strong policy response is needed at this critical juncture to address complex issues that cannot be solved at any single level. This would be an important and much anticipated development that could revitalize the sector and pave the way for even a possible renaissance in India's amazing wood culture.

Keywords Labelled and certified wood · Leakages of R&D efforts · Unscientific utilization · Environmental advantages · National wood-use policy

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Introduction

Notwithstanding the many complex issues that adversely affected a healthy development of the Wood Forest Product (WFP) sector in India, the country continues to sustain its vibrant wood-use culture and is even optimistic in its outlook about promoting it as a favoured material for future use as well. This sentiment is reflected in all the International Conferences, National Seminars/Workshops on wood the country has organized during the last five years. Main drivers of current optimism about wood use appear to be: the many technological innovations in Wood Science and allied fields that could be synergized; breakthroughs in identification of tradable commodities for environmental services in markets/market-like arrangements (e.g. carbon trading); opportunities arising out of International Treaties/Agreements/Initiatives and a worldwide revival of interest in recognizing its aesthetics and other 'option values'.

Concerns about sustainability, biodiversity conservation and climate change are issues currently dominating the environmental debates and policies leading to rapid changes in the way wood is produced, traded, supply chains managed and processed. In a series of papers, Satyanarayana Rao (1998, 2001a, b, 2002a, b, c, 2004, 2005, 2011, 2012) discussed in detail the current scenario of wood utilization in India, strengths and weaknesses of the wood forest sector, the emerging and missed opportunities, importance of developing Institute—Academia—Industry partnerships, certification and labelling of teak wood products, promoting technology based utilization by small and medium-sized enterprises (SMEs), changing role of Forest Product R&D Institutes, bioethics and sustainable development of Forest/Forest industry. Satyanarayana Rao and Remadevi (2006) dealt with problems associated with bio-invasions from imported wood and implementation of W.T.O's Sanitary and Phyto-sanitary Agreement (SPS Agreement) in India. These studies clearly indicate that a range of issues are needed to be addressed and several difficulties overcome in achieving a transition to a more environmentally sound, responsible regime of operation in this sector.

This paper highlights some of the identified areas that need immediate attention at three different levels—(1) Research, research management, research policy and product development; (2) technology transfer/extension, market development and (3) policy support.

Issues Involved and Responses Needed at Different Levels to Enable Wood Product Sector to Harness Emerging Opportunities in India

Research, Research Management (Research) Policy and Product Development at the (R&D) Organization Level

India has an over a century-old Forest Products Research tradition sustained by a number of research institutes functioning under the umbrella organization of the Indian Council of Forestry Research and Education (ICFRE), Ministry of Environment, Forests and Climate Change (MoEF&CC), Ministry of Industries, State Forestry Research Institutes and Forestry Colleges in Agricultural Universities. In recent years and in a limited way, some newly developed industrial laboratories in the private sector are also undertaking this research. Contributions of the Indian Institutes in the field of Wood Science and Technology are well acclaimed and include some remarkable world-class innovations (Anonymous 2006). However, it may be noted that the studies that highlight the environmental advantages of wood and engineered wood products have not received much attention. Of particular significance in this context are the absence of life cycle analysis (LCA) studies and carbon footprint studies for several forest products. LCA studies are needed to validate assumptions on sustainability, as they take into account all the environmental impacts from cradle to grave, i.e., from extraction of timber to the end of product disposal. Carbon footprint assessment studies are needed to generate accurate data that determine the particular wood products capacity to store carbon (extend lock-in periods) and, thereby, its potential to generate carbon credits. Evolving National policies and guidelines for service life prediction of products in different conditions is an essential prerequisite for carrying such studies and remains a major challenge.

At the Research Management and (Research) Policy levels of R&D Institutes/organizations, greater emphasis needs to be placed on an 'eco-efficient approach' in developing processes and products, paying special attention to hitherto neglected aspects like designing, finishing and polishing. Social/socio-economic and market research are other areas that need to be strengthened. Research management and policy also needs to be proactive and supportive of fostering 'knowledge partnerships', interface research and needs to be open to acquiring catching up/or even 'imported' technologies in frontier areas to achieve 'technology leap frogging', where deemed necessary. Suitable mechanisms need to be devised for smooth and effective implementation of such arrangements.

Technology Transfer/Extension/Market Support—Issues Requiring a Cross-Sectoral Approach

Glaring gaps exist between generation, availability (to the user groups) and absorption of wood technologies—a matter of serious concern resulting in huge leakage of efforts and importantly, widespread unscientific utilization. This is particularly conspicuous in operations of the large number of small processing units, most of them in the unorganized sector that consume bulk of timber. Very few studies were undertaken to understand the reasons for such low rates of technology absorption and offer remedial solutions. Much of the technology transfer/extension effort at the R&D Institute level was expended on preparation of technical and semi-technical literature (pamphlets, brochures, bulletins and other publications) and in a few cases, video films, films and in organizing demonstrations, exhibitions workshops/seminars/symposia. While these are important components of any promotional effort, general information such as the volume of the available resource [in case of promotion of a lesser used species (LUS)], sources of reliable supply, cost, potential markets, should also be provided. An effective strategy to introduce a lesser used species or a new processing technique should ideally integrate promotional and marketing strategies which should be institutionalized (Eastin and Wright 1998). Painstaking research by Indian R&D institutes demonstrated that by upgrading/improving the technical characteristics by simple indigenously developed and available techniques, over 50 Indian species could be utilized as alternatives for traditionally favoured species that are either in short supply or highly priced (Anonymous 2006; Rao et al. 2003). This is a major contribution which helps in widening the choice of species for an array of end-use applications. It is annoying to note that needed breakthroughs could be made only in the case of better utilization of rubber wood, poplars and to some extent, eucalypts (in northern India). Achieving wider acceptance of usage of improved timber of other species is a major challenge that needs to be overcome. Since it is a market-creating activity, with all its inherent difficulties, a stepwise, cross-sectoral approach involving market participants—producers, suppliers—importers, traders, processors, distributors (retailers), product manufacturers (local industry)—end users, besides extension staff from the R&D Institutes and managers needs to be evolved, for promotion of each new process or hitherto un-utilized timber species.

John Joseph (2006) considers lack of perception about the capacity, capability of the target groups is a major factor adversely affecting technology transfer efforts as many of the target groups lack resources to meet the incremental investments. Satyanarayana Rao (2012), therefore, advocated ‘hand-holding’ arrangements in the initial stages for SMEs and even ‘performance based incentives’.

The situation, therefore, calls for greater efforts in social/socio-economic and market research and support.

Policy Issues and Prescriptions—At the National Level

Wood products scenario in India has been witnessing several changes during the past three decades. These include logging bans (1996, 1997), liberalization of wood imports (1999) sourcing of domestic raw materials mostly from plantations/trees outside forests, resulting in alterations in species matrix for industrial use, new trends in consumption patterns due to lifestyle changes and requirements of compliance with the standards and terms arising out of international treaties/agreements, etc. (Satyanarayana Rao 2011, 2012). In the past, Indian wood industry withstood competition from an array of other materials—both traditional and modern and forestry itself, in a broader sense, faced several challenges, which were deftly handled by policy makers. The current scenario also calls for a quick response to overcome the difficulties in making a transition to a more environmentally sound regime of operations by the industry, for promoting 'responsible trade' and other elements of sustainable development. Policy support is needed, as a first step in this direction, to enhance the domestic base for supplying eco-labelled wood sourced from certified forests/plantations. The area under FSC forest management unit certification is just 644 ha of private rubber plantations in Tamil Nadu, and the number of chain-of-custody (c-o-c) certified units is a mere 188 (Figures in 2011). These need to be greatly augmented. It is important to note in this context, India does not have a scheme of its own for 'eco-labelling' or 'chain-of-custody' (c-o-c) certification.

It has also to be kept in mind that the increased demand for certified wood may affect the economic prospects of several farm forestry/agroforestry areas as well as certain industrial establishments (e.g. wooden handicraft industry) (WWF India 2011). In general, while there are many supply-sided initiatives, it is to be noted that the focus had been mainly for species used for paper, pulp, fibre and, to some extent, panel products. Plantations for hitherto neglected use categories, especially the socially important species, need be encouraged.

It is paradoxical to note that even while, the National Forest Policy, 1988 (Government of India 1988) recognizes 'utilization' as a priority area, the focus, so far, had been mainly on 'how much produced' and 'how much removed' or 'supplied'. India's National Forest Certification Initiative—the Bhopal-India Process(B-I Process)—deals with utilization aspects under the criteria 6 'optimization of forest resource utilization' and the indicator 6.3 takes into account 'efforts towards reduction in wastage'. To advance 'technology based utilization', vital to promote sustainable wood industry, the all important factor of 'How it (the resource) is utilized' also needs to be accounted and accommodated. As this requires a cross-sectoral approach as noted earlier, and no single agency can effectively deal with it, a major policy thrust is needed to catalyse the different stakeholders to get over their reluctance to adopt new technologies and utilize more improved timber from lesser-known species.

India is a signatory to several important International Agreements/Treaties and Arrangements. The advent of REDD (Reducing Emissions from Deforestation and

Forest Degradation) and REDD + (that includes, in addition, Sustainable Forest Management (SFM) and especially the provisions for conservation and enhancement of forest stocks/carbon sinks in developing countries, initiatives are expected to open up new opportunities for promoting harvested wood products and wood industry. Sensitizing the stakeholders on the implications of these initiatives and enabling them by providing necessary infrastructure and environment are important to make timely adjustments/course corrections.

HRD, education and training issues have not received the attention they merit resulting, especially in the acute shortages of skilled manpower in the manufacturing segment. Innovative approaches like pooling talents from unconventional sources, lateral induction and, importantly, developing/strengthening 'partnerships' may be necessary to improve the position.

As the issues that need to be addressed to promote wood as a sustainable material in India as mentioned above are highly complex as detailed in the foregoing account, the situation necessitates enunciation of a 'National Wood Use Policy', as voiced at several fora (Satyanarayana Rao 2003). Balancing the market and non-market objectives promoting technology-based utilization and creating a favourable operational regime that enables harnessing emerging opportunities could hold the key for a healthy growth and revival of this vital sector (Ganguly 2000; Satyanarayana Rao 2002a, 2011; Satishkumar 1999).

Conclusions

India continues to sustain its amazing wood-use culture, despite mounting pressures on its forests, conflicting interests, missed opportunities and the ever-changing needs. Keeping in tune with the current global trend, India, too is aware of the importance of promoting wood as a sustainable material and is optimistic in its outlook. To realize the same, however, the country needs to address several issues at different levels of operation. The paper discusses some of the important areas that need immediate attention at the R&D, technology transfer and policy support levels. These include in the main, stepping up R&D to highlight environmental advantages of usage of wood (e.g. LCA and carbon footprint assessment studies of harvested wood products), developing an effective strategy to promote technology based, rational utilization of timber and create an enabling climate for responsible trade and an environmentally sound operational regime.

Indian Forest Policy handled several daunting tasks in the past and always avoided being overcome by the seeming hopelessness of situations. With suitable adjustments in operations and where necessary, policy prescriptions, it is argued, India is in a position to revitalize the wood forest sector leveraging its natural advantages (of having over 3000 timber yielding species, including several high-value tropical hardwoods), strengths established over the years (chain of R&D Institutes); a long tradition of forest management, to harness the emerging opportunities. Enunciation of a 'National Wood-use Policy' at this important juncture to

meet the current challenges would be a welcome step in the right direction to ready the Nation for a possible renaissance in its wood-use culture and further the good in wood.

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Part V
Wood and Climate Change

Wood is Good for REDD+!

Ederson A. Zanetti

Abstract My Home my life Program—PMCMV (from Portuguese: Programa Minha Casa Minha Vida) is a Brazilian Federal Government initiative to reduce country's housing deficit. The Ministry of Cities is responsible for the program. The use of wood in housing construction, notoriously those from native species, is regulated by Ministry of Environment. By Decree 318/2014 from Ministry of Cities and Decree 27/2014 from Ministry of Environment, the use of close to 50 different native species' Dead Wood and Harvested Wood Products from sustainable sources became eligible for appliance within My Home My Life Program—PMCMV at Northern region—Amazon region. Housing deficit within the region is estimate between 500,000 and 5 million units considering 2014–2030 time frame. Comparing traditional cement–iron models as baseline scenario and the approved wood model as Smart Growth using IPCC 2006 guidelines, industrial (construction) and AFOLU (REDD, REDD+ and HWP) sectors can generate carbon credits from this technological development influence. This project activity can be included within Brazilian voluntary emission's reduction goals for Amazon region, REDD and REDD+. The inclusion of PMCMV wood housing project activity could generate between 40 million and 400 million tCO2e reductions depending on methodological approach, between 7 and 70% of Brazilian voluntary REDD and REDD+ goals for Amazon region. To make this potential effective, the Ministry of Environment and Ministry of Cities need to implement an adequate MRV methodology for managing carbon along wood houses production chain and develop a PoA project document. At the Brazilian Amazon, Federal Government is promoting the concept that: Wood is Good for REDD!

Keywords Amazon region · Carbon · Dead wood · Harvested wood products · Housing deficit

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Introduction

Normative 318/2014 (Ministry of Cities) and Normative ICMBIO 27/2014 foresee appliance of Dead Wood and Harvested Wood Products for "My Home My Life Program" within Northern Region. Social housing deficit at the region is estimate between 500,000 and 5 million units over 2014–2030. These two Federal regulations created an institutional environmental carbon credits friendly, for both industrial (construction) and AFOLU (REDD+) sectors, as prescribed by IPCC (2006). This project activity can help accomplishing Brazilian voluntary emissions reduction goals within the framework of fighting deforestation and forest degradation at the Amazon, REDD and REDD+. Overall, the inclusion of social wood housing from PMCMV can generate between 40 Million and 400 million tCO2e emissions' reduction, some 7–70% of Brazilian goal for the sector within the Amazon. To make this potential reality, it is necessary to implement an appropriate MRV methodology to follow carbon behavior along social wood housing models' production chains within the region, and to develop a Program of Activities Carbon Project Document.

HWP and Carbon Accounting for REDD and REDD+

With the aim of reducing global GHG emissions, UNFCCC includes forest management and increase of standing stocks project activities among innovations for tropical forests income, social including and environmental protection, the so-called REDD = Reduction of Emission from Deforestation and Degradation (conserves the carbon stocks) and REDD+ = Increase on forest cover, conservation and sustainable forest management (more carbon) and the HWP = Harvested Wood Products. According with FAO (Food and Agriculture Organization FRA 2015), Deforestation means: "changing on land use with reduction of tree crow cover below 10% by hectare" while Degradation is translated as: "change between forest classes (e.g., from "close" to "open") which negatively affects the site and, in particular, reduces its productivity capacity". Intergovernmental Panel on Climate Change (IPCC 2006) guidelines for GHG inventories from different sectors holds Chap. 4 to AFOLU (Agriculture, Forests and Other Land Uses), including accounting procedures for Dead Wood—DW—and Harvested Wood Products—HWP. Within IPCC (2006), Dead Wood (DW) is classified as all kinds of branches, leaves, roots, dead trees and other types of biomass not included as litter or soil. Harvested Wood Products (HWP) are all wood material leaving project activities boundaries—other materials remaining within boundaries are to be accounted as DW. Because of this, wood used within project activity boundaries for fencing, furniture, construction, energy and others must be accounted as DW when determining forest areas carbon sequestration and storage, including from those without a formal Sustainable Forest Management Plan—SFMP. At those areas holding

SFMP, the rule is the same regarding DW, and besides this logs, timber, firewood and others imports and exports are also to be accounted for as HWP.

Methodological issues, UNFCCC project activities related, determined some basic aspects for project activity implementation, including: Estimates and Monitoring; MRV methodology which are robust and consistent and; applicability of the MRV methodology according to IPCC.

Project activity geographical boundaries will determine DW or HWP accountability. REDD project activities incorporate DW to contribute for estimates and monitoring robustness and consistency, according to IPCC (2006). Within a given forest project activity boundary, for example a Brazilian Federal Conservation Unit, DW includes biomass used for fencing, house construction, furniture, energy and others. At subnational and national levels, DW must be accounted for its mitigation effect over deforestation and degradation drivers. As it is with REDD, REDD + project activities at subnational and national levels, DW will contribute to deforestation and degradation combating performance improvement. However, at REDD + it is possible to include strategies to increase tropical forests HWP consumption as an additional alternative to reduce drivers' pressures and increase Brazilian Federal Conservation Units contribution to global climate change mitigation. REDD+ is, by definition, national, which favors strategies focusing on using tropical wood as an alternative to reduce Brazilian forest degradation and deforestation emissions.

Brazil and Regulatory Framework Advances on Wood Housing

My House My Life Program is a Brazil Federal government program producing housing units that after built are sold to families. Enrolled at the program are the Ministry of Cities, CEF (CaixaEconômica Federal) Bank, Federal Official Finance Institutions, D.C., States, Municipalities and construction companies. The ministry of cities is responsible for guidelines, rules and conditions, to define resources distribution among federative units and for monitoring and following of project performance (CEF 2016).

Ministry of Cities' Normative 318/2014 describes about the use of wood in construction and renovation of social housing within "My Home My Life Program (PMCMV)." The normative authorizes construction of wood housing, initially for northern region, for income group 1 (family agricultures, indigenous, etc.). Wood from close to 50 native tropical timber species that can be used at construction (according with specifications) must come from sustainable sources and can be taken from inside geographical boundaries of Federal Conservation Units. ICMBIO's Normative 27/2014 excuses wood from Federal CU's used to cope with normative 318/2014 from presenting a Sustainable Forest Management Plan—SFMP. For Legal Reserves at private properties and other native forestlands,

including timber bought from the market, it is necessary to present forest origin documentation, with harvesting authorizations and fiscal coupons.

Voluntary Carbon Mitigation Goals and HWP

Brazil adopted voluntary goals for emissions reductions within the period 2012–2020 with focus on fighting deforestation. GHG emissions reductions has been a large world concern as it faces climate change and raising on environmental disasters. Among sectors related to these impacts is construction, today one of the largest CO_2 emitters in the planet. For each 1 m^3 of native tropical biomass harvested in Brazil, 0.14 m^3 of final use HWP is generated.

At harvesting, a large portion of aerial biomass carbon is transferred to HWP and will be available at one of the forest products categories. Forest areas biomass volume is used as starting point for HWP carbon estimates, applying specific conversion factors for each destination log production. Estimates related to wood products baseline are available under the format of volumes delivered to industrial plants or in terms of their outputs, comprising industrial logs or primary HWP (boards, planks, panels or paper). Carbon availability at those HWP over the years is the estimate allocating other parameters which indicate carbon amount "in use" and destined to landfills. HWP carbon estimates recycling inclusion relays upon data availability.

Estimates of forest products contribution, in terms of carbon, use generic variables, including (i) domestic HWP and imports (tCO2e/year); (ii) annual variation of HWP produced domestically, including annual variations on exported HWP (tCO2e/year); (iii) annual imports of all kinds of wood and paper (tCO2e/year); (iv) annual exports of all kinds of wood and paper (tCO2e/year); and (v) annual HWP (tCO2e/year). The level of lost on solid products and paper, in a given year, is specified toward the use of a lost constant (k), which by convenience is expressed in terms of half-life in services, in years. Half-life in service describes the number of year necessary for half of the material to change environment, which can be, for example, from a home to landfill, within that sector where it remains stored. Solid wood and paper production, imports and exports are converted from m^3 or tons into tCO2e. For annual estimates calculation, the method uses yield data (Consumption = Domestic Production + Imports—Exports).

Globally forests store circa of 8.4 billion tCO2e and are capable of retaining some further billions, while 4.2–20 billion tCO2e are estimated to be stored within HWP "in use." World wood production includes more than 1.5 billion m^3/year of industrial logs, accounting for something like 1.1 billion tCO2e/year (WGCCFP 2004), with 420 million m^3 of sawed lumber and 220 million m^3 on plywood and panels—representing some 20% of total in long life span forest products, which sequester and store close to 200 million tCO2e each year.

The 3.4 billion m^3 of yearly global harvested wood is equivalent to just 20% of total yields (some 17 billion m^3/year). A lot from what is harvested becomes used for

direct and inefficient burning as fuel wood. Increasing the biomass amount taken from forests and harvesting yields would have a profound positive effect to fight global warming. By the use of extra 2 billion m^3/year industrial woods, it will be possible to reduce between 14 and 31% of all cement and steel GHG emissions, and between 12 and 19% of all fossil fuel consumption by the use of residues from industrial wood production chains for clean energy appliances. With the intensification of sustainable forest management, more CO_2 is sequestered and stored avoiding emissions from alternative materials and still producing renewable energy from harvesting residues. Besides, harvested volumes are renewed. Brazil has by far the largest global stock and growth of "hardwoods" which have the longest life span between tree species, making them relevant suppliers of HWP storing carbon for many years.

United Nations Framework Convention on Climate Change—UNFCCC—was established with the aim of analyzing from the scientific, socioenvironmental and political points of views technological solutions which can be broadly used around the globe to stop CO_2 levels from raising. Being as such is essential that those technologies under transference from developed to developing countries, within anthropogenic global climate change adaptation and mitigation objectives, become useful by local populations on the short, medium and long terms. Those technologies must place a contribution for implementing a sustainable development pattern from social, economic and environmental perspectives. Technologies which contribute to improve economic activity, social inclusion and preserve environmental quality.

HWP Consumption and Trees (Forests)

Brazilian Amazon forest biodiversity rational use from wood species cultivated under contemporary silviculture techniques is key to assure its conservation. DW and HWP consumption for social wood housing construction and renovation assures demand intensification. From one side, native tree species plantations guarantee an increase in stock and yields levels while at the other point construction companies make native trees species wood market available. Along prehistory, Ancient Age, Middle Age, Modern Age and Contemporary Age trees and forests have gone through modifications. These modifications include continental derive, glaciations and inter-glaciations, meteorites, storms, plagues, animals and relationships with men, with results as in the Figs. 1 and 2.

At the begging, there were some 27,000 trees per inhabitant in the planet all of which from native forests, while nowadays the 370 trees available for each person are majorly represented by seminatural (75) and plantation (43) forests—both weren't around even at the Modern Age, the biggest lost occurring from Modern to Contemporary Ages, as the Table portrays:

From the economic and social point of view, the investment for creating a job post within the forest sector is of US$ 600 while US$ 17,000 is needed to do the same at urban areas in Brazil. Diversified native tree species plantations for industrial use is essential to assure an increase on the level of jobs creation, income

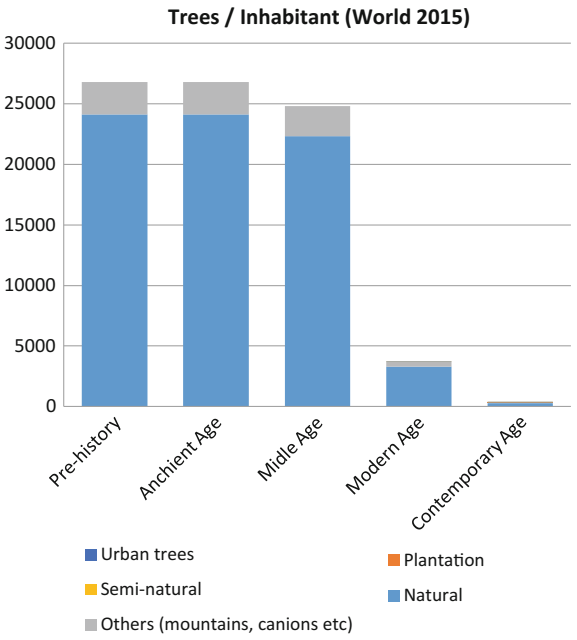

Fig. 1 Trees/inhabitant at the world Author

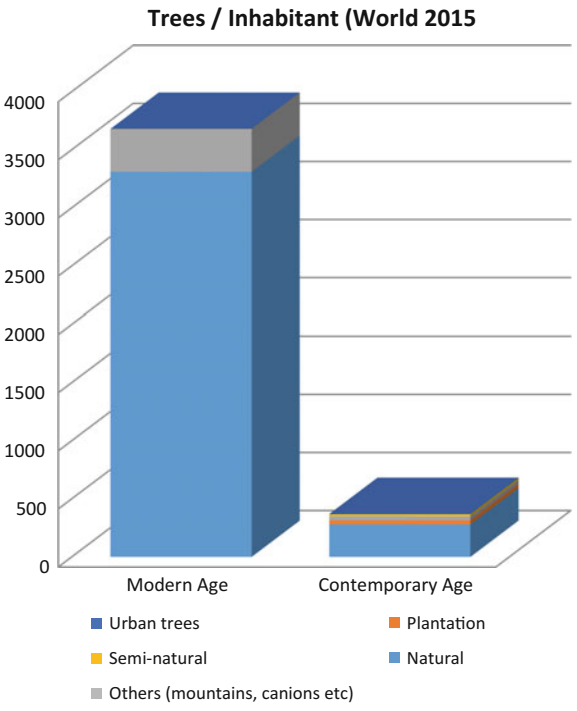

Fig. 2 Trees/inhabitant at the world between the Modern and Contemporary Age *Source* Author

generation and forest biodiversity conservation. There should be a significant increase in the number of species and hectares cultivating native woods in Brazil over the next decade or two.

In Europe and USA, the cultivation and consumption of native forests for industrial use is historical and responsible for stock and yield's levels increase. Selection of individuals, seed collection, nursery production, fertilization, irrigation, breeding and genetic modification turned European stocks from 100 to 300 m^3/ha between the XVIII and XX centuries. US native tree species cultivation at Southern Florida reached 450 m^3/ha from the same 100 m^3/ha. In Brazil, the same has been observed from a variety of wood species be it introduced (*Pinussp*, *Eucalyptus sp,* etc.) or native (*Araucaria angustifolia*, *Schizolobiumamazonicum,* etc.). What is observed is a forest biological reaction to silvicultural treatments determining short- and long-term productivity and stocks increase. Replacing natural regeneration by forest plantations increases standing stocks and summed up the positive effects of contemporary silviculture elevates harvesting volumes. In the world circa of three-fourths forest plantation are from country's native species. Countries with significant natural forest reserves, as Canada and Russia, coordinate project activities by species occurrence's microregions, the Ecological Zones of Occurrence or Ecoregions. At North America, proportion of introduced tree species under cultivation is less than 5%, while in South America less than 5% of forest plantations are from native tree species. Only in Oceania, there will be a little more than 20% of native tree species under cultivation, in Africa they are 60%, in Asia 70% and in Europe 90% of cultivated forests are from native tree species.

Within those regions with larger timber consumption in the planet, the forest cover has increased over the last 25 years, as in Europe, China and India, while within regions with less consumption of industrial logs deforestation is the rule for the same period. It is the consumption of industrial logs which contributes to keep and increase forested areas, reducing deforestation and degradation at large.

For "My Home My Life Program (PMCMV)" and the National Rural Housing Program (PNRH) at Brazilian Northern region, the use of tropical woods from sustainable sources replacing social housing cement and steel, increasing stocks and yields productivity of native timber species silvicultural technologies, commercial expertise at all levels (marketing, Capacity Building, distribution networks, certification, etc.), industrial developments (reconstituted panels, liquid wood, etc.) and adequate finance (Green Bonds, Green Climate Fund, etc.) are essential. Carbon credits to construction sectors for using wood building technologies will contribute to organize a national sectoral strategy for GHG emissions reductions. For gaining international recognition and assure national goals' contribution, this Brazilian construction sector strategy must be aligned to UNFCCC's technical and institutional requirements.

Only the available industrial like forested areas in the Amazon are capable, under SFMP, to contribute for amplifying forest sector participation from today's 4.5 to over 7% GDP, equivalent to an annual income of more than US$ 43 billion. Amazon region ecosystem services market potential has been estimated at around US$ 2–4 trillion/year.

Although there is an apparent competitive advantage when it comes to availability of biomass, Brazil still portrays moderate HWP consumption while the neighboring South American countries have minimum levels. Generally developing countries expand consumption when forest plantations increase their presence, with lower costs and improved technological and managerial procedures, being systematically promoted to the market under political and institutional support.

Brazilian ecosystems sustainable management relays upon technological interventions. With investments directed to appropriate silvicultural technologies, national wood products from Brazilian native tropical timbers will be highly competitive at international Green Economy markets. Brazilian tropical timber species diversity, productivity and qualities being cultivated under contemporary silvicultural techniques are capable of placing native forest sector among world's greatest. Native forest species biodiversity cultivation contribution from the use of Brazilian woods will be a direct result from consumption incentives. National regulations must incentivize the use and consumption of native timber from sustainable sources as a way of assuring forest biodiversity cultivation sustainability.

Increasing forest and HWP carbon sequestration and storage is the same as reducing emissions, and it represents a significant opportunity to private investors on engaging at voluntary Corporate Socioenvironmental Responsibility—CSR—activities or even at international carbon markets. Registered carbon credits can supply an income source for landowners, support rural development and facilitate SFM. Logs produced to supply industry with sustainable sources can receive payments directed to improve technology at silviculture, industry, commerce and funding toward tropical forests. When tropical timber used by society comes from sustainable origins, it increases forestlands carbon sequestration and storage.

Brazil holds the largest stock of hardwoods in the planet. Some of those tropical hardwoods have characteristics that make them therapeutic, comfortable, charming as well as immune to fungi and insect attacks. Brazilian tropical hardwoods, just as softwoods, possess a diversity of qualities which are hard to be reached by any other tree species in the world. Those unique qualities are competitive advantages that can be used to enhance Amazon biodiversity cultivation tropical timber consumption role. With a growing and promoted consumption increase, rural landholders have markets available to justify necessary investments on Brazilian native tropical timber species cultivation. Use of adaptive SFM, biodiversity banking regional strategies implementation and the use of contemporary industries (MDF, HDF, etc.) value aggregation will increase social inclusion chances and, by that, project activity sustainability over time.

HWP and Construction

At construction sector, Brazil has an estimate social housing deficit of between 8 to 10 million units and a growing rate directly linked to population expansion. Applying a volume of 5 m^3/house, the estimated demand reaches 50 million m^3

just for the deficit—representing a total of approximately 25 million tCO2e only on HWP. The use of wood as an alternative material for replacing other materials can generate even more carbon credits from GHG emission's reduction. In a study conducted comparing a traditional social housing model of 52 m^2, built by Parana State Housing Company—COHAPAR—and identified as: "Hose type R1 and R1A—CF52" and a model built by Brazilian Ministry of Environment Forest Products Laboratory—LPF/MMA—called: "Wood Social Housing" also with 52 m^2, the total of GHG emissions was obtained (ZANETTI 2015).

The COHAPAR model already used 5.8 m^3 of HWP—the highest volume for this kind of housing in the country—and was evaluated comparatively to LPF/MMA model which applies 9.2 m^3 of native timbers. The economy in terms of GHG emissions came from using circa of 50% more wood and, for that, carbon stored is 1.7 tCO2/house larger. As by using less materials with production chain higher energy demand, the total emission's reduction is 10.4 tCO2e/house for LPF/MMA model. Altogether 12.1 tCO2e less by unit associated to wood housing. This value gets incorporated at national inventories to accomplish REDD+ voluntary goals as HWP associated to wood housing program from construction sector. Along wood housing production chain, the total GHG emission's reductions impact was estimated at 83 tCO2e/unit. The remaining 71 tCO2e is related to REDD and REDD+ activities from AFOLU sector. Both sectors contributions are valid to comply with Brazilian REDD+ emission's reductions goals for the Amazon region.

For Brazilian Amazon, Normative 318/2014 will contribute to answer a social housing deficit of between 500,000 and 5 million units, considering 2014–2030 time frame. Appliance of tropical woods from sustainable sources at PNHR and PMCMV can generate an emission's reduction impact varying from 41 to 400 million tCO2e, corresponding to something as 7–70% of Brazilian 564 million tCO2e reduction goal. Construction use of HWP represents a reduction of 6–60 million tCO2e, while DW from REDD and REDD+ hold 35–350 million tCO2e, or 6–60% of Brazilian goal. An expanding region and important natural resource base for Brazilian sustainable development, the use of industrial wood from the Amazon can be much more significant to improve northern region country's contribution in the near future.

Wood is Good

"Tackle Climate Change: Use Wood" is a European Parliament program directed to strength societal use of wood as a way of fighting atmospheric CO_2 accumulation. French has "de Bois-Construction-Environment", England the "Wood for Good", Netherlands "Centrum Hout", Denmark "Trae Information", Finland "Puuinfo", Belgium "Wood Forum", Spain's "Viver Con Madera", Australia "Wood Naturally Better" and Austria and Italy "Promo Legno" are few from national, binational and multilateral networks for the promotion of wood use as a form of global climate change mitigation.

International Wood Culture Society (IWCS) is a nonprofit organization formed by wood enthusiasts, dedicated to research, education and promotion of wood culture. IWCS advocates for a harmonious living between people and nature, explores the value of wood use from a cultural perspective and supplies a platform for studying wood culture, encouraging its practice and promotion (IWCS 2016). IWCS established March 21 as Wood World Day, a data to disseminate the value wood aggregates to daily life. IWCS, just as Normative 318/2014, PMCMC and PNHR promote the concept that: "Wood is Good"

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How Good is Wood? Facts and Myths Regarding Wood as a Green Building Material

Arijit Sinha

Abstract Sustainability has been the key driver for decisions concerning building materials. Environmental awareness has increased, and the conscious society and citizens of this world demand more accountability. This has been the driver of the green building movement. The green building programs have been immensely successful—few more than the others. The success has been attributed to their attempt of validating peoples' efforts toward sustainable development by assigning a tangible metrics. Green buildings, in modern vernacular, have become similar to sustainable development. Although similar, sustainable development and green buildings are not the same rather similar. This paper presents author's view on how green building is a subset of sustainable development, which is an all-encompassing concept. An explanation of what constitutes a green building material is discussed, while objectively assessing wood with regard to those criteria. Myths and facts regarding the use of wood in green buildings will be discussed using a life cycle approach. Wood is arguably one of the most sustainable materials. However, there are some facets of wood that impede its acceptance in construction. These impediments and their mitigation strategies are discussed in the paper. Statements concerning sustainability require validation, which can be provided by life cycle analysis (LCA). Many green building programs have certain pitfall and challenges —mostly with respect to practices on material selection and lack of performance monitoring. Materials regardless of its origin have a common starting point, neglecting the environmental benefits of certain materials vis-à-vis another. This paper presents how beneficial LCA can be, when included and integrated into the green building rating system and introduces an integrated design concept for green buildings, especially from a material selection standpoint. Since writing of this article, the major green building programs have altered their methodologies to incorporate LCA in their rating program.

Keywords Carbon sequestration · Life cycle analysis · LEED

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Introduction

Increasing world population coupled with increased economic development of many nations has caused added strain on world's resources. Often economic development is achieved by stressing the environment. Thus, there is increased awareness and realization to conserve energy and resources (Garcia 2011) and use renewable materials (Sinha et al. 2013). Globally, the built environment consumes 60% of the raw materials that are produced by mining the Earth's crust (Bribian et al. 2011). Therefore, sustainability has been a major consideration when choosing building materials. Environmental awareness has increased over time and citizens of this world are demanding more accountability. This has been the driver of the green building movement. The green building programs emerging from the green building movement have been immensely successful. A few more than the others. The success has been attributed to their attempt of validating peoples' efforts toward sustainable development by assigning a tangible metrics.

Current green building standards address a wide array of areas but focus mostly on energy efficiency and indoor air quality. Other areas of consideration in these programs are water management, material production, construction issues, occupant health quality, recycling, reusability, and waste management (Bowyer 2008). In the USA, there are more than 40 green building programs including independent programs led by many local counties. The National Association of Homebuilders (NAHB) worked with these counties and designed their own green building program in 2008 called the National Green Building Program (NAHB 2010). Since the introduction of National Green Building Program, it has emerged as the market leader in the residential sector. On the other hand, in the commercial sector, Leadership in Energy and Environmental Design (LEED) administered by US Green Building Council (USGBC) has been the market leader by a huge margin. At the time of writing the article, LEED for homes was gaining grounds on the NAHB-administered program for residential construction. It is projected that LEED for Homes will soon take over NAHB-administered program.

To earn certification under the LEED program, a building must meet certain prerequisites and performance benchmarks within each category. LEED (2009) contains the following specific rating systems: (1) New Construction (NC); (2) Existing Buildings: Operations and Maintenance; (3) Commercial Interiors; (4) Core and Shell; (5) Retail; (6) Healthcare; (7) Homes; and (8) Neighborhood Development. At the time of writing this article, LEED 2009 was the current version. However, at the time publication of this article, LEED v4.0 has been released and changed some of the point system. This article deals with LEED (2009). Each of the rating systems within the LEED domain is composed of 100 points, which are divided among five categories: Sustainable Sites (26 points); Water Efficiency (10 points); Energy and Atmosphere (35 points); Materials and Resources (14 points); and Indoor Environmental Quality (15 points). Additionally, up to 10 bonus points are possible through innovative design and consideration of regional priorities. To obtain points in LEED, certain prerequisites are mandatory in

all the five categories. After achieving the prerequisites, the points are assigned in a progressive way for incremental level of documented efforts to increase environmental performance. LEED is also the world leader of green buildings. The LEED International Roundtable is composed of representatives from 38 countries (including India) who work to provide global consistency in regional approaches to green building. Each of these 38 countries utilizes LEED rating systems that are catered to the local conditions and practices in their country. LEED has registered projects in 133 countries. In India, the India Green Building Council (IGBC) provides leadership in the green building sector through several different rating systems. One of the primary systems, LEED India, evaluates sustainable site development, water savings, energy efficiency, materials selection, and indoor environmental quality for both new construction and the core and shell of buildings. For residential sector, Green Homes have gained popularity in India. Green Homes have system catered to individual homes, high-rise residential apartments, gated communities, row houses, and retrofit of existing residential buildings. Similarly, Green Townships rating system is for large developments and townships, and the Green Factory Building rating system for industrial complexes.

Green Material

Generally, three criteria are used for assessing whether a material is green—resource management, pollution or indoor environment quality, and performance (Milani 2005; Spiegel and Meadows 2006). These three evaluation categories have significant overlap. For an ideal green material from a resource management standpoint, it should be a natural material derived from renewable sources. The materials extraction and processing should be carried out in an environmentally conscious manner, i.e., process should have low emissions. This is important from a pollution or indoor environment quality standpoint. From a performance standpoint, two things are paramount. One, the material should perform its intended function for a long time, i.e., the material is durable. Durability is becoming a key factor. Materials with low durability cannot stand the test of time to be called green. Globally, the built environment consumes 60% of the raw materials that are produced by mining the Earth's crust (Bribian et al. 2011). Considering a building's life span, more than 90% of the building material's life cycle coincides with the operational phase of the building. This tends to make durability and performance somewhat more important factor for building materials than for many other kinds of products. Second, after the service life of material is over, it should offer value in terms of either full integration into the landscape or can be recycled or down-cycled for a different function. This is important to minimize the environmental impacts of the building material.

Wood is a renewable, strong, and a natural material that would be a natural fit for structures claiming to be green. Wooden structures are aesthetically pleasing. Wood, as a material, also has biophilic traits that help mitigate sick building

syndromes (Fell 2010; Nyrud and Bringlismark 2010). Above all, wood is a sustainable natural resource. Wood is a product of photosynthesis, which uses carbon dioxide and water in the presence of sunlight to build glucose molecule that is the backbone of wood. If sustainable forest practices are enforced, then wood is the most sustainable material on this planet from a resource management standpoint. From pollution standpoint also wood processing are not as energy-intensive processes as other building materials are and emits less pollution. Wood is the only building material that has the ability to sequester and store carbon for a number of years (Bowyer et al. 2007; Buchanan 2006). This makes wood an ideal material from an emissions or pollution standpoint. Because wood can be decomposed, recycled, or reused, wood is one of the rare materials that have the potential of full integration into the landscape after its service life is over.

There are certain impediments for wood being used as a building material of choice in many countries from a performance standpoint. First, wood shrinks and swells with moisture changes. Although the dimensional changes due to moisture can create serviceability issues in structures, these can be accounted for by the structural engineer during the design process. Second impediment is that wood decays. This is a fact but there are ways or provisions in the design to prevent decay. Even if certain degradation due to decay has been encountered proven, mitigating strategies are readily available. Another concern is that wood burns easily and it is a fuel for fire. Wood burns readily initially. However, once the outer layer of wood burns, it produces char, which is insulating in nature and prevents further burning of wood. Testing has shown that a wooden beam under fire can retain its shape and structural integrity for a significantly longer duration than an equivalent steel beam (AWC 1961). There are impediments in terms of physical characteristics but these can be easily dealt with. Wood perhaps exemplifies a green material. Given this wood should be the first choice material for all green building programs, especially the world leader LEED.

LEED and WOOD

The USGBC-administered LEED, although a comprehensive effort in quantifying sustainability metric, has certain pitfalls in terms of how it rates the materials. Certain provisions in LEED and other programs can lead to negative impact on wood and wood products (Bowyer 2008). Materials regardless of its origin have a common starting point in these programs, neglecting the environmental benefits of certain materials vis-à-vis another. For example, materials like concrete and wood are considered equal when being used in a building. While steel has an upper hand as recycled content, recyclability is accounted for (USGBC 2010). This attribute of steel should certainly be accounted for as it is a wonderful property that only steel possess. Similarly, wood should be given more emphasis as due to its biological origin. Moreover, life cycle analysis has shown that wood has less embodied energy and carbon footprint than several other building materials (Puettman et al.

2005). Wood is a renewable material, while the raw materials to make cement and then concrete are a product of energy-intensive process (PCA 2008; van Oss and Padovani 2002; Rajendran and Gambatese 2007). As discussed earlier, steel is preferred over wood and concrete, because of its recyclability and recycled content (USGBC 2010). Steel although is recyclable has higher environmental impacts than wood because the raw material has to be mined and then steel has to be extracted in a blast furnace (IISI 2000). Many experts (Bowyer 2008) consider this stand on steel being given more importance, as a serious error from an environmental standpoint.

LEED assigns extra credit for materials that are "rapidly renewable" (LEED 2009). A material is considered rapidly renewable if it has 10-year period of turn-around or less. For crops like bamboo and trees with smaller rotation, these points can be attained. However, for most wood species, a 10-year or less turn-around time is unheard of, and therefore, this credit is elusive to projects using predominantly wood. Bamboo on the other hand is considered rapidly renewable compared to any hardwood such as maple or oak, and therefore, bamboo flooring is preferred over hardwood flooring as it helps in attaining that credit. No consideration is provided to the amount of energy expended in processing of either material. The scientific background of this credit has been continuously challenged (Bowyer 2007; YPFPG 2008), but the credit criteria still remain in LEED 2009 as well as in the new version.

A challenge that LEED faces is to ensure that the wood used in a project is harvested legally and from sustainable sources. Forest certification ensures this. Forest certification helps LEED by ensuring that the wood has been sourced from a sustainably grown and managed asset after due economic, environmental, and social considerations. Combining forest certification in LEED also ensures that wood harvested illegally (outside the USA) will not receive any credits. Although there are several forest certification program active in the USA, Forest Stewardship Council (FSC) is the only one recognized by the USGBC (LEED 2009). Alternative programs are also very adept in promoting responsible and sustainable forestry practices but are not recognized for their efforts by the USGBC. With FSC wood being limited, it is difficult to earn credits for certified wood. Moreover, it is only wood that requires external validation or certification, while other materials in LEED do not (LEED 2009), despite social and environmental impacts associated with other materials (Bowyer 2007, 2008). The new version of LEED is a bit different in its approach toward certification, but FSC-certified wood is still receiving the highest credits in a new tiered point system that allows for a point for a recognized certification.

Life cycle assessment (LCA) is a tool that uses peer-reviewed aggregated data to provide a rational, quantified approach to determining specific environmental impacts of a product or system through its entire life cycle. If done correctly following all the standard protocols, LCA can provide an objective measure for comparing building design alternatives. LCA has all the facets of becoming a valuable asset in assessing the sustainability of green buildings. There is a widely expressed concern regarding a deficiency in green building standards to allocate

points based on life cycle performance of the products. This issue fundamentally emanates from the fact the material selection criteria do not form an important part of rating systems. Materials are most used in buildings and the built environment. Therefore, they have greater environmental implications. To be fully sustainable, strategies need to be designed for materials' reuse and recycling beyond the building service life. Measures are needed to not only divert material out of the landfills but also to reuse in the built environment albeit at a reduced functionality. Most green building programs neglect the beyond service life characteristics of building materials, which is an incongruity. This is, however, being addressed in the newest version of LEED, where the user will have an opportunity to earn more points with a full building LCA.

Architects Dilemma

An architect or material specifier for a project grapples about which material to specify. On one hand you have a sustainable material such as wood, while on the other steel or other structural material can help provide more credits in LEED to get you over the threshold of certification. An architect has no incentive to specify wood over steel or concrete. A prime example for this is the new Kempegowda International Airport in Bengaluru. It is a LEED gold certified airport with miniscule amount of wood used for structural or decorative purposes. It meets LEED gold standard mainly because biofuels are used for ground force vehicles and installed real-time performance monitoring tool. Both are commendable efforts. It leaves a question open—as to how a building can claim to be green without using natural and sustainable materials?

Concluding Remarks

Climate change induced stresses to the built environment, especially buildings are motivating a paradigm shift toward sustainable development. Sustainable development is based on triple with its triple bottom-line, which is—economic optimization, reduction in environmental impacts, and improvement in human well-being. Green building rating systems are stemming from these transformations. Wood by virtue of its performance in the "greenness" evaluation criteria should be a natural fit for green structures. As wood sequesters carbon as it grows, wood products can have low or sometimes negative carbon footprint. Additionally, wood is a renewable resource provided the forest are sustainably managed. Therefore, the utilization of wood, in all aspects of human existence appears to be the most effective way to optimize the use of resources and to reduce the environmental impact associated with activities pertaining to the built environment. The indifference toward wood in the green building standards is an impediment for its

use in green structures. An architect is always faced with this dilemma of why to specify wood when steel or concrete buildings can earn similar level of green building certification? Green building rating systems need categorical improvements in their way of assessing materials. Need of the hour is an integrated approach to design, where along with energy concerns, long-term performance, environmental metrics, and social consideration are duly weighted. Adoption of life cycle approach to design can lead to this proposed integrated approach. This will mean a paradigm shift in processes and policies pertaining to selection of materials and their utilization since it will require manufacturers of various materials to publically divulge information regarding their environmental impacts over the product life cycle in the form of an environmental product declaration (EPD). Many countries and jurisdictions are now mandating and recommending EPDs for all mass-produced building products. As EPD becomes norm, wood should come out as an environmentally strong material when the entire life cycle is accounted for.

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Carbon Sequestration by Bamboo Farming on Marginal Land and Sustainable Use of Wood Waste for Bioenergy: Case Studies from Abellon Clean Energy

Beena Patel, Bharat Gami and Pankaj Patel

Abstract World needs a new paradigm to solve deep and perilous problems of climate change, energy access and food security through radical innovation and business opportunities along with social good and financial inclusion by following Mahatma Gandhi's philosophy of sustainability. Processed wood waste is a promising renewable energy source which can reduce India's dependency on fossil fuels by tapping through efficient technology and thereby assuring energy security and employment opportunities along with waste utilization. Such system can further be linked and supported by exploiting marginal arable lands through bamboo plantation that sequesters CO_2 and fix carbon in form of biomass. On this concept, case studies on 120 tons/day capacity wood waste pelleting plant and 120 acres Bamboo plantation in Gujarat, India, have been presented. The pellet plant is the only EN*Plus* European standards certified facility in India and Southern Asia that utilizes wood wastes like chips and saw dust from wood processing industries. Pelletized wood waste replaces 1.17 times Indian coal along with reduction in GHG emission by 1.78 kg of CO_2 eq/kg of pellets. In order to capitalize on marginal lands, high-biomass bamboo, yielding 20–25 tones/acre, have been developed and planted in 120 acre as captive farming and agroforestry model at *Aravali* district of North Gujarat, India. The Bamboo, from otherwise unused land, can support handicraft and incense stick industry, opening additional occupational avenues and women empowerment in rural areas. Bamboo waste from handicraft industries can further be utilized for bioenergy projects, sequestering additional 20–25 tons of CO_2/acre-year.

Keywords Agroforestry · Bamboo · Bioenergy · CO_2 sequestration · Sustainable development

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Introduction

India is fifth largest energy consumer and fourth largest petroleum consumer spending 45% of its export earnings for importing energy (Jain 2010). The energy sector plays a key role in the economic development of a country; the worldwide increase in the consumption of energy has led to a scarcity of fossil fuels. This has resulted in the need for exploring renewable energy options. Globally, 19% of the total energy sources are renewable, and 14% of this is predominantly derived from biomass sources (REN21 Secretariat 2011). In the context of a growing global energy crisis, bioenergy has been established with a vision to alternate energy generation sources and develop viable alternate energy sources and generation techniques, leading to a movement toward self-reliance in the global energy sector, especially in India and other emerging global economies. This bioenergy model has been carefully developed in line with the triple bottom-line approach emphasizing on environmental protection, social development and economic prosperity that can be achieved through the following paradigms of business in line with national biofuel policy released by Ministry of New and Renewable Energy (MNRE), Government of India (National Policy on Biofuels 2009):

- Creating a model capable of holistically addressing larger developmental issues, generating income and employment opportunities
- Generating value and opportunities out of waste
- Maintaining food/fodder versus fuel balance, harmony with existing agricultural and land use patterns
- R&D and technology-led innovation
- A focus on long-term sustainability

This integrated and innovation led model for agriculture waste to biopellet production is first of its kind in India (Gami et al. 2011) with established supply chain for agriculture residue collection model involving not for profit organization that interface with farmers for farm residue collection along with marginal land cultivation by bamboo for sustainable biomass supply for bioenergy and livelihood at rural area. Gandhidham near Kandla port is second largest port of the country handling wood log imports. The location is in western part of Gujarat and in proximity of the major cities, well connected to other major cities by means of air, rail and road providing excellent connectivity and logistics. In order to utilize wood waste, 120 tons per day (TPD) wood pellet production plant near Kandla port at Gandhidham has been established as described in Case study-1. Case study-2 describes bamboo farming, its social impact through incense stick production and pellet production utilizing waste from incense stick scrap.

Case Study-1: Wood Waste to 120 Tons/Day Pellet Production Plant

Feed Stock Supply

About 70% of the India's timber requirement is catered through Kandla Port. Gandhidham, located at the proximity of 11 km from Kandla port, is the major hub for timber processing. Approximately 400 saw mill operate in the vicinity of 20–30 km. Around 2.5 m^3/annum of wood is being processed at Gandhidham, which generates about 700–900 tons/day of wood wastes in the form of saw dust and wood strips (GIDB 2005).

Pellet Production

Biopellets from waste wood from saw mills/wood log processing industries was established with imported technology from Europe for better productivity and quality pellet production that has better control over machine through programmable logic controller (PLC). As shown in Fig. 1, the raw materials, wood chips and wood strips are directly sent to chipper machine for reducing the size of feed material. The chipping material (Size $\sim$20 mm) from chipper machine goes to hammer mill and then transferred to mixer. The mixer is equipped with RO water addition unit up to 5%, agitating system and bottom scrapping unit for easy discharge. The mixing unit feeds into pellet mill. The material from mixer is transferred to pellet mills through rotary feeder and conditioner. In pellet press, the two rotors are stable and die is rotating around and cutter cuts the finished material, pellets. Pellet travels to counter flow cooler through peddle conveyor. This cooler cools the pellet up to ambient temperature from 60–80 °C. The cooler is equipped with auto level sensor and grate mechanism for sufficient retention time inside the cooler. The hot air is suck from the cooler and vents it to atmosphere by blower. The moisture is also reduced from 15 to <10%. In downstream, pellets comes in vibrate screen wherein the fine particles are separated and pellets are stored in loose form as well as bagged and sealed with stretch wrapping machine.

Pellet Quality Analysis

The facility is the only European certified wood pellet plant in southern Asia that meets EN*Plus* (EN 14961-2) quality standards for pellets as shown in Table 1 (European Pellet Council 2011). The quality assurance department is equipped with

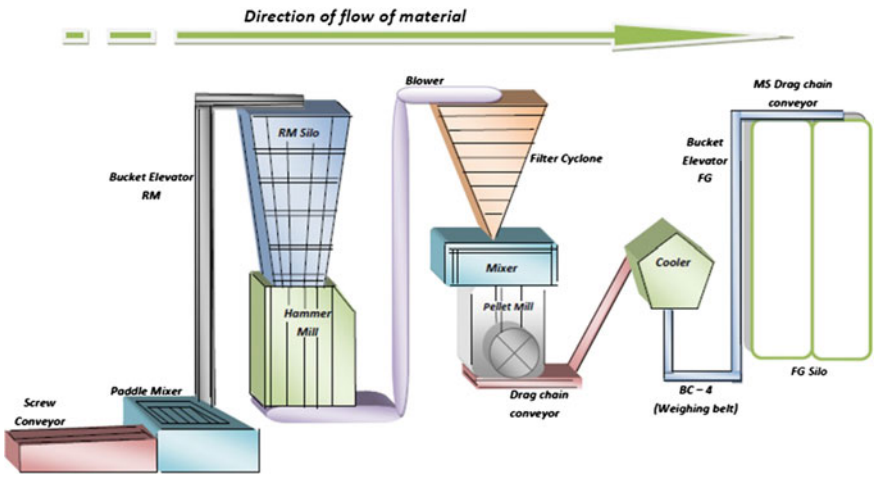

Fig. 1 Schematic diagram of pellet production and process

Table 1 Wood pellets specifications

Sr no.	Property	Unit of measurement	Values
1	Diameter	mm	6–8
2	Length	mm	3–40
3	Moisture	As received, %	≤ 10
4	Net calorific value	As received, MJ/kg	>16.0
5	Ash	% dry	<1.0
6	Bulk density	kg/m^3	≥ 650
7	Nitrogen	% dry	0.3
8	Sulfur	% dry	0.05
9	Chlorine	% dry	0.5
10	Arsenic	mg/kg dry	≤ 1
11	Cadmium	mg/kg dry	≤ 0.5
12	Chromium	mg/kg dry	≤ 10
13	Copper	mg/kg dry	≤ 10
14	Lead	mg/kg dry	≤ 10
15	Mercury	mg/kg dry	≤ 0.1
16	Nickel	mg/kg dry	≤ 10
17	Zinc	mg/kg dry	≤ 100
18	Ash melting behavior	°C	>1200

all required instruments like hot air oven, muffle furnace, analytical balances, bomb calorimeter and ligno tester for durability testing. Ash and moisture contents are checked daily for every raw material that arrives at plant. Nitrogen, sulfur and chlorine content of wood waste are also checked periodically that helps in emission reduction and corrosion protection. Pellets are analyzed for its bulk density and mechanical durability that helps in logistic and integrity of pellets. Moisture, ash and calorific value are monitored as an indicator of energy generation. Diameter and length of pellets are also physically measured as a part of pellet production monitoring (Nielsen et al. 2009) (Table 1).

Product Application

Wood pellets are used at the industrial, community and retail sectors. At industrial level, it is used for utility requirement for industrial units having fluidized bed combustion (FBC) boilers, traveling grate boilers, thermic fluid heaters and hot air generators. The product is suitable for all types of combustion system without modification in combustion system. It significantly improves energy output and thereby efficiency of production. A range of pellet-based cook stoves, hot water generators and burners are available (Fig. 2). At community level, pellets can be used as community cooking in stoves, malls, educational institutions, etc. At retail level, pellets are used at the pellet stoves in domestic/home heating and cooking applications at restaurants, hostels, etc.

Product Benefits

Wood pellets also help users to meet their pollution control obligations and assist them in branding their organizations as clean, green and environmentally responsible. Coal and lignite are replaced by wood pellets at industrial level, while LPG, diesel and kerosene are replaced at community and retail level using pellet-based appliances. One ton of pellet replaces 1.5 tons of lignite as well as replacement of one tone of coal with pellets reduces 1.5 tons of CO_2 emission along with other local pollutants such as sulfur oxide (SO_x), nitrous oxide (NO_x) and suspended particulate matter (SPM) resulting in significantly improved workplace and surrounding environment (Table 2). Pellets show 19–50% cost benefits over other fossil fuels at retail level as shown in Table 3. Pellet used at cooking or heating appliances also significantly reduces emission from urban community and therefore best option as clean fuel for large-scale heat and steam generation at community level.

Table 2 Comparison of energy and emission parameters of pellets with conventional fuels

Fuel	Gross Calorific value (GCV) (kcal/kg)	Displacement with pellets	CO_2 emissions per ton of consumption	Ash %	Sulfur (%)	SO_x (kg/tone)	Moisture (%)	Bulk density (kg/m^3)
Lignite	2500–3000	1.36–1.64	1.64	25–35	1–4	20–80	20–40	650–780
Indian Coal	3500–3800	1.07–1.17	1.52	20–30	0.5–1	10–20	5–20	720–850
Imported Coal	5000–5500	0.75–0.82	1.53	10	0.5–0.7	10–15	5–15	720–850
*Wood Pellets	>4200	1	Carbon Neutral	<5	0.5	10	<10	650

[a]Wood pellets has better GCV compared to lignite and Indian coal; generates 80% less ash and emits less sulfur and Sox. Therefore, pellets are more environments friendly

Table 3 Comparison of energy and economic parameters of pellets with conventional fuels

Pellet use: Saving against fossil fuels and CO_2 emissions reduction						
Particular	Pellet (kg)	LPG (kg)	PNG (m^3)	Diesel (L)	Kerosene (L)	Coal (kg)
Calorific value (kcal): A	4200	12,000	9000	9100	8000	5000
Rate (INR):B	17	108	50	61	50	25
pellet consumption against fossil fuel: C	1	2.9	2.1	2.2	1.9	1.2
Energy: kcal/INR [A/B]	247	111	180	149	160	200
Cost of pellet (INR) required to meet equivalent fossil energy: D [D = C*17 INR-cost of pellets]	–	48.6	36.4	36.8	32.4	20.2
Saving (INR) [B-D]	–	59.4	13.6	24.2	17.6	7.1
Saving %	–	55.03%	27.14%	39.62%	35.24%	19.05%
*CO_2 emissions per ton use of fuel (tCO_2/tonne fuel)	1.67 (carbon neutral as per life cycle of plant)	2.98	2.69	3.19	3.15	1.82

[a]CO_2 emissions are calculated based on efficiency of cooking appliances using respective fuels

Table 4 Processing trial of bamboo for incense stick at Devas, MP, India

Steps	Process Flow	Input RM (kg)	Output FG (kg)	Rejection (kg)		Final FG output %
				Dust	Woody	
1	Cross-cutting	7.32	5.62	0.13	1.57	19
2	Splitting	5.62	5.35	0	0.24	
3	Slicing	5.35	5.24	0.11	0	
4	Stick making	5.24	1.41	3.83	0	
5	Polishing	1.41	1.4	0.01	0	
6	Sizing	1.4	1.39	0.01	0	

RM-Raw material, *FG*-Finished goods

Table 5 *Bamboosa balcooa* proximate and elemental analysis

Sr. No	Parameter		Result	Analysis method	Reference limits as per EN*plus* for wood pellets
1	Moisture (%)		5.5	DIN EN 14,774-2	≤ 10
2	Ash (%)		0.53	DIN EN 14,775(at 550 °C)	≤ 0.7
3	Volatile matter (%)		79.28	–	–
4	Fixed carbon (%)		14.69	–	–
5	GCV (kcal/kg)	Dry bases	4490		–
	GCV (MJ/kg)		18.8	DIN EN 14,918	16.5–19 MJ/kg
	GCV (kWh/kg)		5.22		4.6–5.3 kWh/kg
6	Nitrogen (%)		0.31	DIN EN 15,104	≤ 0.3
7	Sulfur (%)		0.03		≤ 0.03
8	Chlorine (%)		0.01	DIN EN 15,289	≤ 0.02
9	Hemicellulose (%)		23.7	–	–
10	Cellulose (%)		46.81	–	–
11	Lignin (%)		10.26	–	–
12	Silica (%)		1.09	–	–
13	Aluminum–Al (mg/kg)		70	DIN 51,731	–
14	Arsenic–As (mg/kg)		05		<0.8 mg/kg
15	Boron–B (mg/kg)		35		–
16	Calcium–Ca (mg/kg)		185		–
17	Cadmium–Cd (mg/kg)		0		–
18	Cobalt–Co (mg/kg)		0		–
19	Chromium–Cr (mg/kg)		5		<8 mg/kg
20	Copper–Cu (mg/kg)		05		<5 mg/kg
21	Iron–Fe (mg/kg)		15		–

(continued)

Table 5 (continued)

Sr. No	Parameter	Result	Analysis method	Reference limits as per EN*plus* for wood pellets
22	Potassium–K (mg/kg)	80		–
23	Magnesium–Mg (mg/kg)	110		–
24	Manganese–Mn (mg/kg)	50		–
25	Molybdenum–Mo (mg/kg)	50		–
26	Nickel–Ni (mg/kg)	10		–
27	Phosphorus–P (mg/kg)	57		–
28	Lead–Pb (mg/kg)	08		<10 mg/kg
29	Antimony–Sb (mg/kg)	0		–
30	Selenium–Se (mg/kg)	70		–
31	Strontium–Sn (mg/kg)	140		–
32	Zinc–Zn (mg/kg)	44		<100 mg/kg

Table 6 Bamboo economics

Parameters	Units
Waste land utilization	6 m acre
Bamboo production Yield (50% consideration)	12.5 tones/acre/year
Total bamboo production	72.0 m ton/year
Incense stick/tooth pick/chopsticks (19% finish goods from total bamboo yield)	14.4 m ton/year
Incense stick/tooth pick/chopsticks @ 85,000 INR/tone	122,400 INR Cr/year
Energy pellets (@ 80% waste generated from incense stick) of 720 Lac tones/year	57.6 m tons/year
Energy pellets (waste utilization) @ 17,000 INR/tone	97,920 INR Cr/year
CO_2 Emission reduction vis-a-vis fossil	86.4 m tons/year
LPG replacement by pellets	23.00 m ton/year
Direct employment (one person/acre)	6 Million people
Indirect employment (1.5 times of direct employment considering incense stick, pellet production, logistics, marketing, etc.)	9 Million people

Discussion on Case Study-1

India has potential for waste wood processing industries' linkages with renewable energy sector. The timber industry in Gandhidham, Kutch, has grown hikes after granting "Open General License (OGL)" permit in 1995 for import of timber. Gandhidham has now emerged as Asia's largest center for wood-based industries (GIDB 2005; APFSOS 2009). The OGL permit was introduced to reduce burden on

Fig. 2 Pellets based eco-equipment. **a** Industrial pellet burner **b** Green burner **c** *SMART* cooking stove **d** Continuous feeding stove (CFS) **e** *ATOM* cook stove

natural forestry. Carbon emission reductions through renewable fuel and carbon sequestration by adopting plantation are the key transect of the national biofuel policy and national forest policy respectively.

Wood pellets have been successful internationally traded biomass. Ryu et al. (2006) stated that a critical element for biomass fuels to successfully compete with other source is densification; in this sense, wood pellets provide an enhanced heating value of wood per unit volume, low moisture, a more complete and efficient burning with low ash particulate emissions, optimized transportation over long distance with varying application from small-scale residential heating to large scale co-firing in coal power plant (Wahlund et al. 2004; Junginger et al. 2008; Spelter and Toth 2009). Additionally, pellets can be produced from wood waste, forest thinning, other biomass ingredients and wood production by-products (Bergman and Zerbe 2008).

Modern technological advances in the use of pellet fuels in gasifier burners and cook stoves would be important aspects of the energy savings and emission reduction on account of fossil fuel. A range of cook stoves and gasifier burners caters various industries and community cooking. Pellet-based cook stove trials by consumer experienced 20–45% savings on fuel cost with reference to other fossil fuel like kerosene, LPG, diesel, and industrial burner replacing coal, lignite and furnace oil (Tabel 3). The National Programme for Improved Cook stoves (NPIC)

was launched in 1983 with the aim to disseminate mud-based improved cook stoves, equipped with chimneys, and portable metallic stoves to increase the fuel use efficiency and to reduce indoor air pollution. This has been an integral part of national biofuel policy (National Policy on Biofuel 2009). Newer stoves are now being developed and manufactured with the backing of large international companies. These new stoves are designed with the goals of improving the energy efficiency of cooking and reducing indoor air pollution and the labor or cash requirements. Generally, these stoves are made of durable materials that will last 5–10 years or even longer, and many are sold at affordable prices with guarantee. The market potential for biomass stoves in developing countries is large (The World Bank 2011). However, pellet standards are very important criteria to meet its combustion requirement.

Grass pellets contain dramatically high concentrations of ash, nitrogen, sulfur and chlorine than the wood pellets resulting into clinker formation in boiler (Vermont Grass Energy Partnership 2011). To guarantee the consistently high quality of the wood pellets, European pellet certification system certifies product and manufacturing processes. Three wood pellet qualities are defined with the classes ENplus-A, ENplus-A2 and class EN-B that are primarily based on the specifications of the European standard EN 14961-2 (European Pellet Council 2011). The certification system and process audits wood source along with wood quality, and therefore it is very important for traceability of wood supply from forest or processing industry. This helps in reducing immoral practices in wood supply chain.

For consistent quality supply of wood for pellet production, a sustained wood supply is required. This can be achieved by forward integration with agroforestry, farm forestry, sustainable forestry or social forestry. This helps in achieving not only consistent biomass supply but also carbon sequestration, land reclamation and social livelihood by employment through farming and minor forest product generation.

Case Study-2: Bamboo as Bioenergy Feedstock: Large-Scale 120 Acre Bamboo Plantation and Agroforestry Model

To evaluate biomass supply, 120 acre bamboo plantation was raised in 2010 at Modasa Taluka, Aravali district of North Gujarat. Taxonomically bamboo is from family—*Poaceae,* with subfamily—*Bambusoideae*. Bamboo is diverse group, and India is second richest country in world after China in bamboo genetic resources. Total 22 genera covering 136 species of bamboo are reported in India (Sharma 1987). The Northeastern States, Western Ghats, Chattisgarh, Madhya Pradesh and Andaman Nicobar Islands are major bamboo-rich areas in India. Total area covered under bamboo plantation in India is 13.96 Mha. Bamboo is a common term applied

to a broad group of large woody grasses, ranging from 10 cm to 40 m in height. Bamboo has distinct protrusions on the culm, called "nodes" with intermediate parts called "internodes" (Ronald et al. 2013). It is said to be the fastest growing plant on earth, and it can reach up to 40 m high and 30 cm diameter.

Interest in bamboo as a source of biofuel or bioenergy has rapidly increased due to the concerns regarding energy security, oil price volatility and environmental pollution (IEA 2011). Apart from use in bioenergy production, bamboo is widely used for making handicrafts and furniture, building, decorating, paper making and as firewood in developing countries. Its ability to grow on nutrient-poor soils, little requirement of silvicultural management, easy and cost-effective harvesting, vegetative propagation, fast growth and a host of other desirable characteristics like (i) high biomass yield (ii) versatile and diverse uses, (iii) not susceptible for common seasonal changes, (iv) does not fall under forest law for harvesting and cultivation at large scale, (v) absorb more carbon dioxide (CO_2), (vi) produce more oxygen (O_2) and (vii) shallow root structure suitable for soil micro-flora and capable of improving soil ecosystem makes it more suitable candidate for energy plantation.

Bamboo Farming

Large-scale captive bamboo plantation of *Bambusa balcooa* in 120 acre land was initiated in July 2010 at Rameshwar kampa village, District Aravali, North Gujarat. This is first of its kind initiative in India. Bamboo were planted at 450 plants/acre densities with 6.5 feet plant spacing and 15 feet line spacing following standard agriculture practices (Salam and Pongen 2008).

Bamboo Harvesting and Processing

Within three years, 120 acres of marginal land was converted in man-made forest (Fig. 3). The first harvesting was carried out after three years of cultivation in July 2013. From each bamboo plants, average seven poles were harvested having 8 kg average weight of pole; achieving total yield of 25 tons/acre/year.

Incense Stick from Bamboo

The harvested bamboo poles were subjected to incense stick cutting through various bamboo cutting equipments. Table 4 represents stepwise bamboo processing input and output balance in kilograms with final output data. Bamboo pole cross-cutting, splitting, slicing, stick making, policing and sizing are the six major steps required to make incense stick from bamboo. The final product yield is only 19% generating

Fig. 3 Bamboo plantation in 120 acre at Aravali District, Modasa, North Gujarat

80% of woody scrap and dust. This 80% has potential to use for bioenergy pellet production or biopower generation.

Bamboo as an Energy Feedstock

Bamboo scrap generated from incense stick processing was subjected to proximate analysis (European Pellet Council 2011), while fiber and silica analysis was carried out according to published method (IEA Shastry et al. 1999). Our results on bamboo proximate analysis (Table 5) are in line with Scurlock et al. (2000) who showed gross calorific value (GCV) 19 GJ/ton on dry basis with low ash and chlorine contents making them attractive for use in biomass combustion applications. The potassium contents of these bamboo samples were also quite low with the alkali index ranging from 0.1–0.3 that is below the empirical limit of 0.17–0.34 kg/GJ known to cause adverse fouling and slagging in combustion systems (Miles et al. 1996; Baxter et al. 1998). This indicates bamboo is candidate feedstock for solid biofuel. Scurlock et al. (2000) and Littlewood et al. (2013) reported bamboo as feedstock for production of lignocellulose ethanol. Therefore, bamboo has potential for solid and liquid biofuel to take it further through innovative technology and linkages for sustainable bamboo feedstock supply.

Potential of Bamboo Farming/Social Forestry and Bamboo-Based Products

Total geographical area of India is 328.2 m ha out of that only 141 m ha area is under cultivation (Trivedi 2010). On other side India has total 115.4 m acres of wasteland available (Wastelands Atlas 2011). It is a wise idea to replicate bamboo-captive farming on marginal/waste land. Over the period of time, bamboo farming would convert non-productive wasteland into productive land with an added advantage of social and environmental impact. This case study showed 25 ton/acre/year of bamboo yield on marginal land with proper agronomic practice. If we consider only 5% utilization of wasteland from total available Indian wasteland for bamboo farming with only 50% of bamboo biomass yield (12.5 ton/acre/year), then one can achieve 72.0 m tones/year bamboo biomass from 60 lac acres of wasteland. Processing of 72.0 m ton of bamboo for incense stick manufacturing at 20% final product conversion can produce 14.4 m tone/year incense stick/tooth pick/chopsticks, while rest 80% (57.6 m tones/year) goes for energy pellet production. Though incense stick/tooth pick/chopsticks processing industry has low and inefficient productivity (20%), the international market value of these products are very high with rate of INR 85,000/ton (∼ US$ 1300/ton), while in India pellet bulk prizing are currently 17,000 INR/ton (∼ US$ 260/ton). Therefore, through bamboo plantation project, several socioeconomic and environmental benefits can be achieved creating job opportunities from farming, micro-business, bioenergy and greenhouse gas emission reduction using pellets by replacing LPG gas, reducing import of fossil fuel and bamboo sticks, and thereby velocity of money remains within the country rising GDP of the country.

Discussion on Case Study-2

Social forestry was initiated in 1980s as a component of 'community development' programs to assist rural communities and landless people to meet their livelihood needs for fodder, fuel wood, small timber, fruits and minor forest produce through nurseries and tree plantations in common lands and non-forest public lands planned and managed by community. However, the focus on this aspect withered with increasing global attention on biodiversity conservation within forests. The revival of social forestry with stronger linkage between communities for better livelihood opportunities and bioenergy industries for low carbon biobased fuel generation needs to be strengthened. Vedeld et al. (2004), Prasad (2006) observed that forest products contribute between 20–40% of the total income of households in forest areas.

Bamboo incense stick production units' efficiency was poor leading to lots of waste generation during the process as could be seen with 80% waste with only 20% finished product recovery. Indian Forestry Outlook Study (APFSOS 2009)

reported that processing technologies in small-scale sectors are generally handicapped due to inefficient operations, legal restrictions, low output and non-availability of skilled labor subsequently leading to inertia.

With the recognition of climate change as a consequence of anthropogenic carbon emissions, every effort is being made to mitigate impact either by reducing emissions or by working on adaptation to the changing situations. In the era of uncertainties, reduction in greenhouse gases (GHG) and carbon sequestration is the only option available.

Conclusions

Theses case studies indicate that for Indian economic growth, it is required to promote bioenergy projects in linkages with social forestry and bamboo farming through innovative financing mechanisms and developing national carbon sink target enabling India to add its green density. Further such linkage-based projects need to be sanctioned on fast track with single-window clearance. Indian quality standards and certification need to be developed based on the local scenario. This would reduce dishonest practices for biomass supply and create infrastructure and improved technologies for bioenergy projects supported by skill developments in upstream and downstream social livelihood products to improve overall efficiency and marketability. The linkages would address carbon sequestration by bamboo farming along with job creation and revenue from livelihood products while bioenergy projects would address carbon emission reduction dropping down fossil fuel use sinking crude import burden of the country.

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Use Wood—Combat Climate Change

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Abstract The most debated non-political issue in the present world is perhaps the matter related to climate change. Deforestation and reduction in forest cover especially in tropical countries have been said to be closely related to this issue. A close insight reveals that growing and use of timber are the only solution to combat climate change. On an average, a typical tree absorbs, through photosynthesis, the equivalent of 1 ton of CO_2 for every cubic metre growth, while producing the equivalent of 0.7 ton of oxygen. Encouraging the use of wood products can act as a greener alternative to more fossil fuel intensive materials. Substitution of a cubic metre of wood for other construction materials (concrete, bricks) results in significant CO_2 savings. The combined effect of carbon storage and substitution means that 1 m^3 of wood stores 0.9 ton CO_2 and substitutes 1.1 tons of CO_2—a total of 2.0 tons of CO_2. For non-conventional energy, the ultimate source, directly or indirectly, is the sun. The total radiation (energy) reaching to earth from sun is 5.7×10^{24} J per year, and total energy (electricity/heat) produced by burning coal/petroleum per day is 54.37 EJ/year (exa-joule, EJ = 10^{18} J). The best way for tapping solar energy is by growing trees which can give material as well as energy. Growing trees and use it as material and energy source will create a cycle for rotation of carbon in atmosphere, sink and store. The resource from which construction/consumable material and energy being made are from minerals, coal and petroleum which are limited in earth storage and will be exhausted one day or other. We have no other choice but to look at the sun for ultimate need of energy. The major question is whether the vegetative system alone can meet the entire nutrient, energy and material need of living kingdom on the earth. For the nutrient supply, the answer is "YES" and already doing so. The living consumers, whether herbivore or carnivore, whether in aquatic or terrestrial system, are dependent, directly or indirectly, on plants which prepares food through photosynthesis or chemosynthesis by utilizing solar energy. Similarly the average residence time in natural forest from 20–120 years can be brought down to 5–10 years with fast

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growing plantation timber. Agricultural residue, which can also be converted into structural component, can be generated in 3–12 months. Now it is the right time to think and utilize what scheme might be used to manage the resources in nature.

Keywords Climate change · Non-conventional energy · Photosynthesis

Shelter is one of the inseparable necessities of human livelihood. Wood from forest, metal made from minerals, cement made from soil and various synthetic-like plastics made from petro-products are the several materials used for construction of houses. For making shelter, human being has still remained dependent on naturally occurring resources.

Timber, mud and stone were the first to use as housing components to build shelter. As the housing technology advanced, materials such as brick, cement, metal and synthetic were added as housing components. Wood can be used as sole component in making houses or many other structural materials (bridge, tower, etc.), and such uses have been favoured since time unknown because wood is easy to process, even with simple hand tools, has higher weight-to-strength ratio and can be shaped to fit into thousands of end-use applications. However, the use of wood, in excess to growing led to severe deforestation round the world, led to changes in climate due to global warming, emission of excess greenhouse gases, pollution of air, water, etc. This has led to grave concern of the environmentalists, and people in general and various governments/authorities round the world have put ban on felling of trees in forest and use of wood and wood product in many sectors. However, use of other housing and structural materials such as metal, brick, cement, plastic remained unabated, rather boosted to replace wood. All these components for their production and application require high energy and emit poisonous gases and discharge harmful effluents, creating severe environmental deterioration.

Since the beginning of industrial revolution, there has been sharp increase in greenhouse gases into the atmosphere mainly due to CO_2 from burning of fossil fuel. About 55–70% additional greenhouse effect is caused by CO_2. According to most optimistic estimates, the increase in CO_2 in the atmosphere is by 0.5% a year (Beyer et al. 2006). As a result, the mean temperature rise is expected to be 0.1–0.4 °C per decade during the first half of this century (Beyer et al. 2006).

The addition of CO_2 to the atmosphere is about 8 billion tons per year mainly from burning of fossil fuel (Beyer et al. 2006). The current vision of development requires energy of which 85% comes from burning of fossil fuel (Beyer et al. 2006). Hence a reduction or stabilization of current level of emission of CO_2 from burning of fossil fuel would involve politically unacceptable cuts in energy consumption. This has been reflected during signing of Kyoto Protocol by the USA which accounts for highest emission of CO_2 in the world.

Deforestation affects environment and climate change. This concept has gone so deep into people's mind, especially through extreme environmentalists lobby, that felling of trees in forest or in public place is treated as criminal as poaching an

endangered species of animal. The other effect of preaching against use of timber is to promote use of non-wood products made from metal, cement, ceramic, plastic and other synthetic polymeric products obtained from petrochemicals. However, the use of non-wood products has the limitations:

i. Production and use are high energy consuming,
ii. Creates harmful effluent, solid waste, greenhouse and other poisonous gases during production and usable life,
iii. Synthetic polymers are not biodegradable and hence degrade soil and water when dumped after useful life.
iv. Raw materials of all these products are non-renewable and have limited stock on the earth.

Tables 1 and 2 show the quantitative measures of the above said limitations:

If the energy for production of non-renewable comes from burning fossil fuel, for each litre of fuel burnt will produce an extra amount of 2.24 kg. CO_2 to the atmosphere. However, if fuel oil is replaced by natural gas, an amount of gas which produces equal quantity of heat adds only 1.65 kg. of CO_2—an amount significantly less. Then again, production and transport, which requires liquid fuels, will consume 13% of the extra energy.

There are two ways to reduce CO_2 in the atmosphere—either by reducing emissions or by removing CO_2 and storing it, that is, reducing carbon sources and increasing carbon sink. Growing trees absorbs CO_2 and produces O_2. On an average a typical tree absorbs, through photosynthesis, the equivalent of 1 ton of

Table 1 Raw material reserve

Sl no	Resource	Reserve	Duration of availability
1	Natural gas	66,609 Tc f	
2	Coal	847 billion tons	118 years
3	Petro-oil	$1.77 \times 10^{11} m^3$	120 years
4	Iron (2 billion m ton/year extraction)	5% of earth	64 years
5	Line stone (115,000 m ton/year extraction)	10% World sedimentary rock	Adequate
6	Aluminium	8.3% of earth's weight (earth's mass = 5.972×10^{21} tons)	Adequate
7	Uranium	35 m tons as mineral on earth crust	
8	Sea plankton	4.6 billion tons in sea water	
9	Tritium	225 kgs produced in the USA	

Source Reference (1984; OPEC Annual Statistical Bulletin 2004; Kakiuchi et al. 1999)

Table 2 Energy required for manufacture of structural products

Commodity	Net energy required [million kcal (oil equivalent) per ovendry ton]
Lumber	0.73
Wood fence post	1.0
Plywood	1.5
MDF	2.14
Concrete slab	2.15
Concrete block	2.21
Clay brick	2.28
Steel stud	12.68
Steel fence post	12.68
Aluminium siding	50.51
Carpet and pad	9.37
–	–

Source Reference Koch (1992)

CO_2 for every cubic metre growth, while producing the equivalent of 0.7 ton of oxygen (Beyer et al. 2006).

Carbon is being exchanged continuously between the different carbon sources and sinks in a process call "carbon cycle". The global carbon balance is given in Table 3.

CO_2 stored in wood continues to be kept out of atmosphere throughout the initial life of wood products and then beyond, through reuse and recycling (e.g. solid wood to particle board), and finally, carbon in wood is returned to atmosphere as CO_2 by burning or decomposition.

The average life of wood products varies between 2 months in newspaper and 75 years for structural wood. Thus any volume of wood storage will reduce CO_2 in the atmosphere. Thus increasing the use of wood and wood products is one of the simple ways of reducing climate change.

If the forest is left entirely to nature, it will attain a climax stage supporting the soil with the maximum amount of biomass fertility under prevailing condition of

Table 3 Global carbon balance (billion tons of carbon/year)

Elements	Emission	Absorption
Combustion of fossil fuel	6.3	–
Deforestation	1.6	–
Total	7.9	–
Seas and other water bodies	–	2.3
Afforestation and increased biomass	–	2.3
Into the atmosphere	–	3.3
Total	–	7.9

Source Reference Beyer et al. (2006)

rainfall and temperature. Although natural regeneration will occurs, the dead and dying trees will decay or burnt, emitting CO_2 from the stored carbon. Growth is matched by decay, and with no forest management, there would be no increase in carbon storage.

Encouraging use of wood products can act as a greener alternative to more fossil fuel intensive materials. Substituting a cubic metre of wood for other construction materials (concrete, bricks) results in significant CO_2 savings. The combined effect of carbon storage and substitution means that 1 m^3 of wood stores 0.9 ton CO_2 and substitutes 1.1 tons of CO_2—a total of 2.0 tons of CO_2. Thus more wood products replace other materials, the more the so-called substitution effect further reduces CO_2 in the atmosphere. **CO_2 reductions achieved by wood products are eligible under Art. 3.4 of the Kyoto Protocol and the wood working industries may be granted carbon credits in the framework of the emission trading schemes** (Report 2002).

Wood energy is CO_2—neutral and clean:

Wood is an excellent combustible material and has been source of energy since the dawn of human civilization. Wood energy can be derived from wood in raw form, bark, saw mill and shaving residues, residues from panel and furniture manufacturing units, forest residues and wood recovered from consumer products after end of useful life.

Burning of wood provides carbon neutral substitute for fossil fuels. Since it only returns CO_2 that has been from atmosphere for growing of trees, wood combustion does not contribute to global warming or greenhouse gases. Further wood contains little sulphur and nitrogen which contribute to acid rain and also it produces little ash. Thus wood energy is clean.

Energy in the form of heat and electricity is the most needed element required to keep flow of modern civilization. Modern lifestyle will be paralysed without supply of energy in either of the forms. Coal and petroleum are the major source from which energy is being harnessed. Other minor sources are wood and agricultural residue. Energy is also generated from atomic fission, hydro power, wind power and solar radiation. But these are minor sources.

To keep up with the present status of life or better, both materials (as mentioned) and energy (heat, electricity) are required. Resource availability indicates that reserve on the earth is 100–150 years. What then? In fact, human civilization has reached to such a stage with respect to sourcing of energy which is comparable to human in prehistoric days when they adopted practice of cultivation for growing food abandoning collecting/hunting food from natural resource.

The crisis of energy had been felt long back, and search for energy from renewable or perennial sources had been initiated. Success has been obtained in tapping energy as solar energy, wind energy, hydro energy to produce both heat and electricity. Nuclear energy is another potential and successful area. But till today, total energy produced from all these sources compared to energy produced from conventional sources such as coal and petroleum is very negligible.

Table 4 shows the potential availability and present exploitation of renewable energy source in India.

Table 4 Renewable energy source: potential/availability and exploited

Source/technology	Units	Potential/availability	Potential exploited
Biogas plant	Million	12	3.22
Biomass-based power	MW	19,500	384
Efficient wood stoves	Million	120	33.86
Solar energy	MW/km^2	20	1.74
Small hydro	MW	15,000	1398
Wind energy	MW	45,000	1367
Energy recovery from waste	MW	1700	16.20

Source Reference (Ellabbana et al. 2014)

It must be recalled that renewable energy capacity is always much larger than how it can actually deliver. It is variable in supply as far as wind and solar are concerned, and there is yet no inexpensive way to store and use of the energy later. At present, all renewable energy is expensive compared to present ones. It is unlikely in the foreseeable future that renewable energy can meet the vast additional needs of energy. It can only supplement the supply.

Whenever and wherever scientists have looked for non-conventional energy, the ultimate source, directly or indirectly, is the sun. The total radiation (energy) reaching to earth from sun is 5.7×10^{24} J per year, and total energy (electricity/heat) produced by burning coal/petroleum per day is 54.37 EJ/year (exa-joule, EJ = 10^{18} J).

The best way for tapping solar energy is by growing trees which can give material as well as energy. Growing trees and use it as material and energy source will create a cycle for rotation of carbon in atmosphere, sink and store. If wood is burnt, it will not dump extra carbon dioxide as carbon dioxide will be removed from air by growing of trees. Use of conventional fuel such as coal/petroleum/natural gas infuses carbon dioxide and other acidic gases into atmosphere while burning. This does not happen while burning wood.

An Alternate Thinking is now Necessary

In ancient time, as man adopted the process of cultivation for growing food grain, hunting was treated as hated profession as well as cutting of trees; rather, Aryan culture suggested worshipping of trees. Through experience, man of today has learnt that how useful are trees and forest to keep the earth worth living. Felling of trees in forest is being treated as illegal, and this has encouraged use of wood alternative such as metal, cement, plastic, petroleum coal. Use of these materials has definitely helped in quick advancement of the human civilization but simultaneously made the soil, water bodies, air more and more polluted. If this continued at the present rate, days are not far when the world would be unsuitable for healthy living.

Nutrient and other materials are continually recirculated within and among ecosystem, and by and large there are no new inputs or losses from the planet. In terms of material, the earth is a closed system. But both energy and materials are essential to ecosystem structure, function and composition. In terms of cycling of carbon, materials and energy can be inter-converted. Energy is used up and lost as heat as it moves through ecosystem and new energy is continually added to the earth in the form of solar radiation.

Can energy requirement of the world be met from solar energy only? Approximately 5.7×10^{24} J of solar energy are irradiated to the earth's surface on an annual basis. Plants and photosynthetic organisms utilize this solar energy in fixing large amount of CO2 (2×10^{11} tons = 3×10^{21} J/year), while amount consumed by human beings are relatively small [3×10^{20} J/year], representing only 10% of the energy converted during photosynthesis (Hall D.O. Biomass for Energy, ED. Hall. D.O. 1–18 1979).

The low conversion of solar energy into primary energy source for human use is due to lack of effective conversion and storing system. Although large amounts of solar energy are irradiated to the earth's surface, the effective energy concentration (energy/unit area) of solar energy at any one point on the earth surface is small—only about 1 kW/m^2 at most even at noon. Such low effective energy concentration limits the use of solar energy as a primary energy source and elevates the costs associated with its accumulation and transmission.

Still the big issue at the moment is the availability of most renewable energy source to make electricity. Prior to industrial revolution, wood served as a major energy source. The industrial revolution gave rise to the widespread use of both coal and petroleum as energy source. However, all fuel prices, the world over, have been rising in sympathy with those of oil and gas. With increasing quantity of energy requirement, more expensive sources of fuels are inevitable, such as coal, LPG, petroleum, and uranium.

We have no other choice but to look at the sun for ultimate need of energy and material. The life on the earth is surviving and has become sustainable because of sustainable supply of food. We are using the natural process—photosynthesis—to grow food as much as required. Food supply is nothing but cycling of material (mainly carbon in atmosphere) which are being synthesized by nature by using CO_2, H_2O and solar energy. The basic organic molecule—glucose—formed in this way is transformed into various organic compounds. These compounds are utilized by living being for livelihood. At the end of life, organic bodies decompose and carbon goes back to air and water or soil. Ultimately it is the absorption and radiation of solar energy which keeps the life on this earth sustainably.

Nature is distinctly pointing out that energy cannot be sourced other than the sun in indefinite quantity for indefinite period. Today's major source of energy—coal and petroleum—is nothing but the creation of nature through photosynthesis and has buried during some prehistoric days and transformed into coal and petroleum. Unless we learn and adopt the art of tapping solar energy to meet our need for energy and remain increasingly dependent on store source such as coal and petroleum as source of energy, the end of civilization is not far off. Only the part of life

sustainably and totally dependent on nature will survive; the rest of life on the earth, which is dependent on nature's stored for energy and material, will perish.

Before the discovery of coal and petroleum, wood and agro-residues were the only material to be burnt to generate heat. With the discovery of coal and petroleum, these fuels started replacing wood rapidly as fuel. Industrial revolution enhanced the use of coal and petroleum-based fuel (diesel, petrol and natural gas). Automobiles, ship, aeroplanes are totally based on petroleum-based fuel. LPG is rapidly replacing any other domestic fuel for their cleanliness and other advantages of use. Replacement wood by other materials as fuel to generate heat and energy has been greatly augmented by growing environmental consciousness around the world for protection of forest.

Conservation of natural forest is necessary to maintain ecological balance, biodiversity, water sources and to combat global warming. If good consciousness does not work to restrict in discriminate felling of trees or destroying forest, there is reason to impose strictest rule to ban on felling in the forest. But social need for wood cannot be denied.

To meet the social need for wood, almost all countries throughout the world have initiated plantation and supply wood to meet the demand from plantation. In some European countries, Canada and South America, Russia, the plantation is sufficient or surplus to meet the national demand for wood at the present rate of consumption. Most of the countries do not yet use wood for energy generation, although in many European countries energy sector has been advised to utilize 20% of wood as fuel. Many governments have announced subsidies for wood supply to energy-producing units. This has, of course, raised great debate in some countries and is being strongly opposed by wood-based industries. Of course, in those countries the opposition to utilization of wood for energy generation (by burning) is not an environmental issue but rather economical. Supply of wood to energy units at subsidized rate will affect the economy of running the wood-based industries as well as the supply chain.

Compared to effect on economy of the wood-based industries, the disturbance in supply chain of wood is more important because non-sustainable supply may lead to the question of survival of the industry. The question which needed to be answered: Is the net production of timber and consequently supply to both the wood industry and energy units is sufficient to do that on a sustainable basis? Going a step further, if we assume that due to short supply of coal and petro-fuel if all the countries in the world adopt a common policy to utilize wood as source of energy, will the land surface on the earth is sufficient to produce enough trees to meet both the material need and energy need of the growing demand of the society?

The major question is whether the vegetative system alone can meet the entire nutrient and material need of living kingdom on the earth. For the nutrient supply, the answer is "YES" and already doing so. The living consumers, whether herbivore or carnivore, whether in aquatic or terrestrial system, are dependent, directly or indirectly, on plants which prepares food through photosynthesis or chemosynthesis by utilizing solar energy (Table 5).

Table 5 Total surface area on the earth and vegetative cover

Ecosystem type	Surface area ($X10^6$ km^2)	Net primary vegetative production (Pg.)
Forest	31	48.7
Woodland, grassland and savannah	37	52.1
Desert	30	3.1
Arctic alpine	25	2.1
Cultivated land	16	15.0
Human area	2	0.4
Other terrestrial (chaparral, bogs, swamps, mashes)	6	10.7
Subtotal terrestrial	**147**	**132.1**
Lakes and streams	2	0.8
Marine	361	91.6
Subtotal aquatic	**363**	**92.4**
Grand total	510	224.5

Pg = Pedagram = 10^{15}gm = 10^9 tons
Source Reference (Brovkin et al. 2013)

Net primary production (NPP) is calculated as annual harvest. In a cropland, NPP and annual harvest can exceed annual NPP (for example, when a forest is cut down the harvest is of many years of growth), but we can still compute annual averages.

Nature, of its own, may not produce enough to feed all living being on the earth, but man has discovered cultivation where nature's own process of photosynthesis is being utilized to tap solar energy in carbon from air to prepare compounds to store energy and material supply (wood and others). On the land, only human being commands about 40% of the total terrestrial NPP. This has probably never occurred before in earth's history. However, human use of marine productivity is relatively small. Moreover, although major fish stocks are heavily fished and many coastal areas are severely polluted, human impact on the seas is less than on land. As of today, a sustainable food chain has been established to all living consumers to supply from aquatic (sea, river, lake, fisheries) and terrestrial (natural or through cultivation). The sustainable food supply may continue indefinitely unless disturbed by demand—supply gap.

Except human being, no other living being require additional energy other than through food and from sun rays. In respect energy, supply is not sustainable and we are greatly dependent on sources which are stored in the earth. 80% of the energy necessary to run our society comes from fossil fuel. Fossil fuel is nothing but remains of surplus biomass which was buried for long period of time million of years back and had undergone biochemical changes under heat and pressure. It is impossible that under the present rate of consumption of biomass any biomass deposit is pilling up anywhere on the earth. Stored fossil fuel will be exhausted sooner or later, and no fresh store of fossil fuel is going to be created unless the human civilization is completely destroyed and life starts freshly on the earth.

Use of inorganic material such as stone, minerals excavated from earth is being practiced by human being since centuries. Use of stone has been limited due non-availability and competition from more suitable structural products from metal and cement. Mining is going to be restricted in many places.

At the first instance, total dependence on timber for energy and material may look impossible and future may look bleak. But looking at the sustainable ecosystem in the ocean, the above question may be answered. The sustainable supply is not a matter of the quantity in stock at a particular time but the all important aspect of "TIME". Even though the total standing trees may be small, the RATE at which plantation trees produced may be very large. Thus over time it is the total number of plantation trees that is being produced, from whatever may be source or species may be, that is, important for sustainable supply.

A very good example is seen in the ocean where most of the primary production is concentrated in microscopic algae. Algae have short life cycles, multiply rapidly, do not generate much biomass relative to their numbers and are eaten rapidly by herbivores. At any given point of time, the standing crop of algae in an ocean is likely low, but the turnover can be high. Algae populations can double in few days, whereas zooplankton might only reproduce once a year.

Similarly average residence time in natural forest ranges from 20 to 120 years, which can be brought down to 5–10 years with fast growing plantation timber. Agricultural residue, which can also be converted into structural component, can be generated in 3–12 months. Now it is the right time to think and utilize what scheme might be used to manage the resources in nature.

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