Influence of Moisture Absorption on Mechanical properties of Biocomposites reinforced Surface Modified Natural Fibers

M. Ramesh, L. Rajeshkumar, D. Balaji, and V. Bhuvaneswari

Abstract Recent experimentations witnessed natural fibers as a strong alternative reinforcing element for the synthetic fibers due to their biodegradable and ecofriendly nature. Yet natural fibers are characterized by an inherent disadvantage of poor bonding at the interface of fiber and matrix with that of matrix material which renders substandard mechanical and physical properties of the natural fiber composites. Moisture absorption behavior of the natural fibers is one another demerits of using the natural fibers, since this behaviour may pose the natural fibers to absorb moisture from the surrounding environment owing to their hydrophilic nature. Surface modification renders solution to this problem also by making the surface modified natural fiber to be hydrophobic. Various treatment methods of the natural fibers and its reinforcement in polymeric matrices were dealt in detail in the current chapter. Alongside, the effect of moisture absorption of the natural fibers upon the mechanical and physical properties of the natural fiber reinforced composite materials are also discussed. It was observed from different studies that various characteristics like force of adhesion at the interfacial region between the natural fiber and the polymeric matrix enhanced due to surface modification since the surface area of contact increases which in turn enhanced the mechanical behaviour of the composites and minimized the moisture absorption of the natural fiber composites.

Keywords Natural fibers · Surface modification · Biocomposites · Moisture absorption · Mechanical properties

M. Ramesh (\boxtimes)

L. Rajeshkumar · D. Balaji · V. Bhuvaneswari

Department of Mechanical Engineering, KPR Institute of Engineering and Technology, Coimbatore, Tamil Nadu 641407, India

Department of Mechanical Engineering, KIT-Kalaignarkarunanidhi Institute of Technology, Coimbatore, Tamil Nadu 641402, India

[©] The Author(s), under exclusive license to Springer Nature Singapore Pte Ltd. 2022 C. Muthukumar et al. (eds.), *Aging Effects on Natural Fiber-Reinforced Polymer Composites*, Composites Science and Technology, https://doi.org/10.1007/978-981-16-8360-2_2

1 Introduction

Natural fibers are the order of today's experimental materials owing to their various benefits like biodegradability, ease of availability, less in cost and better ability to be recycled. Yet, the moisture absorption behaviour of the natural fibers is considered to be an alarming factor for their usage which lowers the adhesion between matrix and natural fibers and their compatibility (Bhoopathi et al. [2015;](#page-15-0) Ramesh [2018\)](#page-16-0). Natural fibers possess few more demerits like inherent incompatibility with matrix and poor wettability. Possible reason for the incompatibility between matrix and fiber could be the lowest magnitude of adhesive forces existing between the untreated fibers and the matrix (Vimal et al. [2015\)](#page-17-0). Hence in order to improve the fiber and matrix adhesion, fiber surface treatments has become necessary to make the modifications in the surface of the fiber. Fiber surface modification with various chemicals in different concentrations may assist to obtain enhanced fiber matrix adhesion (Jayaramudu et al. [2009;](#page-15-1) Li et al. [2007\)](#page-16-1). As the surface of the fibers were turned to possess more roughness, their adhesion with the matrix increases thus rendering better resultant characteristics.

2 Surface Treatment of Natural Fibers

Surface treatment of the natural fibers may make the natural fibers to dodge the poor surface adhesion and render a better bonding with the matrix element upon surface modification. Various physical and chemical surface modification practices are under existence currently out of which chemical treatment of fiber plays a major role. Surface modification methods like alkaline, benzoylation, permanganate and acetylation treatments are widely carried out to enhance the moisture absorption and mechanical characteristics of the end composites formed out of natural fibers (Ramesh et al. [2017a;](#page-16-2) Ramesh [2016\)](#page-16-3). Surface modification removes micro constituents like pectin, lignin, hemicellulose and cellulose thereby indirectly enhancing the aforementioned properties. Most of the researches during the current days focused in using silane treatment for natural fibers (Ramesh et al. $2017b$). SiH₄ is the chemical formula for silane and they are the commonly used coupling agents for the stabilization of reinforced fibers and the polymeric matrices by blending them. Silane coupling agents remove all the hydroxyl groups in the cellulose at the interfacial region of matrix and the fiber. Silanes were formed from the hydroxyl alcoholic elements with moisture presence and the formed silanol then undergoes reaction with fiber hydroxyl group resulting in stronger covalent bonds along with the cell membrane surface. It was stated by the experimenters that the fiber cramping was minimized by polymeric chains of hydrocarbon due to the formation of cross-linking structure owing to the silane treatment and a covalent bond is established between matrix and fiber (Agrawal et al. [2000\)](#page-15-2).

3 Moisture Absorption of Natural Fibers

Moisture absorption nature of the natural fibers render a hydrophilic nature of fibers and when such fibers were reinforced in hydrophobic matrix it results in poor interfacial characteristics owing to the presence of cellulosic group with higher hydroxyl group (Alvarez et al. [2003\)](#page-15-3). Few experiments employed E-glass and date palm fibers to fabricate hybrid epoxy composites and the fibers were treated to evaluate their moisture absorption behaviour. Date palm fibers were extracted from the meshes around the stem of the palm trees and the fibers were manually extracted from the mesh after which they were washed with distilled water to get rid of dirt and dust. Then the fibers were dried in hot air oven at a temperature of 80 °C for a period of 24 h. Followed by that the fibers were chopped to a length of 2 mm, washed with 3:1 ratio of acetone and ethanol solution respectively and then by distilled water so that the oil, wax and chlorophyll contents existing in the date palm leaf fibers. Finally, 1% sodium hydroxide solution was used to treat the date palm fibers and were dried at atmospheric conditions for 24 h. By making the fiber to undergo alkali treatment, the hydrogen bonding of the fibers were disintegrated thus enhancing the roughness of the fiber surface. Another important aspect of alkali treatment was that it makes the fibers to be devoid of oils, lignin and wax that existed in the external surface of the fiber cell wall. Additionally, the glass fibers were also chopped to a length of 2 mm from continuous glass fiber roves for fiber treatment with the aid of an electronic controlled fiber cutting machine for better accuracy and were dried at 80 °C for a period of 24 h. Such surface modified fibers when used in composite manufacturing, they possessed minimum moisture absorption according to the experimental results (Swain et al. [2018\)](#page-17-1).

4 Mechanical Properties

The experiments were carried out on the hemp fibers whose surface were modified by fungal treatment and reinforced in polyester matrix to evaluate the moisture absorption behavior. Results showcased that the surface modified fibers exhibited enhanced acidic-basic properties and possessed better hydrophobic characteristics. Meanwhile, the treated fibers exhibited better mechanical properties when compared with its counterparts (Ramesh et al. [2020a\)](#page-16-5). It was stated in many studies that the strong adhesion at the interfacial region between matrix and fibers plays a major role in controlling the performance of natural fiber reinforced polymer composites which would be predominant in case of hydrophilic fibers. Experiments conducted using Grewia optiva fibers which had more polar groups on the surface revealed that the untreated fibers possessed hydrophobic nature, weak bonding at the fiber matrix interface and poor fiber wettability with matrix. Various treatments like benzoylation, grafting of vinyl compounds, alkali and silane coupling agents were carried over on the fibers to reduce the fiber hydrophilicity (Gulati and Sain [2006\)](#page-15-4). Results indicated that the untreated fiber composites had a higher dielectric constant when compared with the treated fiber composites. This was attributed to the moisture absorption of the natural fibers from the surrounding and the hydrophilicity rendered increased conductivity (Singha et al. [2013\)](#page-17-2). Hence the surface modification of the Grewia optiva fibers rendered minimum dielectric constant for the resultant bio-composites which was attributed to the reduced polar group orientation in case of the treated fiber. Various aforementioned treatments on Grewia optiva fibers made the fibers to be hydrophobic since the A-OH active groups of the fibers in their backbone were blocked by the surface modification (Kulkarni et al. [1981;](#page-15-5) Singha and Rana [2013\)](#page-17-3).

Few experimenters tried to characterize the treated and untreated banana fiber reinforced polyester composites with respect to their physical, chemical and mechanical characteristics as banana fibers possess various better properties such as better stiffness, strength and appreciable fiber length along with good moisture absorption characteristics. It was stated from the results that the alkali treated banana fibers exhibited better mechanical and moisture absorption behaviour due to the absence of hemicellulose in the surface of the treated fiber (Balaji et al. [2020\)](#page-15-6). Jute fibers reinforced in thermoplastic polyurethane matrix was manufactured with the aid of twin-screw extruder machine with a fiber volume fraction of 30%. Before making the composites, the fibers were oven dried at 100 \degree C for 8 h period to get rid of moisture interference at the time of composite extrusion. Fibers were treated with benzoyl chloride and the parameters used for extrusion of fibers as 100 rpm screw speed, 200 °C as processing temperature and 5 min of mixing time. It was observed from the results that the treated fibers were much hydrophobic when compared with the untreated fibers (Sathish et al. [2018\)](#page-17-4). Studies on bamboo fiber reinforced epoxy composites were carried out to evaluate the mechanical and moisture absorption behaviour of the composites. Bamboo fibers were treated with sodium hydroxide to remove the micro constituents like lignin, wax, hemicellulose, oil and inorganic salts that were encompassed around the surface of the bamboo fiber and then the formed composites were subjected to mechanical and moisture absorption properties evaluation. Owing to the rough fiber surface morphology, the flexural and moisture absorption behaviour of the bamboo fiber composites enhanced and an appreciable chemical and mechanical interlocking was also induced due to chemical treatment. It was also observed that the surface modified bamboo fibers had better interfacial adhesion along with increased hydrophobicity due to the conversion of polymeric bonds into monomers and exposure short crystallites of bamboo fiber (Singha and Rana [2012;](#page-17-5) Barathkumar and Gokulprakash [2018;](#page-15-7) Salem et al. [2020;](#page-16-6) Kushwaha and Kumar [2010;](#page-15-8) Saravana Kumar et al. [2017;](#page-16-7) Indraja et al. [2014;](#page-15-9) Petinakis et al. [2013\)](#page-16-8).

Natural fibers of flax were reinforced with PLA matrix and the mixture was compounded in a melt mixer of model Haake Rheomix at a temperature of 185 °C. Before compounding the matrix with the reinforcement, PLA matrix was dried at 90 \degree C for a period of 2 h in order to reduce the degradation of PLA matrix by hydrolytion. After compounding and fabricating the composites, the moisture content of the composites containing treated and untreated fibers were 1% to 5% (Phuong et al. [2010\)](#page-16-9). Mechanical characteristics of the natural fiber reinforced composites were influenced by various factors like moisture absorption ability of the fiber, fiber

loading and fiber length. It was also noticed that at a low absorption of moisture and at optimum loading condition and length the natural fiber composites had enhanced mechanical characteristics. On the other hand, moisture absorption of the natural fibers influenced the thermal behaviour of the composites. Thermal stability of the composites dropped in case of untreated fiber composites while the composites with treated fibers had better thermal stability. It was stated by the authors that when flame retardants were added as fillers to the above composites, their thermal stability increased further (Sathish et al. [2017\)](#page-16-10).

Various researchers stated that the conductivity of cellulose based natural fibers was due to the hydrophilic nature of the composites. As the hydroxyl group of the natural fibers turned them to be hydrophilic, their moisture absorption rate increases which paved way for the enhanced conductivity of the natural fiber reinforced polymeric composites. It was also noted from the earlier experiments that the alkaline treatment of the natural fibers reduced their dielectric constant and hence the conductivity values which could be attributed to the reduction of orientation polarization of the composites containing alkaline treated natural fibers. As mentioned earlier blockage of active OH group of the natural fibers by the alkaline compounds rendered minimum moisture absorption characteristics of the natural fibers (Yan et al. [2012\)](#page-17-6). Sisal fibers reinforced in polylactic acid matrix were subjected to dielectric constant evaluation and the results showed that the treated sisal fiber composites had better dielectric constant when compared with untreated dielectric constant which could be due to the lignocellulosic constituents present in the sisal fibers. This could also be attributed to the heterogeneous nature of the sisal fiber composites and the chemical constituents present in it. Treated sisal fibers had 43–88% of cellulose, 10–21% of moisture, $10-14\%$ of hemicellulose and $5-12\%$ of lignin content as per the test results (Ramesh and Rajeshkumar [2018\)](#page-16-11).

Dimensional instability and swelling were noted to be end effects of high moisture absorption of fibers and reduced mechanical characteristics and these anomalies resulted due to the weak interfacial adhesion existed in between hydrophobic matrix and hydrophilic fibers. Natural fiber reinforced composites were converted into high performance materials by various physical and chemical surface modifications which indirectly enhances the interfacial characteristics between the natural fiber and matrix (Yan et al. [2012;](#page-17-6) Sethy [2011;](#page-17-7) Orue et al. [2015\)](#page-16-12). Hydrophobicity of the natural fibers can be enhanced by the surface modification through chemicals which also improves the fiber matrix compatibility. Bonding between the fiber and the matrix is also enhanced due to the chemical modification of fiber surface structures which also minimizes the moisture absorption behaviour of the natural fibers. Optimal fiber characteristics could be obtained through the selection of appropriate process parameters and fiber volume fraction. Fiber inherent characteristics like chemical composition, morphology, crystal structure and surface chemistry decides the ultimate properties of the composites and the matrix properties like functionality and nature also emphasizes the same (Singh and Samanta [1163;](#page-17-8) Sigha and Rana [2013;](#page-17-9) Sathish et al. [2019\)](#page-17-10). Biodegradation of plastic matrix resulting in its fragmentation would take place in suitable conditions like oxygen availability, metallic elements presence, moisture absorption, temperature, humidity and the value of pH which

ends up with harmless residue generation which would also be non-toxic (Karthi et al. [2020;](#page-15-10) Mohanty et al. [2005\)](#page-16-13). Cellulose natural fibers were surface modified by acetylene and the process of modification is termed as esterification treatment and is known to be the better method for the cellulose fibers. Moisture that was present in the natural fibers were taken out by the active $CH₃-COO$ acetyl group of the acetylene by undergoing a reaction with the hydroxyl group of the hydrophilic fiber. This renders a better dimensional stability to the natural fibers thereby reducing their hydrophilic nature and improving the dispersion of fiber within the matrix material. As the hydroxyl group of the natural fibers were replaced by the acetyl group during the acetylation, fibers turned more hydrophobic after the acetylation treatment (Chigondo et al. [2013\)](#page-15-11).

4.1 Cyclic Moisture Absorption and Desorption

Few experiments were conducted over the Luffa fibers reinforced in PLA matrix and their hot and cold water absorption and desorption cycles along with few mechanical characteristics like impact and flexural strengths were evaluated. Heat treated Luffa fibers in volume fractions of 5%, 10%, 15% and 20% were reinforced within the PLA matrix and the test were conducted. Luffa fiber composites which exhibited maximum impact and flexural strengths were taken for the analysis of water absorption and desorption where the composites were subjected to a total of 56 cycles of cold and hot water desorption and absorption. Strength of the composites were evaluated after every 14 water absorption and desorption cycles. It was observed from the results that the reduction of flexural and impact strength occurred during absorption and desorption of water and meanwhile impact strength of the Luffa fiber composites faced a major reduction than the flexural strength. SEM images of the Luffa-PLA bio composites were used for the analysis of their microstructure (Sujaritjun et al. [2013\)](#page-17-11). Figure [1](#page-6-0) shows the SEM images of the moisture absorbed samples at different time intervals. The water absorbed matrix layer is clearly visible, which is reported that there has been a maximum amount of water immersed in the first two hours after immersion. It swelled, the the surfaces of the composite specimens are shown. The presence of water molecules within the sample are also visible (Ramesh et al. [2016\)](#page-16-14).

4.2 Polylactic Based Composites

Few experimental results exhibited that the tensile strength and stiffness of the unreinforced polylactic acid (PLA) based composites were not upto the mark for any load carrying application. Hence the use of natural firs which were less in cost, biodegradable, less in weight with high stiffness were recommended as reinforcements particularly for manufacturing methods like 3D printing of natural composites. It was stated that alkali, acid or silane treatments were most often used for the surface modification

Fig. 1 SEM images of the moisture absorbed composite samples after **a** 2 h, **b** 3 h, **c** 4 h, and **d** 5 h (Ramesh et al. [2016\)](#page-16-14)

of the natural fibers in order to improve their interfacial adhesion between matrix and the fiber. Few experiments were carried out on the epoxy modified pine fibers reinforced in PLA matrix which were manufactured by impregnation method and the fiber volume fraction was maintained as 30%. When compared with the unreinforced PLA, epoxy modified pine fibers exhibited an increase of tensile strength and Young's modulus by 20% and 82% respectively which was purely due the surface modification of pine fibers by epoxy treatment. It was also found from the results that 1 wt.% of epoxy modification resulted in less void formation in pine-PLA composites when compared with the pine/PLA composites without epoxy surface modification. SEM images, as depicted in Fig. [2,](#page-7-0) showcases the penetration of epoxy partially into the interior structural pores of the pine fibers which rendered an improved fiber matrix adhesion. It was concluded by the authors that epoxy modification was found to a simple yet effective method of surface modification to obtain better bio composite properties. Figure [2](#page-7-0) denotes the various stages in water absorption of pine fibers with PLA biocompoistes and the morphology of treated fibers which had enhanced moisture resistance (Faruk and Sain [2014;](#page-15-12) Zhao et al. [2020\)](#page-17-12).

Figure [3](#page-7-1) shows the various processing methods and morphology of untreated and treated pine fibers along with the physical properties of various unreinforced

Fig. 2 Moisture absorption behavior of bio composites with natural fibers (Zhao et al. [2020\)](#page-17-12)

Fig. 3 a Processing of PLA based biocompoistes, SEM morphology of **b** Pine fiber surface, **c** Pine fibers cross section, **d**, **e** Epoxy treated pine fiber surfaces, **f** Epoxy treated pine fiber cross section, **g** Variation of tensile strength and Young's modulus of PLA-pine-epoxy composites (Zhao et al. [2020\)](#page-17-12)

and PLA/epoxy reinforced pine fiber composites. Figure [3g](#page-7-1) denotes the variation of tensile strength and Young's modulus of unreinforced PLA (A), PLA reinforced with pine fibers (B), PLA reinforced with pine fibers treated with 0.5, 1, 2 and 4 parts of epoxy (C, D, E, F respectively).

Usually the thermal stability of natural fiber reinforced polymer composites were carried out in a thermogravimetric analyzer of model Q500 in a nitrogen atmosphere at a 20 mL/min flow rate of purge gas. Natural fiber composite samples were heated to temperatures in between 35 and 70 $^{\circ}$ C at a rate of heating of 10 $^{\circ}$ C per minute and was maintained at a temperature of 70 ºC for a time of 20 min in order to get rid of the moisture absorbed by the natural fibers. Again the samples were heated at a rate of 10 ºC/min for a maximum of 700 ºC to assess their behavior. Few experimental results showed that the use of PLA/natural fiber composites face few constraints in temperature related applications owing to their lower flammability resistance and high moisture absorption ability. Diammonium Phosphate (DAP) was one among the commonly used chemical used for the modification of natural fiber surface to improve the flammability resistance of PLA/natural fiber bio composite. Few aspects like low tensile strength, high flammable behavior and moisture absorption hindered the application of PLA/natural fiber composites. Through experimentation, various researchers proposed that this could be improved by various chemical modification methods (Zhao et al. [2020;](#page-17-12) Ramesh et al. [2020b\)](#page-16-15). Figure [4](#page-8-0) denotes the variation of decomposition temperature and peak temperature for the natural fiber reinforced PLA composites.

Few experiments were carried out with PLA matrix reinforced with date palm fibers which were fabricated using a twin screw extruder of Leistriz LSM 34 corotator type having an aspect ratio of 29 and a screw diameter of 34 mm. PLA pellets

Fig. 4 Variation of decomposition and peak temperature (Zhao et al. [2020\)](#page-17-12)

Fig. 5 Ganoderma lucidum fibres reinforced in PLA matrix (Chin-San [2014\)](#page-15-13)

were oven dried at 70 °C for 3 h with a hot air blow and the date palm/PLA ingredients were fed into the extruder hopper with a volumetric feeder at a rate of 4 kg per hour. Venting port at the extruder machine made the mixture devoid of volatile compounds and residual moisture. After extrusion, the mixture was made into pellets by a rotating cutter and the extruded elements were then cooled by feeding them into a water bath (Goriparthi et al. [2012\)](#page-15-14). Cosmetic application of the PLA was observed to be mainly due to the retaining of moisture by the lactate which in turn was due to the hydrophilic nature of the lactic acid. Ethyl lactate possess an appreciable solvency with various polymeric elements and oils which made them suitable for the preparation of antiacne compounds (Chin-San [2014\)](#page-15-13). Experiments were also conducted by few researchers with Ganoderma lucidum fibers reinforced in PLA matrix. Water absorption behavior of the above natural composites were investigated and determined that the surface modification of Ganoderma fibers rendered a hydrophobic composite when compared with the untreated composites. Figure [5](#page-9-0) shows the process of washing the Ganoderma lucidum fibers, treating them and blending them with PLA matrix along with its chemical structure after the surface modification with acetic acid blended in a twin screw extruder (Suprakas and Mosto [2005\)](#page-17-13).

4.3 Kenaf Fiber Composites

Fibers existing in nature plays a significant role for producing green composite for both manufacturing and investigation areas due to its viability. Relations with water and other elements which are convenient for thermodynamic property in natural fiber is a crucial part for the effective outside usage. So for these reasons investigation of both absorption and adsorption nature of kenaf or glass polyester mixture composite

with various stacking order by using water as a medium is a major aim of this task. At the same time investigating the mechanical characteristics of the fiber is also essential part here. Twenty days required for the composite which need to wet completely at actual temperature for conducting moisture absorption experiment. At equilibrium the content of moisture (M_m) , coefficient of diffusion (D_r) and water transport mechanism were the unique factors investigated through this test. Fickian's diffusion law used to calculate water transport mechanism of mixed composites in which polymer relaxation time (t_r) is higher than the characteristic solvent diffusion time (t_d) . Mechanical characteristics like tensile and flexure strength of the protective layer of the composites were derived by using both absorption and adsorption method. The quality of being physically strong was calculated that got reduced with addition of water intake and depends on the arrangement of long thin parts of a kenaf fibre at the protective layer (Suprakas and Mosto [2005\)](#page-17-13). Coefficient (n), coefficient of diffusion (d_x) and moisture gain percentage were the major influencing factors for moisture uptake investigation for various laminates. Those influencing factors were arranged in a column.

Moisture uptake for several composites was explained in the below image with root square value of time (H_r) , at the initial stage, moisture uptake curve is linear, later maintained a value and saturated at the final time period. Few observations have been done that, glass fiber composite possesses a value of 2.314% which is less value of moisture gain and attained the saturation level beyond 288 h. Kenaf composite possesses a larger value of 10.560% for moisture gain and attained the saturation level beyond 432 h. Observations carried out for mixed composite of kenaf /glass and found the moisture gain value which is intermediate of those two composites. Hybrid composite of glass and kenaf fiber results improved moisture gain value than the hybrid composite of kenaf and glass fiber. The reason for this variation is protective layer acting in glass kenaf fiber restricts the dispersion of water particles since it is acting as a protective layer. Fickian's diffusion used to find the absorption of moisture for experimental composites which is having a coefficient (n) nearer to 0.5 which depicts clearly in the table. Transferability of the particles which can dissolve in to the solution of the polymer composite is explained by the coefficient of diffusion. It was observed that diffusion coefficient is linearly proportional to value of moisture gain and found maximum in protective layer of kenaf fiber which is not in a case of glass fiber since the fiber of glass oppose the humidity content (Sorrentino et al. [2007\)](#page-17-14). Decrease of stability of the fibers has been studied by considering tensile and bending characteristics of those fibers with the inclusion of three levels in the below images. It was observed that in glass fibers, maximum elongation stability is more and if we include kenaf means it gets reduced slightly. Kenaf fiber composite possess a reduced elongation stability compare to other fiber composites. The reason for this change is if the laminate strength is more, then it can merge with glass fiber correctly (Mishra et al. [2020\)](#page-16-16).

Figure [6](#page-11-0) denotes the weight loss of the natural fibers due to moisture absorption and the variation of ultimate tensile strength of the natural/synthetic fibers reinforced polymer composites. Absorption of moisture is also one of the reasons for decrement of stability of the fiber composites. In kenaf fiber composites, around fifty one

Fig. 6 Effect of Moisture absorption on the weight loss/gain (Mishra et al. [2020\)](#page-16-16)

percentage of stability reduction was found but if we merge this fiber in to glass fiber, the stability reduction attain the value of thirty seven percentages. Reason for more strength reduction in kenaf fiber composite is bulging of fiber which yield the formation of shear stress in the boundary of the fiber matrix could be the factor for weaken bonding. After dryness, some amount of elongation stability of the composites was retained. This residual strength is more in glass fiber comparatively with both kenaf and kenaf glass fiber composite. In glass fiber returning back of tensile strength is obtained up to the value of 95% which is not in case of kenaf/glass hybrid composite which can obtain only with the value of 87.22%. The reason for this problem is process of becoming older is the nature of kenaf fiber. Plasticizer role is used to regaining the strength of the fiber composite which is used to soft, flexible, increase plasticity, reduce viscosity and friction in processing. This plasticizer role is done by water which could be extracted after dehydration (Prabhu et al. [2020\)](#page-16-17). Figure [7a](#page-12-0), b explains the bending strength and bending modulus of composite samples at the same three various levels. Related nature of lowering in stability and modulus and their revival are also explained (Mishra et al. [2020;](#page-16-16) Gurunathan et al. [2015\)](#page-15-15).

Scanned electron microscopy (SEM) was used to examine the failure state of the broken parts of the composites with the magnification value of \times 350. Figure [8a](#page-12-1) explains the fracture surface of tensile test specimen. In this figure, elongation and empty space of fibers while removing it visualized clearly. Figure [8b](#page-12-1) explains the rupture of vascular fiber bundles and empty spaces because of fiber pull out at fracture surface of flexural test sample (Mishra et al. [2020;](#page-16-16) Alomayri et al. [2014\)](#page-15-16).

Through all discussions and investigations, the following conclusions were derived. Protective layer of all composites for transferring the moisture uses Fickian's

Fig. 7 a Variation of flexural strength w.r.t. composite samples; **b** variation of flexural modulus w.r.t. composite samples (Mishra et al. [2020\)](#page-16-16)

Fig. 8 a Fiber stretching and debonding; **b** fracture surfaces of composite samples (Mishra et al. [2020\)](#page-16-16)

law in which the coefficient value is nearer to 0.5. Maximum value of 7.140×10^8 $\rm cm^2/s$ as a diffusion coefficient is attained by kenaf fiber. Even if that is the case, continuous inclusion of fiber glass decreases the amount of taking up of water. Humiliation of mechanical characteristics is found because of moisture absorption but of merging of glass fiber enhances the reduction as-well-as enhanced the retaining of stability up to 87.22% after the process of drying. Major factors for decrement of stability and tensile constants of fiber composites are based on the pulling out of fiber and de-bonding of fiber–matrix inter-phase (Gokulkumar et al. [2019\)](#page-15-17).

4.4 Pineapple Leaf Fiber Composites

Pineapple leaf fibers (PALF) are being currently used as natural fiber reinforcements widely due to its abundant availability in many regions around the globe. Unique properties including biodegradability, environmental friendly end products, less in cost and weight pose the usage of hybrid fiber reinforced composites to a greater extent. Few experiments were carried out earlier to evaluate the moisture absorption and mechanical characteristics of PALF-G fibers reinforced hybrid composites reinforced in epoxy matrix and the influence of fiber stacking sequence had been analyzed. Tensile, flexural and accelerated moisture absorption tests were conducted for three different stacking sequence such as GPPG, PGPG and PGGP which were used to fabricate the hybrid PALF-G fibers reinforced epoxy composites and the experiments were also carried out in individual PALF and G fiber composites. Fickian diffusion theory was used to evaluate the moisture absorption characteristics of the composites and SEM was used for the characterization of fracture surface morphology of the specimen. Results portrayed that individual PALF composites absorbed larger quantity of moisture than the hybrid composites which in turn degraded the mechanical characteristics of the individual PALF composites while the hybrid composites possessed higher mechanical properties. It was also noticed from the results that the fiber stacking sequence had an appreciable effect over the moisture absorption behaviour of the composites. Mechanical test results indicated that the tensile and flexural behaviour of PALF-G hybrid composites were higher than the individual composites due to the lower moisture absorption of hybrid composites and when compared with unreinforced epoxy composites, the tensile and flexural strength was 153% and 119% higher for the fiber reinforced composites respectively. Amongst the various stacking sequence, PGPG sequence exhibited better mechanical characteristics with their tensile and flexural strength as 120.19 MPa and 169.17 MPa respectively when compared with other sequences. It could be noticed from the SEM images that hybrid composites with PGPG sequence displayed better bonding at the composite interface between matrix and fiber when compared with the other sequences (Ramesh and Rajesh Kumar [2020;](#page-16-18) Mochane et al. [2019;](#page-16-19) Wu [2014\)](#page-17-15).

Moisture absorption behaviour of the PALF-G hybrid composites were evaluated by using the composite specimens of length 50 mm adopting ASTM D5229 standards. A water resistant epoxy coating was applied at each end of the specimen and were sealed with the paint. Specimens were then oven dried at a temperature of 50 ºC for a period of 24 h until all the moisture present in the specimen evaporates and the specimens were atmospheric cooled. Specimens were weighed before and after drying in the oven and the mass values were noted as M_r and M_o respectively. Specimens were then drowned in a bath of water at a temperature of 60 ºC and were measured for their weight at regular interval of time until saturated state was reached (Kharrat et al. [2019\)](#page-15-18). Moisture absorption rate was determined by the following Eq. [1:](#page-13-0)

$$
M_f = \frac{M_t - M_0}{M_0} \times 100
$$
 (1)

where M_t = Mass of the Specimen after time of exposure t, M_0 = Mass of specimen before immersing in water bath. Following equation denoted the Fick's law of diffusion that could be applied to the composite specimen for evaluating its moisture absorption behaviour.

$$
M(T, t) = (M_m - M_i) \left(1 - \exp \left[-7.3 \left(\frac{D_Z \times t}{h^2} \right)^{0.75} \right] \right) + M_i \tag{2}
$$

Experiments conducted in using PALF-G hybrid fibers reinforced in epoxy matrix to determine their moisture absorption behaviour along with the determination of mechanical properties such as tensile and flexural characteristics revealed that the effect of hybridization of fibers had significant effect on the above behaviour and enhanced them. It was also seen that the stacking sequence of the fiber laminates showed greater impact over the composite properties. Fick's law of diffusion could effectively predict the moisture absorption behaviour of the hybrid and individual fiber reinforced composites. The stacking sequence with alternate laminates of PALF and G fibers (PGPG) exhibited lowest moisture absorption which was 10.35% less than the moisture absorption rate of other compositions. Overall from the above discussion it could be seen that the PALF-G hybrid composites with PGPG stacking sequence exhibited better moisture absorption, tensile and flexural properties with better adhesion at the interfacial region in between epoxy matrix and PALF-G hybrid composite reinforcements (Datta et al. [1995;](#page-15-19) Karimzadeh et al. [2020\)](#page-15-20).

5 Conclusion

Broad preamble to actual fiber strengthened composites, the proportion of actual fiber, and modification of surface of the natural fiber and the chemical treatments effect of on natural fiber composites are discussed. Several experiments clearly illustrate the mechanical characteristics of all natural fibers strengthened composites namely; tensile and bending, elongation percentage and finally absorption of water etc. are extensively enhanced. Comparison of several characteristics between surface modified natural fiber and un-treated fiber has been done. Among that, improved tensile and bending strength identified in alkali surface modified natural fiber composites with untreated composites. Sticking bond between natural fiber and matrix got enhanced in case of surface modified fibers and water amalgamation characteristics of natural fiber reinforced composites got decreased. Surface modified fiber composite proven the better tensile properties compared with un-treated fibres. Surface modified fibers has proven enhanced tensile properties comparator combined surface modified fiber which is deriving good property enhancement than the single surface modified method in which alkali method is exceptional. Silane treated surface modified fiber composites possess good improvement in case of impact strength characteristics. Fat, lignin and pectin are the natural fiber impurities can be eliminated easily by using surface modifications of the natural fibers.

References

- Agrawal R, Saxena NS, Sharma KB, Thomas S, Sreekala MS (2000) Activation energy and crystallization kinetics of untreated and treated oil palm fibre reinforced phenol formaldehyde composites. Mater Sci Eng, A 277(1–2):77–82
- Alomayri T, Assaedi H, Shaikh FUA, Low IM (2014) Effect of water absorption on the mechanical properties of cotton fabric-reinforced geopolymer composites. J Asian Ceramic Soc 2(3):223–230
- Alvarez VA, Ruscekaite RA, Vazquez A (2003) Mechanical properties and water absorption behavior of composites made from a biodegradable matrix and alkaline-treated sisal fibers. J Compos Mater 37(17):1575–1588
- Balaji D, Ramesh M, Kannan T, Deepan S, Bhuvaneswari V, Rajeshkumar L (2020) Experimental investigation on mechanical properties of banana/snake grass fiber reinforced hybrid composites. Mater Today Proc. <https://doi.org/10.1016/j.matpr.2020.09.548>
- Vonoth R, Gokulnath KS, Barathkumar KJ, Ahildarshan K, Gokulprakash E (2018) A study of banana fiber: a review. Int J Sci Res Develop 6(09):127–131
- Bhoopathi R, Deepa C, Sasikala G, Ramesh M (2015) Experimental investigation on mechanical properties of hemp-banana-glass fiber reinforced composites. Appl Mech Mater 766:167–172
- Chigondo F, Shoko P, Nyamunda BC, Guyo U, Moyo M (2013) Maize stalk as reinforcement in natural rubber composites. Int J Sci Technol Res 2(6):2277–8616
- Chin-San W (2014) Preparation, characterisation, and biocompatibility of Ganoderma lucidum fibre-based composites with polylactic acid. Compos Sci Technol 103:1–9
- Datta R, Tsai SP, Bonsignore P, Moon SH, Frank JR (1995) Technological and economic potential of poly (lactic acid) and lactic acid derivatives. FEMS Microbiol Rev 16(2–3):221–231
- Faruk O, Sain M (2014) Biofber reinforcement in composites materials.Woodhead Publ Ser 51:454– 487
- Gokulkumar S, Thyla PR, Prabhu L, Sathish S (2019) Measuring methods of acoustic properties [and influence of physical parameters on natural fibers: a review. J Nat Fibers.](https://doi.org/10.1080/15440478.2019.1598913,1-20) https://doi.org/10. 1080/15440478.2019.1598913,1-20
- Goriparthi BK, Suman KNS, Rao NM (2012) Efect of fber surface treatments on mechanical and abrasive wear performance of polylactide/jute composites. Compos Part A Appl Sci Manuf 43:1800–1808
- Gulati D, Sain M (2006) Fungal-modification of natural fibers: a novel method of treating natural fibers for composite reinforcement. J Polym Environ 14(4):347–352
- Gurunathan T, Mohanty S, Nayak SK (2015) A review of the recent developments in biocomposites based on natural fbres and their application perspectives. Compos Part A Appl Sci Manuf 77:1–25
- Indraja Y, Kumar GS, Rao HR (2014) Development and study of properties of natural fiber reinforced polyester composites. Int J Eng Sci Res Technol 410–414
- Jayaramudu J, Reddy KO, Maheswari CU, Reddy DJP, Rajulu AV (2009) Tensile properties and thermal degradation parameters of Polyalthia cerasoides natural fabric reinforcement. J Reinf Plast Compos 28(18):2177–2181
- Karimzadeh A, Yahya MY, Abdullah MN, Wong KJ (2020) Effect of stacking sequence on mechanical properties and moisture absorption characteristic of hybrid PALF/glass fiber composites. Fibers Polym 21(7):1583–1593
- Karthi N, Kumaresan K, Sathish S, Gokulkumar S, Prabhu L, Vigneshkumar N (2020) An overview: Natural fiber reinforced hybrid composites, chemical treatments and application areas. Mater Today Proc 27(3):2828–2834
- Kharrat F, Chaari R, Khlif M, Hilliou L, Covas JA, Haboussi M, Nouri H, Bradai C (2019) The effect of the process on mechanical properties of polylactic acid-date palm leaf fibers composite films produced by extrusion blowing. J Renew Mater 7(9):891–901
- Kulkarni AG, Satyanarayana KG, Rohatgi PK (1981) Electrical resistivity and dielectric strength of plant fibres. J Mater Sci 16(6):1719–1726
- Kushwaha PK, Kumar R (2010) Effect of silanes on mechanical properties of bamboo fiber-epoxy composites. J Reinf Plast Compos 29(5):718–724. <https://doi.org/10.1177/0731684408100691>
- Li X, Tabil LG, Panigrahi S (2007) Chemical treatments of natural fiber for use in natural fiberreinforced composites: a review. J Polym Environ 15(1):25–33
- Mishra C, Deo CR, Baskey S (2020) Influence of moisture absorption on mechanical properties of [kenaf/glass reinforced polyester hybrid composite. Mater Today Proc.](https://doi.org/10.1016/j.matpr.2020.08.100) https://doi.org/10.1016/j. matpr.2020.08.100
- Mochane MJ, Mokhena TC, Sadiku ER, Ray SS, Mofokeng TG (2019) Green polymer composites based on polylactic acid (PLA) and fibers. In: Green biopolymers and their nanocomposites. Springer, Singapore, pp 29–54
- Mohanty AK, Misra M, Drzal LT (2005) Natural fibers, biopolymers, and biocomposites. Taylor & Francis Group, Boca Raton, Florida
- Orue A, Jauregi A, Peña-Rodriguez C, Labidi J, Eceiza A, Arbelaiz A (2015) The effect of surface modifications on sisal fiber properties and sisal/poly (lactic acid) interface adhesion. Compos Part B Eng 73:132–138
- Petinakis E, Yu L, Simon G et al. (2013) Natural fiber bio-composites incorporating poly (lactic acid). In: Masuelli M (ed) Fiber reinforced polymers—the technology applied for concrete repair. InTech, pp 41–59. <https://doi.org/10.5772/52253>
- Phuong NT, Sollogoub C, Guinault A (2010) Relationship between fiber chemical treatment and [properties of recycled pp/BFCs. J Reinf Plast Compos 3244–3256.](https://doi.org/10.1177/0731684410370905) https://doi.org/10.1177/073 1684410370905
- Prabhu L, Krishnaraj V, Gokulkumar S, Sathish S, Sanjay MR, Siengchin S (2020) Mechanical, chemical and sound absorption properties of glass/kenaf/waste tea leaf fiber-reinforced hybrid epoxy composites. J Ind Text. <https://doi.org/10.1177/1528083720957392>
- Ramesh M (2016) Kenaf (*Hibiscus cannabinus* L.) fibre based bio-materials: a review on processing and properties. Prog Mater Sci 78:1–92
- Ramesh M (2018) Flax (*Linum usitatissimum* L.) fibre reinforced polymer composite materials: a review on preparation, properties and prospects. Prog Mater Sci 102:109–166
- Ramesh M, Palanikumar K, Reddy KH (2016) Influence of fiber orientation and fiber content on [properties of sisal-jute-glass fiber-reinforced polyester composites. J Appl Polym Sci.](https://doi.org/10.1002/APP.42968) https://doi. org/10.1002/APP.42968
- Ramesh M, Palanikumar K, Reddy KH (2017a) Plant fibre based bio-composites: sustainable and renewable green materials. Renew Sustain Energy Rev 79:558–584
- Ramesh M, Deepa C, Aswin US, Eashwar H, Mahadevan B, Murugan D (2017b) Effect of alkalization on mechanical and moisture absorption properties of Azadirachta indica (Neem tree) fiber reinforced green composites. Trans Indian Inst Met 70(1):187–199
- Ramesh M, Rajesh Kumar L (2020) Bioadhesives. In: Green adhesives preparation, properties and applications. Wiley, pp 145–164
- Ramesh M, Rajeshkumar L (2018) Wood flour filled thermoset composites. Thermoset Compos Prep Prop Appl Mater Res Forum 38:33–65. <https://doi.org/10.21741/9781945291876-2>
- Ramesh M, Deepa C, Rajesh Kumar L, Sanjay MR, Siengchin S (2020a) Life-cycle and environmental impact assessments on processing of plant fibres and its bio-composites: a critical review. J Indus Textiles 1528083720924730. <https://doi.org/10.1177/1528083720924730>
- Ramesh M, Rajesh Kumar L, Khan A, Asiri AM (2020b) Self-healing polymer composites and its chemistry. In: Self-healing composite materials. Woodhead Publishing, pp 415–427
- Salem TF, Tirkes S, Akar AO, Tayfun U (2020) Enhancement of mechanical, thermal and water uptake performance of TPU/jute fiber green composites via chemical treatments on fiber surface. E-Polymers 20(1):133–143
- Saravana Kumar A,Maivizhi Selvi S, Rajeshkumar L (2017) Delamination in drilling of sisal/banana reinforced composites produced by hand lay-up process. Appl Mech Mater 867:29–33
- Sathish S, Kumaresan K, Prabhu L, Vigneshkumar N (2017) Experimental investigation on volume fraction of mechanical and physical properties of flax and bamboo fibers reinforced hybrid epoxy composites. Polym Polym Compos 25:229–236
- Sathish S, Kumaresan K, Prabhu L, Gokulkumar S (2018) Experimental investigation on mechanical and FTIR analysis of flax fiber/epoxy composites incorporating SiC, A_1O_3 and graphite. Roman J Mater 48:476–482
- Sathish S, Kumaresan K, Prabhu L, Gokulkumar S (2019) Experimental investigation of mechanical and morphological properties of flax fiber reinforced epoxy composites incorporating SiC and Al2O3. Mater Today Proc. <https://doi.org/10.1016/j.matpr.2019.09.106,1-5>
- Sethy SR (2010) A study on mechanical behavior of surface modified natural fiber based polymer composites. B. Tech Thesis. Department of Mechanical Engineering, National Institute of Technology, Rourkela, India, 2011
- Sigha AS, Rana A (2013) Effect of Aminopropyltriethoxysilane (APS) treatment on properties of mercerized lignocellulosic grewia optiva fiber. J Polym Environ 21:141–150
- Singh TJ, Samanta S (2014) Characterization of natural fiber reinforced composites-bamboo and sisal: a review. IJRET: Int J Res Eng Technol. eISSN: 2319-1163|PISSN: 2321–7308
- Singha AS, Rana AK (2012) Ce (IV) ion–initiated and microwave radiation–induced graft copolymerization of acrylic acid onto lignocellulosic fibers. Int J Polym Anal Charact 17(1):72–84
- Singha AS, Rana AK (2013) Effect of Aminopropyltriethoxysilane (APS) treatment on properties of mercerized lignocellulosic grewia optiva fiber. J Polym Environ 21(1):141–150
- Singha AS, Rana AK, Jarial RK (2013) Mechanical, dielectric and thermal properties of Grewia optiva fibers reinforced unsaturated polyester matrix-based composites. Mater Des 51:924–934
- Sorrentino A, Gorrasi G, Vittoria V (2007) Potential perspectives of bio-nanocomposites for food packaging applications. Trends Food Sci Technol 18:84–95
- Sujaritjun W, Uawongsuwan P, Pivsa-Art W, Hamada H (2013) Mechanical property of surface modifed natural fber reinforced PLA biocomposites. Energy Proc 34:664–672
- Suprakas SR, Mosto B (2005) Biodegradable polymers and their layered silicate nanocomposites: in greening the 21st century materials world. Prog Mater Sci 50:962–1079
- Swain PTR, Das SN, Jena SP (2018) Manufacturing and study of thermo-mechanical behaviour of surface modified date palm leaf/glass fiber reinforced hybrid composite. Mater Today: Proc 5(9):18332–18341
- Vimal R, Subramanian KHH, Aswin C, Logeswaran V, Ramesh M (2015) Comparisonal study of succinylation and phthalicylation of jute fibres: study of mechanical properties of modified fibre reinforced epoxy composites. Mater Today: Proc 2:2918–2927
- Wu CS (2014) Preparation, characterisation, and biocompatibility of Ganoderma lucidum fibrebased composites with polylactic acid. Compos Sci Technol 102:1–9
- Yan L, Chouw N, Yuan X (2012) Improving the mechanical properties of natural fiber fabric [reinforced epoxy composites by alkali treatment. J Reinf Plast Compos 425–437.](https://doi.org/10.1177/0731684412439494) https://doi.org/ 10.1177/0731684412439494
- Zhao X, Li K, Wang Y, Tekinalp H, Larsen G, Rasmussen D, Ginder RS, Wang L, Gardner DJ, Tajvidi M, Webb E (2020) High-strength polylactic acid (PLA) biocomposites reinforced by epoxy-modified pine fibers. ACS Sustain Chem Eng 8(35):13236–13247