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# Evaluation of mechanical properties of both benzoyl peroxide treated and untreated teak sawdust reinforced high density polyethylene composites

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Abstract Natural fiber reinforced polymer composites are extensively used in various applications such as automotive interiors, furniture, packaging and construction materials. In the present work, teak or shegun (Tectona grandis) sawdust were used as reinforcements in thermoplastics. Sawdust fibers were chemically treated to reduce their moisture absorption. Sawdust pretreated with alkali was then treated with benzoyl peroxide to provide a more hydrophobic surface that would adsorb less water and have better interfacial adhesion. Reaction parameters such as time, temperature, pH and concentration of benzoyl peroxide were used to get the desired change in functional group of cellulose. FT-IR spectroscopic analyses were done and the results showed the evidence of positive reactions. In this work, chemically treated and untreated teak sawdust reinforced high density polyethylene (HDPE) composites were prepared by compression moulding technique. The effects of this chemical treatment on the physicomechanical properties of the composites were studied. The effect of fiber content on the physico-mechanical

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properties of composites were also studied by preparing the composites with different weight fractions (5, 15, 25, 35 and 45%) of sawdust. Water absorption  $(\%)$ tests for all composites revealed that there was less water absorption for the treated composites than the untreated ones. Scanning electron microscopy (SEM) images of treated and untreated sawdust-HDPE composites showed that better interfacial adhesion has occurred between the fiber and the matrix in benzoyl peroxide treated sawdust-HDPE composites. Mechanical properties (tensile strength, elongation at break and impact strength) were determined and analyzed for all treated and untreated sawdust-HDPE composites. Improved tensile and impact results of treated sawdust-HDPE composites indicated that benzoyl peroxide treatment of sawdust increases the interfacial adhesion between fiber-matrix in the treated sawdust-HDPE composites.

Keywords Saw dust · HDPE · Composite · Tensile strength - Impact strength - Water absorption

# Introduction

The use of natural fibers as a reinforcing material instead of man-made fibers to develop composite materials has attracted significant interest in the last few decades due to their low densities, low cost, nontoxic, non-abrasive and biodegradable properties

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in various applications. Natural fiber reinforced composites can be applied in the plastics, automobile, construction and packaging industries to cut down on material cost. Automotive components, such as dashboards, door panels, seats, and cabin linings were made from natural fiber reinforced polymer composites because of their superior strength/weight and stiffness/weight ratios. Manmade fibers such as glass, graphite (carbon), boron, organic, metallic, and ceramic are heavy, expensive and harmful to the environment. Lignocellulosic fibers offer many advantages over these most commonly used manmade fibers as filler reinforcement in thermoplastic matrix. From the all lignocellulosic fibers, wood fiber is the most broadly used as composite materials reinforcement (Sesan et al. [2016](#page-12-0); Gopinath and Vadivu [2014](#page-12-0); Zivkovic et al. [2011\)](#page-13-0). Wood sawdust is a solid waste material produced from the processing of wood in the timber industries. Sawdust fiber reinforced composites can also be used for furniture, panels, boards, flooring, decking, automotive applications etc. Wood fiber reinforced wood plastic composites (WPCs) combine the favorable performance and cost effective properties of both wood and plastics. Because of these attributes, WPCs are seen as a way to increase the value-added utilization of waste wood and wood of low commercial value (Araga et al. [2011](#page-12-0)). WPC products with improved properties can be obtained if the wood fibers are modified by suitable additives, properly blended with the polymers and carefully processed with suitable molding techniques (Brostow et al. [2010\)](#page-12-0). The properties of the WPCs were also dependent on the wood species and plastic/wood ratios. Selections of wood species are depending on regional availability and cost. The commonly used wood species for commercial WPCs production are pine, maple and oak (Sesan et al. [2016](#page-12-0)). A large amount of wood waste in different form such as wood pulp, fibers or flour has been generated in wood industry. These wood waste forms are very suitable as reinforcement for thermoplastic polymer matrices (Zivkovic et al. [2011](#page-13-0)).

In this research Tectona grandis sawdust were used as reinforcing materials with HDPE matrices to prepare composites by compression moulding technique. Tectona grandis is known as ''Teak or Shegun'' wood. Teak is a fast growing hardwood tree available in Bangladesh. It is native to the South and Southeast Asian countries region but cultivated in many other countries. So, one of the aims of this research is to find out the utilization of teak sawdust sourced from Bangladesh as a raw material for the production of composites.

Brostow et al. [\(2010](#page-12-0)) stated that wood–polymer composites generally exhibit low moisture absorption and high resistance to decay, insect, and UV ray damage. They also stated that the wood surface can be modified to tailor the properties of the materials to greater sustainability for special applications in such areas as building, construction and automotive engineering. Moisture absorbing tendency of natural fibers creates a major problem in natural fiber reinforced composites. Poor resistance against fungal and insect attack, swelling and shrinkage resulting from water absorption and desorption are some of these shortcomings (Ehrenstein and Kabelka [1992](#page-12-0); Araga et al. [2011\)](#page-12-0). This hydrophilic behavior of natural fibers is main demerits of natural fiber reinforced composites which affects on their performance of dimensional stability and durability. Chemical treatment of the natural fibers can overcome these limitations by cleaning the fiber surface, chemically modify the surface, stop the moisture absorption process and increase the surface roughness (Gupta and Kumar [2012\)](#page-12-0). A lot of literature focuses on these limitations through chemical modification techniques of natural fibers and natural fiber reinforced composites (Madhuri et al. [2016](#page-12-0); Macaulay [2014;](#page-12-0) Kord [2011](#page-12-0); Ghani et al. [2016](#page-12-0)). Kallakas et al. [\(2016](#page-12-0)) reported that reduced hydrophilicity and polarity property of woodplastic composites were found by heat treatment of wood fibers. They also investigated and reported that chemical modification of wood fiber with NaOH and 3-aminopropyltriethoxysilane (APTES) increases the mechanical properties of the composites. Thiruchitrambalam et al. ([2009\)](#page-12-0) investigated and reported that sodium lauryl sulphate (SLS) treatment of fibers had provided better mechanical properties of banana/ kenaf hybrid composites. Rowell ([2012\)](#page-12-0) studied on dimensional stability of wood and wood composite and reported that it can be achieved by several methods, including cell wall bulking, cell wall polymer crosslinking and removal of hygroscopic components in the cell wall. Sultana et al. [\(2007](#page-12-0)) reported on oxidation of cellulose in jute to dialdehyde cellulose in jute with sodium periodate and this chemical treatment of jute fibers provides better compatibility between fiber and matrix in oxidized jute-PP composites. To the best of our knowledge, no research has been done on modification of cellulose in sawdust to dialdehyde cellulose in sawdust by chemical treatment.

Effect of mercerization and benzoyl peroxide treatment on morphology, thermal stability and crystallinity of sisal fibers have been studied and reported (Kaushik et al. [2012\)](#page-12-0). They reported on the enhanced thermal stability and crystallinity results of sisal fibers treated with benzoyl peroxide. In this work mercerization and benzoyl peroxide treatment have been carried out with teak sawdust. Major constituents of sawdust are cellulose, hemicellulose, and lignin that are rich in functional groups like hydroxyls. The main aim of this work is to modify cellulose in teak sawdust to dialdehyde cellulose in teak sawdust by benzoyl peroxide treatment. The effects of benzoyl peroxide treatment and sawdust content on the physico-mechanical properties of teak sawdust-HDPE composites have been investigated, and are reported in this paper.

## Materials and methods

### Materials

In this work, high density polyethylene (HDPE) was used as a matrix material to prepare composites. It was manufactured by The Polyolefin Company Pvt. Ltd., Singapore and its commercial name is alkathene or polythene. Melting point of this HDPE was measured and found to be  $130^{\circ}$ C. The sawdust was collected from local timber house (sawmill) named Robel Timber and Sawmill Limited, Sonir Akra, Dhaka, Bangladesh. The supplied sawdust was from Teak (Tectona grandis) wood. Sodium hydroxide (NaOH), sulphuric acid (H<sub>2</sub>SO<sub>4</sub>), benzoyl peroxide  $(C_6H_5CO)_{2-}$  $O<sub>2</sub>$  chemicals from Merck, Germany were used in this work.

#### Processing of sawdust

Sawdust was processed by using several physical methods like shaking, sieving, meshing and washing. Initially the sawdust sample was shaken and clean to remove all undesirable particles to get 100% sawdust. The sawdust was a mixture of many particle sizes. In this experiment the sawdust was sieved with specific meshes are mesh  $70+$ , mesh  $100+$ , mesh  $140+$  and mesh  $120+$  respectively. Sawdust of these four particle size's were taken and mixed thoroughly to make it homogenous mixture using a mixture machine. The sawdust was then washed with distilled water and dried at open atmosphere.

Surface modification of sawdust by chemical treatment

The chemical modification of sawdust was conducted with 5% NaOH solution first. Sodium hydroxide solution (5%) was prepared in distilled water. Dried sawdust was immersed in 5% sodium hydroxide solution for one hour at room temperature  $25^{\circ}$ C. Sawdust to liquor ratio was 1:20 (w/v). At the end of this treatment, the color of sawdust transferred from golden to blood red. The alkali treated sawdust was thoroughly washed in tap water and finally washed with distilled water. The pH of the washed sawdust was found neutral by washing again and again with water. Then the alkali treated sawdust was air-dried first and then dried in oven at 105  $\degree$ C for 6 h to get constant weight. Alkali treated dried saw dust was taken to conduct modification reaction with benzoyl peroxide. A number of reactions were carried out for this chemical treatment with variation of reaction parameters such as time, temperature, pH and concentration of benzoyl peroxide to get the positive result following FT-IR spectroscopic analyses. In benzoyl peroxide treatment, the tensile strength values of composites increased with the increase in concentration of peroxide up to a certain level which was 6% and then remained constant. The reaction parameters were selected and then the reaction was carried out with 6% benzoyl peroxide in acetone solution for 30 min. Then the fiber was thoroughly washed in tap water and finally washed with distilled water and dried in air. Air dried benzoyl peroxide treated and untreated sawdust was placed in an oven for drying at 80  $\degree$ C for 24 h and used for fabrication of composites specimen with HDPE.

FT-IR spectroscopic characterization of untreated and chemically treated sawdust

The infrared spectra of untreated and benzoyl peroxide treated sawdust were recorded on a FT-IR/NIR Spectrometer (Forntier, PerkinElmer, USA). The sample pellets for FT-IR spectroscopy were prepared as follows.

Approximately 0.5 mg of powdered sawdust samples were mixed thoroughly with approximately 100 mg of dried powdered potassium bromide in a small agate mortar pestle. The mixture was taken in a die of specific dimensions. Pellets were made by applying vacuum pressure. IR spectra with all information about absorbance were obtained in the printed form.

## Composite fabrication

Sawdust-HDPE composites were prepared using dried untreated and treated sawdust following the procedure described below.

HDPE polymers were dried in oven for 3 h at 105  $\degree$ C and grinded by a grinding machine. Sawdust was mixed with HDPE in a blender at 400 rpm for 1 min. HDPE matrix and sawdust were taken in different weight fractions (Table 1).

Preparation of composites by compression moulding technique

The dried granulated HDPE and sawdust were mixed and moulded by a compression moulding machine (Paul-Otto Weber Press Machine) at a molding temperature of  $145 \text{ °C}$ . The pressure, temperature, heating time and cooling time of the samples were controlled at the same rate to prepare all composites. The applied pressure was 200–250 kN and heating time was 10 min. After completion of heating the additional pressure of 50 kN was applied to get voids free compression moulded specimen. The specimen was allowed to cool and cure by tap water through the outer area of the heating plates of the hydraulic press machine. Completely cured composite was taken out from the mould and was cut to make specimens of suitable dimension for different tests.

Water absorption test of composites

In order to measure the water absorption of the composites, treated and untreated sawdust reinforced HDPE composites at different weight fractions  $(5-50 \text{ wt\%})$  were prepared and specimens were cut from each composite having dimensions of 39 mm  $\times$  10 mm  $\times$  4 mm. The Testing was conducted in accordance with ASTM D570-99 standard method. Water absorption tests were carried out by immersing specimens in distilled water bath at different conditions according to ASTM D570-99 standard method. Three replicate specimens were tested and the results were presented as average of the tested specimens in Figs. [7](#page-9-0) and [8](#page-10-0) respectively.

# Mechanical properties of the composites

In order to investigate the mechanical properties of the prepared composites the following tests were carried out; (a) tensile (b) Izod impact. For these tests the appropriate ASTM methods were followed.

# Tensile test

An universal tensile testing machine, model: 1410 Titans, capacity: 5 kN, England was used to measure the tensile properties of both the treated and untreated sawdust reinforced HDPE composites at a cross head speed of 10 mm/min. Tensile tests were conducted following ASTM D 3039/D 3039 M-00 ([2002\)](#page-12-0) method and each test was performed until tensile failure occurred except 100% HDPE composite. Six to ten specimens of each composition were tested and the average values were reported by calculating maximum five values.



composites

#### Izod impact test

Impact resistance of any materials can be determined by impact test. In this work, dynamic izod impact tests of the composites were done according to ASTM D 256 by a Universal Charpy/lzod Analogue Impact Tester, Model: QPI-IC-21 J, Qualitest, North America. This machine was used to perform izod impact tests of all the samples. Weight of the hammer of this machine was 4.028 kg, Impact length 327 mm, Mass center distance  $0.302$  m and initial angle  $150^\circ$ . Free rotational angle of the hammer was 147°. The width, thickness and length of the specimen are 17, 2.02 and 80 mm respectively. Notching of the test specimens was done on a milling machine. In this experiment, as the width and thickness are 17 and 2.02 mm so notch cut were made on the 2.02 mm shorter side. So the width of this test specimen will be 2.02 mm and depth will be 17 mm. To conduct this experiment, according to test method, the specimen was supported on a vertical simple beam and was broken by a single swing of the pendulum with the impact line midway between the supports and directly opposite to the notch. Five samples were tested and the average values were reported for each different composite.

Scanning electron microscopy (SEM) images of composites

The fracture surfaces of tensile samples of benzoyl peroxide treated and untreated 30 wt% sawdust-HDPE composites were used to examine the fibermatrix interfacial bonding in the composites. SEM images were taken by scanning electron microscope, model: S-3400N, Hitachi, Japan. The images are presented in the ''Results and discussion'' section.

## Results and discussion

## FT-IR spectroscopic analysis of untreated sawdust

The FT-IR spectrum of untreated sawdust is shown in Fig. [1.](#page-5-0) The peak assignments of the absorption bands corresponding to various groups are summarized in Table [2.](#page-5-0) The FT-IR spectrum of untreated sawdust shows a band in the region near  $1737.05 \text{ cm}^{-1}$ , which is probably due to the CO group of acetyl ester in hemicellulose or aldehyde group in lignin. On the other hand, absorption bands of untreated sawdust give clear peak of cellulose, lignin, and hemicelluloses.

FT-IR spectroscopic analysis of alkali treated sawdust

Sodium hydroxide treatment was done to remove the lignin from cellulose of raw sawdust. In this chemical treatment cellulose is converted to mercerized cellulose. The FT-IR spectrum of alkali treated sawdust is shown in Fig. [2](#page-6-0) and the absorption bands are summarized in Table [3](#page-6-0).

Modification of alkali treated sawdust by benzoyl peroxide and FT-IR spectroscopic characterization

Alkali treated teak sawdust fibers were taken to modify its surface by benzoyl peroxide treatment to make it more active and hydrophobic. Reaction of cellulose in alkali treated sawdust with benzoyl peroxide may be resulted to breaks on the anhydroglucose ring between carbon 2 and carbon 3. This may be converts the two secondary hydroxyl groups into aldehyde groups (Sultana et al. [2007\)](#page-12-0). FT-IR spectral data showed the characteristic bands of aldehyde group at the region of 2928.1 and 2856.9  $cm^{-1}$  due to CH stretching and a small peak at the region of near  $1700 \text{ cm}^{-1}$  due to carbonyl stretching. The spectrum shows the evidence of cellulose in teak sawdust have converted to dialdehyde cellulose in teak sawdust. Sultana et al. [\(2007](#page-12-0)) reported on the similar observation of dialdehyde cellulose in jute fibers with sodium periodate treatment. The peak assignments of the benzoyl peroxide modified teak sawdust fiber are summarized in Table [4](#page-6-0) and the spectrum is shown in Fig. [3](#page-7-0) respectively.

Mechanical properties of untreated and treated sawdust reinforced high density polyethylene (HDPE) composites

Tensile strength, elongation at break, impact strength of the untreated and alkali treated and benzoyl peroxide modified sawdust reinforced high density polyethylene composites have been determined following standard ASTM methods. The results obtained in this study are presented below.

<span id="page-5-0"></span>

Fig. 1 FT-IR spectrum of untreated teak sawdust





# Tensile strength and elongation at break

Tensile strength of 100% HDPE composite is 20.04 MPa and elongation at break is 9.52% which is higher than that of treated and untreated sawdust reinforced HDPE composites. The tensile strengths of the untreated sawdust-HDPE composites increase up

to 15% sawdust loading and then decrease (Fig. [4](#page-7-0)). The elongation at break decreases with increasing sawdust loading (Fig. [5\)](#page-8-0). From the Fig. [4](#page-7-0) it is also observed that the tensile strengths of the benzoyl peroxide treated sawdust reinforced HDPE composites are higher than that of untreated sawdust-HDPE composites. It is also found that the elongation at break

<span id="page-6-0"></span>

Fig. 2 FT-IR spectrum of alkali treated teak sawdust







<span id="page-7-0"></span>

Fig. 3 FT-IR spectrum of alkali treated and benzoyl peroxide modified teak sawdust



Fig. 4 Tensile strength (Mpa) versus sawdust loading (wt %) curves for untreated and benzoyl peroxide treated sawdust-HDPE composites

of treated sawdust-HDPE composites is slightly higher than that of untreated sawdust-HDPE composites. Elongation is inversely proportional to hardness, tensile strength, and modulus of a material. Brostow and Lobland [\(2016](#page-12-0)) stated that elongation and impact strength are inversely proportional to brittleness. They also stated that brittleness is an important property of polymers and polymer-based materials for determining their potential uses. Increasing of the elongation at break of the composites increases the toughness or <span id="page-8-0"></span>Fig. 5 Elogation at break (%) versus sawdust loading (%) curves for untreated and benzoyl peroxide treated sawdust-HDPE composites



ductility of the composites. Accordingly, increase in elongation of the composites shows the decrease in brittleness of those composites.

#### Izod impact strength

Impact strength of 100% HDPE composite is 42 J/m which is lower than that of some treated and untreated sawdust reinforced HDPE composites. The Izod impact strength of the raw and benzoyl peroxide treated composites are shown in Fig. [6.](#page-9-0) It is apparent from the result presented in the figure that the values of impact strength of sawdust-HDPE composites are significantly more than that of unfilled HDPE polymer. Impact strength increases with increasing fiber loading up to 25% and then decreases. Benzoyl peroxide treated composites show better result than untreated sawdust-HDPE composites. Brostow and Lobland [\(2010](#page-12-0)) stated that the lower the brittleness, the higher the dimensional stability of the material and the impact strength of a material is inversely proportional to its brittleness. Hardness and impact strength of composite materials are inversely proportional (Aruniit et al. [2014](#page-12-0)). Benzoyl peroxide treatment of sawdust increases the fracture resistance of treated sawdust-HDPE composites and consequently, reduces their brittleness.

Effect of surface modification of fibers on the mechanical properties of treated sawdust reinforced HDPE composites

All of the results of mechanical properties are significantly more for benzoyl peroxide treated sawdust-HDPE composites than untreated sawdust-HDPE composites. The reasons of these enhancement results may be explained on the basis of hydrophilic nature of sawdust and hydrophobic nature of HDPE matrix. There is very poor interfacial adhesion between hydrophilic sawdust fiber and hydrophobic HDPE matrix. Treatment of sawdust fibers decreases its hydrophilic nature as compared to raw sawdust fibers. This hydrophobic property of treated sawdust increases its interfacial adhesion to HDPE matrix. The improved interfacial bonding between chemically treated sawdust and HDPE matrix in the treated sawdust-HDPE composites increases their mechanical properties as compared to untreated sawdust-HDPE composites.

#### Water absorption

Treated and untreated teak sawdust reinforced high density polyethylene composites at different weight fractions  $(5-50 \text{ wt\%})$  were prepared in order to study the effect of water absorption. The effect of water <span id="page-9-0"></span>Fig. 6 Impact strength (MPa) versus sawdust loading (wt %) curves for untreated and benzoyl peroxide treated sawdust-HDPE composites



absorption on the treated and untreated sawdust loading on the sawdust–HDPE composites have been measured and shown in Figs. 7 and [8.](#page-10-0) It is found from the figures that water absorption increases with increasing fiber loading of the composites. It is also found from the figures that water absorption of untreated sawdust-HDPE composites is higher than that of treated sawdust-HDPE composites. It may be due to presence of lignin and cellulose in untreated sawdust which has hydroxyl groups that can form hydrogen bonds with water molecules. It is also found from the figures that the most significant decrease in hydrophilicity was found for alkali treated benzoyl peroxide modified sawdust-HDPE composites than

Fig. 7 Cold water absorption (%) versus sawdust loading (%) curves for untreated and benzoyl peroxide treated sawdust-HDPE composites



<span id="page-10-0"></span>

Fig. 8 Hot water absorption (%) versus sawdust loading (%) curves for untreated and benzoyl peroxide treated sawdust-HDPE composites

**Sawdust loading (%)**

untreated sawdust-HDPE composites. This may be due to the chemical modification of cellulose in sawdust which converts the more hydrophilic two secondary hydroxyl groups into less hydrophilic aldehyde groups. Lesser water absorption of treated sawdust-HDPE composites increases its mechanical properties and dimension stability. Effect of water absorption on the mechanical properties of natural fiber reinforced plastics composites was investigated by Huner [2015](#page-12-0) and Venkatesh et al. [2016](#page-13-0) and reported that a suitable chemical treatment can reduce the water absorption and increase the mechanical properties and stability of the natural fiber reinforced composites.

## SEM analyses of fracture surfaces

To improve the compatibility between the sawdust fibers and HDPE matrices in composites, sawdust fibers were treated with benzoyl peroxide. The SEM images for untreated 30 wt% sawdust-HDPE composites and benzoyl peroxide treated 30 wt% sawdust-HDPE composites are shown in Figs. [9](#page-11-0) and [10](#page-11-0) respectively. It is clear from the SEM analyses that treated sawdust increased the fiber-matrix interfacial adhesion in composites so stronger interface has been found in SEM image of treated sawdust-HDPE composites. In case of untreated sawdust-HDPE composites, fiber pull-out is more visible in fracture surface than the fracture surface of treated sawdust-HDPE composites. The SEM analyses of composites reveal the evidence of strong interfacial adhesion and compatibility between the treated sawdust and HDPE matrices in treated sawdust-HDPE composites.

#### Conclusion

Benzoyl peroxide treatment of teak (Tectona grandis) sawdust was carried out to improve the adhesion between the hydrophilic sawdust and the hydrophobic HDPE matrices in the sawdust-HDPE composites. Sawdust was pretreated with alkali and then alkali treated sawdust was further treated with benzoyl peroxide. FT-IR spectra confirmed the chemical modification of cellulose in sawdust to dialdehyde cellulose in sawdust by reacting with benzoyl peroxide. The polarity of the aldehyde group is less than that of hydroxyl group. So the hydrophilic tendency of the

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Fig. 9 SEM image of untreated 30 wt% sawdust-HDPE composite



Fig. 10 SEM image of benzoyl peroxide treated 30 wt% sawdust-HDPE composite

treated sawdust fibers was reduced and thus the water absorption of treated sawdust-reinforced composites decreased.

Most of the mechanical properties of the benzoyl peroxide treated sawdust-HDPE composites are higher than those of untreated sawdust-HDPE <span id="page-12-0"></span>composites. Tensile strength, Elongation at break and Impact strength of treated sawdust-HDPE composites show significantly better result than raw sawdust-HDPE composites. So the benzoyl peroxide treatments of sawdust improved the resistance against brittle deformation and fracture of the treated sawdust-HDPE composites than the untreated sawdust-HDPE composites.

Water absorption properties of the composites also show better result for treated sawdust-HDPE composites than raw sawdust-HDPE composites, which will improve the dimensional stability of the composites. Treated sawdust improves the interfacial adhesion between sawdust and HDPE matrix and dispersed uniformly in the composites. The SEM investigations of the composites also confirmed the improvement of interfacial adhesion between the sawdust and HDPE in benzoyl peroxide treated sawdust-HDPE composites. From these observations, it can be concluded that modification of alkali pretreated teak sawdust with benzoyl peroxide enhances the physico-mechanical properties of the teak sawdust reinforced HDPE composites.

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