CELLULOSE CHEMISTRY AND TECHNOLOGY

EFFECT OF ALKALINE TREATMENT ON SAWDUST AS A REINFORCING AGENT FOR POLYMER COMPOSITES

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Material scientists are interested in plant fibers due to their availability, affordability, biodegradability, and strong mechanical properties. They are considered as sustainable alternatives to synthetic fibers in polymer composites; however, their poor adherence to the polymer matrix presents a significant obstacle in this area. This study aims to enhance the hydrophobicity of sawdust by treatment with varying concentrations of NaOH solution (2, 4, 6 and 8 wt%) at 40 °C for 5 hours, which is expected to improve its compatibility with the polymeric matrix. FTIR, XRD, TGA, DTG and SEM characterization techniques were performed, and water absorption and tensile tests were conducted to evaluate the effectiveness of this treatment.

Keywords: sawdust, composite, alkali treatment, adhesion, tensile strength, hydrophilic

INTRODUCTION

Glass and carbon fibers are essential for reinforcing composite materials in the construction, automotive, and aerospace sectors. However, the widespread use of synthetic fibers raises concerns regarding resource and energy consumption, as well as environmental impact.[1] Natural fibers serve as eco-friendly, cost-effective alternatives to synthetic fibers in polymer composites, providing biodegradability and environmental benefits like reduced energy use and renewability. However, they face challenges such as poor bonding with hydrophobic polymers because of interfacial incompatibility resulting from their hydrophilic nature.[2] Alkali, silane, acetylation, permanganate, peroxide, benzoylation, acrylation, acrylonitrile grafting and isocyanate treatments, as well as the addition of maleated coupling agents, are examples of chemical modification techniques used for surface modification of natural plant fibers to improve their various properties. These treatments decrease the fiber's moisture content, eliminate undesirable particles from its surface, and improve the interfacial adherence between the matrix and the fibers.[3]

Numerous researchers have expressed interest in the use of natural fibers to reinforce polymer composites. They achieved promising mechanical property results, supporting the trend of using plant fibers in place of synthetic ones. Laib *et al.*[4] combined Luffa fibers with an unsaturated polyester matrix to create a composite material. Luffa fibers were treated with NaOH, silane, dichromate, permanganate, and by bleaching to improve the adherence at the fiber-matrix interface. The results demonstrated that the composites reinforced with bleached Luffa fibers recorded higher flexural strength by approximately 23.8%, which was the largest increase. Rauf *et al.*[5] developed a composite material using flax fibers reinforced with epoxy. To improve fiber-matrix bonding, they treated the fibers with varying concentrations of silane and fluorocarbons. The optimal results were achieved with 60 g/L silane (20.16% tensile strength increase) and 120 g/L fluorocarbons (34.8% strength increase) compared to untreated fibers. Melouki *et al.*[6] created composites using treated and untreated Alfa fibers to reinforce an unsaturated polyester resin matrix. A 3% NaOH solution was used to treat the fibers at different times (1, 3, 5, and 24 hours). As per the findings, the composite reinforced with 5-hour-treated Alfa fibers exhibited the highest tensile strength value (15.73 MPa), surpassing the composite reinforced with untreated fibers by 11%. Supriya *et al.*[7] studied the impact of potassium permanganate ($KMnO_4$), sodium hydroxide (NaOH), and acetic acid (CH_3COOH) treatments on *Hibiscus rosa-sinensis* fibers. $KMnO_4$ treatment yielded the highest crystallinity (65.77%), best thermal stability (365.24 °C), and lowest material loss (63.11%) by effectively removing lignin and amorphous regions. Acharya *et al.*[8] studied the effects of chemical treatments by alkali, methacryl silane, and potassium permanganate on *Helicteres isora* fibers to evaluate their potential as reinforcement in polymer composites. Alkali treatment improved tensile strength by 4% and modulus by 30%, while silane treatment reduced water absorption by 18%, enhancing fiber-matrix compatibility.

The goal of this study has been to create sustainable and high-performance composite materials by reinforcing unsaturated polyester with sawdust that has been treated with sodium hydroxide (NaOH) solutions at varied concentrations (2, 4, 6, and 8%) for 5 hours at 40 °C. The effects of the treatment were evaluated by FTIR, XRD, TGA, DTG, SEM, water absorption and tensile strength tests. The expected application of these composites is in lightweight automotive interior panels (*e.g.*, door panels, dashboard components), where low density and good tensile strength are critical requirements.

EXPERIMENTAL

Materials

Sodium hydroxide pellets (NaOH) – 98% purity, pH 13-14 (5% w/v aqueous solution), melting point 318 °C, density 2.13 g/cm³ at 20 °C – were received from Prochima Sigma, Tlemcen, Algeria. Glacial acetic acid (CH_3COOH) – 98.5% purity, pH 2.4 (60 g/L aqueous solution), boiling point 117 °C, density 1.048 g/cm³ at 20 °C – was produced by Biochem Chemopharma, France.

The sawdust was supplied by a local sawmill in Bouira, Algeria. Its particle size was determined using 1 mm and 4 mm sieves (Table 1). The bulk density of the sawdust was calculated using the cylindrical method, which is commonly used for powdered materials. The bulk density value was calculated using Equation (1):

$$\rho = \frac{m}{v} \tag{1}$$

where m is the mass of dry sample (g), and v is the total volume of the sample (cm³).[9]

Table 1
Physical properties of sawdust

Property	Sawdust
Particle size	1-4 mm
Bulk density	0.17 g/cm³
Color	Light brown

Table 2
Mechanical and physicochemical characteristics of unsaturated polyester

Property	Value
Appearance	Clear
Shelf life	6 months
Density	1.125 g/cm³
Viscosity 23 °C	550/650 cP
Styrene content	34-37%
Acid value	22 mg KOH/g
Gel time at 23 °C	7-9 min
Tensile strength	63 MPa
Flexural strength	125 MPa
Water absorption	0.18%

The matrix is an orthophthalic, low-reactivity, medium-viscosity, unsaturated polyester resin produced by Poliya Composite Resins and Polymers (Istanbul, Turkey), designed for casting applications. The key properties of this matrix are outlined in Table 2.

Alkali treatment of sawdust

Sawdust was sun-dried for three days to eliminate any excess moisture. It was sieved using 1 and 4 mm sieves to remove solid wood, metal, or plastic particles. Then, it was soaked in NaOH solution with different concentrations (2, 4, 6, and 8 wt%) at a solution ratio of 1:20 (w/v), and it was placed in a water bath at 40 °C for 5 hours. After the alkali treatment, the samples were soaked in a 1% acetic acid solution for 30 minutes to remove excess sodium hydroxide, rinsed with distilled water several times to pH 7, and air-dried for 3 days. Finally, the samples were dried in an oven at 105 °C for 5 hours (Fig. 1). Table 3 lists the codes given to the prepared samples.

Table 3
Codes of the sawdust samples prepared

Sample code	Alkali treatment
USD	Untreated sawdust
TSD2	Sawdust treated with 2 wt% NaOH
TSD4	Sawdust treated with 4 wt% NaOH
TSD6	Sawdust treated with 6 wt% NaOH
TSD8	Sawdust treated with 8 wt% NaOH

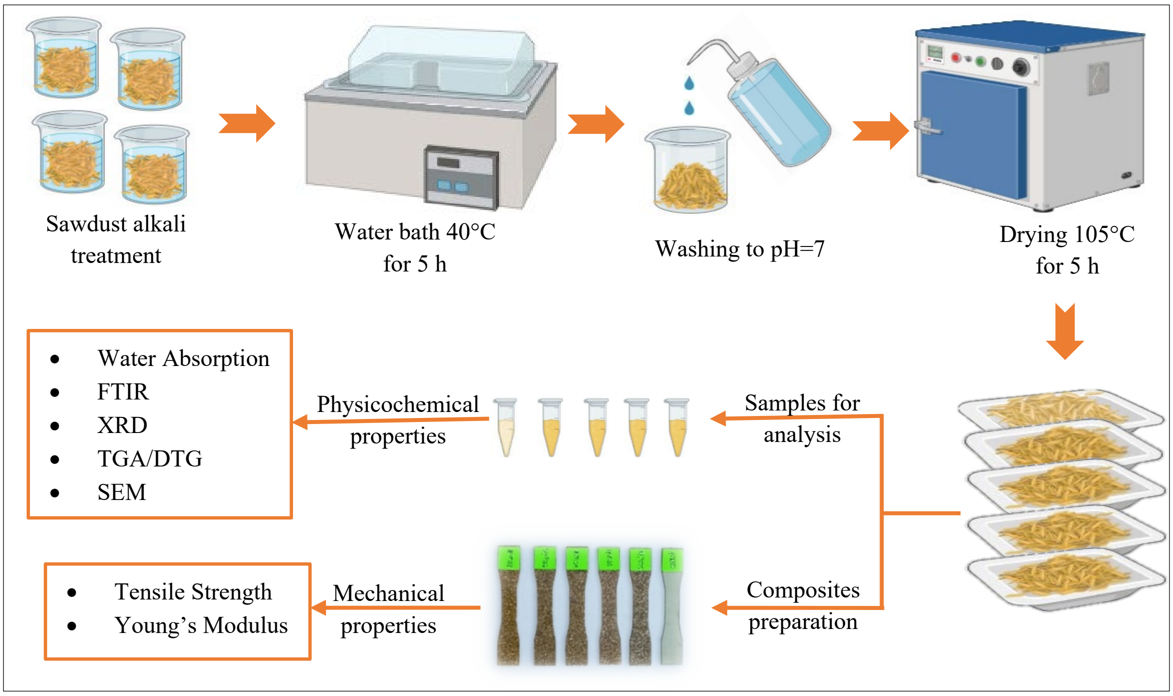

Figure 1: Schematic illustration of the stages of the study

Preparation of sawdust reinforced UP Composite

The hand lay-up method was used to prepare the composites. Unsaturated orthophthalic polyester matrix was utilized, cured with 2% methyl ethyl ketone peroxide. To ensure uniform fiber dispersion, dried fibers were mixed with the resin using a high-speed electric mixer (3000 rpm) for 10 minutes at 25 °C before adding the MEKP hardener. Specimens were fabricated using a silicone mold specifically designed for tensile testing of plastic materials according to the ASTM D638 standard. The dimensions are 200 mm long, and the test sections are 57 mm long, 13 mm wide, and 4 mm thick. The composite material was formulated with a fiber content of 10% (v/v). The mixture was then poured into the mold and subjected to balanced hand pressure to remove air bubbles. The samples were left to dry for 24 hours at room temperature. To obtain better mechanical properties, we dried the samples in an oven at a temperature of 60 °C (Fig. 1), measuring the weight of the samples every hour until constant weights were achieved. Table 4 lists the codes given to the composite materials that were examined.

Table 4
Codes of prepared composite materials

Composite code	Composition
0000/UP	Pure unsaturated polyester
USD/UP	Untreated sawdust/Unsaturated polyester
TSD2/UP	Sawdust treated with 2 wt% NaOH/Unsaturated polyester
TSD4/UP	Sawdust treated with 4 wt% NaOH/Unsaturated polyester
TSD6/UP	Sawdust treated with 6 wt% NaOH/Unsaturated polyester
TSD8/UP	Sawdust treated with 8 wt% NaOH/Unsaturated polyester

Characterization

Water absorption test

Water absorption studies on untreated and alkali-treated sawdust were conducted to determine their hygroscopic characteristics. Approximately 1 g of each sample was immersed in distilled water. After each immersion period, the sample was removed from the container, and excess water on its surface was removed before weighing. The weights of the samples were measured using a precise digital balance with a sensitivity of 0.0001 g. Water absorption was measured at 6 hour intervals until the weight stabilized. The percentage of water sorption in the samples was calculated using Equation (2):

$$\Delta W = \frac{Wf - Wi}{Wi} \times 100 \tag{2}$$

where ΔW is the water sorption (%), Wf *is* the sample weight after immersion in water, and Wi is the sample weight before immersion in water.[10]

Fourier transform infrared spectroscopy (FTIR)

FTIR spectra of sawdust were obtained before and after alkali treatment using an Agilent Cary 630 FTIR spectrometer (India). The measurements were conducted at a resolution of 8 cm^{-1} with 64 scans, focusing on the transmission mode across the 4000 to 400 cm^{-1} range. This approach allows for detailed analysis of molecular changes due to treatment.

X-ray diffraction analysis (XRD)

X-ray diffraction analysis was used to determine the crystallographic properties of both untreated and alkali-treated sawdust, including the value of the crystallinity index (CI). The XRD measurements were carried out using a PANalytical X'Pert PRO powder diffractometer (the Netherlands) with Cu-Kα radiation at operating conditions of 40 kV and 40 mA. The diffractograms were acquired using a step scanning mode of 2 Theta ranging from 10° to 70° and a scanning rate of 0.02° at room temperature. The crystallinity index (CI) was calculated using Segal's equation:

$$CI = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \tag{3}$$

where I_{002} is the intensity of the crystalline phase peak at around 22° and I_{am} is the intensity of the amorphous phase peak at around 18°.[11]

Thermogravimetric analysis (TGA)

Thermogravimetric analysis is used to examine several parameters, including substance breakdown and moisture loss. The technique depends on how temperature variations impact the sample's mass.[12] In this study, both untreated and alkali-treated sawdust samples were subjected to the TGA analysis. Measurements were made using a Mettler Toledo TGA/DSC 3+ (Switzerland). The analysis was performed in the temperature range from 20 to 900 °C at a heating rate of 10 °C/min.

Scanning electron microscopy (SEM)

The untreated and alkali-treated sawdust's surface morphology was studied using a Thermo Scientific Quanta SEM Prisma E electron microscope, manufactured in the USA. The lowest possible image resolution is 7 nm at an accelerating voltage of 3 kV, while the highest is 3 nm at 30 kV. An accelerating voltage of 20 kV was employed to prevent sample degradation, which would occur if the incident electrons' impact speed on the object was too high.

Tensile test

Tensile tests were conducted on the composite specimens to determine their mechanical properties using a Universal Testing Machine (TesT 112.10 kN, made in Germany). The specimens were prepared for the tensile tests in accordance with the standard ASTMD638. The initial load applied was 1 N, and the crosshead speed was 2 mm/min.

RESULTS AND DISCUSSION

Water absorption of untreated and alkali treated sawdust

The water absorption behavior of natural fibers is influenced by several factors, including chemical and physical treatments. Chemical treatments such as alkali, benzoylation, silane, and permanganate treatments can reduce water absorption by natural fibers. Physical modifications, like surface roughening, can also enhance their hydrophobicity.[13,14] As shown in Figure 1, the water sorption rate is notably higher at the start of the test. Over time, the values stabilize and become nearly constant, aligning with the findings previously reported by Gudayu *et al.*[15] In this study, untreated sawdust served as a control. It was noted that alkali treatment reduced the moisture absorption of sawdust. Also, the moisture absorption of the alkali treated sawdust decreased as the alkali concentration increased; these results are consistent with previous reports by Kamaruddin *et al.*,[16] and Begum *et al.*[10]

The untreated sawdust sample showed high water absorption due to its chemical composition, which includes hydrophilic components like cellulose and hemicelluloses. These components contain hydroxyl groups that form hydrogen bonds with water molecules, leading to significant water absorption.[4,10] Alkali-treated sawdust samples showed less water absorption, possibly due to several factors. Alkali treatment can remove hemicelluloses and other hydrophilic components from the fiber surface, reducing its ability to absorb water.[17,18]

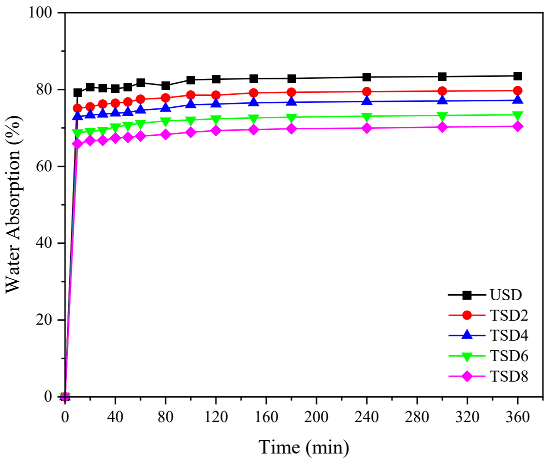

Figure 1: Water absorption behavior of untreated and treated sawdust

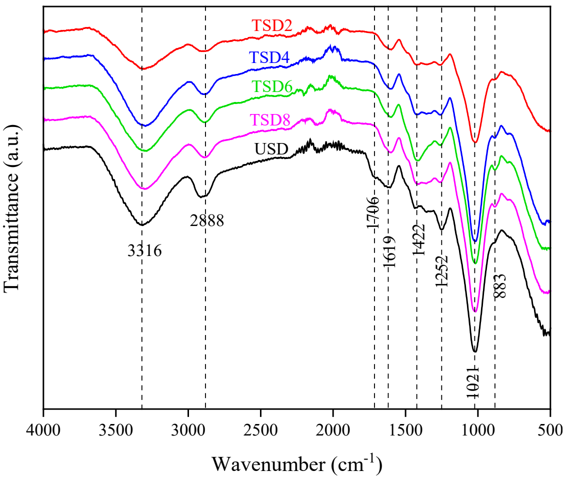

Figure 2: FTIR spectra of untreated and treated sawdust

FTIR analysis of sawdust

The FTIR spectrum of sawdust (Fig. 2) exhibits characteristic absorption bands corresponding to various functional groups present before and after alkali treatment. The broadband absorption at 3316 cm^{-1} indicates the presence of hydroxyl groups, primarily from cellulose and lignin.[19] A substantial decline in peak intensity following the treatment by NaOH, signifying decreased hydrogen bonding and hydrophilic character in the alkali-treated sawdust samples due to the removal of non-cellulosic components.[20] A distinct absorption band at 2888 cm^{-1}, observed in both treated and untreated sawdust, corresponds to the alkyl C-H stretching vibration inherent to cellulose and hemicelluloses components within natural fibers.[16] The absorption band at 1706 cm^{-1}, corresponding to C=O stretching vibrations of acetyl and ester groups of hemicelluloses and aromatic components of lignin, is absent in alkali-treated samples, indicating the effective removal of hemicelluloses and lignin from the sawdust surface.[21,22] An absorption band at 1619 cm^{-1} corresponds to the -OH bending vibration mode of the absorbed water in the crystalline cellulose.[22] The absorption peaks at 1422 cm^{-1} and 883 cm^{-1} correspond to CH2 scissoring vibrations and C-O-C stretching at the β-glycosidic linkage of cellulose, respectively.[23] The peak observed at 1252 cm^{-1} associated with asymmetric stretching of the C-O-C bands in hemicelluloses exhibits a weaker intensity in alkali-treated sawdust compared to the untreated sawdust sample. This reduction in intensity indicates a partial removal of hemicelluloses.[24] The peak at 1021 cm^{-1} is linked to the vibration of C-O bonds in polysaccharides.[25] These findings indicate that alkali treatment effectively removes the non-cellulosic components from the sawdust surface.

X-ray diffraction of sawdust

The diffraction spectra of untreated and alkali-treated sawdust are displayed in Figure 3, while the effects of the treatment on the crystallinity index are shown in Figure 4. Following the treatment, the samples' crystallinity index values indicate an improvement. Comparing the treated to the untreated sawdust, the highest crystallinity index is

75.65% for the sample (TSD4). These findings also demonstrate that increasing the alkali concentration during the treatment has a beneficial effect and raises the crystallinity index (CI). Atangana *et al.*,[26] Arnata *et al.*[27] and Hawanis *et al.*[28] found the same outcomes: as the concentration of NaOH increased, the crystallinity index and mechanical properties of fibers gradually improved. This rise could be due to the effect of alkali treatment on the chemical structure of the plant fibers. Alkali treatment improves the crystallinity index of plant fibers primarily by removing amorphous components, such as hemicelluloses and lignin. This process enhances the proportion of crystalline cellulose, resulting in a more ordered structure. However, excessive treatment can lead to fiber damage and a decrease in crystallinity.

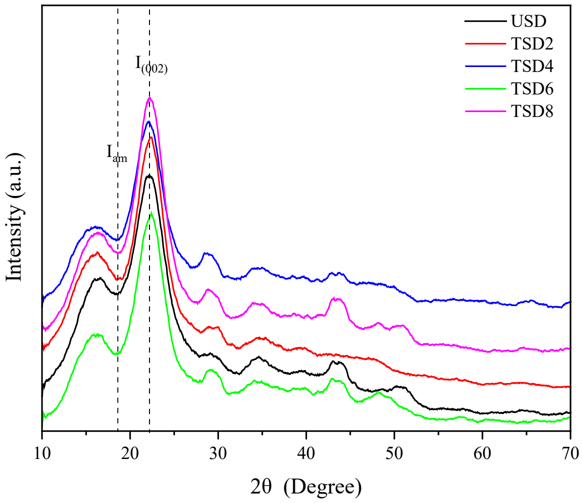

Figure 3: XRD patterns of untreated and treated sawdust

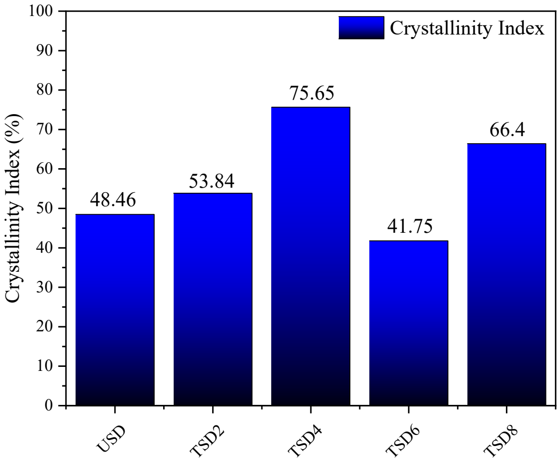

Figure 4: Crystallinity index of untreated and treated sawdust

Thermogravimetric analysis of sawdust

Figure 5 displays the treated and untreated sawdust's thermal stability curves. There are three distinct stages of mass loss. In the first stage, between 35 and 110 °C, the treated and untreated sawdust lost small weight due to moisture evaporation.[29,30] In the second stage, where temperatures ranged from 215 to 390 °C, untreated sawdust underwent significant weight loss at 224 °C, whereas TSD4 started this degradation stage around 239 °C. This weight loss may be explained by the degradation of hemicelluloses and cellulose.[12] Lastly, the third stage, which occurs between 390 and 900 °C, is linked to the breakdown of lignin.[31] The TSD4 sample was noted to have higher thermal stability than the untreated sawdust. Alsafran *et al.*[32] reported similar results. Alkaline treatment increases the thermal stability of natural fibers. Higher degradation temperatures of treated sawdust may be due to the efficient elimination of amorphous materials like hemicelluloses during alkali treatment.[33] Also, alkali-treated sawdust exhibited a lower residual mass (10.69%) compared to the untreated sawdust (14.48%), likely due to the higher lignin and wax content found in the untreated sawdust.[32]

Furthermore, the DTG analysis in Figure 6 indicates that the alkali treatment of sawdust enhances the stability of cellulose at elevated temperatures, resulting in an increase in the maximum thermal degradation temperature of cellulose from 326 °C in the untreated sawdust sample (USD) to 362 °C in the sawdust sample treated with 4 wt% NaOH (TSD4). This improvement in thermal stability can be attributed to the removal of organic impurities, such as pectin and wax, from the natural fiber structure through the alkaline process. Consequently, a greater proportion of cellulosic structures remain in the treated fibers, contributing significantly to their enhanced thermal stability.[34] These findings are consistent with those obtained from X-ray diffraction (XRD) analysis.

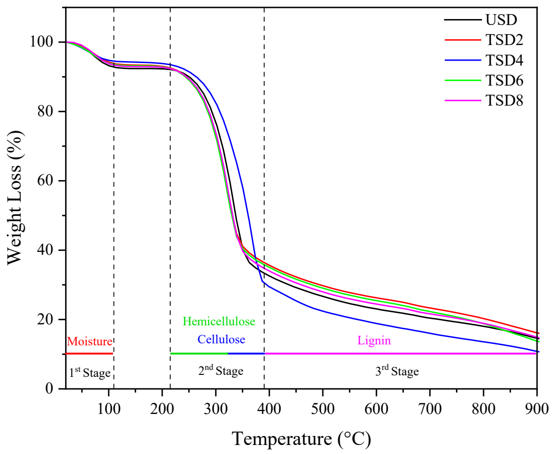

Figure 5: TGA curves of untreated and treated sawdust

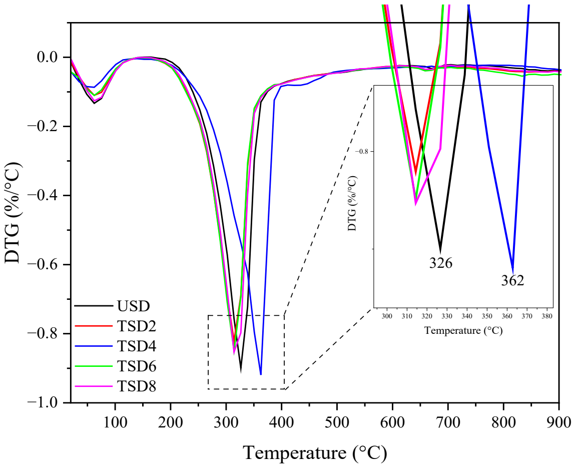

Figure 6: DTG curves of untreated and treated sawdust

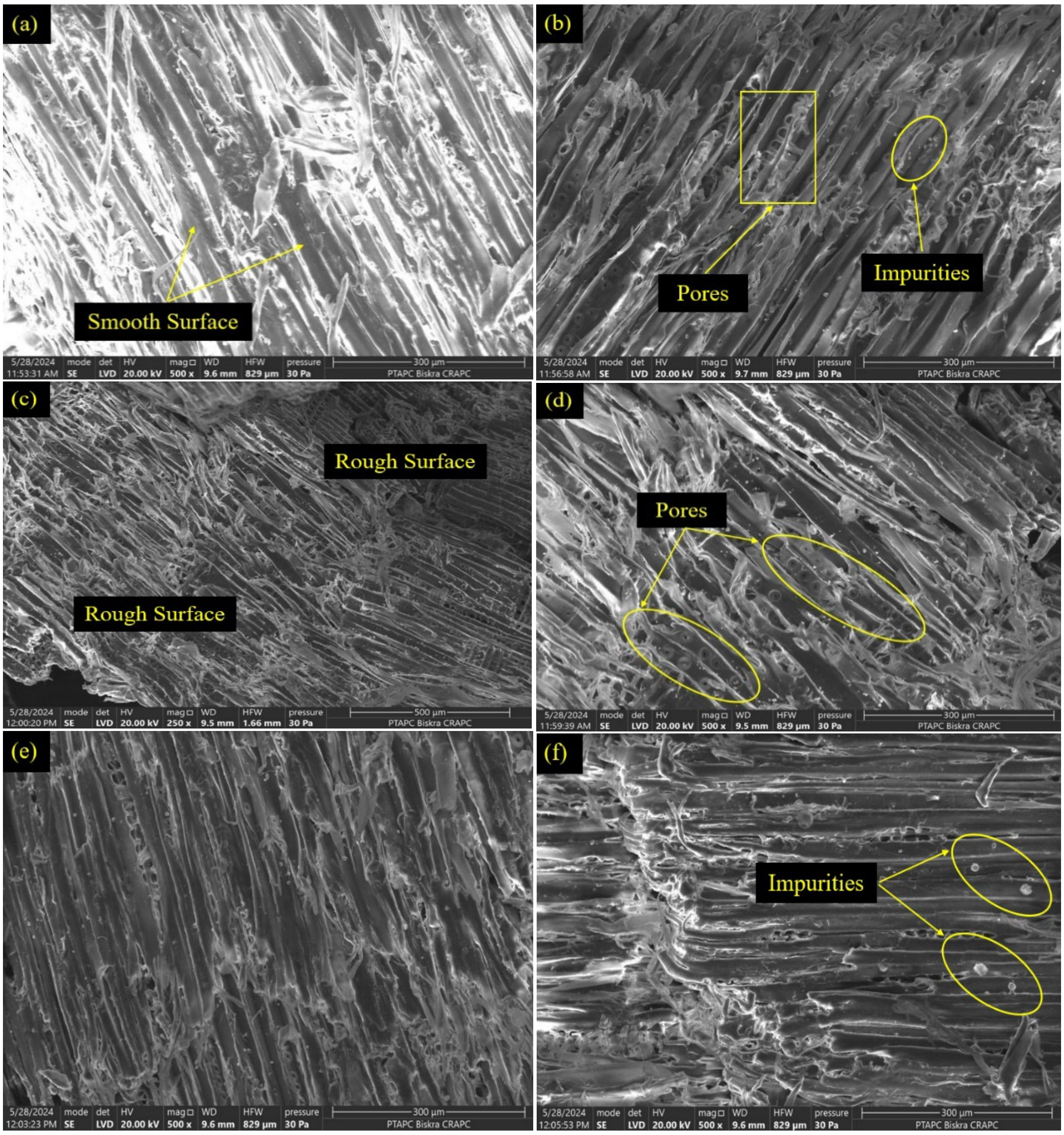

Figure 7: SEM images of (a) USD; (b) TSD2; (c), (d) TSD4; (e) TSD6; (f) TSD8

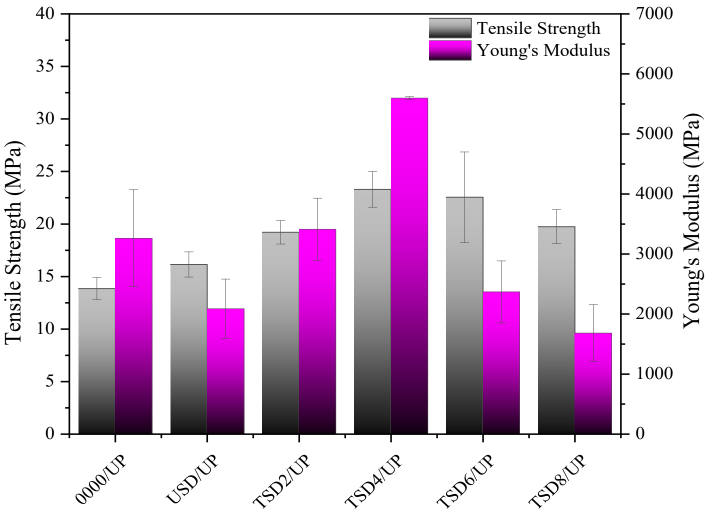

Figure 8: Tensile strength and Young's modulus of the studied composites

SEM analysis of sawdust

Figure 7 displays SEM micrographs of sawdust. The surface morphology of treated and untreated sawdust was examined. Following chemical treatment with sodium hydroxide, the surface of the sawdust exhibits morphological changes, as shown in the micrographs. Untreated sawdust has a smooth surface (Fig. 7(a)), whereas treated sawdust has a rough one (Fig. 7(b)-(c)). Surface roughness increases as the sodium hydroxide concentration increases. The appearance of pores on the surface of sawdust treated with soda is attributed to the removal of certain impurities such as lignin, hemicelluloses, and waxes. This leads to enhanced fiber-matrix adhesion in composites. Similar results have been reported in the literature.[35,36]

Mechanical properties of composites: tensile strength and Young's modulus

Figure 8 shows that alkali treatment of sawdust with varying sodium hydroxide concentrations enhances mechanical properties of sawdust-reinforced unsaturated polyester composites compared to composites reinforced with untreated sawdust. The treatment improves fiber-matrix adhesion by removing impurities like lignin and hemicelluloses, reducing fiber diameter, and increasing surface area. This leads to better cellulose packing and fiber dispersion. The composite reinforced with the sawdust treated with 4 wt% NaOH (TSD4/UP) showed the highest tensile strength of 23.3 MPa, a 44% increase over the composite reinforced with untreated sawdust, highlighting improved mechanical efficiency with higher alkali concentrations. This idea was also confirmed for other natural fibers like piassava,[37] banyan,[33] and kenaf.[34]

Treated sawdust composites also show a higher Young's modulus than the untreated ones. An optimal NaOH concentration improves mechanical properties, including Young's modulus, indicating that chemical treatment enhances the stiffness and overall performance of sawdust-reinforced composites. These findings are consistent with those of Hurtado-Figueroa *et al.*,[38] and Wang *et al.*[39] Composites reinforced with 4 wt% treated sawdust (TSD4/UP) show a double Young's modulus (5599 MPa) compared to the composites with untreated sawdust. However, higher alkali treatment may excessively degrade cellulose, which reduces the mechanical properties and causes a decrease in Young's modulus. This explains the decrease in Young's modulus observed for the composite with 8 wt% treated sawdust (TSD8/UP).[40]

CONCLUSION

This study developed sawdust-reinforced polyester composites with improved mechanical properties by treating sawdust with NaOH solutions (2, 4, 6 and 8 wt%) at 40 °C for 5 hours before composite manufacturing. The alkali treatment roughened the fiber surfaces, enhancing adhesion between the sawdust and the polyester matrix. This led to significant improvements in tensile strength (up to 23.3 MPa) and Young's modulus (5599 MPa). The treatment also increased the crystallinity index of sawdust, with the TSD4 sample reaching 75.65%, indicating a more ordered fiber structure. Thermogravimetric (TGA) and derivative thermal (DTG) analyses confirmed the removal of surface impurities like hemicelluloses and lignin, which contributed to better composite performance. The demonstrated tensile strength improvements (44% increase) combined with enhanced thermal stability (up to 239 °C) from fiber modification make them suitable for lightweight automotive interior panels.

Future research should explore additional surface modifications to further improve sawdust-polyester composites. Expanding mechanical and durability testing, assessing scalability, and combining sawdust with other bio-fillers can enhance performance and application potential. Advanced characterization methods will provide deeper insights, supporting the development of sustainable, eco-friendly composites for diverse industrial uses.

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