



# OPEN Effect of alkaline treatment on the thermal and mechanical properties of sugar palm fibre reinforced thermoplastic polyurethane composites

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This study examines the impact of alkaline treatment on the mechanical, thermal, and morphological properties of sugar palm fiber (SPF)-reinforced thermoplastic polyurethane (TPU) composites. SPF fibers (250  $\mu\text{m}$ ) were treated with sodium hydroxide (NaOH) solutions of 0%, 2%, 4%, and 6% (w/w) for 90 min at room temperature. These fibers were then mixed with TPU in an extruder at 180–200 °C and molded into composite sheets using compression molding at 190 °C, 10 MPa pressure, and a 10-minute holding time. X-ray diffraction (XRD) revealed increased crystallinity in the composites, peaking at 1589 cps with 6% NaOH. Fourier transform infrared (FTIR) spectra showed improved fiber-matrix compatibility, most evident with 2% NaOH. SEM analysis indicated cleaner and rougher fiber surfaces post-treatment, though cracks were observed at higher concentrations. Energy-dispersive X-ray (EDX) confirmed increased oxygen content and sodium residue at elevated NaOH levels, reflecting lignin removal. Thermogravimetric analysis (TGA) demonstrated enhanced thermal stability, with the highest residue mass of 599.16 °C at 6% NaOH. Alkaline treatment notably enhanced SPF/TPU composites, with 2% NaOH providing optimal flexural strength and 6% NaOH maximizing impact strength and thermal stability.

**Keywords** Mechanical properties, Thermal properties, Sugar palm fiber, Thermoplastic polyurethane, Alkaline treatment.

Industrial practices are evolving to produce environmentally friendly products by substituting certain functions of synthetic plastics sourced from petroleum. Products derived from composites are made from natural sources, which are crucial in minimizing carbon footprints and offering readily available properties thanks to their widespread presence in nature<sup>1</sup>. The choice of natural fibers for composite reinforcements significantly

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impacts global environmental protection, promoting sustainable development, preserving living ecosystems, and facilitating the creation of eco-friendly products<sup>2–4</sup>.

Natural fibers like hemp, flax, jute, wood, and various waste cellulosic products that reinforce polymer composites have undergone extensive study as alternatives to synthetic fibers. Polymer composites reinforced with synthetic fiber presented specific environmental health concerns<sup>5</sup>. Applying natural fiber exhibits advantages such as higher load capacity, ease of modification, low cost, and density. It contributes to reduced grinding in processing equipment and is relatively harmless, biodegradable, renewable, and mechanically comparable to inorganic fibers. Additionally, it features low density, biological degradation, health and environmental protection, simple separation, accessibility, enhanced energy recovery, non-corrosive properties, cost reduction, and practical thermal and acoustic insulation attributes<sup>6–10</sup>.

Sugar palm is a natural fiber that contributes to sustainable composites, offering numerous benefits as a raw material for products<sup>11</sup>, such as simple biological degradation, low production costs, high density, reproducibility, non-toxicity, and wear resistance<sup>12</sup>. Sugar palm fiber exhibits a range of applications, such as in bioplastics, food packaging, separators, and seat cushions<sup>13,14</sup>. The role of sugar palm fiber in improving composite performance is influenced by several factors, including changes to the fiber surface structure<sup>5,6</sup>, orientation, fiber distribution, and fiber composition, as well as the bond strength between the fiber and the matrix. Modified fiber demonstrates improved thermal stability. The biocomposite films made from sugar palm, wheat starch, and polyvinyl alcohol (PVA) had 24.6% more mass left over at 495 °C than the fiber that had not been treated, which was 23.3%. This indicates that employing chemical treatment improves the fiber's resistance to thermal degradation<sup>9</sup>. A study by Radzi et al. in 2023 found that treating sugar palm fiber with 6% NaOH for three hours on sugar palm, bamboo, and kenaf with polyester hybrid biocomposites significantly decreased the amount of dirt, lignin, and hemicellulose. This process improves the bond strength between the fiber and the resin matrix while also increasing the fiber's hydrophilicity, which ultimately results in reduced water absorption<sup>11</sup>. On the other hand, the decrease in adhesion at the fiber-matrix interface and more voids significantly contributed to the hastened failure process of the composite<sup>10</sup>.

Although sugar palm fiber (SPF) has been extensively examined as a composite reinforcement with matrices like epoxy and high-impact polystyrene<sup>15–19</sup> research on the application of SPF in thermoplastic polyurethane (TPU) matrices remains quite scarce. In 2019, Afzaluddin et al. reported on sugar palm/glass fibre-reinforced thermoplastic polyurethane hybrid composites, emphasizing their physical and mechanical properties<sup>20</sup>. TPU exhibits distinctive characteristics such as high tensile strength, toughness, durability, and resistance to wear and friction, presenting significant opportunities for applications involving natural fiber-based composites<sup>21</sup>. However, the chemical treatment of SPF might change how the sugar palm fiber and TPU interact, which could influence the composite's mechanical and thermal properties. This phenomenon requires comprehensive and extensive examination. Despite these promising attributes, several knowledge gaps remain regarding optimizing SPF/TPU composites, particularly in understanding the effects of fiber treatment on interfacial bonding, mechanical performance, and long-term stability. Additionally, challenges such as potential fiber degradation, moisture absorption, and processing compatibility need further exploration to unlock the full potential of SPF-reinforced TPU composites. This study seeks to assess the impact of alkali treatment with NaOH solution at concentrations of 0%, 2%, 4%, and 6% on the characterization, mechanical properties, and thermal properties of sugar palm fiber/polyurethane thermoplastic (SPF/TPU) composites.

## Experimental setup

### Material

Sugar palm fiber (SPF) used in this study was purchased from Aceh, Indonesia. The resin used was thermoplastic polyurethane (TPU) type Estane<sup>®</sup> 58,311 (Selangor, Malaysia). The chemical treatment given to the SPF used sodium hydroxide (NaOH) pellet AR media. The chemical composition of SPF under pre-treatment and post-treatment settings was derived from prior research papers, as illustrated in Table 1<sup>22</sup>.

### Preparation of materials

The fibers were washed with water to remove dust and impurities and then dried at atmospheric pressure and room temperature for two weeks. Afterward, they were crushed using a Retsch ZM 200 grinder (Haan, Germany) and subsequently sieved to a suitable size of 250 µm using a FRITSCH auto shaker (Idar-Oberstein, Germany) to make them ideal for the final application<sup>23</sup>.

### Treatment of fibre

Sugar palm fiber in composites goes through stages of cleaning and chemical treatment. First, sugar palm fibers were washed eight times using running water to remove dirt attached to the fiber surface and dry at room

Samples	Cellulose (%)	Hemicellulose (%)	Holocellulose (%)	Lignin (%)	Extractive (%)	Ash (%)
Sugar Palm Fiber	43.88	7.24	51.12	33.24	2.73	1.01
Bleached Fiber	56.67	19.8	76.47	0.27	0.23	2.16
NaOH-Treated Fiber	82.33	3.97	86.3	0.06	–	0.72
Sugar Palm Refined Fiber	88.79	0.04	3.85	0.04	–	0.74

**Table 1.** Chemical composition of sugar palm fibers at different stages of treatment<sup>22</sup>. \.

temperature for 30 min. The second stage is SPF going through chemical treatment using NaOH (0, 2, 4, and 6% (w/w)) to modify the fiber surface structure to increase the interfacial bond of SPF and TPU<sup>24</sup>. SPF is soaked in a plastic container containing NaOH for 90 min. After that, sugar palm fibers were washed thoroughly with distilled water to be free from dirt and residual NaOH<sup>4,15,16</sup>. Finally, sugar palm fibers were left to dry at room temperature for two weeks<sup>15</sup>.

### Composites Preparation

The Thermo Scientific Eurolab 16 extruder machine from Karlsruhe, Germany, was used to make a mixture of 30% SPF and 70% TPU with a rotation speed of 40 rpm and a temperature variation of 180–200 °C<sup>23,25,26</sup>. The finished material mixture was put into a mold measuring 200×200×3 mm and compacted using hot pressing (LOTUS SCIENTIFIC) with a capacity of 25 tons. The machine was set at a pressure of 10 MPa with a temperature of 190 °C and a pressing time of 10 min, then continued with cooling until it reached a temperature of 50 °C. The result, a sheet-shaped composite, was cut according to the size of each test standard.

### X-ray diffraction

The amorphous and crystalline structures of SPF/TPU composites were observed using X-ray diffraction (Rigaku Mini Flex model, Japan) with an X-ray generator tube current of 15 mA and a voltage of 30 kV. The structures formed indicate the interfacial bonding of fibers and matrices<sup>27</sup>.

### Fourier transform infrared spectroscopy

The structural and functional groups of SPF/TPU composites were analyzed using Fourier Transform Infrared Spectroscopy (FTIR) type Thermo Scientific iS50 (Madison, USA). The samples consisted of TPU, composites without fiber treatment, and composites with fiber treatment, where the wave numbers identified were in the range of 4000 to 650 cm<sup>-1</sup>.

### Scanning Electron microscope

Scanning electron microscopy (SEM) analysis is critical in examining the distribution of fibers within the matrix and understanding the nature of the composite failure. The SEM test samples utilized in this investigation were obtained from the fractured surfaces of tensile test specimens assessed in earlier studies<sup>28</sup>. The SEM employed was the Carl Zeiss EVO model from Germany. The samples underwent a coating process involving a thin layer of platinum applied via a sputter coater, resulting in clear contrast images observable under the microscope.

### Energy dispersive X-ray

The SPF/TPU composites were analyzed using energy-dispersive X-ray (EDX) with a HITACHI TM3030 Plus machine from Japan to evaluate the impact of chemical treatment on the composite's elemental composition. The distribution of elements provides insights into the adhesion quality between the fiber and the matrix, which significantly influences their mechanical and thermal properties.

### Thermogravimetric analysis

Thermogravimetric analysis (TGA) was utilized to evaluate the thermal resistance of SPF/TPU composites by monitoring weight loss as the temperature increased, providing insights into the degradation mechanism of the composite. Test specimens weighing 3 g each were prepared and analyzed using a TGA Mettler Toledo (Switzerland) instrument. The testing was conducted at a heating rate of 10 °C/min over a temperature range from room temperature to 600 °C under a nitrogen atmosphere.

### Differential scanning calorimetry

Analyzing the effect of fiber treatment on the thermal behavior and crystallinity level of SPF/TPU composites is a critical aspect in determining the thermal resistance of the composites. Test samples weighing 3 g were prepared in a container and tested using a Perkin Elmer DSC 800 (USA) under nitrogen atmospheric conditions with a flow rate of 10 ml/min, a heating rate of 10 °C/min, and a temperature range from room temperature to 300 °C.

### Flexural testing

The flexural test was conducted on the SPF/TPU composite to assess its ability to resist stress before structural failure and its capacity to retain deformation. The test specimens, each measuring 130×13×3 mm, were fabricated following ASTM D790 guidelines, with five samples in total. Each sample was tested using an Instron 3369Q3720 universal testing machine, employing a three-point bending setup and a crosshead speed of 2 mm/min.

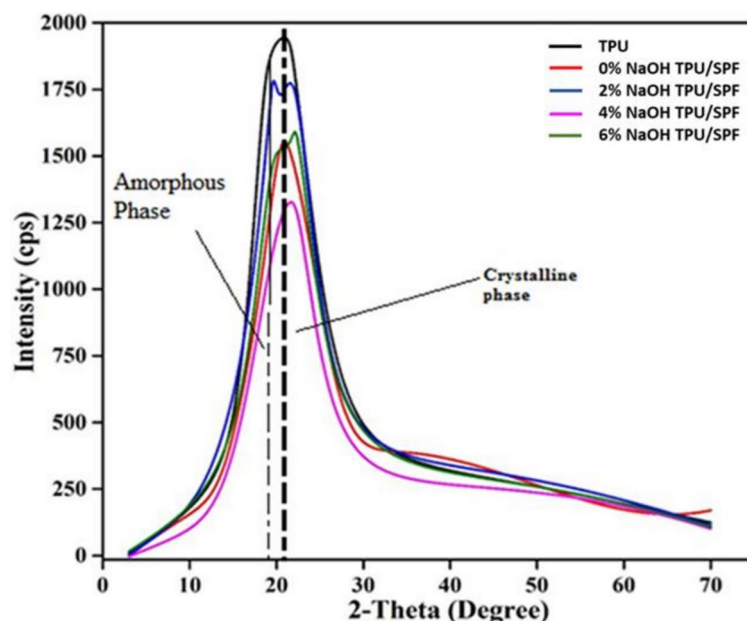
### Impact testing

The impact test aims to assess the ability of the SPF/TPU composite to absorb energy from a sudden load before the material breaks. The testing was performed according to ASTM D256 standards, with five specimens prepared, each measuring 63×13×3 mm. The specimens were tested using a Zwick/Roell 5113 impact tester (Ulm, Germany) with a 15 J capacity to observe the fracture surface of the material.

## Results and discussion

### X-ray diffraction

The X-ray diffraction (XRD) analysis for pure TPU, untreated SPF/TPU (0% NaOH), and SPF/TPU subjected to NaOH treatment at concentrations of 2%, 4%, and 6% are illustrated in Fig. 1. The XRD analysis demonstrated two prominent peaks for TPU at 19.07° and 21.95° (theta), suggesting the existence of a carbon phase. The peak



**Fig. 1.** XRD diffractograms for pure TPU, untreated SPF (0% NaOH), and treated SPF (2, 4, and 6% NaOH) reinforced TPU composites.

observed at 21.95° theta signifies the crystalline phase of TPU, whereas the peak at 19.07° theta reflects the amorphous characteristics of TPU.

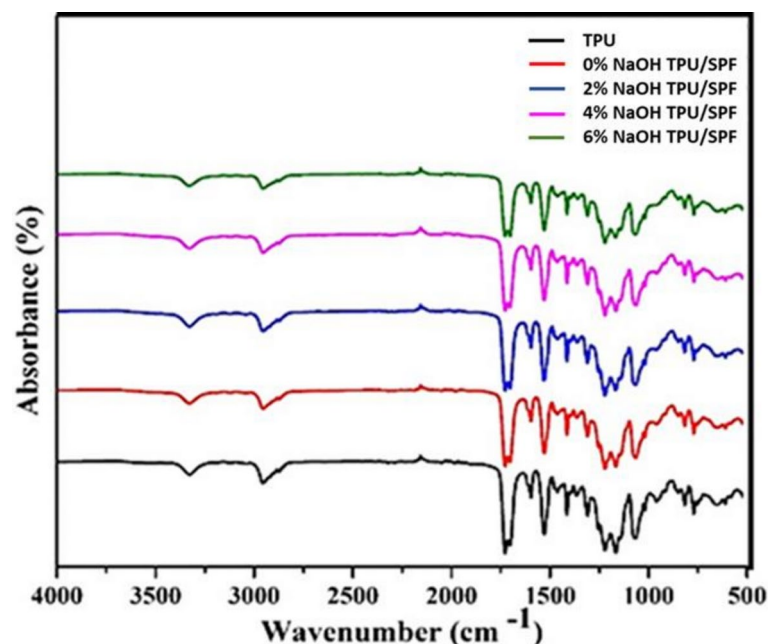
After incorporating SPF into the matrix, notable alterations were observed in the diffraction pattern. One of the three peaks in the untreated SPF/TPU composite (0% NaOH) disappeared after being treated with NaOH solution. This suggests that the chemical treatment modified the structure of the material. The fibers' crystallinity index increased because non-cellulosic compounds like lignin and hemicellulose were removed using chemicals and machines. Additionally, the reduction of the humps following alkali treatment suggests that the amorphous chains have organized into crystalline regions. The sharpness of the peaks indicates a higher crystallinity index<sup>29</sup>. Le Troëdec et al. also evaluated the degradation of amorphous hemicellulose from the fiber surface in their study concerning the treatment of lime straw and hemp fiber<sup>30,31</sup>.

A peak intensity of about 1589 cps was seen after 6% NaOH was applied. This clearly shows that the chemical treatment significantly improved the crystallinity of the SPF/TPU composite. The observed rise may be because amorphous parts like hemicellulose and lignin were removed, making the composite material's structure more regular. This further substantiates the hypothesis that the chemical treatment of the fibers improves the fiber-matrix interaction by eliminating amorphous barriers, leading to a composite with a more uniform structure<sup>32</sup>.

### Fourier transform infrared spectroscopy

Figure 2 displays the fourier transform infrared spectroscopy (FTIR) spectra for both pure TPU and SPF/TPU composites that have been treated with varying concentrations of NaOH (0%, 2%, 4%, and 6%). The standard absorption peaks related to O–H, N–H, CH<sub>2</sub>, CH<sub>3</sub>, and C=O functional groups of the fibers and polymer matrix can be found in Table 2. A prominent broad band in the range of 3,420–3,200 cm<sup>−1</sup>, linked to O–H stretching vibration, shows a shift from 3,326.2 cm<sup>−1</sup> in pure TPU to 3,327.55–3,328.91 cm<sup>−1</sup> in the SPF/TPU composites. This change signifies an improvement in hydrogen bonding and the compatibility between the fibre and matrix, particularly at 2% NaOH<sup>29</sup>.

The N–H peak (1,590–1,650 cm<sup>−1</sup>) consistently measured around 1,596 cm<sup>−1</sup>, suggesting that the treatment with NaOH did not alter the interaction of this group. The CH<sub>2</sub> and CH<sub>3</sub> stretches (3,000–2,800 cm<sup>−1</sup>) had a slight change in position, going from 2,956.5 cm<sup>−1</sup> in pure TPU to a range of 2,955.77–2,956.22 cm<sup>−1</sup> in SPF/TPU. This shows that the interactions are stable. In pure TPU, the non-bonded C=O band (1,740 cm<sup>−1</sup>) moved from 1,726.9 cm<sup>−1</sup> to a range of 1,726.26–1,726.43 cm<sup>−1</sup>. The corresponding C=O peak (1,690 cm<sup>−1</sup>) moved from 1,701.12 cm<sup>−1</sup> to a range of 1,701.53–1,702.05 cm<sup>−1</sup><sup>33,34</sup>. This change shows that the NaOH treatment strengthened the chemical interactions between the fibers and the TPU matrix<sup>35</sup>. No new peaks emerged, indicating that the interaction observed was physical, facilitated by increased hydrogen bonds. This suggests that the primary adhesion mechanism between the fibers and the TPU matrix is dominated by physical interaction rather than chemical bonding. The peak shifts, particularly in the C=O and CH<sub>2</sub>/CH<sub>3</sub> areas, indicate improved interfacial adhesion through enhanced hydrogen bonding. This reinforcement contributes to the composite's improved mechanical and thermal performance, particularly at 2% NaOH treatment, by strengthening fiber-matrix compatibility without introducing new covalent interaction<sup>36–39</sup>.



**Fig. 2.** FTIR of TPU, 0% NaOH SPF/TPU, 2% NaOH SPF/TPU, 4% NaOH SPF/TPU, and 6% NaOH SPF/TPU composites.

Peak location (cm <sup>-1</sup> ) *	Chemical structure	Motion	Pure TPU	0% NaOH SPF/TPU	2% NaOH SPF/TPU	4% NaOH SPF/TPU	6% NaOH SPF/TPU
3,420–3,200	O–H	Stretching	3326.2	3328.72	3327.55	3328.55	3328.91
1,590–1,650	N–H	Bending	1596.2	1596.14	1596.35	1596.27	1596.21
3,000–2,800	CH <sub>2</sub> and CH <sub>3</sub>	Stretching	2956.5	2955.99	2955.77	2956.04	2956.22
1,740	C=O	Non-bonded urethane Stretching	1726.9	1726.66	1726.26	1726.29	1726.43
1,690	C=O	Associate urethane	1701.12	1702.05	1701.83	1701.60	1701.53
1,550–1,510	H–N–CO	Combined motion	1529.27	1528.53	1529.45	1529.24	1528.72

**Table 2.** FTIR result of pure TPU, 0% NaOH SPF/TPU, 2% NaOH SPF/TPU, 4% NaOH SPF/TPU, and 6% NaOH SPF/TPU composites.

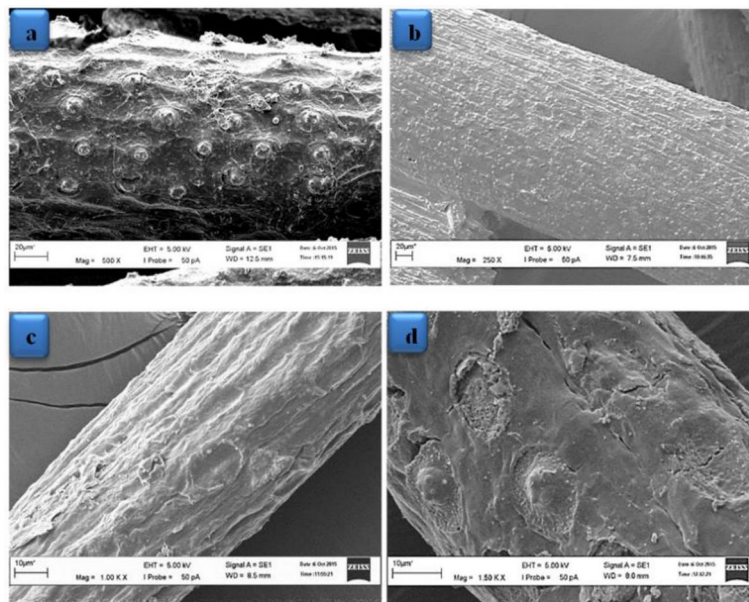
### Scanning electron microscopy

Figure 3 shows what the scanning electron microscopy (SEM) saw on the surface of the fiber before it was treated with 0% NaOH and after it was treated with 2, 4, and 6% NaOH. The untreated surface of the SPF, illustrated in Fig. 3a, exhibits a significant accumulation of dirt, comprising oil, wax, and various other contaminants. The SPF treated with NaOH concentrations of 2%, 4%, and 6%, as illustrated in Figs. 3b–d, exhibits a progressively cleaner and rougher surface<sup>40</sup>. This demonstrates that NaOH treatment eliminates dirt and non-cellulosic layers that obscure the fiber, including oil and wax. This process enhances the cleanliness of the fiber surface while also developing a rougher surface structure, which can improve the mechanical interaction between the fiber and the matrix in the composite<sup>4</sup>. Increasing the NaOH concentration from 2 to 4% and then to 6% results in forming a few cracks on the fiber surface, with the 6% NaOH concentration exhibiting higher cracks. Cracks may lead to fiber brittleness, influencing the dimensions and alignment of the fibers within the matrix and their capacity to withstand stress<sup>32</sup>.

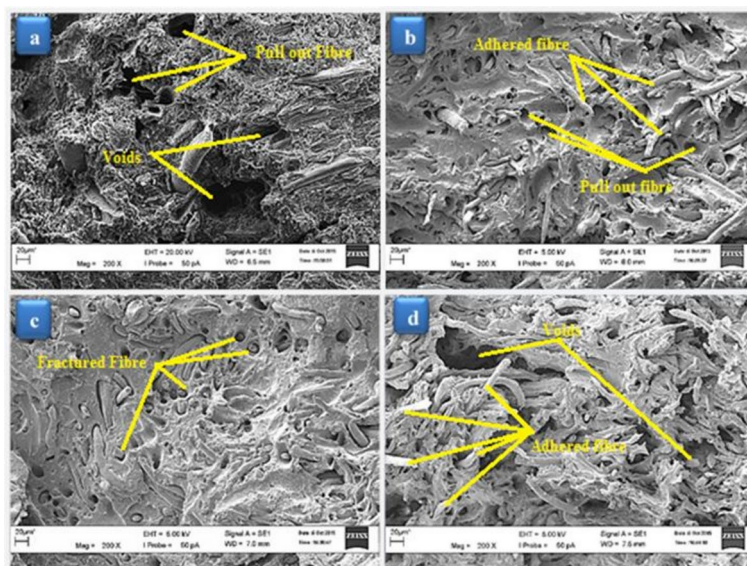
The SEM observation of SPF/TPU composites treated with NaOH reveals a nuanced relationship between the quality of the fiber-matrix interface and the mechanical properties, as illustrated in Fig. 4. In 2019, Allaeddin et al. identified various failure models that may arise in composites, such as fiber-matrix debonding, fiber pull-out incidents, fiber breakage, matrix cracking, and matrix yielding<sup>41</sup>. In the sample lacking NaOH treatment (0%) depicted in Fig. 4a, there is an apparent prevalence of pull-out fibers and significant voids. The inadequate adhesion between the fiber and matrix leads to an uneven stress distribution, resulting in diminished mechanical properties during bending and impact assessments<sup>32</sup>.

At a 2% NaOH concentration (Fig. 4b), SEM shows a clear improvement in adhesion (stuck fibers), but some fibers are still pulling away. This treatment eliminates a significant portion of lignin and hemicellulose, enhancing the interaction between the fiber and the matrix. The flexural strength at this concentration shows that the fiber-matrix interface is working well to support the stress distribution<sup>42</sup>. Nonetheless, the tensile strength observed





**Fig. 3.** SEM of (a) SPF with 0% of NaOH, (b) treated SPF with 2% of NaOH, (c) treated SPF with 4% of NaOH, and (d) treated SPF with 6% of NaOH.



**Fig. 4.** SEM of (a) 0% NaOH SPF/TPU, (b) 2% NaOH SPF/TPU, (c) with 4% NaOH SPF/TPU, (d) and 6% NaOH SPF/TPU.

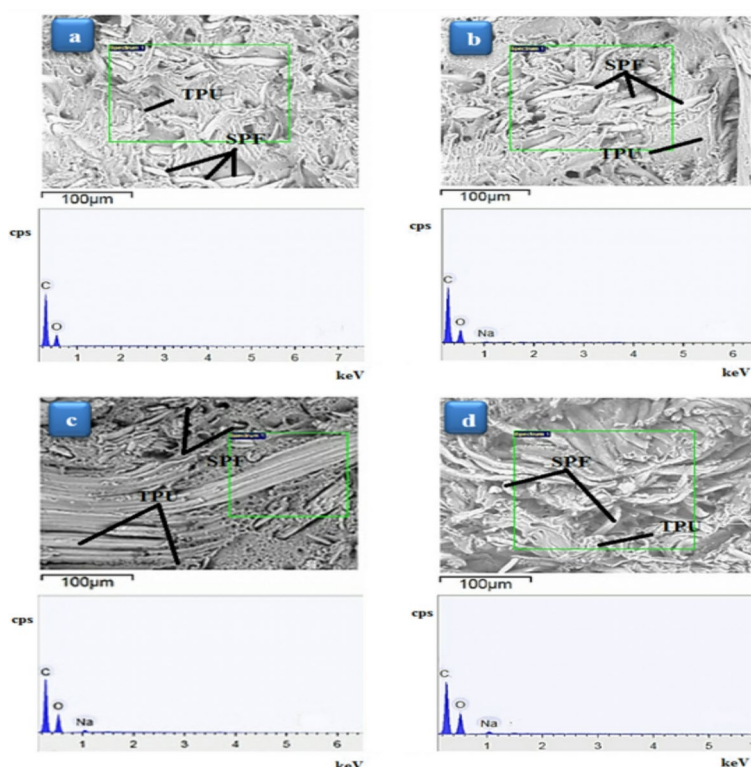
at this concentration is inferior to that of the 6% NaOH treatment (Fig. 4d), as indicated in earlier research. This may be attributed to the TPU matrix's insufficient penetration, which has not yet reached an optimal level to cover and absorb the fibers adequately<sup>43</sup>. Consequently, this results in a diminished strengthening effect from the fibers in the tensile direction<sup>28</sup>.

Figure 4c shows a concentration of 4% NaOH. The SEM results show multiple fiber fracture phenomena that point to strong fiber-matrix adhesion. Nonetheless, the mechanical findings indicate a reduction in flexural strength relative to a concentration of 2% and the lowest impact strength observed. The observed phenomenon can be attributed to the rapid absorption of impact energy, leading to more pronounced damage to the fibers with minimal energy dissipation. The fiber fracture phenomenon happens rapidly and is not accompanied by additional energy dissipation mechanisms like matrix fracture or plastic deformation, which diminishes the material's capacity to endure high-impact energy<sup>44</sup>.

At a concentration of 6% NaOH, the SEM analysis reveals minimal voids, a prevalence of adhered fibers, and a more uniform distribution of fibers. In 2020, Sabarinathan et al. found that the application of silane treatment

Concentration of NaOH	Element	Weight%	Weight% $\sigma$	Atomic %
0% NaOH	Carbon	58.635	0.628	65.487
	Oxygen	40.702	0.630	34.127
	Natrium	—	—	—
2% NaOH	Carbon	58.265	0.649	65.275
	Oxygen	40.263	0.653	33.86
	Natrium	1.472	0.115	0.862
4% NaOH	Carbon	56.982	0.549	64.112
	Oxygen	41.276	0.553	34.865
	Natrium	1.741	0.103	1.024
6% NaOH	Carbon	55.629	0.697	62.845
	Oxygen	42.523	0.702	36.064
	Natrium	1.848	0.132	1.091

**Table 3.** Elements analysis on the surface of SPF/TPU composites.

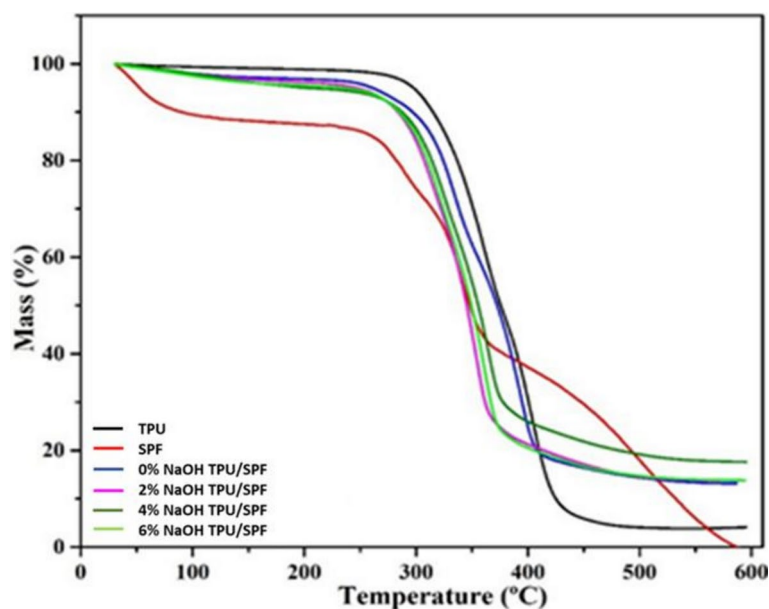


**Fig. 5.** EDX of (a) 0% NaOH SPF/TPU, (b) 2% NaOH SPF/TPU, (c) with 4% NaOH SPF/TPU, (d) and 6% NaOH SPF/TPU.

to fish tail palm fibre/vinyl ester resin with compositions of 10, 20, and 30 wt% caused a decrease in void content from 2.34, 2.67, and 2.99% to 1.98, 2.10, and 2.50%<sup>38</sup>. The earlier studies showed that this condition led to the highest tensile strength across all samples. This is because the better interfacial adhesion makes it easier for stress to move from the matrix to the fibers during tensile testing<sup>36,45</sup>. Moreover, the impact strength peaks at this concentration, as the TPU matrix demonstrates superior capability in absorbing impact energy due to robust interactions with the fibers. Nevertheless, the flexural strength was diminished compared to the 2% and 4% concentrations, likely because of fiber degradation stemming from excessive alkali treatment that influenced the fiber orientation within the matrix<sup>46</sup>.

### Energy dispersive X-ray spectroscopy

The element composition on the surface of the untreated SPF/TPU composite (0% NaOH) and after NaOH treatment at concentrations of 2%, 4%, and 6% were evaluated through energy dispersive x-ray spectroscopy (EDX) analysis, as tabulated detailed in Table 3; Fig. 5. The findings indicate that the carbon element is predominant in the untreated SPF/TPU composite (0% NaOH), accounting for a weight% of 58.635%, whereas



**Fig. 6.** TGA for pure TPU, SPF, treated and untreated with different NaOH concentrations (0, 2, 4, and 6%).

Samples	Residual Mass (%)		
	T <sub>26.99</sub> /°C	T <sub>303.00</sub> /°C	T <sub>599.16</sub> /°C
Pure TPU	100.0	94.18798	4.1
SPF	99.51	71.89	0.003774
0% NaOH SPF/TPU	99.98	90.45	13.15
2% NaOH SPF/TPU	99.65242	94.30348	13.84
4% NaOH SPF/TPU	99.73518	94.46382	17.56
6% NaOH SPF/TPU	99.76049	94.76698	13.81

**Table 4.** Residual mass percentage of pure TPU, SPF, and SPF/TPU composites (different treatment concentrations of NaOH solution).

oxygen contributes 40.702%. This composition highlights the prevalence of the TPU polymer matrix and SPF fibers, which retain considerable amounts of hemicellulose and lignin content<sup>47</sup>.

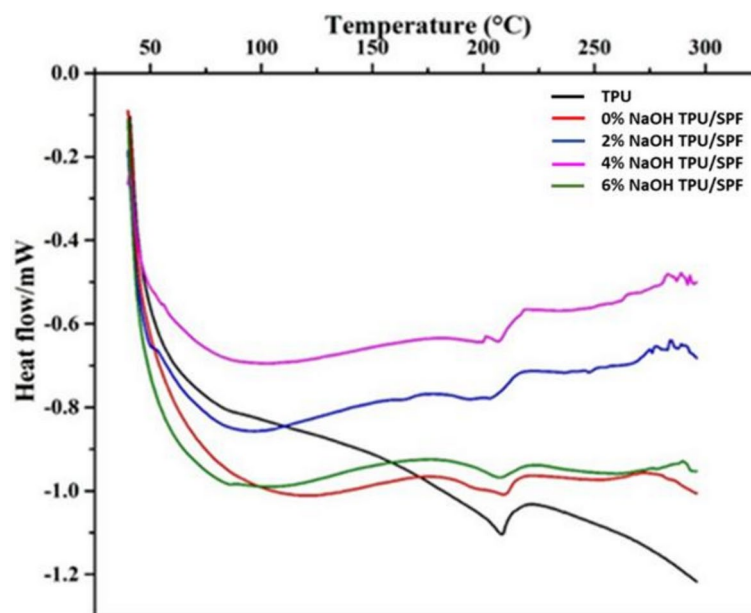
Following the treatment with NaOH, notable alterations in the elemental composition were observed<sup>48</sup>. The composite subjected to 2% NaOH treatment exhibited a slight reduction in carbon content to 58.265%, alongside a decrease in oxygen content to 40.263%. The sodium content of 1.472% suggests residual NaOH solution adsorbed on the fiber surface. As the concentration of NaOH rose to 4% and 6%, the carbon content diminished further, reaching 56.982% and 55.629%, respectively. The oxygen content steadily increased, reaching 42.523% in the 6% NaOH treatment. Furthermore, the sodium content rose to 1.741% with 4% NaOH and 1.848% with 6% NaOH.

The reduction in carbon content and the rise in oxygen indicate the partial extraction of amorphous hemicellulose and lignin from the surface of SPF fibers following NaOH treatment<sup>49</sup>. The treatment with 6% NaOH led to a more significant rise in oxygen content than the 4% and 2% treatments, suggesting that new hydroxyl groups were revealed due to the breakdown of the fibers' amorphous components. The presence of sodium residue in the NaOH-treated samples indicates that, despite the washing of the fibers, some residual NaOH solution remained on the surface. This residue may enhance the roughness of the fiber surface, potentially leading to robust mechanical interactions between the fibers and the TPU matrix within the composite. Nonetheless, elevated sodium levels may also enhance the hygroscopic properties of the composite, a factor that must be considered in real-world applications<sup>50</sup>.

### Thermogravimetric analysis

The thermogravimetric analysis (TGA) results are shown in Fig. 6. It indicates that the SPF/TPU composite treated with NaOH is more thermally stable than the untreated composite and pure SPF. Table 4 displays the mass residue data at significant temperatures (26.99 °C, 303.00 °C, and 599.16 °C) for conditions lacking NaOH treatment (0%) and for NaOH treatments of 2, 4, and 6%. The test on pure TPU showed that it had a significant mass residue of 94.19% at 303.00 °C and 4.1% at 599.16 °C, showing that it is very resistant to thermal degradation. In contrast, pure SPF exhibited a residue of only 71.89% at 303.00 °C and was nearly depleted at





**Fig. 7.** DSC analysis for pure TPU, untreated (0% NaOH) and treated 2, 4, and 6% NaOH of SPF/TPU composite.

Composites	T <sub>g</sub> (°C)	T <sub>m</sub> (°C)	Energy of fusion (J/g)	Crystallinity (C) %
TPU	197.27	208.64	39.8027	20.224
0% NaOH SPF/TPU	198.54	204.61	33.441	16.992
2% NaOH SPF/TPU	182.47	203.32	32.668	16.599
4% NaOH SPF/TPU	190.00	200.85	30.560	15.528
6% NaOH SPF/TPU	192.09	207.39	37.577	19.094

**Table 5.** Values of the T<sub>c</sub>, T<sub>m</sub>, the (ΔH<sub>exp</sub>), and C % for pure TPU, untreated SPF/TPU, and treated SPF/TPU composites with different NaOH concentrations.

599.16 °C (0.0038%), attributable to the significant presence of hemicellulose and lignin, which are prone to degradation. A 0% untreated SPF/TPU composite showed better thermal stability, with a mass residue of 90.45% at 303.00 °C and 13.15% at 599.16 °C. This indicates that TPU affects the thermal properties of SPF<sup>4,51</sup>.

Applying alkali treatments using NaOH enhanced the thermal stability of the composite<sup>52</sup>. At NaOH concentrations of 2% and 4%, the residue at elevated temperatures (599.16 °C) rose to 13.84% and 17.56%, respectively. The observed increase is because wax, lignin, and hemicellulose are removed from the fiber surface. This makes the fiber less amorphous and increases the crystalline structure of cellulose. The increased crystalline cellulose enhances hydrogen bonding and optimizes the distribution of thermal energy through a more stable structure<sup>53</sup>. The treated fibers are less likely to absorb water, which makes it easier for them to stick to the TPU matrix<sup>54</sup>. At a NaOH concentration of 6%, the residue at a temperature of 599.16 °C was observed to decrease to 13.81%. This is probably a result of excessive fiber degradation, which compromises the fiber structure as a reinforcement<sup>51</sup>.

### Differential scanning calorimetry

Differential Scanning Calorimetry.

The Differential Scanning Calorimetry (DSC) results show essential information about the thermal changes that happen in SPF/TPU composites, such as the crystallization temperature (T<sub>c</sub>), melting temperature (T<sub>m</sub>), melting energy, and crystallinity level (Fig. 7). The numbers show how changing the NaOH concentration (0, 2, 4, and 6%) affected the composite's thermal properties and the levels of crystallinity that went with them<sup>55</sup>, as shown in Table 5.

The melting temperature of pure TPU is 208.64 °C, accompanied by a maximum crystallinity level of 20.224%. If the SPF/TPU composite was not treated with 0% NaOH, it has a slightly lower T<sub>m</sub> of 204.61 °C and a crystallinity level of 16.992%. The observed decrease indicates SPF fibers' influence on the TPU matrix's crystallization process. The treatment with NaOH reveals differences in thermal properties. The composite subjected to 2% NaOH treatment exhibits a reduced T<sub>m</sub> of 203.32 °C compared to the untreated composite. The melting energy has decreased to 32.668 J/g, with a crystallinity level of 16.599%. The observed decrease

could be attributed to the partial extraction of hemicellulose and lignin, which influences the fiber and matrix interaction<sup>56</sup>.

The treatment with 4% NaOH resulted in a  $T_m$  of 200.85 °C and a crystallinity level of 15.528%. Changing the chemicals in the fiber means that increasing the concentration of NaOH might cause the TPU matrix to become less crystallized. In the case of the 6% NaOH treatment, the melting temperature ( $T_m$ ) was observed to rise to 207.39 °C, while the crystallinity level attained 19.094%. The observed rise might be because the fibers are now more compatible with the matrix after being treated with NaOH. This, in turn, may make the thermal interaction within the composite stronger<sup>57</sup>. The DSC results indicated that the treatment with NaOH influenced the thermal transition of the composite<sup>59–62</sup>. When  $T_m$  and crystallinity went down during the first treatment, the structure of the fibers changed. On the other hand, when they went up during later treatments, the matrix and fibers were better able to work together. This finding fits with earlier research that showed that chemical treatment could change the fibers' size and orientation within the matrix<sup>57</sup>, changing the composite's mechanical and thermal properties<sup>61</sup>.

### Flexural properties

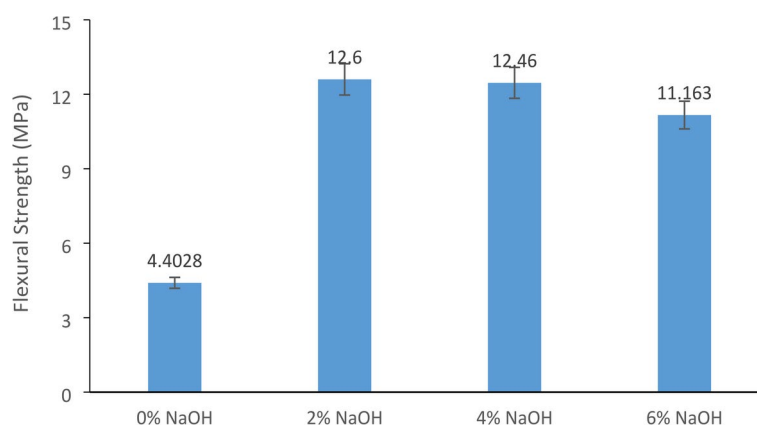
Figure 8 shows how the composite's flexural strength changes with different amounts of SPF immersion media, precisely at 0%, 2%, 4%, and 6% NaOH. An observable enhancement in the flexural strength value of SPF/TPU treated with NaOH solution is noted, in contrast to the untreated SPF/TPU composite (0 wt% NaOH). The flexural strength of the composite with untreated SPF measures 4.4028 MPa. In contrast, the flexural strength of the treated composite shows an increase to 12.6, 12.46, and 11.163 MPa at NaOH concentrations of 2, 4, and 6%, respectively. This finding indicates that NaOH treatment enhances the compatibility between fibers and TPU matrices<sup>44</sup>. Moreover, the uniformity of SPF within the matrix significantly improves flexural strength<sup>15</sup>.

The few empty spaces seen with SEM and the uniform distribution of SPF in the composite with a 2% NaOH concentration make the surface contact between SPF and the matrix stronger. This improvement allows the matrix to effectively transfer stress to the fibers, decreasing the likelihood of composite failure before fracture<sup>49</sup>. Increasing the amount of NaOH from 2 to 6%, on the other hand, lowers the flexural strength because SPF breaks down in the matrix<sup>4</sup>. Cracks start appearing on the fiber surface, making the fibers brittle and easily separate from the matrix when loaded. This causes a low and uneven stress transfer mechanism<sup>7</sup>.

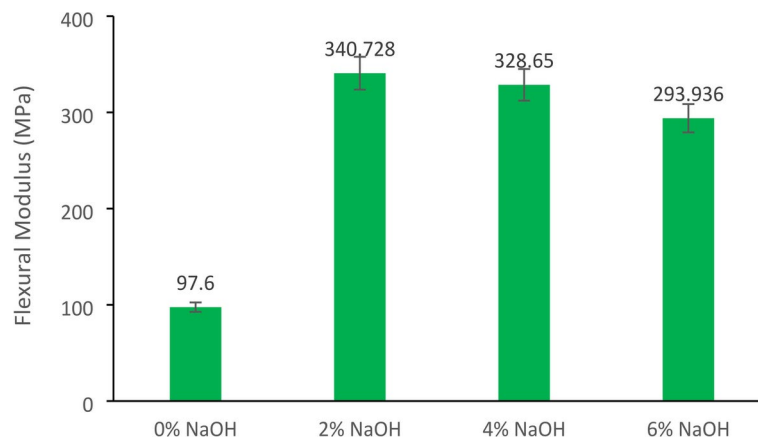
Figure 9 illustrates how varying NaOH concentrations (0, 2, 4, and 6%) influence the flexural modulus of SPF/TPU composites. A flexural modulus of 97.6 MPa was found when 0% NaOH was applied to SPF. This value increased significantly after 2%, 4%, and 6% NaOH treatments, giving 340.728, 328.65, and 293.936 MPa values in that order. The treatment with NaOH effectively eliminates impurities from the fiber surface, which enhances the interfacial interaction between SPF and TPU, consequently leading to an increase in the flexural modulus of SPF/TPU composites<sup>17,36</sup> and raising the NaOH concentration from 2 to 4% and then to 6% results in a reduction of the flexural modulus. The degradation of too short fibers leads to a disturbance in fiber orientation, which subsequently reduces the flexural modulus<sup>32,62</sup>. Additionally, SEM observations indicate that treatment with 4% and 6% NaOH leads to the formation of cracks on the fiber surface, initiating the cracking process and hastening the composite failure mechanism<sup>63</sup>.

### Impact strength

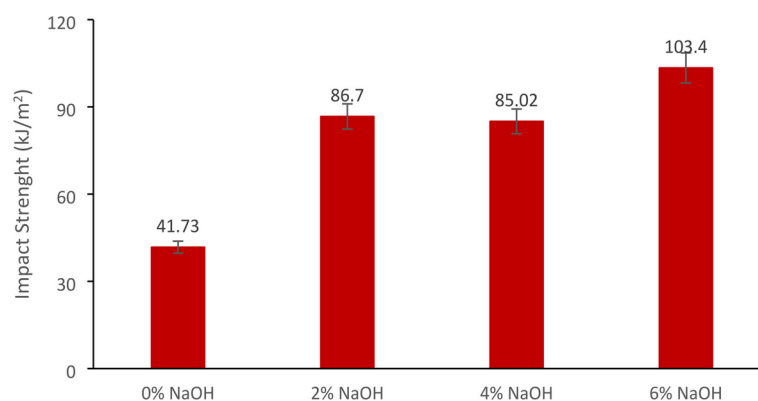
The influence of NaOH treatment at concentrations of 0, 2, 4, and 6% of SPF on the impact strength value of the SPF/TPU composite is illustrated in Fig. 10. The composite exhibited an impact strength of 41.73 kJ/m<sup>2</sup> without NaOH treatment, which increased to 86.7, 85.02, and 103.4 kJ/m<sup>2</sup> after SPF was immersed in NaOH solutions of 2, 4, and 6% before the mixing and molding process. The alteration of the fiber surface can effectively eliminate the components of hemicellulose, lignin, and wax layers, enhancing moisture resistance. This process influences the fiber's roughness and fosters a robust bond between SPF and TPU<sup>64</sup>. The implications of this are significant for improving the impact strength of the composite, given the substantial energy needed to induce fracture failure of the fiber<sup>58</sup>.



**Fig. 8.** The flexural strength variation of SPF/TPU composites with SPF treated at different NaOH concentrations.



**Fig. 9.** The flexural modulus of SPF/TPU composites using SPF treated with NaOH concentrations of 0%, 2%, 4%, and 6%.



**Fig. 10.** The impact strength of SPF/TPU composites with SPF treated at different NaOH concentrations.

Applying 6% NaOH on SPF demonstrates a significantly higher impact strength than the 2% and 4% concentrations. This effect can be attributed to removing a more significant amount of amorphous components<sup>29</sup>, which enhances the elasticity and energy absorption capacity of the composite<sup>65</sup>. The composition of 4% NaOH exhibits a lower impact strength than 6% and 2%, likely attributed to inadequate fiber distribution and fiber brittleness within the matrix, which diminishes elastic resistance and the composite's capacity to absorb impact energy<sup>59</sup>. Furthermore, the low impact strength value may result from the significant kinetic energy absorbed by the composite, leading to an increased likelihood of matrix cracks, fiber fractures, and more extensive permanent indentation in the impacted region<sup>66</sup>.

## Conclusions

This investigation assessed the impact of alkali (NaOH) treatment at varying concentrations of 0% (control), 2%, 4%, and 6% on the mechanical, thermal, and morphological characteristics of sugar palm fiber (SPF) composites reinforced with thermoplastic polyurethane (TPU). The XRD analysis results indicated that the treatment with NaOH enhanced fiber crystallinity, achieving a peak intensity of 1589 cps at a 6% NaOH concentration, in contrast to the control sample. The FTIR spectrum demonstrated enhanced compatibility between the fiber and matrix at a 2% NaOH concentration, indicated by a shift in the O–H and C=O bands, while no new peaks appeared. SEM observations indicated that NaOH effectively altered the fiber surface to exhibit increased roughness; however, at concentrations of 4% and 6%, microcracks were observed that could potentially impact fiber orientation, composite density, and the quality of matrix-fiber adhesion. The findings were validated by EDX analysis, which revealed an increase in oxygen content and sodium residues on the fiber surface following alkali treatment. The maximum flexural strength was obtained with 2% NaOH treatment, demonstrating a 25% increase relative to the control sample. The treatment with 6% NaOH resulted in the highest impact strength, attaining a value of 12.8 kJ/m<sup>2</sup>, which represents a 30% increase over the untreated sample. The treatment with 6% NaOH demonstrated optimal thermal stability, exhibiting the highest mass retention of 18.6% at a temperature of 599.16 °C, as indicated by TGA analysis. Nonetheless, elevating the NaOH concentration resulted in the degradation of lignin and hemicellulose, a factor that must be taken into account in material design for specific applications. The results indicate that a 2% NaOH concentration yields optimal performance for

enhancing flexural strength, whereas a 6% NaOH concentration is preferable for applications that demand high impact strength and thermal stability. It is advisable to conduct additional studies to investigate the integration of alkali treatment with various modification techniques, including the application of coupling agents or physical treatments like plasma, to enhance fiber-matrix adhesion and thermal stability. Furthermore, an examination of additional parameters like wear characteristics, environmental durability, and resilience to extreme conditions, including heat and humidity, might be conducted to broaden the applicability of this composite across diverse industries.

## Data availability

Data availability We ensure that all research data are contained within the article.

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### Author contributions

D.B.: conceptualization, supervision; A.A.M. and S.P.: writing—original draft, investigation; A.I.I.: Writing- review and editing, validation; J.P.S.: methodology, investigation; M.R.M.R.: formal analysis, data curation; S.S.: methodology, resources; T.C.: formal analysis, validation; D.F.F.: formal analysis, data curation; S.A.F., M.I.A. and Q.M.: Administration, Funding, Validation. All authors have read and agreed to the published version of the manuscript.

### Declarations

### Competing interests

The authors declare no competing interests.

### Ethical statement

This research study did not involve the use of animals and humans.

### Additional information

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