Effect of Surface Treatment on the Mechanical Properties of Sugar Palm/Glass Fiber-reinforced Thermoplastic Polyurethane Hybrid Composites

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Effects of various surface modifications were evaluated relative to the mechanical properties of sugar palm fiber/glass fiber (SPF/GF) reinforced thermoplastic polyurethane (TPU) hybrid composites. The 6 wt.% alkaline, 2 wt.% silane, and combined 6 wt.% alkaline-2 wt.% silane treatment of SPF were carried out for 3 h to improve the fiber/matrix interaction of SPF/GF with TPU. The SPF and GF were fixed at 30 wt.% and 10 wt.% fiber loading, respectively, and were fabricated using the melt compounding method followed by hot compression in a moulding machine. Mechanical properties, such as tensile, flexural, and impact strength, were evaluated using a universal testing machine and an Izod impact tester. The untreated and treated hybrid composites were characterized by FTIR spectroscopy. The tensile, flexural, and impact strength of the combined 6 wt. % alkaline-2 wt. % silane treatment was improved 16%, 39%, and 18%, respectively, as compared to the untreated SPF/GF reinforced TPU hybrid composites. Moreover, the scanning electron microscopy (SEM) showed a good fiber and matrix interfacial bonding in the hybrid composites. Thus, this treated hybrid composites could be suitable for fabricating automotive parts.

Keywords: Sugar palm fibers; Thermoplastic polyurethane; Sugar palm composites; Mechanical properties; Silane treatment

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INTRODUCTION

In light of environmental protection, the use of renewable resources and product biodegradability has driven many manufacturing industries to focus on using natural resources, especially fiber-rich plants, for the production of polymer composites. This trend has led to the use of plant fibers for reinforcement in polymer composites rather than relying on synthetic fibers (Westman *et al.* 2010; Ahmad *et al.* 2015). Economics, as well as their superior properties, has enticed many manufacturing industries into using synthetic fibers to reinforce plastics, and natural fibers has become a priority in the composite industry only after considering their low cost, low density, and high strength (Jawaid and Khalil 2011). Plant fibers are naturally grown and sown in suitable environments throughout the world. Natural plant fibers can be extracted from the plant's stem, leaf, and fruit. Some examples of plant fibers include kenaf, flax, jute, bamboo, hemp, abaca, sisal, and many more.

For use in polymer composites, the hydrophilicity of plant fibers has caused a compatibility issue. This has driven many studies to report on the extent of enhanced interfacial bonding either by way of plasticizing fibers or modifying the surface (Khalil *et al.* 2013; Rashid *et al.* 2016; Rajadurai 2016; Rashid *et al.* 2017). The main problem encountered is an issue of fiber-matrix-adhesion due to the incompatibility between the hydrophilic natural fibers and the hydrophobic polymer matrix. This problem can be improved by chemically treating the fiber surfaces. Silane treatment is a common method to clean and modify the fiber surface to have a lower surface tension and enhanced interfacial adhesion between a natural fiber and the polymeric matrix. Previous work by other researchers employed mercerization (Maleque *et al.* 2012; Afdzaluddin *et al.* 2013; Atiqah *et al.* 2014; Asim *et al.* 2016), silane (Abdelmouleh *et al.* 2007; Gharbi *et al.* 2014; Zhou *et al.* 2014; Zahari *et al.* 2015), and combined alkaline-silane treatment (Asumani *et al.* 2012; Ramamoorthy *et al.* 2015; Asim *et al.* 2016). For instance, Bakar *et al*. (2015) reported on alkaline-treated and untreated long unidirectional kenaf/woven Kevlar hybrid composites and found an improvement in the tensile strength but weaker impact properties. The tensile improvement was due to the improved interfacial bonding of the fiber.

In recent years, the incorporation of natural fibers like kenaf (Maleque *et al.* 2012; Atiqah *et al.* 2014), sugar palm (Sapuan *et al.* 2013; Misri *et al.* 2015), bamboo (Zuhudi *et al.* 2016), banana (Haneefa *et al.* 2008), jute (Acharya 2014), and coir (Kumar *et al.* 2009) with glass fiber has received much attention from research and other various industries due to the mechanical property improvements they provide compared with glass fiber composites. The addition of natural fibers with synthetic fibers improves the strength properties of both the natural and glass fibers. Sharba *et al.* (2015) studied the effects of fiber orientation on the mechanical properties and fatigue life of glass/kenaf hybrid composites and found that unidirectional kenaf hybrid composites have higher tensile and compressive strength than woven kenaf. Uma Devi *et al.* (2012) investigated the tensile and impact properties of short pineapple leaf/glass reinforced polyester composites and found that the tensile strength of the hybrid composites increased 28%, with the addition of 10 vol.% glass fiber. Hamouda *et al.* (2015) developed hybrid composites from coir fiber and woven glass fiber-reinforced polyester. Physical property testing revealed less thickness swelling for the hybrid composites compared to that of coir/polyester composites. Moreover, the tensile and flexural strength also were increased 88% and 298%, respectively, compared to that of coir/polyester composites.

There are several studies that investigate surface modifications of sugar palm fiber (Bachtiar *et al.* 2008; Leman *et al.* 2008; Ishak *et al.* 2009; Rashid *et al.* 2016). To date, there have been few or no attempts to study treated sugar palm/glass fiber reinforced thermoplastic polyurethane hybrid composites. The aim of this research is to evaluate the effects of various treatments, such as 6 wt.% alkaline (TNSP), 2 wt.% silane (TSSP), and combined 6 wt.% alkaline-2 wt.% silane (TNSSP), on the mechanical properties of SP/G-reinforced TPU hybrid composites. The hybrid composites have a constant sugar palm/glass weight fraction of 30%/10%. The tensile, flexural, and impact properties were determined and post tensile testing morphology was also characterized to be able to recommend the optimum formulation of the hybrid composites.

EXPERIMENTAL

Materials

Estane® 58311 TPU in pellet form with a density of 1.13 g/cm^3 and E-glass fiber with a length of 6 mm and a density of 2.55 g/cm^3 were supplied by Innovative Pultrusion Sdn. Bhd. (Negeri Sembilan, Malaysia). The sugar palm fiber (SPF) was collected from sugar palm trees in Jempol, Negeri Sembilan, Malaysia.

Preparation of sugar palm fiber

Sugar palm fiber (SPF) was cut into lengths of approximately ≤8 cm to 10 cm. The fibers then were purified with tap water several times to get rid of any impurities attached to the SPF. The SPF was kept in open air and then dried in an air circulating oven at 60 °C for 48 h. The dry SPF was ground to a size of 10 mm to 15 mm using a plastic crusher machine (Suzhou Poks Machinery Co., Ltd., Jiangsu, China) followed by a pulverizing machine and then the SPF particles were sieved to attain 125 µm to 250 µm particles.

The SPF was modified to enhance the fiber-matrix bonding of the hybrid composites. The SPF was treated using alkaline, silane, and combined alkaline-silane treatments. The detailed procedure for these surface modifications is described below.

In the alkaline treatment, the SPF particles (125 µm to 250 µm) were immersed in a 6 wt.% alkaline solution with distilled water for 3 h at room temperature. Then, the SPF was rinsed with distilled water until a neutral pH was obtained. The fibers were oven-dried for 48 h at 60 °C. This procedure is based on the modification method described by Atiqah *et al.* (2014).

In the silane treatment, the SPF particles (125 µm to 250 µm) were immersed in a 2 wt.% silane solution for 3 h. To obtain the silane treatment, 3-aminopropyl-tri ethoxy silane (APS) was mixed with a mixture of methanol and water (90/10 w/w) from the hydrolysis process. To obtain a solution pH of 3.5, the mixture was stirred with acetic acid for 10 min. The fibers were measured by weight percentage and soaked in this solution for 3 h under agitation. After this time, the SPF were washed with distilled water and decanted. The fibers were then oven-dried at 60 °C for 72 h to remove any moisture. This procedure is based on the modified method described by Atiqah *et al.* (2017). The chemical reaction of alkaline (1) (Vasquez *et al.* 2016) and silane treatment (2-3) (Agrawal *et al.* 2000) are given as follows.

$$\text{Fiber-OH} + \text{NaOH} \rightarrow \text{Fiber-O-Na}^{+} + H_2O \quad (1)$$

$$CH_2CHSi(OC_2H_5)_3 \xrightarrow{H_2 0} CH_2CHSi(OH)3 + 3C_2H_5OH \quad (2)$$

$$CH_2CHSi(OH)_3 + \text{Fiber- OH} \rightarrow CH_2CHSi(OH)_2\text{O-Fiber} + H_2O \quad (3)$$

In the combined alkaline-silane treatment, the samples were treated with a 6 wt.% alkaline treatment followed by a 2 wt.% silane treatment, as in the second procedure.

Fabrication of hybrid composites

The SPF/GF/TPU composites were prepared using a melt-mixing compounding method followed by hot-press moulding. The drying process was carried out for sugar palm particles of the size 125 µm to 250 µm, glass fibers, and thermoplastic polyurethanes in pellet form in an electric oven at 80 °C for 48 h. The chemical treatment of four sets of 30/10 wt.% SPF/GF-reinforced TPU composites is described in Table 1. The process of homogenization was performed at the optimum processing parameters of 40 rpm for 11 min at 190 °C using a Haake poly drive R600 (Thermo Electron Scientific, Karlsruhe, Germany). The compounds were produced with SPF/GF content of 30/10 by weight. The TPU polymer was mixed until steady torque was reached, and then the GF was added to the TPU melt. At a rotor speed of 40 rpm, additional runs with SPF were carried out at 190 °C. When the temperature reached 195 °C for the first time, the mixer was opened for a few seconds to reduce the temperature again to prevent the thermal degradation of SPF. To avoid the effect of residence time in the mixer on SPF degradation, mixing was stopped for both feeding methods immediately after SPF feeding and the compound was removed from mixer to cool down to room temperature. The hot-press moulding was performed using a Vechno Vation 40 ton compression molding machine (Carver, Inc., Wabash, IN, USA). The samples were preheated for 7 min, fully pressed for 10 min at 190 °C, and then cold-pressed for 5 min at 25 °C (El-Shekeil *et al.* 2012). The development of untreated and treated hybrid SPF/GF-reinforced TPU composites is depicted in Fig. 1.

Table 1. Chemical Treatment Parameter Employed on SPF/GF/TPU Hybrid Composites

Designation	TPU (wt. %)	Sugar Palm Fiber (SPF) (wt. %)	Glass Fiber (GF) (wt. %)	Treatment	Soaking Time (h)	Temp (°C)
UTSP[1]	60	30	10	Untreated	-	-
TNSP[2]	60	30	10	Treated with 6 wt. % Alkaline	3	23
TSSP[3]	60	30	10	Treated with 2 wt. % Silane	3	23
TNSSP[4]	60	30	10	Combined 6 wt. % alkaline-2 wt. % Silane	3	23

[1] Untreated sugar palm
[2] Treated NaOH sugar palm
[3] Treated Silane sugar palm
[4] Treated NaOH and silane sugar palm

Characterizations

Fourier transform infrared spectroscopy(FTIR)

FTIR spectra of untreated and treated SPF/GF/TPU hybrid composites was recorded to distinguish new absorption bands if any during surface modification of SPF. For sample preparation, the specimens were cut with size of 10 x 10 x 3 mm and then analyzed using FT-IR Spectrometer (Perkin Elmer Spectrum 100 series spectrophotometer, USA) equipped with attenuated total reflectance (ATR) capability. The spectra were recorded with a resolution of 2 cm^{-1} in the range of 4000 to 400 cm^{-1}.

Tensile test

The tensile test was performed on flat dog-bone-shaped sample per the ASTM D638 (2010) test standard using a universal testing machine (AMETEK Lloyd Instruments Ltd., West Sussex, UK). The specimen was tested by a calibrated universal testing machine with a speed of 50 mm/min. The average of six samples was used for all tests.

Flexural test

The flexural properties of developed SPF/GF/TPU hybrid composites were evaluated according to the ASTM D790 (2003) (3-point bending) standard. The test was executed using a universal testing machine (AMETEK Lloyd Instruments Ltd., West Sussex, UK) universal testing machine with a span length of 50 mm and a crosshead speed of 12 mm/min.

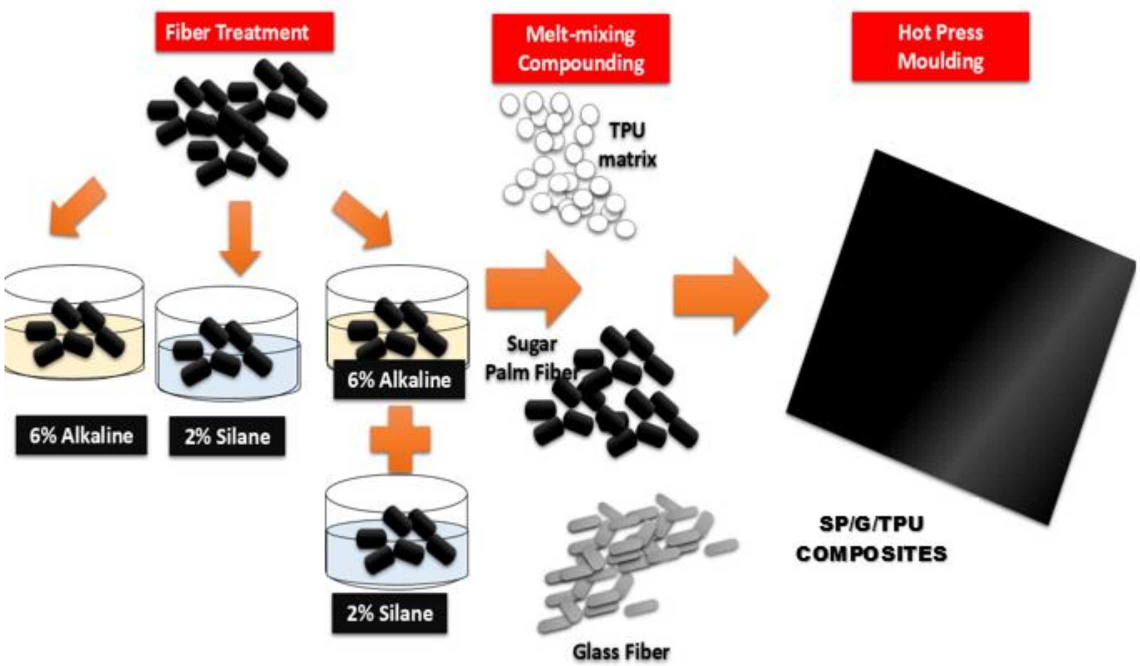


Fig. 1. Development of untreated and treated hybrid SPF/GF-reinforced TPU hybrid composites

Impact test

Standard notched Izod impact test (INSTRON CEAST 9050, Norwood, MA, USA) specimens were cut from the developed SPF/GF/TPU hybrid composite plates using an abrasive water-jet machine (Excel WJ 4080, OMAX, Corporation Kent, Washington, USA) according to ASTM D256 (1996). Averages of five samples were taken to present the final impact strength on select treated and untreated SPF/GF/TPU hybrid composites.

Scanning electron microscopy (SEM)

Morphological investigations were performed on the untreated and treated SPF/GF/TPU hybrid composites with an SEM machine (HITACHI S-3400N, Hitachi, Ibaraki, Japan). The SEM instrument was used at an emission current of 58 μA, an acceleration voltage of 5.0 kV, and the working distance was set to 6.2 mm. Before the SEM analysis, samples were coated with gold (Hitachi High-Technologies GLOBAL, Kyoto, Japan).

RESULTS AND DISCUSSION

Tensile Properties

In this study, to evaluate the efficiency of fiber modification on sugar palm fiber, mechanical properties, such as tensile strength, were evaluated. As shown in Fig. 2, the tensile strength for untreated sugar palm fiber was 21.15 MPa, which was lower than the other treated fiber hybrid composites. When the SPF fiber was treated with 6 wt.% alkaline, 2 wt.% silane, and the combined 6 wt.% alkaline-2 wt.% silane, the tensile strength was increased to 21.90 MPa, 23.52 MPa, and 24.46 MPa, respectively. The maximum tensile strength for the treated hybrid composites was found for the TNSSP. The tensile strength of the TNSSP was improved by 16% compared to the UTSP hybrid composites. The tensile strength for the treated hybrid SPF/GF-reinforced TPU was higher than that of the untreated hybrid composites, which may have been due to good interfacial bonding between the treated fiber and TPU matrix, as shown in Fig. 3(d). The changes in the fiber surface after surface modification of the fiber improved the properties of the composites, which correlated with the effect of various treatments on the tensile properties of the composites. A similar mechanism for various alkaline-silane treatments providing tensile strength improvement was found by Farsi (2010).

As shown in Fig. 2, the tensile modulus the SPF/GF-reinforced TPU composites increased after the combined silane-alkaline treatment. The presence of treated sugar palm fiber reduced the polymer chain mobility, which led to increased stiffness in the hybrid composites (Ku *et al.* 2011). Moreover, the tensile modulus of the TSSP of SPF/GF/TPU composites was higher than both the UTSP and other treatments. This was mainly due to the strong interfacial bonding adhesion that exists between the SPF with GF and TPU matrix of hybrid composites. This is consistent with what has been observed for surface modification that was employed on other natural fibers (Lee *et al.* 2009; Oliveira and Marques 2014). Moreover, other researchers also found that the composites that employed alkaline-silane treatments showed improved compatibility with curaua fiber with PP due to the mixture of both treatments induces the mechanical interlocking between the fiber and matrix (Oliveira and Marques 2014).

Scanning Electron Microscopy

Figure 3 displays the SEM images after tensile testing of UTSP, TNSP, TSSP, and TNSSP treated hybrid SPF/GF-reinforced TPU composites. Observable differences were visible between the SEM figures of the aforementioned treatment samples (Fig. 3). More fibers were pulled out in the UTSP sample compared to others, leaving more holes, leading to poor fiber/matrix adhesion (Luo *et al.* 2016). Fragments and impurities that existed on the surface of UTSP (Fig. 3(a)) were removed *via* alkaline treatment. The surface of the fiber TNSP (Fig. 3(b)) was smoother than UTSP fiber, whereas the surface of TNSP showed nodes, and only a slight fibrillar structure was present (Fig. 3 (b)). The surfaces of the sugar palm fiber samples were covered with a TPU matrix and showed good interfacial bonding, as shown in Figs. 3(b), (c), and (d). This was due to fiber modification by the various treatments that were employed on the sugar palm fiber. The presence of more nodes and fibrils on the surface of the sugar palm fibers, as in Fig. 3(d), was a result of the 6 wt.% alkaline-2 wt.% silane treatment that yielded deeper indentations among the sugar palm fibers. The SEM observations of the treated SPF/GF-reinforced TPU hybrid composites were in agreement with the mechanical properties testing (Fig. 2), demonstrating that surface treatment improved the properties of the hybrid composites.

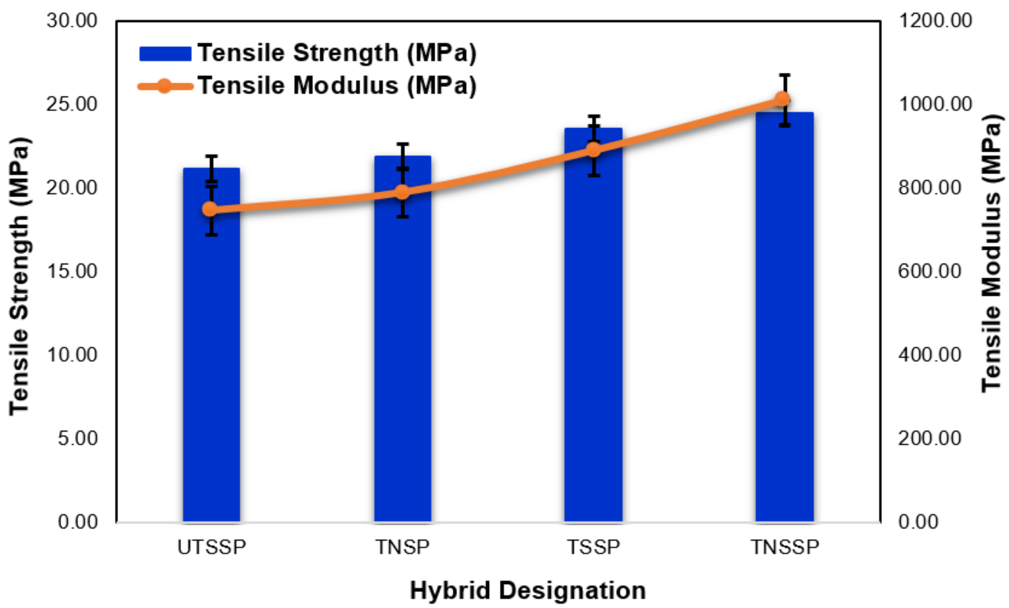


Fig. 2. Tensile properties of untreated and treated hybrid SP/G-reinforced TPU composites

Flexural Properties

Figure 4 shows the flexural properties of the untreated and treated hybrid SPF/GF-reinforced TPU hybrid composites. The flexural strength and modulus of the untreated and chemically-treated SPF hybrid composites depended on the type of fiber surface modification.

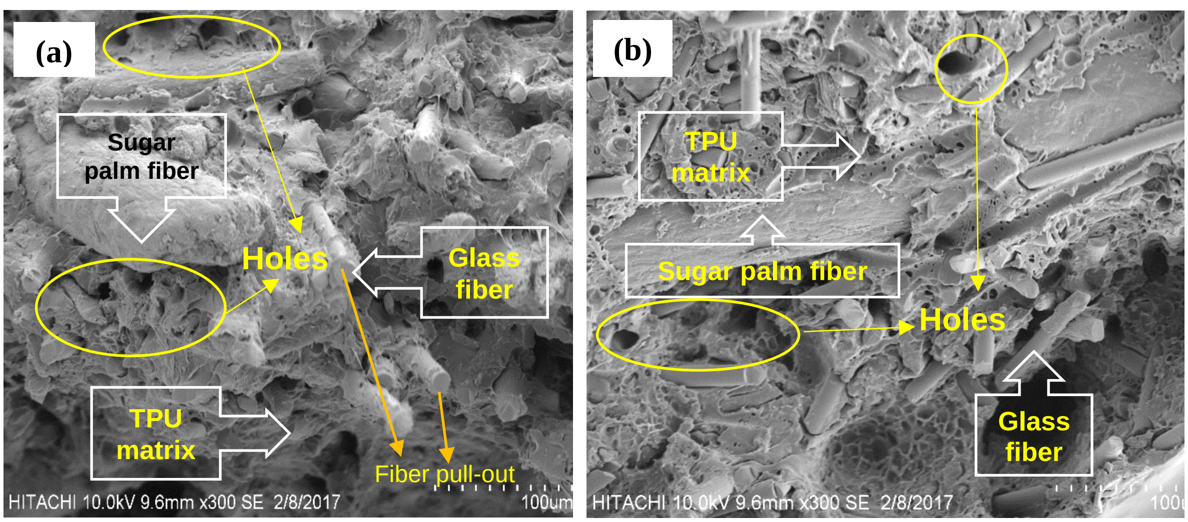
The trend of the flexural strength is shown in Fig. 4 and indicates that the combined TNSSP treatment provided good wetting to the GF-reinforced TPU composites. The incorporation of treated 30 wt.% of sugar palm fiber led to improved properties, especially in both flexural strength and modulus as compared to UTSP. The flexural strength of the TNSSP improved 39% compared to that of the UTSP hybrid composites. This may have been attributable to employing the 6 wt. % alkaline- 2 wt.%

silane treatment, which may reduce the cell wall thickening of the fiber that leads to the better adhesion between fiber and matrix as observed by earlier investigators (Lee *et al.* 2009). Other researchers also have highlighted that fiber treatment plays an important role in influencing the flexural properties (Li *et al.* 2007; Sathishkumar *et al.* 2013).

As shown in Fig.4, the flexural modulus of UTSP, TNSP, TSSP, and TNSSP were 266.70 MPa, 355.37 MPa, 395.63 MPa and 413.42 MPa. This corresponded to a 33.2%, 48.3%, and 55% increase in flexural modulus of TNSP, TSSP, and TNSSP as compared to UTSP, respectively.

This improvement in flexural modulus was attributed to the stress transfer from the low modulus of untreated SPF to the high modulus of the TNSSP. Moreover, the increase in flexural strength and modulus of TNSP was by 38% and 55% as compared than UTSP, which generates active groups or clean surfaces that form bonds with the silane molecules (Farsi 2010).

In fact, more chemical bonds formed among the cellulose hydroxyl groups on the silane molecules and the fiber surfaces compared to that of the untreated of fiber. This finding was in agreement with other research on hemp fiber using various alkaline/silane treatments that resulted in improved mechanical properties (Patel *et al.* 2010).



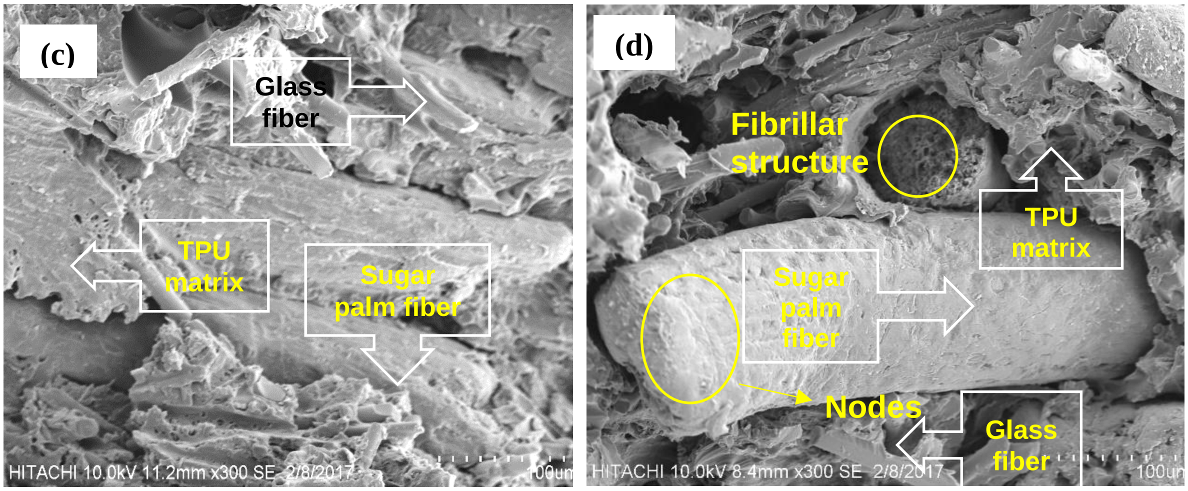


Fig. 3. SEM pictures of (a) UTSP, (b) TNSP), (c) TSSP), and (d) TNSSP

Impact Properties

The impact test results of untreated and treated SPF/GF-reinforced TPU hybrid composites are illustrated in Fig. 5. The energy absorption was determined using the Izod impact machine test (INSTRON CEAST 9050, Norwood, MA, USA). As shown in Fig. 5, the maximum impact strength was obtained for the TNSSP, followed by TSSP and TNSP compared to UTSP with glass fiber-reinforced TPU hybrid composites. The impact strength of the TNSSP improved 18% compared with the UTSP hybrid composites.

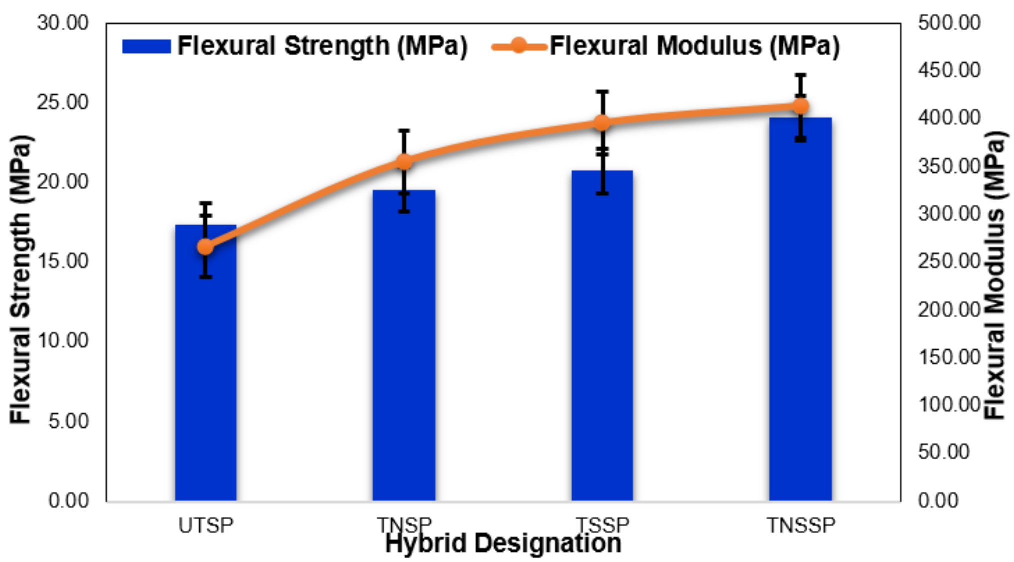


Fig. 4. Flexural properties of untreated and treated SPF/GF-reinforced TPU hybrid composites

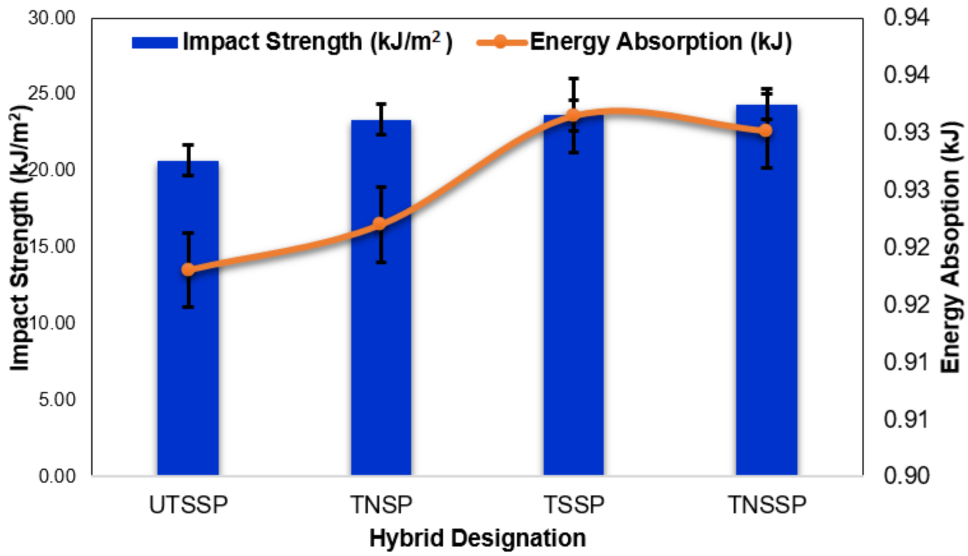


Fig. 5. Impact properties of untreated and treated hybrid SPF/GF-reinforced TPU composites

It was evident that the surface modifications improved the impact strength of hybrid composites. The alkaline treatment provided a 12% improvement in the impact strength compared to that of the UTSSP. Moreover, a noticeable increase in impact strength was observed with the combined alkaline-silane (TNSSP) treated SPF/GF-reinforced TPU composites. The incremental impact strength was due to the generation of moisture resistance and surface roughness by expelling hemicellulose, lignin, pectin, wax, and *etc*. from the alkaline treatment (Li *et al.* 2007). Furthermore, the alkaline pretreatment followed by silane treatment improved the nucleation density of the cellulose part of the fiber, yielding a trans-crystalline interphase region of small crystals. There was improved adhesion bonding of fiber and matrix in alkaline-silane fiber treated relative to untreated fiber composites, which allows the polymer matrix to effectively surround and adhere to the fibers (Huda *et al.* 2008; Sajna *et al.* 2014).

Fourier Transform Infrared Spectroscopy(FTIR)

FTIR spectra of the UTSP, TNSP, TSSP, and TNSSP are presented in Fig. 6. The wide band at 3330 cm^{-1} at the UTSP was attributed to the OH^- groups present in the cellulose and hemicellulose of the fiber. After post treatment of sugar palm fiber, the OH groups were substituted as –ONa groups in 6 wt.% alkaline treatment and –O-Si-R in 2 wt.% silane modification. The decreased intensity of –OH band was decreased when the sugar palm was treated as in TNSSP at 3324 cm^{-1} as compared to UTSP. The stretching band at 1740 cm^{-1} is indicative of the lignin in natural fiber. The removed band at this frequency in TNSP, TSSP, and TNSSP showed there were changes after fiber treatment. These findings are similar to other works that showed chemical changes after modification of fiber (Sareena *et al.* 2012; Shaniba *et al.* 2017). For TSSP and TNSSP, there were stretching frequencies at 1590, 1172, and 1098 cm^{-1}, as shown in Fig. 6. The band at 1590 and 1098 cm^{-1} existed at 2 wt.% silane and combined 6 wt.% alkaline-2 wt.% silane showed C=C bonding of silane bonding. The characteristics absorption peak at 1172 cm^{-1} is due the presence of Si-OEt, which confirms the reaction of 3-aminopropyltriethoxysilane with silane fiber treated (Shaniba *et al.* 2017).

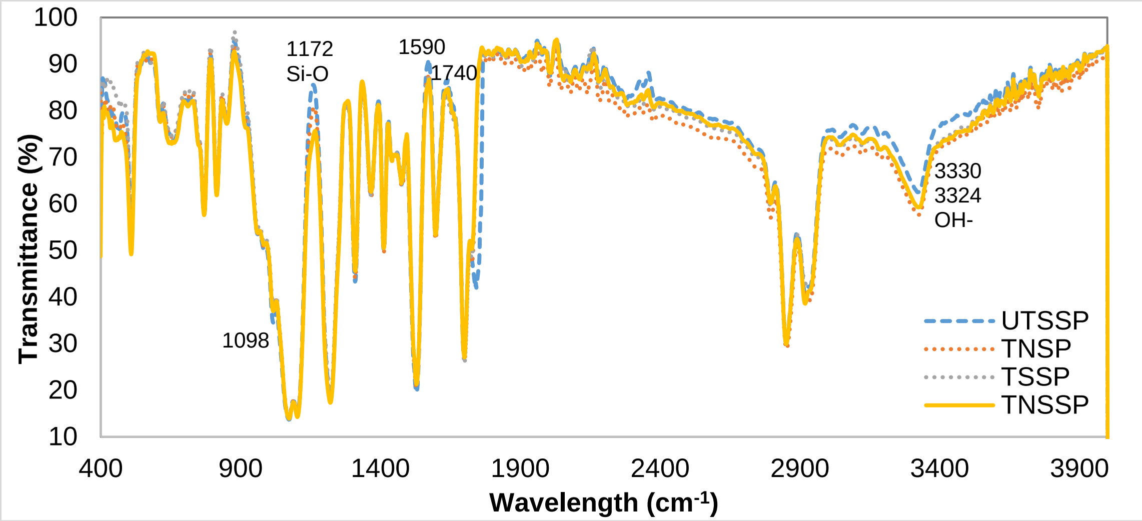


Fig. 6. FITR spectra for UTSP, TNSP, TSSP, and TNSSP

CONCLUSIONS

The effect of various fiber modifications on the mechanical properties of sugar palm fiber glass fiber (SPF/GF)-reinforced thermoplastic polyurethane (TPU) hybrid composites was investigated.

1. The results clearly indicated that treating sugar palm fiber improved its mechanical properties. Regarding the mechanical properties of the hybrid composites, it was observed that the combined 6 wt.% alkaline-2 wt.% silane treatment (TNSSP) was the most effective combination considering the improved tensile, flexural, and impact properties of SPF/GF-reinforced TPU hybrid composites.
2. These results were also supported by morphological studies by SEM and FTIR. The SEM morphology of the treated SPF/GF-reinforced TPU hybrid composites was better than that of the untreated fiber. This was because 6 wt.% alkaline (TNSP), 2 wt.% silane (TSSP), and combined 6 wt.% alkaline-2 wt.% silane (TNSSP) treatments enhanced the compatibility between the sugar palm fiber with glass and TPU matrix.

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