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Experimental Investigation of Effect of Fiber Length on Mechanical, Wear, and Morphological Behavior of Silane-Treated Pineapple Leaf Fiber Reinforced Polymer Composites

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Abstract: The development of the best properties in polyester composite from pineapple leaf fiber (PALF) as a reinforcing material is a subject of interest. The properties of PALF are reliant upon fiber length, wherein technical difficulties in production of long fibers and processing for better characteristics in polyester composites possess inherent challenges. The PALFs are subjected to silane treatment for altering fiber properties. This research attempts to analyze the impact of silane-treated PALF with varying fiber lengths (5, 10, 15, 20, and 25 mm) on the performance of natural fiber composites (NFC) properties. Open mold and hand lay-up techniques were employed to develop the polyester composites. The prepared PALF-based polyester composites were examined for different properties (impact, flexural, tensile strength, and wear rate). Coefficient of friction and wear studies are performed on the prepared composites subjected to different loads (10, 20, and 30 N) via a pin on disc test rig. Polymer composite fracture surfaces were analyzed to observe the interfacial bonding between fibers and matrix via scanning electron microscopy (SEM). SEM results showed that the application of silane treatment resulted in better surface topography (fiber length of 5–10 mm showed smooth surface resulted in crack proliferation possessing low fracture toughness of 15–32 MPa; whereas a 15–20 mm fiber length resulted in better fiber–matrix bonding, improving the fracture toughness from 42–55 MPa) as a result of change in chemical structure in PALF. The 20 mm length of PALF resulted in better properties (flexural, tensile, impact, and wear resistance) which are attributed to fiber–matrix interfacial bonding. These properties ensure the developed polymer composites can be applied to walls, building insulation, and artificial ceilings.

Keywords: pineapple leaf fiber; polyester resin; hand layup method; mechanical properties; wear properties; SEM

1. Introduction

NFCs are gaining worldwide attention across various industries due to their supremacy in biocompatible properties and widespread applications over synthetic composites [1,2]. Natural fibers (NFs) decompose naturally in the environment, and also ensure distinguished benefits such as non-toxicity, low cost, reusability and recyclability [3,4]. NFs are derived from plants (cellulose, hemicellulose and lignin-derived stalk, leaf, wood, fruit, bast, grass), animals (wool or hair, silk, feathers, etc.), and minerals (asbestos, ceramics, and metal). Based on the use, the fibers are blended with thermosetting and thermoplastic [5–7]. Note that the fibers derived from minerals are avoided due to serious health issues [8]. The plant fibers resulted in better stiffness and strength than that obtained from animal fibers [9]. Therefore, an extensive study is required to prepare plant-based composites with the help of matrix material—either the thermoplastic (vinyl ester, epoxy, and polyester) or thermosetting matrix (polypropylene and polyethylene resins) [10,11].

Polyester is one of the world's most commonly used polymers which offers excellent mechanical properties (good fatigue resistance, low friction coefficient, and better impact strength and flexural strength) and is therefore found in distinguished applications (textiles, packing and labeling, components for the automotive industry) [12,13]. High-strength natural fiber reinforcing the polymer matrix to develop a composite material is referred to as 'natural fiber polymer matrix composites' (PMCs). PMCs are generally lightweight, wherein the fibers provide enough rigidity and strength [14]. The composite's mechanical and physical properties are reliant upon fiber properties, preparation of fibers, mixing ratio (reinforcement and matrix material), fiber orientation and selection, fiber length, orientation and weight ratio, degree of interfacial bonding, and the intermingling of the fibers and fabrication process [15–17]. Fiber geometry play a vital role in preparing composites wherein impact toughness is the dominant factor contributing towards aerospace applications [17]. The diverse nature of natural-fiber-reinforced polymer composites demonstrates a vital role in locomotive applications (aerospace, automotive, and construction industries) [18]. Note that the selection of appropriate parameters which influence the properties of polymer composites are of industrial relevance.

In India, two popular agricultural wastes—pineapple leaf and banana fiber—are promising alternate materials for synthetic fiber [19,20]. Banana fibers are extensively used in industries for distinguished applications (shipment cables, apparel clothing, cordage, etc.) due to intrinsic properties such as biodegradability [21,22]. Banana fibers possess lower tensile strength than that obtained from pineapple leaf fiber due to lower cellulose content (48–50% for banana, and 70–82% for pineapple) [23]. To date, pineapple leaves are less utilized for fiber production, wherein their leaves contain cellulose properties possessing the best mechanical properties with biocompatibility [24–26]. Cellulose offers better strength, stability, stiffness, and strength for fibers, wherein reinforcement in composites offers biocompatibility with enough strength and properties that could result in energy efficiency [26]. Elongation at the composite splits is prolonged when the pineapple leaf fiber is combined with particulate filler [27]. Techniques such as increasing rubber chewing time, manipulating dispersal of particulate filler, and degree of crosslinking could alter the stress–strain curves correspond to reinforcing PALF rubbers [28,29]. However, the above methods commonly resulted in fiber pull-out failure. Note that these properties are treated as a benchmark, wherein attempts are made to reinforce rubbers with aramid fiber [30,31]. The resulting composites demonstrated outstanding mechanical properties without any sign of fiber pull-out. PALF showed the strengthening agent would not compete with the rubber composites containing aramid fiber [28,32]. To ensure better results, there are better matrix-reinforcement (fibers) interfacial adhesion characteristics and the contrast is observable under significant strain or deformation [33]. The composite modulus is defined by both the modulus of fiber and its aspect ratio, as with short fiber reinforced composite [34,35]. For PALF, the fibers are prepared to the highest aspect ratio, and that could compensate for the inferior modulus [36,37]. PALF demonstrated lower modulus than reinforced aramid fiber rubbers. Thereby, fiber bundles are divided into simple micron size and a shape that ensures the best impact while keeping the length the same [38,39]. To alter the external surfaces of the fiber, silane (inorganic compound) treatment is to be carried out [40,41]. The silane treatment carried out both on kenaf fiber and pineapple leaf fiber resulted in better tensile strengths than untreated fibers in composites [42]. The silane-treated fibers exhibited better strength identical to those obtained for alkali-treated fiber in different experimental conditions [43]. The properties of PALF composites subjected to silane treatment are affected by the percent of fiber volume, fiber length, surface treatment, fiber–matrix ratio, fiber weight, etc. [19,44,45]. Good flexural, impact, and tensile properties were produced by PALF composites with 30 wt % [10]. It was observed that many surface treatment techniques (NaClO2, acetylation, KOH, NaOH, isocyanate and binding agents) progress towards attraction between fiber and matrix that could produce enhanced properties (i.e., mechanical) in composites [46]. In addition, different engineering methods—such as injection molding, vacuum-assisted resin transfer molding, hand layup technique, and compression molds—are commonly applied in industrial practice due to their simplicity and ensure strong properties [47,48]. The following key observations are drawn from the above literature: (a) PALF has greater potential for yielding better properties and also ensures sustainability by utilizing agricultural waste for fabricating parts. (b) Silane treatment modifies external surfaces of fibers that could be capable of exhibiting better properties in composites. (c) The PALF properties are often reliant on fiber length, wherein technical difficulties in production of long fibers and processing for better characteristics in polyester composites possess inherent challenges. (d) The effect of fiber length with silane treatment has not been investigated much in literature. Therefore, the present work is focused on the application of silane-treated pineapple leaf fiber with different fiber lengths were investigated on morphology, and properties (wear, tensile, flexural, impact, and so on). The optimal fiber length for better composite properties is recommended for ease of production of composites and practical utility in industries.

The research work is as follows: Section 2 describes the PALF material, properties, extraction, silane treatment, and preparation of composite specimen. Section 3 describes the characterization (tensile, flexural, impact, and wear test) of composite specimens. Section 4 describes the mechanical (tensile, flexural, impact, and wear test) and fractography characterization of composite specimens. Section 5 offers concluding remarks on the obtained results.

2. Materials and Methods

2.1. PALF Material

India stands in the fifth global position for the production of pineapple [49]. Pineapple grows primarily in the northeast (Assam and Tripura) and southern parts of the Karnataka province of India [50]. The size of the plants and fruits among different parts of India. After plucking the pineapple fruits, the plants are treated as waste. Thereby, plant fibers can be extracted from leaves (usually about 1 m long) which are treated as an agricultural by-product. The fiber extracted from pineapple leaf possesses approximately similar mechanical strength to jute fiber [51]. Pineapple fruits yield between 2 and 3 years, and after plucking fruits the plant leaves are collected from Bindiganavile, Karnataka, India. The fibers are extracted from pineapple leaf and that same fiber used to fabricate polyester composites. NFs are extracted via three methods, namely chemical, manual, and mechanical [52,53]. A mechanical method of extracting fibers is often advantageous in obtaining high-quality fibers in a very short interval of time [54]. From the extracted pineapple leaf sheath, the fiber bundles are extracted from the plant's pseudostem. The retained water content in the fibers is removed via a sunlight drying process. The polyester resin and PALF physical and mechanical characteristic details are presented in the literature [55].

2.2. Preparation of PALF with Silane Treatment

The polymer matrix possesses hydrophobic features, whereas natural fibers as hydrophilic reinforcement material raises problems in polymer composites. To limit said disadvantages and minimize hydrophilic characteristics, the fibers are treated with different chemicals such as sodium hydroxide [56], acetic acid [57], silane [56], benzoyl peroxide [58], potassium permanganate [59], stearic acid [60], cellulose powder [61], polymer coating [62], bleaching [63], and so on. Silane-treated woven natural fiber possesses greater resistance to penetration of high impact, breaking strength, and toughness which are useful for bulletproof composites [56]. Silane is a multifunctional molecule, wherein triethoxy silane is employed to modify the fiber surface. The triethoxy silane is mixed with an ethanol–water blend to prepare a solution. The fiber solution ratio is maintained at 20:1. The solution is maintained at a continuous pH value equal to 4 for a predetermined time. Three separate time treatment durations—2, 4, and 6 h—are maintained wherein the fibers are submerged in a solution. The fibers are later washed with water to diffuse the pH level and then oven-dried (to remove excess water or humidity) for 36 h at 70 °C. After ensuring the fiber extraction process, the PALF was later treated with silane possessing a molecular weight of 193.25 g/mol from Vikram Resin and Polymers Ltd. Bangalore, India. Silane coupling agents reduce the cellulose hydroxyl groups in fiber–matrix interface and ensure the PALF fibers adhere to the matrix material that could stabilize the composites. The impurities (white patches) that can be seen in the untreated PALFs are reduced or absent with the silane-treated fibers (refer to Figure 1). The silane-treated fibers might result in better interfacial adhesion in composites.

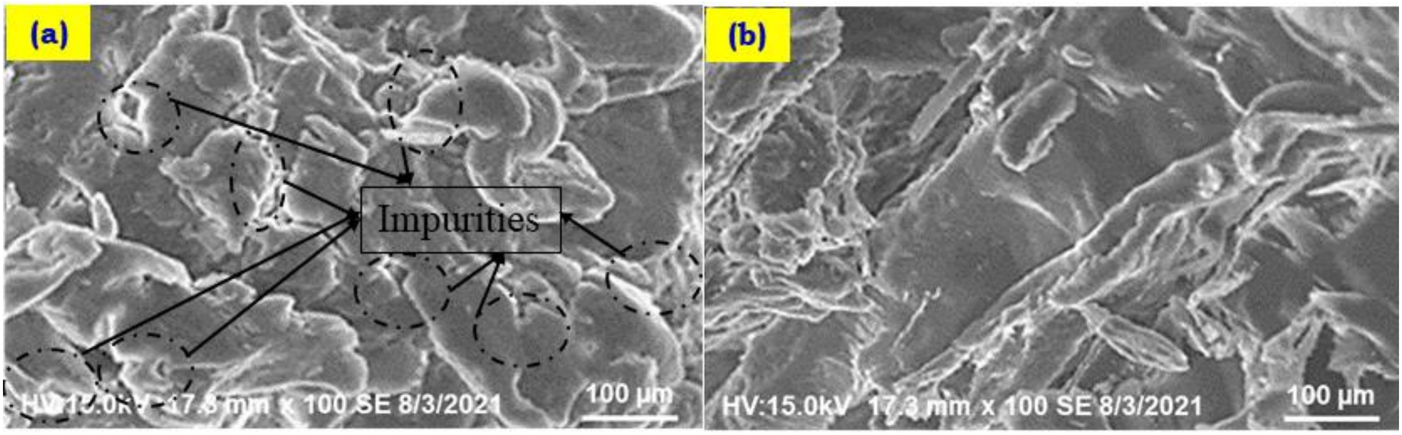

Figure 1. SEM micrograph of pineapple leaf fiber (**a**) without silane treatment and (**b**) with silane treatment.

2.3. Preparation of Composite Specimen

Hand lay-up is proven as a simple and cost-effective technique to fabricate composites possessing the combination of higher fiber volume with longer fiber size [64]. PALF is a reinforcement and polyester resin employed as a matrix material used to prepare polyester composites via hand lay-up technique. Experiments are conducted and analyzed with different fiber lengths (5, 10, 15, 20, and 25 mm) on properties (strengths: impact, tensile, and flexural, and wear) of polyester composites. The natural fiber composites are prepared using a metal die and three replication experiments are performed to increase the efficiency of measurements. The die for preparing the polyester composite specimens is presented as shown in Figure 2.

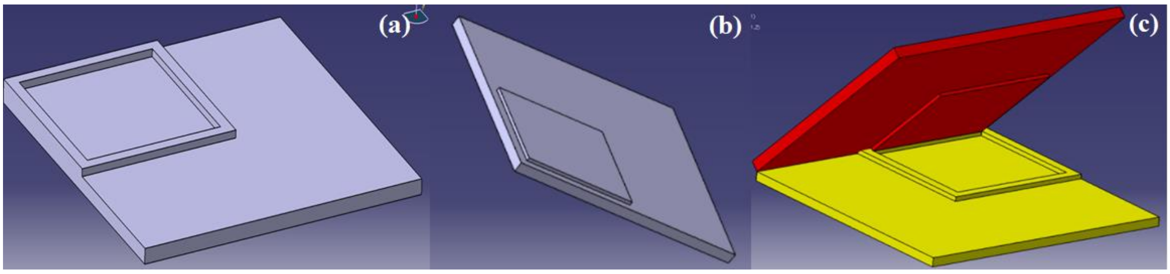

Figure 2. (**a**) Lower mold half, (**b**) upper mold half, and (**c**) open mold.

3. Characterization of Composites

PALF is inexpensive, biocompatibility ensuring better properties (high strength and rigidity), and therefore being used in fabricating parts in various industries—such as agricultural industry, thermal insulation, automotive, medical and biomedical, building and non-structural applications [65–67]. Such applications are reliant upon properties of composite materials [65]. Mechanical strength and toughness are important properties to widen the applications. The methodology employed to prepare pineapple leaf fiber composites and fabrication of composites according to test sample sizes (tensile, flexural, and impact) is presented in Figure 3.

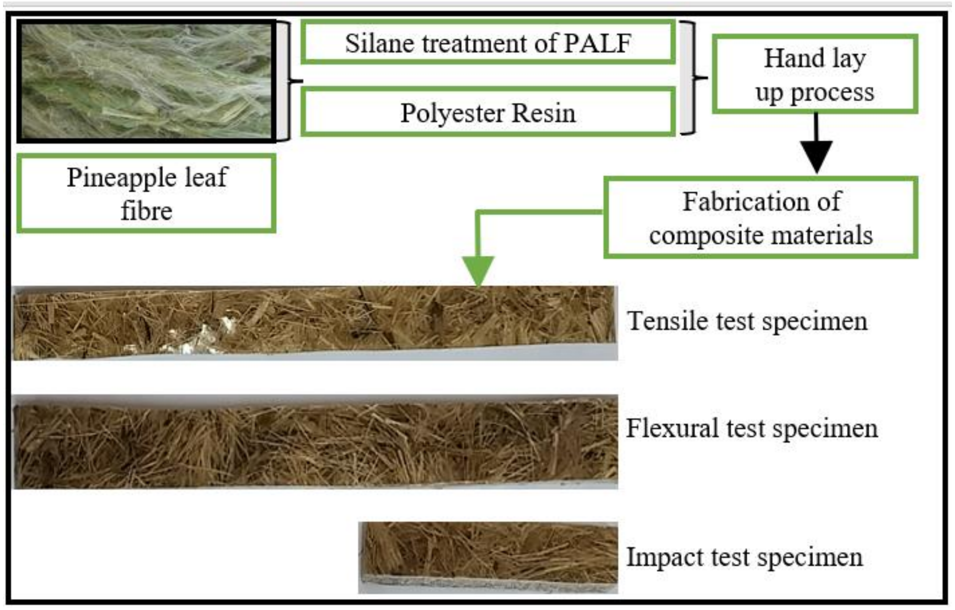

Figure 3. Methodology employed to prepare pineapple leaf fiber composites and test samples.

Therefore, the present work investigated the following properties.

3.1. Tensile Test

The samples are prepared to dimensions of 120 × 20 × 3 mm, which resembles the ASTM D3039 [68]. In each replicate experimental condition, two composite test samples are prepared for each fiber length of a sample. The average values corresponding to six test specimens were examined via universal tensile testing equipment are used for the analysis of the influence of fiber length on tensile strength characteristics. The dimensions of the samples are presented in Figure 4.

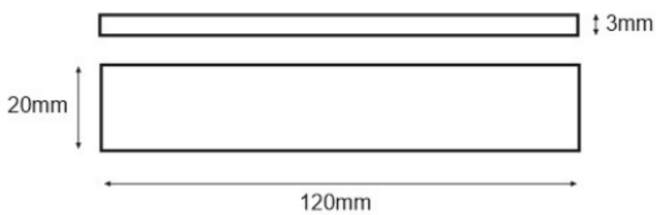

Figure 4. Tensile test specimen.

3.2. Flexural Test

Flexural tests are conducted for the samples prepared to dimensions of 125 × 12.7 × 3 mm, which resemble the ASTM D790 [69]. The average values of a total of six specimens are examined (composed of two test samples in each of three replicates) for each fiber length. The tests are carried out via universal testing equipment possessing specimen with a length and crosshead maintained equal to 65 mm and 12 mm/min, respectively. In three-point bending experiments the outer rollers were 65 mm apart and examined at 0.3 mm/min [70]. The digital gauge helps to record the specimen deflection and flexural parameters (flexural modulus, stiffness, and strength). Figure 5 presents the sample dimensions of the flexural test specimen.

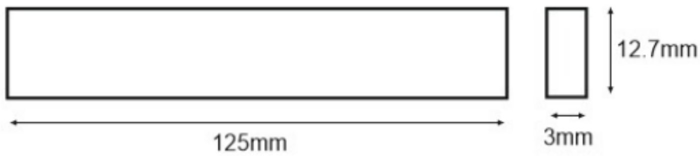

Figure 5. Flexural test specimen.

3.3. Impact Test

The toughness of the polyester composite samples was evaluated using impact tests. The polyester composite samples measure 70 × 15 × 8 mm which resembles the ASTM D256 standard [71]. For each fiber length, the average values of the six impact measurements are recorded and the same is used for performing analysis. The prepared test samples are inserted into the equipment and allow the pendulum to swing until it fractures or breaks. The maximum energy absorbed to fracture the samples is recorded which determines the impact energy and specimen behavior at higher deformation loads. Figure 6 shows the dimensions pertaining to the impact test specimen.

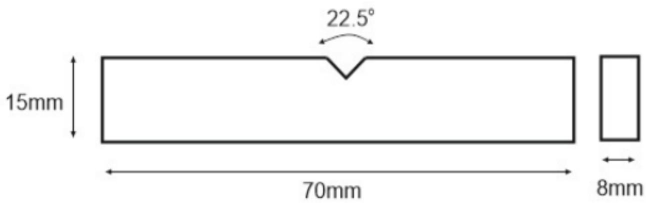

Figure 6. Dimension of impact test specimen.

3.4. Wear Test

Pin-on-disc wear testing equipment is employed to examine the wear properties for specimens prepared to a dimension (pin size of 1 cm diameter and length of 3 cm) of PALF composite samples. ASTM G-99 standard is applied to test the polymeric materials. A total of six wear test samples were prepared and the average values pertaining to each fiber length were used for performing analysis. The weight loss corresponds to specimen weights before and after wear examination were recorded via digital weighing balance.

3.5. SEM Examination

The morphology characteristics of fractured surfaces of tensile and flexural samples were investigated via SEM (JEOLJSM 5800). The samples were sliced into smaller pieces and were sputter-coated using an ultra-thin coating of electrically conducting metal to enhance the conductivity of the specimens.

4. Results and Discussion

4.1. Tensile Test

The effects of silane-treated PALF polyester composites subjected to tensile strength examinations are presented in Figure 7. Tensile properties (Young's modulus, tensile strength, and elongation at break) tend to increase with the increased length of fibers up to 20 mm. This trend occurs due to lower fiber lengths possibly being insufficient to spread uniformly in a polyester matrix, which could result in lesser stress transfer from the matrix to the fibers. This depicts the individual length of fibers in the polyester composite matrix that alter the tensile properties. Similar results are reported in the literature [38]. In general, PALF contains less cellulosic content (i.e., vary with fiber length), making the fiber less flexible or more elastic. The SEM morphology on tensile fractured surfaces is evident that voids, cracks, and fiber pullout resulting in debonding matrix and fiber adhered interface (refer to Figure 7). Higher tensile properties (Young's modulus, strength, and elongation at break) are observed for 20 mm of fiber length, wherein there is a strong existence between matrix and fiber. The reduction in tensile strength is observed at 25 mm of fiber length. This occurs probably due to the difficulty for the resin to penetrate between the spaces corresponding to fiber and resin, resulting in poor wetting characteristics and therefore reducing the efficacy of stress transfer of the matrix–resin interface [72]. Increased fiber loading in polyester composites resulted in better tensile modulus and therefore displayed better elasticity for the fiber length of 20 mm (refer to Figure 7b,c).

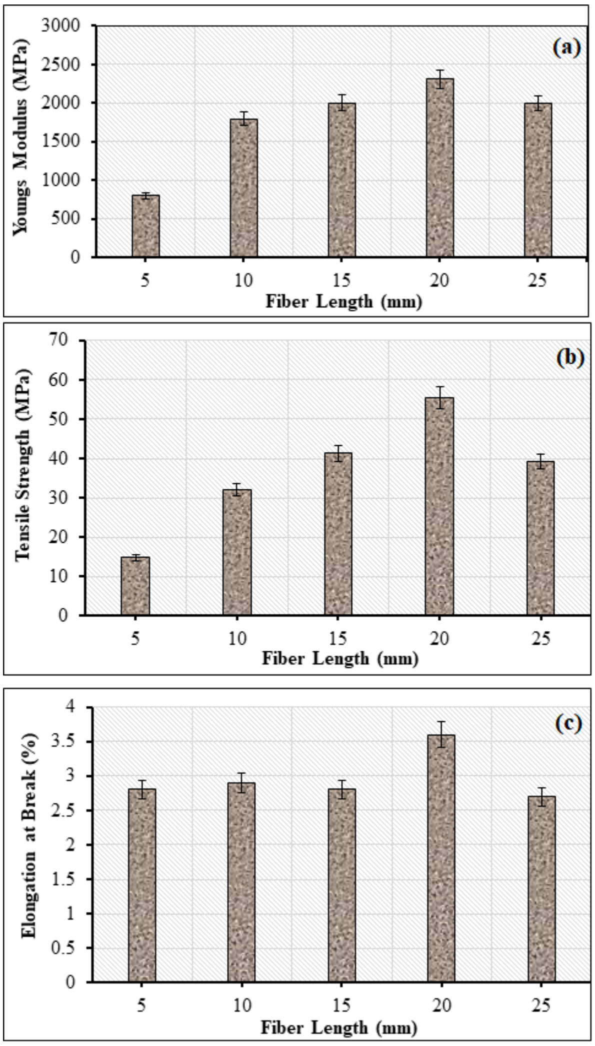

Figure 7. Tensile tests results for (**a**) Young's modulus vs. fiber length, (**b**) tensile strength vs. fiber length, (**c**) percentage of elongation vs. fiber length.

4.2. Flexural Test

The performance of flexural properties of 5, 10, 15, 20, and 25 mm of PALF fiber length in polyester composites are presented in Figure 8. Short fiber length (i.e., 5 mm) in composite samples resulted in higher flexural strength which primary control the microcrack propagation [73]. Furthermore, short fibers also increase the number of fibers which bind closely in the composite samples and therefore increase the flexural strength [74]. Flexural strength corresponding to 10–15 mm fiber length in the polyester composites is lesser compared that of 20 mm fiber length specimens. The reduction in flexural strength and modulus are attributed to short fiber polyester composites experiencing cluster formation and uneven mixing between the fibers and matrix. The uneven distribution of fiber in the composite samples is incapable of sustaining or passing stress from the resin matrix. The highest flexural strength, observed at 20 mm fiber length, is attributed to better interfacial bonding at the matrix–resin interface. Similar results are observed with sisal fiber polymer composites [75]. The use of polypropylene matrix improves properties (flexural modulus and flexural stress) with fiber length, wherein problems encountered related to fiber-to-fiber repulsion and dispersion are treated insignificant, which improve composite properties as a result of better interfacial relationship [65]. Improved adhesion characteristics (between fiber and polyester resin matrix) could alter the fiber surface modification with silane treatment. Silane-treated fibers are free from impurities, resulting in improved matrix–fiber adhesion characteristics and causing fiber failure that could sustain higher loads. The flexural modulus with a 20 mm fiber length is found to be 45 GPa compared to another fiber length. Short fiber possessing dimensions between 5 and 20 mm of fiber length resulted in flexural strength variation between 12 and 45 GPa. A similar trend was observed for specific flexural stiffness, wherein the length of fiber kept at 20 mm resulted in 0.3×10^6.

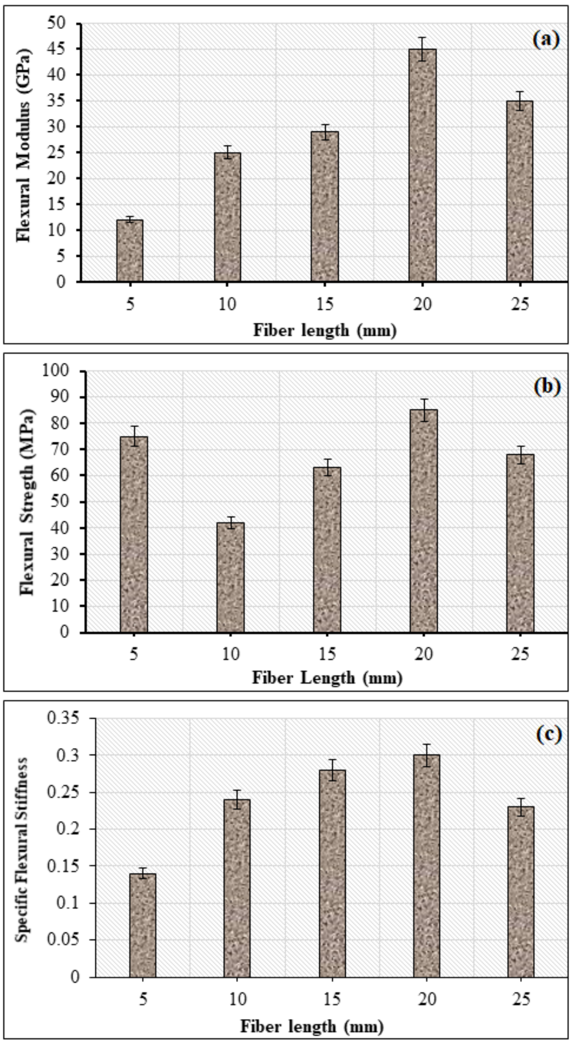

Figure 8. Flexural tests results for (**a**) flexural modulus vs. fiber length, (**b**) flexural strength vs. fiber length, (**c**) specific flexural stiffness vs. fiber length.

4.3. Impact Test

Figure 9 depicts the performance of impact strength corresponding to different fiber lengths of PALF-reinforced polyester composites. Higher impact strength (i.e., 27.2 KJ/m^2)

was observed corresponding to the fiber length of 25 mm than that of 5–20 mm fiber lengths—i.e., 18.4 to 23.9 KJ/m^2. This occurs due to the long length of the fibers ensuring significant interfacial adhesive bonding at the fiber–matrix interface in composites. Note that the interfacial fiber–matrix closeness in fiber-reinforced polymer composites plays a vital role in polyester composites [76]. The fiber in the composite matrix transmits the stress to reinforce absorption fibers, but if the closeness between fiber and matrix is weak, the transfer will not be efficient. The strong interfacial closeness of the fiber–matrix interface enables improvements of the energy preoccupation throughout the impact loading. Longer fiber length requires more energy to pull the fibers out than short fibers during the fracture of specimens [77]. Furthermore, short fibers possess more fiber ends in the composite specimens which act as a stress concentration point and therefore reduced strengths are observed among 5–15 mm fiber lengths [78]. The probable reasons for lower energy absorption through impact are fiber pull out, debonding, and fracturing of the fibers. In addition, strain energy is due to the debonding of the fiber and the fracturing of the fiber is relative to the debonding length. The composites reinforced by the PALF fiber displayed very strong impact strength possessions, which can be attributed to the hollow in the heart of pineapple leaf fibers that helps withstand impact and preserve the composite structure, even preventing fiber debonding and fracture. Figure 9 shows the impact strength vs. fiber length.

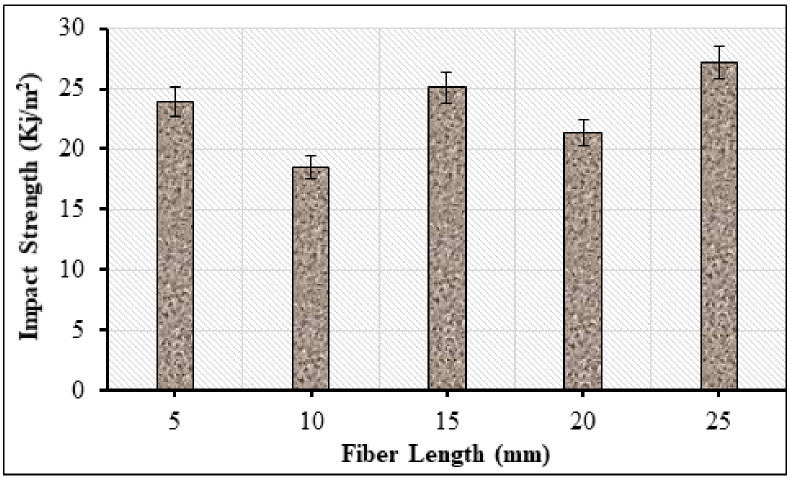

Figure 9. Impact strength vs. fiber length.

4.4. Wear Test

The pin-on-disc wear test rig equipment was used wherein PALF composite samples rub against a hardened steel disc with limited roughness. The composite sample is placed on the disc, with four screw fastenings vertical to the spinning disc with the aid of a sample holder and screws, and a lever is attached to the load. Figure 10 shows the fiber length variation with reference to the coefficient of friction. The coefficient of friction decreases from 0.8 to 0.76 for fiber lengths of 5, 10, 15, and 25 mm (refer to Figure 10). The lower values of the coefficient of friction correspond to a fiber length of 20 mm with a value equal to 0.74. Increase in fiber length results in higher wear resistance (less wear rate) in composite samples (refer to Figure 10b). This occurs because the fibers offer greater resistance to wear than matrix and therefore the wear rate is controlled by fibers. The function of matrix is to tightly hold the fibers, where composites are subjected to mechanical and thermal stresses [79]. The coefficient of friction was highly correlated with the wear rate of composite samples (except for fiber length of 25 mm). This occurs because wear debris produced during sliding might become disoriented and pose difficulty for ease of pin movement which finally increases the coefficient of friction [79,80].

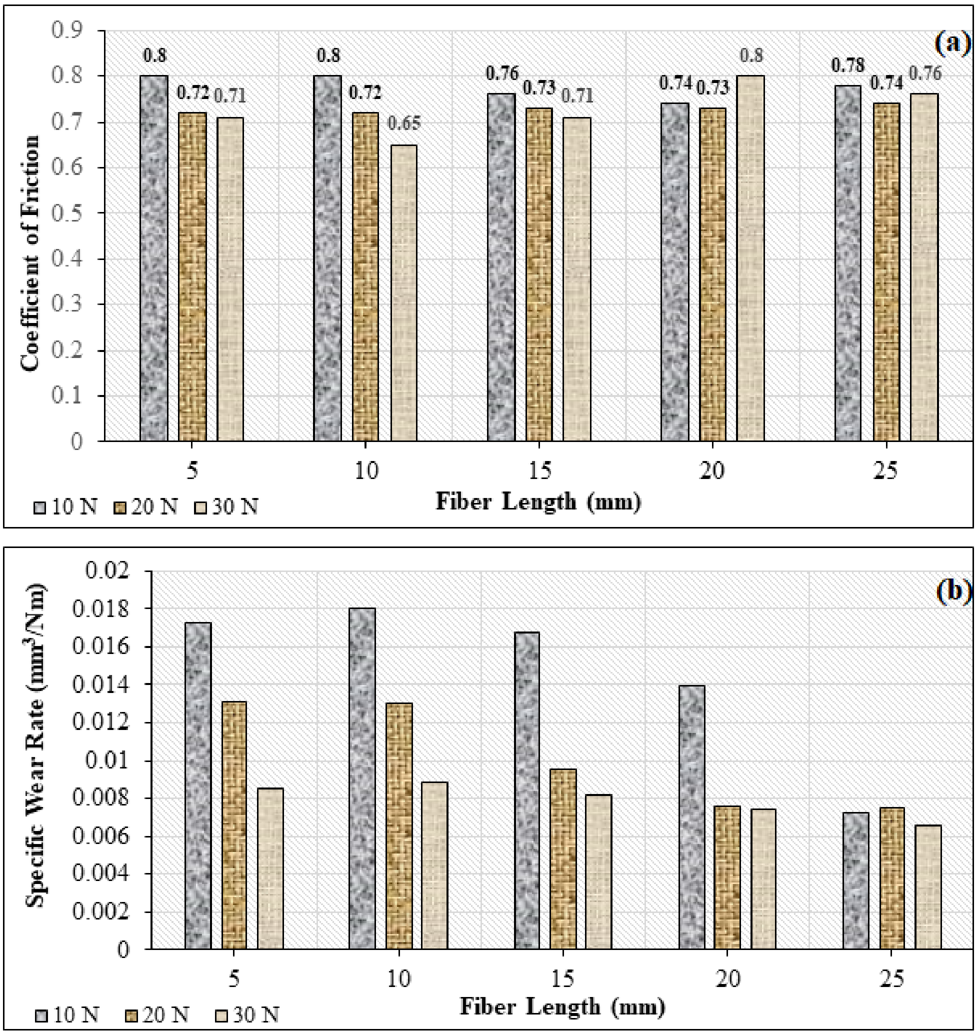

Figure 10. Fiber Length vs. (**a**) coefficient of friction (**b**) specific wear rate (mm^3/Nm).

4.5. SEM Examination on Fractography Analysis

A significant difference in the fractured surfaces of different fiber length specimens is observed at microscopic analysis. PALF fiber possessing 10 mm exhibits an entirely smooth surface that demonstrates rapid crack proliferation and low fracture toughness (refer to Figure 11a). It was also noticed that, compared to the long fiber length specimens, the short fibers exhibited rougher surfaces. Mechanically, the short fiber length specimens obstruct and slow down the dissemination of flaws. A narrow surface shape is shown by the short-fiber-length specimen broken surface. A good indication of improved fracture toughness is provided by the narrow surface form. SEM morphologies were also exposed, and the addition of polyester could alter the crack formation mechanism due to better closeness and crosslinking effect between PALF and polyester (refer to Figure 11b,c). PALF can weave into the polyester chains and eventually generate robust barriers to stop crack propagation.

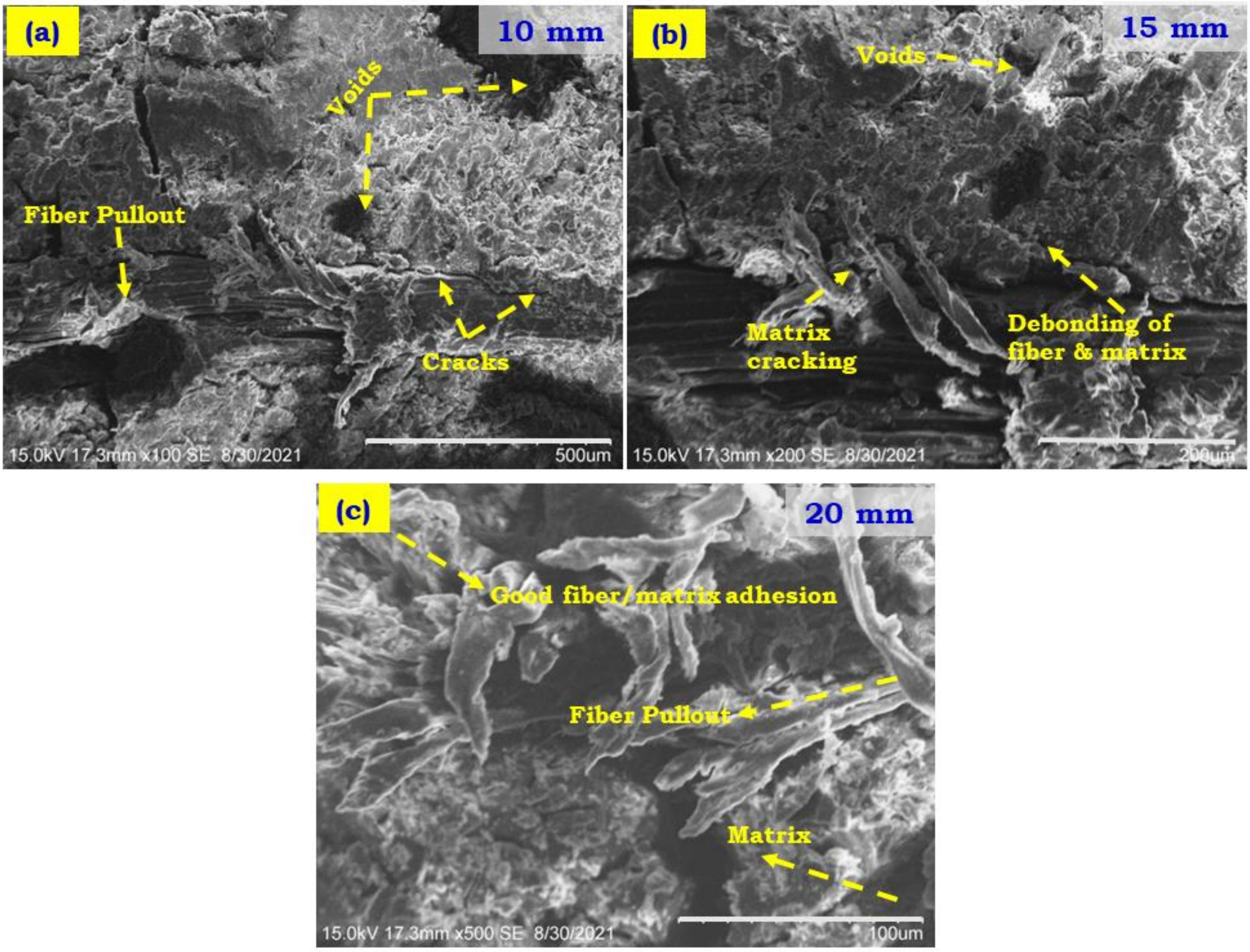

Figure 11. SEM morphologies tensile fractured surfaces of fiber length possessing 10, 15, and 20 mm.

Figure 12a–c explains the multi-cellular nature corresponding to silane-treated PALF. The silane-treated pineapple leaf fiber was stacked in a parallel fashion in a joined fiber bundle which can be seen in fracture morphology in the flexural specimen (refer to Figure 12a–c). During the fracture phase, the PALF was pulled out of the polymer matrix, resulting in huge voids near to the scale bar. This clearly indicates that the interfacial adhesion between matrix and reinforcing PALF was weak, wherein the silane-treated PALF composites lower impact and tensile strength. Note that fiber agglomeration takes place for the fiber length of 10 mm, resulting in weak bonding between the fiber and matrix, hence failure occurs before taking the maximum load. The results show that, at 20 mm fiber length, the maximum flexural strength was observed. This could be due to better fiber–matrix adhesion than the other samples studied. The active load transfer is observed wherein better bonding between the fracture starts and fiber pullout is minimum. The optimal flexural strength was observed for 20 mm fiber length.

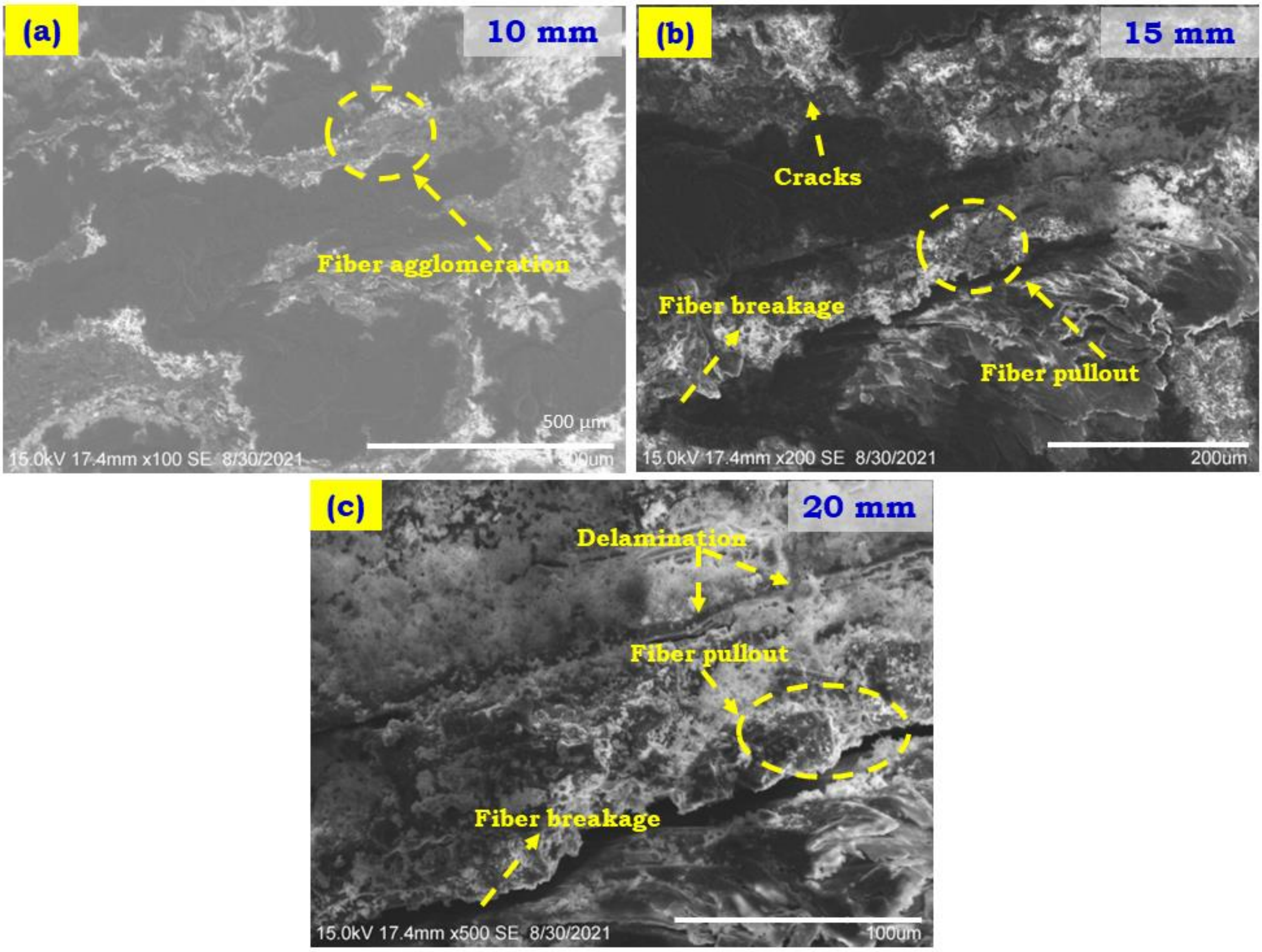

Figure 12. SEM morphologies of flexural fractured samples of 10, 15, and 20 mm PALF fiber length.

5. Conclusions

The PALF polyester composites are often reliant on fiber length, wherein production of long fibers that could process better properties in polyester composites possess inherent challenges. Furthermore, PALF polyester composites were prepared to know the influence of fiber length on the properties (impact, tensile, flexural, and wear rate) and the findings are presented as discussed below:

- The hand lay-up molding technique was applied to fabricate polyester PALF composites, bBefore fabricating silane treatment to modify the surface of fibers was carried out. The silane-treated fibers possess potential advantages to improve the mechanical strength of composites by developing roughness on the fiber surface wherein there exists a greater probability to improve the matrix–fiber adhesion.
- The 20 mm fiber length of PALF exhibited a maximum tensile strength of 57.4 MPa, flexural strength of 45 GPa, and low wear resistance properties. The reason for decreased properties after 20 mm fiber length in polyester PALF composites is attributed to the closeness of the fiber–matrix interface and internal friction caused by the fibers. The agglomeration of fibers in the polyester matrix limits the flexibility of polymer chains wherein there is an inferior plasticity which reduces the damping characteristics. SEM morphology of the tested fractured surface of PALF composite samples revealed a perfectly smooth surface, indicating rapid crack propagation and low fracture toughness.

- The fiber length possessing 20 mm resulted in low coefficient of friction equal to 0.74 with a wear rate of 0.014 mm^3/Nm for 10 N, 0.0076 mm^3/Nm for 20 N, and 0.0075 mm^3/Nm for 30 N. Note that the impact strength of polyester composites is found to be 27.2 KJ/m^2.
- Overall, PALF polyester composites with 20 mm fiber length and fiber loading produced better properties than other composites. The pineapple leaf fiber is manually extracted, and manufacturing materials for potential future uses is cost-effective.

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