

# EFFECT OF COUPLING AGENTS OF THE INTERFACIAL STRENGTH AND MECHANICAL PROPERTIES OF KENAF/POLYMER COMPOSITES

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### Abstract

The surfaces of kenaf fibers were treated with three different silane coupling agents. Among them, the most effective one for the property improvement was 3-glycidoxypropyltrimethoxy silane (GPS) when it was applied to the kenaf fiber surfaces at 0.5 wt%. Various thermoplastic (polypropylene: *PP)* and thermosetting (unsaturated polyester: UPE) composites with untreated and treated chopped kenaf fibers were fabricated using compression molding technique. The result demonstrates that the interfacial. flexural, tensile. and dvnamic mechanical properties of both kenaf/polymer composites importantly depend on the GPS silane treatments done at different concentrations. The result was also agreed with each other among their interfacial shear strength, flexural properties, tensile properties, storage modulus, with support of fractography of the composites.

#### **1** Introduction

Recently, there has been an increasing interest in the use of natural fiber-reinforced polymer composite materials (NFRP) or biocomposites. They have been attracted attention as potential alternative of glass fiber-reinforced polymer composites (GFRP) during the last years. This is because of many advantages of natural fibers over glass fiber reinforcement, for example, natural abundance, low cost, low density, biodegradability, carbon dioxide reduction in nature, acceptable specific mechanical properties, damping and insulation characteristics, good processing stability, etc. However, they also have some drawbacks, for instance, weak interfacial strength between natural fiber surfaces and a polymer matrix, fiber variability, surface irregularity, and finite fiber length[1].

The weak bonding at the interfaces between the natural fibers and the polymer matrix is surely a critical cause of the reduction of the properties and performances of the composites. Therefore, a number of chemical and physical surface modification studies on a variety of natural fibers have been devoted to understand and improve the interfacial strength between natural fibers and the polymer matrix[2-4].

In particular, as one of the chemical modification methods, a silane coupling agent, which is generally used in glass fiber composites, is a chemical substance capable of reacting with both the reinforcement and the polymer matrix of a composite material[5]. It acts as an interphase between the polymer phase and the fiber phase forming a chemical bridge between the two phases.

Kenaf (*Hibiscus cannabinus*) is an herbaceous annual plant that can be grown under the wide range of weather condition, for example, it grows to more than 3 m within 3 months even in moderate ambient conditions. Kenaf has been usefully utilized for a long period of time, traditionally as rope, canvas, sacking, etc. In general, kenaf fiber is composed of 45-57% cellulose, 21-23% hemicellulose, 8-13% lignin and others like pectin and waxes[6,7].

Polypropylene (PP) has been most frequently used as thermoplastic matrix resin in conventional GFRPs. This is due to its cost effectiveness, good mechanical properties, easy processibility, versatile applications, etc. Recently, PP has also been widely used with natural fibers in various NFRP systems[8].

Unsaturated polyester (UPE) resins are the most widely used thermosetting matrix in NFRPs as

well as in conventional polymer composite industries because of their good mechanical, chemical, and weather resistant properties, especially when reinforced with fibers[1,9].

In the present study, kenaf/PP and kenaf/UPE composites were fabricated by means of compression molding technique using chopped kenaf fibers surface-modified with three different silane coupling agents. The goal of the study is to investigate the effect of silane coupling agents on the interfacial shear strength, flexural properties, tensile properties, and dynamic mechanical properties of the composites through the surface modification.

### 2 Experimental

#### 2.1 Materials

Straight kenaf fibers. supplied from Bangladesh, were surface-treated with different silane compounds. The treated-kenaf bundles were cut to 10 mm long in average for fabricating randomly oriented kenaf/PP and kenaf/UPE composites. PP fibers in the entangled fluffy form (P-4203, 10 denier) were supplied from Kolon Glotech Co., Korea. The average PP fiber diameter was about 20-25 µm. The tenacity was 1.5-4.5 g/denier. The ortho-type UPE resin containing a 35 wt% styrene monomer as crosslinking agent and also diluent was purchased from Sewon Chemical Co., Korea. Methyl ethyl ketone peroxide (MEKP) was used as catalyst. E-Glass fibers supplied from Hankuk Fiber Co., Korea were used for comparing the properties with kenaf reinforced polymer composites.

#### 2.2 Fiber Surface Treatment

Three different silanes from Aldrich were used as coupling agents, which are 3glycidoxypropyltrimethoxy (GPS). silane 3aminopropyltriethoxy silane (APS), and 3methacryloxypropyltrimethoxy silane (MPS). The concentrations of each silane were 0.1, 0.3, 0.5, 1, 3, and 5 wt% regarding the weight of kenaf fibers. Straight kenaf fiber bundles were treated by dipping them into each silane solution (ethanol:water = 1:1) for 2 h at ambient temperature and then completely dried and chopped.

#### 2.3 Composite Processing

The PP fibers and chopped kenaf fibers were dried at  $80^{\circ}$ C for 6 h in a vacuum oven. Prior to

composite processing and then they were uniformly mixed. After mixing, the kenaf fiber and the PP compounds were placed in a stainless steel mold and melted using a hot-press at 180°C for 20 min, holding at 1000 psi for 10 min. Finally, the compression-molded composite was cooled down to ambient temperature, maintaining the applied pressure. During the composite process, the PP fibers can be transformed into the PP matrix through the melting.

Kenaf/UPE composites were fabricated using compression molding technique at  $50^{\circ}$ C and then further cured at  $80^{\circ}$ C. A pressure of 1000 psi was applied. A matched-die mold with cavity dimensions of 100 mm x 150 mm was used. The thickness of each composite was varied according to the requirement of analytical method.

The fiber contents were 40 vol% in kenaf/PP and 30 vol% in kenaf/UPE composites. For comparison, glass fiber/PP and glass fiber/UPE composites (GFRP) were fabricated by the same molding method with the corresponding fiber length and loading as in the kenaf fiber composites.

#### 2.4 Characterization

#### 2.4.1 Resin Microdroplet Formation and Single Fiber Microbonding Test

A universal testing machine (UTM, Instron 4467) was used for a single fiber microbonding test. The load cell was 100 N and the crosshead speed was 2 mm/min. The grip distance was 20 mm. Figure 1 shows a schematic illustration of a single fiber microbonding test including a thermoplastic PP microdroplet and a thermosetting UPE resin microdroplet formed respectively on a single kenaf fiber filament and compares optical micrographs observed before and after the test. Prior to resin microdroplet formation, kenaf fibers with a relatively uniform fiber diameter around 30 µm were selected because cellulose-based natural fibers including kenaf may, in general, have variable fiber diameters at different locations due to the irregular fiber surfaces

Each test was performed with about 30 specimens. The average value of the interfacial shear strength (IFSS) for each composite specimen was obtained from all the test results using the following equation.

$$\tau = F/(\pi \cdot D_f \cdot L_e) \tag{1}$$

Here  $\tau$  is the IFSS. F is the force required for debonding the resin microdroplet from the single kenaf fiber filament while tensile loads are applied.  $D_f$  is the diameter of the measuring fiber. And  $L_e$  is the fiber length embedded in the resin microdroplet.



Figure 1. (a) Schematic illustration of a single fiber microbonding test, (b) kenaf/PP composite model specimens, and (c) kenaf/UPE composite model specimens for single fiber microbonding test.

### 2.4.2 ATR-FTIR

Attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectra were recorded using a Jasco 300E spectrophotometer in the range of 1250-700 cm<sup>-1</sup>.

#### 2.4.3 Flexural Test

Three-point bending tests were performed according to ASTM D790M-86 using a universal testing machine (Instron 4467). A load cell of 30 kN was used. The crosshead speed was 0.85 mm/min and the span-to-depth ratio was 16:1. The average values of the flexural strength and modulus of each composite were obtained from about 10 specimens.

## 2.4.4 Tensile Test

Tensile tests were performed according to DIN 53455 using a universal testing machine (Instron 4467). The specimen dimensions were 150 mm x 15 mm x 5 mm and the gauge length was 100mm. A load cell of 30 kN was used. A crosshead seed of 10 mm/min was used. The average values of tensile strength and modulus of each composite were obtained from 10 specimens.

## 2.4.5 Dynamic Mechanical Analysis

Dynamic mechanical analysis (DMA Q800, TA Instruments) was done purging nitrogen gas with a heating rate of 5  $^{\circ}$ C/min at a fixed frequency of 1 Hz and with an oscillation amplitude of 20  $\mu$ m. A

single cantilever bending mode was used. The specimen dimensions were 35 mm x 12 mm x 2 mm.

## 2.4.6 Scanning Electron Microscopic Observation

Scanning Electron Microscopy (SEM, JSM-6380) was used to observe the fracture surfaces of each composite with the kenaf fibers treated at different silane concentrations. All specimens were gold-coated before observation. The acceleration voltage used was 5 kV.

#### **3 Results and Discussion**

Figure 2 depicts the interfacial shear strength (IFSS) result obtained by means of single fiber microbonding tests with a kenaf single fiber embedded in the PP and UPE microdroplet. The IFSS vales of untreated kenaf/PP and kenaf/UPE composites were significantly improved not only by the type of silanes but also their concentrations applied. In both kenaf/PP and kenaf/UPE composites, the greatest improvement of the IFSS was obtained with both GPS and MPS, especially at 0.5 wt% conc. At 0.5 wt% of GPS and MPS, the treated kenaf/PP composites showed the IFSS about 88% greater than the untreated counterpart, whereas the treated kenaf/UPE composite with 0.5 wt% GPS exhibited the IFSS about 110% greater than the untreated. The IFSS was lower, indicating that the value was decreased with decreasing the concentration below 0.5 wt% and increasing it above 0.5 wt%. As a result, it may be described that use of the GPS coupling agent was most effective for increasing the interfacial adhesion between the kenaf and the polymer matrix, based on the single fiber microbonding test result. In addition, GPS is cheaper and easier to handle than APS and MPS. Therefore, only GPS was utilized for treating kenaf fibers to fabricate kenaf/PP and kenaf/UPE composites using a compression molding method hereinafter.

Figure 3 presents ATR-FTIR spectra observed with the untreated and GPS-treated kenaf fibers in the 1250-700 cm<sup>-1</sup> range. It was found that there was a slight change in the absorption bands around 1050 cm<sup>-1</sup> and 765 cm<sup>-1</sup>. A slight increase in the peak around 1050 cm<sup>-1</sup> is attributed to the presence of the asymmetric stretching of -Si-O-Si- and/or to the -Si-O-C- bonds. Also, the absorption band around 765 corresponds to the -Si-C- symmetric stretching bond[2,10].

The peak assignment is in agreement with those reported in other studies dealing with glass

surfaces treated with the same silane coupling agents[11]. This suggests that the condensation reaction between the hydrolyzed silane and the kenaf fiber surface can take place.



Figure 2. Interfacial shear strengths measured for (a) kenaf/PP and (b) kenaf/UPE composites with kenaf fibers treated with various silanes at different concentrations.



Figure 3. ATR-FTIR spectra for (a) untreated and (b) GPS-treated kenaf fibers.

In general, flexural deformation occurs at the outside surface of the test specimen. In a fiber reinforced composite system, the specimen is deflected until a rupture occurs in the outer fibers and the fibers located in the specimen surface may sensitively respond for the applied load.

Figure 4 depicts the variation of the flexural properties measured from three-point flexural tests for various kenaf/PP and kenaf/UPE composites prepared using kenaf fibers treated at different GPS contents. The single data (PP & UPE) on the far left in Figure 4 are for PP and UPE controls without kenaf fibers, respectively. The figures also include the flexural data measured from E-glass/PP and E-glass/UPE composites (GFRP) with the corresponding glass fiber length and loading for comparison.

The flexural strength of PP and UPE were decreased with incorporation of chopped untreated kenaf fibers in the composite system. This is because the presence of cellulose-based kenaf with cellular structure in the PP and UPE matrices lowered the strength. In fact, the presence of pores and voids in the composite is detrimental to the mechanical strength. However, the flexural strength of untreated kenaf/PP kenaf/UPE composites and was significantly improved with the GPS treatment. The greatest improvement on the flexural strength of the untreated (0 wt%) composite was obtained from kenaf/PP and kenaf/UPE composites treated with 0.5 wt% GPS, as found in the IFSS result. The flexural modulus of kenaf/PP and kenaf/UPE composites was greatly improved by incorporating chopped kenaf fibers. The greatest improvement of the modulus was also found with 0.5 wt% GPS.

The flexural strength of untreated kenaf/PP composite was increased from 38 MPa to 48 MPa by the fiber treatment with 0.5 wt% GPS. This value is quite comparable with the GFRP value (about 47 MPa). The flexural modulus was also improved up to about 2 GPa after the treatment with 0.5 wt% GPS. It is higher than the GFRP (about 1.8 GPa). In the case of kenaf/UPE composite, the GPS silane treatment effect was somewhat greater than in the kenaf/PP composite. The flexural strength of untreated composite was largely improved from 75 MPa to 105 MPa after the treatment with 0.5 wt% GPS. This result is also comparable with the GFRP value (about 110 MPa). The flexural modulus (7.9 GPa) of GPS-treated kenaf/UPE composite was also comparable with E-glass/UPE composite (about 8 GPa).

The variation of the flexural properties as a function of GPS contents is quite consistent with that of the interfacial shear strength result described earlier. It demonstrates that the GPS treatment done to the kenaf fiber surfaces strongly affects not only on the interfacial adhesion between the kenaf fibers and the thermoplastic PP or the thermosetting UPE in the composites but also on the flexural properties.



Figure 4. Variations of the flexural strength and modulus measured for (a) kenaf/PP and (b) kenaf/UPE composites with kenaf fibers treated at different GPS contents. The figures also include the data for PP, UPE, GFRP with PP, and GFRP with UPE, respectively.

Figure 5 shows the variation of the tensile properties measured for various kenaf/PP and kenaf/UPE composites prepared using kenaf fibers treated at different GPS contents. The tensile strengths of PP (24.5 MPa) and UPE (about 32 MPa) controls were slightly decreased by incorporating the chopped kenaf fibers. However the tensile strength of untreated kenaf/PP and kenaf/UPE composites was increased with the GPS treatments. The greatest improvement was with the both composites treated at 0.5 wt% GPS, as similarly found in the flexural study. The tensile strength of untreated kenaf/PP composite was increased about 17% from 23 MPa to 27 MPa after the treatment. The strength is comparable to that of E-glass/PP composite of about 27.5 MPa. Also, the tensile modulus of kenaf/PP and kenaf/UPE composites was varied against the GPS content, as similarly examined in the tensile strength. The maximum modulus obtained with 0.5 wt GPS was about 3.9 GPa, which is slightly higher than that of E-glass/PP composite.

In the case of kenaf/UPE composite, the variation of the tensile properties as a function of GPS content is almost similar with the result from kenaf/PP composite, exhibiting the maximum properties at 0.5 wt% GPS.

The mechanical study indicates that the tensile properties as well as the flexural properties of kenaf/PP and kenaf/UPE composites strongly depend on the GPS silane content used for modifying kenaf fibers. It is noted that the flexural and tensile properties of the untreated composites were significantly enhanced by reinforcing with surface-modified kenaf fibers, exhibiting the greatest values of the strength and the modulus with the 0.5 wt% GPS treatment. The greatest strength and modulus of kenaf/polymer composites were comparable to those of E-glass composites with the corresponding fiber length and loading.



Figure 5. Variations of the tensile strength and modulus measured for (a) kenaf/PP and (b) kenaf/UPE composites with kenaf fibers treated at different GPS contents. The figures also include the data for PP, UPE, GFRP with PP, and GFRP with UPE, respectively.

Figure 6 shows the variation of the logarithmic storage modulus (E') as a function of temperature for PP control, untreated and GPS-treated kenaf/PP composites, and E-glass/PP composite. The storage modulus of PP control was greatly increased over the measuring temperature range due to reinforcing effect by the chopped kenaf fibers of 40 vol%. In addition, the storage modulus of the untreated composite was further enhanced by the fiber surface modification. Interestingly, the modulus of 0.5 wt% GPS-treated kenaf/PP composite was comparable to E-glass/PP counterpart, especially at above glass transition temperature.

In order to inspect quantitatively the dynamic mechanical property change with the GPS content, the storage moduli obtained at four specified measuring temperatures (-30  $^{\circ}$ C, T<sub>g</sub>, 40  $^{\circ}$ C, and 80  $^{\circ}$ C) were summarized in Table 1. The reason for selecting such specified temperatures is following: - $30^{\circ}$ C at the glassy region, T<sub>g</sub> at the glass transition temperature, 40°C at far above the glass transition, and  $80^{\circ}$ C at a possible use temperature. It can be seen that the storage modulus of untreated kenaf/PP composite was significantly increased by the fiber treatment with 0.5 wt% GPS at all specified temperatures, showing the maximum value of E'. The overall tendency of the dynamic mechanical storage modulus variation as a function of GPS content was quite similar to that seen from the interfacial and quasi-static mechanical results described earlier. This implies that the untreated kenaf fibers should be modified with an appropriate silane concentration to improve the storage modulus as well as the flexural and tensile properties of kenaf/PP composites.



Figure 6. Variations of the storage modulus of PP control, untreated and treated kenaf/PP composites, and an E-glass/PP composite as a function of temperature.

Table 1. A comparison of the storage moduli of PP, untreated and treated kenaf/PP composites, and an E-glass/PP (GFRP) at specific temperatures

< MPa >	PP	Untreated	0.5 wt%	1 wt%	3 wt%	GFRP
E' at -30°C	3171	4170	5489	5385	4567	6052
E' at T <sub>g</sub>	3330	4036	5325	5244	4426	5768
E' at 40°C	2053	3152	4153	4173	3459	4141
E' at 80°C	1507	2661	3489	3510	2889	3473

Figure 7 presents the variation of tan  $\delta$  as a function of temperature for PP, kenaf/PP composites, and an E-glass/PP composite. The tan  $\delta$  peak height was surely reduced due to reinforcing kenaf fibers but it did not follow the tendency of the storage modulus change shown in Figure 6. The glass transition temperature, which can be normally determined from the tan  $\delta$  peak temperature, of the composites was in the range of  $-9^{\circ}C \sim -5^{\circ}C$  and it was not changed significantly by the surface treatment.



Figure 7. Variation of the tan  $\delta$  curves of PP control, untreated and treated kenaf/PP composites, and an E-glass/PP composite as a function of temperature.

Figure 8 shows the variation of the storage modulus as a function of temperature for PP, untreated and treated kenaf/UPE composites, and an E-glass/UPE composite. Table 2 lists the storage moduli measured at 40 °C, 60 °C, 80 °C, and T<sub>g</sub>. Here each temperature was selected since 40 °C and 60 °C are frequently exposable temperature upon use, particularly in many parts applications and 80 °C is known to be the almost maximum use temperature of UPE. As shown, the storage modulus of UPE was significantly increased by introducing chopped kenaf fibers to the UPE matrix, due to a reinforcing effect.

The storage modulus of untreated kenaf/UPE composite was largely increased by about 40% after the GPS silane treatment at 0.5 wt%, giving the

greatest storage modulus among the specimens studied. The higher storage modulus of the silanetreated composite than the untreated is due to the increased interfacial adhesion between the kenaf fibers and the matrix, as reported using various surface modification methods for different natural fibers in other papers[12-16]. This tendency was also quite consistent with the interfacial and mechanical results described above. The result demonstrates that silane coupling agent done on the natural fibers significantly influences not only the interfacial behavior, but also the quasi-static and dynamic mechanical properties of the kenaf/UPE composites.



Figure 8. Variations of the storage modulus of UPE control, untreated and treated kenaf/UPE composites, and an E-glass/UPE composite as a function of temperature.

Table 2. A comparison of the storage moduli of UPE, untreated and treated kenaf/UPE composites, and an E-glass/UPE (GFRP) at specific temperatures

< MPa >	UPE	Untreated	0.5 wt%	1 wt%	3 wt%	GFRP
E' at 40°C	3479	4041	5644	5275	3972	6145
E' at 60°C	2980	3707	5315	4995	3597	5532
E' at 80°C	1172	2308	4427	3316	2316	3473
E' at T <sub>g</sub>	148.2	759.5	1918	1703	703.2	1385

As described earlier, in Figure 9 the tan  $\delta$  peak height of UPE was greatly reduced by incorporation of kenaf fibers. This is because the fibers restrict the movement of polymer molecules. However, the peak height was not strongly dependent on the silane treatment done.

The improvement on the mechanical properties of kenaf/polymer composites is because the interfacial adhesion between the kenaf fibers and the PP or the UPE polymer matrix has been effectively



Figure 9. Variation of the tan  $\delta$  curves of UPE control, untreated and treated kenaf/UPE composites, and an E-glass/UPE composite as a function of temperature.

increased by GPS silane treatment. The bifunctional silane molecules act as a link between the resin and the cellulose by forming a chemical bond with the surface of cellulose through a siloxane bridge while its organo-functional group bonds to the polymer resin. This co-reactivity with both the kenaf and the thermosetting UPE resin via covalent bonds may provide some molecular continuity across the interfacial region of the composites[2].

Although there is no undergoing chemical reaction between the natural fibers and the thermoplastic resin during molding process. complete inertness of the fiber-matrix system cannot be attained during molding because there are some possible grafting reactions at molding temperatures. Therefore, the silane coupling agent may, to some extent, react with polypropylene at molding temperature through an insertion reaction into C-H bonds of the polymer. This can explain why the interfacial and mechanical properties of the untreated kenaf/PP composite were improved after the silane treatment of kenaf fiber surfaces[17,18]. Another plausible reason is that the wettability of the melted PP to the kenaf surfaces may be enhanced by the silane treatment as a sizing material.

As shown earlier, the interfacial shear strength of untreated and treated kenaf/polymer composites strongly depends on the surface treatment level with GPS. The fractographic result in Figure 10 supports qualitatively the interfacial behavior, which is related with the mechanical behavior in terms of flexural, tensile, and storage modulus properties above-mentioned. Figures 10 and 11 display SEM micrographs observed from the fracture surfaces of the untreated and treated composites. Each microscopic observation was just focused on a representative single kenaf fiber surrounded with the PP and UPE matrices. The single kenaf fiber consisted of a number of cells, the fiber surfaces were undulated, and the shape and diameter of the single fiber were varied fiber by fiber, as similarly found in other cellulose-based natural fibers. Figures 10 and 11 exhibit the typical fracture pattern of a ductile thermoplastic and a brittle thermoset, respectively. In addition, it was likely that the fiber surfaces fractured depend on the GPS content used.

With a closer inspection of Figures 10 and 11, it was found that the untreated composite specimen (a) had the poor interfacial adhesion between the kenaf fiber and the PP or UPE matrix. One can see some gaps or interstices between the natural fiber and the surrounding matrix. On the other hand, in the case of 0.5 wt% GPS (b), there was a close interfacial contact between the fiber and the matrix. In addition, the pull-out fiber length after fracture was relatively shorter than in the other cases. It was also observed that the kenaf fiber was broken together with the PP matrix upon fracture, indicating the cohesive failure and the strongest interfacial adhesion between the fiber and the matrix. In both composite cases, the interfacial gap between the kenaf fiber and the surrounding matrix becomes greater with increasing GPS content again. The pullout fiber length also becomes longer again.



Figure 10. SEM micrographs observed for kenaf/PP composites fabricated with kenaf fibers treated at various GPS contents. ( $\times$ 1200); (a) untreated, (b) 0.5 wt% GPS, (c) 3 wt% GPS, (d) 5 wt% GPS.



Figure 11. SEM micrographs observed for kenaf/UPE composites fabricated with kenaf fibers treated at various GPS contents. ( $\times$ 1200); (a) untreated, (b) 0.5 wt% GPS, (c) 3 wt% GPS, (d) 5 wt% GPS.

#### **4** Conclusions

In the present study, we have attempted to increase the interfacial and mechanical properties of kenaf/PP and kenaf/UPE composites through the surface treatments with different silane coupling agents at various concentrations. As a result, the following conclusions were obtained.

Among the three silane coupling agents used, the most effective one for the property improvement was 3-glycidoxypropyltrimethoxy silane (GPS) when it was applied at 0.5 wt% GPS for the kenaf surface treatment, resulting in the increased interfacial shear strength, flexural, tensile, and dynamic mechanical properties of the both kenaf/PP and kenaf/UPE composites.

The quasi-static and dynamic mechanical properties of PP and UPE controls were enhanced by incorporating chopped kenaf fibers and further increased with the silane treatment, due not only to reinforcing fiber effect but also to the improved interfacial adhesion between the kenaf fibers and the thermoplastic PP or thermosetting UPE matrix.

The mechanical properties of kenaf/PP and kenaf/UPE composites were comparable to those of E-glass/PP and E-glass/UPE composites with the corresponding fiber length, fiber loading, and processing method.

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