



Effect of Immersion Temperature on Mechanical, Water Absorption and Morphological Properties of Sodium Hydroxide Modified Fluted Stem Pumpkin Fiber Reinforced Polyester Bio-composites

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Authors' contributions

This work was carried out in collaboration among all authors. Authors OAO and IOM designed the study, performed the statistical analysis. Author TUJ wrote the protocol, and wrote the first draft of the manuscript. Authors EN and OFO managed the analyses of the study and managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

The effect of immersion temperature on mechanical, water absorption and morphological properties of sodium hydroxide treated and untreated fluted pumpkin stem fiber (FPF) polyester bio-composites was investigated. Random matted FPF reinforced polyester composites were fabricated using a hand layup molding process, with 30wt% of FPF treated under the temperatures of 30°C, 40°C and 60°C were investigated. Fourier transform infra-red spectroscopy (FTIR) and Scanning electron microscope (SEM) analysis were employed in characterizing the microstructure and morphology of the materials. Mechanical tests showed that the treated FPF at 30°C improved the tensile modulus by 21.17% (1100-1300 Nmm⁻²) compared to the untreated FPF. The treated FPF at 45°C improved the tensile strength by 36.4% (14.42-19.71MPa), improved flexural strength by 14.77% (63.23-72.57MPa), improved impact strength by 48.28% (3.48-5.15kJm⁻²), improved compressive strength by 35.01% (17.99-24.29MPa) compared to the untreated FPF. Discrepancies in properties emanating from immersion temperatures were found to show enhanced properties at 45°C than 30°C and 60°C immersion temperatures. It was found that the highest improvement in tensile, flexural, compressive and impact strength was attained at a temperature of 45°C while the optimum tensile modulus was obtained at a temperature of 30°C for the FPF treated polyester composites compared to the untreated composites. The percentage water absorption for the treated FPF polyester composites decreased above 45°C. The initial increase from 30-45°C was ascribed to poor wetting of the fiber by the resin. The results obtained from FTIR and SEM confirmed that the chemical modification of the composites was successfully performed.

Keywords: *Fluted pumpkin fiber; polyester; mechanical; temperature; alkali treatment; water absorption.*

1. INTRODUCTION

Recently, plastic composites reinforced with natural fibers have gained popularity as regards their offer of improved properties. Because of their flexibility and strength properties, natural fibers are widely established and accepted in different industries of the research community as better and more environmentally friendly reinforcement materials. However, the complete usage and adoption of natural fibers composites in automotive and other construction industries have been significantly hampered by the weak fiber-matrix contact, water sorption capacity and low durability which negatively affect their mechanical properties [1]. These have triggered the interest of researchers in finding ways of making up for the deficiencies in the composite (fiber-matrix). Researchers have proven experimentally surface treatment of natural fibers as an effective method of bypassing the deficiencies associated with natural fiber composites [2-6]. Sisal, areca, hemp, flax, kenaf, coir, bagasse, jute, cotton, bamboo, banana, pineapple, and henequen are a few varieties of natural fibers available in nature and have been used since human evolution. These fibers are commonly used as reinforcements in bio-composites for different applications such as in aircraft, aerospace, automotive, marine, and other industries [7]. The availability, low cost,

recyclability, biodegradability, low density as well as satisfactory mechanical properties offered by sisal fiber [8], has made it a choice as a reinforcement material in this study. Sisal fibers are grown throughout the world, but expansively in tropical and sub-tropical regions and are generally used for making materials such as ropes, handicrafts, mats, carpets, papers and textile fibers.

Recently, most studies carried out across the globe have proven sisal fibers suitable to produce sisal-based composites with tolerable physical and mechanical properties. However, past works have established that natural fiber-based composites, such as sisal fiber composites are susceptible to heat and moisture when subjected to varying environmental conditions which are capable of negatively affecting the properties of the fiber itself [7,8]. More so, sisal fiber composites possess other drawbacks like the incompatibility that exists between the hydrophobic polymers and hydrophilic natural fibers. These drawbacks necessitated chemical modification of natural fibers to improve their adhesion to the matrix. Different surface treatment techniques such as mercerization (alkali treatment), isocyanate treatment, acrylation, benzoylation, latex coating, permanganate treatment, acetylation, silane treatment, thermal treatment and peroxide

treatment have been recommended by many researchers [9]. To improve the interpenetration of the polymer matrix in the fiber, the most popular method of treatment, which is alkali treatment, was applied to natural fibers aiming to reduce the incompatibility that naturally exists between them [10]. More so, previous works on surface treatment of natural fibers have established that alkali treatment removes surface impurities, which leads to an increase in the amount of cellulose exposed on the fiber surface, hence, opening more sites for good interlocking between the fiber and the matrix.

“The polymer resin is an effective holding medium for fibers. It also helps to transfer the load to fiber reinforced composites. The resin, its chemical composition, and physical properties are known to affect the processing, fabrication, and ultimate properties of a composite material. They are easily fashioned into any desired shape, are well-suited with most other materials and cure readily (by heat or catalyst) into an insoluble solid. There are two major types of polymer matrix/resin: thermoplastic and thermoset polymer resin. Although there are many different types of resin utilized in the composite industry, polyester, phenolics and epoxy resins are the most commonly used thermoset. Among these polymers resins, polyester was chosen and used in the fabrication of natural fibrebased composites to produce suitable composite material parts with adequate mechanical and physical properties” [11,12].

Previous studies have reported the mechanical properties of natural fibers to determine their potential to be used as reinforcement in commercial polymer composites. Sreekumar et al., [13] modified sisal fiber using sodium hydroxide, heat treatment, benzoylation and silanization to improve interfacial bonding with polyester resin. After the results of the tensile, flexural and impact properties of all polyester composites reinforced with various treated fibers with those of untreated fiber composites, he found that all the treatment methods increased the flexural, tensile and impact properties of the treated fiber reinforced polyester composite when compared with the untreated. The scanning electron microscopy was also applied in studying the morphology of the treated sisal fiber. Senthilkumar [14] studied “the effects of alkali treatment at various concentrations (0,3,6,9%) of sodium hydroxide solution on the mechanical properties of sisal fiber reinforced epoxy composites. The tensile and flexural

strength of alkali-treated composites were improved, compared to the untreated fiber composites. The impact strength of the treated fiber composites was decreased compared to the untreated ones”. Gupta et al., [15] investigated “the tensile and flexural properties of sisal fiber reinforced epoxy composites. The composites fabrication was performed at various fiber volume contents of 15, 20, 25, and 30% of sisal fiber. The orientations of the fibers in the composite were in unidirectional and mat form. The result of the experiment showed that the tensile and flexural strength (132.73MPa and 288.6MPa) of the sisal fiber epoxy composites, both in unidirectional and in mat form were found to be maximum at 30% of sisal fiber content. Composite in the unidirectional orientation of fibers was found to give better tensile and flexural properties in comparison to the mat form”. Oladele et al., [16] studied the effect of extraction by soil retting and chemical treatment on the mechanical properties of sisal fiber reinforced polyester composites. The treatments were conducted at an elevated temperature of 70°C for 2hrs using 2% solution each of sodium hydroxide, potassium hydroxide, hydrogen peroxide and ethanol. Sisal reinforced polyester composites were developed using both treated and untreated fibers for a test of mechanical properties. The results showed that potassium hydroxide treated fiber reinforced polyester composite, followed by that of ethanol treated, gave the best enhancement of the tensile and hardness properties of the sisal reinforced polyester composites than other treatments. Boopathi et al., [17] studied “the influence of curing temperature of 40,60,80,100°C and fiber volume fraction of 15,30,45% on the mechanical properties of sisal fiber reinforced polyester composites. The result of the test conducted for the tensile and impact strength of the fabricated composite sheets showed that the tensile strength of the composites increased with the rise in curing temperature and reached a maximum of 27.5 MPa at 60°C. Also, the impact strength of 36kJ/m² of the sisal fiber reinforced polyester composite was found to be better with the composite fabricated at 30 % fiber volume fraction and increased gradually with increasing curing temperature and decreased after attaining a maximum of 80°C”. In another investigation, Bichang’a, et al., [18] evaluated “the effect of alkali treatment on the mechanical properties of a woven sisal fabric reinforced epoxy composite fabricated at 40 % fiber weight fraction. It was inferred that chemical treatment of sisal fabric with 4 % (w/v) sodium hydroxide solution for 1hr

at room temperature improved the mechanical properties of the composite". Further, Samuelet al., [19] reported that "the mechanical properties of ukam and sisal fiber reinforced composites were significantly influenced by the alkali treatment of the fibers".

Studies have reported that "natural fibers showed great sensitivity to temperature and thermal degradation expected to occur" [20]. Therefore, the behavior of composite material produced with fibers treated at different temperatures is an important factor to be considered, to determine which temperature should produce the most suitable material properties. Investigations have been made on thermal degradation of polymer matrix composites, the curing temperatures of composites, the mechanical fabrication and mechanical testing temperatures of the composites. The results showed that temperature contributed to the improvement in the properties of composites.

However, the effect of temperature on composite material properties is less explored because the immersion temperature and treatment has not been exhausted. Moreover, literature review showed that no work has been done using fluted pumpkin/polyester composites.

The negative effect of temperature on natural fiber properties cannot be overemphasized. Since, the application of natural fibers composites in water environments is inevitable. Therefore, the objective of the study was to investigate the mechanical, water absorption and morphological properties of fluted pumpkin stem fibers reinforced polyester bio-composite as a function of sodium hydroxide treatment and immersion temperature of the bio-composites. The novelty of the study is to investigate the effect of chemical modification and immersion temperature on the properties of underutilized and ubiquitous fluted pumpkin stem fiber agro waste material.

2. MATERIALS AND METHODS

2.1 Materials

Fluted pumpkin fiber (FPF) was obtained locally from the Nnamdi Azikiwe University Farm. Other materials included the treatment agents: Unsaturated polyester resin, methylethylketone peroxide (MEKP), cobalt naphthenate, sodium hydroxide and distilled water supplied by Henkoff Chemicals Ltd., Abuja, Nigeria. Petroleum Jelly

was used as a mold release agent to prevent the composite from sticking to the mold during de-molding. A hand brush was used to smear the mold release agent. Plies and paper tapes were used to form the shape of the mold.

2.2 Fiber Surface Treatment

Retted fluted pumpkin fibers were immersed in 5% sodium hydroxide (NaOH) solution in a water bath for 1hour at immersion temperatures of 30, 45, 60°C. For the preparation of the NaOH aqueous solution, 40grams of NaOH pellets were dissolved in 800ml of water for each treatment temperature of each of the fibers. For the treatment of each of the fibers a beaker containing 800ml of aqueous solution of NaOH and 80grams of fluted pumpkin fibers was placed in a water bath for each treatment temperature. This process was repeated successively for the various temperatures. After complete treatment with alkali, the fibers were rinsed thoroughly with distilled water and sun dried for 48hours. The fibers were further dried in an oven at 60°C for 1hour to ensure complete drying. The treated fibers were finally stored in desiccators for composite preparation.

2.3 Composite Fabrication

A hand lay-up method was used for the composites molding. A mold of smooth polished metal sheet of dimension 300 x 300 x 5mm was made, coated with mold release agent for ease of removal of the composite laminate after curing, followed by pouring of the composites that comprise of polyester resin and randomly spread FPF fibers. For proper curing process and chemical reaction between resin and fiber, cobalt naphthenate and methylethylketone peroxide were used as accelerator and catalyst. To ensure that the resin was evenly spread across each fiber and removal of possible air bubbles that could be trapped within the composite, roller was used to press the composites. The composite was allowed to cure uniformly at room temperature for 6 hours after which they were cut for mechanical tests.

2.4 Tensile Test

Dumbbell type tensile test specimens were prepared by cutting into the required dimension 200 x 20 x 5mm according to the ASTM D638 standard. A computerized universal testing machine (model: KIC-2-1000-C) with a crosshead motion of 50mmmin⁻¹ was used with a

maximum load rating of 50kN. Specimens treated at different temperatures were tested. Tests were carried out until failure occurred at room temperature. Stress versus strain graphs were generated and plotted.

2.5 Flexural Test

The flexural strength test was done in a three-point flexural setup according to ASTM D 790-10. The specimen size was cut with dimension 150 x 20 x 5mm in line with the machine requirement. The test was carried out using a computerized Universal Testing Machine (UTM) at a crosshead motion of 50mmmin⁻¹. A concentrated load (point load) was placed at the centre of the specimen until it fractured and broke. Stress versus strain graph was generated and ultimate bending strength was noted.

2.6 Impact Test

The impact test was carried out according to ASTM D 256 standard and the specimens were prepared and cut to dimension 66 x 15 x 5mm. During the testing process, the specimen was loaded in the testing machine and allowed the pendulum until it fractured or broke. The energy needed to break the material was noted and the impact strength was determined using the following equation.

$$G_c = \frac{U}{A} = \frac{\text{energy of fracture (J)}}{\text{area of cross-section (m}^2\text{)}} \left(\frac{J}{m^2} \right) \quad (1)$$

The values obtained were used for discussion.

2.7 Compressive Test

A compressive test that measures the capacity of a material to withstand loads tending to reduce size was conducted as per ASTM D3410 using a computerized universal testing machine with a crosshead speed of 50 mm/min. The specimen size was cut to dimension 150 x 20 x 5 mm. The tests were carried out at room temperature and each test was performed until failure occurred. Stress versus strain graph was generated and ultimate compressive stress was noted. In each case, three samples of the composite were tested, and the average value was reported in MPa.

2.8 Water Absorption Test

Water absorption test was studied to show the most prone to water absorption condition. The

specimens were allowed to post-cure under room temperature to a constant weight (M_0). The specimens were then immersed in water at room temperature and periodically taken out (every 24hrs) for up to 120hours. This process was followed by surface drying with tissue paper and weighed using an analytical balance (M_1) [21], before returning to the water again. Three samples were used for each measurement and the average value was recorded. The percentage of water uptake $\Delta M_{(t)}$ was calculated using the following formula:

$$\Delta M_{(t)} = \frac{M_1 - M_0}{M_0} \times 100\% \quad (2)$$

Where $M_{(t)}$ is the moisture uptake, and M_0 and M_1 are the mass of the specimen before and after immersion in water, respectively.

2.9 Scanning Electron Microscopy (SEM)

To ascertain the structural changes that occurred between the fiber and the matrix of the specimens, a scanning electron microscope (SEM) with equipment model- JSM-7900F was used to take micrographs of the specimen at various magnifications.

2.10 Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR technique was utilized in order to help identify the functionality of compounds in the composite materials and to investigate sample compositions. The FTIR analysis was performed using an Agilent infrared spectrometer. The unmodified and modified fibers were analyzed using an attenuated total reflectance (ATR) mode of operation, over a 650-4000 cm⁻¹ wave number range, at a resolution of 4 cm⁻¹.

3. RESULTS AND DISCUSSION

3.1 Mechanical Properties

Fig. 1 shows the effect of alkali treatment on tensile strength of fluted pumpkin fiber/polyester composite at 30°C, 45°C and 60°C immersion temperatures. As illustrated, composites prepared from treated fluted pumpkin fiber showed enhanced mechanical properties compared to the untreated sisal fiber. The tensile strength showed the best improvement with alkali treatment at 45°C by 36.40% from 14.42 MPa to 19.71 MPa. It was observed that beyond 45°C

the tensile strength of the composite decreased. The result emphasized that treatment of fluted pumpkin fiber at high alkali temperature above 45°C can lead to a decrease though not too significant of the tensile strength of the treated fiber. At the optimum temperature the fiber-matrix wettability was high which resulted in better adhesion. This could be because of the effective

removal of most hemicelluloses and lignin with unstable structures and retaining the cellulose with the most stable shape [22]. Owen et al. [23] reported a similar result in which alkali treatment of sisal fiber recorded the tensile strength of 15.64MPa for fiber treated with 2% NaOH, while the composites with untreated fiber had a tensile strength of 8.37MPa.

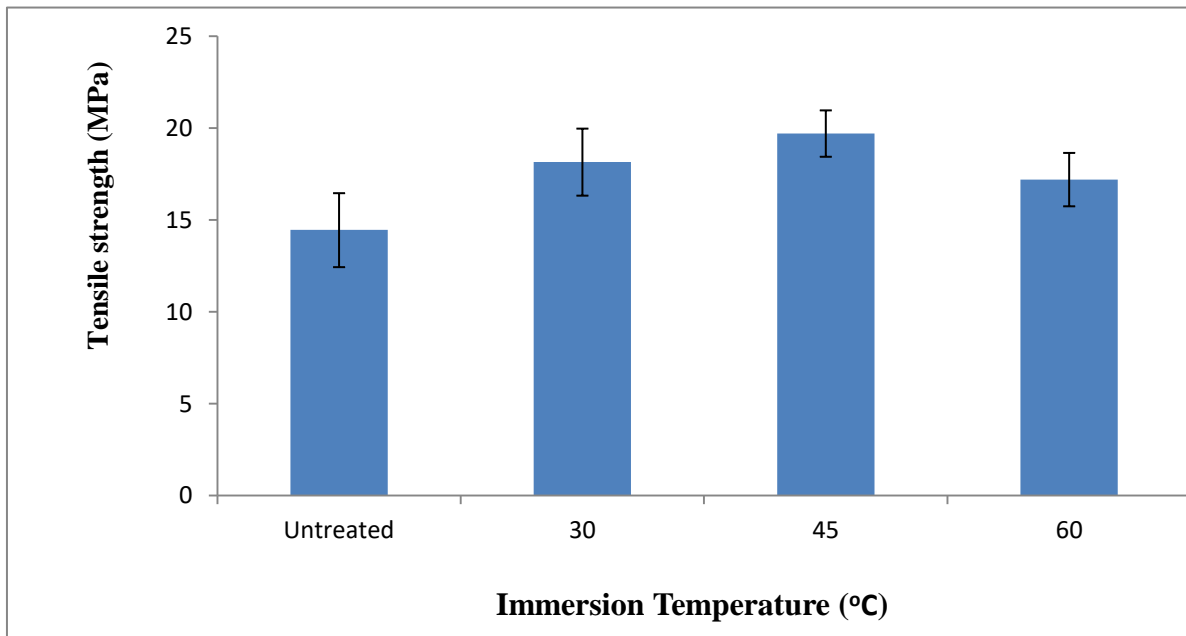


Fig. 1. Effect of immersion temperature on tensile strength of fluted pumpkin fiber reinforced polyester composite

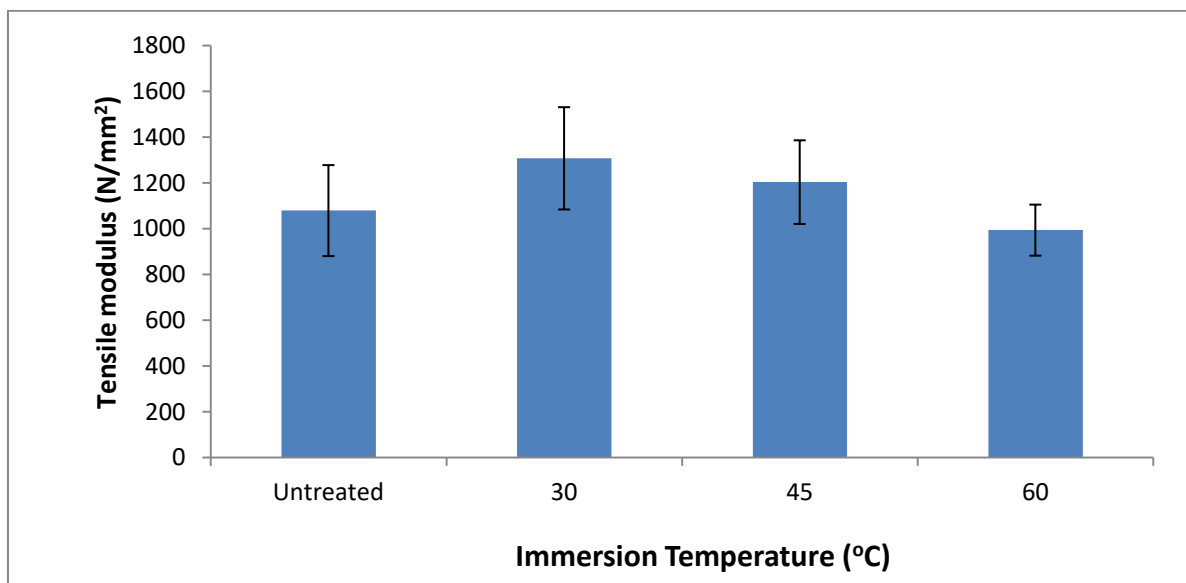


Fig. 2. Effect of immersion temperature on tensile modulus of fluted pumpkin fiber reinforced polyester composite

The effect of alkali treatment on tensile modulus of fluted pumpkin fiber/polyester composite at 30°C, 45°C and 60°C immersion temperature is shown in Fig. 2. Treatment of fluted pumpkin fiber caused an increase in tensile modulus, at 30°C immersion temperature the tensile modulus of the treated composite increased by 21.17% from 1100-1300Nmm⁻² compared to the untreated fluted pumpkin fiber. Above 30°C, there was a decline in tensile modulus. The improvement may be due to alkali mercerization that removed surface impurities from the fluted pumpkin fiber, causing an increase in the crystallinity and effective surface area of the sisal fiber [24]. This finding comparably agrees with the result of Bichang'a et al., [18] on the effect of alkali treatment on tensile modulus of woven sisal reinforced epoxy composite in which 31.19% increment was reported.

In Fig. 3, there is an increase in the immersion temperature from 30°C to 45°C and then a decrease with further increment in temperature. Fluted pumpkin fibers treated at 45°C with the alkali gave the optimum flexural strength of its reinforced composite. The result showed that flexural strength improved from 63.23MPa to 72.57MPa at a rate of 14.77%. A decrease in

flexural strength above 45°C was due to the damaging of the cellulose that constitutes most fibers, thus leading to low interfacial adhesion of fibers with matrix. To achieve enhanced flexural strength means that the interfacial bonding between the fiber and the matrix is important.

Fig. 4 shows the effect of immersion temperature of alkali treated and untreated fluted pumpkin fiber on the impact strength (kJ/m²) of fluted pumpkin fiber reinforced polyester composite at 30°C, 45°C and 60°C immersion temperature. It was found that the impact strength of the composite reinforced with untreated fluted pumpkin fiber was 3.48kJ/m². Thus, it improved with alkali treatment of fluted pumpkin fiber by 48.28% to the maximum value 5.15 kJ/m² at 45°C. This could be due to the deteriorating of the fiber at a high temperature which affected the interfacial properties of the composite, thereby lowering its energy absorption capacity. The result obtained here is in line with the work of Owen et al., [23] in which he studied the effect of alkali concentration on the mechanical properties of sisal/fiber reinforced epoxy composites. In their result, the maximum impact strength was 8.5 kJ/m² at 5% NaOH compared to 5.8 kJ/m² for the untreated composite.

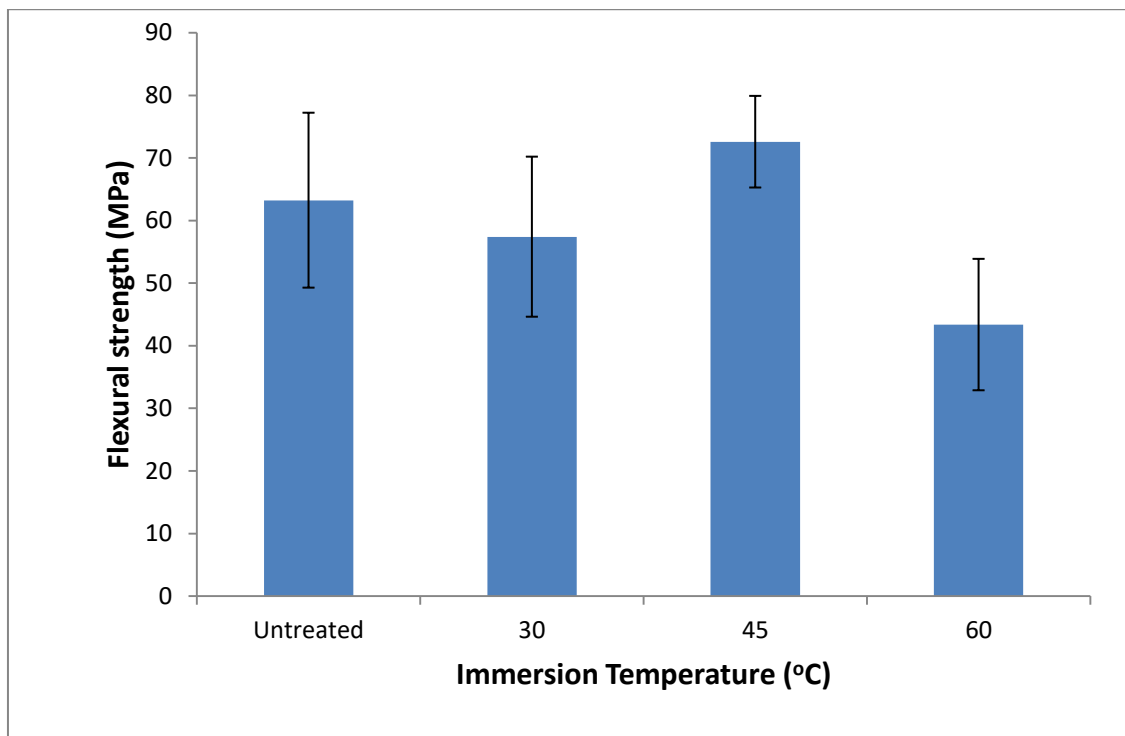


Fig. 3. Effect of immersion temperature on flexural strength of fluted pumpkin fiber reinforced polyester composite

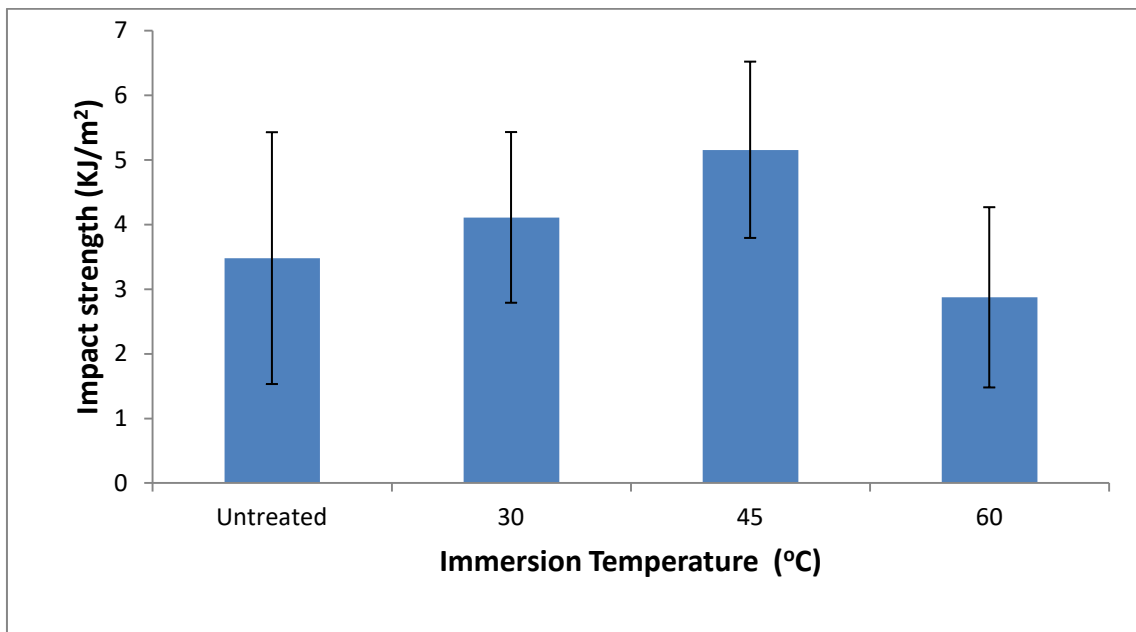


Fig. 4. Effect of immersion temperature on impact strength of fluted pumpkin fiber reinforced polyester composite

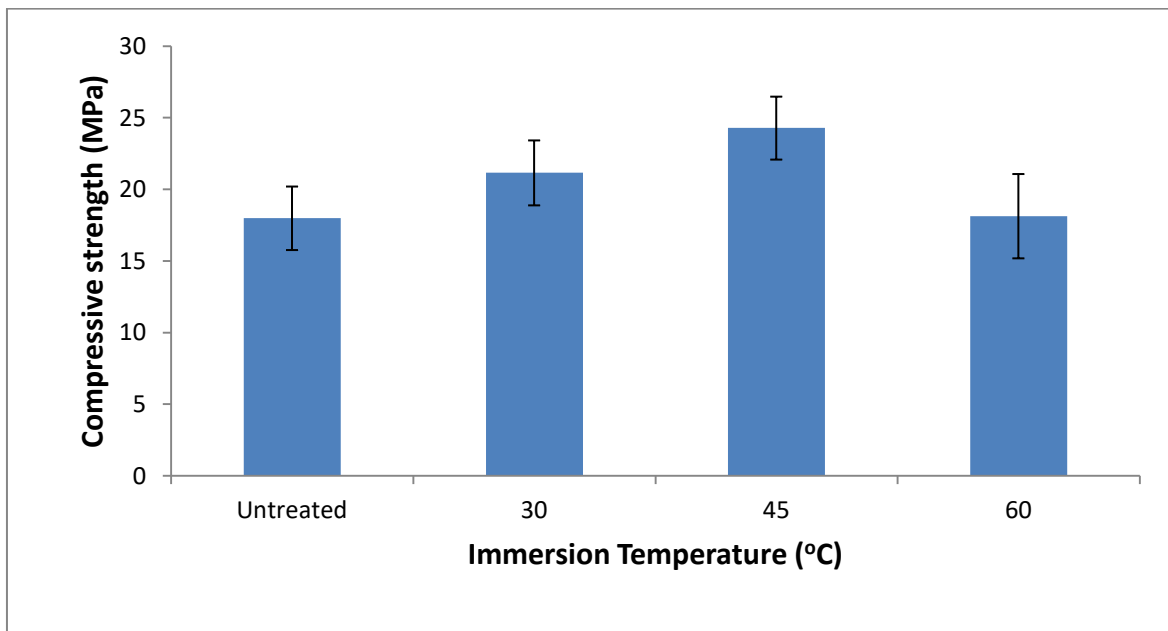


Fig. 5. Effect of immersion temperature on compressive strength of fluted pumpkin fiber reinforced polyester composite

Fig. 5 shows the effect of immersion temperature of alkali treated and untreated fluted pumpkin fiber on the compressive strength of fluted pumpkin fiber reinforced polyester composite at 30°C, 45°C and 60°C immersion temperature. From the result, compressive strength for the untreated fluted pumpkin/polyester composite

was 17.99 MPa. It was observed that the compressive strength of treated fluted pumpkin fiber reinforced polyester composites increased to 24.29MPa at 45°C. It was observed that the compressive strength of the fluted pumpkin fiber reinforced polyester composite decreased drastically beyond 45°C.

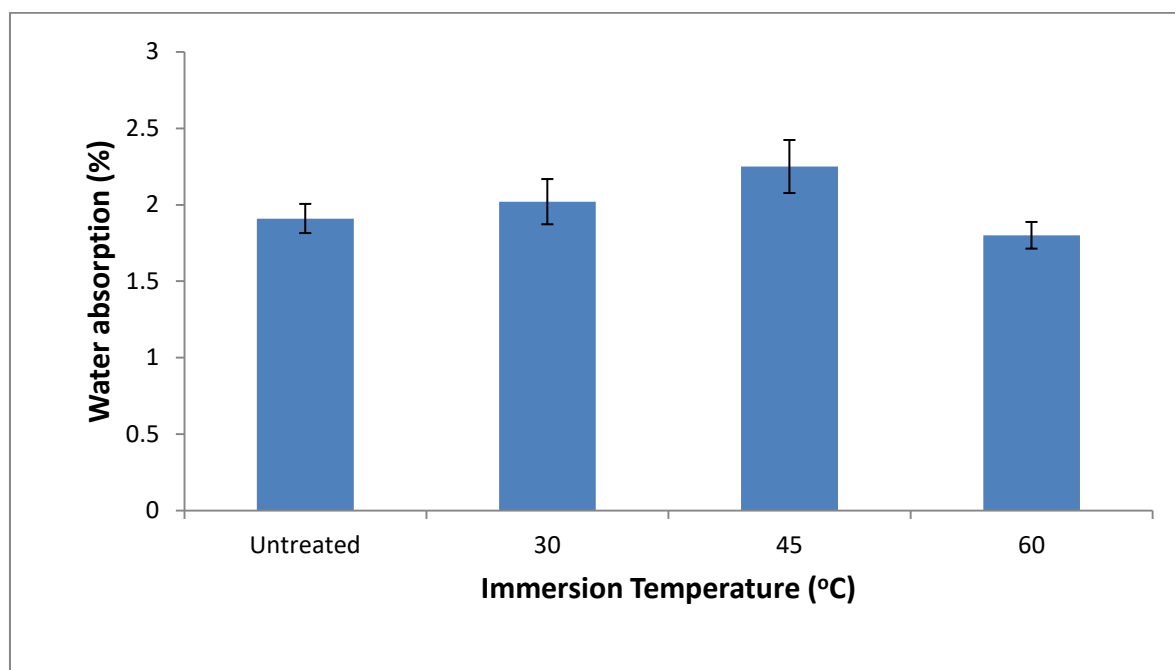


Fig. 6. Effect of immersion temperature on water absorption of fluted pumpkin fiber reinforced polyester composite

Fig. 6 shows that the water absorption percent of the treated fluted pumpkin fiber/polyester composites increased with increasing immersion temperature up to the 45°C immersion temperature, beyond which it decreased. The initial increase from the immersion temperatures 30°C to 45°C could be attributed to the poor wettability of the fiber by the matrix, which is necessitated by the partial removal of fiber surface impurities. This led to the formation of hollow cavities on the surface of the composite sample, which increased its water sorption capacity.

3.2 Fourier Transform Infra-red Spectroscopic Analysis (FTIR)

The effect of sodium hydroxide treatment on the fluted pumpkin fiber surfaces was observed using FT-IR Spectroscopy. The comparison of the representative FTIR spectra of the untreated fluted pumpkin fiber and the treated fluted pumpkin fiber (Fig. 7a–7b) show a reduction in O-H stretching intensity. Also, shifting of the peaks from 3293cm⁻¹ and, 3283cm⁻¹, could be observed for the untreated sample, the alkali treated sample, at the wave numbers mentioned above. This is likely due to the breaking of hydrogen bond between the O-H groups of cellulose and hemicellulose molecules (Saha et al., 2010). Peaks at 2920cm⁻¹, and 2857cm⁻¹,

predominantly arise from C-H stretching (Jonoobi et al., 2010). The absorption peak at 1721cm⁻¹ is associated to carbonyl C=O stretching of acetyl groups of hemicellulose (Haque et al., 2009). The absorbance peak at 1441cm⁻¹ (Saha et al., 2010) were assigned to -CH₃ asymmetric deformation of lignin. However these peaks are detected in the alkaline treated sisal fiber very weakly this weakening indicates the removals of lignin. After the mercerization process, the bands at 1718cm⁻¹ and 1245cm⁻¹ attributed to the stretching vibrations of C=O and C-O groups respectively disappeared these kinds of groups are present in lignin and hemicellulose structures (Fávaro et al., 2010). "Increase in peak intensity at 1022cm⁻¹ suggested that NaOH treatments increase hydroxyl group concentration on the fiber, which would promote more active sites for fiber/matrix interface" (Mwaikwambo and Ansell, 2012).

3.3 Scanning Electron Microscopy (SEM)

Fig. 8a and 8b showed the tensile fracture surface of treated and untreated fluted pumpkin fibers reinforced polyester composite samples. As shown in Fig. 8a, it was found that the surface of the treated tensile samples had wrinkles, many gaps, micro-voids on the fiber surface, a little fiber pullout from the bundle. The micro-voids and spaces on the treated fiber surface

provide access to the polyester resin that showed good adhesion between fiber and matrix because of fiber treatment. Fig. 8b showed the tensile tested specimen of untreated fluted pumpkin fiber composite enclosed by impurities such as pectin, lignin, hemicelluloses, and waxes on their surface. These surface impurities

reduced the area of contact between fluted pumpkin fiber and resin. Therefore, the high values of tensile strength reported for treated composites in this study may be due to the rougher surfaces created on the fiber surfaces that created a more interlocking site for adequate interpenetration of the resin into the fiber.

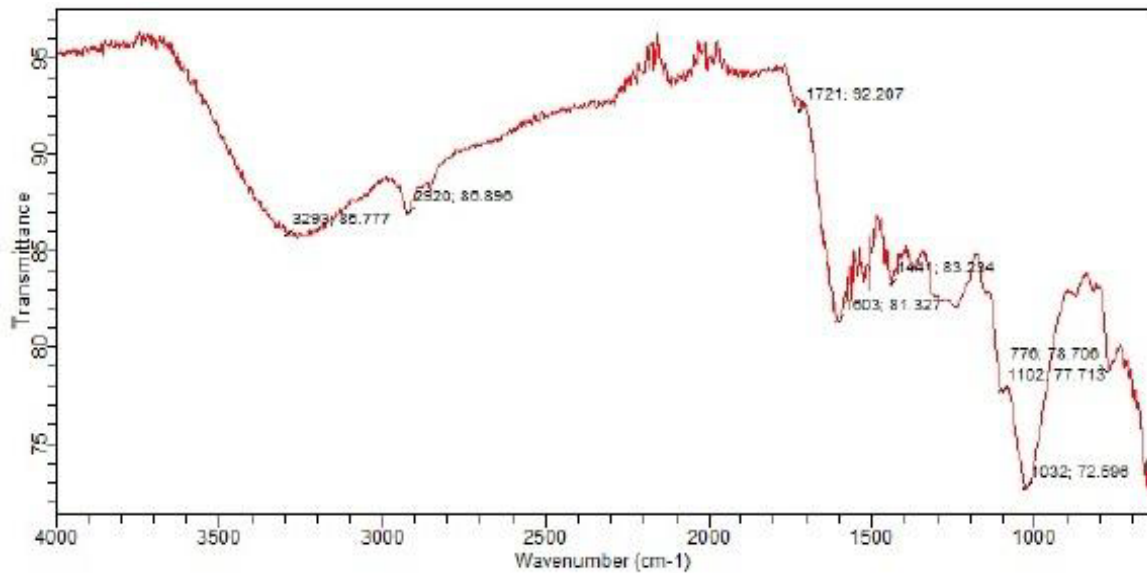


Fig. 7a. FTIR spectra of untreated fluted pumpkin fiber

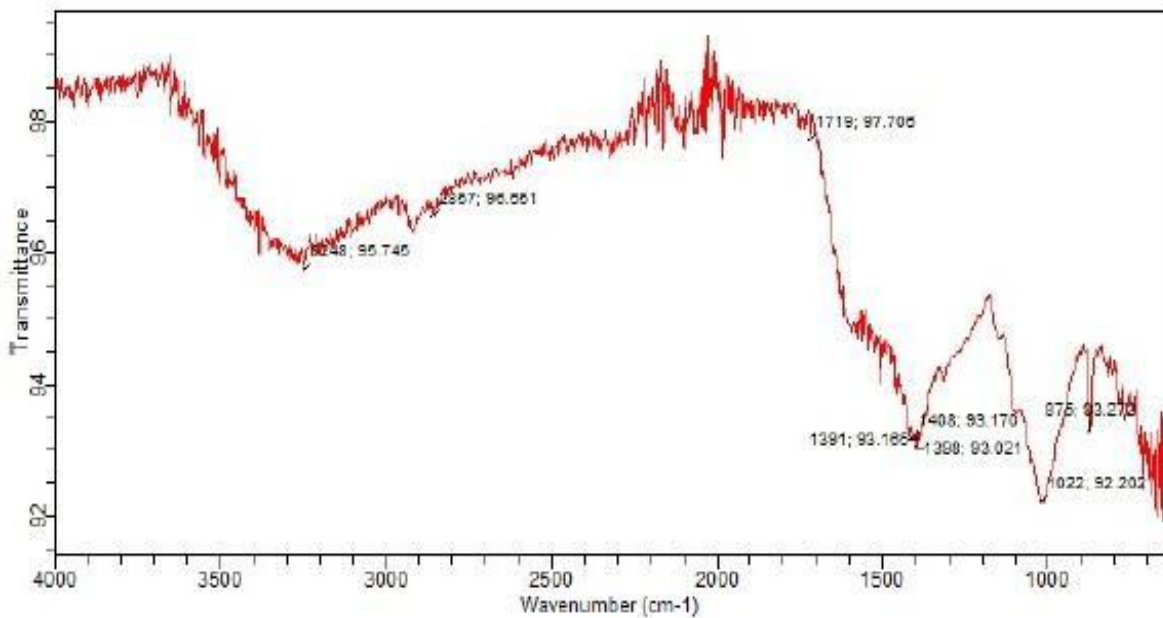


Fig. 7b. FTIR spectra of sodium hydroxide treated fluted pumpkin fiber

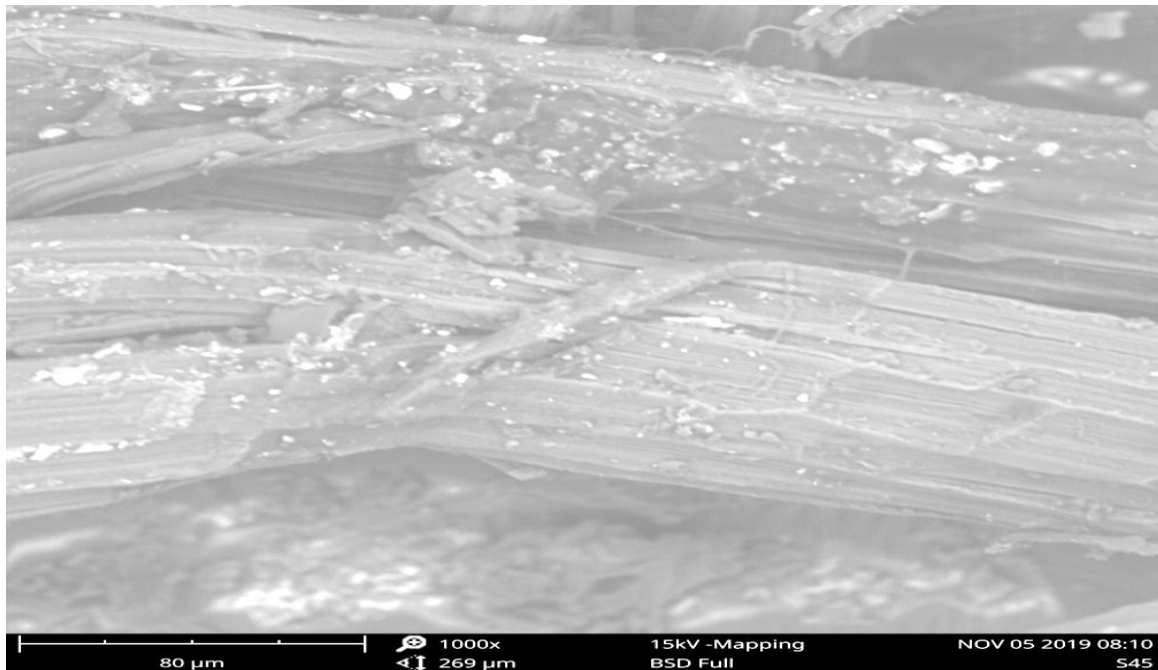


Fig. 8a. SEM image of fracture surface of treated fluted pumpkin fiber polyester composites under tensile condition

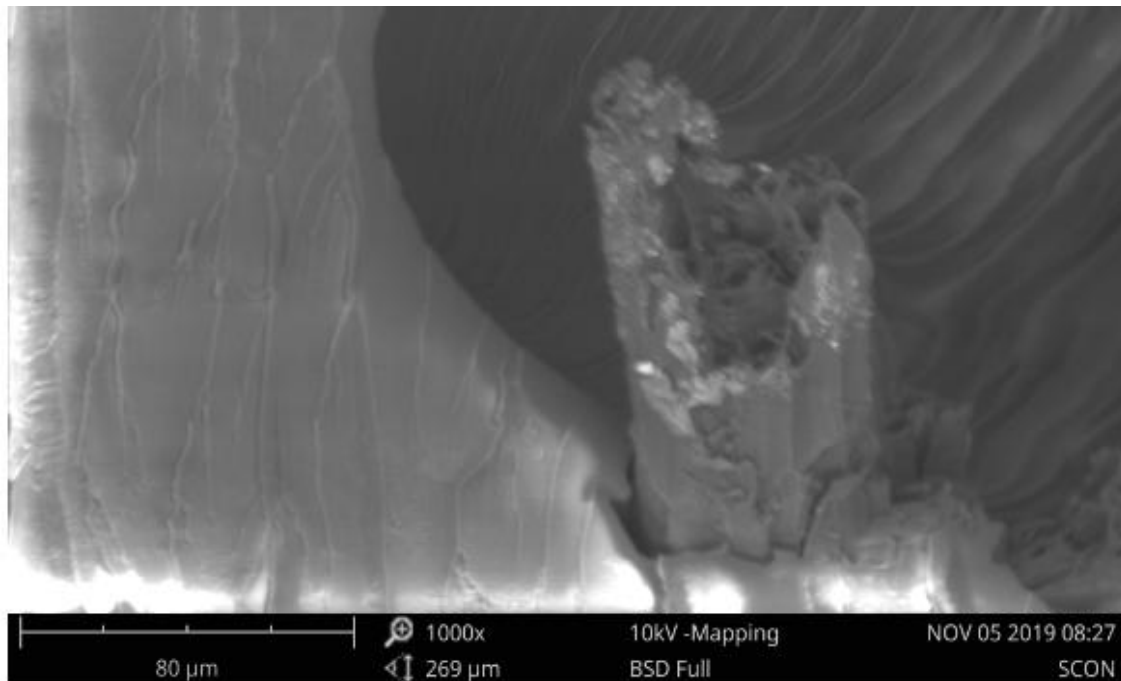


Fig. 8b. SEM image of fracture surface of untreated fluted pumpkin fiber polyester composites under tensile condition

4. RELEVANCE OF THE RESEARCH

The study's primary contribution is reporting that fluted pumpkin stem fibers are viable, eco-friendly, alternative raw materials of low cost for

the production of polyester composites. The evaluation of its mechanical and water absorption properties indicate its potential as engineering material. The study shows that improved properties can be achieved through the

alkali treatment of the fluted pumpkin stem fibers at the required temperature, thus an application of the composite produced could be limited to non-structural use such as ceiling boards, electronic, food packaging materials and parapet walls.

5. CONCLUSION

This study has established that chemical treatment is essential for the interfacial compatibility and improvement in mechanical and water absorption properties of fluted pumpkin stem fiber reinforced polyester composites. The enhancement is because of wrinkles, many gaps and micro-voids the treatment created on the fluted pumpkin fiber surface at the different immersion temperatures, which provided access to polyester resin interpenetration. It is worth mentioning that this study has shown that the immersion temperatures of sodium hydroxide treated fluted pumpkin fiber will affect the tensile strength, tensile modulus, flexural strength, impact strength, compressive strength, and water absorption capacity of the composites. Fourier transform Infra-red Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) analysis conducted on the untreated and treated fluted pumpkin fiber polyester composites supported to the improved effect on the tested properties of the composites. Higher temperature causes excessive peeling, deteriorating the fiber by damaging the cellulose that reduces the strength of the composite.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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