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Extraction of Fibers from *Saccharum munja* Grass and Its Application in Composites

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ABSTRACT: With growing environmental awareness, ecological concerns and new legislations, natural fiber-reinforced plastic composites have received increasing attention during the recent decades. The natural fiber composites have many advantages over traditional glass fiber composites, including lower cost, lighter weight, environmental friendliness, and recyclability. This article reports the findings of the studies done on a new fiber, hitherto unexplored, extracted from *Saccharum munja* grass. The extracted fibers were further treated using sodium hydroxide to improve its performance in composites. Both treated and untreated fiber-reinforced composites were prepared by hand lay-up process using unsaturated polyester resin. Mechanical properties and thermal behavior of the composites were evaluated. The improvement in properties was found for alkali-treated fiber composites. © 2014 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2014**, *131*, 40829.

KEYWORDS: cellulose and other wood products; composites; mechanical properties

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INTRODUCTION

Fiber composite materials such as glass fiber, carbon fiber, and aramid have been widely used in automotive, marine, aerospace, aircraft industries, and are now being used in structural and infrastructure application such as windmill, roof, bridge, girder, railway sleepers, and floating river walkway.[1] Because of their excellent tensile, toughness and thermal resistance properties, and their hydrophobic nature, composite has variety of applications. Various types of composites can be engineered according to their applications. But limitations for the fibers in vogue are their high cost, nonrecyclability or biodegradability, and supply chain management. These limitations forced researchers to pay attention to more eco-friendly fibers. It was found that the use of natural fibers can overcome the limitations.

Natural fibers provide numerous benefits such as low cost, light weight, high specific modulus, and no health hazards of composites reinforced with synthetic fibers such as glass, carbon, and aramid fibers. These advantages of natural fiber composites place the natural fiber composites among the high-performance composites having economical and environmental advantages.[2]

A hurdle in the use of natural fibers as reinforcement in composites is that it has insufficient interface quality with the polymer matrix. The insufficient interface can be owing to the presence of lignin and other covering matter on the natural fibers. This makes the fibers inaccessible for matrix bonding. The surface modification of the fibers by chemical treatment is the solution worked on and is one of the largest areas of current research.

Different studies have been carried out on different fibers to overcome the hurdle. Hemp, sisal, jute, and kapok natural fibers were modified by using mercerization and acetylation and improved the performance of natural composites by promoting better fiber to resin bonding.[3] The effect of alkali treatments on the flexural properties of polyester matrix composite reinforced with alfa fibers was studied and it was found that flexural strength and flexural modulus improved by 23–57 MPa and from 1.16 to 3.04 GPa, respectively, for 10% of NaOH for 24-h processed alfa fiber composite compared to the untreated fiber composite.[2] Woven bamboo mat was treated with maleic anhydride, benzoyl chloride, benzyl chloride, and flexural properties of maleic anhydride-treated bamboo polyester composites were improved by nearly 50%. The tensile strength and modulus of benzoylated bamboo fiber polyester composite improved by 71 and 118%, respectively.[4]

The variety of natural fibers and their availability depends on the richness of flora and fauna of the place. India is a country

with rich and abundant flora and fauna. There are various natural resources available which show potential use as reinforcement in composites. Grass is in plethora all over the country. There are various types of grasses available which have very good potential to use as reinforcement in composites.

Munja is one such grass which shows a very good potential to be used as reinforcement. It is commonly known as Kana or sarkanda or Munja. The botanical name of this grass is *Saccharum m unja*. It is distributed over North India and North West India and it is also found in Pakistan and Afghanistan. This grass is found in arid areas and along river banks in India. The grass is tall, panicles silky, and greenish brown. The grass grows in excess and up to 7 feet in height. It is planted in field dividing area because it forms extensive root network that binds the soil/pebbles and prevents soil erosion from fields. This grass cannot be used for cattle feed because the leaf blades can wound the cattle. It is used to make fire during winters or also used to make ropes, brooms, and huts. Overall, the grass does not have any notable scale commercial application.[5]

In this research, the fiber obtained from *S. munja* grass was used as reinforcement. This grass has neither been used previously as textile fiber nor as reinforcement in the composite. In our research study, a thriving attempt has been made to separate fibrous material (*S. munja* fiber) from *S. munja* grass and use of *S. munja* fiber as a reinforcing material in composites. Because of the newness/originality of the obtained fibers, the properties of fiber are studied prior to its application in composite. It was found that *S. munja* has very good reinforcing properties. The composites formed seem to have potential to supplement common fiber-reinforced composites.

EXPERIMENTAL

Extraction of Fibers from *S. munja* Grass

As shown in Figure 1(a), *S. munja* grass is grown on the border of fields to avoid soil erosion. This grass was obtained from a village in East Rajasthan, India. The grasses were cut from bottom and left open in sun light for 15 days to dry at a climatic temperature of 35°C. The stem of the grass (known as sarkanda) was separated from the leaves manually. The culms (outer covering of the stem) were separated manually from the dried stems. The dried stem can be used for making decorative items or furniture. Then culms were further dried in sun light for 7 days. The bundle of culms is shown in Figure 1(b). This bundle was then beaten with a wooden hammer to convert culms into fibers. Thus, *S. munja* fibers (SMF) were extracted from the grass. The bundle of fibers obtained from *S. munja* grass is shown in Figure 1(c).

Treatment of Fibers

SMFs extracted from the grass were cut to a length of 200 mm and treated with 5% of sodium hydroxide for 2, 4, 6, and 8 h in water bath using material to a liquor ratio of 1 : 30 on weight basis at room temperature. Then they were washed multiple times to remove soluble impurities and remaining sodium hydroxide. After washing, treated fibers were neutralized with acetic acid and dried at room temperature for 48 h. After drying, combing was carried out for unidirectional alignment of fibers. The difference in weight of the fibers before and after NaOH treatment was 22.8% for 8-h alkali-treated samples. Before fabrication of composite, fibers were dried in an oven at 105°C for 2 h. The complete removal of moisture from fibers was confirmed by constant weight of the fibers. These fibers were directly used for the fabrication of composites. Tensile and thermal properties of the fibers were studied and reported.

Composite Fabrication

A stainless steel mold of size 200 mm × 150 mm × 3 mm was prepared and the dried fibers were laid aligned unidirectional. In all, 10% of weight fraction of fibers was used to make composite sheet. In brief, 3% of cobalt accelerator and 1% of methyl ethyl ketone peroxide hardener were mixed with sufficient quantity of unsaturated polyester resin (UPR). This mixture was then poured into the mold. Plastic squeeze was used for uniform application. A silicon roller was rolled to remove entrapped air from the fibers. The mold was left for 24 h at room temperature for curing of composite. After 24 h, the unidirectional fiber-reinforced composite sheet was separated from mold. Specimens for tensile, flexural, and impact strength testing were cut on vertical cutting machine according to the required dimensions. In total, five types of composites were made and characterized. For each type of composite, three batches of fiber samples were used.

Fourier Transform Infrared Spectroscopy

Fiber samples were grinded and converted into powder form. This powder was mixed with KBr in the ratio of 1 : 9 without

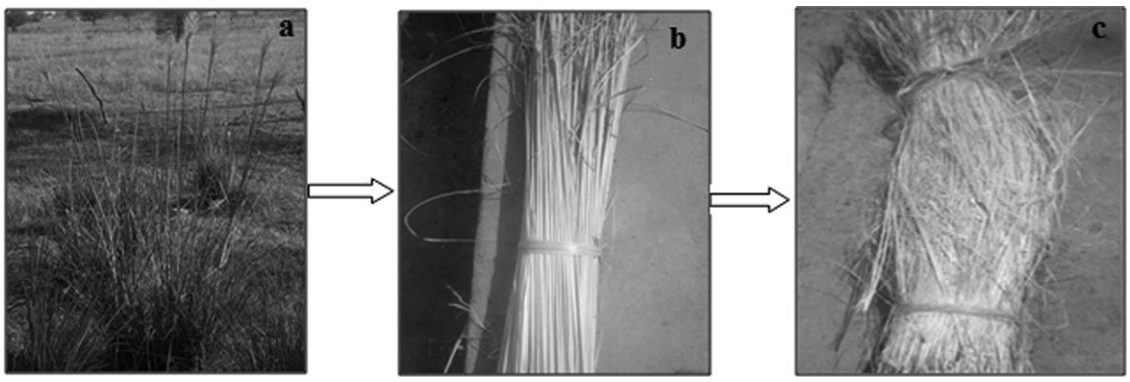

Figure 1. Extraction of fibers from *S. munja* grass. (a) *S. munja* plant. (b) Bundle of culms of *S. munja*. (c) Fibers of *S. munja*.

drying and pellets were prepared. The Fourier transform infrared spectroscopy (FTIR) spectra of the untreated and the alkali-treated samples were recorded in the region of 3750–700cm^{-1}on a Shimadzu 8400S FT-IR instrument with 32 scans in each case at a resolution of 4 cm^{-1}.

Mechanical Testing

Tensile, flexural, and izod impact tests were carried out according to the ASTM Standard. For each test and type of composite, five specimens were tested and the average values are reported. Tensile tests were conducted according to ASTM D 638 using a Universal Testing Machine (Lloyd LR) at a crosshead speed of 5 mm/min. Flexural tests were carried out according to ASTM D 790 using the same testing machine mentioned above at a crosshead speed of 2 mm/min. Izod impact tests of notched samples were conducted according to ASTM D 256 using a Tinius Olsen Impact Tester (model 104) which has metallic hammer. Tensile strength of fibers was measured according ISO 5079 method on Tinius Universal Testing Machine at a crosshead speed of 30 mm/min.

Scanning Electron Microscopy

The interfacial bonding between SMFs and UPR matrix in manufactured composites was examined using a scanning electron microscope (SEM) (model JEM 5400). Fibers samples were mounted on the metallic stub and coated with gold ions for 6 min and thickness of gold coating was approximately 10 Å. The accelerating power in the SEM was kept in the range of 3 kV.

Thermogravimetric Analysis

Thermogravimetric analysis (TGA) of fiber samples and composite sample was done with Shimadzu 8400S and the degradation of the sample with respect to temperature was studied. The experiment was done by using 8 g of samples from 30 to 500°C with a heating rate of 20°C/min for fiber sample and 10°C/min for composite sample. The nitrogen gas was used as purge gas.

RESULTS AND DISCUSSION

Characterization of SMF

SMFs are characterized for tensile and thermal properties. The surface characteristic of the fibers is also reported to study the effect of alkali treatment on the fiber surface.

Tensile Properties. The tensile properties of SMFs obtained are shown in Figure 2. A comparison of untreated SMFs with alkali-treated fibers can be seen in this figure. As it is observed, after alkali treatment tensile strength of the fiber was increased up to 16% after 8-h alkali treatment in comparison with untreated fiber. After NaOH treatment, impurities and some part of hemicelluloses and lignin were removed, which increase the strength of fiber.

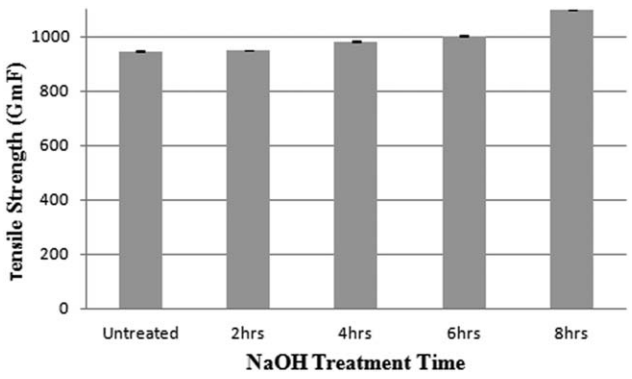

Figure 2. Effect of NaOH treatment on tensile strength (kgf) of fibers.

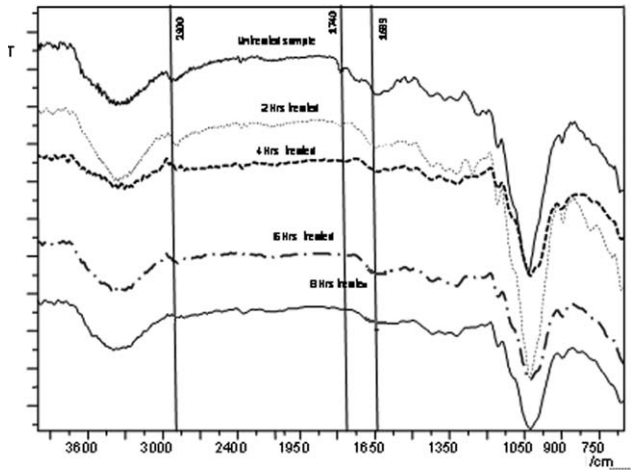

Figure 3. FTIR spectra of untreated and treated SMFs.

Fourier Transform Infrared Spectroscopy. The spectra of untreated SMFs and alkali-treated SMFs are shown in Figure 3. The untreated SMF shows a peak at 1740 cm^{-1} assigned to C═O, which disappeared when the fibers were treated with the NaOH aqueous solution. The observation shows the removal of hemicelluloses by alkali treatment. The vibrational peak around 2900 cm^{-1}, belonging to the C—H stretching vibration in cellulose and hemicelluloses, decreased after NaOH treatment. It indicates that part of the hemicelluloses was removed.[6] Furthermore, the peak around 1633 cm^{-1} for C—O assigned to the ester and ether crosslink between cellulose and lignin, or cellulose and hemicelluloses, disappeared during NaOH treatment. This is owing to the removal of acid, lignin, and other fiber constituents.[7] Though these groups attached through lignin contents are removed, their presence in the cellulose structure of fiber shows some intensity around that region.

Scanning Electron Microscopy. Figure 4(a) shows the SEM micrograph of untreated SMFs. The impurities were clearly observed on the surface of untreated fiber. The modification of fiber by sodium hydroxide (NaOH) effectively removed the impurities from the fiber surface. Figure 4(b–d) shows the SEM micrograph of the surfaces of alkali-treated SMFs for different time periods. It was found that the treated fiber surfaces are cleaner than untreated fibers.[8] Figure 4(e) shows the SEM micrograph of 8-h-treated SMFs that presented the cleanest fiber surface. It was observed that almost all impurities have been removed from the fiber surface. The increase of soaking time in NaOH showed more clean and rough surface of the fiber.

Thermogravimetric Analysis. The fiber degradation process was divided into four stages: moisture evaporation, hemicelluloses degradation, cellulose degradation, and lignin decomposition.

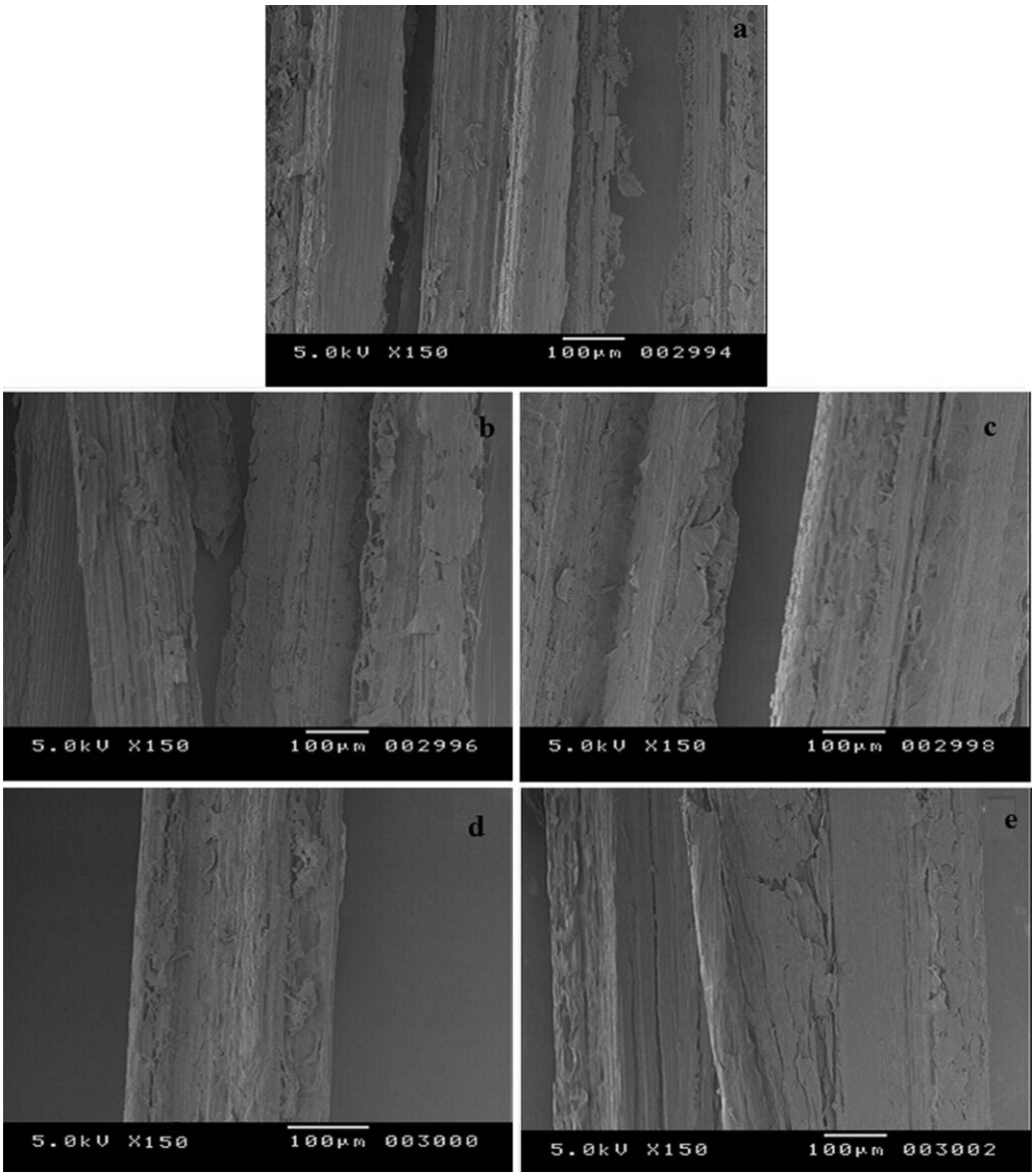

Figure 4. SEM image of SMFs. (a) Untreated SMFs. (b) Two-hour alkali-treated SMFs. (c) Four-hour treated SMFs. (d). Six-hour alkali-treated SMFs. (e) Eight-hour alkali-treated SMFs.

Moisture is present in the fiber in two forms: free water and linked water. Free water is generally attached on the fiber surface and evaporates at lower temperature (25–150°C). However, the linked water forms chemical bonds with the hydroxyl groups present in hemicelluloses and lignin, and evaporates at higher temperatures.[9] After the removal of free water, in the temperature range of 150–500°C, the degradation process begins in the cellulose, hemicellulose, and lignin constituents. The different degradation stages during the thermal degradation of fiber are shown in Figure 5(a,b). It can be observed that the cellulose decomposition process (weight loss) was very mild until around 250°C. Then, a sharp and large weight loss occurred at about 350°C. In the temperature range of 250–350°C, the cellulose was entirely decomposed owing to the breaking down of its molecular structure. This was owing to the fact that most of the cellulose structure was crystalline, which is strong and resistant to hydrolysis. The crystalline structure contains strong intramolecular and intermolecular hydrogen bonds which requires higher energies to be broken down.[10] The decomposition of hemicelluloses is at about 200°C.The thermal decomposition of lignin took place over a broader temperature range (150–450°C) than the cellulose and hemicelluloses. During heating, lignin formed aromatic hydrocarbons, hydroxyl, and phenolic compounds. All these components contained —OH groups which became unstable at higher temperatures. These unstable groups underwent further reactions among themselves which resulted in structural rearrangements owing to radical–radical interactions.[11] It was found that thermal degradation temperature (weight loss, 50%) of untreated SMFs was 310.25°C and for alkali-treated (5% NaOH for 8 h) SMFs it was increased to 340.28°C. Alkali-treated fibers showed higher decomposition temperature compared to the untreated fibers.

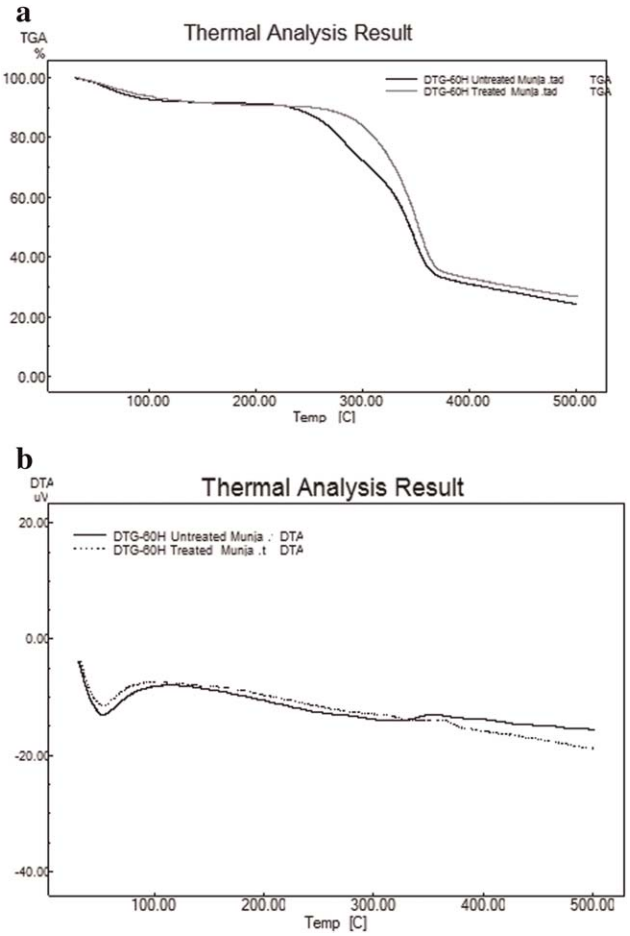

Figure 5. (a) TGA of SMFs. (b) Differential TGA of SMFs.

For the main fiber decomposition region (250–350°C), the alkali-treated fibers had less weight loss than the untreated fibers. These results indicate that alkali treatments removed portions of hemicelluloses and lignin constituents from the fiber. Owing to this fact, the decomposition process mainly occurred on the cellulose which in turn increased the overall degradation temperature of the treated fibers.

Characterization of Composites

Tensile Properties. As shown in Figure 6, the tensile strength of the composite is greater than the standard (neat resin), which means that there is definitely an improvement in the tensile strength with the inclusion of fiber material. Furthermore, with increase in the treatment time of NaOH from 2 to 8 h, the tensile strength value also increased. The tensile strength of untreated SMF composite is 59.13 MPa and treated SMF composite (for 8 h) is 80.69 MPa. Tensile strength of alkali-treated SMF composites increased by about 36.46% as compared to untreated SMF composites. Tensile strength of 6-h alkali-treated fibers and 8-h-treated fibers is almost similar. This could be because of no further improvement of fiber properties after 6-h treatment; if treatment time is increased for more than 8 h, then there are chances of deterioration in fiber strength and other fiber properties. Hence, from the results it can be said

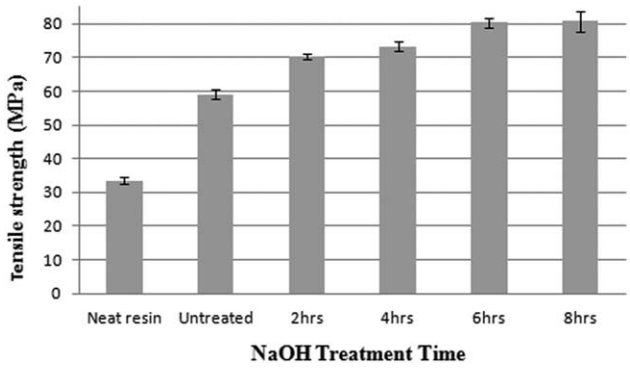

Figure 6. Effect on NaOH treatment on tensile strength of SMF/UPR composites.

that alkali treatment time of 6 h gives good and optimum results.

The improvement in tensile strength of SMF composites was owing to the removal of impurities because of alkali treatment. Alkali treatment leads to fibrillation which causes the breaking down of the fiber bundle into smaller fibers which results in decrease in fiber diameter and thereby increases the aspect ratio. Therefore, the development of a rough surface topography and enhancement in aspect ratio offers better fiber–matrix interface adhesion and an increase in mechanical properties.[12]

Results of tensile modulus are shown in Figure 7. Modulus of untreated SMF composites was found to be 2663 MPa. After the treatment time of 8 h, tensile modulus of SMFs was 3836.2 MPa which shows improvement in modulus of alkali-treated SMF composite. The increase in tensile modulus is 44%. The increase in tensile modulus shows that the stiffness of the composite is increasing with the use of fibers as reinforcement. UPR with SMFs as reinforcement is becoming stiffer and could withstand higher stress at the same strain portion. The treatment with alkali shows further increase in tensile modulus. Low stiffness means that fibers are unable to absorb the tensile forces owing to the inefficiency of bonding between fiber and matrix.[13] Thus, the results show better interfacial bonding between UPR matrix and SMFs after alkali treatment of fibers. This means that the fiber served well as reinforcement and shared the major load for matrix.

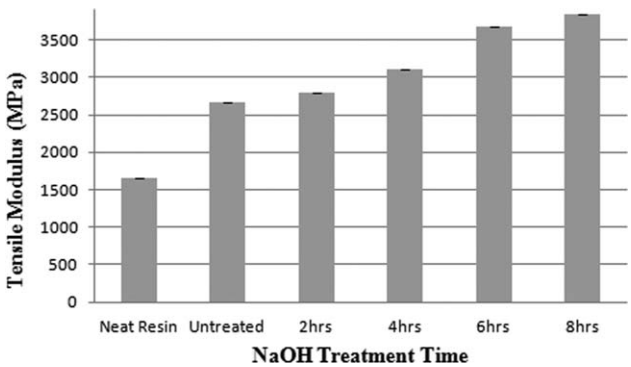

Figure 7. Effect on NaOH treatment on tensile modulus of SMF/UPR composites.

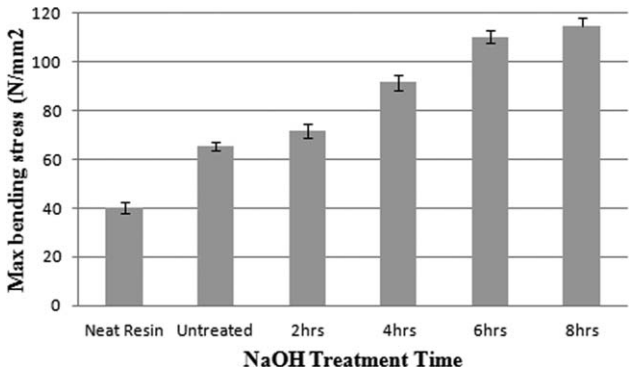

Figure 8. Effect of NaOH treatment on flexural strength of SMF/UPR composites.

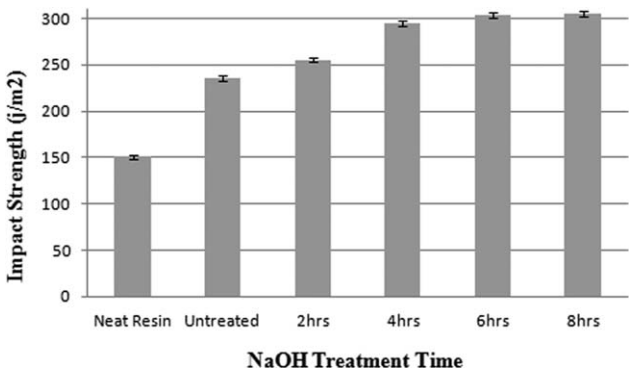

Figure 10. Effect of NaOH treatment on izod impact strength of SMF/UPR composites.

Flexural Properties. The flexural strength of untreated SMF composite was 65.34 N/mm^2 and after alkali treatment of the fibers for 8 h, flexural strength of composite was 114.93 N/mm^2 as shown in Figure 8 which is 76% improvement in flexural strength. The observations made earlier for the tensile strength on the effect of fiber–matrix adhesion are also seen clearly here. The fiber surface treatments also have effect on the flexural strength. The increment of interfacial area of contact was beneficial for the flexural strength. This indicates that the increase in area of contact between the fiber and the matrix improves the level of adhesion.[14]

Figure 9 shows the flexural modulus of SMFs/UPR composites for untreated and the alkali-treated fibers. It is seen that the improvement of the flexural modulus emerges when the time of treatment increases. The modulus of untreated SMF composite is 4106 MPa and alkali-treated SMF composites is 6452 MPa, and hence improvement in modulus is about 57%. The flexural modulus is the stiffness parameter. After the alkali treatment, SMFs provide improved crystallinity of cellulose and remove part of the hemicellulose and lignin content beyond the removal of impurities. This suggests that fiber becomes relatively ductile after the removal of some hemicellulose and lignin. This can result in higher fiber stiffness owing to the increased crystallinity of hard cellulose.

Izod Impact Strength. Figure 10 shows that impact strength of untreated SMF composite was found to be 236.1 J/m^2, whereas for 8-h alkali-treated SMF composites, it is 305.4 J/m^2, showing improvement of about 29%. Impact strength of neat UPR is 151 J/m^2 which is very less than the fiber composites. Fibers play a vital role in impact strength of the composites as they act as stress-transferring medium and resist crack formation.[15] SEM images of SMFs before alkali treatment show that the surface of fiber is covered with a layer of substance, which may include pectin, lignin, and other impurities. After alkali treatment, important modification done is the disruption of hydrogen bonding in network structure, thereby increasing surface roughness. The removal of lignin made more cellulosic sites available for contact with the resin which led to better mechanical interlocking.[16] Higher impact strength of alkali-treated SMF specimen was owing to good interface bonding between fiber and matrix.

Thermogravimetric Analysis. As shown in Figure 11, the UPR matrix is thermally stable up to 394°C. The composites started degrading at lower temperatures than UPR. This is the result of the presence of SMFs which degrades at lower temperatures. The degradation of the polymer backbone in the 8-h alkali-treated composites sample occurs at a higher temperature than the other alkali-treated samples. The alkali treatment brings about an increased surface roughness in the fiber which results in better mechanical interlocking between the filler and the matrix. As a result, the fiber may retard the movement of free radicals formed during the initiation of degradation, or it may interact with

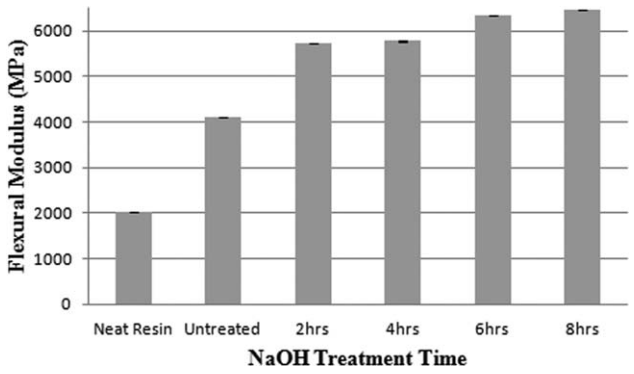

Figure 9. Effect of NaOH treatment on flexural modulus of SMF/UPR composites.

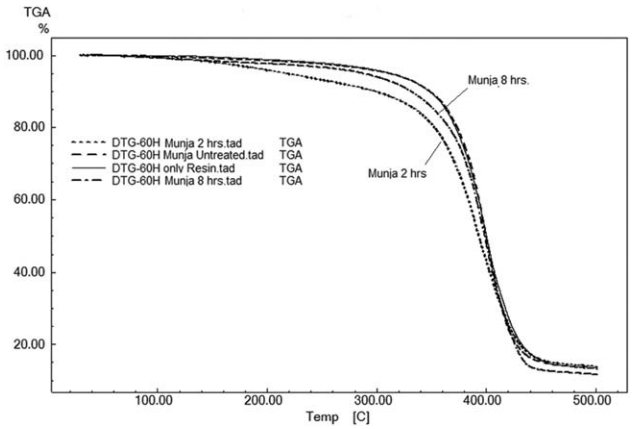

Figure 11. TGA of SMF/UPR composites.

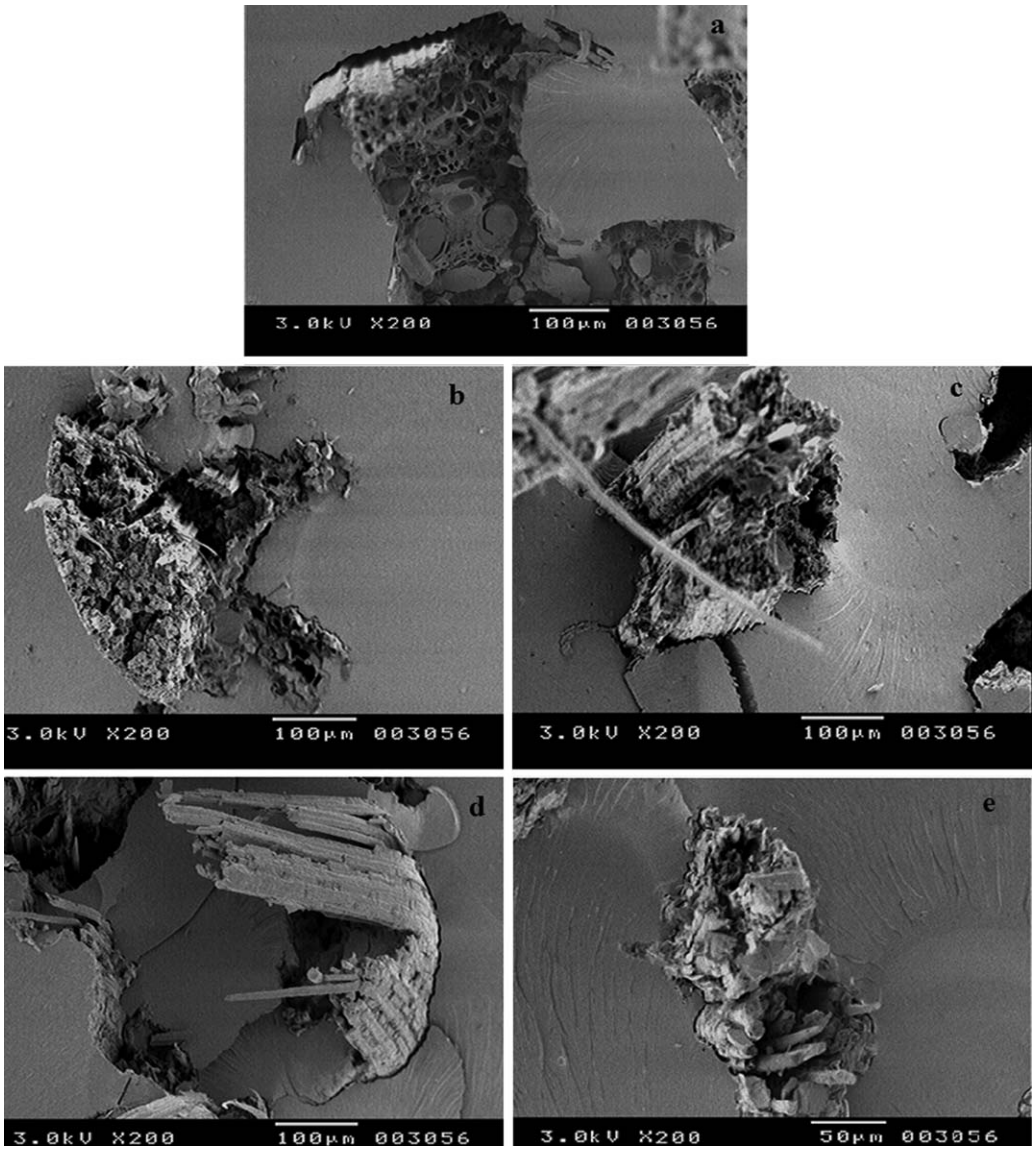

Figure 12. SEM image of SMF composites. (a) Untreated SMF composite. (b) Two-hour alkali-treated SMF composite. (c) Four-hour treated SMF composite. (d) Six-hour alkali-treated SMF composite. (e) Eight-hour alkali-treated SMF composite.

volatile degradation products and in the process slow down their diffusion out of the sample. Thus, the alkali treatment increases the thermal resistance of the fiber composite.

Scanning Electron Microscopy. The results of scanning electron microscopy of impact fractured surface of SMF composites illustrates the improvement of the interaction between the fiber and the matrix. SEM images/micrographs of the treated fiber composites show that the fibers are closely adhered to the matrix. Comparing Figure 12(a–e), it is observed that the number of pores in untreated fiber composite is higher than that in composite made using alkali-treated fibers. Also, the pore size is higher. Thus, the presence of pores is indicative of weak interaction between untreated fiber and UPR resin. Although SEM images of treated fibers indicate that the distance between fiber and matrix has reduced to large extent, resulting in improved interfacial bonding between fiber and matrix. Improved interfacial bonding between the fiber and the matrix can be attributed to the removal of lignin, hemicelluloses, and other structural effects, which causes rough surface morphology of fibers. This finding corresponds with the results in improvement in mechanical properties. Thus, alkali treatment results in significant change in morphology of the fiber surface and is effective for increasing the available contact between the fibers and the UPR matrix. The enhancement of impact strength of the treated fiber composite as compared to untreated fiber composite can also be attributed to the flexibility of the interface molecular chains, resulting in comparatively greater energy absorption.

CONCLUSIONS

Use of natural fibers as reinforcement in composite is a very important and significant approach in composite industry and SMF has potential to be one such fiber. Tensile properties, flexural,

and impact strength of the fibers show comparable values with the reinforcing fibers used today. There are various ways of chemical modification of fibers to improve the performance of fibers in composite and the modification of SMFs by alkali treatment has shown improved fiber performance in composites. Commercialization of the fibers and their use in composite can give complementary income in agriculture at minimal investment costs.

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