Polymer Modified Grass Fiber, Part 1: Characterization of Grass Fiber and Assessment of Properties of Polymer Modified Fiber

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ABSTRACT: Natural fibers are used for reinforcement of ecofriendly green composites. These grass fibers have wide range of applications for the preparation of various domestic goods and handicraft items. But the chemical composition of the grass fiber has not been reported; therefore composition analysis of grass fiber was carried out. Morphology of the dry grass was studied by using scanning electron microscopy. Since the grass fiber contains around 22% water-soluble matters, we have studied its influence on the tensile strength and aging characteristics. The tensile strength of the grass fiber was measured before and after water leaching. This study has shown a definite scope for the suitability of grass fiber in composite applications. To improve the strength of grass fiber, modification with various polymers such as P-F resin, polyurethane and acrylamide was carried out. Moisture absorption property of unleached, water-leached and different polymer modified fiber was also evaluated. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 104: 1095–1103, 2007

Key words: fiber; mechanical properties; polymer modification

INTRODUCTION

Natural fibers are forecast to grow 15–20% per year in automotive applications, and up to 50% or more per year in some building products. Around 75% of this volume is wood fiber; the remainder is composed of agricultural fibers such as kenaf, jute, hemp, flax, banana, pineapple, and sisal. These fibers including coconut coir, grown in many areas, are the major source of natural fibers. Sisal, pineapple, and banana fibers are cellulose-rich (>65%) and show tensile strength, modulus and failure strain comparable with other cellulose rich fibers like jute and flax whereas the lignin-rich (>40%) coir fiber is relatively weak possessing low failure strain. These fibers are extensively used for cordage, sacks, fishnets, matting and rope, and as filling for mattresses and cushions (e.g., rubberized coir).[1]

Various studies have been made to understand the structure, mechanical properties, and effect of chemical treatments on jute,[2,3] coir,[4] sisal[5] etc. Rials and Wolcott[6] have reviewed the physical and mechanical properties of agro-based fibers such as flax, hemp, jute, kenaf, ramie, cotton, and other grass fibers such as sugarcane bagasse, bamboo, esparto, and sabai.

Rowell[7] has elaborated in a review the various opportunities for composites from agro-based resources and English et al.[8] have shown how the agro-based fibers are processed into composites.

To improve the strength of grass fiber polymer modification is necessary. In general, the lignocellulosic materials are hygroscopic and undergo degradation when exposed to various natural degrading agencies. Rowell[9] has extensively reviewed the chemical modification of agro-based fibers. From the published literature it has been observed that for improving/altering the properties, agro-based fibers have been modified in three major ways: modification with chemical compounds having reactive functional groups, modification through composite preparation using some matrix polymers and chemical grafting of suitable polymers by graft copolymerization. It has been known that certain polymer, e.g., phenol-formaldehyde, generate chemical bonding with lignocellulosic reinforcements. Because of the high polarity of phenol-formaldehyde resin, strong dipole-dipole and Vander Waals forces are developed and chemical reactions occur between fiber components and the resin. Bisanda and Ansell[10] studied the effect of silane treatment to improve the adhesion between sisal fiber and epoxy matrix. Mukhopadhyay et al.[11] studied the influence of poly(methyl methacrylate) and poly(ethyl acrylate) grafting on the tensile properties of jute fiber. Graft copolymerization of vinyl monomers onto natural fibers have been studied using peroxydisulfate.[12–15] Agarwal and

Sreenivasan[16,17] have studied graft copolymerization of methyl methacrylate (MMA), acrylonitrile (AN), vinyl acetate (VA) and styrene onto jute fibers by gamma radiation. Mishra et al.[18] grafted MMA onto chemically modified jute fibers.

But nothing is reported in the literature about the characteristics and properties of the grass fiber (*Cyperus Tegetum* Roxb) of Cyperaceae family. This grass is an agricultural product of India. It grows in lateritic soil throughout the year preferably in the low land areas. Dried sticks are used for making domestic products like floor mats, window curtains and various other handicrafts. Such grass products have adequate strengths and are durable up to 2–5 years depending on the nature of handling and use. Like other natural products such as sisal, ramie, jute, and hemp containing long fibers that are being used for making composites, grass may also be used in place of them because of its good mechanical strength. So this investigation was planned to study the composition analysis and property evaluation for knowing its suitability in composite preparation. The modification of grass sticks with different polymers was carried out to see the influence of such modification on the properties of the polymer modified grass fiber.

EXPERIMENTAL

Materials

A fully grown matured sun dried grass stick (*Cyperus Tegetum* Roxb) is ∼ 75–80 cm long, ∼ 2–3 mm dia, yellowish in color with light greenish tint and specific gravity of 0.64. The grass fibers were thoroughly cleaned before any treatment. For composition analysis neutral detergent solution (NDS) and acid detergent solution (ADS) were used. The NDS is an aqueous solution of disodium ethylenediamine tetra acetate (E. Merck India), sodium borate decahydrate (Qualigens Fine Chemicals, Glaxo), sodium lauryl sulfate (S.D. Fine Chemical India), 2-ethoxy ethanol (Ranbaxy Laboratories) and disodium hydrogen phosphate (S.D. Fine Chemical India). The ADS is an aqueous solution of cetyl trimethyl ammonium bromide in $1N$ sulfuric acid (E. Merck India). For modification of grass with polymers water-leached grass was taken in all cases. Phenol (S. D. Fine Chem, India), 37–41% formalin solution (Qualigens, India), neopentyl glycol (NPG) (Fluka AG, Germany), polyethylene glycol (PEG 400) (E. Merck, India), toluene diisocyanate (TDI) (E. Merck, Germany), dibutyltin dilaurate (DBTDL) (Fluka AG, Germany), ceric ammonium nitrate (CAN) (E. Merck, India), sodium hydroxide (S. D. Fine Chem, India) were used as received. Acrylamide (S. D. Fine Chem, India) was crystallized from ethyl alcohol solution.

Water-soluble matters

Water-soluble components were estimated by immersing the weighed grass fiber in distilled water where fiber to water ratio was 1 : 300 for 24 h at 25°C. Finally the water-leached samples were dried at 50°C for 24 h and weighed. Total weight loss was expressed as percent water-soluble matters.

Cellulose and hemicellulose

Hemicellulose, cellulose and lignin were estimated following the method described elsewhere.[19] For estimation of these components the grass was refluxed separately with NDS and ADS followed by washing the insoluble part with hot water and acetone in each case. The insoluble parts obtained after treatment with NDS and ADS are termed as Neutral Detergent Fiber (NDF) and Acid Detergent Fiber (ADF) respectively. The hemicellulose content of grass fiber was obtained as a difference of NDF and ADF. The cellulose content of grass fiber was obtained as the difference of ADF and residue after extraction of ADF with 72% sulfuric acid.

Lignin content

The lignin content of grass fiber was obtained as the difference of residue after extraction of ADF with 72% sulfuric acid and ash obtained by igniting ADF.

Ash content

Ash content of grass fiber was estimated by burning a weighed amount of grass fiber on an alumina crucible in a muffle furnace at 750°C for 3 h. The remaining residue was expressed as percent ash.

Hydroxyl value

The hydroxyl value of grass fiber was estimated by a standard technique[15] using acetic anhydride-pyridine mixture and back titration of the acetic acid by standard potassium hydroxide solution. The hydroxyl value was expressed as mg KOH/g.

Infra red (FTIR) spectroscopy

FTIR study was carried out for grass fiber samples using a Nexus 870 Nicolet FTIR spectrophotometer. For FTIR analysis dry powdered sample was used.

Scanning electron microscopy

The surface and inner morphology of the dry and polymer modified grass fiber sample were evaluated by scanning electron microscopy (SEM) using JEOL,

TABLE I
The Composition Analysis of Dry Grass

Components	% by weight
Hemicellulose	21
Cellulose	15
Lignin	40
Water soluble matters	22
Ash	2

JSM 5800 both for water-leached and polymer modified samples.

Methods for modification of grass Fibers

Modification with phenol formaldehyde resin

Water-leached grass fiber sticks were soaked with the solution of phenol (1.0 mol), formaldehyde (formalin) (1.2 mol) and sodium hydroxide catalyst (2% with respect to phenol) for different time periods at room temperature (25°C) for 6 h. The resin content of the grass fiber was varied by varying the concentration of phenol and formaldehyde in the soaking solution keeping the same mole ratio of phenol : formaldehyde (1 : 1.2). The resin was formed *in situ* within the grass fiber matrix by heating the soaked and subsequently air dried grass stick at 150°C for 7 min. Polymer loading within the grass stick was varied by diluting the prepared (concentrated) resol with water to obtain resol solutions of different solid contents while the soaking time was kept constant for 6 h.

Modification with polyurethane resin

For treatment of grass fiber with polyurethane resin, initially a prepolyurethane was prepared from neopentyl glycol (0.5 mol), PEG 400 (0.5 mol) and TDI (1.5 mol). The prepolyurethane synthesis was carried out in two steps. In the first step, NPG was reacted with TDI in THF at 25°C for 1 h in presence of DBTDL catalyst (6 drops 0.1 mol). In the second step, after 1 h, PEG 400 was added and the reaction was continued for further 40 min. In typical batch, 0.52 g NPG, 2.0 g PEG, 2.61 g TDI, and 20 mL dry THF solvent were used for prepolyurethane synthesis. This prepolyurethane (10.07%) solution in THF was used for soaking the dry grass sticks. The grass sticks were then soaked for different time periods at 25°C to vary the resin content. After soaking for a specified time period the samples were cured in atmospheric moisture for 24 h followed by final curing at 80°C for 5 h. The self stability of this prepolyurethane is quite good and no change was observed on storage in a dry condition for 30 days.

Modification with acryl amide monomer

Grass sticks were modified with acryl amide monomer by grafting of *in situ* polymerized polyacrylamide onto grass fiber surface. For this modification the grass sticks were first soaked with aqueous solution of 0.05M CAN in 0.04 (N) HNO_3 for 30 min at room temperature followed by immersing the CAN soaked grass stick in 0.5% acrylamide solution under nitrogen atmosphere for polyacrylamide grafting. Here also to vary the resin content the CAN treated grass stick was kept immersed for two different time periods (1 and 2 h).

Tensile strength

Tensile strengths of different polymer modified grass stick specimens were evaluated with a KMI (India) tensile testing machine at a crosshead speed of 50 mm/min at ambient condition. Before testing, the specimens were kept in a desiccator for 2 days. The polymer modified grass stick (cylindrical) specimens (10 cm long and 0.2–0.3 cm dia) were gripped for measuring the tensile properties.

Moisture absorption characteristics of polymer treated grass stick

The moisture absorption characteristic of polymer treated grass stick samples was measured by keeping the samples in a chamber having relative humidity (RH) of 97%. The weight gain due to moisture absorption was expressed as %moisture absorption.

RESULTS AND DISCUSSION

Composition analysis

The composition of dry grass is given in Table I. Grass was found to contain about 22% water-soluble matters when it was leached in distilled water for 24 h at room temperature. Some sugars and soluble minerals may represent these water-soluble matters. Hemicel-
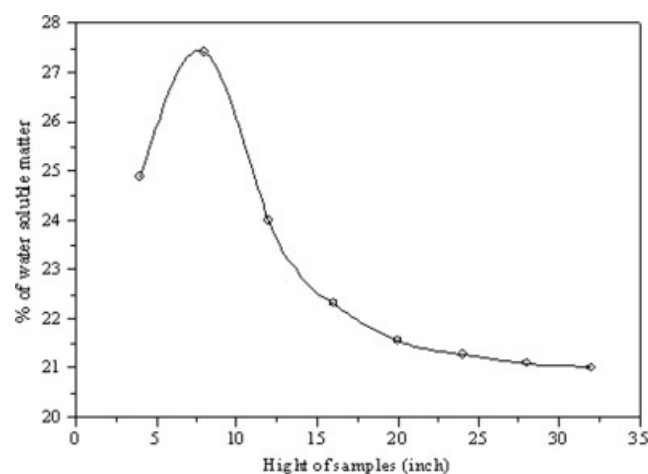

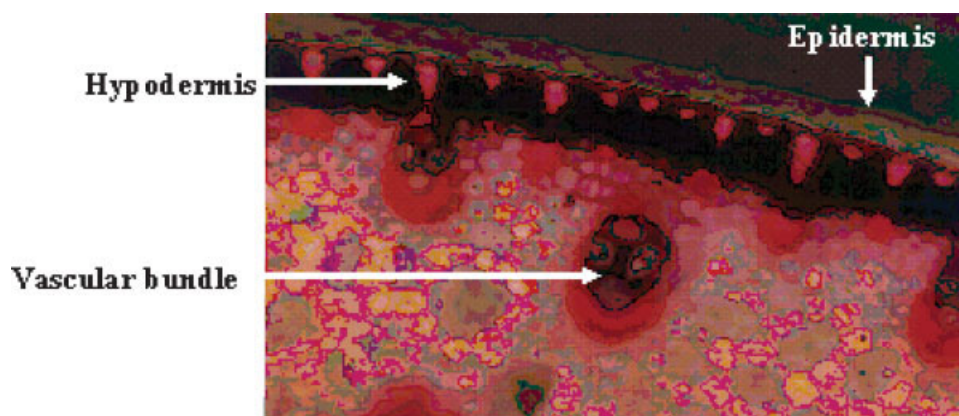**Figure 1** Variation of % water-soluble matter along the length of the grass fiber.

Figure 2 Optical micrograph of stained green grass fiber section. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

lulose, cellulose and lignin were estimated following the method described elsewhere.[19] It was also interestingly observed that the amount of water-soluble matter in the grass is not uniform along its length. Percent water-soluble matter gradually decreases from bottom to top along its length (Fig. 1).

The composition analysis data also show that there is quite a substantial quantity of lignin (40%) whereas the cellulose content is lower. Such high amount of lignin probably makes the grass so stiff. As is seen from Figure 2 that the maximum lignin is concentrated in the epidermis region stained by phlorogucinol. This lignin makes the outer skin of the grass rigid while the inner core contains a soft tissue. 15% cellulose in the grass remains dispersed in the lignin matrix representing the outer core of the grass as a natural composite. Water-soluble part of this grass is also quite high (22%). Although the components of the water-soluble part were not analyzed but when left exposed to ambient atmosphere showed fungal growth. This observation gave an important indication of the influence of such water-soluble matters towards performance life of the grass products.

FTIR spectroscopic analysis

FTIR vibrational bands of unleached and water-leached grass fiber samples are shown in Figure 3. In water-leached sample shifting of H bonded -OH stretching frequency value to the higher region indicates that intermolecular H-bonding decreases because of the removal of water-soluble sugars. Peaks at 2923 cm^{-1} correspond to C—H stretching in cellulose and hemicellulose present in grass fiber. Such peak is more intense in unleached sample than that in the water-leached sample. The decrease of peak intensity in water-leached sample is due to the removal of water-soluble portion from grass fiber. The peak intensity at 1735 cm^{-1} of water-leached grass fiber has

decreased due to removal of water-soluble part after water leaching. Asymmetric C—O—C stretching band at 1057 cm^{-1} indicates the presence of the chain of anhydroglucose ring, i.e., cellulose. The CH$_2$ bending vibration at 1440 cm^{-1} is due to the presence of lignin in both the water-leached and unleached grass fiber samples.

Strength of dry grass stick

It was thought that the strength might not be uniform along the entire length of the grass stick. Therefore, the tensile strength was measured at its different lengths from the root end to the top using five specimens at a time. The results of tensile strengths of the different portions of the grass stick along its length are shown in Figure 4. It is found from the figure that middle portion of the grass along its length has maximum strength. It was also observed by others[20] that in case of natural fiber, the tensile strength of the speci-
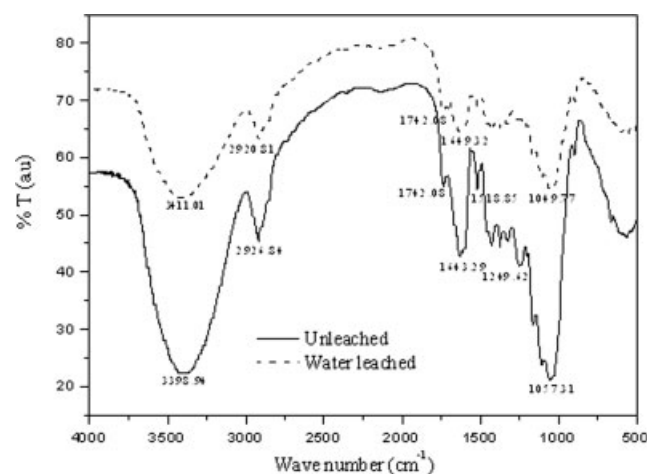

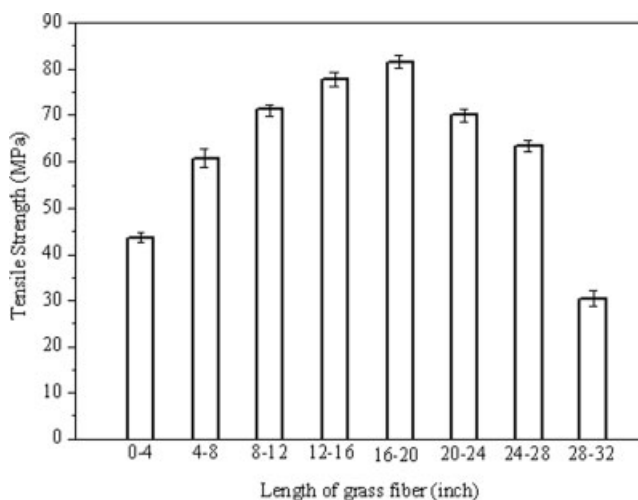**Figure 3** FTIR Spectrum of unleached and water-leached sample.

Figure 4 Variation of tensile strength of grass fiber along its length.

men vary from its bottom to top along its length. Although in case of flax fiber the tensile strength varies along its length but in case of pineapple-fibers the tensile strength remains almost unchanged along the length.[21] This type of variation in mechanical properties may be due to the inhomogenity or presence of defect in natural fiber.[20,21] The grass stick was assessed for its tensile strength before and after leaching with water (Fig. 5). From the figure it is evident that water-leached sample shows higher tensile strength compared to that of unleached grass fiber sample.

The performance stability of the grass fiber samples was evaluated by a simulated test using an accelerated aging oven in the laboratory. Both the dried water-leached and water-unleached samples were put in a constant temperature air-aging oven at 70 ± 2°C
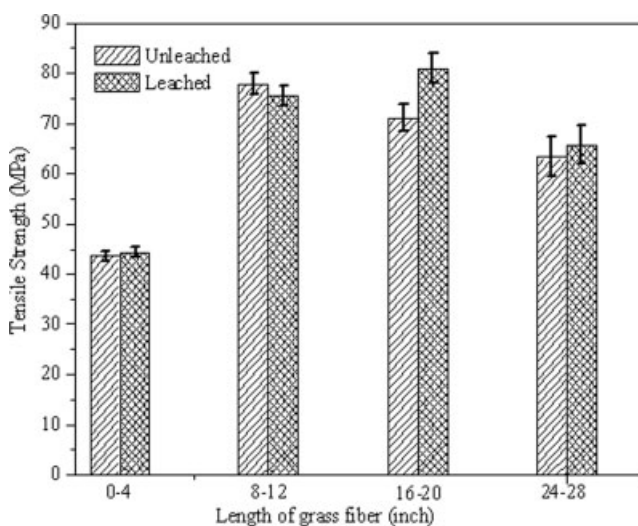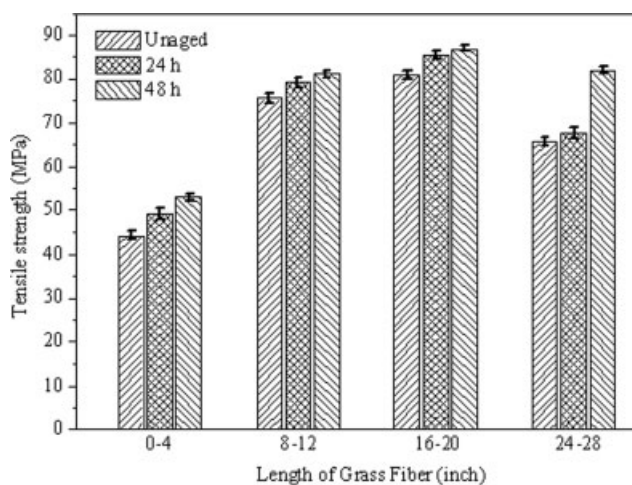

Figure 6 Aging of water-leached grass fiber at 70°C for 24 and 48 h.

for various duration. After keeping the samples in the aging oven for specified period, those were taken out and cooled to room temperature by keeping in a desiccator. Next the aged samples were subjected to tensile strength measurement.

The leached samples show better mechanical strength after 24 and 48 h aging at 70 ± 2°C than the original value (Fig. 6). But in case of unleached sample there is a large decrease in tensile strength with the increase of aging time (Fig. 7). So the thermal aging behavior of the two samples (leached and unleached) is of opposite nature.

This type of aging behavior can be explained by the following reason. Removal of water-soluble materials leads to the creation of some void spaces inside the bulk of the grass fiber. So on application of tensile stress some alignment of fibers occur in the bulk of the sample along its length compared to that effect in
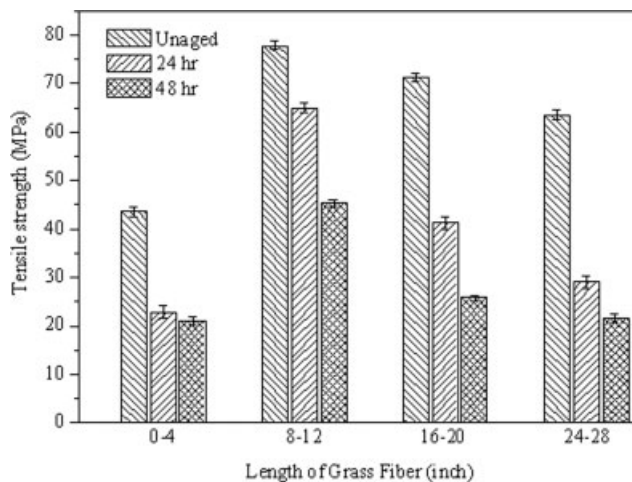

Figure 5 Tensile strength of grass fiber before and after water leaching.

Figure 7 Aging of unleached grass fiber at 70°C for 24 and 48 h.

TABLE II
Tensile Strengths of Various Polymer Modified Grass Fiber Samples

Soaking material	Solid content (%)	Polymer	Soaking time (h)	Polymer loading (%)	Tensile strength (MPa)
None (control)	–	–	–	–	42.0
P-F resin (Resol) Viscous	67.96	P-F resin	6	105	35.6
	61.33	P-F resin	6	80	39.4
	40.46	P-F resin	6	35	67.0
	22.56	P-F resin	6	28	54.9
Prepolyurethane solution in THF	10.07	Polyurethane	6.0	20	103.6
	10.07	Polyurethane	10.0	32	78.4
	10.07	Polyurethane	16.5	40	54.6
Acrylamide monomer	5	Polyacrylamide	1.0	14	116.57
	5	Polyacrylamide	1.0[a]	25	68.85
	5	Polyacrylamide	2.0	15	116.00
	5	Polyacrylamide	2.0[a]	30	67.98

[a] Soaked under vacuum.

the unleached sample as a result of which leached sample has shown better or almost comparable tensile properties. From the results of the accelerated aging test of the samples it is apparent that the water-soluble materials remaining in grass fiber has some definite action on the aging process. The presence of water-solubles has shown pronounced influence on faster degradation of the unleached grass fiber. So the aging property of the unleached sample is poor, whereas in leached sample the absence of such type of materials has increased the thermal stability of the grass fiber.

Modification of grass fiber by treatment with various polymers

The main disadvantage of the grass in its product applications is its stiffness and environmental degradation. These limitations may be avoided by incorporation of polymer into the grass matrix. It was thought that the polymer modified grass would behave similar to that of fiber reinforced polymer composite although the polymer would not remain as continuous phase, rather the polymer would fill the tubular voids of the grass matrix. Grass matrix being a polar cellulosic polymer, incorporation of polar polymers into such tubular voids of the grass matrix was supposed to develop interfacial bonds for enhancing the strength of the modified grass. Internal morphology of grass is such that the incorporated polymer may also remain as bundles of filaments. But it has been found from the morphology of the grass that introduction of high molecular weight polymers into grass matrix is a difficult task because penetration of polymers into the bulk of the grass is a diffusion controlled process. Large polymer molecules cannot penetrate uniformly into the tubular void spaces of grass matrix. So it was anticipated that *in situ* synthesis of polymers from their respective

monomers within the grass matrix might be convenient way of modification with polymers. When soaked the grass sticks with polymer solutions at different dilutions we observed a nonuniform polymer impregnation into the grass. This phenomenon led us to seek for other convenient method for uniform polymer impregnation. We tried to incorporate phenol formaldehyde resin, polyacrylamide and polyurethane. After modification with such polymers tensile strengths of such modified polymers were measured and the results are presented in Table II.

Modification of grass fiber with phenolic resin (resol)

The modification of grass fiber with phenolic resin was carried out in presence of excess formaldehyde to provide potential crosslinking. In basic pH, the phenol and formaldehyde taken in the mole ratio 1 : 1.2 can form crosslinked network structures and bonding with grass fiber, subsequently enhancing the fiber mechanical strength. Grass fiber and phenolic resin reaction was carried until gel point at 150°C for 7 min. From the results in Table II it is observed that resin incorporation into the grass decreases if the grass is soaked with dilute solution of phenol-formaldehyde resin. If concentration of phenol-formaldehyde solution is increased then more amount of resin is introduced in the micro pores of grass. But tensile strength (67 MPa) is maximum for 35% resin content. The tensile strength of the control sample (unmodified grass fiber) is 42 MPa. This is because of the fact that if resin content is very high then grass will acquire brittleness and ultimately tensile strength will be low but for medium resin content, tensile strength will be maximum and simultaneously it will be flexible in nature. The variation of tensile strength with polymer loading is also shown in Figure 8.
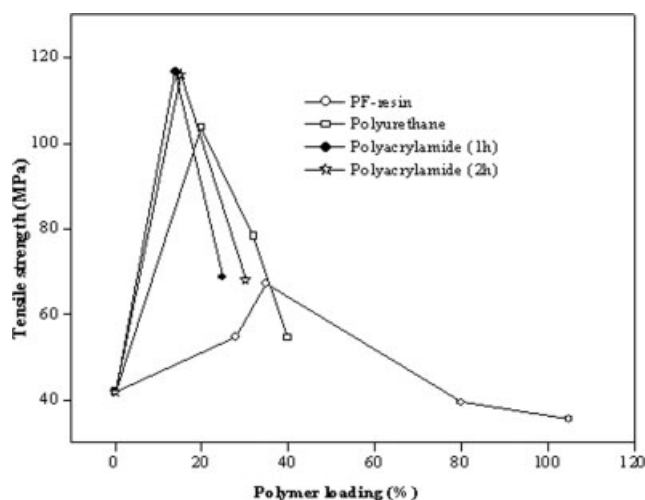
Figure 8 Variation of tensile strength with different polymer loading in grass fiber.

Modification of grass fiber with polyurethane

In the typical modification process of grass fiber by polyurethane initially low molecular weight prepolymer was synthesized. This low molecular weight prepolymer was easily impregnated into the grass fiber and finally this low molecular weight prepolyurethane might form a three dimensional net work structure with free hydroxyl groups of grass fiber ultimate enhancement of the fiber mechanical property. The variation of resin content in the grass on tensile strength of the same for different soaking times is given in Table II and Figure 8. From the data in Table II it is observed that the tensile strength of the polyurethane modified grass depends on the resin content and with increase in resin content tensile strength of the *in situ* generated polyurethane-grass fiber composite decreased remarkably. This phenomenon can be explained by chemical crosslink and physical anchoring of natural fiber and polyurethane resin. Beyond a particular resin concentration physical anchoring plays a predominant role over chemical crosslink. But physical anchoring has less influence than chemical crosslink in composite's ultimate property. For that reason tensile strength of the materials drop remarkably after a certain resin concentration. This decrease in tensile strength can not be explained by ductile or brittle fracture of resin. As seen from Figure 8, 20% or less resin content may provide highest tensile strength.

Modification of grass fiber with polyacrylamide

Modification with polyacrylamide was done by soaking the grass with acrylamide monomer followed by *in situ* polymerization of the monomer within the matrix. It was anticipated that this technique would over-

come the problem of impregnation of high molecular weight polymer into the bulk of the grass. The aqueous solution of the monomer was soaked into the bulk of grass stick with or without applying vacuum. Polymer loading was higher in case of monomer soaking in vacuum. It is seen from Table II and Figure 8 that with higher loading of polymer, the tensile strength is lower than that with higher amount of polymer, as is the observation with other polymers.

Hydroxyl value of grass fiber

Hydroxyl value of grass indicates its moisture absorption characteristics and the scope for modification with polymer. Hemicellulose contains large number of hydroxyl groups in the repeating unit.[22] It has been found that the hydroxyl content of the unleached grass fiber is 475 and that of the water-leached sample is 335. This happens due to the loss of soluble sugars during water leaching.

Moisture absorption of polymer treated grass fiber

The extent of moisture absorption of unleached, water-leached and polymer modified fibers in 97% RH at 25°C is shown in Table III. From the results it is evident that % moisture absorption of unleached grass fiber is more than double than that of water-leached sample. The presence of large amount of water-soluble material in unleached sample which contain large number of hydroxyl group enhances the moisture absorption characteristics of unleached grass fiber compared to that of the water-leached grass fiber sample. The polymer treated grass fibers were exposed to 97% RH in closed chamber. Such environment was created by putting a saturated solution of potassium sulfate[23] within the chamber.

It is seen from Table III that after modification with polymer, the moisture absorption characteristics of grass fiber composite reduces a substantial amount due to the decrease in hydroxyl functionality by reacting with modified polymer. Polyacrylamide modified grass fiber has shown maximum moisture absorption over other polymer modified samples because of hygroscopic nature of polyacrylamide.

TABLE III
Moisture Absorption of Unleached, Water-Leached, and Polymer Modified Grass in 97% RH at 25°C

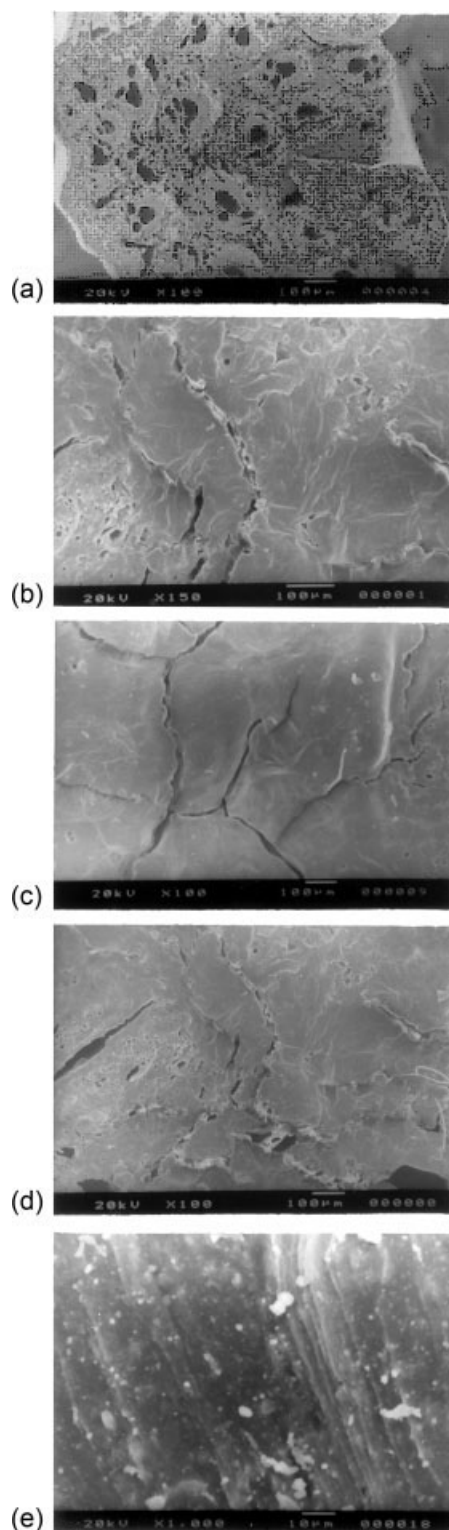| Type of grass fiber	% Moisture absorption
Unleached	23.8
Water leached	10.3
Polyurethane modified grass fiber	8.3
PF resin modified grass fiber	14.0
Polyacrylamide modified grass fiber	25.4

Figure 9 (a) SEM photograph of water leached grass fiber cross section. (b) SEM photograph of phenolic resin modified grass fiber cross section (Fiber : Polymer = 2.8 : 1). (c) SEM photograph of polyurethane modified grass fiber cross section (Fiber : Polymer = 5 : 1). (d) SEM photograph of polyacryl amide modified grass fiber cross section (Fiber : Polymer = 7 : 1). (e) SEM photograph of tensile fractured surface of polyurethane modified grass fiber (Fiber : Polymer = 5 : 1).

SEM of grass fiber

SEM pictures of water-leached and polymer modified grass fiber samples are shown in Figure 9(a–d). In water-leached grass fiber sample we found that a mesh like structure was present on the outer surface of the grass. From cross-sectional view of water-leached grass sample it is seen that the pore size increases due to the removal of hemicellulose. From Figure 9(a–d) it is evident that after modification with various polymers the pores were filled up by polymer and there was no holes on the fiber surface which ultimately increased the strength of the fiber. Figure 9(e) represents the tensile fractured surface of polyurethane modified grass fiber sample. From the figure it is clear that unidirectional ripples (lines of reinforcement) are observed in the tensile fracture surface.

CONCLUSIONS

The tensile strength of the grass varies along its length having maximum strength at the middle portion. Water leaching of grass has improved the strength as well as the aging resistance due to removal of soluble carbohydrates, which cause fungal attack. The SEM study of water-leached sample has revealed an etched surface and porous morphology of the grass. This indicates a possibility of better resin adhesion, if used for modification of grass, for improved properties. Modification by *in situ* polymer formation in optimum amount within the grass bulk has indicated properties improvement.

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