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ORIGINAL CONTRIBUTION

Jute Fiber Graft Copolymers Euphorbia Latex Composites: Physico-Mechanical Studies

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ABSTRACT

For the synthesis of bio-composite materials for useful applications, it becomes necessary to modify the surface of natural fibers through chemical treatments. Morphology, structure and properties of natural fibers have an obvious effect on the mechanical properties of the bio-composite materials. It is thus necessary to know the morphology, thermal stability and crystalline behavior of original and modified fibers. In present paper, jute fibers (JF) were chemically modified with tetramethyl silane(TMS) through microwave radiations induced graft copolymerization. Various reaction parameters were optimized to get maximum grafting (>90%). Thermal stability and crystalline behavior of original and modified fibers were investigated. Physico-mechanical and thermal studies showed that surface of jute fibers becomes rough and amorphous through graft copolymerization and thermal stability has been found to be increased. Microwave radiation induced grafting showed a diminutive effect on the crystalline behavior of the jute fibers as optimum time to get maximum grafting is very less (40 min) in comparison to conventional grafting. Synthesized graft copolymers were used as reinforcing material in preparation of Euphorbia latex biocomposites. It has been observed that graft copolymers improved the interface between fiber and matrix and enhanced the mechanical strength of composites.

KEYWORDS— Jute fibers; Grafting; thermal stability; crystallinity; mechanical properties

1. INTRODUCTION

Graft copolymerization is a technique used to transform the properties of natural polymers. Modifying the properties of natural polymers through graft copolymerization has been reported by Sabaa and Mokhtar [1] and various other prominent workers [2-5]. In view of the fact that the prospect of fabrication of newer monomers at low cost is very uninviting, so the modification of a wide array of existing synthetic and natural polymer through graft copolymerization technique for incorporating highly specific properties is of great importance. Graft copolymerized fibers are better in dyeing, printing, chemical resistance, water repellency, fiber strength, abrasion resistance etc. Studies of different workers shown that the grafting procedure are completely different from the values observed for conventional solution polymerization, for example, binary mixtures of monomers: ethyl acrylate/acrylic, acid acrylonitrile/styrene and acrylamide/ styrene. Thermal stability, crystallinity and morphology of the graft copolymers were too studied [3-4]. Methyl methacrylate has been reported to polymerize under microwave irradiation using very low concentration of initiator [6]. In a recent report, grafting of acrylamide [7] and acrylonitrile [8] onto chitosan and acrylonitrile onto guar gum [9] was done under microwave conditions, in very short reaction time and in absence of any redox initiator.

Mechanical properties of thermoplastic composites reinforced with acrylate grafted baggase cellulose fibers were studied. It has been found that best results could be obtained with

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polymethyl methacrylate (PMMA) grafted cellulose fibers because of better fiber-matrix adhesion. The modulus of polyvinyl chloride composites is increased when grafted or ungrafted cellulose are used as reinforcement but the composites with PMMA grafted cellulose present the higher modulus [10]. The concept of using renewable resources[11-13] for the production of plastics and composite for construction application has been started since the beginning of our civilization. In recent years, fiber reinforced plastic composite have been widely accepted as materials for structural and non-structural applications. The main reason for the interest in fiber reinforced plastic for structural applications is due to the high specific modulus strength of the reinforcing fiber. Glass fiber is most commonly used as reinforcement followed by carbon and boron fiber. The later fibers are prohibitively expensive and their use is justified in high value aerospace applications. Even glass fiber is more expensive. Therefore it is worth-while to explore the possibility of utilizing the cheaper materials as reinforcement. Resin and fiber obtained from renewable resources[14-21] can provide the desired alternatives that would reduce the dependence of petroleum feedstock. In this direction, extensive research studies have been continued on variety of natural fibers[22-33] with significant outcome for their industrial uses especially in construction application. Like other natural fabrics the scope for using jute fabric in place of traditional glass fiber partly or fully stems from lower specific and higher specific modulus of jute(1.29 and 40 GPa respectively)[34] compared with those of glass(2.5 and 30 GPa respectively). Beside its much lower cost, low density, renewable nature, low abrasion, the ease with which it can be processed and handled and low energy requirement for production and processing of jute (only 2% of that require for glass fiber)[11,12] make it an alternative fiber for use as reinforcement in preparing composites. However the potential of jute in high value moulded composite has remained largely unrealized in view of poor wettability[35] with organic resin due to presence of polar hydroxyl group results in weak interfacial adhesion in composite of poor strength and lesser environment resistance especially under humid condition. Thus it is imperative to modify functional moieties present in jute fiber by suitable chemical treatment for better impregnation and improved interfacial adhesion with organic adhesives. Marginal to good improvements both in dry and wet strength have been reported with the application of some interfacial agents[35]. Dr.Mitra et. al. has tried to pretreatment of fabric with low molecular weight phenol formaldehyde and CSNL modified phenol formaldehyde condensate and similar other formaldehyde resin before it is made into composite. CSNL modified phenolic resin behaves better in reducing the moisture regain but strength of composite is found to be increased.

Natural latex obtained from different plants can be used as matrix as a whole or with compatible polymer for composite making of better strength. Here the used latex is collected from Euphorbiaceae family such as Euphorbia royleana, Euphorbia nerifolia and Euphorbia cadicifolia along with Hevea brasiliensis[36] and India can easily be a leading source for exploitation. The main constituents of Euphorbia latex[37,38] is resinous mass(65-80%) containing tetracyclic triterpinoid, protein(10-20%), cis-1,4 polyisoprene (5-10%) and small amount of ash.

Green composites reinforced with S. spontaneum fiber and its different graft copolymers were fully biodegradable and environment friendly. Mechanical properties including tensile strength, compressive strength and Modulus of elasticity (MOE) and modulus of Rupture(MOR) of different reinforced samples were significantly improved in comparison to pure jute matrix composite. Maximum compressive strength and wear resistance was found in case of fibers grafted with TMS [39]. The present study emphasizes on utilization of Euphorbia latex coagulum for the development of reinforced composite board on grafted jute fiber. Jute fiber reinforced E. latex composites were prepared by compression molding technique in which good interfacial adhesion is generated by fiber surface modification. The jute fibers were modified

through graft copolymerization with TMS. The short grafted fibers were then spread between the alternate layers of E. latex resin by hand lay-up method to obtain the thermoplastic composites. E. latex composites reinforced with TMS grafted jute fibers showed better mechanical properties.

Since grafting on to JF under microwave radiations is not enough reported in literature, so in this chapter we report the grafting of TMS onto JF fibers and graft copolymers were used as reinforcing material for the synthesis of biocomposites.

2. MATERIALS AND METHODS

2.1.Woven jute fabric fabric

Jute fiber and commonly cultivated herb in eastern India For the work an extensively distributed jute fiber of weight 1400 – 1600 GSM and 1.5 mm thickness was used for this project as shown in figure 1. Jute fabric is available in the market (collected from Gloster Ltd., 21, Strand Road Kolkata) in roll form and this roll was cut to size as per the dimension required for the study. The strength properties are given in table 1.

Table 1: Mechanical properties of Control jute

Type of fabric	Physical properties		Mechanical properties						
	Density (Kg/m^3)	Diameter (μm)	Tensile strength (MPa)		Young's Modulus (GPa)		Specific Strength (MPa/gm^{-3})	Specific Modulus (GPa/gm^{-3})	Failure strain(%)
			Ave.	M.I.[a]	Ave.	M.I.[a]	Ave.	Ave.	
Jute	1290[c]	29 – 52	380	152	25.2	7.1	293.2	18.1	1.38

M.I.(Minimum Individual).

[a] The observations number was about 50

[c] Density of jute is calculated from the specific gravity referred to Ref.[7].

[Tensile tests were conducted using a small-capacity testing machine (ASG-H/Ez Test- 00: shimadzu) at 1 mm/ min of crosshead speed].

2.2. Euphorbia Latex

Euphorbia latex was collected from foothills of Himalayas around the region of Sahastradhara, Dehradun, India. The milky latex was coagulated by adding 5% aqueous solution of tannic acid. The coagulated latex was washed several times with pure water to remove tannic acid till the pH of the material become 7 (checked with E. Merck pH paper). Ultimately the material is dried in a hot air oven at a temperature 60^0C[26]

2.3. Tetrmethyle Silane

99.5% purity TMS by Sigma Aldrich were collected from Sarada glass and chemicals, 33, Dixon lane, Kolkata – 14.

Purification of jute fibers

Jute fibers (Corchorus olitorius) were washed with detergent in order to remove impurities and then Soxhlet extracted with acetone for 48 hrs. to remove waxes, lignin and other impurities.

Graft copolymerization

Jute fiber (500 mg) were immersed in 1000 mL of distilled water for 24 h prior to its grafting under the influence of microwave radiation. A definite percentage of TMS in acetone were added to the reaction mixture. The reaction mixture was stirred and transferred to the microwave equipment operating at 220W microwave powers for a specific time interval. The different reaction parameters such as TMS concentration, reaction time etc. were optimized and the excess was separated from the grafted fiber by soxhlet extraction in order to obtain maximum graft yield. The graft copolymer obtained was dried in the hot air oven at 50^0C until a constant weight was obtained. The percent graft yield (G) and percent grafting efficiency (% GE) were calculated as follows [**3**]:

$$\%G = \frac{Final\ \ weight\ of\ \ jute\ \ fiber - Initial\ \ weight\ of\ \ jute\ \ fiber}{Initial\ \ weight\ of\ \ jute\ \ fiber} \quad \text{.....(1)}$$

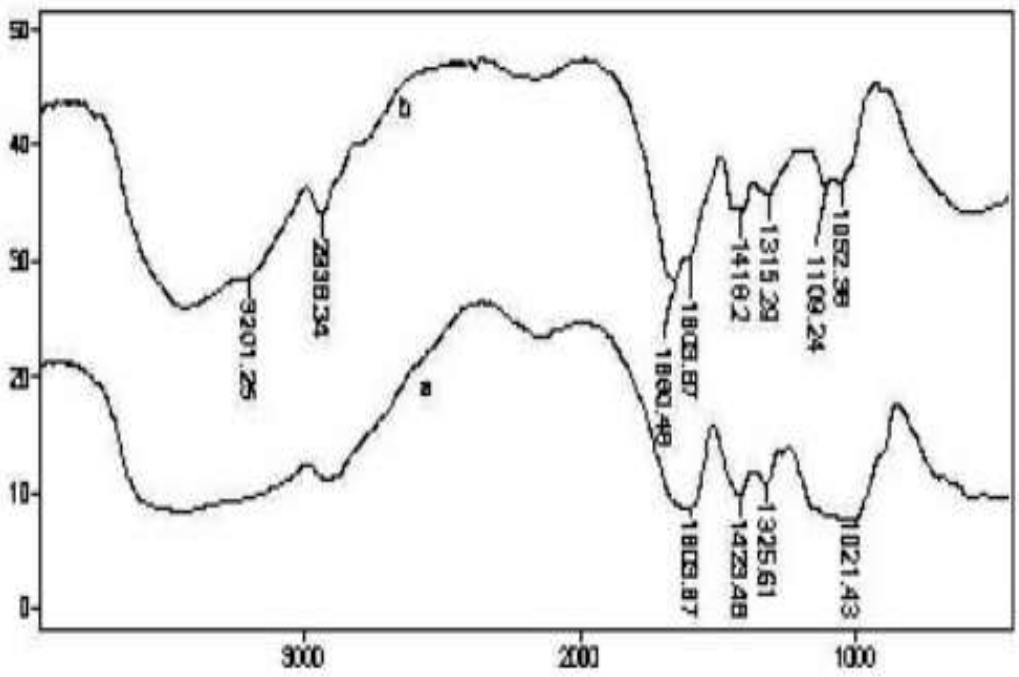
$$\%GE = \frac{Final\ \ weight\ of\ \ jute\ \ fiber - Initial\ \ weight\ of\ \ jute\ \ fiber}{Weight\ of\ \ monomer} \quad \text{.....(2)}$$

Preparation of composites

E. Latexmatrix based composite were prepared by mixing a particular ratio (10:0.5) of resin with Jute fiber. The matrix was mixed with control jute and grafted jute fiber and then placed in the mold of a particular dimension (length 40-80 mm and cross section 5 × 5 mm). Degasification of sample was carried out in compression molding machine and the samples were put for curing at 100^0C for 10 minutes under a specific pressure 12 kg.cm^{-2}. Composites thus prepared were subjected for different mechanical studies. The number of specimen used for the determination of mechanical properties was three in each case. Tests were conducted at ambient laboratory condition (temperature: 35^0C and RH: 50%).

Characterizations

Infra red spectroscopy (IR): IR spectra of the ungrafted and grafted jute fiber were taken with KBr pallets on a Perkin Elmer Lamda-35 (USA) UV VIS spectrophotometer. IR spectra of ungrafted and grafted natural fiber are useful in explaining the grafting of a particular monomer on to the fiber. The presence of a new peak in spectra of grafted fiber confirms grafting on to jute fibers.



IR spectra of the control and grafted jute fiber were taken with KBr pallets on a Perkin Elmer Lamda-35 (USA) UV VIS spectrophotometer.

Thermo gravimetric analysis: Thermo gravimetric analysis and differential thermal analysis were carried out in nitrogen atmosphere at a heating rate of 10˚C min^{-1} using Perkin Elmer, (Pyris Diamond) thermal analyzer. The rate of nitrogen supplied was 200 $ml.min.^{-1}$

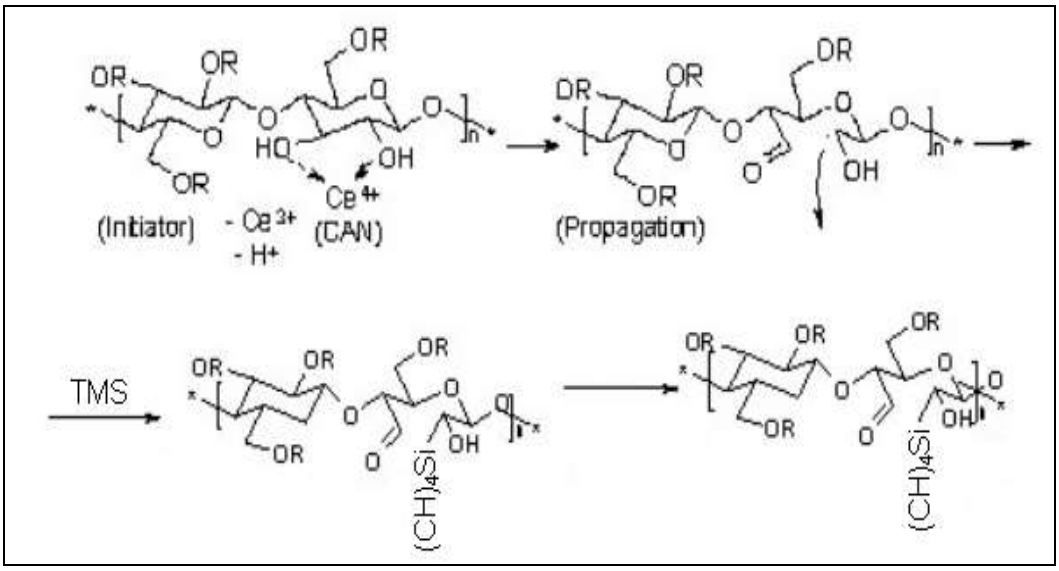
Results and discussion

Grafting yield of jute fiber directly immersed in TMS solution is very low. This is because of the fact that jute being a cellulosic fiber with lignin does not readily bonded with TMS. In order to overcome this barrier jute fiber has been immersed in water for 24 hrs. so as to open the active sites of the cellulosic back bone and to open the functional group and grafting has been carried-out by using solvent-TMS mixtures rather than pure TMS which enhances the deep penetration of dissolved latex inside the polymer matrix and considerably increases the graft yield. The success of this method is due to the combination of two effects: (i) reduction in viscosity of the reaction medium which helps in the easy movement of TMS molecules towards the active site of jute and (ii) easy and greater accessibility of the matrix to the open jute fiber. Modification of the jute fibers has been carried out under MWR induced grafting in aqueous medium using Ceric ammonium nitrate (CAN) in the presence of nitric acid CAN-HNO_3 as initiator as per following mechanism.

Mechanism

In polymeric materials containing cellulose as a building block, C_2, C_3 and C_6 hydroxyl groups and C—H sites are the active centers for grafting of polymeric chain onto polymeric backbone. Ceric ammonium nitrate takes part in redox reaction with Ce^{4+}. Ceric(IV) ion induce active center in cellulose polymeric chain by oxidizing the basic glucose molecule. It oxidizes and produces the free radical of cellulose polymeric chain. Ceric ions form the chelate complex with

cellulose molecule through C_2 and C_3 hydroxyl group of glucose unit. Ce^{4+} ion is reduced to Ce^{3+} by transfer of electron from cellulose molecule.



found to be 45 min (**Table 1**). With further increase in time a decline in percentage grafting has been observed, which may be due to hydrogen attraction reaction along with some other side chain reaction.

Optimization of different reaction parameters for grafting of ethyl acrylate on to jute

Grafting of different vinyl monomer on to the jute fibers may be influenced by the presence of the number of active sites on to the fiber surface. The reaction parameters such as reaction time, Initiator ratio (CAN – HNO_3), concentration used to carry out graft copolymerization of TMS were optimized. The optimized reaction parameters for grafting of TMS on to JF are given below in **Table 1**.

Effect of TMS concentration

In Jute fiber grafting increases with increase in TMS which become maximum (65.8%) at 0.369 mol dm^{-3} and then decreases with further increase in concentration (**Table 1**). This may be due to excessive formation of TMS at higher concentration of it.

Effect of Reaction Time

The optimum reaction time is found by varying reaction time between 40 to 50 minutes. The optimized time of grafting on to jute fiber is

Effect of CAN Concentration

The effect of variation in CAN concentration was studied and the results are given n in **Table 1**. CAN concentration was increased from 3.85 to 7.26 × 10-3 mol dm-3. It is evident that %G increases with an increase in the CAN concentration, and reaches maximum value of 91.8%, at 6.41 × 10^{-3} mol dm^{-3} of CAN. These increasing trends of the grafting parameters indicated that ceric ions exclusively participate in the formation of active sites on the cellulose up to this concentration of ceric ions, and beyond it, no more active sites are formed on the cellulose. Further increase in CAN concentration is accompanied by a decrease in the %G .The decreasing trend in %G beyond 6.41 × 10-3 mol dm^{-3} concentration of ceric ions may be assumed to be due to its participation in the termination reactions with growing grafting and propagating chains on the cellulose.

Effect of HNO_3 concentration

With increase in nitric acid concentration there is a decrease in percentage grafting. This is due to the reason that at higher concentration of acid causes a degradation of backbone chains of cellulose and graft chains. Higher acid concentration also causes an oxidation of the free radicals formed which results in an increased bonding (**Table 1**).

About 0.5 g jute fibers were immersed in 100 mL of water for 24 h prior to their grafting under microwave radiations. The fiber was then mixed with known amount of binary monomer mixture and definite amount of CAN – HNO_3 (6.41 × 1 0-3 mol dm^{-3} + 0.313 mol dm^{-3}) at optimum reaction time and TMS concentration.

Table 1: Optimization of different reaction parameters for grafting of TMS on to jute

Sl. No.	TMS (mol dm^{-3})	Time (min)	Initiator Ratio × 10^{-3} mol dm^{-3}	Grafting (%)	GE (%)
1	0.092	45	4.81+0.235	23.2	24.7
2	0.184	45	4.81+0.235	29.9	15.9
3	0.276	45	4.81+0.235	46.4	16.4
4	0.369	45	4.81+0.235	65.8	17.5
5	0.461	45	4.81+0.235	32.8	6.98
6	0.369	35	4.81+0.235	65.0	17.3
7	0.369	40	4.81+0.235	81.8	21.8
8	0.369	50	4.81+0.235	50.2	13.3
9	0.369	55	4.81+0.235	47.2	12.5
10	0.369	40	3.85+0.235	47.0	12.5
11	0.369	40	5.55+0.235	87.4	26.6
12	0.369	40	6.41+0.235	91.8	30.0
13	0.369	40	7.26+0.235	74.6	19.8
14	0.369	40	6.41+0.157	58.4	15.5
15	0.369	40	6.41+0.313	88.8	23.6
16	0.369	40	6.41+0.392	79.8	21.2
17	0.369	40	6.41+0.470	42.4	11.3

Table 2: Optimization of concentration of CAN-HNO_3 and TMS on % grafting

TMS (0.369 mol dm^{-3})+ CAN-HNO_3			
Sl. No.	CAN Conc.(mol dm^{-3})	Grafting (%)	GE (%)
1	0.235	106.2	45.2
2	0.282	118.4	42
3	0.329	135.2	36
4	0.375	82.8	22

High percent graft yield in case of TMS and CAN-HNO_3 due to the presence of strong acceptor result in generation more free radical sites. In case of E. laex, low percent grafting is due to miscibility of polar and non polar interaction and thereby resulting in generation of less number of free radical sites on the backbone polymer.

FT-IR spectroscopy:

The IR spectra of jute fiber showed a broad peak at 3413.27cm^{-1} with relative absorbance of 81.9% due to bonded OH groups in cellulose and at 2922.64 cm^{-1} with relative absorbance of 81.6% due to -CH stretching in cellulose and hemicelluloses. Another peak at 1644.81 cm^{-1} may be due to C-C bond. The carbon-oxygen single bond also has an

absorption in the fingerprint region, varying between 1000 cm^{-1} and 1350 cm^{-1} depending on the molecule in it. The wave number in finger print region at 1067.72 cm^{-1} may be due to C-O stretching. Because C-H bond is found in almost all organic compounds, so it is not much important. What it means is that a trough just under 3000 cm^{-1} can be ignored, because it is probably just due to C-H bond (**Fig. 3a**). The carbon-oxygen double bond C=O, is one of the really useful absorption, found in the range 1670-1760 cm^{-1}. Its position varies slightly depending on what sort of compound it is in. In raw jute fibers a trough is observed in region 1670-1760 cm^{-1}.

In case of Jute-TMS a peak at 1863.56 cm^{-1} with relative absorbance of 96.5% Jute-TMS high absorbance in this region indicating a special bond between C-Si. This extra hump confirms the grafting of it on to jute fiber (**Fig. 3b**). Other peaks at 3433.90 cm^{-1} due O-H stretching, 2981.32 cm^{-1} due to C-H stretching and 1111.64 cm^{-1} may be due to C-O group are similar to the raw fiber. Other peak at 3434.55 cm^{-1} is due to O-H group is broader than in raw fiber which indicates the presence of alcoholic as well as carboxylic O-H group in the fiber. The peaks at 2922.64 cm^{-1} due to C-H stretching, 1160.3 cm^{-1} due C-O stretching etc (**Fig. 3**).

Table 3: FT-IR data of raw and grafted jute fiber

Wave Number (cm^{-1})	Functional Group	Relative % absorbance	
		Raw fibre	Jute- TMS
3600-3200	-OH stretching	81.9	96.5
2962-2850	-CH streching	81.6	95.7
1860-1863	-C-Si streching	-	96.8
1470-1350	-CH bending	77.6	93.5
1350-1000	C-O stretch	80.2	95.0

Morphological studies

Scanning electron microscopic provides an excellent technique for the study of surface morphology of original and chemically modified jute fibers. It has been observed that surface morphology of original jute fibers differs in smoothness and roughness than grafted jute fibers. Fig. 4a and b The SEM of original and grafted jute fibers. These micrographs clearly showed the difference in their surface morphology. The original fiber (**Fig. 4a**) is free from any deposition of TMS and surface is very smooth in comparison to modified fibers. Modified jute fibers (**Fig. 4b**) Showed that considerable amount of TMS are grafted and their surface becomes rough [**13**].

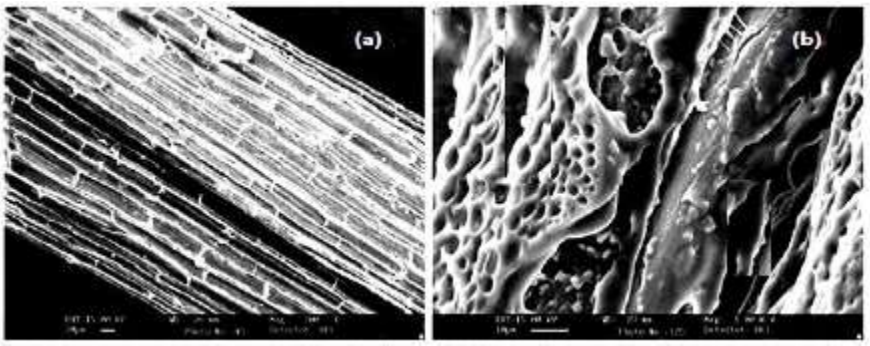


Figure 4: SEM of (a) Control jute fiber (b) Jute-TMS were carried out on Electron Microscopy Machine (LEO 435 VP)

Thermal gravimetric analysis:

TGA of jute fiber and TMS graft jute were carried out at a rate of 100^0C min.$^{-1}$ in nitrogen as a function of percentage weight loss versus temperature. In TGA of controll jute fiber, two stage decomposition has been observed, with maximum weight loss between 100 and 300^0C (16.6%) and 300 to 374^0C (59.5%). The first stage decomposition may be due to loss of moisture and second stage decomposition is due to cellulosic and lignin degradation. The initial and final decomposition temperatures of raw fiber are 100 and 300^0C respectively. In the two stages decomposition the first stage at 100 to 300^0C may be due to breakdown of hemicellulose and glycosidic linkage of cellulose whereas the second stage of decomposition at 300 to 400^0C may be related to the degradation of grafted jute chains on to the fiber surface. From the above result it is clear that there has been increase in the thermal stability of fiber upon grafting.

Table 4: Rate of weight loss per minute at different temperatures through TGA analysis

Sl.No.	Sample	Temp. (^{O}C)	Rate of wt. loss (mg/min)	Temp (^{O}C)	Rate of wt. loss (mg/min)	Temp. (^{O}C)	Rate of wt. loss (mg/min)
1	SHF	60	0.172	350	1.188	-	-
2	SHF-g-poly(EA)	61	0.136	356	1.142	393	0.651
3	SHF-g-poly(EA+MMA)	54	0.096	362	0.970	397	1.555
4	SHF-g-poly (EA+AA)	54	0.075	355	0.981	399	0.994

4.4 *water absorption test:*

The water absorption test was carried out as per of ASTM D-570. The test results indicate that the composite from grafted jute fabric shows comparable water absorption property with the control table 4

Table 4: Water absorption test of composite of size 2 inches diameter

Nature of Jute	E latex	After 2 hrs. (%)	After 24 hrs. (%)
Control Jute	100:00	5.30	7.20
Grafted Jute	100:00	5.65	7.08

The specimens are dried in an oven then placed in desiccators to cool and weight immediately.

4.5 *Flexural test:*

For flexural test the specimens were cut from molded sheet. The dimension of the test specimens was 127 mm x 12.7 mm x 3.0 mm. Flexural strength of coagulum modified polyester jute fabric composites specimens were tested on universal tensile machine (Instron, 4302 model, UK) at the crosshead speed of 5 mm/min and at a span length of 96 mm. Three-point bend test were performed to measure flexural strength. Temperature was maintained at 23±2°C as per the guidelines of ASTM D-790. Flexural strength property increase though strength decreases with the increase in percentage of latex coagulum as shown in table 5 and figure 7, indicate utilization of such type of mixed glue from bio-adhesive will be effective for the preparation of flexi composite.

Table 5: Tensile and flexural strength of composite

Nature of Jute	E latex	Av. Tensile Strength (MPa)	Elongation at Break (mm)	Flexural Strength (MPa)
Control Jute	100:00	50	1.85	22.2
Grafted Jute	100:00	83	1.92	28.6

*Av. tensile strength is obtained from three samples Tests were done by universal tensile machine (Instron, 4302 model, UK)

4.6 Tensile strength of compressed impregnated jute fabric:

The tensile test was performed according to ASTM D638. The samples were 150 mm long 25 mm wide with thickness of 3 mm. three specimens were tested on universal tensile machine (Instron, 4302 model, UK) at cross head speed of 0.5 mm /min. Load displacement curves were generated for each sample. For measuring elongation, two marks along the central load axis were made at a distance of 15 mm, on either side from the centre of the sample. The test results showed that better bonding with in case of TMS treatment on jute.

5. CONCLUSION

Graft copolymerization is preeminent method for the surface modification of cellulose fibers. Through graft copolymerization, surface of jute fibers becomes rough and amorphous and thermal stability has been found to be increased. Microwave radiation induced grafting Showed a diminutive effect on the crystalline behavior of the jute fibers. Graft copolymers reinforced composites Showed enhanced mechanical properties, thermal stability and they improved the interface between jute fibers and Euphorbia latex. Jute polyester resin composite is known to all; here an attempt has taken to replace polyester resin with compatible natural bio-base latex from euphorbia plant which is abundantly available in all over India.

Comparable water absorption property, The flexural strength, tensile strength with the control jute and improvement in case of graft jute fiber indicate better bonding due to blocked of polar hydrophilic functionalities and bonding between non polar-non polar interaction between TMS grafted jute and E. Latex as the glue is hydrophobic in nature.

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