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Effect of monomer (EGDMA) on the mechanical properties of jute fiber by using additives: Photocuring with UV radiation

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Abstract : For the biodegradability of jute (Corchorus capsularis) in environmental awareness has given emphasis to develop the properties. The physico-mechanical properties of jute yarns were treated by EGDMA in along with photoinitiator (2%) and cured under UV radiation intensities. As the radiation-induced graft copolymerization is one of the most important methods to modify natural polymers Concentration of monomer; soaking time and radiation intensity were optimized with respect to mechanical properties. The Photocuring method has enhanced tensile strength (TS factor Tf = 2), elongation at break factor, (Ef = 1.4) and polymer loading (20%) as affected with 5% EDGMA (5 minutes soaking time). Urea (0.5 - 2%) was added to the opti-

INTRODUCTION

Jute (*Corchorus sp.*) is a callused based natural fiber which mainly used as hessian clothes, ropes,

mized solution (5% EGDMA) where 1% Urea showed the best properties (PL = 2.4 %, Tf = 2.3). KMnO₄ treated yarns produced the best results (PL = 29%, Tf = 2.6, Ef = 1.7) along with Urea (1%). This is a unique achievement in the sense that the mechanical properties of the jute yarns can be improved by curing of KMnO₄ treated yarns followed by addition of additive (urea) rather than curing of jute yarns without pretreatment or any additive. Both KMnO₄ and Urea are very cheap. This method of modification of the jute yarn appears to be economical. **© Global Scientific Inc.**

Keywords : Modification; Jute; UV-radiation; Monomer concentration; EGDMA; Additives.

gunny bags, shopping bags, backings of tufted carpets, cheap floor coverings and matting etc. The natural polymer is biodegradable, abundantly available, easily decomposable in the environment and eco-

friendly. Jute and traditional jute products are facing stiff competition from artificial fiber and synthetic products. This situation can improve if new technologies are developed, diversified jute products would be produced according to customer's choice and market demand. Upon the public demand for survival of jute in competition with the synthetic fibers, it must be constantly developed and improved in physical property to retain its glorious status. Scientists have attempted over the years to modify its quality and characteristic properties, both genetically and by improving its end products, with a view to diversifying its applications in various fields. The environmentally conscious citizens are therefore reverting to the use of natural polymers wherever possible in place of man-made synthetic polymers to minimize pollution of the environment. The main reason is that the synthetic polymers are non-degradable, durable and sustainable in the environment, causing pollution after its end uses. The energy intensive nature of their production and their resistance to biodegradability has led to renewed interest in jute. Jute has the advantages of being both renewable by agro-efforts and environmental friendly due to biodegradability. Nevertheless, attempts have been continually taking place to improve natural polymers both genetically and chemically. Thus natural polymers are treated with different materials in different modes to increase their strength, durability and sustainability while possessing their inherent degradable character to maintain an environment free of pollution^[1-5].

Modification of jute grafting for use in composites was reported^[6] where moisture absorption of jute fibers reduced significantly by treatment with graft copolymerization with methyl methaciylate (MMA). The tensile strength and Elongation at break of jute fiber decreased on grafting whereas the initial modulus increased with increasing percentage of grafting. Gamma radiation Co-60) was used on graft Copolymerization of vinyl monomers, MMA, acrylonitrile, vinyl acetate and styrene onto jute fiber^[7]. Ferric sulphate used as initiator and photographed with MMA copolymerization on bleached jute fiber^[8]. The ethylenediamine (EDA) showed the effect onto jute fiber. The physico-chemical proper-

ties of the jute fiber such as weight loss, swelling moisture absorption and tensile properties were influenced by EDA. Changes in the fine structure of jute were assessed from X-ray and IR studied^[9]. Physical and mechanical properties of UV cured jute fiber were improved by urethane acrylate. The role of plasticizers and water uptake were investigated. Through plasticizers the tensile properties of the UV cured films were decreased, they substantially enhanced both tensile strengths by 300%. Water and moisture take up at different relative humidity conditions decreased significantly^[10]. The optimum conditions for grafting were obtained and proved that MMA grafted was thermally more stable than the original fibers. The processing and characterization of jute fiber reinforced thermoplastic polymers were investigated to verify their possible application in reinforcement of thermoplastics^[11]. The radiation induced jute plastic composite; preparation and IR studies were performed. Jute plastic composite has been prepared with tossa jute yams and MMA mixed with methanol at different properties using Co-60 gamma sources irradiation. Incorporation of a minute amount of (1%) additive like N-Vinyl pyridine, Tripropyleneglycol diacrylate and trimethylolpropane triacrylate, or urea substantially increased the grafting of MMA onto jute yarn. The TS, water absorption and moisture absorption at different relative humidities has been detected. Both TS, EB, decreased with increased grafting. The IR study reviled that MMA has been grafted onto jute cellulose at the sites of OH groups of the jute cellulose backbone^[12]. The degradable property of UVcured hessian cloth (jute) was determined and the enhancement of tensile strength and elongation was detected. Loss of these properties due to simulating weathering was also measured. For degradation samples to be buried in water and mud for a long period and soil retained their tensile properties^[13]. The natural fiber reinforced biodegradable matrix composite and their effect of surface modifications of jute on the performance of jute-biopol composites were studied^[14].

Due to the awareness of polymeric scientist for the sustainable development of cellulosed based jute composite materials need to be improved the physi-

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cal and mechanical properties for better application in diverse field of biodegradable materials. Graft copolymerization is a novel technique for the development of cellulosed based materials. Radiation is a very convenient method for graft initiation cause in allows considerable degree of control of structural factors of the polymer by careful selection of the dose and dose rate. This is why jute is photografted with (Ethylenglycol di-methylacrylat) EGDMA monomer to improve its characteristics.

MATERIALS AND METHODS

Materials

Cellulosed based jute yarns. Ethylenglycoldimeth acrylat (EGDMA), methanol (CH₃OH) extra pure (Merck, Germany); acetone (CH₃COCH₃), Urea, Potassium permanganate (KMnO₄) (BDH, UK) and the photoinitiator; benzyl dimethyl ketal (IRGACURE 651) was supplied by Ciba Geigy.

Methods

Sample preparation

Locally collected jute yarns were cut into 25cm and washed with acetone to remove the foreign particles. Then dried in the oven at 105^o C for about 24 hrs to remove moisture and stored in the desiccators. The weight of the individual fiber was taken by the electronic balance separately.

Soaking solution

A series of formulation was prepared using EGDMA (3-20% v/v) monomer in methanol (95-78% v/v) along with 2% photoinitiator IRGACURE – 651. The 100 ml beakers were taken for different formulations i.e. EGDMA of 3, 5, 10 and 20% gradually and mixed with MeOH of 95, 93, 88 and 78% respectively by adding 2% photoinitiator. The mixtures were stirred thoroughly by a glass rod until a clear transparent solution was obtained and the beakers were covered with foil paper to resist degradation of photoinitiator. The beakers were marked as (M_1-M_4) 3, 5, 10, 20% EGDMA solutions.

Determination of polymer loading

The weighted and dried samples were kept immerged in different formulations to determine the polymer loading. Ten individual weighted fibers were taken for each formulation. The fibers were marked according to their weights and dipped in each of the solutions and kept immerged for different period of time (soaking times). The soaking time varied from 3-20 minutes (3, 5, 10, 20 min). After soaking in the formulations, they were passed under UV (ultraviolet) curing lamp for different radiation doses (2, 4, 6, 8, 10 passes). The UV manicure Machine (IST Technik, Germany) with intensity of the lamp 2 kW 9.5 A current and the wave length 254-313 nm a conveyer speed of 4m/min. For each pass the samples moved 0.50 m under the UV conveyer belt speed^[15]. It was necessary to pass the treated substrates through the UV radiation several times to ensure curing. The irradiated fibers were then left in dark for 24 hrs to complete of the polymerization. After 24 hrs the fibers were washed with acetone for about 5 minutes to remove unreacted monomer and dried in the oven for 2 hrs at 105° C. The dried fibers were then cooled in the desiccators and were weighed for the second time in the electronic balance. The polymer loading of EGDMA treated jute fibers were calculated by deducting the final weight from the initial weight.

PolymerLoading(%) =
$$\frac{W_t - W_0}{W_0} \times 100$$
, Where, W_t

= weight of the treated dry sample, W_0 = weight of the dry sample before treatment. The polymer loading of individual fiber was added to calculate the polymer loading of each of the formulations for each of the soaking times.

Determination of tensile properties

Various tensile properties such as tensile strength and elongation at break of both treated (T₁, E₁ respectively) and untreated virgin yarns (T₂, E₂ respectively) were measured with the universal testing machine (INSTRON, model 1011, UK). The increment in tensile properties after UV curing was expressed as tensile strength factor Tf (Tf = T₁/T₂) and elongation factor Ef (Ef = E₁/E₂).

Surface treatment with KMnO₄

Different KMnO₄ solution containing 0.03, 0.05, 0.1% (v/v) respectively were prepared. Virgin jute

yarns were subjected to pretreatment with KMnO_{4} . The fibers were then dipped in the beakers containing solutions for 5 minutes. Then they were washed with running water until KMnO_4 was removed completely. After drying the fibers were treated with monomer. The polymer loading, tensile properties were determined.

Addition of additive

An additive urea was added to impress better properties to the treated samples. For this purpose different small concentration of urea (0.5, 1 and 2% w/v) was incorporated with the optimized formulation during the treatment of the pretreated jute yarn. The grafting procedure was same as described previously. Tensile properties of these treated samples were determined. Water uptake, weathering effect and soil degradation studies were also performed.

Determination of water uptake

For the water uptake; both treated and virgin samples fibers were immerged in water in a static bath at 25°C for 30 minutes. The fibers were taken out of the water container at constant time interval and wiped carefully and then weighed. The weight gain of the samples was calculated.

Simulated weathering test

The control and treated fibers were subjected to simulated weathering test by using weather tester (Model: QUV, Q-Panel Company, U.S.A, Volts: 230, Hz: 50, Watts: 150) for 600 hours which was performed in alternating cycles of sunshine over 4 hrs ($65\pm2^{\circ}$ C), dews and condensation 2 hrs ($45\pm2^{\circ}$ C). The samples were then dried in the oven for half an hour, weighed and their mechanical properties were measured.

Determination of degradable properties in soil and water environment

To monitor the degradable properties of the samples, the fibers were weighed individually. Some samples were buried in the soil (20% moisture) and some were immerged in the water and washed thoroughly with running water. Then the fibers were dried in the oven and the loss of weight was calculated. Degradability was monitored for 60 days in 15 days interval.

IR analyses

The acetone-extracted jute yarn were dried at 105°C for 24 hrs and powdered in a mortar. Fourier transforms infrared spectroscopy (FTIR) spectra are characterized by the pure potassium bromide pellet technique on RX1 (Model IR-470, Shimadzu, Japan, region (4000-400 cm). The jute compounds for IR analysis were frequently obtained by mixing and grinding a small amount of the powdered jute materials (1 mg) with dry and pure KBr (200 mg). The thorough mixing and grinding were accomplished in a mortar by a pistler. The powdered mixture was then compressed in a metal holder under a pressure for 8-10 tonnes to produce a pellet. The pellet was then placed in the path of the infrared beam for measurements.

Statistical analysis

For measuring the data 5 samples in each replicate were tested. Analysis of variance and Duncan's multiple-range tests were used to perform statistical analysis of all results using PASW Statistics Base 18 software (SPSS Inc., Chicago, IL, USA). Differences between means were considered to be significant when $p \le 0.05$.

RESULTS AND DISCUSSIONS

Optimization of grafting condition of monomer EGDMA (UV curing method) Optimization of monomer concentration (EGDMA)

Polymer loading

Polymer loading (PL) values of the yarns were calculated at the weight gain (%) after they were cured under UV radiation. The PL values of the EGDMA treated yarns at different radiation intensities as a function of monomer concentration are shown in the Figure 1. The lowest values were observed at low monomer concentration where increases with EGDMA concentration and is maximum (20%) with 5% EGDMA at 6th pass of UV radiation. At low monomer concentration the vinyl monomer promotes the rapid propagation reaction with the help of photoinitiator leading to network polymer structure through graft co-polymerization reaction via their double bond. As the EGDMA concentration is increased the amount of residual concentration is also increased with consequence of faster rate of formation of three dimensional network structures causing restricted mobility. After the attainment of the maximum the decrease in PL values at higher monomer concentration may be caused by two factors. At higher EGDMA concentration radical-radical recombination process may be dominating creating more homo-polymer rather than monomer + cellulose backbone reaction. The other reason could be that the swelling of the cellulose backbone with MeOH is insufficient due to low methanol concentration. As a result monomer molecules are incapable of penetrating the cellulose molecules in the presence of low solvent concentration. This may cause a smaller number of reacting sites at the cellulose backbone and thus continue to reduce the active sites as MeOH concentration decreases with higher EGDMA concentration. The crosslinking rate especially during the early stages of radiation is proportional to EGDMA concentration. The EGDMA increase the radical-radical reaction termination and hence decrease the extent of scission reaction and oxidation.

Tensile properties

The tensile properties like tensile strength and elongation at break are expressed as tensile strength

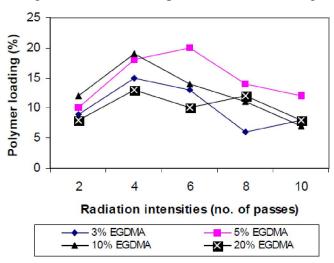


Figure 1 : Polymer loading values of the treated jute yarn against radiation intensities as a function of EGDMA monomer concentration, soaking time 5 minutes

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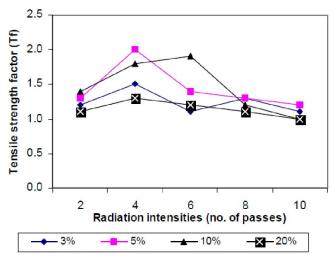


Figure 2 : Tensile strength factor of the treated jute yarn against radiation intensities as a function of monomer concentration, soaking time 5 minutes

factor (Tf) and elongation at break factor (Ef). The tensile strength factor Tf is measured as T_1/T_2 where, T_1 is the tensile strength of the treated yarn and T_2 is that of the untreated yarn. The tensile strength values are plotted in the Figure 2 against radiation intensities as a function of monomer concentration. The figure indicates that the Tf values increase with the increase of monomer concentration which is the maximum (2.0) with 5% EGDMA and after that it decreases with the increase of Tf with the EGDMA concentration. The increase of Tf with the EGDMA concentration. And the decrease is because of the radical-radical recombination reaction among the growing EGDMA molecules.

The elongation up to the breaking point of treated and untreated jute yarns samples were expressed as elongation factor (Ef) as $Ef = Eb_t/Eb_u$, where Eb_u and Eb_t are the elongation at break of the untreated and treated yarns respectively. The enhancement of elongation is depicted in Figure 3 against reaction time as a function of monomer concentration. The highest enhancement of elongation (1.4) was achieved by the 4 UV passes with 5 % EGDMA.

Effect of surface treatment with KMnO₄

Polymer loading

The roles of KMnO_4 on mechanical properties improvement of jute were described^[16]. Jute yarns were subjected to surface treatment with KMnO_4 .

The yarns were soaked for different soaking times (1, 5 and 10 minutes) with KMnO₄ of varying concentrations (0.03, 0.05, 0.1 % w/v respectively). Then they were treated with 5% EGDMA and giving UV radiation. Among the different concentrations, 0.05% KMnO₄ at 5 minute of soaking at 4th pass produced the best results (PL = 21% and Tf = 2.5). The results of PL with respect to KMnO₄ concentration and radiation intensities are shown in the Figure 4. This cellulose free radical is quite stable which supports the higher rate of reaction with monomer molecules. Thus permanganates induce grafting of EGDMA on to cellulose molecules of jute yarns.

Tensile properties

The tensile properties (*Tf* and *Ef*) of the treated yarns with respect to $KMnO_4$ concentration are

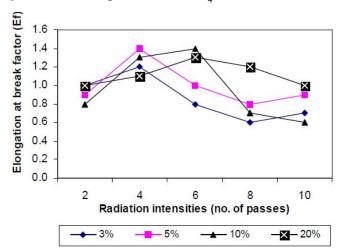


Figure 3 : Elongation at break factor of the treated jute yarn against radiation intensities as a function of monomer concentration, soaking time 5 minutes

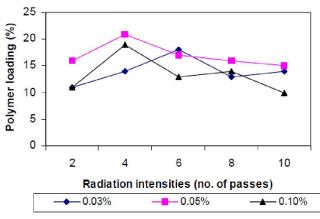


Figure 4 : Effect of KMnO₄ on the polymer loading of treated jute yarn, treating time 5 min [5% EGDMA]

shown in the Figure 5-6. The highest Tf (2.5) is achieved with 0.05% (KMnO₄) concentration at 4th pass. At low concentration of KMnO₄, there occurs minor change in the PL values, but more in the tensile properties. Both PL and mechanical properties increase with KMnO₄ concentration and is highest (1.9) with 0.05% KMnO₄. On increasing the concentration the properties decrease very much due to the degradation of yarn. The radiation intensity (number of passes) causes the degradation of the properties.

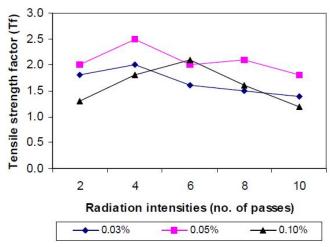


Figure 5 : Effect of $KMnO_4$ (concentration) on the tensile strength factor of the treated jute yarn, soaking time 5 minutes

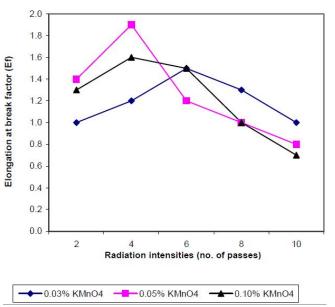


Figure 6 : Effect of $KMnO_4$ (concentration) on the elongation at break factor (Ef) of treated jute yarn, soaking time 5 minutes

Effect of additive on the EGDMA jute yarns

To improve the properties an additive Urea of different concentrations (0.5 - 2% w/v) was added for optimization solution concentration and its effect on the properties. Enhanced properties (PL=24%, Tf = 2.3) were achieved with 1 % Urea. The results of PL, Tf and *Ef* are presented in the Figure 7-9 respectively. Urea possesses >C=O group adjacent to a nitrogen atom having a lone pair of electrons which are activated to form a bridge between the monomer and the cellulose through the additive. Oxygen has more affinity towards electrons; thus, the electron clouds are densely populated around the oxygen atom of >C=O groups, thereby pulling more electrons towards the oxygen from the

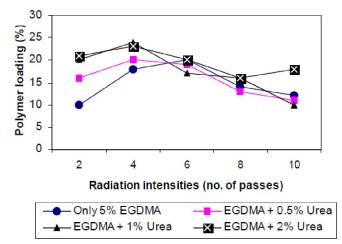


Figure 7 : Effect of urea on the polymer loading values of the treated jute yarn as a function of urea concentration, soaking time 5 minutes

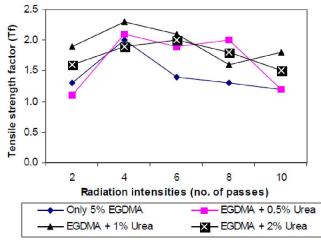


Figure 8 : Effect of Urea on the tensile strength factor of the treated jute yarn as a function of urea concentration, soaking time 5 minutes

area of the nitrogen atom or its vicinity, creating some favorable conditions for the augmentation of the monomer and the additive units with the cellulose backbone polymer of the substrate. Urea is an inclusion compound whose properties would assist partitioning by complexing with monomer. This can lead to an increase in monomer concentration at a grafting site and thus enhance reactivity at that site.

Effect of pretreatment with KMnO_4 and addition of Urea

Again urea was incorporated with 5% EGDMA solution when the permanganate treated fibers were treated with the optimized formulation. Then more enhancement of properties were observed with 0.05% KMnO₄ and 1% Urea at 5 minutes of soaking

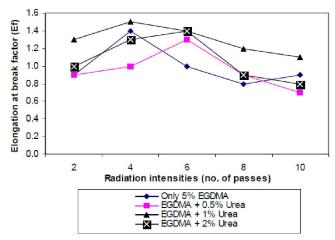


Figure 9 : Elongation at break factor (Ef) of the treated jute yarn against radiation intensities as a function of urea concentration, soaking time 5 minutes

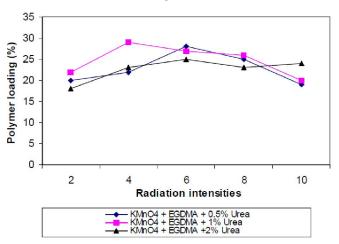


Figure 10 : Elongation at break factor (Ef) of the treated jute yarn against radiation intensities as a function of urea concentration

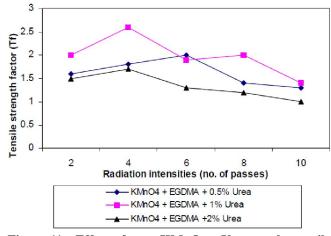


Figure 11 : Effect of urea $KMnO_4$ + Urea on the tensile strength factor (Tf) of the treated jute as a function of urea concentration

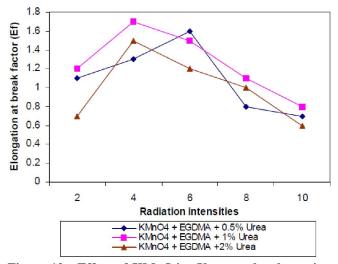


Figure 12 : Effect of KMnO4 + Urea on the elongation at break factor (Ef) of the treated jute yarn against radiation intensities as a function of urea concentration

which are shown in the Figure 11-12. The PL value reached to 29% (Figure 10) and the Tf was 2.6 (Figure 11) and the Eb was 1.7% (Figure 12).

Water uptake

Water uptake values of the virgin and treated yarns were calculated by immerging the yarns in water contained in a static bath at the room temperature. The yarns were taken out of water after constant time interval and their weight gain were calculated. The results of water uptake values of the treated and untreated yarns are shown in the Figure 13. All the treated fibers toke up water within the first 20 minutes of soaking time and then the values were constant. But the untreated fibers continued to take up water through out the period of monitoring (110 minutes). The minimum amount of water was taken up by the 5% EGDMA treated yarns and the highest by virgin yarns. The hydroxyl groups of the cellulose molecules were filled up by the monomer molecules. So the water uptake value of that system was lowest.

IR spectra of jute

The characteristic features of the infrared (IR) spectra of jute fibers are due to its lignin and hemicellulose constituents. The IR spectra of jute yarn were taken with good resolution and are shown in the Figure 14; peak assignments are described^[17]. The Hbonded O-H stretching causes the board band between 3600 and 3200 cm⁻¹. The O-H is present in the cellulose and hemicellulose of the jute fiber. The usual glucose linkage in the cellulose is characterized through the C-H stretching around the 2940-2890 cm⁻ ¹ band and 1377 cm⁻¹. The linkage vibration is also observed at 895 cm⁻¹. The acetylated xylan bands are noticed at 1728 and 1246 cm⁻¹. The band at 1740 cm⁻¹ ¹ is due to the hemicellulose constituent (carboxylic and acetyl functionalities and ester linkages). It is necessary to mention that the C=O stretching of the uronic acid of the hemicellulose, and the unconjugated keto groups of the lignin can be observed around 1700 cm⁻ ¹, but the conjugated aryl carbonyl stretching can be noticed at 1644 cm⁻¹. The asymmetric C-H deformation present in methyl and methylene groups is ob-

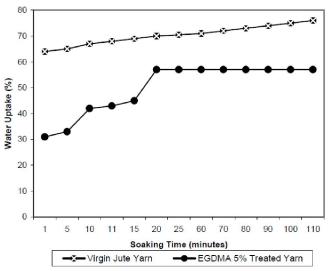


Figure 13 : Water uptake values of virgin jute yarns and 5% EGDMA treated jute yarn

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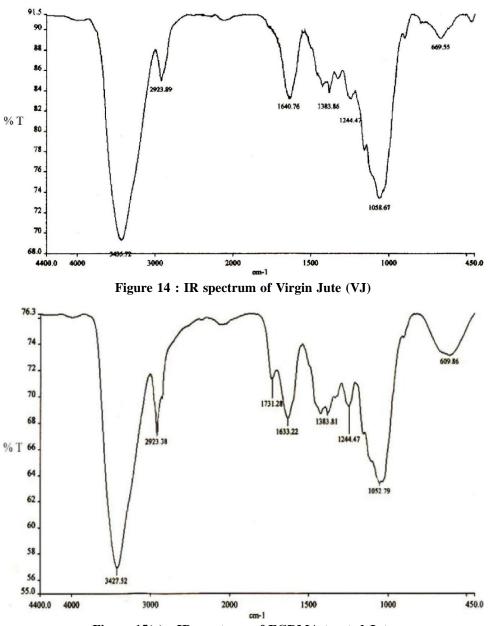


Figure 15(a) : IR spectrum of EGDMA treated Jute

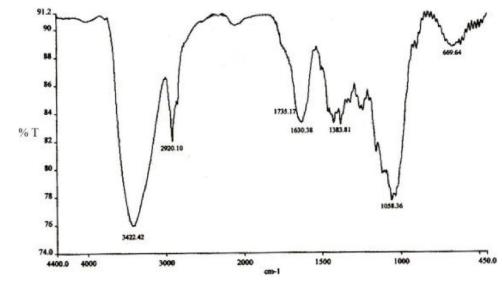
served by the 1454 cm⁻¹ band. The aromatic skeleton of lignin is observed at 1418 cm⁻¹ and 1497 cm⁻¹. The peak at 1318 cm⁻¹ is attribute to the aromatic ring breathing with C-O stretching in syringyl units of lignin; but the peak at 1111 cm⁻¹ is due to the aromatic C-H in plane deformation of guaiacyl and syringyl units. The anti-symmetric bridge oxygen stretching is observed at the 1156 cm⁻¹ peak.

IR Spectra EGDMA grafted jute

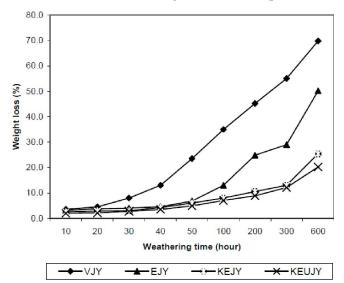
The IR spectra of EGDMA grafted jute yarns in the presence of MeOH are shown in Figure 5.4 & ________ with the increased grafting, the characteristic polymer peaks become prominent. The polymer is formed through the reaction of EGDMA. The C=O vibration at 1728 cm⁻¹ increases substantially with enhanced polymer loading; this is because the C=O groups are present with the acrylated group of EGDMA molecule. This observation indicates the presence of EGDMA moiety with the jute cellulose, demonstrating a concomitant grafting of EGDMA with the jute matrix. This is, therefore, a direct evidence of grafting copolymer of EGDMA and jute.

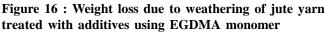
Simulating weathering effect

Virgin jute yarn (VJY), jute yarns treated with 5%









EGDMA (EJY), 0.05% KMnO₄ + EGDMA (KEJY), KMnO₄ + EGDMA + Urea (1%) (KEUJY) were exposed to accelerating weather tester over a period of about 600 hrs to study the degradation properties. The weight loss and the reduction of tensile properties particularly TS and Eb of the sample were periodically determined which are shown in Figure 16 respectively. Different formulations cause different extents of strength loss but the pattern is similar for all the cases. The highest weight loss (69%) was found in for the control jute yarn and the 50, 25 and 20 % were observed for the treatment with EGDMA + VJY (virgin jute yarn), KMnO₄ + EGDMA + VJY and Urea + KMnO₄ + EGDMA + VJY respectively.

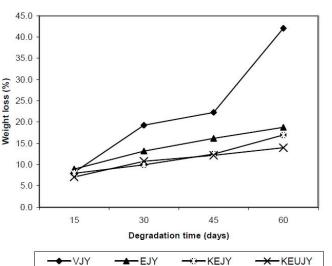


Figure 17 : Weight loss due to soil degradation of jute yarn treated with additives using EGDMA monomer

Soil and water degradation

Weight loss was monitored for both soil & water, the virgin samples showed the highest loss of weight within this period of time Figure 17. The treatment is effective for degradation while the weight loss increases due to the treatment EGDMA, KMnO₄ and urea respectively. The highest weight loss 42, 19, 17 and 14 % was achieved for the control, EGDMA, EGDMA + KMnO₄ and EGDMA + KMnO₄ + Urea respectively.

CONCLUSION

The physico-mechanical properties of jute yarns

were treated with methanolic solution of EGDMA in along with photoinitiator IRGACURE 651 (2%) and cured under UV radiation. Thermal treatment was also done with Benzoyl peroxide catalyst at a temperature of 80°C. Concentration of monomer; soaking time and radiation intensity were optimized with respect to mechanical properties. The Photocuring method has enhanced tensile strength (TS factor Tf = 2), elongation at break factor, (Ef = 1.4)and polymer loading (20%) were achieved with 5% EDGMA (at 5 minutes of soaking time). Urea (0.5 -2%) was added to the optimized solution (5% EGDMA) and 1% Urea showed the best properties (PL = 2.4 %, Tf = 2.3). KMnO₄ treated yarns produced the best results (PL = 29%, Tf = 2.6, Ef = 1.7) along with Urea (1%).

In the case of thermal curing method enhanced tensile strength (Tf = 1.8, Ef = 1.4) and polymer loading (13%) were achieved at the temp. of 80° C in 30 minutes of reaction time. The KMnO₄ treated yarns produced the best results (PL = 20%, Tf = 2.6, Ef = 1.5). This is a unique achievement in the sense that the mechanical properties of the jute yarns can be improved by curing of KMnO₄ treated yarns followed by addition of additive (urea) rather than curing of jute yarns without pretreatment or any additive. Both KMnO₄ and Urea are very cheap. This method of modification of the jute yarn appears to be economical.

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