

γ – Irradiated Jute Reinforced Polypropylene Composites: Effect of Mercerization and SEM Analysis

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Abstract

Jute fibres (*Corchorus olitorius* L.), an environmentally and ecologically friendly product, were chemically modified and treated as surface treatment which was carried out by mercerizing jute fabrics with aqueous solutions of NaOH (5, 10 and 20%) at different soaking times (30, 60 and 90 minutes) and temperatures (0, 25 and 50°C). These mercerized jute fabric reinforced polypropylene composites were fabricated by composition molding technique and equated with virgin jute fabric reinforced polypropylene composites, fabricated by same technique. The above composite samples were compared by evaluating the mechanical parameters such as tensile strength, tensile modulus, bending strength, bending modulus. The effect of mercerization on weight and dimension of jute fabrics was studied. Mechanical properties of mercerized jute-PP composites were measured and found highest at 20% NaOH at 0°C for 60 min soaking time. Alkali treatment helped in the development of hydrophobicity and reduction in volume fraction of the porosity. This may be due to the better fibre matrix interface adhesion caused due to the fibre surface treatment by alkali. The optimized formulation was irradiated by γ radiation at different dosage (100, 150, 200, 250 & 500 Krad). Among them 250 Krad showed highest mechanical properties.

Keywords: Jute fiber; Polyethylene; Composite; Tensile strength; Bending strength; Bending modulus

Introduction

Environmental awareness and depletion of the petroleum resources are among vital factors that motivate a number of researchers to explore the potential of reusing natural fiber [1-4] as an alternative composite material in industries such as automotive component [5,6], packaging materials [7], insulation [8,9], acoustic absorption panel [10,11] and building materials [12,13]. Natural fibers are available in abundance, low cost, lightweight polymer composite and most importance its biodegradability features [14-17], which often called “ecofriendly” materials. Handling of the natural fibers causes little concerns in terms of health, safety and energy can be recovered from it in an environmentally friendly way. Natural fibers like jute, kenaf, flax, hemp, coir and sisal have already earned a testimony of success as reinforcing material in engineering markets such as in automotives, construction as well as in packaging industries.

Among all the natural fibers, jute appears to be the most useful, economical and exhibits moderately higher mechanical properties. High cellulose content and low microfibril angle of jute fiber provide good reinforcement in polymer matrix. Moreover it is fully bio degradable and eco-friendly. However, jute fiber due to hydrophilic nature is inherently incompatible with hydrophobic thermoplastics, such as polyolefin. So, jute fibers suffer poor interfacial adhesion with non-polar hydrophobic matrix. To overcome this limitation, development of strategies for surface modification of natural fibers or polymer matrices is needed in order to create a strong adhesion at the interfaces of two different phases. Many attempt such as physical and chemical treatments have been adopted to improve the interfacial adhesion. Mercerization is one of the most used chemical treatments of natural fibers when they are used as reinforcement in polymer matrices [18-21]. Treatment of jute and sisal fibers with 5% aqueous NaOH solution showed significant change on the crystallinity of fibers. Alkaline treatment also significantly improved the mechanical and dynamic mechanical behaviours of natural fibre reinforced composites.

The major fibers constituents (α -cellulose, hemicellulose and lignin) of the untreated and alkaline-treated jute fibre samples were determined by Exequiel S. Rodríguez et al. [22]. The results are listed as in Table 1.

Gassan et al. [23] observed that shrinkage of the fibers during mercerization had the most significant effect on the fiber structure and as a result, on the fiber mechanical properties such as tensile strength, modulus and toughness. Rajulu et al. [24] showed that the maximum tensile strength, modulus and density of the mercerized lignocelluloses fabric were attained at 4 hour of alkali treatment when fabric were treated with 5% aqueous NaOH solution for 0, 2, 4, 6 and 8 hours. The alkali treatment also increased the surface roughness of the fibers. Khan et al. [1] prepared a series of composites jute, mercerized jute and manmade cellulose tyre cord yarn Codenka in polypropylene/ethylene block copolymer (PP) with maleic acid anhydride grafted PP (MAPP) as a coupling agent. They observed that the partial substitution of jute instead of Codenka leads to enhance stiffness properties of the composites. On the other hand, impact strength decreases with the increasing jute fraction. Liu et al. [25] showed that combination of NaOH and MAPP emulsion is a good adhesion promoter for jute fiber mat/PP composite. The present research work deals with the extensive study on the properties of mercerized jute fabrics in terms of weight and dimension at different soaking time and temperature. The consequence of the mercerization on the mechanical and thermal properties of the mercerized jute composites was explored.

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Received May 19, 2016; **Accepted** June 16, 2016; **Published** June 26, 2016

Citation: Islam Bossunia MT, Poddar P, Hasan MM, Hossain MT, Gulenoor F, et al. (2016) γ – Irradiated Jute Reinforced Polypropylene Composites: Effect of Mercerization and SEM Analysis. J Material Sci Eng 5: 266. doi:10.4172/2169-0022.1000266

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Component (%)	Treated	Untreated
α -Cellulose	80.3	71.1
Hemi-cellulose	9.6	15.9
Lignin	7.6	11.8
Other components (ash, water, pectins)	2.5	1.2

Table 1: Major fibers constituents of the treated and untreated alkaline treated juice fibre sample.

Mercerization Treatment and γ Irradiation

Mercerization is an alkali treatment process. It is widely used in textile industry [26]. The standard definition of mercerization as proposed by ASTM D1965 is: the process of subjecting a vegetable fiber to an interaction with a fairly concentrated aqueous solution of strong base, to produce great swelling with resultant changes in the fine structure, dimension, morphology and mechanical properties [2]. Therefore, mercerization is a chemical modification process that changed the chemical constituent behavior in natural fiber. The effect of alkali on cellulose fiber is a swelling reaction, during which the natural crystalline structure of the cellulose relaxes. Native cellulose (i.e., cellulose as it occurs in nature) shows a monoclinic crystalline lattice of cellulose-I, which can be changed into different polymorphous forms through chemical or thermal treatments.

The type of alkali and its concentration will influence the degree of swelling, and hence the degree of lattice transformation into cellulose-II. It has been reported that Na⁺ has got a favorable diameter, able to widen the smallest pores in between the lattice planes and penetrate into them. Consequently, sodium hydroxide (NaOH) treatment results in a higher amount of swelling. This leads to the formation of new Na-cellulose-I lattice, a lattice with relatively large distances between the cellulose molecules, and these spaces are filled with H₂O molecules. In this structure, the OH groups of the cellulose are converted into O-Na-groups, expanding the dimensions of molecules. Subsequent rinsing with water will remove the linked Na-ions and convert the cellulose to a new crystalline structure, i.e., cellulose-II, which is thermodynamically more stable than cellulose-I. NaOH can cause a complete lattice transformation from cellulose-I to cellulose-II. Addition of aqueous sodium hydroxide (NaOH) to natural fiber promotes the ionization of the hydroxyl group to the alkoxide. The following reaction takes place as a result of alkali treatment [27-29]:



As reported in much literature, natural fiber chemical constituent consists of cellulose and other non cellulose constituent like hemicellulose, lignin, pectin and impurities such as wax, ash and natural oil [30,31]. This non cellulose material could be removed by appropriate alkali treatments, which affect the tensile characteristic of the fiber [29,32].

Mercerization was found to change fiber surface topography, and the fiber diameter was reported to be decreased with increased concentration of sodium hydroxide concentration [33]. Mercerization treatment also results in surface modifications leading to increase wet ability of coir fiber polyester resin as reported by Prasad et al. [34]. It is reported in that alkaline treatment has two effects on the henequen fiber: (1) it increases surface roughness, resulting in better mechanical interlocking; and (2) it increases the amount of cellulose exposed on the fiber surface, thus increasing the number of possible reaction sites [35]. Consequently, alkaline treatment has a lasting effect on the mechanical behavior of natural fibers, especially on their strength and stiffness. In

order to find out the effect of γ radiation, optimized formulation was irradiated at different dosage (100, 150, 200, 250 & 500 Krad).

Materials and Experiment

Materials

Jute fibers (Hessian Cloth) were supplied by Jute Corporation, Dhaka, Bangladesh. Area density of jute fabrics is 0.0224 g/cm². Sodium hydroxide and Polypropylene pellets (Trade name: Cosmoplene) were procured from Merck, Germany and Polyolifen Company, Singapore. Pte Ltd., respectively. The melt flow index (g/ 10 mins) and density (g/ cm²) of PP are 3.0 and 0.9 respectively.

Surface modification of jute fabrics

Jute fabrics were cut into the size of 40×20 cm and treated with different concentrations of NaOH (5, 10, and 20%) in aqueous medium for soaking time of 30, 60 and 90 mins at room temperature. Jute fabrics were also soaked with NaOH solution at 0°C, 25°C and 50°C for a specific concentration and soaking time. After soaking jute fabrics were washed with running tap water until there was no trace amount of NaOH present in the fabrics. The detection of NaOH was carried out by litmus test. The jute fabrics were then dried in an oven at 105°C until the fabrics reached a constant weight. After drying jute fabrics were kept in a desiccator for composite fabrication and γ irradiation.

γ Irradiation of mercerized jute fiber

The optimized formulation was irradiated by γ radiation (Co-60) from 100 Krad to 500 Krad doses at a dose rate of 300 Krad / h.

Fabrication of composites

PP sheets of desired size (15 cm × 13 cm) were prepared by compressing PP granules in the heat press (Carver, Inc., USA, Model 3856) at 190°C for 5 mins under 5 ton pressure. Jute fabrics were cut into small pieces of 15×13 cm size. For composite fabrication, sandwich was made by alternatively placing four layers of pre-weight jute fabrics inside five layers of PP sheets. The sandwich was then kept between two steel plates with a composite fabrication mold (thickness: 2 mm). The sandwich with this arrangement was compressed in the heat press at the same conditions applied for PP sheet preparation. After being heat pressed, PP sheets and composites were cooled to another press (Carver, Model: 4128) operated in a cooling mode. The average thickness of the composites is 2 mm.

Mechanical testing

Tensile test of composite sample was carried out by a universal testing machine using a gauge length of 20 mm and cross head speed of 10 mm/min. Three point bending strength of the composites sample was measured in the same machine with a span length of 58 mm and cross head speed of 2 mm/min. The test was carried out according to DIN (Deutsches Institut fur Normung meaning German Institute for Standardisation) 53455 and DIN 53452 standard methods, respectively. Impact strength (Charpy) of the composites and PP was carried out in an impact tester (MT-3016, Pendulum type, Germany) following ASTM (American Society for Testing and Materials) D 6110-97. All the results were taken as the average value of five samples.

Scanning electron microscopic (SEM) investigation

The surfaces of the composites of both treated and untreated composite samples were examined using a Hitachi S-4000 field emission scanning electron microscope, operated at 5 kV.

Results and Discussion

Effect of mercerization on physical properties of hessian cloth

Weight of jute cloth: Weight of the jute fiber is largely depends on the concentration of NaOH and the time period of treatment. Weight of the jute fiber decreases with the increasing of concentration of NaOH. At the same concentration of NaOH the weight depends on the time periods. The weight of jute fiber follows a decreasing trend from 5% to 20% of NaOH and at the same concentration it decreases from 30 min to 90 min. the maximum weight loss is 20.37% which is for 20% NaOH (90 min) (Figure 1).

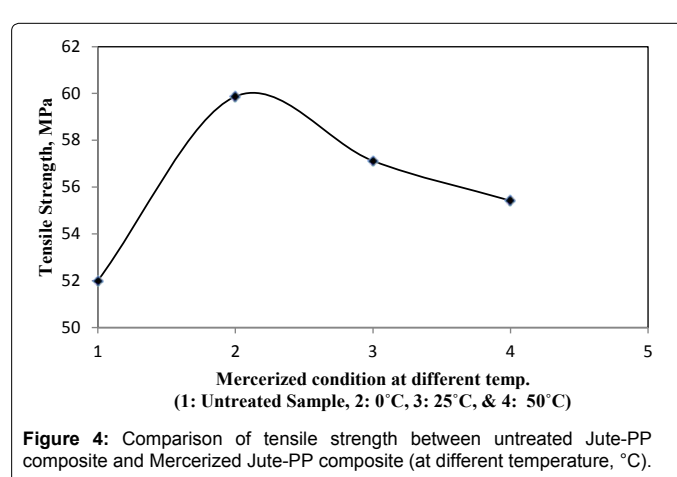
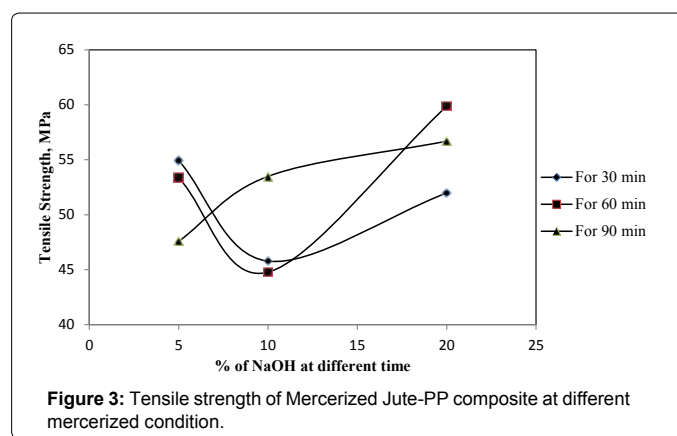
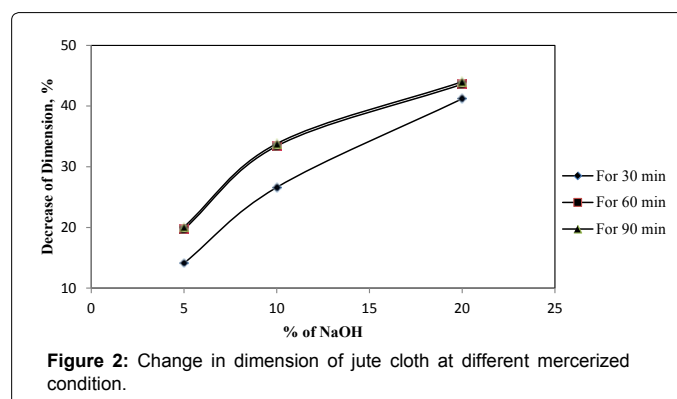
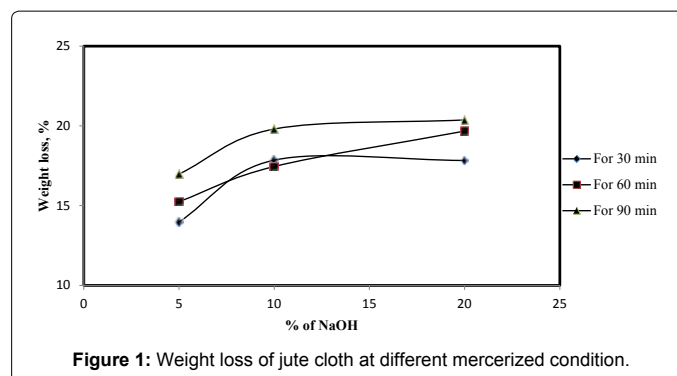
Dimension of jute cloth: The dimension of the jute fiber are also depends on the concentration of NaOH and the time period of treatment. Dimension of the jute fiber decreases with the increase of concentration of NaOH. At the same concentration of NaOH the dimension depends on the time periods. The dimension of jute fiber follows a decreasing trend from 5% to 20% of NaOH and at the same concentration it decreases from 30 min to 90 min. the highest value obtains is 44.01% which is for 20% NaOH (60 min) (Figure 2).

Effect of mercerization on mechanical properties of jute composites

Tensile strength of jute/PP composite: Mechanical properties of composites were strongly influenced by adhesion between the matrix and fibers. The Figures 3 and 4 showed that the treatment with NaOH had the highest effect on tensile strength (TS) producing composites with the best tensile properties. The tensile strength also depends on the concentration of NaOH, time period and finally on temperature. It is clearly indicated in Figure 3 and 4, tensile strength is highest in 60 min for mercerized condition of 20% NaOH and for 20% conc. NaOH (60 min) it is higher at 0°C (59.87 MPa) than 25°C and 50°C. This may be due to the interfacial adhesion between jute fiber and PP was increased by treatment of the jute fibers with alkali and it is optimum for 20% conc. NaOH (60 min) at 0°C mercerized condition.

Bending strength of jute/PP composite: The dependency of bending strength on mercerization of jute fiber, of jute composite is investigated. Mercerized jute composites have higher bending strength than the untreated jute composites for max cases. The bending strength also depends on the concentration of NaOH, time period and finally on temperature. At 5% and 10% concentration of NaOH, it decreases from 30 min to 90 min. while at 20% conc. NaOH, it is highest for 60 min. On the other hand, for 20% conc. NaOH (60 min) it is higher at 0°C than 25°C and 50°C. This value is 72.41 MPa. Observation proves that there is a poor interaction between fiber and matrix in the untreated composites. In the chemically treated composites, interaction between fibers and matrix is strong and fibers are uniformly spread in the composites thus prevented to form a bundle (Figures 5 and 6).

Bending–E-modulus of jute/PP composite: Bending- E-modulus of both untreated and chemically treated composites was studied (Figure 7). Mercerized jute composites have higher bending -E-modulus than the untreated jute composites showed in Figure 8. The variation of Bending –E-modulus with the concentration of NaOH, time period and finally on temperature is given in Figures 7 and 8 respectively. The mercerized jute composite with different mercerized condition of concentration of NaOH did not show the same pattern with increase of time period. The highest value obtained is 1.96 GPa at 0°C (for



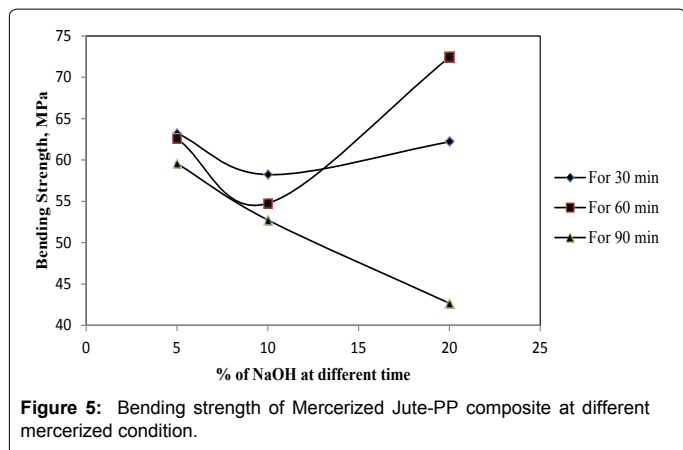


Figure 5: Bending strength of Mercerized Jute-PP composite at different mercerized condition.

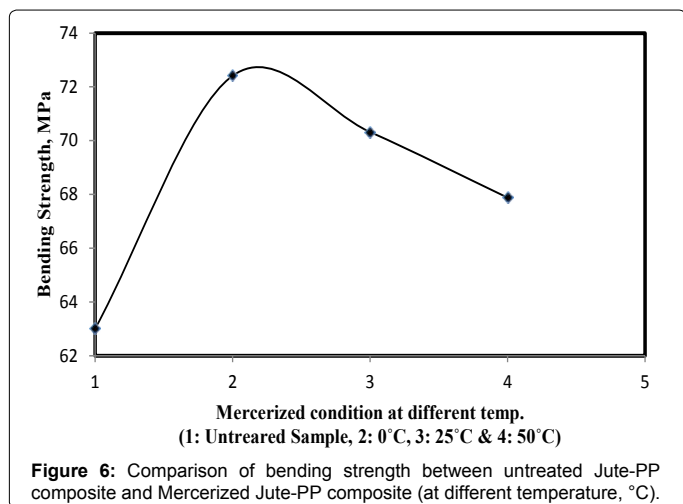


Figure 6: Comparison of bending strength between untreated Jute-PP composite and Mercerized Jute-PP composite (at different temperature, °C).

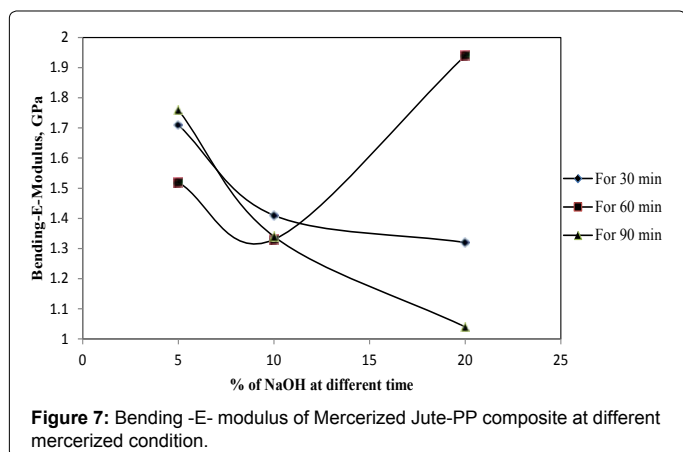


Figure 7: Bending -E- modulus of Mercerized Jute-PP composite at different mercerized condition.

20% NaOH treated for 60 min.) This may be due to elasticity of jute fibers which depends simultaneously both on amounts of fiber and chemical treatment process. The significant information of this part of research is the improvement of BE for jute/PP composites. Hence, it can be concluded that using chemical treatment have the potentially to improve BE to the required range.

Tensile strength and bending strength of γ irradiated composites: Effect of γ radiation on mechanical properties of optimized (20% NaOH

treated at 0°C for 60 min.) mercerized jute composites at different doses was studied. The highest value of TS and BS was 58.36 and 70.47 MPa respectively for 250 Krad. Whereas the TS and BS for untreated sample was 52 and 63 MPa respectively. γ radiation dose may be due to the intercross-linking between the fiber and matrix which enhance the mechanical properties of the composites (Figure 9).

SEM analysis

Figures 10 and 11 indicates some gaps between jute fiber and matrix which is responsible for the low mechanical properties. From the treated SEM image it is found that gaps between fiber and matrix are not observed. Due to NaOH treatment, organic constituents of jute such as lignin, pectin, traces of metals and impurities, etc. might be dissolved and the surface of the cellulose in jute become sleek which enhanced fiber matrix adhesion. Again, γ radiation may generate some active sites and reduce more moisture which might be responsible for better fiber matrix bond.

Conclusion

Mercerization of jute fiber carried out in different concentration (5, 10 and 20%) of NaOH and for different time periods (30, 60, 90 min) and also at different temperatures (0°, 25° and 50°C). Polypropylene (PP) based composites are fabricated by using untreated and treated jute fibers by compression molding. Tensile strength (TS), bending strength (BS) and Bending E Modulus (BE) of the composites studied.

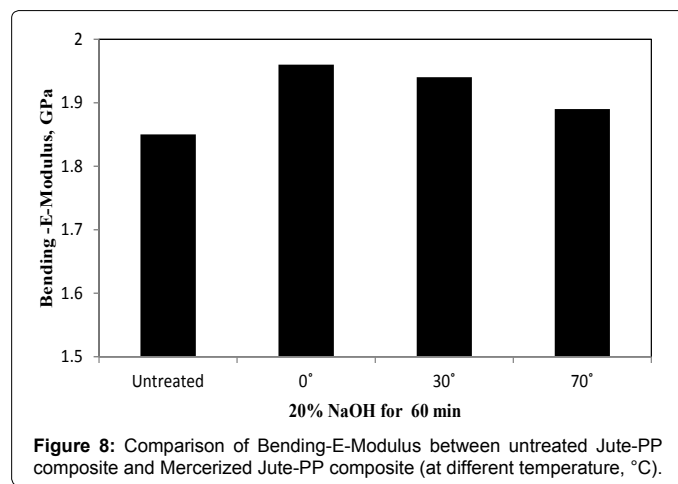


Figure 8: Comparison of Bending-E-Modulus between untreated Jute-PP composite and Mercerized Jute-PP composite (at different temperature, °C).

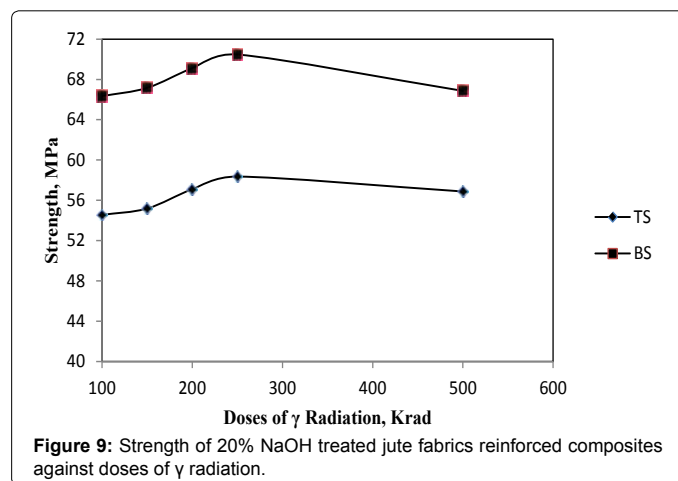


Figure 9: Strength of 20% NaOH treated jute fabrics reinforced composites against doses of γ radiation.

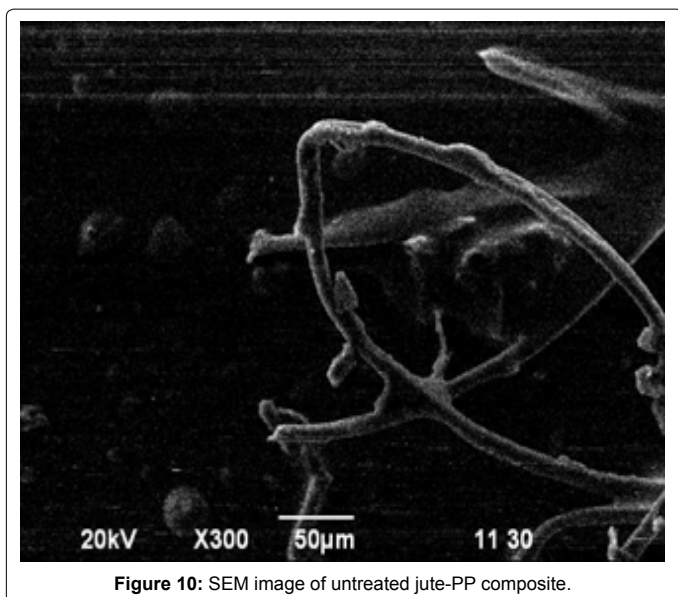


Figure 10: SEM image of untreated jute-PP composite.

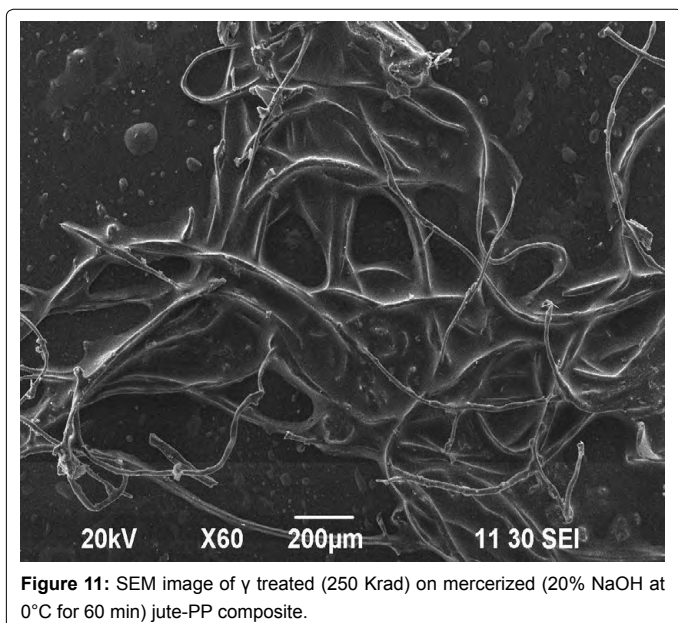


Figure 11: SEM image of γ treated (250 Krad) on mercerized (20% NaOH at 0°C for 60 min) jute-PP composite.

At 0°C temperature the tensile strength, bending strength and Bending E Modulus are the highest for 20% NaOH treated jute composite with a time period of 60 min. It decreases when treated at 25 and 50°C. Again, optimized (20% NaOH treated for 60 min.) mercerized jute fibers were irradiated under γ radiation and mechanical properties were measured. But the mechanical properties of the composites were found lower than that of 20% NaOH treated for 60 min jute PP composites. SEM images supported the idea that the improvement of interfacial fiber matrix adhesion was most likely responsible for those improvements.

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