Surface Treatment of Areca-Nut Fiber Using Silane and Gamma Irradiation: Fabrication of Polycaprolactone Based Composite

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Abstract: Composite materials enjoy great attraction in material science. The objective of this research was to fabricate natural fiber reinforced polymer matrix composites and to increase performances through physical and chemical methods. Areca nut fiber (50 % by weight) reinforced polycaprolactone (PCL) based unidirectional composites were prepared by compression molding. Tensile strength (TS), tensile modulus (TM), bending strength (BS), bending modulus (BM) and impact strength (IS) of the composite were found to be 32 MPa, 685 MPa, 45 MPa, 820 MPa and 15 kJ/m^2, respectively. Six different formulations (F1-F6) of vinyl trimethoxy silane (VTMS) (1-6wt%) along with methanol (97-92%) and photo-initiator Darocur-1173 (2%) were prepared in order to modify the surface of areca nut fiber. Areca nut fiber were soaked for 15 min in different formulations and then exposed under gamma radiation of various doses (250-1000 krad). Composite made of areca nut fiber treated with 4% silane and at 500 krad dose of gamma radiation performed the highest mechanical properties. TS, BS, TM, BM and IS of the composite were found to be 43 MPa, 64 MPa, 1015 MPa, 1423 MPa and 20 kJ/m^2, respectively. Polymer loading (PL) of the gamma treated and untreated fiber was studied. Degradation test of the composites was carried out for 6 weeks in soil medium and it was found that the treated composite retained much of its original strength. It can be concluded that silane and gamma radiation are important tools to improve the mechanical property of the composite.

Keywords: Gamma Radiation, Silane Monomer, Areca-Nut Fiber, Polycaprolactone, Composite.

INTRODUCTION

Compositing is a useful technique for the modification of physico-mechanical properties of polymeric materials [1]. Natural fiber reinforced composite has been enjoying tremendous attraction all over the world due to its biodegradability, bioresorbility, availability, etc [2]. Among all the natural fiber reinforcing materials, areca nut fiber materializes as a promising material because of its cheap cost, easy availability, nontoxicity, biodegradability and eco-friendly manner. The areca nut fiber is predominantly composed of cellulose and varying proportions of hemicellulose, lignin, pectin and protopectin [3]. Polycaprolactone (PCL) is a thermo plastic bioresorbable polymer with a low melting point (59–64^{o}C). PCL is degraded by water via hydrolysis [4]. The main commercial application of PCL is in the manufacture of biodegradable bottles and films, synthetic wound dressings, encapsulants for drug release systems, contraceptive implants, etc. [5-6]. PCL is a potential applicant in the field of tissue engineering for its bioresorbility [7-9]. Various physical and chemical treatments are employed to modify the surface properties of the polymer. Among them, physical treatment, such as ionizing or nonionizing radiation can introduce better surface cross-linking between natural and synthetic polymer, and reduce hydrophilic nature of the polymer [10-12]. Gamma irradiation exerts positive effect on the mechanical performance of polymer through cross-linking [13]. Surface modification of the polymers can be carried out by the monomer treatment. The silane monomer vinyl trimethoxy silane (VTMS) induced cross-linking using their double bonds. Silane coupling agents belong to a class of organosilane compounds having at least two reactive groups of different types bonded to the silicon atom in a molecule [14]. The materials are used as promoters, dispersing agents, cross-linking agents, surface modifiers etc. [15]. It is used in various polymers like polyethers, polyurethanes , polyesters, etc. as cross-linking agent to improve the properties such as tear resistance, elongation at break, abrasion resistance , etc. [16-17]. The ultimate goal of the present study is to modify the surface characteristics of the areca nut fiber through gamma irradiation and monomer treatment followed by the fabrication of PCL matrix based areca nut fiber reinforced composite.

EXPERIMENTS

Preparation of PCL Sheet

The PCL matrix sheets were prepared by compression molding. The granulated PCL was

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purchased from Sigma-Aldrich, UK. 15 gm PCL granules were placed in between two steel plates. The steel panel was then placed in a heat press (Carvar, USA) and operated at 100 ^{o}C for 5 min to melt the polymer. A 5 bar consolidation pressure was applied for 1 min. The mold was then removed from heat press and was cooled for 1 min in a separate press under 5 bar pressure at room temperature. The prepared sheet was then cut into desired size (100 mm x 60 mm x 0.4 mm) for composite fabrication and stored at polyethylene bag.

Treatment of Fiber with VTMS Monomer and Gamma Irradiation

Area nut fiber (collected from local area of Bangladesh) was soaked into six formulations (F1-F6) of VTMS (purchased from Fluka, Switzerland) varying its percentages from 1-6 wt% along with methanol (97-92%) and photo-initiator Darocur-1173 (2%) for 15 min. Table **1** outlines the composition of the six formulations. The monomer treated fiber was then irradiated under gamma radiation (Co-60) from 250 to 1000 krad doses at a dose rate of 300 krad /hour.

Table 1: Composition (%w/w) of the Prepared Six Monomer Formulations

Formulation	VTMS (%)	Methanol (%)	Photo-initiator Darocur-1173 (%)
F1	1	97	2
F2	2	96	2
F3	3	95	2
F4	4	94	2
F5	5	93	2
F6	6	92	2

Fabrication of Composites

Composite laminates were prepared by sandwiching four layers of areca nut fibers (silane and gamma irradiation treated or untreated fibers) in between five sheets of PCL. Fibers were placed unidirectionally between the sheets of PCL and the resulting sandwich construction was fixed tightly using adhesive tape in order to curb the movement of fibers during hot pressing. This sandwich panel was then placed in between two steel plates and heated at 110 ^{o}C for 5 min to soften the polymer prior to pressing at 100 ^{o}C and 3 bar pressure for 1 min. The steel mold was then cooled to room temperature in a separate cold press. The fiber volume fraction of the composites was calculated to be 50%. The prepared composites were sealed in a polyethylene bag prior to various tests.

Mechanical Properties of the Composites

Composites were cut into desired dimensions for determining the TS, TM, BS, and BM. Tensile and bending properties of the composites were evaluated using the Hounsfield series S testing machine (UK) with a crosshead speed of 1mm/min at a span distance of 25 mm. The load capacity is 500n; efficiency is within ± 1%. The dimensions of the test specimen were (ISO 14125): 60mm×15mm×2mm. IS (Charpy) of the composites was measured using Impact tester (MT-3016, Pendulum type, Germany). All the results were taken as the average value of at least five samples.

Polymer Loading (%) of the Areca Nut Fibers

Polymer loading (PL) was calculated based on mass gained after soaking in the monomer formulations. The initial mass of the areca nut fibers was measured. Then the fibers were soaked in F1–F6 formulations for 15 min and then were cured under gamma irradiation of various intensities (250-1000 krad). The final mass of the cured fibers was measured again. The PL (mass gain) of the fibers of six formulations was determined on the basis of mass gained by the fibers. The PL was measured by the following equation: PL (%) = $(W_t - W_o)/W_o \times 100$, where W_o is the mass of the untreated dry fiber and W_t is the mass of the cured dry fiber.

Soil Degradation Tests of the Composites

Composite samples were buried in soil (having at least 25% moisture) for different periods of time (up to 6 weeks) to find out the degradation nature of the composites. The buried samples were withdrawn carefully after a fixed period of time. Then it were washed with distilled water to remove the adjunct dusts and dried at 105 ^{o}C for 6 hr. The dried samples were kept at room temperature for 24 hr and then measured their mechanical properties.

RESULTS AND DESCRIPTIONS

Mechanical Properties of the Composites

Mechanical properties of the composites are important factors to decide its spectrum of application in several fields. Mechanical properties play a vital role to select a composite for both commodity and engineering purposes. Areca nut fiber reinforced PCL

Table 2: Mechanical Properties of the Areca Nut Fiber Reinforced PCL Based Unidirectional Composite (50 wt% fiber)

Mechanical Properties of the Composite Material				
	Tensile Strength (TS)	**Tensile Modulus (TM)**	**Bending Strength (BS)**	**Bending Modulus (BM)**
PCL Sheet	15 MPa	424 MPa	20 MPa	590 MPa
Composite	32 MPa	685 MPa	45 MPa	820 MPa

based unidirectional composite was prepared by compression molding. The fiber content of the composite was 50% by weight. Mechanical properties of the composites such as TS, BS, TM and BM were measured and illustrated in the Table **2**. From the Table **2**, it is clear that fiber reinforcement was occurred and resulted in improved mechanical properties of the composites. TS, BS, TM and BM of the PCL sheet were found to be 15 MPa, 20 MPa, 424 MPa and 590 MPa, respectively. Composite gained a 113% increased TS and a 125% increased BS over that of matrix PCL sheet. TS, BS, TM and BM of the composite were found to be 32 MPa, 45 MPa, 685 MPa and 820 MPa, respectively. It was found that TM and BM also improved significantly due to incorporation of areca nut fiber into the matrix PCL sheet. TM and BM improved 62 % and 39%, respectively, than that of the PCL matrix. Impact strength of the composites was tabulated in the Table **3** and the result was found satisfactory. From the present study, it is obvious that mechanical properties of the composites were superior to those of the matrix material. The increased in mechanical properties is due to the reinforcement of areca nut fiber with the matrix PCL. Areca nut fiber has successfully reinforced the mechanical properties of PCL.

Table 3: Impact Strength (IS) of the Areca Nut Fiber Reinforced PCL Based Unidirectional Composite (50 wt% fiber)

Impact Strength of the Composite Material	
PCL/ Areca Nut Fiber Composite	Impact Strength (IS)
	15 kJ/m^2

Polymer Loading

The PL values of the fiber were evaluated as a mass gain after soaking in the monomer solution. The fiber was soaked in six formulations (F1-F6) for 15 min and then dried in an oven at 105 ^oC for 2 hrs. The mass gained in respect to initial mass was measured after soaking in the monomer formulations. PL (%) values of the fiber in six formulations for 15 min soaking time was presented in Figure **1**. From the Figure **1**, it is clear that PL values were increased gradually with the increasing of VTMS concentration (%) up to a certain limit and then it reached maximum value of 8.2% for F4 formulation and then it decreased to 6.2% for F6 formulation. For F1, F2, F3 and F5 formulations the PL values were found to be 3.2%, 4.5%, 6.0% and 7.1%, respectively.

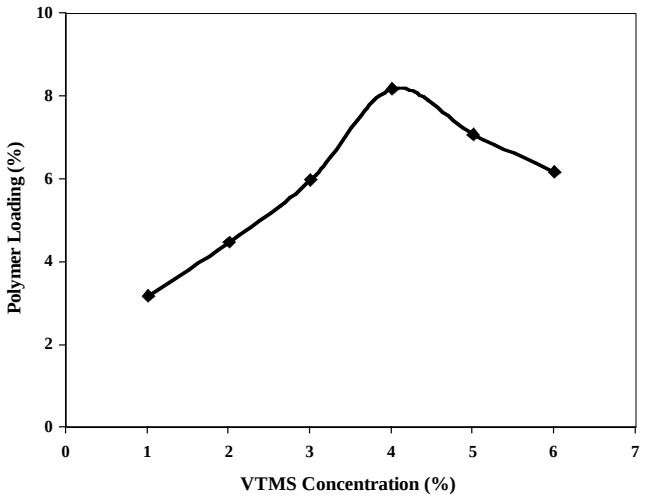

Figure 1: Polymer loading (%) of the fiber in response to six formulations at 15 min soaking time.

The mass gain is due to the incorporation VTMS monomer in to –OH groups of areca nut fiber due to the formation of hydrogen bonding. The degree of polymerization reaction increases with the increase of monomer concentration up to a certain limit. The highest PL value was obtained for 4% VTMS concentration in solution. At low VTMS concentration (1%, 2% and 3%), the percentage PL value is less because VTMS monomer promotes rapid free radicals propagation reaction with the help of photo initiator leading to network polymer structure through graft copolymerization reaction. As VTMS concentration increased, the amount of residual concentration is also increased with consequence of faster rate of formation of 3D network structure causing restricted mobility [18].

At higher VTMS concentration, the radical–radical recombination reaction among growing VTMS

molecules may lead to poly (VTMS) which may be dominant and hinder more silane monomers to be reacted with hydroxyl groups of areca nut fiber [18, 19].

The PL values of the fiber were evaluated against gamma irradiation dose. The fibers were soaked in the six formulations for 15 min. Soaked fiber was then cured under gamma irradiation dose at different intensities (250-1000 krad). Figure **2** depicts the polymer loading of the fiber in response to six formulations at 15 min soaking time against gamma irradiation dose. It is obvious that, PL values were increased with the increasing intensity of gamma dose up to a certain limit and attained a maximum and then they were decreased with higher intensity doses. The maximum value was found at 500 krad dose; but after that the PL values were decreased for all formulations. As an instance, 17.5 % PL value was obtained for F4 formulation (4% silane) at 500 krad. But PL was reduced to 14.6% at 1000 krad for the same formulation. All formulations showed the same patterns against gamma irradiation treatment. The PL values were found to be 6.6%, 8.0%, 10.7%, 10.2% and 9.7%, respectively, for the formulations of F1, F2, F3, F5 and F6, respectively, at 250 krad dose.

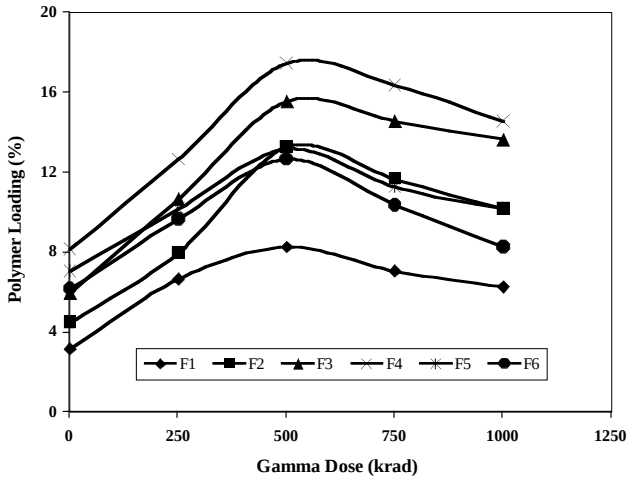

Figure 2: Polymer loading (%) of the fiber in response to six formulations at 15 min soaking time against gamma irradiation.

The maximum PL values for F1, F2, F3, F4, F5 and F6 formulations were found to be 8.3%, 13.3%, 15.6%, 17.5%, 13.2% and 12.7%, respectively, at 500 krad dose. PL values improved 123%, 195%, 160%, 113%, 86% and 104%, respectively, for F1, F2, F3, F4, F5 and F6 formulations due to 500 krad irradiation dose employments compared to untreated one. The increasing trend in polymer loading with irradiation dose is due to getting sufficient energy for initiation and propagation of polymerization of monomers as well as cross-linking and grafting of monomers with the cellulose backbone of areca nut fiber [20, 21]. On the contrary, the decrease in polymer loading at higher doses could be regarded as the result of photo-degradation of polymers by the higher energy of radiation [22, 23].

Optimization of Formulation and Radiation Dose

Areca nut fiber was soaked in the prepared six formulations for 15 min and cured under gamma irradiation of different doses (250-1000 krad). The unidirectional composites were fabricated using treated fiber and PCL by compression molding. The mechanical properties of the composite were evaluated for six formulations against gamma irradiation dose. The graphical representations are provided in Figures **3**, 4, 5, 6 and 7. TS and BS of the composite for six formulations against gamma irradiation are plotted in Figures **3** and **4**. The highest TS and BS values were found for F4 formulation at 500 krad dose. TS and BS values were increased at low intensity of gamma irradiation dose and attained maximum values at 500 krad dose and then decreased at higher doses for all formulations.

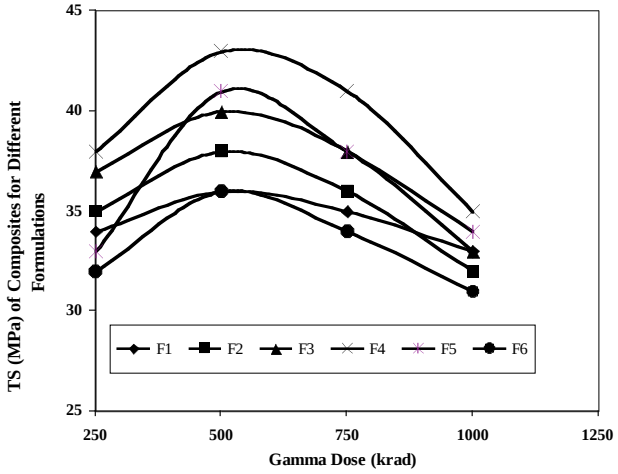

Figure 3: TS of the monomer treated composite as a function of gamma irradiation.

From the Figure **3**, the TS values were found to be 34 MPa, 35 MPa, 37 MPa, 38 MPa, 36 MPa and 32 MPa, respectively, at 250 krad, whereas, 36 MPa, 38 MPa, 40MPa, 43 MPa, 41 MPa and 36 MPa, respectively, at 500 krad for F1, F2, F3, F4, F5 and F6 formulations, respectively. TS values improved 5.9%, 8.6%, 8.1%, 13.2%, 13.9% and 13.5%, respectively, for F1, F2, F3, F4, F5 and F6 formulations at 500 krad doses with respect to 250 krad doses. The highest BS

value was found for F4 formulation at 500 krad dose as shown in Figure **4**. BS values of F1, F2, F3, F4, F5 and F6 formulations were found to be 53 MPa, 58 MPa, 60MPa, 65 MPa, 60 MPa and 57 MPa, respectively, at 500 krad dose. From the Figure **4**, it can be clearly stated that, the increasing and decreasing trends for BS values with respect to gamma irradiation dose intensity are similar to those of TS values patterns. BS values were increased al lower intensity and reached highest level at 500 krad dose and then were decreased with higher intensity compared to 500 krad dose. Bending strength enjoyed 8.2% increased BS for F1, 16% increased BS for F2, 17.6% increased BS for F3, 16.1% increased BS for F4, 15.4% increased BS for F5 and 16.3% increased BS for F6 formulations at 500 krad dose compared to 250 krad dose.

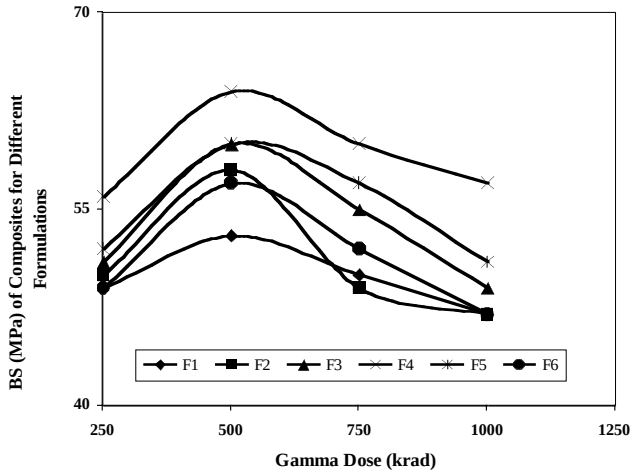

Figure 4: BS of the monomer treated composite as a function of gamma irradiation.

TM and BM of the composite for six formulations as a function of gamma irradiation dose are shown in Figures **5** and **6**. TM and BM values were found maximum at 500 krad for all formulations and they were decreased at higher doses than 500 krad. From the Figure **5**, it can be observed that TM values were found to be 811 MPa, 834 MPa, 951 MPa, 1015 MPa, 820 MPa and 801 MPa, respectively, for F1, F2, F3, F4, F5 and F6 formulations at dose intensity of 500 krad. At 500 krad dose, TM values gained 13.7%, 11.9%, 18.7%, 10.7%, 6.2% and 11.2% of increased TM over those of TM values at 250 krad dose. BM values, as shown in Figure **6**, were found to be 1253 MPa, 1302 MPa, 1356 MPa, 1423 MPa, 1330 MPa and 1310 MPa, respectively, for F1, F2, F3, F4, F5 and F6 formulations at 500 krad dose. At 500 krad dose, BM improved 22.5%, 16.3%, 16.2%, 15.3%, 15% and 14.4%, respectively, for F1, F2, F3, F4, F5 and F6 formulations over those of 250 krad dose. Both TM and BM values were initially increased at lower dose and reached highest value at 500 krad and then were declined at higher dose. The increasing pattern of mechanical properties at lower dose gamma irradiation is due to the grafting and cross-ling of hydroxyl group of areca nut fiber, PCL and silane monomer. At higher doses, photo-degradation of polymer dominant over cross-linking which ultimately plays role to lower the mechanical properties of the composite [22, 23].

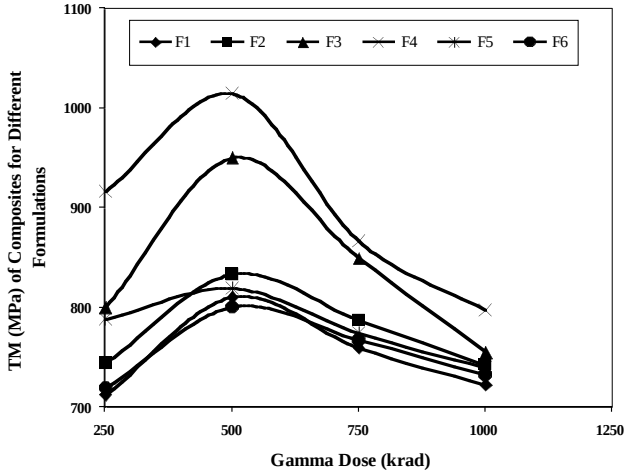

Figure 5: TM of the monomer treated composite as a function of gamma irradiation.

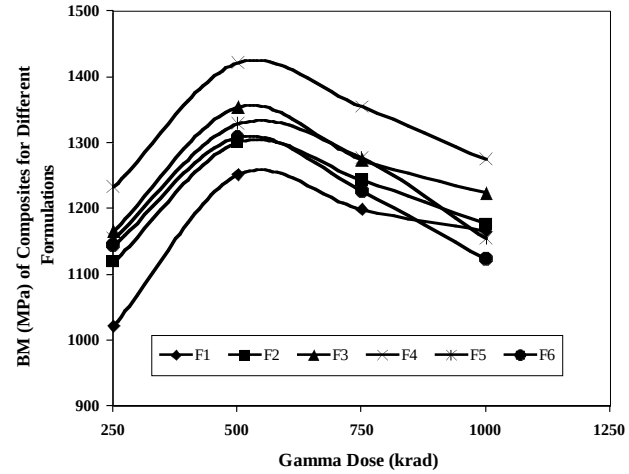

Figure 6: BM of the monomer treated composite as a function of gamma irradiation.

Figure **7** describes the IS values of the composite against gamma irradiation dose. Monomer treatment and gamma irradiation dose slightly increased IS values of the composite. The IS values were found to be 17 kJ/m^2, 18 kJ/m^2, 19 kJ/m^2, 20 kJ/m^2, 18kJ/m^2 and 17 kJ/m^2, respectively, for F1, F2, F3, F4, F5, and F6 formulation at 500 krad dose. Silane applied to the fiber was intended to act as a coupling agent to

improve the fiber matrix adhesion. Moreover, gamma irradiation favored cross-linking between hydroxyl group of fiber and silane which ultimately increased the IS values. Thus the formulation F4 at 500 krad dose was considered as the optimum condition.

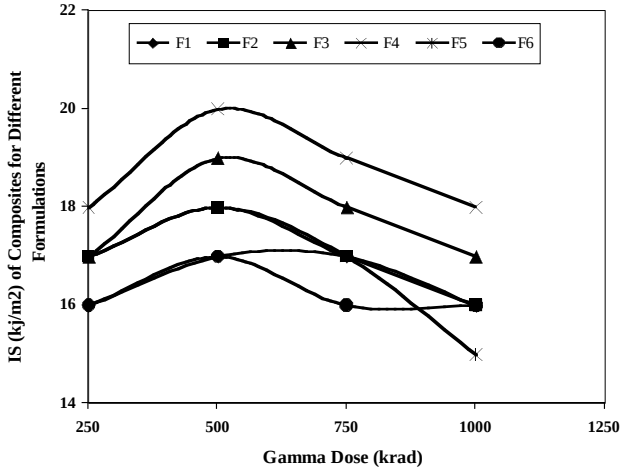

Figure 7: IS of the monomer treated composite as a function of gamma irradiation.

Degradation Properties of the Composites

Degradation nature of the composites is required to find the suitability of its utilization. Degradation of the compression molded composite was performed for up to 6 weeks in soil medium containing at least 25 % moisture. After drying, the composites were subjected to mechanical test. TS and TM of the untreated and treated composite (optimum) are plotted against degradation time and are shown in Figures **8** and **9**. It is clear that both strength and modulus were decreased slowly with time. From the Figure **8**, TS values of the untreated composites were found 25 MPa, 17 MPa and 11 MPa, respectively, for degradation time of 2 weeks, 4 weeks and 6 weeks. Treated composite observed TS values of 35 MPa, 24 MPa and 19 MPa, respectively, for 2 weeks, 4 weeks and 6 weeks degradation period. Figure **9** showed TM values of 560 MPa, 478 MPa and 411 MPa, respectively, after a degradation time of 2 weeks, 4 weeks and 6 weeks for the untreated composites whereas this values were 820 MPa, 633 MPa and 507 MPa for the treated composites. Untreated composite retained 33% and 40%, respectively, its initial TS and TM after 6 weeks of degradation. But treated composite retained much higher TS (45% of original strength) and TM (50% of original modulus) compared to untreated composite after 6 weeks degradation time. The decreased in strength and modulus is due to bio-degradable nature of areca nut fiber and PCL [3, 4]. Lower strength and modulus lost for the treated composite compared to untreated one was due to the grafting and cross-linking of cellulose of areca nut fiber with silane monomer which reduced hydrophilic nature of fiber.

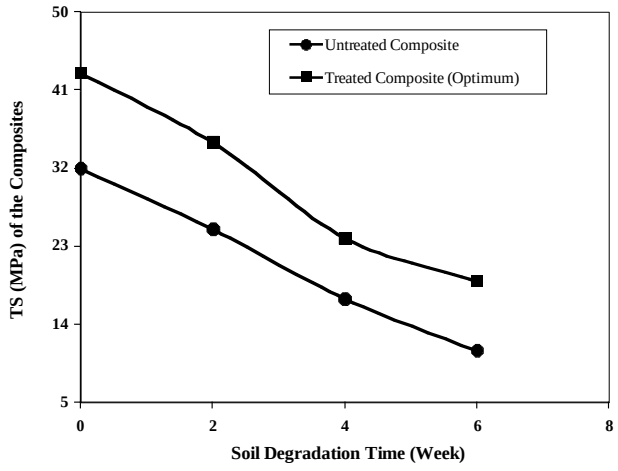

Figure 8: TS of the untreated and treated composite (optimum) as a function of soil degradation time.

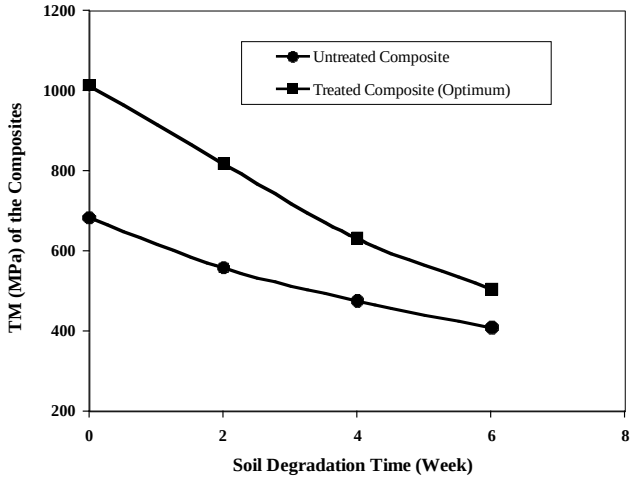

Figure 9: TM of the untreated and treated composite (optimum) as a function of soil degradation time.

CONCLUSION

Areca nut fiber reinforced PCL based unidirectional composites were prepared by compression molding. TS, TM, BS, BM and IS of the composite were found to be 32 MPa, 685 MPa, 45 MPa, 820 MPa and 15 kJ/m^2, respectively. The areca nut fiber was treated with VTMS and gamma irradiation dose to modify the polymeric character of the fiber. Compression molded composite was prepared with this surface modified fiber and PCL matrix. Composite made of areca nut fiber treated with 4% silane and at 500 krad dose of gamma irradiation performed the highest mechanical

properties. It can be concluded that VTMS and gamma irradiation have a positive impact on the performance of the composite.

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