

Role of Pretreatment with Potassium Permanganate and Urea on Mechanical and Degradable Properties of Photocured Coir (*Cocos nucifera*) Fiber with 1,6-Hexanediol Diacrylate

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ABSTRACT: Coir fibers were modified with 1,6-hexanediol diacrylate (HDDA) using ultraviolet (UV) radiation. Concentration of HDDA, soaking time, and radiation dose were optimized and found to be 30% HDDA in methanol along with photoinitiator Irgacure-500 (2%) and 120 min of soaking time registered as the better performance (polymer loading (PL) 7%, tensile strength factor, $T_f = 1.50$). Urea of different concentrations (0.5–2%) were incorporated with 30% HDDA to monitor its effect on the properties and 1% urea produced the best results (PL = 25%, $T_f = 1.82$). For the improvement of the properties, the fibers were subjected to surface treatment with potassium permanganate (KMnO_4)

of different concentrations at various treating times. Enhanced properties (PL = 86%, $T_f = 4.42$) of the fibers treated with KMnO_4 (0.05%) were obtained. The KMnO_4 treated fibers were again treated with HDDA (30%) solution along with urea (1%) and found to produce the best results (PL = 100%, $T_f = 4.5$). Water uptake and degradable properties of the treated and virgin fibers were obtained. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 100: 4361–4368, 2006

Key words: coir fiber; natural fiber; photocuring; UV radiation; biodegradable

INTRODUCTION

With the advent of synthetic fibers, the use of natural fibers like coir, jute, and sisal have been diminished to such an extent that many industries dealing with natural fiber products had to be closed. But natural fibers have many advantages compared with man-made fibers, for example, low weight, recyclability, and biodegradability. They are renewable raw materials and have relatively high strength and stiffness and cause no skin irritation.¹ But there are some disadvantages, such as moisture uptake, quality variations, and low thermal stability. Coir is a natural fiber, which is obtained from the husk of the fruit of the coconut palm. *Cocos nucifera* L. Cellulose is the main constituent of this fiber (43%),² which is a hydrophilic glucan polymer consisting of linear chain of 1,4- β bonded anhydroglucose unit, and also contains alcoholic hydroxyl groups.³ These hydroxyl groups form intermolecular hydrogen bonds inside the macromolecule itself and in other cellulose macromolecules. Therefore, coir fi-

ber is hydrophilic in nature and this is the most important disadvantage of this fiber. Chemical modification may inactivate these groups. Previously pineapple leaf fiber-reinforced polyethylene composites were treated with potassium permanganate (KMnO_4) (as a strong oxidizing agent) and improved properties were achieved.⁴ Several processes like chemical treatments,^{5–8} photochemical treatments,^{9–14} plasma treatment,¹⁵ and so forth have been developed to modify polymers and fiber surfaces. To improve the physico-mechanical properties, a pretreatment of the fiber surface or the incorporation of surface modifier during grafting process is required. Several studies have been reported to improve physico-mechanical properties of natural fibers, using different impregnating solution under gamma radiation^{9,10} and UV radiation.^{11–14,16} In the same way, coir fibers were also modified and enhanced properties were achieved.¹⁷ It was previously observed that both polymer loading (PL) and tensile strength of wood plastic composite were enhanced using additives containing a carboamide group.^{18–20} Urea is very cheap and contains this group, and so it was selected as an additive. The present study deals with the modification of the mechanical and degradable properties of coir fibers as

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TABLE I
Optimization of Monomer Concentration (Soaking Time 120 min)

No. of UV passes	Formulations											
	5%			10%			30%			50%		
	PL	T_f	E_f	PL	T_f	E_f	PL	T_f	E_f	PL	T_f	E_f
2	4.47	0.40	2.11	7.30	0.66	0.59	8.65	0.44	0.41	14.1	0.42	0.66
4	13.1	0.65	1.15	9.17	0.83	1.02	17.0	0.99	0.67	15.1	0.69	0.88
6	13.1	0.90	1.06	13.2	0.91	1.32	13.7	1.50	1.01	14.2	0.66	0.68
8	10.2	1.02	0.76	14.2	0.89	1.39	6.67	0.87	0.50	13.2	0.56	0.67
10	9.17	0.40	0.32	11.3	0.54	0.38	3.10	0.59	0.46	12.1	0.19	0.61

well as to observe the effect of surface treatment and additive.

EXPERIMENTAL

Materials

Coir fiber (brown) was collected from local market of Bangladesh. The difunctional monomer 1,6-hexanediol diacrylate (HDDA) and swelling agent methanol were procured from Merck (Germany). Irgacure-500 of Ciba-Geigy (Switzerland) was used as a photoinitiator. Additive urea and KMnO_4 were procured from British Drug House Lid (8011).

Method

Coir fibers were washed with acetone, dried in the oven at 105°C for 1 h to remove the moisture, and cooled in the desiccators. Four different formulations were prepared using monomer HDDA of different proportions (5, 10, 30, and 50%) and photoinitiator (2%) in methanol (swelling agent). The dried fibers were immersed in these formulations for different soaking times (5, 10, 30, 60, 120, and 180 min) and were then cured under UV lamp (2 kW power, IST, Technik, Germany) at different radiation intensities (2, 4, 6, 8, and 10). After 24 h, the fibers were washed with acetone to remove the unreacted HDDA. The weight gain of the fibers was calculated as PL, and the mechanical properties (tensile strength and elongation at break) were calculated by the Universal Testing Machine (INSTRON, model 1011, UK). Urea of different concentrations (0.5–2%) was added to the optimized solution. Again the fibers were pretreated with KMnO_4 of different concentrations (0.01–1%) at different treating times (1, 5, and 10 min) before they were treated with the optimized formulation. Afterwards they were again treated with the same formulation incorporated with urea of optimized concentration.

Water uptake

Water uptake values of treated and untreated fibers were calculated by dipping the fibers in a static bath

containing water at 25°C . The fibers were taken out of water after a constant time interval, wiped very well, and weighed.

Simulated weathering test

The loss of PL and the tensile properties by the impact of simulated weathering performed at alternating cycles of sunshine, dews, and condensation were determined with the help of an Accelerated Weathering Tester (model Q.U.V., Q-Panel Co.) The temperature during the treatment varied between $(65 \pm 2)^\circ\text{C}$ (sunlight) and $(45 \pm 2)^\circ\text{C}$ (condensation) through alternating cycles of 4 h sunlight and 2 h condensation for a period of about of 120 h. The loss of properties due to the weathering treatment was determined.

Soil and water degradation

The treated and virgin coir fibers were kept in soil (20% moisture) and water for a period of 2 months (60 days). The change of tensile properties caused by these treatments was periodically noted to determine the degradable character of the samples in these environments.

RESULTS AND DISCUSSIONS

Polymer loading

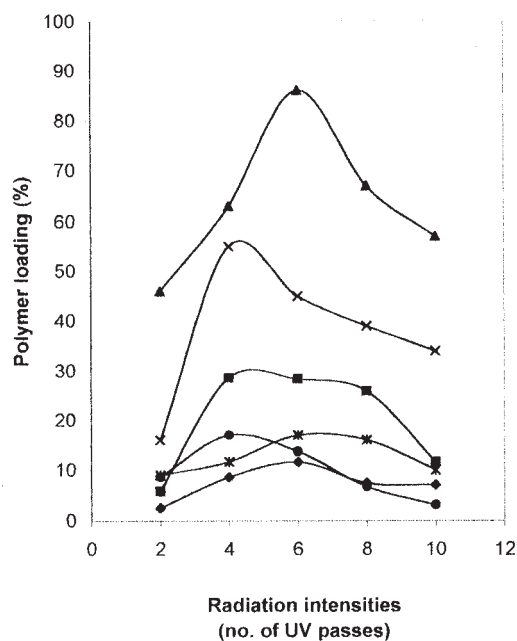
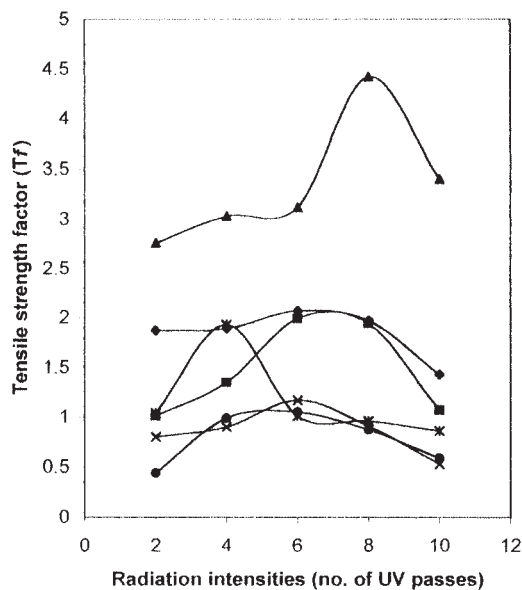
PL values of the fibers were calculated as the percentage weight gain, after they were cured under UV radiation. The PL values of the HDDA-treated fibers at different radiation intensities, as a function of monomer concentration are shown in the Table I. The values are low at low monomer concentration, increase with HDDA concentration, and reach the maximum with 30% HDDA (17%) at 6th pass of 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 copolymerization reaction via their double bond.²¹ As the HDDA concentration increases, the amount of residual concentration is also increased with consequence of faster rate of formation of three-

TABLE II
Optimization of Soaking Time

No. of passes	Soaking time (min)																	
	5			10			30			60			120			180		
	PL	T_f	E_f	PL	T_f	E_f	PL	T_f	E_f	PL	T_f	E_f	PL	T_f	E_f	PL	T_f	E_f
2	1.6	0.54	1.51	6.9	0.77	0.82	7.0	0.49	0.33	7.61	0.65	0.64	8.65	0.44	0.41	12.12	0.51	0.87
4	7.3	0.66	0.89	11.2	0.79	0.71	16.14	0.71	1.14	7.9	0.91	0.89	17	0.99	0.67	16.38	0.66	1.19
6	6.1	0.91	0.72	8.8	0.97	0.41	11.12	0.88	0.67	8.5	0.88	1.15	13.7	1.5	1.01	9.7	0.71	1.10
8	6.0	0.89	0.61	6.8	0.80	0.41	10.39	0.86	0.65	13.1	0.76	0.75	6.67	0.87	0.50	8.82	0.39	0.89
10	5.2	0.83	0.41	5.3	0.53	0.30	7.5	0.54	0.50	8.1	0.56	0.43	3.1	0.59	0.46	7.9	0.26	0.75

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 HDDA concentration, radical-radical recombination process may be dominating creating more homo polymer rather than monomer + cellulose backbone reaction. The second 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 HDDA concentration. The crosslinking rate, especially during the early stages of radiation, is pro-

portional to HDDA concentration. The concentration of HDDA increases the radical-radical reaction termination and hence decreases the extent of scission reaction and oxidation.²² The decrease in PL value at higher monomer concentration could be associated with the fact that the homopolymerization reaction between monomer and monomer-radicals is dominant than that of the monomer and coir cellulose reaction. Again, the values of PL are presented in the Table II as a function of soaking time. At low soaking time, PL value is quite low; it increases with the increase in time with the same monomer concentration (30% HDDA). The highest value is obtained with 120 min of soaking. After that, it decreases with time. The diffusion of monomer into the fiber, the swelling of trunk polymer, and the Trommsdorff effect of solvent on graft polymer radicals affect the radiation induced graft copolymerization reaction of vinyl monomers


Figure 1 PL of treated coir fiber against radiation intensities as a function of KMnO_4 concentration, treating time 1 min.

Figure 2 Tensile strength factor (T_f) of treated coir fiber against radiation intensities as a function of KMnO_4 concentration, treating time 1 min.

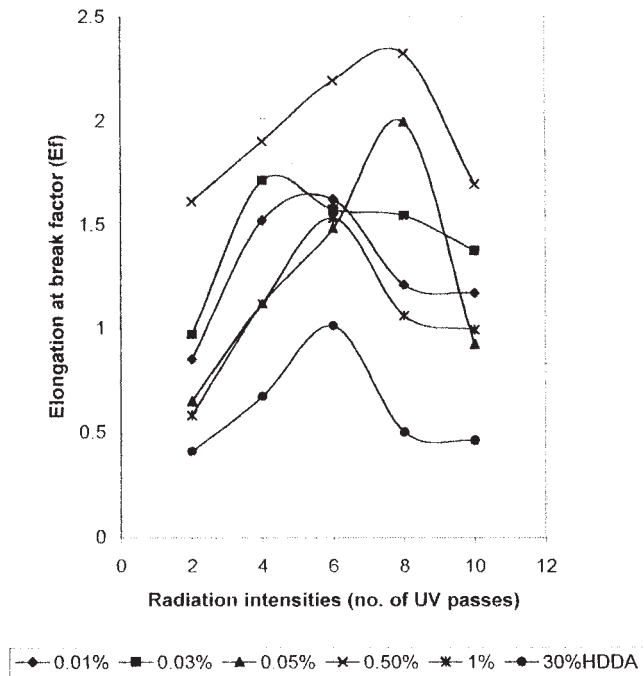


Figure 3 Elongation at break factor (E_f) of treated coir fiber against radiation intensities as a function of KMnO_4 concentration, treating time 1 min.

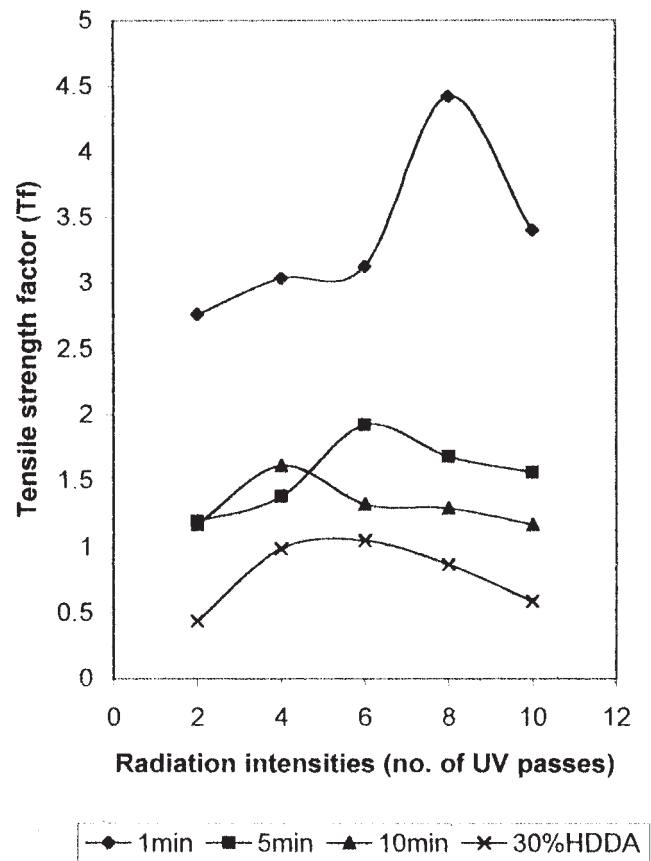


Figure 5 Tensile strength factor (T_f) of treated coir fiber against radiation intensities as a function of treating time in KMnO_4 solution (0.05%).

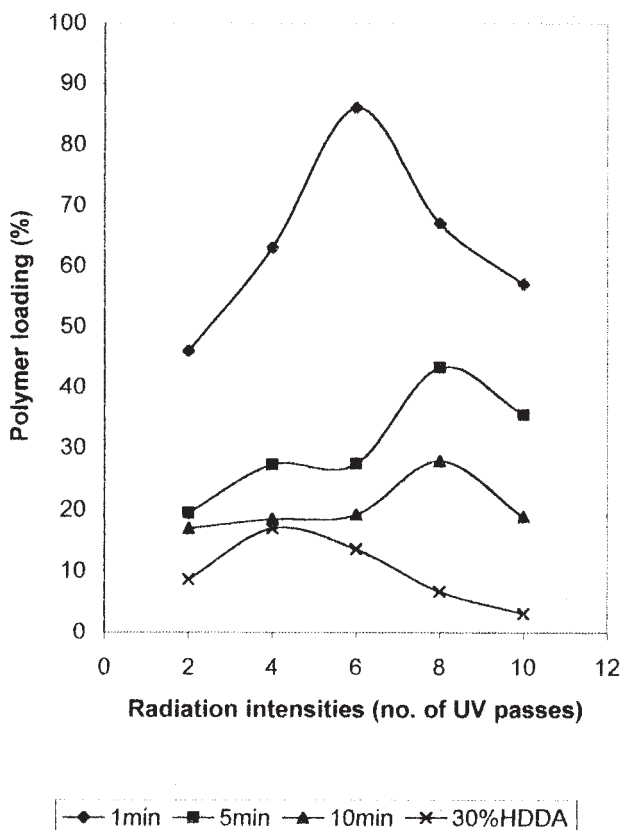


Figure 4 PL of treated coir fiber against radiation intensities as a function of treating time in KMnO_4 solution (0.05%).

onto cellulose backbone.^{23,24} Swelling increases the cross sectional area of the fiber and the fiber surface becomes lustrous. As a result, monomer can easily diffuse into the fiber and react with cellulose in low swelling time. The decrease in PL values with radiation dose after the maximum was achieved could be caused by the radiation degradation of higher UV doses.²⁵

Tensile properties

Tensile properties of the treated fibers were expressed as tensile strength factor and elongation at break factor. The tensile strength factor T_f was calculated as T_1/T_2 , where T_1 and T_2 are the tensile strength of the treated and untreated fibers, respectively. The values of T_f are shown in the Tables I and II. The values are low with lower monomer concentration and soaking time. Thirty percent of HDDA at 120 min of soaking time produced the highest T_f (1.50). Increase of T_f with HDDA concentration may be due to the higher rate of polymerization and the decrease is due to radical-radical recombination reaction among the growing HDDA molecules. The T_f increases with soaking time

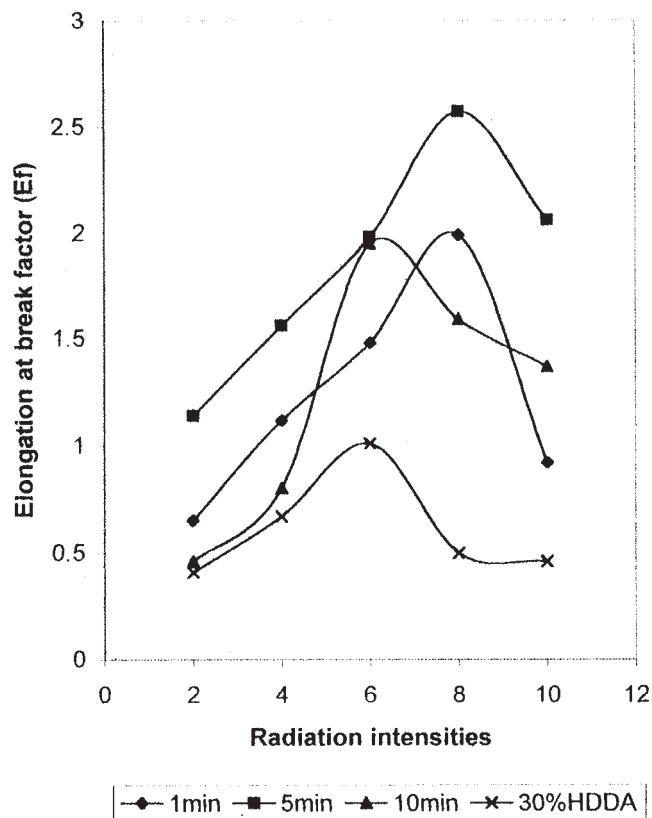
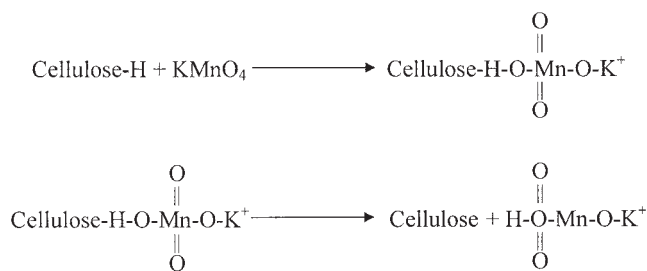


Figure 6 Elongation at break factor (E_f) of treated coir fiber against radiation intensities as a function of treating time in KMnO₄ solution (0.05%).

up to 120 min, and then decreases again. The increase is due to increased swelling leading to increased diffusion of monomer into the sites of reaction and a greater amount of grafting. T_f is low at lower doses of radiation, is highest at 6th pass, and then it decreases again because of some degradation of fibers with higher doses. Elongation at the break point was expressed as elongation at break factor E_f , where $E_f = E_1/E_2$ (E_1 , elongation at break of treated fiber and E_2 , elongation of untreated fiber). The values of E_f are tabulated in Tables I and II. The



Scheme 1

highest value (2.11) was achieved with 5% HDDA at 120 min of soaking.

Effect of surface treatment with KMnO₄

Coir fibers were subjected to surface treatment with KMnO₄. The fibers were treated for different times (1, 5, and 10 min) with KMnO₄ of varying concentrations (0.01, 0.03, 0.05, 0.5, and 1%). The loss of treated fibers was about 4%. Then, they were cured with 30% HDDA. The results of PL, T_f , and E_f with respect to KMnO₄ concentration are presented in Figures 1–3, respectively. Again those results with respect to treating times in KMnO₄ solution are shown in Figures 4–6, respectively. Among the different concentrations, 0.05% KMnO₄ at 1 min of treating time produced the best results (PL = 86% and $T_f = 4.42$). Both highest PL and T_f are achieved at 6th pass of UV radiation. Because of the presence of KMnO₄ as a powerful oxidizing agent, the cellulose molecules of coir fibers undergo reactions, which can be shown as Scheme 1.²⁶

This cellulose free radical is quite stable, which supports the higher rate of reaction with monomer molecules. Thus, permanganate induces grafting of HDDA on to cellulose molecules of coir fibers. 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 with 0.05% KMnO₄. On further increasing the concentration of the latter, properties decrease very much because of the degradation of fiber.

TABLE III
Effect of Urea on PL, T_f , and E_f of the Heated Coir Fiber

No. of UV passes	Concentration of urea (%)								
	0.5%			1%			2%		
	PL	T_f	E_f	PL	T_f	E_f	PL	T_f	E_f
2	9.48	0.57	0.57	13.9	1.55	1.12	7.0	0.56	0.55
4	19.8	0.66	0.95	16.6	1.82	1.25	13.4	0.63	0.73
6	16.9	0.88	0.97	20.0	1.73	1.69	19.4	0.77	0.92
8	14.1	0.58	0.77	25.2	1.35	1.04	13.7	0.64	1.99
10	8.67	0.32	0.36	17.6	1.20	0.94	9.01	0.56	0.78

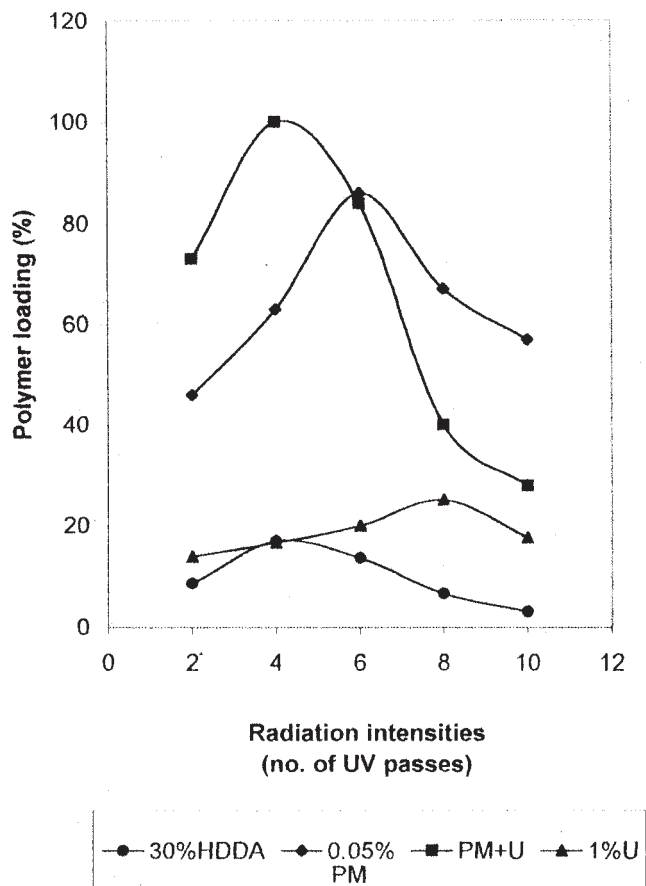


Figure 7 Effect of urea on the PL of KMnO_4 pretreated coir fibers.

Effect of additive (urea) on the properties of coir fibers

An additive urea of different concentrations (0.5–2%) was added to the optimized solution during the treatment of the fibers with that formulation. Enhanced properties (PL = 25%, T_f = 1.82) were achieved with 1% Urea. The results of PL, T_f and E_f are presented in the Table III. Urea possesses $\text{C}=\text{O}$ groups adjacent to a nitrogen atom having lone pair of electrons, which are activated to form a bridge between the monomer and the cellulose through the additive. Oxygen has more affinity toward electrons; thus, the electron clouds are densely populated around the oxygen atom of $\text{C}=\text{O}$ groups, thereby pulling more electrons toward the oxygen from the 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.¹⁸ Urea is an inclusion compound whose properties would assist partitioning by complexing with the monomer. This can lead to an increase in the monomer concentration at a grafting site and thus enhances reactivity at that site.²⁰ Again urea was also incorporated with 30% HDDA solution when the fi-

bers treated with 0.05% KMnO_4 were treated with that formulation. Then, more enhancements of the properties were observed, which are shown in the Figures 7–9 presenting PL, T_f and E_f , respectively. The PL value reached to 100% and the T_f was 4.5.

Water uptake

Water uptake values of the virgin and treated fibers were calculated by immersing the fibers in water contained in a static bath at the room temperature. The fibers were taken out of water after constant time interval and their weight gain was calculated. The results of water uptake values of the treated and untreated fibers are shown in the Figure 10. All the treated fibers took up water within the first 10 min of soaking, and then the values were constant. But the untreated fibers continued to take up water throughout the period of monitoring. The minimum amount of water was taken up by the KMnO_4 + urea system (5.3) and the highest by virgin fibers (94.5). It should be noted that KMnO_4 + urea system also possesses the highest PL (100%). The hydroxyl groups of the cellulose molecules were filled up by the monomer molecules. So, the water uptake value of that system was lowest.^{27,28}

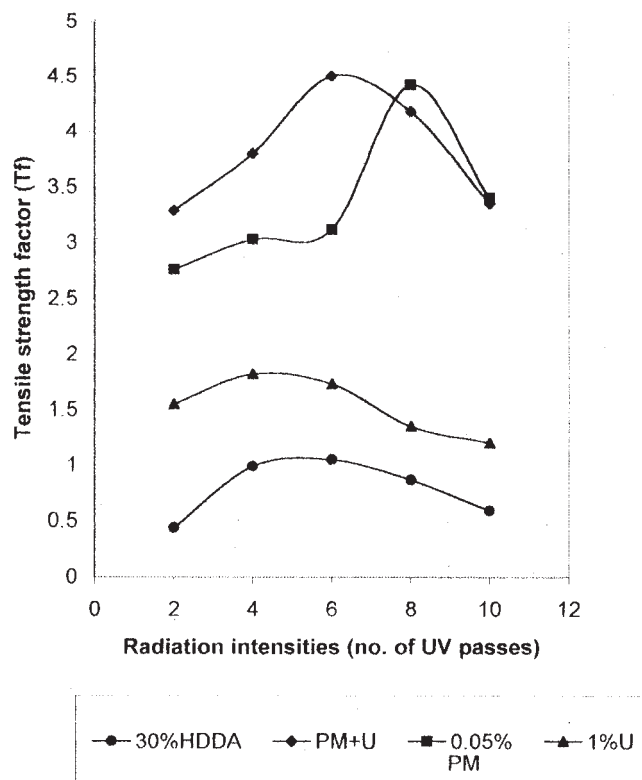


Figure 8 Effect of urea on the tensile strength factor of KMnO_4 pretreated coir fibers.

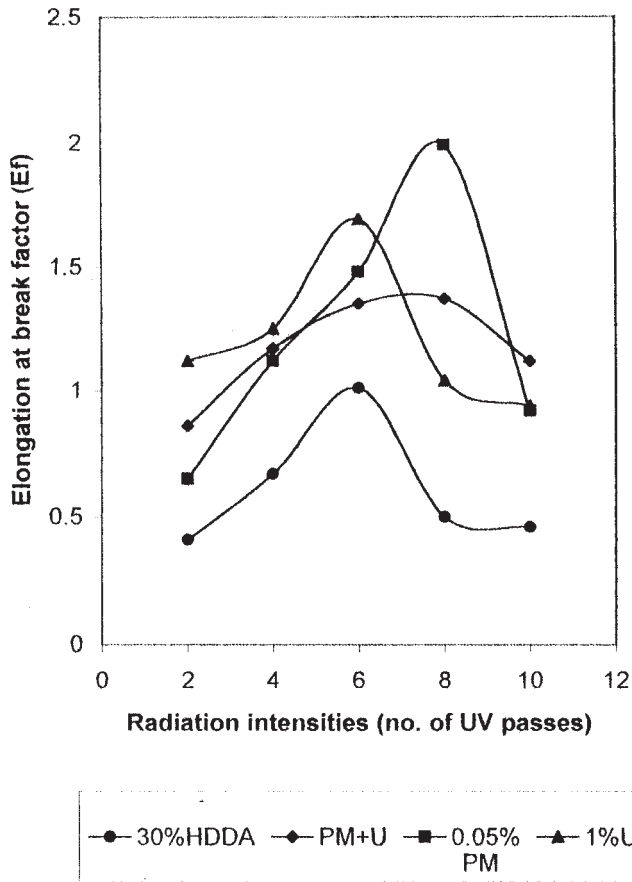


Figure 9 Effect of urea on the elongation at break factor of KMnO₄ pretreated coir fibers.

Simulated weathering test

The treated and untreated fibers were subjected to simulated weathering test to monitor its effect on the properties of the fibers. The loss of properties of the virgin fibers was much higher than that of treated fibers. After the highest period of observation, the minimum loss of properties was achieved with HDDA + U system (weight loss 2.5%) and KMnO₄ + urea system (TS loss 7.6%). The results are shown in the Table IV.

TABLE IV
Loss of Properties in Simulated Weathering Condition

Hours	Loss of properties														
	Virgin			H			H + U			H + KMnO ₄			H + KMnO ₄ + U		
	Wt	TS	EB	Wt	TS	EB	Wt	TS	EB	Wt	TS	EB	Wt	TS	EB
6	2.12	2	26.11	1.26	4	5.85	1.37	2	2.16	1.3	4.3	12	1.9	3.1	11
12	2.64	5	48	1.76	16	7.8	1.77	5	4.86	1.7	5.6	15.3	2.1	3.8	15.2
20	2.76	16	26.75	2.06	17	11.7	1.89	6	7.02	2.4	8.1	17	2.3	4.2	17.9
40	3.2	22	19.1	2.64	20	13.17	1.94	8	10.88	2.8	8.9	18.6	2.6	5.8	19.5
80	3.66	27	9	2.88	25	16.68	2.03	9	12	3.3	10	19	3.1	6.1	23
120	4.05	29.8	7.1	2.92	28	18.39	2.54	10	15	3.7	11.6	21	3.5	7.6	26.5

H, HDDA (30%); U, urea (1%).

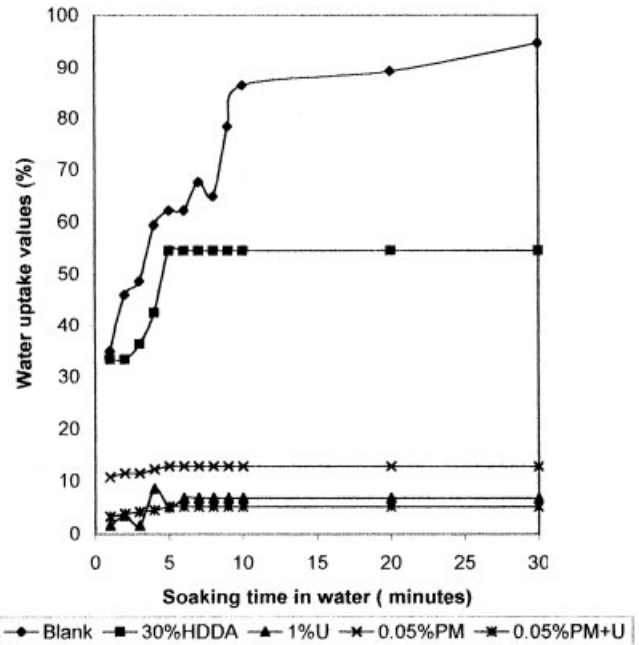


Figure 10 Water uptake values of treated and untreated coir fibers against the soaking time in water.

Degradation in soil and water

The degradation of the properties of both treated and untreated fibers were also monitored in soil and water environment. The degradation of the properties of untreated fibers was maximum. Loss of properties was higher in soil than in water. The results are tabulated in the Tables V and VI.

CONCLUSIONS

To improve the physicochemical properties, coir fibers were treated with a single monomer 1,6-hexanediol diacrylate (HDDA) in MeOH solution along with photoinitiator Irgacure-500 (2%) and cured under UV radiation. Concentration of monomer, soaking time, and radiation intensity were optimized with respect to mechanical properties. Enhanced tensile

TABLE V
Loss of Properties in Soil

Aging time (days)	Loss of properties														
	Virgin			H			H + U			H + KMnO ₄			H + KMnO ₄ + U		
	Wt	TS	EB	Wt	TS	EB	Wt	TS	EB	Wt	TS	EB	Wt	TS	EB
15	7.6	6	32	9.2	4.9	13	8.07	3.1	11.5	11.7	4.2	7.9	7.1	4.7	5.6
30	19.1	8.3	50	13.5	7.7	16	11.15	6.2	14	12.7	6.5	8.3	7.5	5.9	7.4
45	20.5	12.5	56	17.25	11.8	17	12.01	9.6	17	17.7	8.7	10	9.5	7.6	8.6
60	42.1	14	62	20.89	12.7	22	14.22	11.9	20	17.9	10.4	13.2	16.5	9.3	10

H, HDDA (30%); U, urea (1%).

TABLE VI
Loss of Properties in Water

Aging time (days)	Loss of properties														
	Virgin			H			H + U			H + KMnO ₄			H + KMnO ₄ + U		
	PL	TS	EB	PL	TS	EB	PL	TS	EB	PL	TS	EB	PL	TS	EB
15	6.2	5	22	5	4	11	44	3	9	4.2	5.0	5.6	4.8	3.7	6.7
30	17	7	27	7	5	14	8	5	12	5.9	5.8	8.2	5.5	4.1	7.3
45	23	10	34	11	8	18	10	7.5	13	6.2	6.4	9.0	7.2	4.9	8.6
60	29	12	45	16	10	19	13	9	17	10.24	7.2	10.6	8.7	6.5	10.2

H, HDDA (30%); U, urea (1%).

strength (50%) and PL (17%) were achieved with 30% HDDA at 120 min of soaking time. Urea (0.5–2%) was added to the optimized solution (30% HDDA) to improve the properties and 1% urea showed the best results (PL = 25%, T_f = 1.82). For further upgrading of the properties, the fibers were subjected to surface treatment with KMnO₄. The KMnO₄ treated fibers produced the highest enhancement of properties (PL = 100%, T_f = 350%) along with urea (1%). The degradable properties of the treated sample were monitored in different weathering conditions.

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