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Effect of Surface Treatment on the Mechanical Features of Lady's Finger Fibers Reinforced Polymer Composites

Haydar U. Zaman*, Ruhul A. Khan

Institute of Radiation and Polymer Technology, Bangladesh Atomic Energy Commission, P.O. Box 3787, Savar, Dhaka, Bangladesh

A B S T R A C T

Lady's finger fiber (LFF)-reinforced polypropylene (PP) based unidirectional composites were prepared by hot press molding and their mechanical features were evaluated. LFFs were treated with UV light at different intensities and then composites were manufactured. The mechanical features of irradiated LFF/PP composites were significantly enhanced compared to the untreated part. Irradiated LFFs were treated with a 2-hydroxyethyl methacrylate (HEMA) solution and cured with UV light. HEMA concentration and no. of UV passes were optimized for grafting and mechanical features. To further advance the features, the fibers were refined with various concentrations of alkali (NaOH) solution for 0.5 h before curing. LFFs were then grafted with an optimized HEMA solution and cured with the same UV-pass. Grafting of the alkaline treatment composite shows better mechanical features than the optimized HEMA treatment composite. SEM, water uptake, and weather testing of composites were also investigated..

Keywords: Lady's finger fiber, polypropylene, composite, alkali-treatment, and mechanical properties

1. INTRODUCTION

Experienced growing environmental awareness in recent decades has stimulated industry interest in the use of natural fibers as a reinforcing ingredient in polymer composites (Amran, 2015; Zaman et al., 2011b; Zaman et al., 2010). NFs have a low price, low-density, appropriate definite strength, good thermal insulation features, and materials serve equipment and reduce velum, and they deliver a renewable resource and can be recycled without impact ecological loss. Cellulose is the most important chemical constituent of NFs, particularly in composite making (Khan et al., 2010). Cellulose-based fibers have been used as plastic composites for structural and non-structural applications. The most associated bottlenecks were weak fiber matrix adherence by fiber, incompatibility with some polymer matrix, and moisture absorption by the fiber (Riccieri et al., 1999). To improve fiber-matrix adhesion, a pretreatment or surface modifier of the fiber surface needs to be included during processing. Some methods have been developed to fix the surface of the fibers, such as chemical treatment (Gowda et al., 1999), photochemical treatments (Furtado et al., 2020). Chemicals can effectively interlock with cellulose and activate the hydroxyl group that can induce the necessary properties of the polymer. Many coupling agents are often used to promote interactions and bonds between fibers and matrix. Alkali treatment is a process that enhances mechanical properties. Alkali treatment involves the removal of lignin, hemicellulose, and pectin from hydrophilic substances that alter the status of hydrophobic substances. Due to the huge damage to hemicellulose, the fibers can lose their cement capacity and as a result, they become separated from each other and turned into fine (Mohanty et al., 2000). Radiation is a highly appropriate procedure for graft initiation. Polymer materials for UV radiation treatment have the advantages of space decrease, no solvent emission, and rapid processing. Several processes related to the physical and mechanical properties of NFs have been reported to advance the use of various elite

* *Corresponding author.*
E-mail address: *haydarzaman07@gmail.com*

solutions under UV radiation (Rahman et al., 2019). Earlier studies have shown that the treatment of NF has significantly developed the mechanical properties of NF-reinforced composites (Zaman et al., 2011a; Zaman et al., 2009).

Of all the natural fibers, the lady's finger (*Abelmoschus esculentus*) is a monocotyledon herbaceous plant under the Malvaceae family, mainly present in Bangladesh and some other tropical regions of the world. Fiber can be extracted from the extrinsic layers of the stem. It contains 60-70% α-cellulose, 15-20% hemicellulose, 5-10% lignin, 3-5% pectin, and some water-soluble substances related to jute, flax, pineapple leaf fibrous, etc. (Alam and Khan, 2007). Fiber currently has no economic value, as it is a victim of plant burns. Recent research on their thermal and mechanical behavior has pointed out some possibilities as reinforcement in polymer-matrix composites (De Rosa et al., 2010). However, the main use of lady's finger fibers in materials has been limited to employing a moisturizer such as mucilage. Lady's finger mucilage may be a source of polysaccharides that can be used for the synthesis of biodegradable polymers using polyacrylonitrile, for example, with appropriate chemical grafting. The use of these fibers in composites will probably be considered.

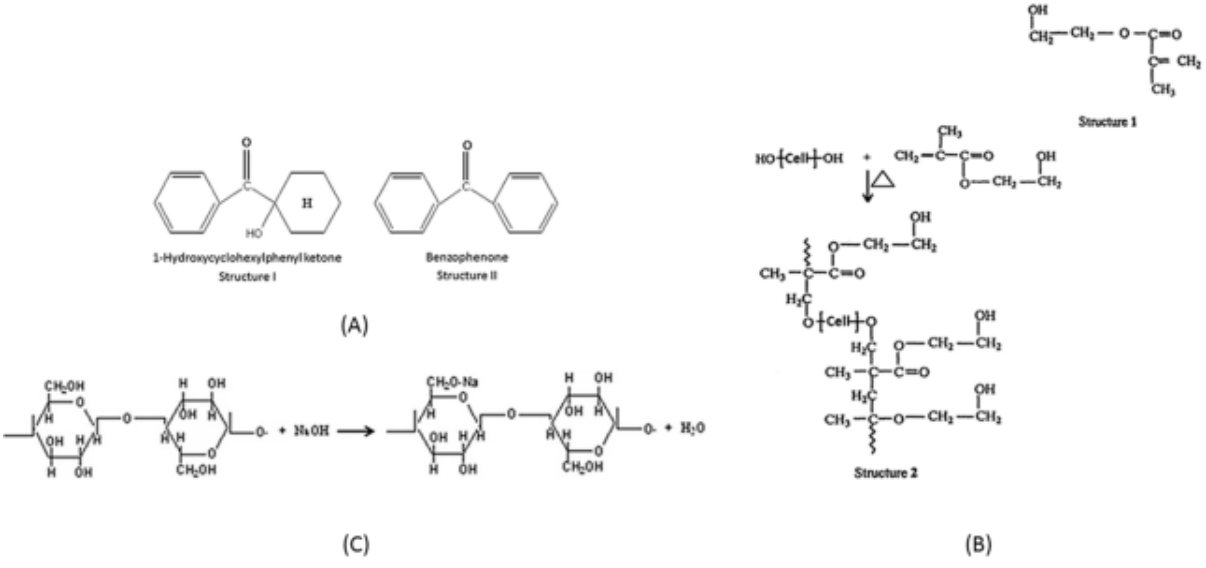
In this study, PP was chosen as a thermoplastic resin because it has several outstanding features such as transparency, superior surface strength, high impact strength, high heat distortion temperature, and dimensional stability. PP is also very suitable for filling, reinforcing and blending. PP with fibrous natural fibers is one of the potential routes to create natural synthetic polymer composites. In this present study, unidirectional lady's finger fiber- PP composites were manufactured and physicomechanical properties were evaluated. To improve the mechanical properties of the composites, lady's finger fibers were treated with HEMA under UV light before and after being treated with sodium hydroxide.

2. EXPERIMENTAL

2.1. Materials

The thermoplastic polymer matrix PP was obtained from MTBE (Malaysia) Sdn. Bhd. as the pellets form with a specific gravity of 0.91-0.92 and melting temperature of 160-170ºC. Lady's finger plant was obtained from Manikganj District (Bangladesh). After five months old and around 2 m high plants were collected. After collection, the plant was placed underwater for bacterial decay. The fiber was separated from the stacks by water retting for about 15 days. Then the fibers were washed several times using distilled water. They were dried in open air and reserved in a clean containers. HEMA was obtained from E. Merck, Darmstadt (Germany). Swelling solvent methanol (MeOH) was purchased from E. Merck, Darmstadt (Germany). Photoinitiator, Irgacure-500 procured from Ciba-Geigy (Switzerland). Irgacure-500 is a mixer of 1-hydroxycyclohexyl phenyl ketone (structure I) and benzophenone (structure II) [Scheme 1 (A)] was collected from Ciba-Geigy (Switzerland).

Scheme 1- (A) Structure of 1-Hydroxycyclohexylphenyl ketone and Benzophenone; (B) Reaction mechanism between cellulose and HEMA; (C) Proposed reaction of cellulose molecule with alkali with the production of the water molecule.



2.2 Methods

2.2.1 Fiber surface treatments

Washed-dried LFFs have been exposed to UV light; 2 kW power with a wavelength of 254-313 nm at the current of 50 amp and with various intensities (20-70 UV-pass) (UV curing device; IST technique, Germany). The source of UV light is a conductive belt, a mercury lamp inside which rotates around and once it touches the lamp, it is considered a pass, and the conductor speed is 4 m/min. Carrier distance 1 m. The composite samples were exposed to UV light and the no. of UV passes was calculated.

The alkaline treatment of NFs is called mercerization. Mercerization solution uses 5-25% NaOH in water. LFFs were immersed in mercerized solution at 15-60ºC for 0.5 h. Mercerized fibers were then thoroughly cleaned several times in water to detect any traces of NaOH. However, the final washing was done with 2% acetic acid to handle the last trace of NaOH. Finally, the washed and dried fibers were reserved in an air dryer for 24 hours.
A set of formulations was arranged and the formulated compositions are presented in Table 1.

Table 1: Composition of different formulations based on 2-hydroxyethyl methacrylate (HEMA).

Materials	Formulations (w/w %)			
	H_1	H_2	H_3	H_4
HEMA	5	10	15	20
Methanol	93	88	83	78
Photoinitiator	2	2	2	2

Exposed LFFs were dripped in this formula and cured with UV light of varying intensity (20-70 UV pass). The curing samples were removed in hot benzene for 48 h to measure the amount of grafting. The grafting percentage was determined by the formula:
Grafting (%) = $[(W_1-W_2)/W_1] \times 100$
Where W_1 and W_2 are weighed afore and after hot benzene removal. For better comparison, mercerized fibers (15% NaOH) were dipped in these formulas and permitted to stand for 5 min.

2.2.2 Preparation of composite specimens

The PP sheet (0.25-0.30 mm thickness) was made from PP pellets using a hot press machine at 170ºC with a pressure of 8 MPa for 5 min. In the manual rotation method, the LFFs were set lengthwise on the PP sheet. PP sheet layers (PL) stacked LFF layers (LFL) were formed in steps similar to PL-LFL-PL..., the outer layers were made by two layers of PP sheet. Composite samples were made using a hot press at 180ºC for 7 min at a pressure of 8 MPa and then cooled to another press with the help of two steel plates.

2.2.3 Characterizations

Tensile features of the composite sample were measured using Shimadzu UTM (Model AG-1, Japan) with electrical weight cells of 6 kN following the ASTM-D 638-03 standard. The tensile test was achieved at a crosshead speed of 10 mm/min and a gauge length of 20 mm. According to ASTM-D 256 standards, the Izod impact strength was performed using an impact machine (model, Toyo Seiki Co., Japan). The dimensions of the sample were 63.5 × 12.7 × 3 mm^3. All experiments were achieved at 23°C ± 2°C and relative humidity of 50 ± 5%. Reported values averaged over five measurements.
The SEM micrographs of untreated and treated composite samples were analyzed by a Zeiss, Evo 50 scanning electron microscope. The fracture edges of the specimens were embedded in an aluminum spit and covered with a thin layer of gold to disperse the electric charge throughout the test.
Water absorption of untreated and treated composite samples was determined by dipping the sample in a glass beaker covering water at 23°C. After a constant time break, the sample was taken out of the water, drained, and weighed.
The composite sampling was conducted by the Accelerated Weathering Tester (Model Q-U-V, Q-Panel Company, USA). Untreated, 10% HEMA treatment and alkali + 10% HMA treatment specimens were tested. The treatment differed between 60°±2°C (sunshine) and 40°±2°C (condensation) over 4 h of sunshine and 2 h of condensation for a period of 600 h. The specimens were dried in an oven for 0.5 h and measured in their tensile properties due to weather testing.

3. RESULTS AND DISCUSSION

3.1 Morphological study

Figure 1 displays the SEM diagrams of the fracture surfaces of untreated (a), 10% HEMA treatment (b), and alkali + 10% HEMA treatment (c) composites, respectively. For PP/LFF composites (Fig. 1, a), they appear to be isolated from the PP matrix and have a relatively large pull-out compared to other treatment composites. It was observed that the interfacial structure of this composite could not effectively stress transfer. This observation was consistent with the low tensile strength values given in Table 2. Significant differences in fiber-matrix interaction between alkaline with HEMA treatment and HEMA treatment composites are indicated by SEM observation. Better bonding has been observed for alkali + 10% HEMA treatment composite than for 10% HEMA treatment composite. Even if fiber pullout was observed in both cases, a significant amount of matrix residue was found in the fiber in a composite of alkali + 10% HEMA treatment. In the case of HEMA treatment, the fiber was available in the form of pullout packages and was randomly distributed by the matrix. Thus, the composite of HEMA treatments shows a greater amount of pullout. It was revealed that the alkali + 10% HEMA treatment composites were surrounded by a PP matrix, which indicates a good fiber-matrix bond (Fig. 1, c). Here, the cementing elements were slightly

removed from the multi-cellular matrix and the isolate cells became further eminent. A portion of the hemicellulose and lignin was removed from the fiber cell walls after NaOH treatment. Reducing hemicellulose can improve the mechanical features of the composite.

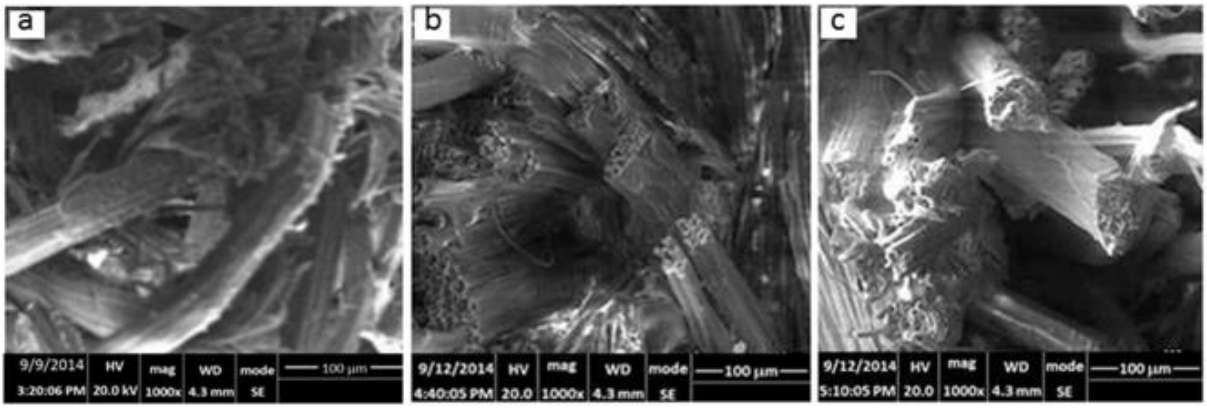


Fig. 1- SEM images of the fracture surfaces of (a) untreated (UC), (b) 10% HEMA treatment (HTC), and (c) alkali + 10% HEMA treatment (AHTC) composite.

3.2 Effect of UV light on the tensile features of the composites

Washed-dried LFFs were exposed to varying intensities (20-70 UV pass) and then composites were manufactured under UV light. The effects of UV light on the mechanical features of the exposed-LFF/PP composites were measured and presented in Table 2. We have noticed significant changes in the tensile features after LFFs were exposed to UV light. Growing the UV pass in the fiber increases the tensile strength (TS), tensile modulus (TM), and impact strength (IS) of the specimens. The maximum TS (42.9 MPa), TM (0.97 GPa), and IS (13.9 kJ/m^2) were found after 40 UV pass fibers, which was 18% 14%, and 32% higher than untreated composite. This may be due to a growth in the tensile features of the specimen with UV-passing, which may lead to intercross-links among the surrounding cellulose particles, which in turn increases the strength of the NF. It increased the tensile features by a certain amount with UV pretreatment and then decreased as a result of two opposite events, namely, photo-crosslinking and photodegradation, which occur simultaneously under UV light (Marcovich). The fusion reaction stabilizes the free radicals in the lower passes; as an outcome, photo-crosslinking or inter-crosslinking occurs in the surrounding cellulose particles. This UV-pass higher in time can break the main chain and reduce the polymer fragment. Consequently, the tensile features decline with higher UV passes.

Table 2: Mechanical features of exposed-LFF/PP composites against dissimilar UV passes.

Materials	**No. of UV passes**	**Properties**		
		TS (MPa)	TM (GPa)	IS (kJ/m^2)
Irradiated LFF/PP composites	0	36.5 ± 0.7	0.85 ± 0.04	10.5 ± 0.5
	20	38.3 ± 0.6	0.88 ± 0.06	11.9 ± 0.6
	30	40.6 ± 0.9	0.91 ± 0.03	12.7 ± 0.4
	40	42.9 ± 0.5	0.98 ± 0.05	13.9 ± 0.6
	50	41.3 ± 0.7	0.97 ± 0.04	12.7 ± 0.7
	60	39.8 ± 0.6	0.95 ± 0.06	12.3 ± 0.5
	70	38.8 ± 0.8	0.92 ± 0.07	11.9 ± 0.4

3.3 Effect of HEMA on the mechanical features of the composites

The exposed LFF (40 UV pass) was soaked for 10 min at different concentrations (5-20%) of HEMA with MeOH and cured with UV light of varying intensities (20-70 UV pass). The outcomes of grafting are illustrated in Figure 2 (A) as opposed to the no. of UV passes as a function of HEMA concentration. Grafting quantities measure the amount of cross-linking between fibrous monomers, and we noticed that grafting increases with UV pass, achieves maximum value at a certain UV pass, and then gradually decreases with UV pass.

In almost all cases, most grafting samples were treated with 10% HEMA at the 50th UV pass. The maximum grafting value (5.6%) was found by 10% HEMA followed by 15% HEMA solution. During concentrations above 10%, the amount of grafting of LFFs with HEMA decreased because grafting through a double bond with a photoinitiator of the monomer directed them into the network polymer structure to promote rapid free-radical propagation (Datta et al., 1997). The reduction in grafting with an increase in HEMA can be interpreted by the fact that during upper HEMA concentrations, a radical-radical recombination procedure may predominate, which may produce more homopolymers instead of the monomer + LFF cellulose backbone reaction (Khan et al., 1996). Minimum grafting value (2.8%) was obtained for 5% HEMA solution at 50th UV pass. From now on all experiments were performed using this optimized condition.

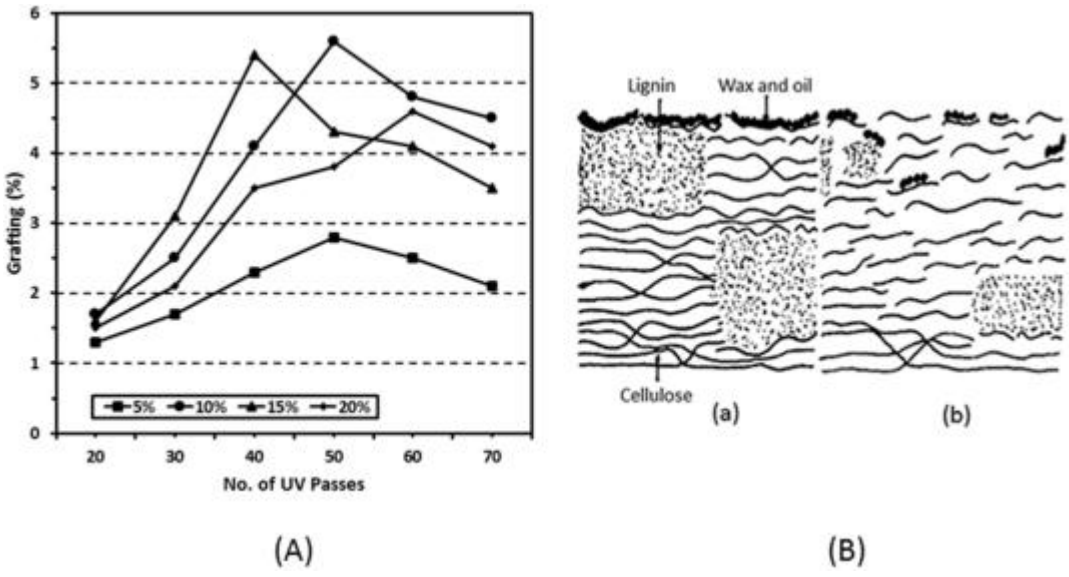


Fig. 2- (A) Grafting (%) of HEMA in LFF against the intensity of radiation related to HEMA concentration; (B) Typical structure of (a) untreated and (b) alkalized cellulosic fiber.

Mechanical features of 10% HEMA treatment exposed-LFF/PP composite have been assessed and illustrated in Table 3. The mechanical features increased with increasing UV light up to a specific value and then decreased with increasing UV pass. The maximum TS (46.3 MPa), TM (1.02 GPa), and IS (14.6 kJ/m^2) were found to be 26%, 20%, and 39% over the untreated composite. The photoinitiator produces free radicals when exposed to UV rays. This free radical initiates a free radical reaction between the monomer and -OH groups of fibers and thus enhances the fiber-matrix bond. The vinyl group of acrylate moiety of HEMA reacts with the -OH group of cellulose spine via graft-copolymerization (Scheme 1, B). This will reduce the hydrophilic nature of LFF, which is attributed to superior mechanical features.

Table 3: Mechanical features of 10% HEMA treated exposed-LFF/PP composites against dissimilar UV passes.

Materials	No. of UV passes	Properties		
		TS (MPa)	TM (GPa)	IS (kJ/m^2)
UC	0	36.5 ± 0.7	0.85 ± 0.04	10.5 ± 0.5
	20	38.5 ± 0.6	0.90 ± 0.06	11.4 ± 0.6
	30	41.8 ± 0.5	0.94 ± 0.04	12.8 ± 0.4
	40	43.5 ± 0.7	0.98 ± 0.05	13.7 ± 0.8
HTC	50	46.3 ± 0.6	1.02 ± 0.07	14.6 ± 0.6
	60	45.1 ± 0.7	1.00 ± 0.06	13.5 ± 0.8
	70	43.4 ± 0.5	0.96 ± 0.08	12.7 ± 0.5

UC: Untreated composite; HTC: 10% HEMA treated composite

3.4 Effect of alkali (NaOH) treatment on the features of the composites

Alkaline treatment progresses fiber-matrix bonding by eliminating lignin, waxy elements, and oils to cover the outer surface of the fiber cell wall (Fig. 2B, a). It makes the fibers express and gives the fibers a reasonable topography (Fig. 2B, b). The alkaline treatment increases the disparity of the exposure of the cellulose to the surface and the amount of cellulose released on the fiber surface in good mechanical interlocking. The NaOH reaction with cellulose is presented in equation 1.

$$\text{Cell-OH} + \text{NaOH} \rightarrow \text{Cell} - \text{O}^{-}\text{Na}^{+} + \text{H}_2\text{O} + [\text{surface impurities}] \quad (1)$$

The following reactions occur due to alkaline treatment (Scheme 1, C). It is noteworthy that the native cellulose I produced by alkaline short-length crystals depolymerizes the molecular structure (Fig. 2B, b). As a result, LFFs had long-term effects on mechanical behavior, especially in the case of alkali treatment (Rwawiire and Tomkova, 2015).

Washed-dried LFFs were divided by different alkali concentrations (5-25%) and at a dissimilar temperatures from 15°C to 45°C. The percentage of weight loss was determined before treatment with HEMA and is illustrated in Figure 3 (a). The percentage of weight loss due to alkaline treatment increased after raising the temperature to a certain value and decreased after reaching the maximum value. Maximum weight loss (10.5%) was obtained at a temperature of 25°C and 15% alkaline solution. LFFs were modified with a 15% alkali solution at 25°C and wholly cleaned and dried. The fibers were then grafted

with 10% HEMA and cured by UV light (50 UV-pass). Grafting values (Gr), TS, TM, and IS are presented in Table 4. The outcomes exhibited that grafting (Gr) and mechanical features were increased in the alkaline treatment composite and that the Gr was about 12%, 13% of TS, 26% of TM, and 25% of IS more than the 10% HEMA treatment composite.

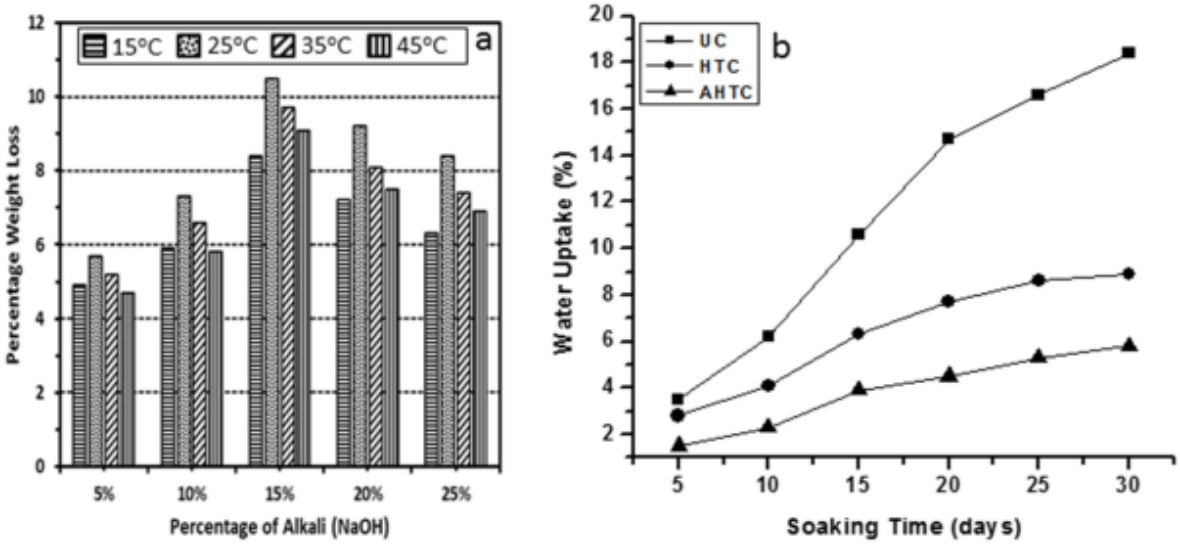


Fig. 3- (a) The effect of alkaline treatment with weight loss of LFFs at different alkali (NaOH) concentrations as the temperature function of the solution; (b) The water uptake quality of untreated and treated composite as compared to the time of soaking in water.

Table 4: Polymer grafting (%) and mechanical features of alkali + 10 % HEMA treated exposed-LFF/PP composites against dissimilar UV passes.

Materials	No. of UV passes	Properties			
		Grafting (%)	TS (MPa)	TM (GPa)	IS (kJ/m^2)
UC	0	-	36.5 ± 0.7	0.85 ± 0.04	10.5 ± 0.5
	20	3.1	40.6 ± 0.5	0.93 ± 0.05	13.2 ± 0.6
	30	3.9	43.5 ± 0.7	0.97 ± 0.06	15.4 ± 0.8
	40	5.2	48.2 ± 0.8	1.10 ± 0.07	16.8 ± 0.7
AHTC	50	6.3	52.2 ± 0.7	1.28 ± 0.05	18.3 ± 0.5
	60	5.7	51.3 ± 0.6	1.26 ± 0.07	17.4 ± 0.7
	70	4.9	50.7 ± 0.5	1.23 ± 0.08	16.1 ± 0.5

Untreated composite; AHTC: alkali + 10% HEMA treated exposed LFF/PP composite

3.5 Water uptake of the composites

The water absorption of the LFF/PP composite samples (20 × 10 × 2.5 mm^3) was determined at 25°C for about 30 days in a glass beaker and is presented in Figure 3 (b). The treated samples take water within 25 days of soaking and after that, the values remain almost stable. However, untreated specimens continue to take water throughout the observation. Alkaline + 10% HEMA treatment samples (AHTC) and untreated samples (UC) adopted low water content (5.8%) to high water content (18.4%), respectively. The water absorption of the treated specimens is reduced because HEMA reacts with the –OH group of cellulose and therefore decreases the hydrophilic nature of LFF. It can be noted that the HTC sample shows good water resistance. It is compatible with TS, TM, IS, and Gr standards.

3.6 Weather effects

Untreated and treated samples were exposed to simulated sunshine and severe weather tests over 600 hours of condensation in the periodic cycle. The TS and TM of the specimens were determined periodically. Figures 4 (a) and 4 (b) presented a decrease in TS and TM of the specimens due to weather. The loss of TS of untreated specimens in most observations was about 30%, whereas the treatment composite of 10% HEMA (HTC) and alkali + 10% HEMA (AHTC) was about 14% and 11%, respectively. Similarly, the TM loss of the untreated sample was about 26.3%, whereas the HTC and AHTC sample was about 13.7% and 8.6%, respectively. Weather tests showed that untreated specimens lost tensile features (TS and TM), but treated specimens retained tensile features for decades despite exposure to intense weather for 600 hours. The alkali + 10% HEMA treatment specimen demonstrates further weather

acceptance and is further durable than other specimens.

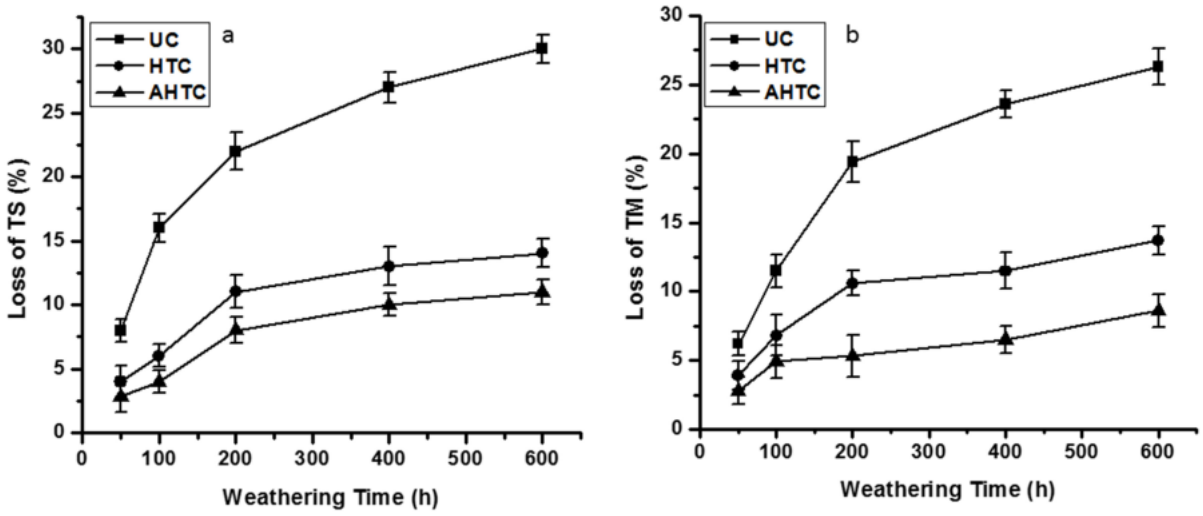


Fig. 4- (a) Loss of tensile strength of untreated and treated LFF/PP composites due to weather simulation; (b) Loss of tensile modulus of the untreated and treated composite due to weather simulation.

4. CONCLUSIONS

From the above we can conclude the following:

(1) In current works, LFF/PP specimens were made by hot press method. The mechanical features of the exposed-LFF/PP specimen (at 40 UV pass) show better results than untreated ones.

(2) Exposed LFFs were treated with HEMA solutions (5-20%) and cured with UV light of varying intensity (20-70 UV pass). Significant improvement in tensile features has been observed after HEMA treatment.

(3) Alkaline treatment LFFs were grafted with optimized HEMA solution (10% HEMA) and cured with UV light (50 UV pass). Grafting in LFF specimen with alkaline treatment shows better mechanical properties than 10% HMA treatment specimen.

(4) Water uptake behavior of treated specimens showed a significantly lower tendency than that of the untreated samples. Weather studies have shown that the TS and TM of treated specimens may be lesser than the treated specimens regarding their decay time.

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