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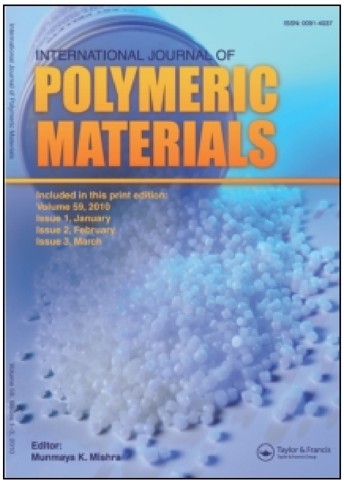

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Mercerization of Flax Fiber Improves the Mechanical Properties of Fiber-Reinforced Composites

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Mercerization of Flax Fiber Improves the Mechanical Properties of Fiber-Reinforced Composites

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In this article, modification of mercerized flax (MFx) through graft co-polymerization with methylmethacrylate (MMA) using ferrous ammonium sulphate–potassium per sulphate (FAS–KPS) redox initiator has been reported. Water uptake and moisture absorbance properties of methylmethacrylate grafted mercerized flax (MFx-g-MMA) and mechanical behavior of raw flax, mercerized flax, and MFx-g-MMA fibers reinforced—polystyrene matrix–based composites also have been evaluated. Four reaction parameters, reaction temperature, reaction time, initiator molar ratio, and monomer concentration, have been optimized to get maximum graft yield. Maximum graft yield of 138.35% has been obtained at optimum reaction conditions. The graft co-polymers thus formed were characterized by FTIR, TGA, and SEM techniques. Mercerized flax fiber reinforced showed better results than raw flax and MFx-g-MMA fibers reinforced composites.

Keywords: composites, flax, graft-copolymer, mechanical behavior, mercerization, moisture absorbance, water uptake

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INTRODUCTION

Polymers constitute a very important class of materials in the present day civilization. Many conventional materials in industry have been replaced by polymers in various technologies. Desirable and targeted properties can be imparted to meet the requirement of specialized applications by modification of variety of natural and synthetic polymers. The history of polymer modification by co-polymerization began around 1910 when it was discovered that copolymers of olefins and dienes yielded better elastomers than polyolefins or polydiens alone. However, it was not until the 1930s when Staudinger made his pioneering contribution to the field of copolymerization, and in the 1940s, when wartime shortage of natural rubber created the necessity for synthetic rubber, that this field really came in to eminence. Today, well armed with a fairly good understanding of the kinetics and mechanism of copolymerization and equipped with experience gained over the last seventy years, polymer scientists who create new macromolecules by combining known monomers in multitudinous combinations are so proficient at this work that the day does not seem very far away when the true customary synthesis of polymers with accurately predetermined properties will be a reality. Graft co-polymers of cellulosics when used in separation and enrichment technology have advantages over the conventional ones due to chemical resistance, radiation stability, low cost of preparation, low density, and so on. Cellulose is of special interest due to its abundance in nature. Graft copolymerization of vinyl monomer onto polymeric materials, including cellulose and cellulose derivatives, has been the subject of extensive studies for about four decades [1–2].

Graft co-polymerization of different vinyl monomers has been successfully attempted onto both natural [3–5] and synthetic polymers [6–8]. Misra et al. have extensively studied the modification of natural polymers such as rayon [9], wool [10–12], gelatin [13–14], as well as synthetic polymers such as poly (vinyl alcohol) PVA [15–17], and polyamide-6 [18].

Improvements in mechanical and tribological properties can be attained through the incorporation of different fiber reinforcement. These fillers, when compounded with the pertinent base resin, yield an almost limitless array of compounds that provide increasing load carrying capacity, reduced coefficients of friction, improved wear life, higher mechanical strengths, improved thermal properties, greater fatigue endurance and creep resistance, and excellent dimensional stability and reproductability. Composites are recent class of engineering materials and have found numerous applications in different fields. Fiber-reinforced plastics of thermoplastic materials offer a number of technical advantages for variety of engineering, aerospace, and automotive applications. Because of the advantages of natural fibers over conventional reinforcement materials, such as renewability, low cost, abundance, high specific resistance, and non-abrasiveness [19–20], these fibers have recently attracted the attention of research workers. Therefore, in some technical applications, natural fiber–reinforced composites appear to be an alternate material to glass fiber–reinforced plastics.

Although flax fibers have extensively been used in industries for the preparation of composite materials no information is available on the graft co-polymerization of mercerized flax (Mercerized flax means alkaline-treated flax) with methylmethacrylate and their applications as reinforcement materials with polymer-based matrix composites. Also, as only few reports are available on polystyrene-based matrix composites, it was therefore considered worthwhile to prepare polystyrene-based matrix composites using raw flax, mercerized flax, and MFx-g-MMA fibers as a reinforcing agent. The composites thus formed have been further evaluated for mechanical properties such as wear resistance, tensile strength, and compressive strength.

EXPERIMENTAL

Materials

Purification of Materials

flax fiber was purified by refluxing in acetone for 72 h in soxhlet apparatus [21]. MMA monomer was purified by washing with 5% NaOH and subsequently drying over anhydrous Na_2SO_4 followed by distillation. Middle fraction was used for grafting. KPS (BDH) was used as received and FAS (BDH) was recrystallized from hot water.

Reinforcing Agent

flax fibers were given the shape of a thread of 0.5 mm diameter by twisting them.

Matrix

High molecular weight thermoplastic resins produced generally by the free-radical polymerization of styrene monomer [$C_6H_5CHCH_2$], which can be initiated by heating alone, but more effectively by heating in the presence of free-radical initiator (such as benzoyl peroxide [$(C_6H_5CO)_2O_2$]. Typical processing techniques are modified mass polymerization or solution polymerization, suspension polymerization, and expandable beads. Major markets for polystyrene are in consumer and institutional products, electrical/electronic uses, and building/construction.

Gel-like material of PS was prepared with the help of chloroform, as PS is easily dissolved by organic solvents.

Composite Sample Die

For making composite samples, a die of aluminum having a length of 40 mm and cross-section of 5 × 5 mm was used.

Methods

Mercerization of Flax Fiber

Mercerization of flax was done as per the standard procedure [22].

Graft Co-Polymerization

Activation of flax fiber (0.2 g) was done at room temperature by immersing it in 100 ml of distilled water for 24 h prior to carrying out graft copolymerization. A definite ratio of FAS-KPS was added to the flax fiber and water mixture as an initiator in reaction flasks. A known amount of monomer was added to each reaction flask. The reaction mixtures were stirred at 25, 35, 45, 55, and 65°C keeping other reaction parameters (reaction time, initiator ratio, and monomer concentration) constant. After a pre-determined reaction time, the ingredients were filtered out and the residue was extracted for 48 h with acetone to remove the homopolymer (PMMA). Graft co-polymers thus obtained were dried at 50°C to a constant weight and then optimum reaction temperature was noted down. Optimization of other reaction parameters, that is, reaction time, initiator ratio, and monomer concentration was then carried out in order to get the maximum graft yield (Table 1).

The percent grafting (Pg) and percent efficiency (Pe) were calculated from the increase in the weight of flax fiber sample in the following manner:

$$\%\ \text{Grafting (Pg)} = \frac{\text{Wg} - \text{W}}{\text{W}} \times 100$$

$$\%\ \text{Efficiency (Pe)} = \frac{\text{Wg} - \text{W}}{\text{Wm}} \times 100$$

where W, Wg, and Wm are weights of ungrafted fiber, grafted fiber, and monomer, respectively.

TABLE 1 Evaluation of Optimum Reaction Parameters for Grafting of MMA onto Mercerized Flax

Sr. no.	Temp. (°C)	Time (min.)	$[MMA] \times 10^{-3}$ mol/lt	Molar ratio FAS: KPS	P_g	P_e
1	25	120	1.96	0.50 : 1	0.00	0.00
2	35	120	1.96	0.50 : 1	0.00	0.00
3	45	120	1.96	0.50 : 1	27.75	2.83
4	55	120	1.96	0.50 : 1	24.75	2.55
5	65	120	1.96	0.50 : 1	10.70	1.09
6	45	60	1.96	0.50 : 1	2.25	0.23
7	45	90	1.96	0.50 : 1	48.70	4.97
8	45	150	1.96	0.50 : 1	56.25	5.74
9	45	180	1.96	0.50 : 1	20.40	2.08
10	45	210	1.96	0.50 : 1	39.10	3.99
11	45	150	1.96	0.25 : 1	60.15	6.14
12	45	150	1.96	0.75 : 1	7.90	0.81
13	45	150	1.96	1.00 : 1	0.00	0.00
14	45	150	1.96	1.25 : 1	0.00	0.00
15	45	150	1.96	1.50 : 1	0.00	0.00
16	45	150	0.98	0.25 : 1	0.00	0.00
17	45	150	1.47	0.25 : 1	1.10	0.15
18	45	150	2.45	0.25 : 1	36.90	3.01
19	45	150	2.94	0.25 : 1	138.35	9.41
20	45	150	3.43	0.25 : 1	88.55	5.1

Moisture Absorbance Study of Mercerized Flax (MFx) and MFx-g-MMA Fibers

Moisture absorbance studies at various humidity levels were carried out as per the method reported earlier [23]. Moisture absorbance percentage was found out by placing a known weight (W_i) of dry grafted and ungrafted samples in a humidity chamber (Swastik make) for about 2 h and then the final weights (W_f) of the samples exposed at different humidities ranging from 20 to 100% were taken.

The percent moisture absorbance was calculated from the increase in initial weight in the following manner.

$$\%\ \text{Moisture absorbance}\ (\%M_{abs}) = \frac{W_f - W_i}{W_i} \times 100$$

Water Uptake Study of MFx and MFx-g-MMA Fibers Through Capillary Action

Small wicks of the grafted and ungrafted flax fibers of 0.5 mm diameter were prepared by hand and initial mark with ink pen was drawn on the wick and then it was dipped into a beaker containing water. Rise of water through capillary action in each fiber wick was noted down at different time intervals.

Preparation of Composites

A die was firstly half filled with the PS gel material. Five numbers of flax fibers (0.1 g), each of 0.5 mm diameter were arranged longitudinally within the die and then the remaining half of the die was filled with the PS gel. Samples so prepared were cured in an hot air oven at 50°C for 5 h. This was followed by air drying for about 1.5 h. These samples were subjected for different mechanical properties, that is, wear resistance, tensile strength, and compressive strength.

Wear-Test

Wear testing was done as per ASTM D 3702 on DUCOM machine (TR-20LE) designed for ISO-9001. The friction disc was cleaned with emery paper at a speed of 4 m/s. Initial weight of the sample was taken and the sample was fixed in the jaws of the wear-testing machine. Then the machine was set to display zero wear and friction. The speed was varied from 1 m/s to 8 m/s and the machine started giving definite trends at 4 m/s. Further testing was done at 4 m/s with varying loads of 0.2 kg, 0.5 kg, 1.0 kg, 1.5 kg, and 2.0 kg. For each load the machine was allowed to run for 15 min and the readings were recorded. After 15 min, the sample was taken out and the final weight was noted hence the actual weight loss due to wear was recorded.

Tensile Strength Test

The tensile strength testing was carried out as per the ASTM D 3039 on Universal Testing Machine (model H25 K-H, by Hounsfield). The composite sample of 4 cm length was clamped in two jaws of the machine. Each end of the jaws covered 1 cm of the sample. Tensile stress was studied for the remaining 2 cm portion of the sample. Readings for force and extension were set at zero. Strain rate was fixed at 10 mm per min and total extension range was 25 mm. Tensile stress was applied till the failure of the sample, and the total extension per unit force was noted.

Compressive Strength Test

Compressive strength of the samples was studied by using the procedure described in ASTM D 3410 on Universal Testing Machine (model H25 K-H, by Hounsfield). Composite sample was held between two platforms. The strain applied was at the rate of 10 mm per min whereas the total compression range was 7.5 mm. Initially readings for force and compression were set at zero and the compression stress was applied till the complete failure of the sample. Total compression per unit force applied was noted.

Evidences of Grafting

Characterization of Ungrafted and Grafted Fibers by FTIR Spectroscopy

IR spectra of the MFx fiber and MFx-g-MMA fibers were taken with KBr pallets on Bomem, Hartmann, & Braun (MB Series) spectrophotometer. flax fiber showed a broad peak at 3420 cm^{-1} due to bonded $-OH$, 2907 cm^{-1} due to $C-H$ stretching and at 2925.5, 1458.6, and 1055.87 cm^{-1} arising from $-CH_2$, $C-C$, and $C-O$ stretching, respectively. On grafting, IR bands due to characteristic functional groups incorporated onto flax have been witnessed apart from the previously listed bands. MMA; 1730 cm^{-1} due to carbonyl group of MMA.

Characterization of Ungrafted and Grafted Fibers by Scanning Electron Microscopy (SEM)

The scanning electron microscopic analysis of the MFx fibers and MFx-g-MMA was carried out on a LEO Electron Microscopy Machine (Sr.No.435-25-20). On comparing the scanning electron micrographs of ungrafted flax and its graft co-polymers, it has been observed that upon grafting a considerable amount of polymer is deposited onto the grafted flax surface.

Characterization of Ungrafted and Grafted Fibers by Thermogravimetric Analysis/Differential Thermal Analysis (TGA/DTA)

Thermogravimetric analysis and differential thermal analysis of the MFx and MFx-g-MMA fibers were carried out in air on a thermal analyzer LINSEIS (L-81 11). TGA of ungrafted and grafted flax has been studied as a function of wt%. loss v/s temperature (Table 2). Cellulosic flax degrades by dehydration, depolymerization, and glycosan formation. In case of mercerized flax, two-stages of decomposition have been found in temperature ranges from 307.2–345.4°C with the weight loss of 31.5% and 345.4–559.3°C with the weight loss of 39.33%. The former stage is attributed to loss by dehydration and depolymerization processes, whereas the later stage is attributed to loss by volatilization process.

MFx-g-poly (MMA) fiber also degrades by two-stage decomposition. After the initial loss of moisture, decomposition continues beyond 277°C with weight loss of 59.5% until 331.6°C and a weight loss of

TABLE 2 Thermal Behavior of Ungrafted and Grafted Mercerized Flax

		Thermogravimetric data					DTA peaks (°C)	
Sr. no.	Cellulosic	Degrad stages (°C)	Weight loss (%)	Residue (%)	IDT (°C)	FDT (°C)	Exo	Endo
1	MFx	307.2–345.4	31.5	29.17	307.2	559.3	309.5	360
		345.4–559.3	39.33				465.6	
2	MFxMMA	277–331.6	59.5	3.83	277	446.6	346.8	370
		331.6–446.6	36.67				415.1	

36.67% in temperature range from 331.6–446.6°C. The above mentioned results show a decrease in thermal stability after grafting, which may be due to disturbance in original crystal lattice of fiber because of impregnation of monomer chains in the matrix.

In the last region between 513.9–600°C, weight of residue (29.17%) almost remains constant in mercerized flax fiber and it is attributed to char formation whereas in MFx-g-poly (MMA) almost total degradation take place.

DTA of ungrafted mercerized flax is also different from that of MFx-g-poly (MMA).The exothermic peak at 309.5°C in flax fiber corresponds to decomposition stage between 307.2–345.4°C and exothermic peak at 465.5°C corresponds to second decomposition stage 345.4–559.3°C in TGA curve. Sharp exothermic peak arising from 230.8°C is due to the onset of degradation of MFx-g-poly (MMA) chains and this peak extends to 346.8°C with extensive heat loss. Another exothermic peak at 415.1°C is observed. These peaks correspond to the region between 277–331.6°C and 331.6–446.6°C in the TGA curve, respectively. Beyond the second exothermic peak, the degradation of grafted fiber proceeds smoothly with constant weight loss of the backbone polymer.

RESULTS AND DISCUSSION

Mechanism

C_2, C_3, and C_6 hydroxyls and C$-$H groups (Figure 1) are active sites for the incorporation of polymeric chains through grafting onto cellulosic fibers like flax.

KPS is known to take part in a redox reaction with Fe^{2+} (from FAS) through the following chemical equation:

$$Fe^{2+} + {}^{-}O_3S-O-O-SO_3^{-} \longrightarrow Fe^{3+} + SO_4^{2-} + SO_4^{-}$$

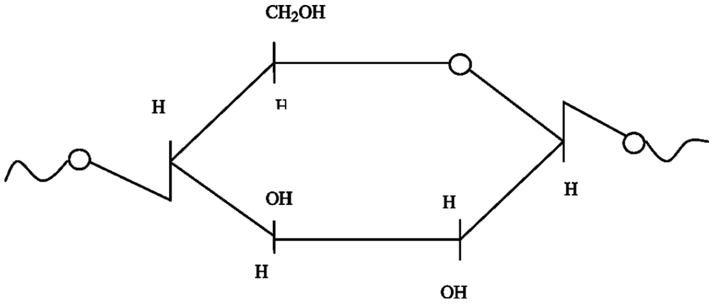

FIGURE 1 Structure of cellulose.

Interaction of SO_4^{-} with H_2O generates $OH^{\bullet}$ and these free radicals are responsible for grafting of polymeric chains onto the flax backbone, thereby resulting in the formation of copolymer along with homopolymer [24]. It can be explained through the following mechanism:

$$SO_4^{-} + H_2O \longrightarrow HSO_4^{-} + OH^{\bullet} \tag{1}$$

$$SO_4^{-} + \mathrm{MFxCell{-}OH} \longrightarrow HSO_4^{-} + \mathrm{MFxCell{-}O}^{\bullet} \tag{2}$$

$$\mathrm{MFxCell{-}OH} + OH^{\bullet} \longrightarrow \mathrm{MFxCell{-}O}^{\bullet} + H_2O \tag{3}$$

$$M + OH^{\bullet} \longrightarrow OH{-}M^{\bullet} \xrightarrow{nM} OH{-}(M)^{\bullet}_{n+1} \tag{4}$$

$$OH{-}(M)^{\bullet}_{n+1} + \mathrm{MFxCell{-}OH} \longrightarrow \mathrm{MFxCell{-}O}^{\bullet} + OH{-}(M)_{n+1}{-}H \tag{5}$$

$$\mathrm{MFxCell{-}O}^{\bullet} + M \longrightarrow \mathrm{MFxCell{-}O{-}M}^{\bullet} \xrightarrow{nM} \mathrm{MFxCell{-}O{-}}(M)^{\bullet}_{n+1} \tag{6}$$

$$\mathrm{MFxCell{-}O{-}}(M)^{\bullet}_{n+1} + {}^{\bullet}(M)_{n+1}{-}OH \longrightarrow \mathrm{MFxCell{-}O{-}}(M)_{2n+2}{-}OH \tag{7}$$

$$\begin{gathered} \mathrm{MFxCell{-}O{-}}(M)^{\bullet}_{n+1} + Fe^{3+} \longrightarrow \mathrm{MFxCell{-}O{-}}(M)_{n+1}{-}Fe^{2+} \\ \text{(Graft copolymer)} \end{gathered} \tag{8}$$

$$\begin{gathered} OH{-}(M)^{\bullet}_{n+1} + M^{\bullet} \longrightarrow OH - (M)_{n+1}{-}M \\ \text{(Homopolymer)} \end{gathered} \tag{9}$$

where MfxCell−OH = mercerized flax cellulose and M = monomer.

On the basis of this mechanism it can be concluded that both $SO_4^{\cdot}$ and $OH^{\cdot}$ are involved in graft copolymerization. However, in case of grafting carried out in the absence of swelling agent, initiation of reaction by SO_4^- (Eq. 2) is unlikely as the concentration of persulphate used is very small, whereas in presence of swelling agent, SO_4^- reacts with H_2O to generate $OH^{\cdot}$, which can initiate the grafting reaction. The resulting polymeric chain abstracts hydrogen atom from the backbone polymer and generates the macro-radical (MFxCell-$O^{\cdot}$) and the grafting propagates further. On the other hand, $OH^{\cdot}$ can directly generate macro-radical (MFxCell-$O^{\cdot}$) by hydrogen abstraction. Because initiation is a faster reaction than hydrogen abstraction by $OH^{\cdot}$ the generation of macro-radical by the former process is more likely to take place. Termination of grafting may occur by either process (7) or (8) or both.

Presence of Fe^{3+} has great impact on graft yield as it is involved in the termination of growing chains. Because Fe^{3+} is produced by the reaction between FAS with KPS, the relative amounts of KPS and FAS in the initiating system should influence the graft yield. Hence, concentrations of initiator and monomer, variations of time and temperature affect the graft yield as these factors determine the relative population of various radical species generated in different steps during the course of reaction.

Optimization of Reaction Parameters for Graft Co-Polymerization of Monomer MMA Onto Mercerized Flax

The availability of the number of active sites on the polymer backbone affects the extent of the grafting. Other variables that affect the grafting are the various reaction parameters like reaction time, reaction temperature, molar ratio of initiator, and concentration of monomer. Extensive studies were made to optimize these four reaction parameters (Table 1)

Effect of Reaction Temperature

Reaction temperature has been found to play an important role in getting maximum graft yield. Figure 2 describes the effect of reaction temperature on percentage of grafting of MMA onto mercerized flax. Increase in temperature beyond optimum reaction temperature, that is, 45°C resulted in the decrease in graft yield, which could be due to increase in viscosity of reaction medium and subsequently hindrance in the movement of the radicals to the polymeric backbone active sites [25]. Considerable homo-polymerization and occurrence of various hydrogen abstraction reactions at high temperature also resulted in the wastage of monomer by its participation in side-chain reactions

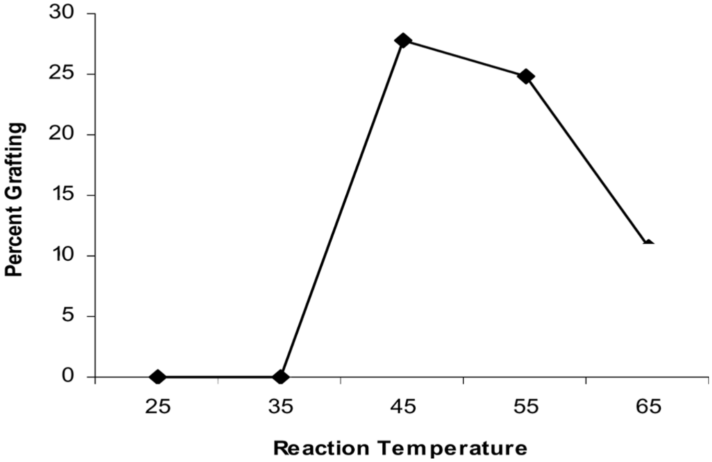

FIGURE 2 Effect of reaction temperature on grafting. Mercerized flax fiber, 0.2 g; [MMA], 1.96×10^{-3} mol/lt; time, 120 min; FAS-KPS, 0.50:1.

[26]. At high temperature, the fiber also loses weight and strength due to removal of water soluble constituents of the fiber [27].

Effect of Reaction Time

Reaction time greatly affects the graft yield. Percentage of grafting of monomer as a function of time is presented in Figure 3. The decrease in graft yield beyond optimum reaction time (150 min) is due to the predominance of homo-polymerization over graft co-polymerization and other side reactions [26]. Difficulty in the mobility of the radicals to polymeric backbone active sites is also created due to increase in the viscosity of reaction medium on further increase in reaction time beyond optimum [25].

Effect of Molar Ratio of Initiator

Graft yield was greatly affected by molar ratio of initiator FAS-KPS, as shown in Figure 4. The percent grafting was found to decrease with increase in molar ratio (beyond critical value), which is evident from the mechanism that at higher molar ratio more and more Fe^{3+} ions are generated, which result in the termination of growing chains (Eq. 8). On the other hand, if FAS-KPS molar ratio falls below the critical value, then there is less generation of OH^- ions, which obviously results in decreased formation of macro-radical (MFxCell-O^-) ions and hence decreased percent grafting (Eq. 3) [22].

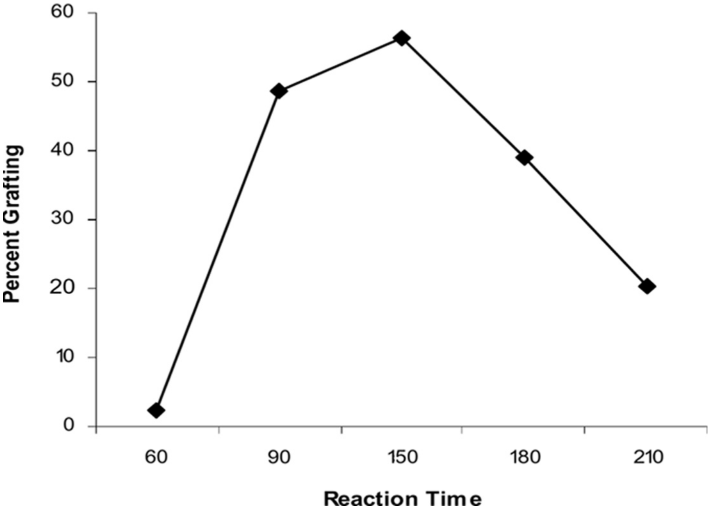

FIGURE 3 Effect of reaction time on grafting. Mercerized flax fiber, 0.2 g; [MMA], 1.96×10^{-3} mol/lt, temperature, 45°C; FAS-KPS, 0.25 :1.

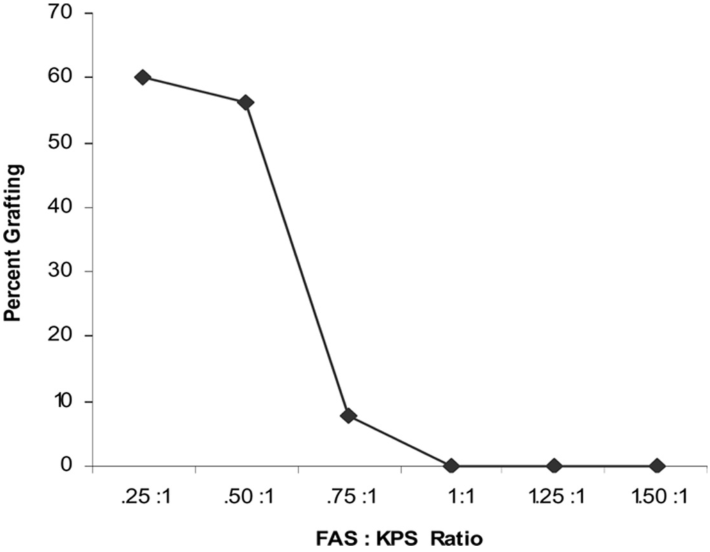

FIGURE 4 Effect of concentration of initiator (FAS:KPS ratio) on grafting. mercerized flax fiber, 0.2 g; [MMA], 1.96×10^{-3} mol/lt, temperature, 45°C; time, 150 min.

Effect of Monomer Concentration

Figures 5 depicts the effect of concentration of monomer MMA on percentage of grafting onto mercerized flax. From Figure 5, it is evident that there is a sudden increase in the percentage of grafting when the concentration of MMA is increased from 2.45×10^{-3} to 2.94×10^{-3} mol/lt, where maximum percentage of grafting (138.35%) was observed. After reaching maximum value of grafting, a further increase in monomer concentration results in decrease in the graft yield, which probably occurs due to prefered formation of homopolymer over the graft co-polymerization [28].

Moisture Absorbance Study of Mercerized flax (MFx) and MFx-g-MMA Fibers

It has been observed that the graft co-polymerization of MMA onto mercerized flax has a great impact on the moisture absorbance behavior when compared with that of mercerized flax, as clearly shown in Figure 6. With increase in graft yield, there has been a decrease in percent moisture absorbance (%Mabs.), which is due to the fact that

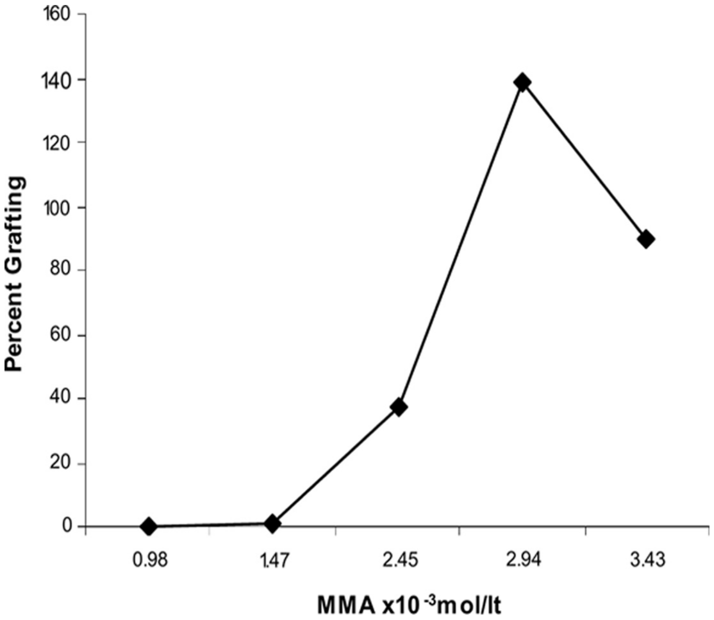

FIGURE 5 Effect of monomer concentration on grafting. mercerized flax fiber, 0.2 g; FAS-KPS, 0.25:1, temperature, 45°C; time, 150 min.

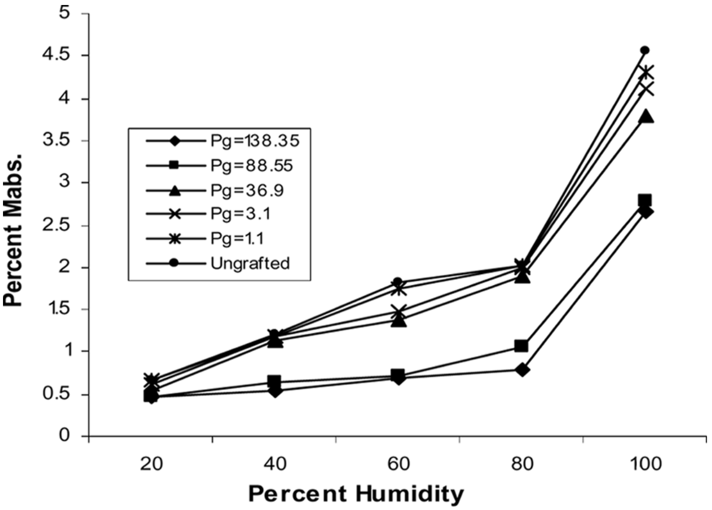

FIGURE 6 Effect of grafting on moisture absorbance at different humidity levels.

with increase in graft yield, the sites for maximum moisture absorbance are blocked [28].

Water Uptake Study Through Capillary Action

It has been observed that water uptake of the fiber decreases with increase in percent grafting, as shown in Figure 7. This is due to the fact that graft co-polymer chains of MMA grafted onto flax fiber have less affinity for water as compared to hydroxyls and other functional groups present in ungrafted fiber. Therefore, the resistance of fiber toward water increases with increase in grafting [28].

Mechanical Behavior of flax Reinforced and Polystyrene Matrix–Based Composites

Strength of composites depends on the adhesion of fiber reinforced and matrix, which in turn depends on several factors, one of which is the roughness of fiber. Mercerization increases the surface roughness resulting in better mechanical interlocking and the amount of cellulose exposed on the fiber surface. It also increases the number of reaction sites and allows better fiber wetting. Strength of composites also depends on the strength of reinforcing fiber because when a load is applied there is localized plastic flow in matrix, which transfer the load to the fibers embedded in it. The fibers inhibit slip and crack

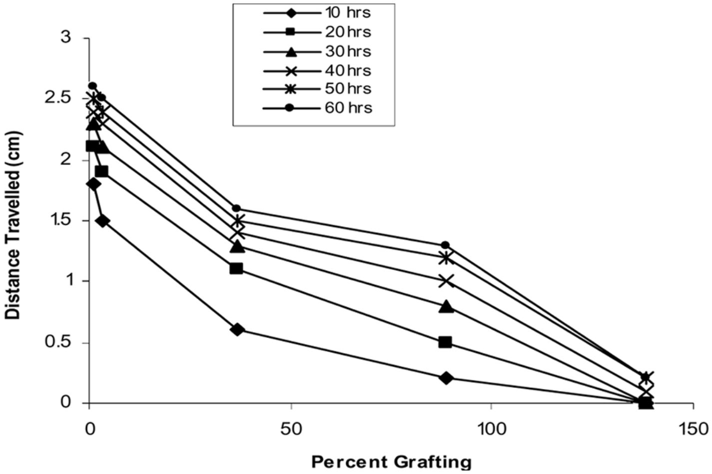

FIGURE 7 Effect of grafting on water uptake.

propagation, thereby increasing mechanical properties. Mercerization has a lasting effect on the mechanical behavior of flax fibers, especially on in fiber strength and stiffness [29]. Therefore, increase in both roughness and strength of fiber result in increase in the strength of the composite. Grafting in the presence of redox initiator results in increase surface flaws and hence weakens the fiber. Grafting tends to increase the strength of composite by increasing the roughness of fiber that is, by providing better adhesion between fiber and matrix, but at the same time it also tends to decrease the strength of the composite as a result of weakening of fiber. Predominance decides the effect of grafting on the strength of composite.

Wear Resistance

It has been observed that in the case of polystyrene, material loss takes place by the mechanism of abrasion and micro-cutting up to 1.5 kg load. Increasing the load to 2.0 kg leads to increase in wear rate by about 1.5 times, which may further be attributed to frictional heat generated due to sliding. It has been observed that reinforcement with mercerized fiber improves the wear resistance significantly as compared to reinforcement with graft co-polymers. It is evident from Table 3 that wear rate is reduced more on reinforcement of the polystyrene with mercerized flax fiber as compared to raw flax and graft copolymers. Higher wear resistance of mercerized flax fiber–reinforced composites is due to the fact that on mercerization the fiber

TABLE 3 Wear Rate of Raw, Mercerized and Grafted Mercerized Flax Reinforced Composites

	Wear resistance (gm/m × 10^{-4})				
Sample code	0.2 Kg	0.5 Kg	1 Kg	1.5 Kg	2 Kg
Polystyrene	6.21 ± 0.11	7.91 ± 0.06	8.86 ± 0.15	10.33 ± 0.18	16.76 ± 0.01
Raw Flax reinforced	1.24 ± 0.02	2.04 ± 0.04	4.42 ± 0.05	8.12 ± 0.09	15.85 ± 0.05
Mercerized Flax(MFx) reinforced	0.71 ± 0.07	1.12 ± 0.10	1.54 ± 0.08	2.4 ± 0.04	6.15 ± 0.16
MFx-g-MMA reinforced	1.74 ± 0.08	3.12 ± 0.09	2.41 ± 0.06	6.66 ± 0.15	7.16 ± 0.1

swells and reduces surface flaws, whereas in case of graft co-polymers there is in increase in surface flaws due to grafting, thereby resulting in weakening of the fiber [22]. Moreover, morphology of the fiber changes due to grafting. Martins et al. [30] reported that after mercerization, the fiber surface gets changed. The parenchyma cells get partially removed, and the fibrils start to split, because of the alkali action. As a result, the effective surface area available for contact with matrix increases.

Compressive Strength

In compression, the adhesive and stiffness properties of binder are crucial in order to maintain the fibers as straight columns and prevent buckling. Fiber-reinforced composite compression failure occurs when the fibers exhibit (often sudden and dramatic) buckling. The main mode of failure of composites under longitudinal compressive load arises due to buckling of columns or micro buckling. Generally micro buckling is preceded by de- bonding of constituents and micro cracking of matrix.

Better results have been observed in case of reinforcement with mercerized flax fiber than raw and grafted fibers. Mercerization is usually used to produce high-quality fibers [31] and it also improves the fiber-matrix adhesion as it removes the natural and artificial impurities [32]. Poor results in case of grafted fibers are due to the fact that grafting result in weakening of fiber.

In case of reinforcement with MFx, composites could bear a load of 500 ± 7.2 Newton force with compression of 1.3 ± 0.05 mm whereas composites reinforced with MFx-g-poly (MMA) could bear a load of 200 ± 4.6 Newton force with compression of 0.8 ± 0.05 mm, respectively (Table 4). Pure polystyrene could withstand a load of 100 ± 5.5 Newton force with a compression of 0.27 ± 0.02 mm.

TABLE 4 Compressive Strength of Raw, Mercerized, and Grafted Mercerized Flax Reinforced Composites

Sample code	Compressive strength (N/mm^2)	Compression (mm)
Polystyrene	100 ± 5.5	0.27 ± 0.02
Mercerized Flax(MFx) reinforced	500 ± 7.2	1.3 ± 0.05
MFx-g-MMA reinforced	200 ± 4.6	0.8 ± 0.05
Raw Flax reinforced	400 ± 3.6	0.9 ± 0.04

Tensile Strength

Tensile strength of a material is the maximum amount of tensile stress that it can be subjected to before it breaks. The failure of composite under longitudinal tensile load is initiated by breaking of fibers at their weakest points. When some fibers break, the load carried by them is passed on the remaining ones. As the load further increased, the number of broken fibers also increases; thereby some loci of composite may become too weak to support the increased load which, in turn, may lead to complete rupture of the composite. Behavior under tensile load is highly dependent on the stiffness and strength of the fiber.

Composites reinforced with mercerized flax (MFx) fiber showed better result in comparison to composites reinforced with raw and grafted fibers (Table 5), which could be due to decrease in surface flaws on mercerization. Composite reinforced with MFx fibers failed at 175 ± 2.6 Newton force with extension of 4.7 ± 0.04 mm whereas composites reinforced with MFx-g-Poly(MMA) failed at 165 ± 2.6 Newton force with extension of 0.89 ± 0.02 mm. Pure polystyrene could not withstand even the load of 125 ± 3.6 Newton force with an extension of 0.42 ± 0.05 mm.

TABLE 5 Tensile Strength of Raw, Mercerized, and Grafted Mercerized Flax Reinforced Composites

Sample code	Tensile strength (N/mm^2)	Extension (mm)
Polystyrene	125 ± 3.6	0.42 ± 0.05
Mercerized Flax(MF) reinforced	175 ± 2.6	4.7 ± 0.04
MF-g-MMA reinforced	165 ± 2.6	0.89 ± 0.02
Raw Flax reinforced	175 ± 4.6	4.3 ± 0.01

CONCLUSION

Grafting of MMA onto flax has increased the resistance toward water because MMA grafted onto flax fiber has less affinity for water than the ungrafted fiber. Mercerized flax fiber reinforced showed better mechanical properties than raw flax and MF-g-MMA-reinforced composites. Reduction of surface flaws due to mercerization is responsible for higher strength. Grafting in the presence of redox initiator results in increase in surface flaws and hence weakens the fiber.

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