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Enhancement of hydrophobic character of lignocellulosic fibers through graft-copolymerization

Amar Singh Singha[*] **and Raj K. Rana**

Department of Applied Chemistry, National Institute of Technology Hamirpur, Hamirpur 177005, H.P., India

[*]Corresponding author. Tel: (+91) 98058 45675; Fax: (+91) 1972 223834; 98058 45675; E-mail: singhaasnit@gmail.com (A.S. Singha), rajrananith@gmail.com (R.K. Rana).

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ABSTRACT

Owing to hydrophilic behavior lignocellulosic fibers, e.g. Agave is unacceptable in various fields of applications. In the present communication attempts have been made to enhance hyrdrophobicity of these fibers through surface modification using methyl methacrylate as vinyl monomer. The grafting reaction initiated by ceric ion was carried out in vacuum. The various reaction parameters such as reaction time, temperature, monomer concentration, initiator concentration and vacuum were optimized to obtain the maximum percent graft yield. The grafted samples were evaluated for the hydrophobic character and other physico-chemical properties such as swelling behavior in different polar and nonpolar solvents alongwith chemical resistance behavior. The grafted products were further characterized by different techniques such as FT-IR, SEM, X-RD and thermogravimetric studies. Copyright © 2010 VBRI press.

Keywords: Agave; hydrophobic; vacuum; SEM; graft-copolymerization.

A.S. Singha is Professor & Head of Department of Chemistry at National Institute of Technology Hamirpur (H.P.) India. He did his B.Sc. M.Sc. M.Phil and PhD in organic chemistry from Himachal Pradesh University, Shimla, India. He started his career as a Lecturer form DAV College Kangra (H.P.) in the year 1982. He moved to National Institute of Technology Hamirpur (then known as Regional Engineering College, REC) in the year 1986. He became Assistant Professor in the year 1990 and Professor in the year 1998. He has been associated with the institute as Dean Academic (2002-07), Dean Planning and Development (2007-2009) and Registrar of the Institute for one year. His area of research includes synthesis and characterization of polymer resin based composites, modification of natural fibers and reaction mechanisms. He has about 210 publications to his credit.

R.K. Rana is Senior Research Fellow in the Department of Chemistry National Institute of Technology Hamirpur (H.P.) India. He has obtained Bachelor's degree in Science from Himachal Pradesh University in 1996 and Master's Degree in Industrial Chemistry from Institute of Basic Sciences Khandari Campus Agra University in 2000. His area of research is chemical modification of lignocellulosic fibers, synthesis and characterization of polymer resin based composites.

Introduction

The utilization of biomass has gained an increased importance due to threats of limited petroleum availability and concerns about the environmental pollution. Eco friendly, renewable and biodegradable composite materials prepared by using lignocellulosic fibers of plant origin have become the most required material in the present time [**1**]. The uses of natural fiber reinforced composites are expanding day by day opening new application windows. These natural fiber reinforced composites have proved of great importance in many areas such as automobile, aerospace, sports, marine industry, packaging and construction industries etc. However hydrophilic natures of hydroxyl groups present on the cellulose molecules hamper the application of the native lignocellulosic fibers as tailored material. This has resulted in an increasing demand for surface modification of these cellulosics in order to develop hydrophobic character [**2-3**]. Therefore efforts are on to develop new processes and efficient methods to reduce the hydrophilicity of the cellulosic fibers and thus improving the compatibility of natural fibers with polymer matrices [**4**]. A number of methods for surface modification of lignocellulosic fibers such as mercerization, silane treatment, acetylation, benzoylation, plasma treatment etc.

have been reported in literature [**5-11**]. Among various methods of surface modification of cellulosic fibers graft copolymerization is an attractive method to impart a variety of functional groups on the polymer backbone.

Grafting of vinyl monomers onto the cellulosic materials provides a potential method for altering the physico-chemical and mechanical properties of the polymer substrates. Depending upon the structure of the grafted monomer the cellulosic materials can be made hydrophobic [**12**], heat resistant [**13**] and resistance to microbial attack [**14**]. The graft copolymerization of vinyl monomers onto lignocellulosic fibers is generally considered to take place in three steps. The first step is the generation of reactive sites on the surface of substrate. Second step is the addition of monomer to the substrate and third step is the propagation of monomer chain on the backbone polymer. Graft copolymerization reactions are generally carried out in aqueous medium. In water the amorphous regions on the cellulosic fiber undergo swelling which result in the easy and rapid diffusion of the monomer radicals onto backbone and hence leads to more grafting efficiency [**15**]. Grafting efficiency can be increased either by pre swelling of cellulosic fibers or by carrying out the grafting reaction in a medium in which cellulosic fibers undergo swelling.

Graft efficiency also depends upon the conditions in which the graft copolymerization reaction is carried out. The role of reaction conditions is very important in determining the percent graft yield as it affects the diffusion of the monomer radicals onto the cellulosic backbone and thus determines the time of completion of the grafting reaction. A large number of studies on graft copolymerization of vinyl monomers onto lignocellulosic fibers under different conditions such as in air, under the influence of microwave radiations and under pressure are available in literature [**16-19**].

In the present communication graft copolymerization of methyl methacrylate onto *Agave americana* fibers was carried out in vacuum. The graft copolymers thus obtained were evaluated for hydrophobic character and other physico-chemical properties such as swelling behavior and chemical resistance. The graft copolymers were then characterized by fourier transform infra red (FT-IR) spectroscopy, scanning electron microscopy (SEM), thermogravimetric analysis (TGA) and X-ray diffraction (X-RD) studies. Agave fibers were obtained from the leaves of wild growing perennial plant belonging to Agavaceae family.

Agave plant has more than 200 species all over the world. *Agave americana* species is native to Himachal Pradesh, a hilly state of Himalayan region and found in large excess in the vicinity of National Institute of Technology Hamirpur. In local language it is called as '*Kyor*' and utilized by people for domestic purposes like making of ropes, bags, mats etc. The present work ensures the commercial utilization of this natural wealth which is of immense importance keeping in mind the day by day depleting petroleum resources and increasing environment threats. The surface modified fibers will be used as reinforcement in place of toxic synthetic fibers in the preparation of biocomposites.

Experimental

Materials

Methyl Methacrylate (MMA) was received from CDH India and was purified by first washing with 5% NaOH and then drying over anhydrous Na_2SO_4. Finally it was subjected to distillation and the middle fraction of distillate was used for the further studies. All other reagents were of analytical grade and were used as received. Ceric ammonium nitrate (CAN) is obtained from Merck chemicals and was used as initiator. Acetone supplied by Rankem laboratories was used for extraction of homopolymer from graft copolymer.

Weighing was performed on digital balance of Shimadzu Libror AEG-220 make. Grafting reaction was carried out in vacuum oven supplied by Sunbeam India. Humidity chamber of Swastika make was used to evaluate the hydrophobic character of graft copolymers.

Sample preparation

Leaves were collected from the Agave plant and immersed in flowing water till outer cuticle, and waxes on the covering layer of leaves were removed. The leaves were then taken out and rubbed gently in order to remove the cementing materials and separating the fiber bundles. The fibers thus obtained were washed with mild detergent to remove impurities involved during extraction of fibers. The fibers were then transferred to hot air oven maintained at 80˚C for 12 hours and finally soxhlet extracted with acetone for 72 hours. The dried fibers were then cut into pieces of length 85-100 mm and 0.5 g of fiber was used as sample in each grafting reaction. Double distilled water was used as solvent during the grafting reaction.

Grafting reaction

Fibers were subjected to preswelling in distilled water for 24 hours before graft copolymerization. Preswelling of fibers activates the reactive sites on the surface and also results in the faster diffusion of the monomer radicals from the reaction medium. After swelling the fiber samples were transferred to the reaction kettle containing 100 ml of distilled water used as solvent in the grafting reaction. A definite amount of CAN and concentrated nitric acid was added to the reaction mixture which was followed by the addition of definite amount of monomer in small fractions with constant stirring. The reaction kettle was placed in vacuum oven maintained at definite temperature for a definite interval of time. On the completion of grafting reaction samples were taken out and homopolymer formed was soxhlet extracted with acetone. The grafted samples were dried in hot air oven at 60˚C and percent graft yield and grafting efficiency was calculated as per following equation (1, 2).

$$\%\ Grafting\,(P_g) = \frac{W_g - W}{W} X\ 100 \qquad (1)$$

$$\% \ Efficiency \ (P_e) = \frac{W_g - W}{W_m} X \ 100 \qquad (2)$$

Where, W, W_g and W_m are the weights of raw fiber, grafted fiber and monomer respectively. Various reaction parameters such as reaction time, temperature, vacuum, concentration of initiator, nitric acid and monomer were optimized to get maximum graft yield.

Reaction mechanism

Grafting reaction proceeds through redox mechanism in which Ce(IV) ions are reduced to Ce(III) ions by the transfer of electron from cellulose molecule and hence free radical sites are generated on the fiber surface at which monomer radicals attack to form graft copolymers (Eq. 9). Homopolymer is also formed by combination of monomer free radicals in the reaction medium [**20**].

$$R + Ce^{4+} \rightarrow [C] \rightarrow R^{*} + Ce^{3+} + H^{+} \qquad (3)$$

$$M + Ce^{4+} \rightarrow [D] \rightarrow M^{*} + Ce^{3+} \ H^{+} \qquad (4)$$

$$M^{*} + nM \rightarrow (M)^{*}_{n+1} \qquad (5)$$

$$(M)^{*}_{n+1} + R \rightarrow (M)_{n+1} - H \ + R^{*} \qquad (6)$$

$$R^{*} + M \rightarrow RM^{*} \qquad (7)$$

$$RM^{*} + nM \rightarrow R(M)^{*}_{n+1} \qquad (8)$$

$$R(M)^{*}_{n+1} + Ce^{4+} \rightarrow R(M)_{n+1} + Ce^{3+} \qquad (9)$$

$$(Graft\,copolymer)$$

$$(M)^{*}_{n+1} + Ce^{4+} \rightarrow (M)_{n+1} + Ce^{3+} \qquad (10)$$

$$(M)^{*}_{n+1} + M^{*} \rightarrow (M)_{n+1} - M \qquad (11)$$

$$(Homopolymer)$$

Where R= -C-CH_2OH of *Agave americana* fiber, M = monomer (MMA), C = fiber and ceric ion complex
D = Monomer and ceric ion complex.

Measurements and characterization

Fourier transform infrared (FT-IR) spectra of raw and grafted fibers were recorded on Perkin Elmer FT-IR spectrophotometer using KBr pellets. The samples were exposed to IR radiations in the range of 4000 to 500 cm^{-1} with a resolution of 2 $cm^{-1.}$

TGA/DTA measurements were made on Perkin Elmer Pyris Diamond thermal analyzer in air. All the samples were subjected to a constant heating rate of 10˚C/min. Thermal studies were performed over a temperature range of 25˚C to 800˚C in the air with flow rate of 200ml/min. Signal calibrated smoothed curves were used for further analysis.

Surface topology of the graft copolymers was studied with the help of scanning electron microscopy. SEM studies were made on Leo scanning electron microscopy machine 435 VP. Before focusing electron beam on the samples they were coated with Gold suspension in order to make them conducting. The image resolution was set at 500 X.

X-ray studies were carried out on Brucker D8 Advance X- ray diffractometer, using Cu Kα (1.5418Å) radiation, Ni filter and a scintillation counter as a detector at 40 KV and 40 mA on rotation from 5˚ to 50˚ at 2θ scale.

Finely powdered samples placed in central cavity of sample holder made up of polymethyl methacrylate (PMMA) were used for X-Ray diffraction studies. Crystallinity index was measured by using wide angle X-ray diffraction counts at 2θ scale close to 22˚ and 18˚. Crystallinity index measures the orientation of the cellulose crystals with respect to the fiber axis. The counter reading of peak intensity close to 22˚ and 18˚ represents the crystalline and amorphous region in cellulose respectively [**21**]. Percent Crystallinity and crystallinity index (C.I.) was calculated as per method reported [**18**].

$$\% \ C_r = \frac{I_C}{I_C + I_A} X \ 100 \qquad (12)$$

$$C.I. = \frac{I_C - I_A}{I_C} \qquad (13)$$

Optimization of grafting parameters

Various reaction parameters such as reaction time, temperature, vacuum, concentration of initiator, monomer and nitric acid upon which the graft yield depends were optimized before evaluating the hydrophobic character and other properties of the graft copolymers. When grafting reaction was carried out under optimum conditions, percent grafting of 15.2% was obtained.

Grafting time

Grafting reactions were carried out for different time intervals ranging from 30 to 150 minutes. It was observed that percent graft increased initially as reaction time increases, reached maximum at 90 minutes and then decreased again with further increase in the reaction time. The variation of percent grafting with time has been represented in **Table 1**.

As the reaction time increases, more and more monomer radicals are generated and get sufficient time to interact with fiber backbone resulting in increased percent graft yield. However after reaching the optimized value further increase in the reaction time, P_g decreases due to the reason that the lesser number of the reactive sites are available for grafting. Also viscosity of the reaction medium increases due to homopolymer formation and thus monomer radicals could not diffuse easily on to the fiber backbone. Therefore, the percent grafting decreases with increase in time after optimized value.

Table 1. Optimization of grafting time

Sr. No.	Grafting Time (Min)	Percent Grafting (P_g)	Percent Efficiency (P_e)
1.	30	7.8	2.05
2.	60	10.0	2.63
3.	90	12.2	3.24
4.	120	11.5	3.05
5.	150	5.6	1.49

Reaction temperature

Table 2 gives the variation of graft yield with temperature. It is clear from the table that temperature has a considerable effect on the grafting reaction. Graft yield increases as temperature is increased. It becomes maximum at 45˚C which is optimum temperature for grafting reaction. However, graft yield decreases when we proceed beyond this temperature.

Table 2 Optimization of reaction temperature.

Sr. No.	Grafting Temp. (˚C)	Percent Grafting (P_g)	Percent Efficiency (P_e)
1.	25	9.8	2.60
2.	35	13.4	3.56
3.	45	15.2	4.03
4.	55	12.5	3.32
5.	65	11.3	2.98

The initial increase in the graft yield with rise in temperature could be due to increase in the kinetic energy of the molecules which resulted in the faster diffusion of greater number of monomer radicals onto fiber backbone. However further rise in temperature beyond optimum value, collisions among the radicals increase which causes hindrance in the movement of radicals towards the reactive sites on the fiber backbone.

Effect of vacuum

Graft copolymerization is supposed to take place under soft conditions when carried out under vacuum. It is probably due to the reason that grafting reaction under vacuum proceeds without the influence of environmental conditions. Graft yield increases initially with increase in vacuum. It may be due to the reason that as vacuum increases chances of interaction between monomer radicals and active sites on fiber backbone increase. However after reaching the optimum value, graft yield decreases with further increase in vacuum. It may be due to the increase in the interactions among the monomer radicals at high vacuum resulting in the formation of dead polymer. **Table 3** depicts the variation of percent graft yield with vacuum.

Table 3 Optimization of vacuum.

Sr. No.	Vacuum (mm/Hg)	Percent Grafting (P_g)	Percent Efficiency (P_e)
1.	250	10.6	2.78
2.	350	11.2	2.96
3.	450	12.8	3.42
4.	550	11.5	3.05
5.	650	9.7	2.55

Table 4. Optimization of initiator concentration.

Sr. No.	[CAN] × 10^3 Mol/L	Percent Grafting (P_g)	Percent Efficiency (P_e)
1.	4.38	4.5	1.19
2.	6.58	8.7	2.29
3.	8.77	15.2	4.03
4.	10.96	11.4	2.99
5.	13.15	9.8	2.60

Concentration of initiator

When the initiator concentration increases initially more and more Ce(IV) ions are produced in the reaction medium which result in the generation of greater number of reactive sites on the fiber backbone and monomer radicals in the reaction medium [Eq. 3, 4]. Hence graft yield increases with the initial rise in the initiator concentration. After reaching the optimized value when initiator concentration is further increased, free radical monomer chain is terminated due to presence of large excess of Ce(IV) ions which react with monomer radicals. **Table 4** represents the data for optimization of initiator concentration.

Concentration of nitric acid

The percent grafting increases with increase in the concentration of the nitric acid. However, increasing nitric acid concentration beyond optimum value resulted in the hydrolysis of the fiber backbone and also in the degradation of the monomer radical. Hence, percent grafting decreases with further increase in the concentration of nitric acid. Optimization of nitric acid concentration has been shown in **Table 5**.

Table 5. Optimization of nitric acid concentration.

Sr. No.	$[HNO_3] \times 10$ Mol/L	Percent Grafting (P_g)	Percent Efficiency (P_e)
1.	0.69	7.9	2.09
2.	1.38	10.5	2.78
3.	2.07	11.6	3.08
4.	2.77	15.2	4.03
5.	3.46	9.0	2.39

Table 6. Optimization of monomer concentration.

Sr. No.	$[MMA] \times 10$ Mol/L	Percent Grafting (P_g)	Percent Efficiency (P_e)
1.	0.45	7.8	2.06
2.	0.91	10.8	2.87
3.	1.36	11.9	3.14
4.	1.81	15.2	4.03
5.	2.26	10.5	2.78

Monomer concentration

Increase in the concentration of monomer results in increase in the graft yield up to certain value known as optimum value. Further increase in the monomer concentration results in the decreased graft yield (**Table 6**). This behavior is because of the reason that as we increase concentration of monomer more and more monomer radicals are available in the reaction mixture to participate in the grafting reaction hence graft yield increases. However, increasing the monomer concentration beyond optimum value leads to the formation of homopolymer which predominates over the graft copolymerization.

Study of hydrophobicity of grafted fibers

Hydrophobic character of grafted fibers was evaluated in terms of percent swelling in different polar and nonpolar solvents and percent moisture absorbance under different humidity levels.

Swelling behavior

A definite weight of sample was immersed in a solvent for 24 hours and after taking out the sample, excess of solvent was removed .The final weight of the sample was noted down and percent swelling was calculated as per method reported [**18**]. The swelling studies were performed in water, ethanol, DMF and CCl_4.

Moisture absorbance behavior

Before subjecting to different humidity conditions each sample was made moisture free by placing in hot air oven at 60˚C for 12 hours. Moisture absorbance behavior was studied under different humidity levels ranging from 40% to 95%. A sample of known weight was placed in the humidity chamber maintained at a definite humidity level for 2 hours. The sample was then taken out and final weight was noted down immediately. Percent moisture absorbance was calculated as per method reported [**18**].

Evaluation of chemical resistance

Chemical resistance of graft copolymers was studied as a function of percent weight loss by immersing the known weight of grafted sample in the solutions of HCl and NaOH of different normalities ranging from 0.5N to 2.0N for 24 hours. The sample was taken out and dried to constant weight in hot air oven. The final weight of the sample was noted down and percent weight loss was calculated as per method reported earlier [**18**].

Results and discussion

Evidence of Graft Copolymerization

SEM: Scanning electron microscopy gives the morphological evidence of the graft copolymerization. From SEM images it was observed that grafting leads to the change in the surface topology of the fiber. The surface of the fiber becomes rough due to the attachment of the polymer chains over it. **Fig. 1** and **2** represent the scanning electron micrographs of raw and grafted fibers respectively which were recorded at a magnification of 500 X.

FT-IR: FT-IR spectrum of raw *Agave americana.* fiber showed a broad peak at 603 cm^{-1} (due to out of plane –OH bending), 898 cm^{-1} (due to β-glycosidic linkage), 1257.9 cm^{-1} (due to -C-O-C and –C=O stretching in xylan side substituent and lignin aromatic C=O stretching), milder peaks at 1381.2- 1432.0 cm^{-1} (due to -CH, $-CH_2$ and $-CH_3$ bending), 1505.8 cm^{-1} (lignin aromatic ring vibration and stretching), 1652.7 cm^{-1} (due to H-O-H bending of absorbed water and for lignin C-H deformation), 2138.7 cm^{-1} (due to O-H stretching of absorbed moisture), 2368.5 cm^{-1} (due to C-H stretching in polysaccharide chains),2918.1cm^{-1} (for C-H stretching vibration of aliphatic methylene groups) and 3397.1 cm^{-1} (due to bonded -OH group). However in case of Agave-g-Poly (MMA) an additional peak at 1735.3 cm^{-1} was observed, which is peculiar to the carbonyl group (>C=O) of ester stretching vibrations. The above results suggest that MMA has been grafted onto the agave fiber through covalent linkages. The FT-IR spectra of raw and grafted fibers have been given in **Fig. 3**.

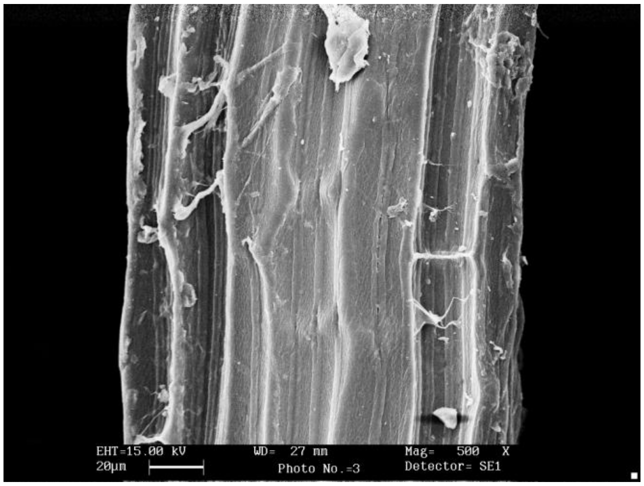

Fig. 1. SEM of raw fiber.

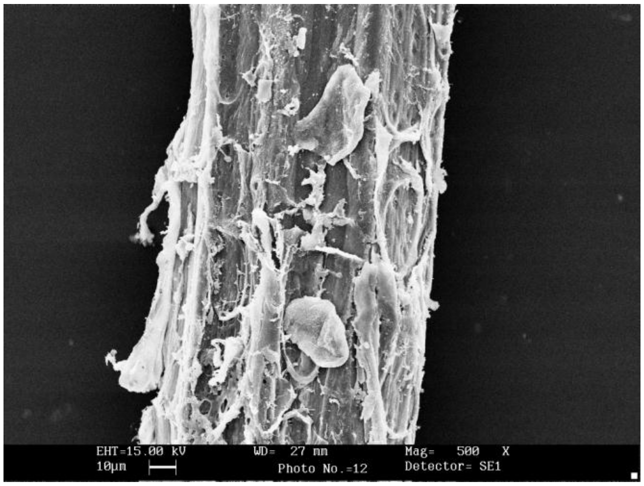

Fig. 2. SEM of grafted fiber.

XRD: **Fig. 4** represents the diffractogarms of raw and grafted fibers. It is evident from the figure that raw fiber gave peaks at 22.03˚ and 14.22˚ with relative intensities of 97.6 and 46.1 respectively. Also grafted fiber gave peaks at 22.25˚ and 17.54˚ with relative intensities of 100 and 45.7 respectively. **Table 7** represents the crystallinity index and percent crystallinity of raw and grafted fibers. It is clear from the table that there was no appreciable change in the crystallinity of the fiber upon grafting under vacuum. It may be explained by the fact that the grafting process mainly occurs in the amorphous regions of the fibers without any remarkable disturbance in the crystal lattice [**22**].

Thermal analysis: The effect of grafting of methyl methacrylate on the thermal stability of Agave fibers was studied with the help of thermogravimetric technique. **Fig. 5** and **6** represent TGA/DTA curves for raw and grafted fibers respectively. The results reveal increase in thermal stability of the fiber upon grafting.

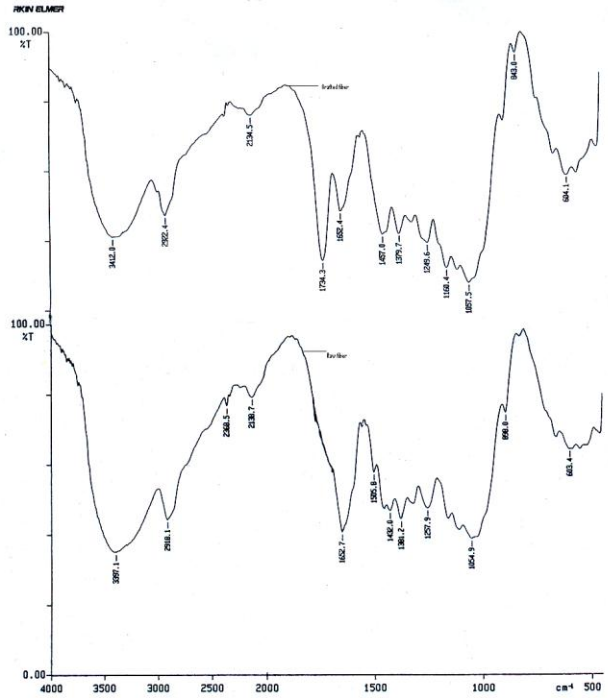

Fig. 3. FT-IR spectra of raw and grafted fiber.

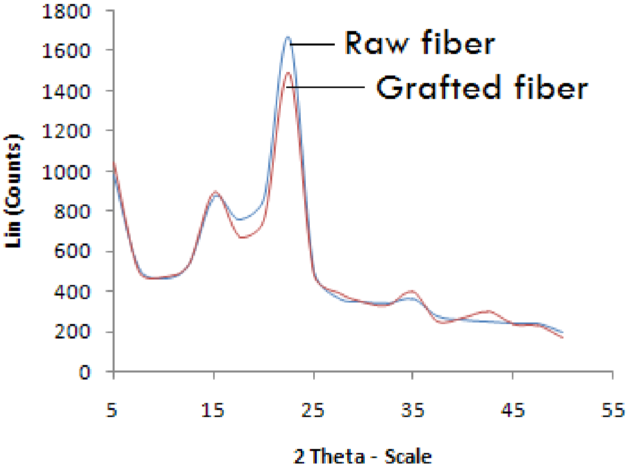

Fig. 4. X-RD of raw and grafted fiber.

Table 7. % Crystallinity and crystallinity Index (C.I.) on raw and grafted fibers.

Sample	$I_{C\ 22.03^\circ}$	$I_{A\ 14.22^\circ}$	% Cr	C.I.
Raw fiber	97.6	46.1	67.92	0.53
	$I_{C\ 22.25^\circ}$	$I_{A\ 17.54^\circ}$	% Cr	C.I.
Grafted fiber	100	45.7	68.6	0.54

The initial and final decomposition temperatures of raw fiber were 240°C and 432°C respectively. Raw fiber exhibited two stage decomposition, with the maximum weight loss between 240°C - 346°C (50.6 %) and 346°C -

432°C (32.3 %). The first stage of decomposition may be due to cellulosic decomposition and the second stage may be due to the degradation of lignin and oxidation of char.

The initial and final decomposition temperatures of grafted fiber are 270°C and 477°C respectively. In case of grafted fibers the first stage decomposition takes place at 270°C to 352°C with weight loss of 78.4 %. It can be attributed to the breakdown of hemicellulose and glycosidic linkages of cellulose whereas the second stage of decomposition occurred between 352°C to 477°C with weight loss of 21.3 %. This may be related to the degradation of grafted poly (MMA) chains onto the fiber surface. TGA results are also well supported by DTA.

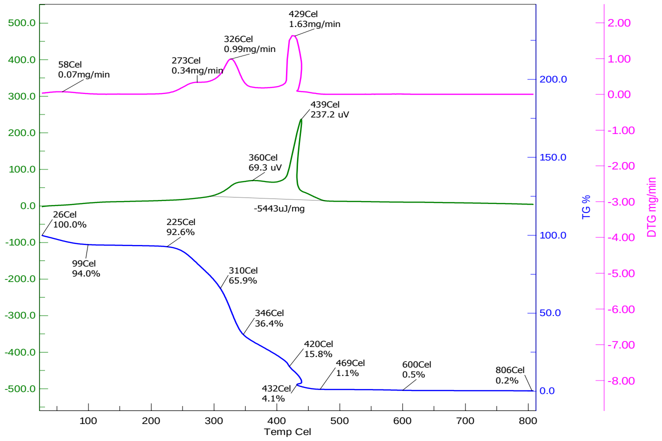

Fig. 5 Thermogram of raw fiber.

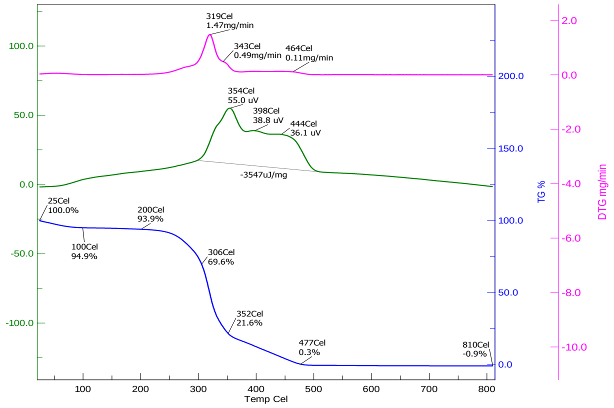

Fig. 6. Thermogram of grafted fiber.

Hydrophobic character of graft copolymers

The swelling studies were performed on raw fibers as well as samples of grafted fiber having different percent graft yields. The results of swelling studies have been shown in **Table 8**. It is evident from the table that raw fiber shows maximum swelling in water. As percent graft yield increases % swelling of grafted fiber in water decreases and reaches to 29.4% at percent graft yield of 15.2%. Moreover raw fiber has 15.8% swelling in CCl_4. With the increase in graft yield percent swelling in CCl_4 also increased and reaches to 85% at graft yield of 15.2%.

Moisture absorbance studies were performed in the humidity chamber under humidity levels ranging between 40% to 95%. The results of humidity studies have been shown in **Table 9**. At a particular humidity level % mab was maximum for raw fiber and it decreases with increase in the percent graft yield. At 95% humidity raw fiber absorbed 42% moisture whereas grafted fiber having 15.2% graft yield absorbed only 16.3% moisture.

Table 8. Swelling studies in different solvents.

Sr.		% Swelling			
No.	Pg	Water	Ethanol	DMF	CCl_4
1.	0	93	41.5	38.4	15.8
2.	4.5	65	33.4	39.3	47.4
3.	8.7	56	26.7	52.2	58.7
4.	11.6	37.5	20.3	63.4	76
5.	15.2	29.4	16.8	66.3	85

Table 9. Moisture absorbance studies.

Sr. No.	Pg	% Moisture absorbance at different humidity levels			
		40%	60%	80%	95%
1.	0	5.9	8.8	19.6	42
2.	5.6	4.7	6.2	12.8	27.8
3.	9.8	2.4	5.3	11.8	24.0
4.	11.6	1.8	4.9	9.7	20.2
5.	15.2	1.2	3.3	6.9	16.3

From the above discussion it is clear that grafting results in decrease in the hydrophilic character of lignocellulosic fibers. However there is enhancement of the hydrophobic character of these fibers as a result of grafting of methyl methacrylate. Also the hydrophobic character increases with increase in the percent graft yield. The above behavior of grafted fiber may be due to the fact that hydrophobic poly (MMA) chains are incorporated on the surface of lignocellulosic fibers as a result of graft copolymerization. Therefore affinity of grafted fibers towards –OH group of water and other polar groups decreases which leads to the decrease in the hydrophilic character.

On the other hand these hydrophobic poly (MMA) chains are more solvolysed by non polar solvents such as DMF and CCl_4.

Chemical resistance studies

Resistance to Hydrochloric Acid (HCl): **Table 10** represents the acid resistance studies in terms of weight loss of the fiber when immersed in HCl of different strengths for 24 hours. It is clear from the table that grafted fibers are more resistant to the attack of acids and hence show lesser weight loss as compared to raw fibers.

Table 10. Acid resistance studies.

Sr. No.		% Weight Loss (HCl)			
	P_g	0.25N	0.5N	0.75N	1.0N
1.	0	8.2	13.7	16.0	18.9
2.	4.5	7.0	10.6	12.8	16.3
3.	5.6	6.4	10.1	12.2	15.8
4.	8.7	5.5	9.6	11.7	14.6
5.	12.5	4.5	8.3	10.3	14.1
6.	15.2	3.8	6.2	9.5	13.4

Table 11. Base resistance studies.

Sr. No.		% Weight Loss (NaOH)			
	P_g	0.5N	1.0N	1.5N	2.0N
1.	0	18.6	22.0	27.0	34.5
2.	4.5	13.4	17.7	20.0	28.7
3.	5.6	12.7	16.3	18.7	26.3
4.	8.7	9.8	13.2	16.9	20.4
5.	12.5	7.8	10.8	13.6	16.4
6.	15.2	5.6	9.3	12.8	14.9

Resistance to Sodium Hydroxide (NaOH): The effect of sodium hydroxide of different strengths on both raw and grafted fibers have been shown in **Table 11**. Base resistance behavior of raw as well as grafted fibers has been reported in terms of percent weight loss. It is observed from the results of base resistance studies that grafting results in the increase in base resistance of Agave fibers.

From the above studies it is observed that chemical resistance of lignocellulosic fibers increases on graft copolymerization with (MMA). It can be explained by the reason that most of the reactive sites on the surface of fibers which are prone to attack by chemicals are blocked by poly (MMA) chains. Therefore grafted fibers exhibit more resistance to acids and bases as compared to raw fibers where reactive sites are unoccupied and easily attacked.

Conclusion

From the present work it was concluded that graft copolymerization of MMA onto Agave fibers has been found to be an efficient way to enhance the hydrophobic character of these fibers. Moreover hydrophobic character of lignocellulosic fibers increases with the increase in the extent of percent grafting. From X-RD studies it was concluded that there in no appreciable change in the crystal lattice of the fibers after graft copolymerization. Thermal stability of lignocellulosic fibers increases after grafting of MMA onto fiber which is a good sign for application of these fibers especially at elevated temperatures. Grafted fibers are more resistant to the chemicals as compared to raw fibers.

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