Studies on Fiber Properties of Sodium Hydroxide-Treated and Grafted Murga Fibers—Microscopical Study

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Synopsis

Murga fiber (purified) treated with sodium hydroxide shows crimp. Above 20% NaOH concn no further change in crystallinity is marked. Incorporation of AN onto fiber matrix through grafting has been carried out. The surface is characterized by a scanning electron microscope.

INTRODUCTION

Murga fiber belongs to the family of *Liliaceae*, a plant group producing long leaves from which the fiber is extracted. The botanical name of this fiber is Sanseveria Ruxvergina. The long vegetable fibers produced in the world are basically used in manufacture of agricultural ropes and other crude applications.

In the recent past, the increasing production of vegetable fibers and with the ever-increasing cost of production of synthetic fibers have necessitated further research on the commercial vegetable fibers, with potential end use in industry in the developing countries. This murga fiber is widely grown in various part of Orissa, a state of India. The present investigation has been carried out on murga fibers (both NaOH-treated and AN-grafted). The crimp and some mechanical properties have been studied.

MATERIALS AND METHODS

Murga fibers were freed from organic impurities with Soxhlet extraction for 24 h in benzene and alcohol mixture (1:1), washed repeatedly with distilled water, and finally dried in air.

The ultimate single cells were obtained by treating with sodium hypochlorite in concd HCl and again 3% sodium sulfite (hot). They were washed throughly in distilled water and dried in air.

Sodium hydroxide used was of AnalaR grade. Methyl methacrylate mono-

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mer was purified by the usual method. The water used was triple-distilled. Mn(III) soln was prepared according to the method by Vogel.[3]

Procedure

Vinyl monomer, water, and purified fiber and substrate were placed in a reaction vessel (a Pyrex tube fitted with a $B_{24/29}$ socket carrying a $B_{24/29}$ cone with inlet and outlet tube); nitrogen was passed for 30 min. The vessel was placed in a thermostat to attend the required temperature. The oxidant [Mn^{+3}] was added, and the induction period noted. The reaction was carried out for 3 h. The polymerization was terminated by adding hydroquinone solution. The fibers were taken out, washed thoroughly in running water, dried in air, and refluxed in acetone for 8 h in a Soxhlet apparatus to remove the homopolymers. The fibers were repeatedly washed with distilled water and air-dried. The homopolymers were dried and weighed.

Percentage of grafting, conversion percentage, and graft efficiency were calculated as follows:

$$\text{graft efficiency} = \frac{\text{wt grafted sample} - \text{initial wt}}{\text{wt monomer}} \times 100$$

$$\%\ \text{of grafting} = \text{percentage of synthetic polymer in grafted copolymer}$$

$$\text{conversion}\ \% = \frac{\text{product}}{\text{monomer}} \times 100$$

Scanning Electron Microscopic Studies

Photograph of silver-coated samples before and after treatment of chemicals were taken with the help of a Cambridge-Stereoscan electron microscope. Samples were prepared by standard methods.

RESULTS AND DISCUSSIONS

Treatment with Aqueous NaOH at Different Concentrations

Treatment of Soxhleted fiber with aqueous NaOH (5, 10, 15, 20%) in the slack conditions were carried out at room temperature. There was no change in the structure of the fiber when the concn of NaOH was less than 10% at any length of time, but, as the concn was increased, an appreciable change in the internal structure was noticed. The development of an extensive and very fine crimp was found. At 20% NaOH, the result was very interesting, showing remarkable changes in the degree of crystallinity and orientation, and above this concn no further change in the structure was noticed.

Formation of crimp may be due to the internal stress developed as a result of nonuniform transverse swelling along the length of individual murga filaments. Crimp develops in order to neutralize the internal stresses. All natural long vegetable fibers are bound together by a cementing material, mostly thought to be lignin. The dimensional variability is enormous among the ultimates from the same fiber as well as those from other fibers. The nonswellable lignin contents are responsible for the nonuniform crimp,

and also the possibility of formation of crosslinkages with easily swellable amorphous regions of cellulosic components of the fiber. The higher lignin contents in leaf fibers resisted the internal stresses due to nonuniform swelling so that less crimp was observed; but when a subsequent chemical treatment removes lignin to a greater extent, a change in amorphous region to crystalline one is noticed, thus showing fine crimp. This is in affirmation to the view of Chakrabarty[1] in the case of jute.

For a further development in the study of the fiber properties, a maiden attempt was taken by incorporating vinyl monomers onto the vegetable fibers. Incorporation of vinyl monomers onto natural protein fibers as well as synthetic polymers have proved to be very much encouraging in introducing many additional desirable properties considering their end uses. Keeping in mind the cost of production and the end uses of the grafted vegetable fibers, few modifications, by grafting them with AN within a suitable experimental conditions, were attempted to compare their behavior towards bacterial growth, moth, acid, alkali, salt, temperatures, weathering, and fire resistance.

The Effect of the Monomer Variation on Grafting of the Murga Fiber

The graft copolymerization results are presented in Table I. A plot of percentage of grafting at different concentration of monomer was made.

The percent of grafting at different concentrations of monomer, metal ion, and substrate has been plotted (Fig. 1). The maximum graft yield is obtained at a monomer concentration of 1.1308*M*, after which the graph shows a decline. This is probably due to incompatibility of the monomer with the polymer; as such the polymer chains are easily available to the metal ion for termination. A fairly higher homopolymer content was obtained, with a low percentage of grafting in comparison with other protein fibers (a much greater percentage is obtained in the case of silk; the result has been sent for publication). The incorporation onto fiber matrix is probably difficult due to lignin coating.

Without metal ion, there is absolutely no grafting, which shows the indispensability of Mn(III) for the initiation of polymerization. A plot of % of grafting at different concentrations of metal ion at a fixed concentration of monomer (Fig. 2) shows there is no appreciable variation in the % of grafting at a higher concentration of grafting, which is inconclusive; but a termination is operative since the highest % (7.36) is much lower in comparison with the monomer variation.

The effect of substrate on graft % is plotted (Fig. 3). The graft percentage remains unchanged afterwards (EDTA = 2 mL). This can be attributed probably due to the termination by oxidant and mutual at higher concentration of substrate.

The graft efficiency calculation shows that lignocellulosic fiber responds to the incorporation of monomers onto its backbone less appreciably but certainly not negligibly. Lower conversation of monomer to polymer in case of grafted sample clearly indicates the higher grafting efficiency. More works are in progress to increase grafting % and efficiency.

TABLE I
Graft Copolymerization of Vinyl Monomer onto Murga Fibers initiated by Manganese(III)

Effect of monomer on grafting %[a] (see Fig. 1)

Expt no.	Monomer $\times 10^{-2}$	Conversion of monomer to polymer	% of grafting	Graft efficiency
1	15.08	2.0	3.41	3.24
2	37.7	11.0	9.08	10.2
3	75.4	25.7	11.00	12.3
4	113.08	26.9	14.32	15.7
5	150.8	25.4	9.0	10.7
6	188.5	21.4	5.2	7.4
7	226.2	20.2	4.2	6.1

Effect of Mn(III) on grafting %[b] (see Fig. 2)

Expt no.	Mn(III) $\times 10^{-2}M$	Conversion of monomer to polymer	% of grafting	Graft efficiency
1	0.35	—	1.68	1.66
2	1.75	0.32	2.44	02.7
3	3.5	8.0	11.0	12.3
4	7.0	41.68	5.70	7.8
5	10.5	57.08	6.38	8.2
6	17.5	60.93	7.38	8.7

Effect of EDTA on grafting %[c] (see Fig. 3)

Expt no.	EDTA $\times 10^{-4}$	Conversion of monomer to polymer	% of grafting	Graft efficiency
1	2.5	45.66	2.61	2.8
2	5.0	52.41	11.00	12.3
3	7.5	25.1	5.22	7.4
4	10.0	19.3	5.76	7.8
5	12.5	9.18	5.76	7.8
6	15.0	3.19	2.76	2.9

[a] [EDTA] = $5 \times 10^{-4}M$; Mn(III) = $3.5 \times 10^{-2}M$; murga = 0.088 g; temp = 50°C, time = 3 h.

[b] [Monomer] = $0.7539M$; [EDTA] = $5 \times 10^{-4}M$; murga = 0.088 g; temp = 50°C; time = 3 h.

[c] [monomer] = $0.7539M$; Mn(III) = $3.5 \times 10^{-2}M$; murga = 0.088 g; temp = 50°C; time = 3 h.

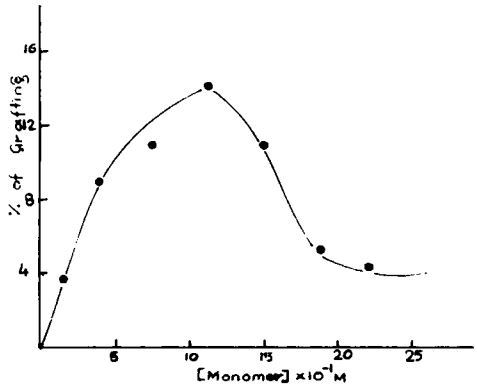

Fig. 1. Effect of monomer on grafting %: [EDTA] = $5 \times 10^{-4}M$; Mn(III) = $3.5 \times 10^{-2}M$; murga = 0.088 g; temp = 50°C; time = 3 h.

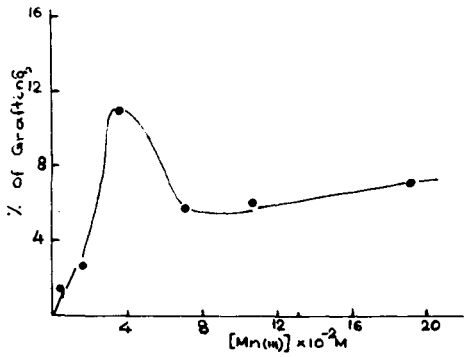

Fig. 2. Effect of Mn(III) on grafting %: [monomer] = 0.7539*M*; [EDTA] = $5 \times 10^{-4}M$; murga = 0.088 g; temp = 50°C; time = 3 h.

The surface structure of murga fibers both untreated and MMA-treated fibers was investigated by means of a scanning electron microscope. Two distinct morphological changes were observed between natural and grafted fibers. The grafted fibers showed clusting of polymers randomly throughout the fiber length. Secondly, some of these polymer clusters looked like small plates portuding out from the fibers. A definite incorporation of monomer on the walls of the ultimate cell were noticed.

CONCLUSION

Ligno cellulosic fiber (murga) has shown remarkable affinity for grafting. The results are encouraging. Further development in graft efficiency and % of grafting will be extremely useful, and full work in this respect is in progress.

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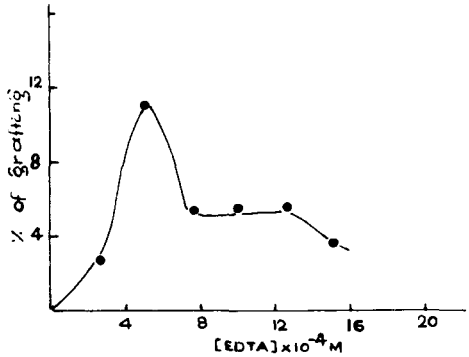

Fig. 3. Effect of EDTA on grafting %: [monomer] = 0.7539*M*; Mn(III) = $3.5 \times 10^{-2}M$; murga = 0.088 g; temp = 50°C; time = 3 h.

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