Acrylonitrile Monomer Grafting on Gray Cotton to Impart High Water Absorbency

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ABSTRACT: Acrylonitrile (AN) monomer was directly grafted onto gray cotton yarn using a $KMnO_4$ —HNO₃ redox system by a chemical initiation technique. The major factors affecting polymerization of AN, such as KMnO₄, nitric acid, and AN concentration as well as the reaction time and temperature of the polymerization, were studied. The water absorbency of grafted fibers depends strongly on their posttreatment. Saponification of AN-g-cotton with a hot sodium hydroxide solution developed superabsorbency. Absorbency with distilled water and a saline solution (0.9% NaCl) was studied. Grafting cellulose with AN using a KMnO₄/nitric acid redox system proceeds by a free-radical mechanism. Use of cotton in the gray form as a starting material rather than a bleached one helps in the better deposition of MnO_2 with subsequent acceleration of the grafting reaction, resulting in better whiteness of the grafted products. The enhancement in the whiteness index could be interpreted in terms of the oxidation of natural coloring matter, which happens to be the objective of the bleaching process. Grafting of AN onto gray cotton results in the elimination of several preparatory treatments including the conventional bleaching operation, ultimately reducing the water consumption as well as minimizing the unwanted effluent generation. The work has the potential of promoting cost-effective and environmental friendly technologies and techniques. © 1999 John Wiley & Sons, Inc. J Appl Polym Sci 72: 887-894, 1999

Keywords: gray cotton; acrylonitrile; grafting; infrared; superabsorbent; saline solution

INTRODUCTION

There has been considerable interest in recent years in the development of materials with the capacity for absorbing massive amounts of water. These polymers find potential use in disposable diapers, sanitary napkins, bandages, bed pads, etc. Most of the commercial uses of superabsorbents are concerned with blood, urine, and other fluids rather than with pure water.¹ A number of such materials have been prepared by grafting vinyl monomer onto starch,^{2–7} guar gum,⁸ and cellulose^{9–14} followed by alkaline hydrolysis to enhance water absorbency. Of the chemical initiators used for the synthesis, the Mn(IV) ion in the presence of an activator has proved to be an effective initiator.¹⁵

Huge quantities of loom waste are generated in textile mills during the weaving of gray cotton fabrics. The loom waste is a mixture of size (starch) and pieces of gray cotton fibers. If the loom waste is to be converted into superabsorbent polymers through a graft copolymerization reaction, then the grafting reaction has to be carried

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out on gray cotton, rather than on a bleached one. The present work was undertaken with a view to develop improved conditions for vinyl graft copolymerization onto gray cotton waste. A literature survey shows that there has been very little work done on grafting on gray cotton fibers.

EXPERIMENTAL

Materials

Gray cotton hank $(2/40^{\rm s} \text{ count})$ was used. KMnO₄ and HNO₃ were reagent grade. Acrylonitrile (AN) was supplied by S.D. Fine Chemicals Ltd. (Bombay, India) and was twice distilled before use. *N,N*-Dimethylformamide (S.D. Fine Chemicals Ltd.) was used as such without any further treatment. A nonionic wetting agent (NYNOL-N) was supplied by Rachana Chemicals (Bombay, India).

Treatment of Cotton Yarn with Potassium Permanganate

Unless otherwise indicated, the treatment of gray cotton yarn with KMnO_4 and the grafting of the so-treated yarn was carried out as described below: Two grams of gray cotton yarn was impregnated in an aqueous solution containing specific concentrations of KMnO_4 (0.005–0.075*N*) and 0.2% of a nonionic wetting agent. The treatment was carried out at 55°C for a period of 30 min at a material-to-liquor ratio of 1 : 100 with continuous shaking to avoid the heterogeneity in the MnO_2 deposition on the yarn. After this treatment, the yarn was thoroughly washed with distilled water and squeezed between two filter papers before immersing in the polymerization solution.

Bleaching of Gray Cotton Yarn

Gray cotton yarn was subjected to a standard scouring treatment and then bleached using H_2O_2 = 1.2% at pH 10 for 50 min at 90°C.

Grafting Procedure

The KMnO₄-treated cotton was placed into a reaction flask containing varying concentrations of AN (0.4-8.0 % w/v) and nitric acid (0.2-2.0*M*). The flask contents were kept in a thermostatic water bath at different temperatures $(30-80^{\circ}C)$ and for different reaction time intervals (10-60 min). After the desired reaction time was over, the sample was removed and washed with cold water several times. The sample was then squeezed and dried in an oven at 105°C for a period of 3 h and cooled over P_2O_5 in a desiccator. The homopolymer was extracted with dimethylformamide for a period of 24 h at 35°C.

Determination of MnO₂ Quantity onto Cotton Yarn

The amount of MnO_2 deposited onto the cotton yarn was determined by adding 10 mL of standard oxalic acid (0.1*N*) and 10 mL of a 2*N* H₂SO₄ solution to the cotton yarn treated with permanganate in a conical flask. The mixture was gently heated to above 60°C and then titrated against a KMnO₄ solution of 0.01*N* until a light pink color appeared due to unreacted potassium permangnate (self-indicator):

The amount of
$$\mathrm{MnO}_2$$
 deposited $= rac{V imes 0.1 imes 100}{W}$

where V is the volume (mL) of KMnO_4 equivalent to MnO_2 in the sample and W is the weight (g) of the sample used.

Estimation of Percentage of Graft Add-on

Gravimetric Method

The percentage of graft add-on was calculated on a dry-weight basis. This was determined by storing the heated material at 105°C for a period of 3 h in a weighing bottle over P_2O_5 at room temperature in a vacuum dessicator until a constant weight was obtained:

Percent graft add-on =
$$\frac{(W_2 - W_1)}{W_1} \times 100$$

where W_2 is dry weight of the grafted sample and W_1 is the dry weight of cotton yarn after the KMnO₄ treatment.

Nitrogen Estimation

The nitrogen content of the grafted sample was estimated by Kjeldahl's method¹⁶ and the percent grafting was determined as follows:

Graft add-on (%)

 $= 3.79 \times \text{Nitrogen content of the samples (N\%)}$

		Graft Add-on (%)		
$\begin{array}{c} \text{KMnO}_4 \\ \text{Concentration } (N) \end{array}$	$\begin{array}{c} Amount \ of \ MnO_2 \ Deposited \\ (meq/100 \ g \ Sample) \end{array}$	Gravimetric Method	Nitrogen Analysis	
0.005	3.24	12.16	11.98	
0.015	6.26	18.12	17.78	
0.025	12.06	24.84	24.26	
0.035	14.74	29.46	28.76	
0.045	16.68	30.12	29.86	
0.050	17.86	36.64	36.13	
0.075	17.94	36.18	35.76	

Table I Effect of KMnO₄ Concentration on Percent Graft Add-on of Gray Cotton Yarn

Reaction conditions for KMnO₄ treatment: weight of hank (gray): 2.000 \pm 0.015 g; temperature: 55°C; time: 30 min; nonionic wetting agent 0.2%; M : L ratio = 1 : 100

Reaction conditions for grafting: weight of hank (gray): 2.000 \pm 0.015 g; monomer (AN) = 6.4% w/v; HNO₃ = 0.8*M*; *M* : *L* ratio = 1 : 100; temperature: 60°C; time: 60 min.

Base Hydrolysis

Grafted fibers of about 2.0 g were introduced into 100 mL of a boiling NaOH solution (3%) with constant stirring. A graft copolymer was formed rapidly as a slurry and had a typical red-orange color, which then slowly faded to light yellow as the reaction neared completion. It was filtered while hot on a Buchner funnel and washed until pH 9 and then dried and ground to 20 mesh.

Infrared (IR) Confirmation

The grafting was confirmed by IR analysis. The IR spectra were recorded in a KBr phase using a JASCO FT/IR-5300 Model spectrophotometer employing the KBr palette technique of O'Connor et al.¹⁷

Water and Saline Absorbency (Filtration Method)

A dry sample (20-mesh size) of 0.1 g was immersed in 50 mL distilled water for 30 min and poured into a 40-mesh sieve of known weight. Water was allowed to drain for 1 h; then, the hydrated sample and the screen were weighed. Water absorbency was calculated as follows:

Absorbency, g H₂O/g sample =
$$\frac{(c-b) - a}{a}$$

where *a* is the weight of the dry sample; *b*, the weight of the sieve, and *c*, the weight of the swollen sample plus the weight of the sieve. A similar technique was used to study saline solution absorbency, where the dry sample was immersed in 50 mL of a saline solution (0.9% NaCl) for 30 min.

RESULTS AND DISCUSSION

Effect of KMnO₄ Concentration on the Amount of MnO₂ Deposited

Cotton hank was treated with different concentrations of the MnO_4 solution (0.005-0.075N) as described in the Experimental section. The cotton sample treated with $KMnO_4$ was monitored for MnO_2 deposition. Table I shows the relation between the amount of MnO_2 deposited onto the cotton sample as a function of $KMnO_4$ concentration. The data show that the MnO_2 deposit increases by increasing the $KMnO_4$ concentration up to 0.05N. Above this concentration, it was found that no marked increase in the MnO_2 deposition was observed and, hence, the range of the $KMnO_4$ concentration was restricted to 0.005-0.075N.

It is logical to assume that MnO_2 particles are deposited uniformly all over the cellulose located at certain sites, most preferably at the hydroxyl groups of cellulose. Once the accessible hydroxyl groups are occupied by MnO_2 particles, extra deposition of MnO_2 will be difficult, and if deposited at all, the particles would be easily removed during washing. This would explain why the amount of MnO_2 deposited onto the cotton sample remains constant at higher concentrations of $KMnO_4$, especially above 0.05N.

Tentative Mechanism of Grafting

It was shown above that when gray cotton yarn was treated with $KMnO_4$, a substantial amount of MnO_2 was deposited onto the yarn. Gray yarn

	Graft Add-on (%)		
HNO ₃ Concentration (M)	Gravimetric Method	Nitrogen Analysis	
0.2	22.42	21.86	
0.4	25.38	24.84	
0.6	30.12	29.62	
0.8	37.76	36.42	
1.0	32.62	31.82	
2.0	30.82	29.42	

Table II	Effect of	f HNO ₃	Conc	entrati	on on
Percent (Graft Add	l-on of	Gray	Cotton	Yarn

Reaction conditions: weight of hank (gray): 2.000 ± 0.015 g; $[MnO_2] = 17.86$ meq/100 g sample; monomer (AN) = 6.4%w/v; M : L ratio = 1 : 100; temperature: 60° C; time: 60 min.

turns brownish and then dark brownish depending on the concentration of $\rm KMnO_4$ used in the solution.

It has been reported¹⁸ that radical formation occurs during a polymerization initiated by potassium permanganate in the presence of an acid through the reduction of Mn(IV) to Mn(III) and/or Mn(II). Thus, during vinyl copolymerization on cellulose (Cell-OH) under the catalytic influence of potassium permanganate in the presence of an acid (HR), creation of a free-radical species occurs, which may be represented as follows:

$$Mn(IV) + HR \rightarrow Mn(III) + H^{+} + R^{\bullet}$$
 (1)

$$Mn(IV) + H_2O \rightarrow Mn(III) + H^+ + HO^{\bullet} \quad (2)$$

Once these free-radical species (\mathbf{R}^{\bullet}) are created, they produce a cellulose macroradical via direct abstraction of a hydrogen atom from the hydroxyl group of cellulose:

$$Cell-OH + R^{\bullet} \rightarrow Cell-O^{\bullet} + RH$$
(3)

Cellulose macroradicals may also be formed by direct attack of Mn(IV) or Mn(III) ions on the cellulose molecule via abstraction of a hydrogen atom:

$$Cell-OH + Mn(IV) \rightarrow Mn(III) + H^{+} + Cell-O^{\bullet}$$
(4)

$$Cell-OH + Mn(III) \rightarrow Mn(II) + H^{+} + Cell-O^{\bullet}$$
 (5)

In the presence of a vinyl monomer, the cellulose macroradical is added to the double bond of AN,

Table III	Effect of Monomer (AN)
Concentra	ation on Graft Add-on
of Gray C	otton Yarn

	Graft Add-on (%)		
Concentration of AN (% w/v)	Gravimetric Method	Nitrogen Analysis	
0.4	2.52	2.46	
1.6	4.37	4.12	
3.2	18.12	17.98	
4.8	28.98	27.74	
6.4	36.34	35.68	
8.0	33.43	32.86	

Reaction conditions: weight of hank (gray): 2.000 ± 0.015 g; $[MnO_2] = 17.86$ meq/100 g sample; $HNO_3 = 0.8M$; M : Lratio = 1 : 100; temperature: 60°C; time: 60 min.

resulting in covalent bond formation between the monomer and the cellulose with the creation of a free radical on the monomer, that is, a chain is initiated. Subsequent addition of a monomer molecule to the initiated chain propagates grafting onto the cellulose:

$$\begin{array}{cccc} Cell & -O' + CH_2 = \stackrel{H}{\underset{X}{\overset{C}{\overset{}}}} & \longrightarrow Cell - O - CH_2 - \stackrel{H}{\underset{X}{\overset{C}{\overset{}}}} & (6) \\ Cell & -O - CH_2 - \stackrel{H}{\underset{X}{\overset{C}{\overset{}}}} + CH_2 = \stackrel{H}{\underset{X}{\overset{C}{\overset{}}}} & \longrightarrow Cell - O - CH_2 - \stackrel{H}{\underset{X}{\overset{C}{\overset{}}}} & - \stackrel{H}{\underset{X}{\overset{}}} & (7) \\ & \xrightarrow{Monomer} & Cellulose Graft Copolymer \end{array}$$

Table IVEffect of PolymerizationTemperature on Percent Graft Add-onof Gray Cotton Yarn

	Graft Add-on (%)		
Polymerization Temperature (°C)	Gravimetric Method	Nitrogen Analysis	
30	22.12	21.64	
40	24.32	23.62	
50	30.82	29.82	
60	37.74	36.86	
70	36.72	35.94	
80	35.75	35.12	

Reaction conditions: weight of hank (gray): 2.000 ± 0.015 g; $[MnO_2] = 17.86$ meq/100 g sample; $HNO_3 = 0.8M$; monomer (AN) = 6.4% w/v; M : L ratio = 1 : 100; time: 60 min.

	Graft	Graft Add-on (%)		
Polymerization Time (min)	Gravimetric Method	Nitrogen Analysis		
10	18.76	17.88		
20	24.46	23.82		
30	27.56	26.88		
40	31.82	30.42		
50	34.42	33.68		
60	37.76	36.33		

Table VEffect of Polymerization Time onPercent Graft Add-on of Gray Cotton Yarn

Reaction conditions: weight of hank (gray): 2.000 \pm 0.015 g; [MnO₂] = 17.86 meq/100 g sample; HNO₃ = 0.8*M*; monomer (AN) = 6.4% w/v; *M* : *L* ratio = 1 : 100; temperature: 60°C.

Effect of MnO₂ Deposited on the Percent Graft Add-on

Table I gives data on the effect of the $\rm KMnO_4$ concentration on the percent graft add-on. It can be seen that the graft add-on increases significantly by increasing the $\rm KMnO_4$ concentration up to 0.05N. Above this concentration, the graft add-on almost levels off. The enhancement of grafting by increasing the $\rm KMnO_4$ concentration signifies the necessity of a certain amount of $\rm MnO_2$ deposition on the gray cotton yarn to produce, ultimately, cellulose macroradicals capable of initiating grafting. On the other hand, the leveling of grafting after a certain $\rm KMnO_4$ concentration could be associated with

(a) A lower rate of monomer diffusion from the aqueous phase to the fiber phase by virtue

of an excess amount of deposition of colloidal particles of MnO_2 ,

(b) A higher production of inhibiting oxygen at a higher MnO_2 concentration due to the side reaction as shown by the following equation:

$$MnO_2 + 2H^+ \rightarrow Mn(II) + H_2O + O \qquad (8)$$

(c) A faster rate of chain termination particularly via bimolecular collision between two chain radicals, because of the abundance of primary free-radical species.

Effect of Concentration of HNO₃

Table II shows the effect of the concentration of nitric acid on the percent graft add-on. It was found that the percent graft add-on increases with increasing concentration of an acid, attaining a maximum at 0.8*M*. The fall in the graft add-on above this concentration could be attributed to various conditions, such as:

- (a) The coagulation of a colloidal homopolymer in the solution and in the fiber structure increases at lower pH. This retards the diffusion of both the monomer and the initiator into the fiber phase.
- (b) Side reaction, given in (8), increases at a higher acid concentration. Thus, besides consuming some MnO_2 (required for the production of initiating species), it also leads to a constant production of inhibiting oxygen.

Sample No.	$\underset{(N)}{\mathrm{KMnO}_4}$	HNO ₃ (M)	Wetting Agent (%)	Monomer (AN) Concentration (% w/v)	Graft Add-on (%)	Whiteness Index (Hunter Whiteness)
1^{a}		_	_	Untreated	0.0	29.123
2	0.05	0.8	0.2	0	0.0	70.421
3	0.05	0.8	0.2	1.6	4.37	40.308
4	0.05	0.8	0.2	3.2	18.12	38.589
5	0.05	0.8	0.2	4.8	28.98	35.764
6	0.05	0.8	0.2	6.4	36.34	32.266
7	_		_	3.2	—	45.120
8	_		_	6.4	—	43.357
9^{b}		—		_	_	76.585

Table VI Effect of Solution Composition on the Whiteness Index and Percent Graft Add-on of Gray and Eleached Cotton Yarn

^a Untreated cotton yarn (gray).

^b Bleached cotton yarn.

Substrate		Graft Add-on (%)		
	MnO_2 (meq/100 g cellulose)	Gravimetric Method	Nitrogen Analysis	
Gray	17.86	36.76	36.33	
Bleached ^a	11.56	27.45	26.86	

Table VII Comparison of AN Graft Add-on of Gray Cotton Yarn with Bleached Cotton Yarn

Reaction conditions for KMnO₄ treatment: KMnO₄ = 0.05N; wetting agent: 0.2%; temperature: 55°C; time: 30 min; M : L ratio = 1 : 100. Reaction conditions for grafting: monomer (AN) = 6.4% w/v; HNO₃ = 0.8M; M : L ratio = 1 : 100; temperature: 60°C; time: 60 min.

^a Bleached cotton yarn.

Effect of Monomer Concentration

Table III shows the effect of AN concentration on the percent graft add-on. The AN concentration used ranged from 0.4-8.0% w/v. As the concentration of AN increases, the percent graft add-on also increases. This could have been associated with the higher availability of monomer molecules around the cellulose macroradicals. Cellulose macroradicals are relatively immobile, and for grafting to occur, the monomer molecules need to be in close proximity to them.

Effect of Reaction Temperature

Table IV shows the effect of temperature $(30-80^{\circ}C)$ on the graft add-on. It is clear that the percent graft add-on increases by increasing the polymerization temperature from 30 to 60°C. This favorable effect of higher temperature on grafting could be associated with the following:

- (a) Better decomposition of the redox system, giving rise to more free radicals;
- (b) Greater swellability of cotton cellulose;
- (c) Increased mobility of the monomer molecules;
- (d) Higher diffusion from the aqueous phase to the fiber phase; and
- (e) Higher rate of initiation and propagation of the graft chain.

Effect of Reaction Time

Table V presents the relationship between the reaction time and graft add-on within a given time range (10-60 min.). It is evident from the results that at 60° C graft add-on continues to increase with increase in time up to 60 min. This can be explained in terms of a faster rate of monomer depletion and a shortage of cellulose macro-

radiacals, as well as structural changes in the cellulose as the grafting reaction proceeds at the highest temperature.

Optimal Condition of Grafting

About 37.76% graft add-on may be obtained by maintaining the following reaction conditions: $KMnO_4 = 0.05N$; $HNO_3 = 0.8M$; monomer (AN) = 6.4% w/v; M : L ratio = 1 : 100; temperature = 60°C; and time = 60 min.

Whiteness Index (Hunter Whiteness) of Grafted Sample

An interesting feature of the present work (Table VI) is that the whiteness index is increased from 29.123 to 70.421 by merely immersing gray cotton yarn in the blank grafting bath containing MnO_2



Figure 1 Water and saline solution absorbency of hydrolyzed poly(AN-*g*-cellulose).



Figure 2 IR spectra of (A) gray cotton and (B) grafted (cell-g-AN) gray cotton.

in the absence of the AN monomer. Some improvement in the whiteness index of gray cotton is observed when it was given the same treatment but in the presence of AN. The first and last treatments bring about a graft add-on of 4.37 and 36.34% along with a whiteness index of 40.308 and 32.266, respectively. Sample 9 is comparable with sample 2. The enhancement in the whiteness index by treating the gray cotton containing MnO_2 deposits could be interpreted in terms of the oxidation (bleaching) of natural coloring matter present in the substrate.

Comparative Behavior of Gray Cotton Yarn and Bleached Cotton Yarn Towards Grafting

Table VII shows that the comparison between percent graft add-on onto the gray and bleached cotton gives interesting results. MnO_2 deposition on gray cotton (17.86 meq/100 g cellulose) is higher than on bleached cotton yarn (11.56 meq/ 100 g cellulose). This may be because the gray cotton yarn facilitates the deposition of MnO_2 . The presence of natural starchy matter in the gray cotton yarn seems to be responsible for this observation in the presence of wetting agents. The wetting agents also neutralize the negative effect of an oily substance present in the gray substrate. The desizing and scouring of gray cotton yarn removes oily substances and starch during the bleaching process. The wetting agent, in the absence of starch, therefore, enhances the deposition of MnO_2 only marginally. Gray cotton yarn turned dark brown after $KMnO_4$ treatment, while only a slight coloration could be observed in the case of bleached cotton. Subsequently, the percent graft add-on is higher by 30 percentage points in the case of gray cotton yarn (27.45%).

Water Absorbency and Saline Absorbency

An absorbency test with distilled water and a saline solution (0.9% NaCl) was carried out for selected samples and the results are summarized in Figure 1. The results show that the absorbency could be obtained up to 60 g H_2O/g of the fiber sample and 36 g saline solution/g fiber sample for the maximum graft yield (37.74 %) of AN-g-cellulose.

This observation is in agreement with the results reported by Chen et al.¹⁹ The fluid absorbency of superabsorbents is influenced by the concentration of the electrolytes present in the fluid. A water-absorbent system consists of a constant quantity of a polymer network with a variable quantity of an aqueous component. The osmotic pressure attributable to the polymer network is the driving force for water absorption and, consequently, the swelling of the polymeric network. At the swelling equilibrium, the chemical potential of water in the polymer equals to that of the water surrounding the polymer. Addition of a salt (NaCl) to the polymer solution leads to network contraction as indicated by the lowering of the viscosity and the chemical potential of the water surrounding a polyelectrolyte polymer. Therefore, absorbent polyelectrolyte polymers cannot imbibe as much salt water as pure water alone.

IR Spectra

The IR spectra of grafted cotton cellulose are given in Figure 2. Based on general consider-

ations for the assignment of variable absorption bands of cotton cellulose, it was observed that the spectrum of grafted cellulose shows a new band at 2243 cm^{-1} , which is the characteristic C=N absorption band. This testified to the existence of the grafting reaction.

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