Homogeneous Graft Copolymerization of Acrylonitrile onto High α-Cellulose in a Dimethyl Acetamide and Lithium Chloride Solvent System

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ABSTRACT: Homogeneous graft copolymerization of acrylonitrile (AN) monomer onto high α-cellulose was investigated in a lithium chloride/*N*,*N*-dimethyl acetamide (DMAc/LiCl) solvent system. Benzoyl peroxide (BPO) and azobisisobutyronitrile (AIBN) were used as radical initiators. By varying temperature, time, and monomer concentrations in grafting reactions, the optimum conditions for both initiator systems were fixed. The graft yield for the AN–BPO system was higher than that for the AN–AIBN system. The optimum conditions of reactions were at temperatures of 70 and 60°C with initiator concentrations of 0.4% (0.36 mmol) and 2% (1.24 mmol) for the AN–AIBN and AN–BPO systems, respectively, at a monomer concentration of 5% (14.1 mmol) solution. The number of grafts per cellulose chain was in the range from 2.2 to 1.1 for AN–BPO and 0.5 to 2.1 for the AN–AIBN system. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 89: 630–637, 2003

Key words: initiators; graft copolymers; infrared spectroscopy; membranes; α-cellulose

INTRODUCTION

The modification of cellulose and its derivatives by graft copolymerization represents a useful way to improve the quality of the natural material and to extend the range of their uses. Many workers[1–12] have carried out graft copolymerization of different vinyl monomers onto cellulose by a heterogeneous reaction system. In such a system, it is generally observed that grafting takes place primarily in the amorphous region of the cellulose, leaving the crystalline part ungrafted, and as such the grafting yield is very low.[13,14] Because of inadequate grafting, the grafted products always exhibit differences in composition and structure.[15–18]

However, with the development of nonaqueous solvent systems, the cellulose can be dissolved molecularly and, as such, the molecular mass of the cellulose takes part during chemical reaction/modification. The nonaqueous solvents for cellulose consist of a variety of binary and ternary solvent systems, in which cellulose can be dissolved without degradation and formation of any derivative. Systems such as dimethyl sulfoxide/paraformaldehyde (DMSO/PF), *N*,*N*-dimethyl formamide/nitrous oxide (DMF/N_2O_4), and *N*,*N*′-dimethylacetamide/lithium chloride (DMAc/LiCl) are some of the nondegrading, nonaqueous solvent systems from which cellulose can be isolated in pure form after dissolution.[19–21] Therefore, grafting of cellulose in such homogeneous systems possesses no complexities.[22–24]

During the past few years, work on homogeneous grafting of vinyl monomers onto cellulose and some cellulose derivatives dissolved in nondegrading solvent systems such as DMSO/PF[24–27] and DMSO/PhMe[28,29] have been tried using different initiators [e.g., ammonium persulfate (APS), potassium persulfate (KPS), azobisisobutyronitrile (AIBN), and benzoyl peroxide (BPO)] but so far, work on homogeneous graft copolymerization of high α-cellulose dissolved in a DMAc/LiCl solvent system has not been investigated. It is reported that cellulose can be dissolved in a high concentration up to 16% in solvent systems such as DMAc/LiCl and *N*-methyl-2,2-pyrolidine/lithium chloride (NMP/LiCl) without degradation of the polymer chain.[30]

In the present study we describe the homogeneous graft copolymerization of acrylonitrile (AN) onto high α-cellulose in a DMAc/LiCl solvent system. AIBN and BPO were used as initiators. The effect of varying reaction time and temperature and concentrations of initiators and monomer were studied to optimize the conditions under which grafting would occur most effectively. The grafted products obtained under optimum conditions were analyzed by infrared spectroscopy and their molecular weights and the number of grafts per cellulose backbone were determined.

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TABLE I
Graft Copolymerization of AN onto Cellulose in BPO System for 3 h at Different Reaction Conditions[a]

Temperature	Total conversion of monomer to polymer (%)				Graft yield (%)				Grafting efficiency (%)			
(°C)	a	b	c	d	a	b	c	d	a	b	c	d
30	0.39	0.46	0.40	0.36	1.67	3.00	2.67	02.00	74.53	81.82	74.89	72.17
40	1.84	2.40	2.16	1.81	7.67	13.33	10.33	10.00	67.23	70.69	69.54	64.51
50	2.68	3.36	2.86	2.59	13.00	18.00	15.33	13.67	62.00	66.67	65.23	61.43
60	4.21	5.54	4.98	4.15	25.33	27.67	26.00	22.33	57.86	61.94	58.62	60.20
70	4.88	5.91	5.56	4.65	27.00	32.00	31.00	25.33	65.41	67.13	64.57	62.12
80	2.65	3.47	3.23	2.62	18.33	21.00	16.00	12.00	70.12	75.00	71.23	68.19

[a] Concentration of BPO: a, 1% (0.62 mmol); b, 2% (1.24 mmol); c, 3% (1.86 mmol); d, 4% (2.48 mmol).

EXPERIMENTAL

Materials

High α-cellulose pulp extracted from *Hibiscus cannabinus* containing 94.40% α-cellulose and 0.04% ash was taken for this study.[31] The pulp was dried to less than 5% moisture before use. DMAc, LiCl, AN, BPO, AIBN, potassium per-iodate (KIO_4), and hydroquinone were analytical-grade chemicals from BDH, India, and were used without further purification.

Methods

Dissolution of cellulose

Powdered high α-cellulose (10 g, 61.7 mmol based on glucose unit) was added to 500 mL (5380 mmol) of DMAc in a round-bottom flask, fitted with a short path condenser. The flask was then heated to a temperature of 150°C for 26 min. Then 25 g (589.6 mmol) of LiCl was added and the system was again heated to 165°C for 10 min. The system was then cooled to room temperature and stirred overnight for complete dissolution.[21] The clear solution obtained was 2% by weight and used for further work.

Grafting

All the polymerization reactions were carried out in nitrogen atmosphere. A three-neck 250-mL round-bottom flask fitted with a temperature-controlled water bath was used for the reactions. To the reaction flask containing 15 mL 2% (1.85 mmol) cellulose solution and 15 mL each of BPO and AIBN of concentrations varying from 1 to 4% (0.62–2.48 mmol) and 0.2–0.8% (0.18–0.72 mmol), respectively, were added. Purified nitrogen gas was bubbled through the reaction mixture for 30 min at 20°C. Varying amounts of the monomer in the range of 2–7% (5.7–19.8 mmol) were added and the polymerization reactions were carried out at different temperatures between 30 and 80°C for 2–7 h. The reactions were terminated by the addition of hydroquinone.[24] The polymerization mixture was poured into cold distilled water with vigorous stirring. The crude grafted products were Soxhlet extracted with DMF for 24 h to remove any adherent homopolymer. The extracted cellulose was then dried to a constant weight and kept in a desiccator over P_2O_5.

The graft yield, total conversion of monomer to polymer, grafting efficiency, and number of graft per cellulose chain were calculated on oven dry weight of the cellulose from the increase in weight after grafting by using the following relations:[32]

$$\text{Graft yield, GY(\%)} = \frac{C - A}{A} \times 100 \qquad (1)$$

TABLE II
Graft Copolymerization of AN onto Cellulose in BPO System for 5 h at Different Reaction Conditions[a]

Temperature	Total conversion of monomer to polymer (%)				Graft yield (%)				Grafting efficiency (%)			
(°C)	a	b	c	d	a	b	c	d	a	b	c	d
30	0.87	1.0	0.65	0.62	8.67	10.67	6.67	6.00	74.3	82.05	76.92	72.00
40	2.01	2.01	1.81	1.78	19.67	20.33	17.67	16.67	72.84	75.31	72.60	69.49
50	2.43	2.63	2.26	2.13	21.67	24.67	20.33	23.00	66.33	69.81	67.03	62.73
60	4.56	4.98	4.42	4.19	38.00	44.33	36.67	35.33	61.96	66.17	61.80	58.60
70	4.98	5.83	5.28	4.83	47.00	58.33	47.33	43.00	70.20	74.46	67.70	66.10
80	2.85	3.59	2.43	2.31	27.67	37.67	23.67	22.00	72.20	77.93	72.49	71.00

[a] Concentration of BPO: a, 1% (0.62 mmol); b, 2% (1.24 mmol); c, 3% (1.86 mmol); d, 4% (2.48 mmol).

TABLE III
Graft Copolymerization of AN onto Cellulose in AIBN System for 3 h at Different Reaction Conditions[a]

Temperature (°C)	Total conversion of monomer to polymer (%)				Graft yield (%)				Grafting efficiency (%)			
	a	b	c	d	a	b	c	d	a	b	c	d
30	10.55	14.14	22.29	23.68	3.30	8.33	4.33	3.33	2.32	2.94	1.37	1.05
40	13.97	17.73	26.93	27.32	10.00	11.67	11.00	6.67	5.31	3.64	3.39	1.82
50	22.30	29.94	35.91	39.49	13.38	16.00	15.00	11.67	4.46	3.72	3.57	2.19
60	39.13	41.61	58.82	61.61	17.33	23.00	16.00	13.33	3.29	3.46	2.53	1.60
70	48.52	52.14	67.80	69.81	14.67	18.00	12.00	11.00	2.24	2.31	1.64	1.17
80	54.62	60.80	78.33	79.64	5.67	8.67	6.67	5.00	0.77	1.00	0.79	0.46

[a] Concentration of AIBN: a, 0.2% (0.18 mmol); b, 0.4% (0.36 mmol); c, 0.6% (0.54 mmol); d, 0.8% (0.72 mmol).

Total conversion of monomer to polymer,

$$\mathrm{TC}(\%) = \frac{B - A}{D} \times 100 \quad (2)$$

$$\text{Grafting efficiency, GE}(\%) = \frac{C - A}{B - A} \times 100 \quad (3)$$

No. of grafts per cellulose chain

$$= \frac{\text{Molecular weight of cellulose}}{\text{Molecular weight of grafted PAN}} \times \frac{\mathrm{GY}}{100} \quad (4)$$

where A is the weight in grams of the original cellulose taken for the reaction, B is the weight in grams of the grafted cellulose, C is the weight in grams of the grafted products after extraction, and D is the weight in grams of the monomer charged.

IR studies

IR spectra of the grafted and ungrafted cellulose samples were recorded on a Perkin–Elmer spectrometer (Model 580 B, Perkin Elmer Cetus Instruments, Norwalk, CT) using KBr disk technique, in the range 4000–400 cm^{-1}, with a resolution of 2 cm^{-1} using five scans per sample.

Determination of molecular weights of α-cellulose and grafted PAN

The molecular weights (M_v) of α-cellulose and grafted PAN were determined viscosimetrically by using a Ubbelohde dilution viscometer at 30°C. The M_v of α-cellulose was calculated after measuring intrinsic viscosity $[\eta]$ of cellulose solution using the following relation[33]:

$$[\eta]_{\text{DMSO-PF}} = K \times M_n^{\alpha} \quad (5)$$

where the values of K and α were taken as 15.85×10^{-4} and 0.625, respectively, at 30°C. The M_v of cellulose was calculated at 19×10^4.

For determining the M_v of PAN, the grafted cellulose samples were first oxidized[34] by immersing in 50 mL aqueous saturated $\mathrm{KIO_4}$ solution at room temperature for a period of 6 h. To this mixture was then added 20 mL of 2.5M NaOH and the reaction was allowed to continue for 24 h. After this period, the remaining residue was recovered by filtration and washed with distilled water. The whole filtrate was then taken and methanol was added to it until the complete precipitation of PAN was obtained. The PAN was then recovered by filtration and dried. The dried PAN thus obtained was dissolved in DMF and used for viscosity measurement.

TABLE IV
Graft Copolymerization of AN onto Cellulose in AIBN System for 5 h at Different Reaction Conditions[a]

Temperature (°C)	Total conversion of monomer to polymer (%)				Graft yield (%)				Grafting efficiency (%)			
	a	b	c	d	a	b	c	d	a	b	c	d
30	14.55	16.41	16.93	24.14	3.33	10.00	4.33	3.33	2.12	2.16	3.17	1.02
40	16.52	20.12	26.85	29.73	6.67	13.33	8.67	6.67	2.98	2.74	4.00	1.67
50	25.70	38.08	44.94	49.94	14.67	23.33	14.33	11.67	4.24	3.13	2.37	1.74
60	43.03	51.70	58.97	61.61	33.33	36.67	18.33	13.67	2.32	2.70	2.31	1.64
70	49.85	57.27	64.34	72.14	13.33	20.00	16.33	12.00	1.98	1.64	1.88	1.23
80	60.68	67.18	69.63	80.80	10.00	16.67	12.67	10.00	1.23	1.04	1.35	0.92

[a] Concentration of AIBN: a, 0.2% (0.18 mmol); b, 0.4% (0.36 mmol); c, 0.6% (0.54 mmol); d, 0.8% (0.72 mmol).

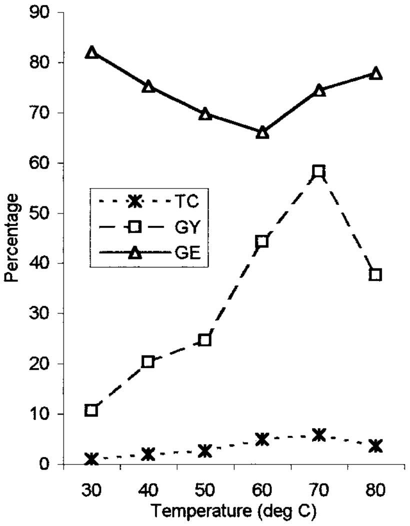


Figure 1 Effect of polymerization temperature on grafting of AN with BPO: grafting time, 5 h; cellulose, 2% (1.85 mmol); AN, 14.1 mmol; BPO, 1.24 mmol.

Intrinsic viscosities [η] (in mL g^{-1}) of the isolated PAN were measured at 30°C. The viscosity-average molecular weight was calculated by the following equation[35]:

$$[\eta]_{\mathrm{DMF}} = 20.9 \times 10^{-3} M^{0.75} \quad (6)$$

RESULTS AND DISCUSSION

Effect of temperature

The graft copolymerzation of AN onto high α-cellulose was carried out at different temperatures ranging from 30 to 80°C with a variation of 10°C for each reaction. The data on weight gain with respect to reaction temperature for AN–BPO and AN–AIBN systems are shown in Tables I and II and III and IV for 3 and 5 h reaction periods, respectively. Figures 1 and 2 represent the effective temperature at a fixed concentration of BPO and cellulose in 5 and 3 h, respectively. At both reaction times, GY and TC were maximum at 70°C, which was the most effective temperature for the AN–BPO system. However, GE decreased as the temperature increased to 60°C and then gradually tended to increase. Figures 3 and 4 show the temperature dependency of these three parameters for the

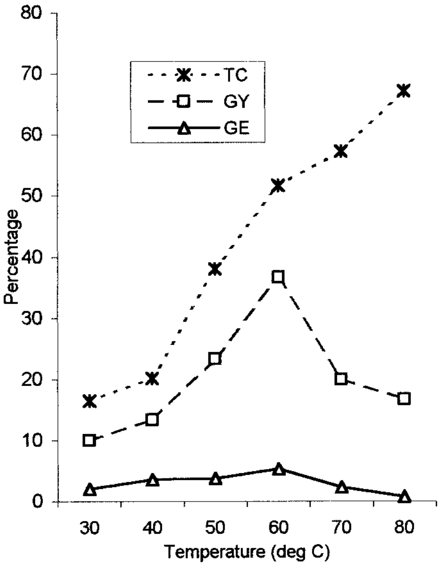


Figure 3 Effect of polymerization temperature on grafting of AN with AIBN: grafting time, 5 h; cellulose, 2% (1.85 mmol); AN, 14.1 mmol; AIBN, 0.36 mmol.

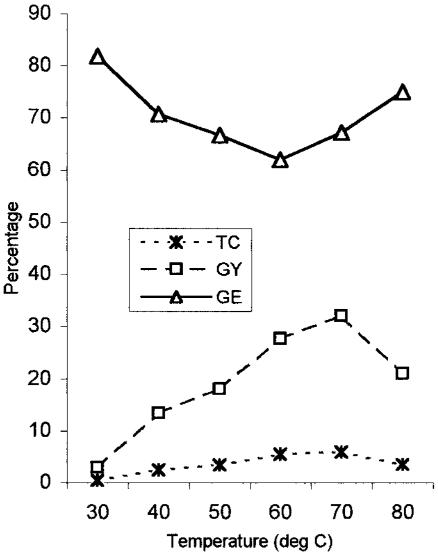


Figure 2 Effect of polymerization temperature on grafting of AN with BPO: grafting time, 3 h; cellulose, 2% (1.85 mmol); AN, 14.1 mmol; BPO, 1.24 mmol.

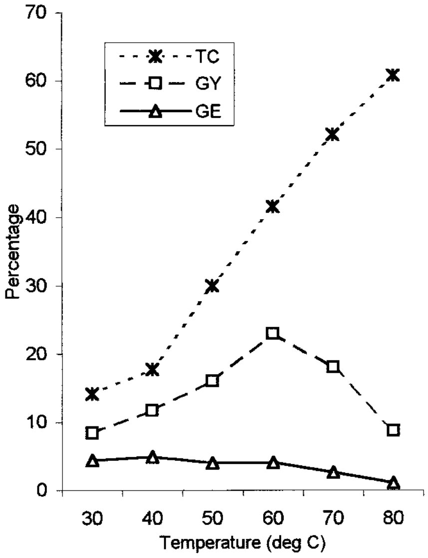
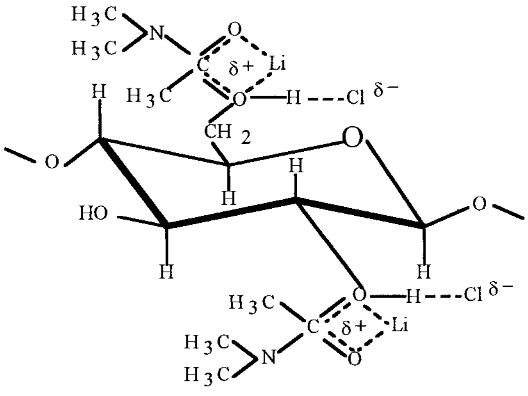


Figure 4 Effect of polymerization temperature on grafting of AN with AIBN: grafting time, 3 h; cellulose, 2% (1.85 mmol); AN, 14.1 mmol; AIBN, 0.36 mmol.



Scheme 1

AN–AIBN grafting system in 5 and 3 h respectively. In this monomer–initiator system, TC was higher than either GY or GE. TC increased as the temperature was raised. GY and GE were maximum at 60°C, after which they decreased gradually with the increase of temperature. GE for the AN–AIBN system was maximum at 8%. However, grafting of cellulose was reported to be less effective with AN in an AIBN/PF–DMSO solvent system.[24,26] Possibly this was attributable to the solvent system. In the DMAc/LiCl solvent system, the primary and to some extent the secondary hydroxyl groups of cellulose were solvated, with the possible mechanism of interaction with cellulose as shown in Scheme 1.[36–40]

Effect of initiator concentration

Grafting reactions were carried out by varying the BPO concentration from 1 to 4% (0.62–2.48 mmol). Figure 5 indicates the effect of BPO concentrations in the 5-h reaction time, keeping all other conditions of the reactions constant. With the increase of BPO concentration up to 4% (2.48 mmol), it was observed that at 2% (1.24 mmol) GY and TC increased simultaneously and then decreased gradually, whereas GE remained almost constant. At a high concentration of BPO, the viscosity of the system decreased because of degradation of the cellulose backbone.[25] The decrease of GY at high concentrations of BPO might be attributable to the gradual termination of grafting on primary hydroxyls of cellulose macroradicals. On the other hand, when AIBN concentrations were varied from 0.2 to 0.8% (0.18–0.72 mmol) (Fig. 6), the most effective concentration of AIBN was 0.4% (0.36 mmol). With the increase of AIBN concentrations, TC increased, whereas GY and GE were maximum at 0.4% (0.36 mmol) and then gradually decreased.

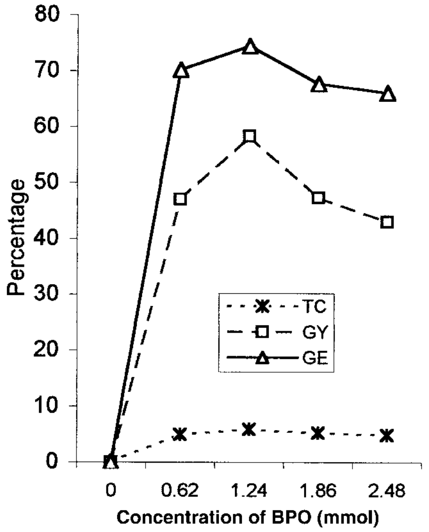


Figure 5 Effect of initiator concentration on grafting of AN with BPO: grafting time, 5 h; cellulose, 2% (1.85 mmol); AN, 14.1 mmol; temperature, 70°C.

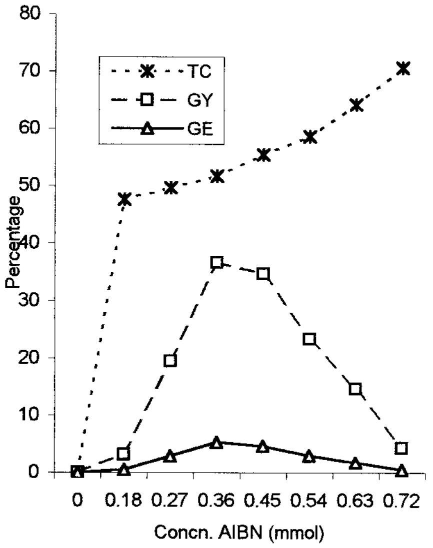


Figure 6 Effect of initiator concentration on grafting of AN with AIBN: grafting time, 5 h; cellulose, 2% (1.85 mmol); AN, 14.1 mmol; temperature, 60°C.

Effect of monomer concentration

The concentration of AN was varied in the range 2–7% (5.7–19.8 mmol). Figure 7 shows GE, GY, and TC of the grafted products in an AN–BPO system. TC and GY were maximum at 5% (14.1 mmol) and then decreased gradually. Similarly, in the AN–AIBN system, the most effective concentration of AN was at 5% (14.1 mmol) with respect to TC, GY, and GE (Fig. 8).

Effect of time

Figure 9 and 10 show the effect of polymerization reaction time on grafting in the AN–BPO and AN–AIBN systems, respectively. Maximum GY was obtained at the 5-h reaction in both systems. In the AN–BPO system, when grafting time was increased,

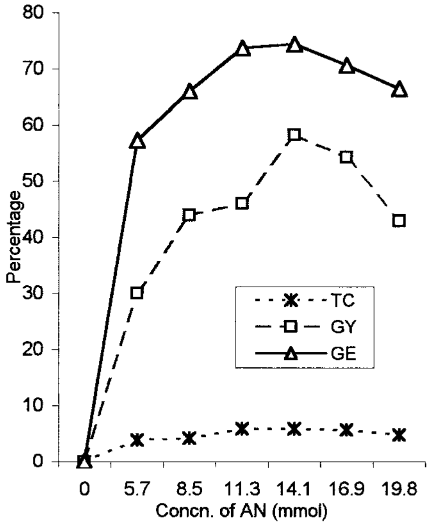


Figure 7 Effect of monomer concentration on the grafting of AN with BPO: grafting time, 5 h; cellulose, 2% (1.85 mmol); BPO, 1.24 mmol; temperature, 70°C.

TC, GY, and GE increased up to 5h, after which it remained unchanged. For the AN–AIBN system, however, TC increased with the increase of time, whereas GY was maximum at 5 h and then dropped.

IR studies

IR spectra (Fig. 11) of the ungrafted regenerated cellulose and grafted cellulose were recorded. The grafted products [Fig. 11(b)] showed a characteristic band at 2231 cm^{-1}, peculiar to the nitrile group C≡N, which was absent in the regenerated α-cellulose [Fig 11(a)], thereby indicating the formation of PAN-grafted cellulose.[41]

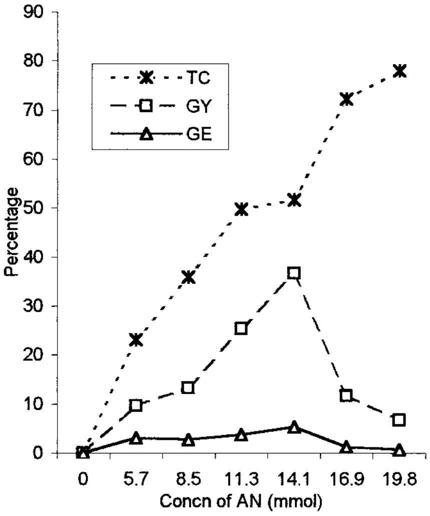


Figure 8 Effect of monomer concentration on grafting of AN with AIBN: grafting time, 5 h; cellulose, 2% (1.85 mmol); AIBN, 0.36 mmol; temperature, 60°C.

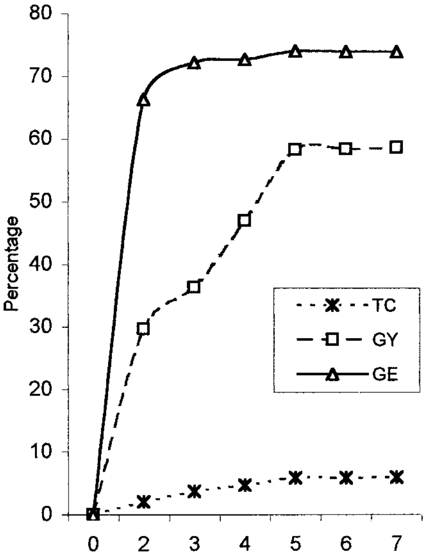


Figure 9 Effect of polymerization time on grafting of AN with BPO: cellulose, 2% (1.85 mmol); AN, 14.1 mmol; BPO, 1.24 mmol; temperature, 70°C.

Effect of grafting on molecular weights

The molecular weights of PAN extracted from grafted samples prepared under different temperatures at the 5-h reaction were determined and are reported in Tables V and VI for both initiator systems.

The molecular weights of extracted PAN increased in the AN–BPO system with an increase in temperature of the reaction, whereas the number of grafts decreased (Table V). The GY and M_v increased with temperature, whereas the number of grafts tended to

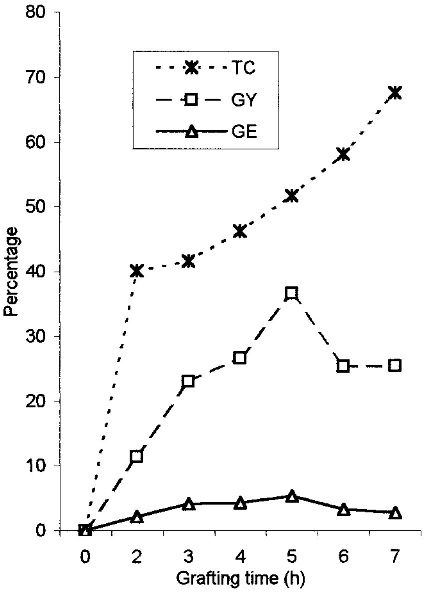


Figure 10 Effect of grafting time on grafting of AN with AIBN: cellulose, 2% (1.85 mmol); AN, 14.1 mmol; AIBN, 0.36 mmol; temperature, 60°C.

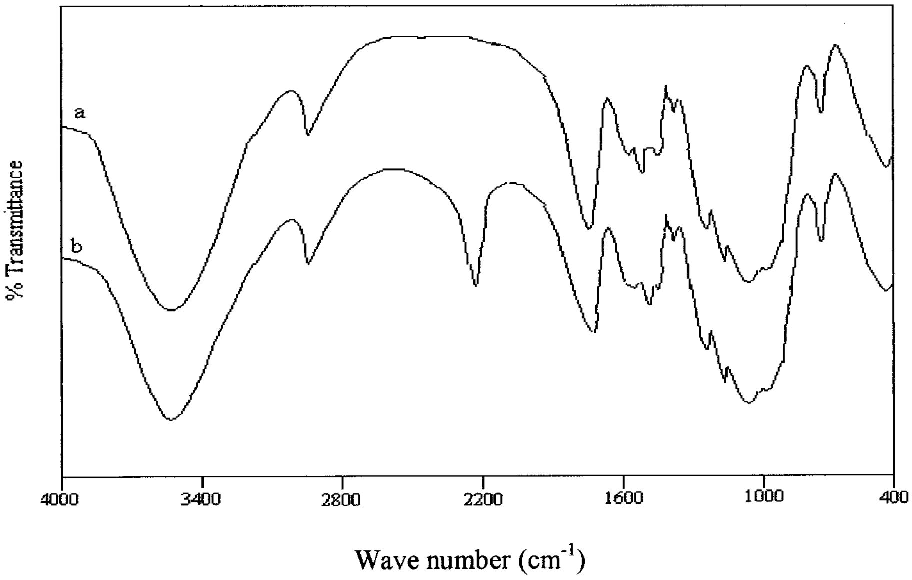


Figure 11 IR spectra of (a) regenerated high α-cellulose; (b) AN-grafted cellulose.

decrease.[25] The molecular weight of some PAN samples in the AN–AIBN system are tabulated in Table VI. In this case, the molecular weights of grafted PAN decreased with an increase in temperature, whereas the number of grafts per cellulose chain increased up to 60°C and then remained constant. This might be attributable to the reduced number of chains transferring from the PAN homopolymer to the cellulose backbone in this monomer–initiator system.

TABLE V
Graft Yields, M_v and Number of Grafts with Varying Temperatures of Reactions in AN-BPO System[a]

Temperature	Graft yield	M_v of grafts[b] ($\times 10^{-4}$)	Number of grafts
30	10.67	0.90	2.2
40	20.33	1.93	2.0
50	24.67	2.51	1.9
60	44.33	4.86	1.7
70	58.33	7.01	1.6
80	37.67	6.55	1.1

[a] Cellulose, 2% (1.85 mmol); AN, 5% (14.1 mmol); BPO, 2% (1.24 mmol); time, 5 h.
[b] M_v of cellulose = 19×10^4.

TABLE VI
Graft yields, M_v and number of grafts with varying temperatures of reactions in AN-AIBN system[a]

Temperature	Graft yield	M_v of grafts[b] ($\times 10^{-4}$)	Number of grafts
30	10.00	3.90	0.5
40	13.33	3.50	0.7
50	23.33	3.30	1.3
60	36.67	3.20	2.1
70	20.00	2.86	1.3
80	16.67	2.50	1.3

[a] Cellulose, 2% (1.85 mmol); AN, 5% (14.1 mmol); AIBN, 0.4% (0.36 mmol); time, 5 h.
[b] M_v of cellulose = 19×10^4.

CONCLUSIONS

Homogeneous graft copolymerization of AN onto high α-cellulose in a DMAc/LiCl solvent system can be carried out by using BPO and AIBN initiators. That grafting took place is evident from the IR spectra of the grafted cellulose. A nonconventional plant like *Hibiscus cannabinus* has been globally identified as a potential source of fiber for future years. High α-cellulose extracted from such an important plant may be a good fiber source for producing grafted cellulose for the preparation of, for example, high efficiency cellulose membranes.[42]

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