Phase-Transfer-Agent-Aided Polymerization and Graft Copolymerization of Acrylamide

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ABSTRACT: The use of phase-transfer catalysts, with water-insoluble initiators, for polymerization and graft copolymerization reactions was explored. The polymerization of a water-soluble vinyl monomer, acrylamide (AAm), and the graft copolymerization of AAm onto a water-insoluble polymer backbone, isotactic polypropylene (IPP), with a water-insoluble initiator, benzoyl peroxide (BPO), and a phase-transfer catalyst, tetrabutyl ammonium bromide ($Bu_4N^+Br^-$), were carried out in a water/xylene binary solvent system. The conversion percentage of AAm into polyacrylamide (PAAm) and the percentage of grafting of AAm onto IPP were determined as functions of various reaction parameters, such as the BPO, AAm, and phase-transfer-catalyst concentrations, the amounts of water and xylene in the water/xylene mixture, the time, and the temperature. The graft copolymer, IPP-*g*-PAAm, was characterized with IR spectroscopy and thermogravimetric analysis. By a comparison of the results of the phase-transfer-catalyzed graft copolymerization of AAm onto IPP and the preirradiation method, it was observed that the optimum reaction conditions were milder for the phase-transfer-catalyst-aided graft copolymerization. Milder reaction conditions, including the temperature, the time of reaction, and a moderate initiator (BPO), in comparison with high-energy γ-rays, led to better quality products, and the reaction proceeded smoothly with high productivity. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 91: 2364–2375, 2004

Key words: phase transfer catalysis; copolymerization; addition polymerization; isotactic; poly(propylene) (PP); thermogravimetric analysis (TGA)

INTRODUCTION

Phase-transfer catalysis is known to be a very convenient and useful method in organic synthesis. It has also been used in the chemical modification of polymers for the synthesis of various functional polymers.[1] However, very few studies have been reported on the use of phase-transfer catalysts (PTCs) in polymerization processes.

The free-radical polymerization of acrylic acid and other vinyl monomers in solution or in bulk with water-soluble initiators such as potassium oxydisulfate and potassium persulfate has been extensively used for emulsion polymerization processes. However, the insolubility of such initiators in organic solvents and monomers makes their use impractical for solution and bulk polymerization. Recently, it has been found that such water-insoluble initiators can be used effectively for bulk or solution polymerization when used with certain phase-transfer agents, such as quaternary ammonium salts or macrocyclic polyethers (e.g., crown ethers).[2] The kinetics of the phase-transfer-agent-assisted free-radical polymerization of *o*-tolyl methacrylate with potassium persulfate as a water-insoluble initiator and tributyl benzyl ammonium chloride as a PTC were determined in an ethyl acetate/water biphase system at 60°C.[3] The preparation of a new polyether with an oxetane pendant group by polycondensation proceeded very smoothly in aromatic solvents, catalyzed by quaternary ammonium or phosphonium salts such as tetrabutyl ammonium bromide ($Bu_4N^+Br^-$) or tetrabutyl phosphonium bromide, to afford polymers with high yields and high molecular weights.[4] Nakamura and Wang[5] synthesized a high-molecular-weight polyester with reactive groups in the side chain through water-phase/organic-phase interfacial polycondensation with PTCs. The copolymerization of methyl methacrylate with styrene was carried out in water/ethyl acetate with a $K_2S_2O_8$/onium salt catalyst. The reactivity ratio of the copolymerization was calculated.[6] Gupta and Mandal[7] reported the polymerization of methyl methacrylate with $K_2S_2O_8$ as an initiator and $Bu_4N^+Br^-$ as a PTC at 60°C. In this article, we report on the polymerization of acrylamide (AAm) and the graft copolymerization of AAm onto isotactic polypropylene (IPP) with benzoyl peroxide (BPO) as a water-insoluble initiator and $Bu_4N^+Br^-$ as a PTC in a water/*p*-xylene biphase solvent system.

EXPERIMENTAL

Materials and method

IPP in the form of beads was received from Indian Petrochemical Corp., Ltd. (Baroda, India). The beads were recrystallized from *p*-xylene, and the powdered
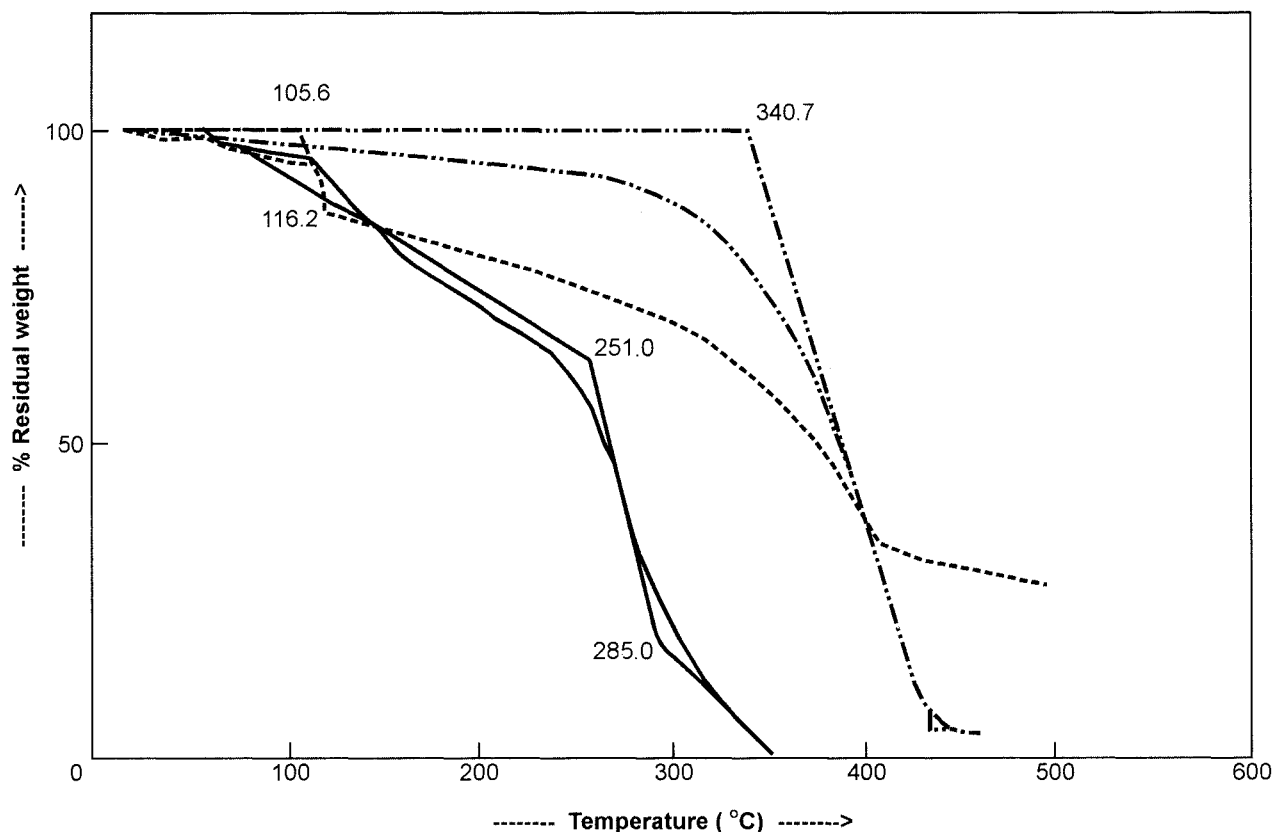

Figure 1 Primary thermograms of (– · · –) IPP, (—) PAAm, and (–) IPP-*g*-PAAm.

IPP was used in all grafting reactions. AAm (Merck) was dried before use. BPO (SD Fine Chemicals) and $Bu_4N^+Br^-$ (Sisco) were used as received. Distilled water and *p*-xylene were used as solvents for the biphase system.

Polymerization of AAm

A definite amount of AAm (100 mg) was dissolved in a known volume of water. To it, a known solution of BPO in *p*-xylene and a definite amount of PTC were added. The reaction mixture was refluxed under constant stirring for different time periods. After the stipulated time period, the reaction was stopped, and an excess of methanol was added to precipitate the unreacted monomer and the homopolymer polyacrylamide (PAAm). The conversion percentage of AAm to

PAAm was calculated from the initial increase in the weight of AAm as follows:

$$\text{Conversion (\%)} = \frac{W_2 - W_1}{W_1} \times 100$$

where W_1 and W_2 are the weights of AAm and PAAm, respectively.

Graft copolymerization of AAm onto IPP

Recrystallized IPP (100 mg) and a known amount of BPO, dissolved in a definite amount of *p*-xylene, were placed in a reaction flask. To it was added a definite amount of AAm, dissolved in a known amount of water. A definite quantity of PTC was added to the

TABLE I
TGA of IPP, PAAm, and IPP-*g*-PAAm

| Sample | IDT (°C) | FDT (°C) | DT (°C) at every 10% weight loss | | | | | | | | | | Residue (%) |
			10	20	30	40	50	60	70	80	90	100	
IPP	310	432.8	289.75	334.05	357.74	374.36	387.47	398.51	408.47	417.92	427.24		5
PAAm	251.8	285.0	118.35	148.40	196.40	238.90	258.15	268.80	272.05	291.99	311.29	347.69	0
IPP-*g*-PAAm			115.35	207.35	292.99	341.64	375.22	394.18	449.88				25

TABLE II
Graft Copolymerization of AAm onto IPP Under Different Reaction Conditions

Sample	IPP (mg)	AAm (mg)	BPO (mg)	PTC (mg)	Water (mL)	p-Xylene (mL)	Observation
1	100	300	150	—	20	—	No reaction
2	100	300	150	—	—	20	No reaction
3	100	300	150	—	10	10	No reaction
4	100	300	150	70	20	—	No reaction
5	100	300	150	70	—	20	No reaction

reaction mixture. The reaction flask was placed in the water bath at a constant temperature, under constant stirring, for a definite time period. After the stipulated time period, the flask was removed, and an excess of methanol was added to precipitate the unreacted monomer, PAAm, and the graft copolymer, IPP-g-PAAm. The reaction mixture was filtered and washed thoroughly with water to remove the homopolymer. The grafted IPP was dried at 45°C until a constant weight was obtained. The percentage of grafting was calculated from the initial increase in the weight of the original IPP in the following manner:

$$\text{Grafting (\%)} = \frac{W_2 - W_1}{W_1} \times 100$$

where W_1 and W_2 are the weights of the original IPP and grafted IPP, respectively, after the complete removal of the homopolymer.

Evidence of grafting

IR spectroscopy

From a comparison of the IR spectra of IPP and IPP-g-PAAm, it was observed that the peaks at 1675 cm^{-1} of >C=O and at 3350 cm^{-1} of the amide group due to —NH stretching in the IR spectrum of the grafted IPP were absent in the spectrum of unmodified IPP. This confirmed the formation of the graft of AAm onto IPP.

Thermogravimetric analysis (TGA)

TGA of IPP, PAAm, and IPP-g-PAAm was carried out in air at a heating rate of 10°C/min, and the primary

thermograms are presented in Figure 1. The initial decomposition temperature (IDT), final decomposition temperature (FDT), and decomposition temperature (DT) for every 10% weight loss are presented in Table I. It can be observed from the thermograms that all three substrates showed different patterns of decomposition. Polypropylene showed great stability toward the initial rise in temperature until the start of the initial decomposition at 310°C. The polymer further degraded continuously, with a small temperature difference for every 10% weight loss. The temperature difference continued decreasing with a rise in the temperature, and this indicated a faster rate of decomposition. The final decomposition began at 432.8°C, at which temperature almost 93% of the polymer decomposed. The residual weight at 436.39°C was 5%.

PAAm, however, showed a double stage of decomposition. During the first stage of decomposition, the initial loss of weight between 70 and 120°C was due to moisture desorption. Beyond this, the decomposition continued at a fast rate and with a high temperature difference for each 10% weight loss until a 45% weight loss. This loss was due to the bonded water in the polymer. The second stage of decomposition began at 251.8°C, beyond which further decomposition occurred at a faster rate with a continuous decrease in the temperature difference for every 10% weight loss until a 70% weight loss, beyond which the temperature difference again rose. Complete decomposition occurred at 348°C with no residue.

The primary thermogram of IPP-g-PAAm showed a very different course of decomposition. A water desorption inflexion was observed between 70 and 106°C, beyond which a sharp decrease was observed

TABLE III
Polymerization of AAm Under Different Reaction Conditions

Sample	AAm (mg)	BPO (mg)	PTC (mg)	Water (mL)	p-Xylene (mL)	Observation
1	300	150	—	20	—	No reaction
2	300	150	—	—	20	No reaction
3	300	150	—	10	10	No reaction
4	300	150	70	20	—	No reaction
5	300	150	70	—	20	No reaction
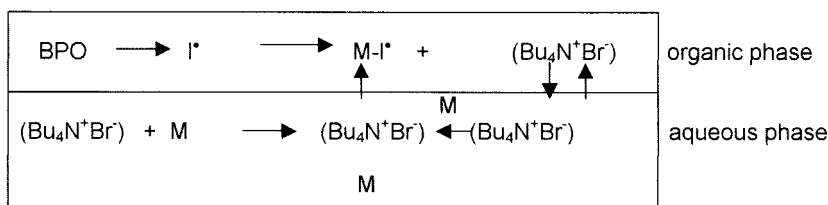
Scheme 1 PTC mechanism for the polymerization of AAm.

with a 5% weight loss until 116.2°C. This weight loss may also have been due to the bonded water in the grafted PAAm. After this, the decomposition took place at a slow rate, with the temperature difference for every 10% weight loss being much higher than either that of the backbone (IPP) or the grafted polymer (PAAm). The residual weight percentage was much higher (25% at 498.39°C).

The DT values of the grafted polypropylene were lower than those of the original polymer up to a 60 wt % loss but became higher thereafter. The lower rate of decomposition, the higher temperature difference for every 10% weight loss, and the much higher residue left for IPP-*g*-PAAm showed that the grafting of AAm onto IPP increased the thermal stability of the backbone polymer.
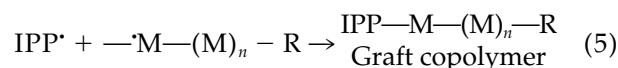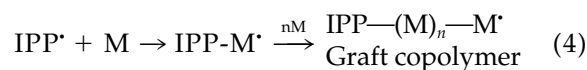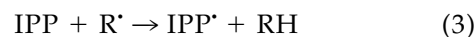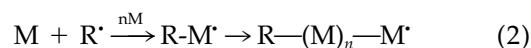
Role of the PTC

To establish that both the polymerization and graft copolymerization occurred via a phase-transfer mechanism, we performed a series of experiments with or without the addition of a PTC ($Bu_4N^+Br^-$) and in water, *p*-xylene, or a binary solvent system consisting of water and *p*-xylene.

In all cases, the reaction mixture was stirred for 2 h at 60°C after the reaction; an excess of methanol was added to precipitate the unreacted monomer, PAAm, and the graft copolymer, IPP-*g*-PAAm. The precipitates dissolved upon treatment with water and *p*-xylene, and this indicated that either AAm or IPP precipitated (Table II). The graft copolymer, IPP-*g*-PAAm, if formed, would have been insoluble in both solvents. Similar reactions were carried out in the absence of IPP, and it was found that the polymerization of AAm also did not occur (Table III). A quantitative amount of

AAm was obtained after the addition of methanol to the reaction mixture. It was evident from these observations that the polymerization of AAm and the graft copolymerization of AAm onto IPP with quaternary ammonium bromide took place via a phase-transfer mechanism.

RESULTS AND DISCUSSION

BPO is a known free-radical initiator for carrying out the polymerizations of various vinyl monomers and graft copolymerization reactions.[8,9] The general mechanism for the polymerization of AAm and the graft copolymerization of AAm onto IPP can be depicted as follows:

$$BPO \rightarrow R^{\bullet} \tag{1}$$

$$M + R^{\bullet} \xrightarrow{nM} R\text{-}M^{\bullet} \rightarrow R\text{---}(M)_n\text{---}M^{\bullet} \tag{2}$$

$$IPP + R^{\bullet} \rightarrow IPP^{\bullet} + RH \tag{3}$$

$$IPP^{\bullet} + M \rightarrow IPP\text{-}M^{\bullet} \xrightarrow{nM} \begin{array}{c} IPP\text{---}(M)_n\text{---}M^{\bullet} \\ \text{Graft copolymer} \end{array} \tag{4}$$

$$IPP^{\bullet} + {}^{\bullet}M\text{---}(M)_n - R \rightarrow \begin{array}{c} IPP\text{---}M\text{---}(M)_n\text{---}R \\ \text{Graft copolymer} \end{array} \tag{5}$$

$$R\text{---}(M)_n\text{---}M^{\bullet} + {}^{\bullet}M\text{---}(M)_n\text{---}R \rightarrow \begin{array}{c} RM_{2n+2} \\ \text{Homopolymer} \end{array} \tag{6}$$
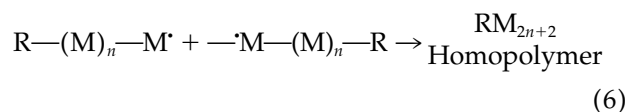
where R · is a radical species generated from BPO.
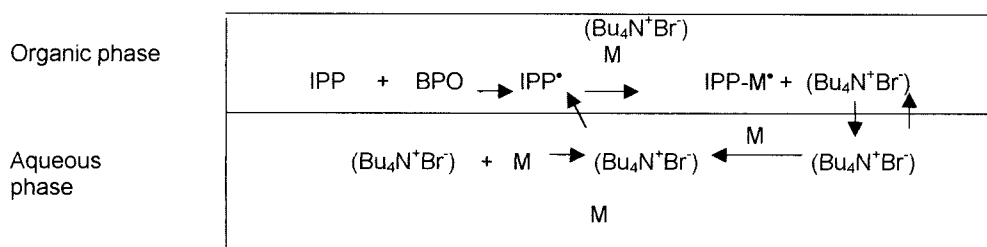
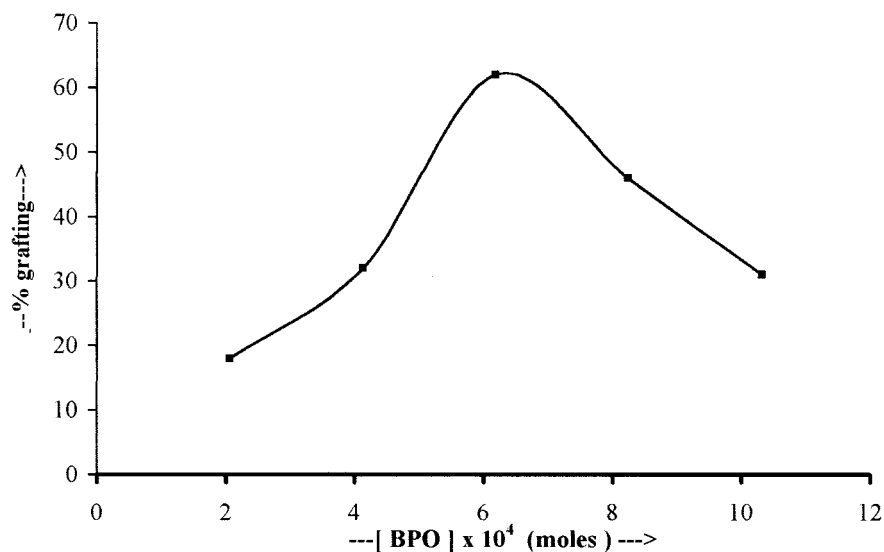**Scheme 2** PTC mechanism for the graft polymerization of AAm onto IPP.

Figure 2 Effect of the BPO concentration on the grafting percentage of AAm onto IPP.

The interaction of the reactants, that is, the backbone polymer IPP, the monomer AAm, and the initiator, BPO, distributed between the two phases, the organic phase and the aqueous phase, takes place with the involvement of the PTC $Bu_4N^+Br^-$. The transfer of the reactant from the aqueous phase to the organic phase by the PTC can be visualized, as shown in Schemes 1 and 2. IPP, a hydrocarbon polymer, and BPO, soluble in p-xylene, are in the organic phase. BPO decomposes to give free radicals, which abstract tertiary hydrogen from IPP; this results in the formation of active IPP macro free radicals, at which the grafting of the vinyl monomer, AAm, can take place. The PTC, being soluble in both phases, transports the water-soluble monomer to the organic layer. The initiation of the monomer in the organic phase takes place by the initiator radical (step 2) or by the macroradical present in the organic phase (step 4), which can lead to the production of the graft copolymer (steps 4 and 5) or the homopolymer (step 6).

The mechanism of the polymerization of AAm is the same, except for the reactions involving IPP. It involves the generation of radicals from the initiator (step 1), the initiation and propagation of the monomer (step 2), and termination to give the polymer (step 6).

The polymerization of AAm to PAAm and the graft copolymerization of AAm onto IPP powder by a phase-transfer-catalyzed mechanism have been studied as functions of various reaction parameters, and the results are explained in light of the proposed mechanism.
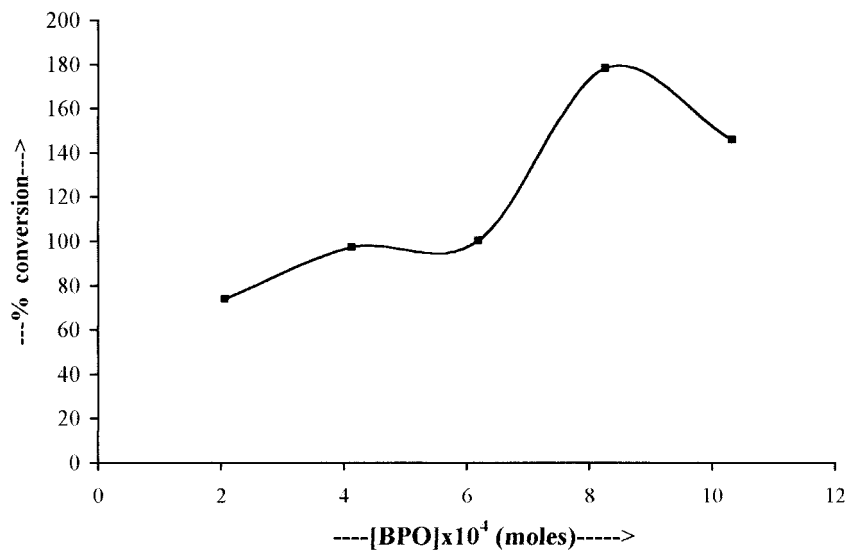

Figure 3 Effect of the BPO concentration on the conversion percentage of AAm to PAAm.
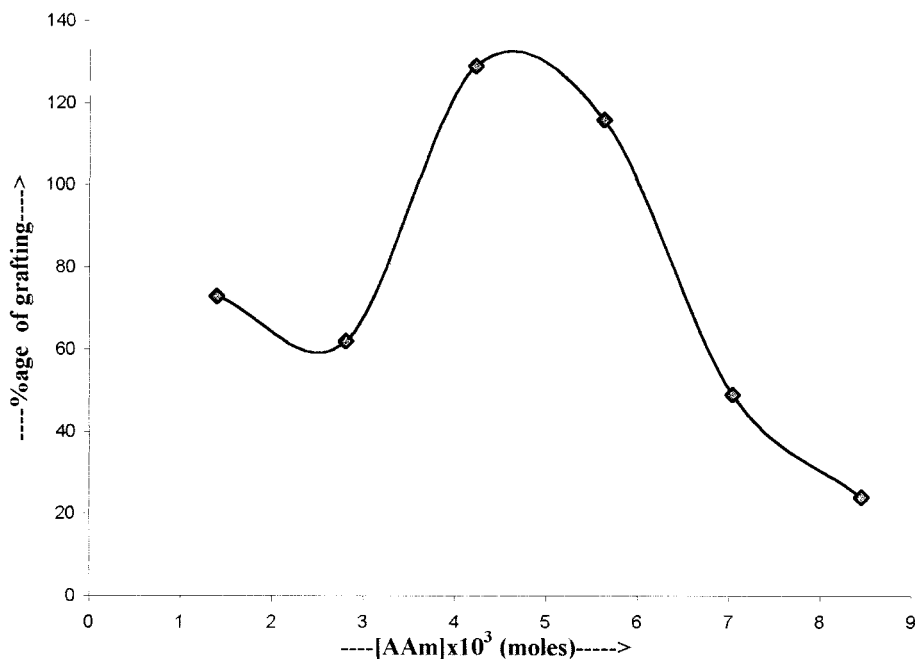
Figure 4 Effect of the monomer concentration on the grafting percentage of AAm onto IPP.

Effect of the BPO concentration

The graft copolymerization of AAm onto IPP and the polymerization of AAm to PAAm were studied as functions of the BPO concentration, and the results are presented in Figures 2 and 3, respectively. It can be observed from Figure 2 that the grafting percentage of AAm onto IPP increases with an increase in the BPO concentration, being maximum (62%) at [BPO] = 6.94 $\times$ 10^{-4} mol, beyond which it decreases. The conversion percentage of AAm to PAAm also increases with an increase in the BPO concentration, being maximum (178.5%) at [BPO] = 8.2 $\times$ 10^{-4} mol, and decreases thereafter (Fig. 3). The decrease in the grafting and conversion percentages beyond the optimum BPO concentration may be due to the fact that, at higher concentrations, BPO undergoes induced decomposition, leading to a decrease in the radical concentration and thereby reducing the initiation processes and affecting both the grafting and polymerization reactions:
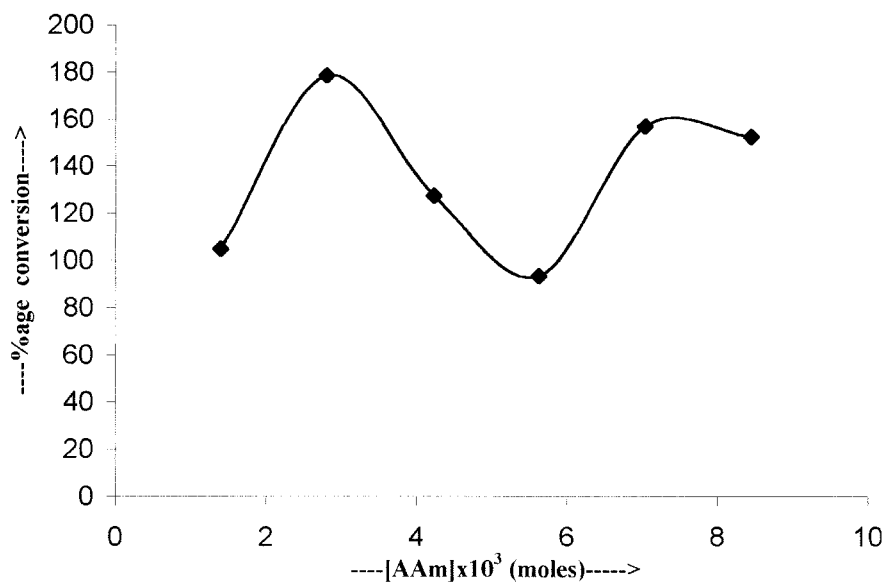

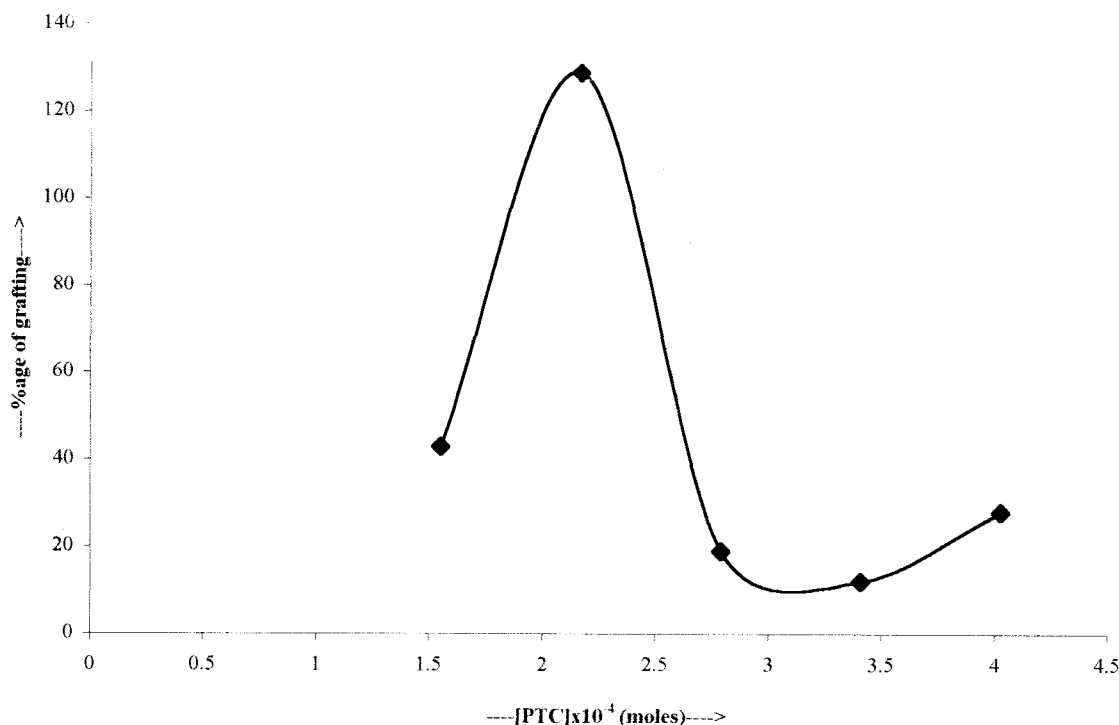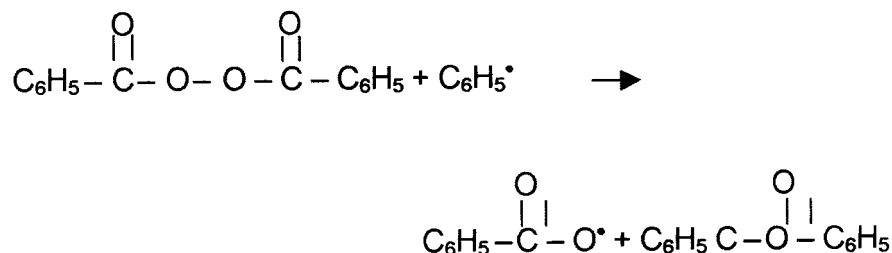**Figure 5** Effect of the monomer concentration on the conversion percentage of AAm to PAAm.

Figure 6 Effect of the PTC concentration on the grafting percentage of AAm onto IPP.

Similar observations were made during the BPO-initiated grafting of acrylonitrile and methyl acrylate onto starch.[10] It has further been observed that the maximum grafting percentage of AAm (62%) onto IPP is obtained at a lower BPO concentration (6.94×10^{-4} mol), whereas a higher conversion percentage (178.5%) of AAm is obtained at a higher BPO concentration (8.2×10^{-4} mol). This may be attributed to the fact that the initiation of the monomer is much faster than the generation of active sites on the polymeric backbone via hydrogen abstraction, and this leads to a higher monomer conversion. During grafting, the competition between the homopolymer formation and graft copolymerization leads to a lower grafting percentage, with a maximum at a lower BPO concentration. At a higher BPO concentration, more of the homopolymer is formed at the expense of the graft copolymer.

Effect of the monomer concentration

Figures 4 and 5 represent the effects of the monomer concentration on the grafting percentage of AAm onto IPP and the conversion percentage of AAm to PAAm, respectively. Both the grafting percentage (Fig. 4) and conversion percentage (Fig. 5) increase with increasing monomer concentration and decrease thereafter. The maximum percentage of grafting (129%) is obtained at [AAm] = 4.32×10^{-3} mol, and the maximum conversion of AAm (178.2%) is obtained at [AAm] = 2.81×10^{-4} mol. The decrease in the percentage of grafting at a higher monomer concentration is due to preferential homopolymer formation. The higher rate of propagation (18,000 L/mol s^{-1}) and the higher rate of termination (14.5×10^{6} L/mol s^{-1}) of AAm[11] lead to higher polymer formation than the graft copolymer. However, a decrease in the conversion percentage
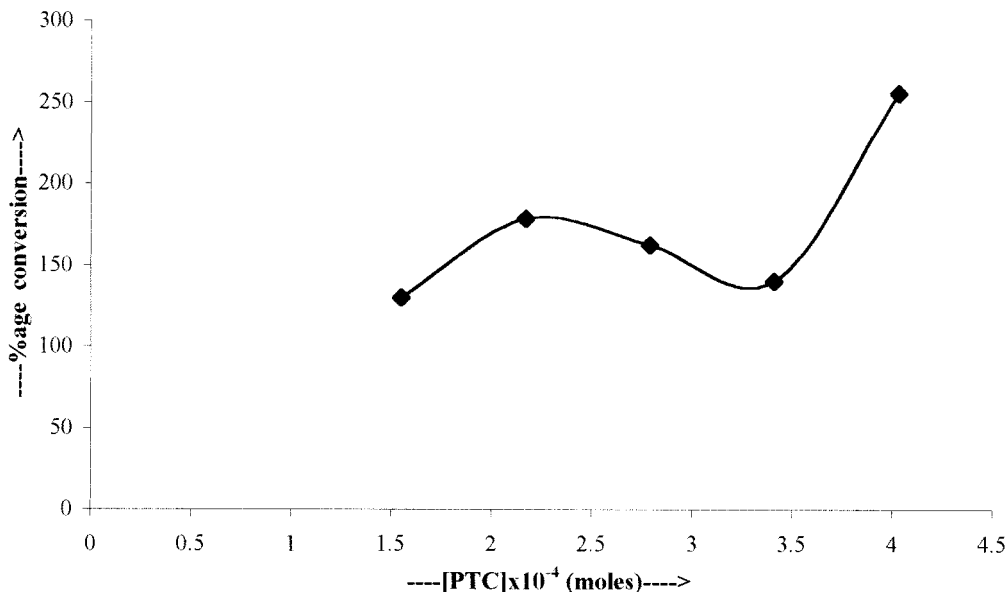
Figure 7 Effect of the PTC concentration on the conversion percentage of AAm to PAAm.

beyond the optimum value of the monomer concentration can be attributed to the various chain-transfer reactions, the monomer-transfer constant being high (ca. 0.6×10^{-4}).

Effect of the PTC concentration

The effect of the PTC concentration, $Bu_4N^+Br^-$, on the percentage of grafting of AAm onto IPP is presented in Figure 6, and its effect on the conversion percentage of AAm is presented in Figure 7. Both the percentage of grafting of AAm and the conversion percentage of AAm show an increase with an increasing concentration of PTC. The maximum percentage of grafting (129%) and the maximum conversion percentage (178.5%) are obtained at [PTC] = 2.17×10^{-4} mol. A further increase in the concentration leads to a reduction in either of the processes. However, at a higher PTC concentration, beyond 3.5×10^{-4} mol, a second rise in the percentage of grafting and conversion percentage can be observed, the rise being sharp in the polymerization process. The mechanism of the reac-
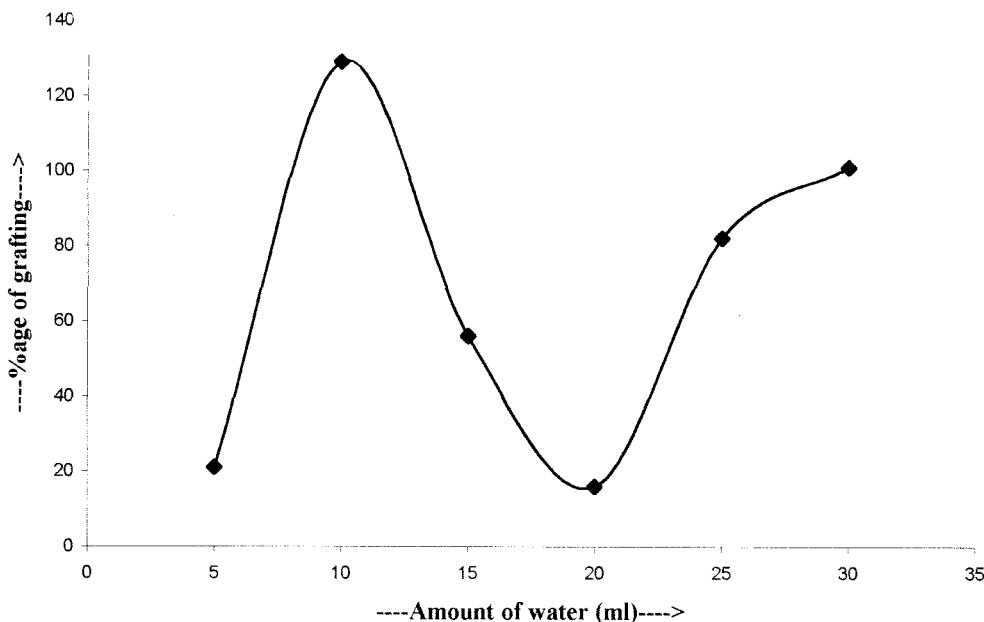

Figure 8 Effect of the variation of the amount of water in a water/xylene mixture on the grafting percentage of AAm onto IPP.
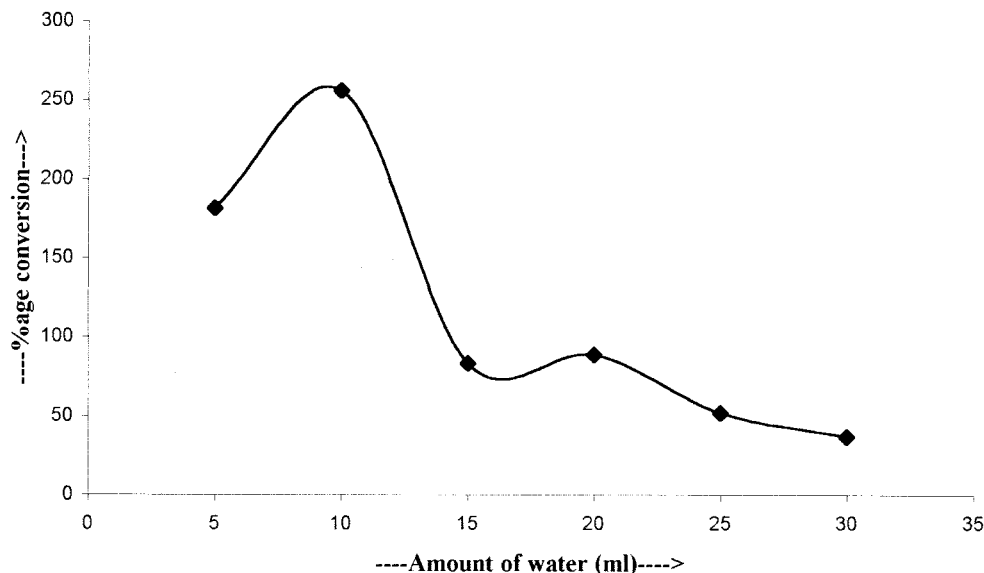
Figure 9 Effect of the variation of the amount of water in a water/xylene mixture on the conversion percentage of AAm to PAAm.

tion involves the transportation of the monomer from the aqueous phase to the organic phase by the PTC. An optimum catalyst concentration (2.17×10^{-4} mol) promotes both reactions, giving the maximum percentage of grafting and the maximum conversion percentage. An increase in the PTC concentration beyond the optimum increases the transportation of the monomer to the organic phase, and this may lead to more homopolymer formation. The homopolymer, PAAm, being soluble in water, increases the viscosity of the medium, leading to a decrease in grafting. The transfer of the monomer to the organic phase is also thus restricted, and the monomer reacts with growing polymeric chains, leading to homopolymer formation. This leads to a sharp increase in the monomer conversion, as observed at a higher PTC concentration.

Effect of the amount of water and *p*-xylene in the water/*p*-xylene solvent system

The percentage of grafting of AAm onto IPP (Fig. 8) and the conversion percentage of AAm to PAAm (Fig. 9) were studied as functions of the variations in the amounts of both water and *p*-xylene in a water/xylene solvent system, with the volume of one of them kept fixed at 10 mL. The percentage of grafting of AAm and
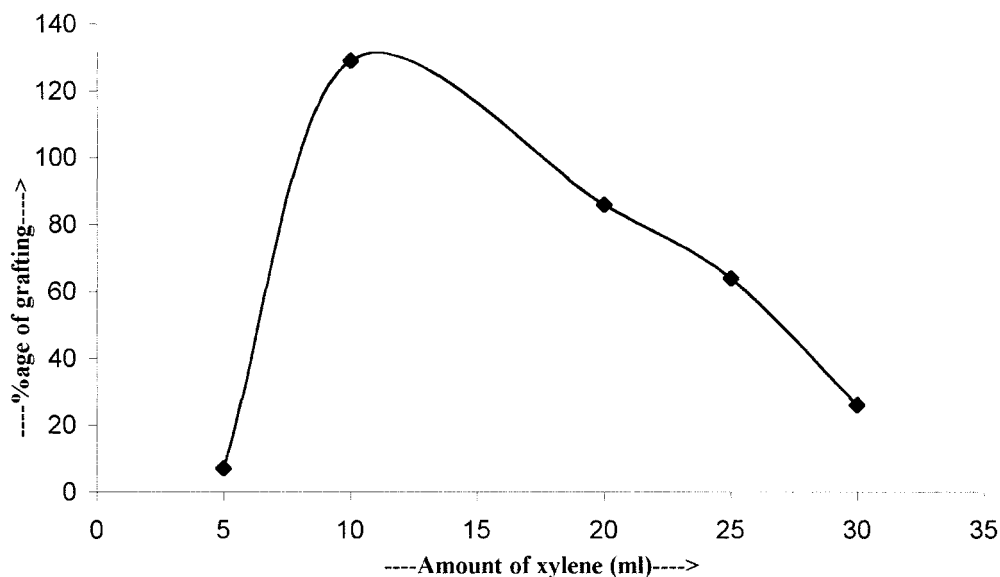

Figure 10 Effect of the variation of the amount of xylene in a water/xylene mixture on the grafting percentage of AAm onto IPP.
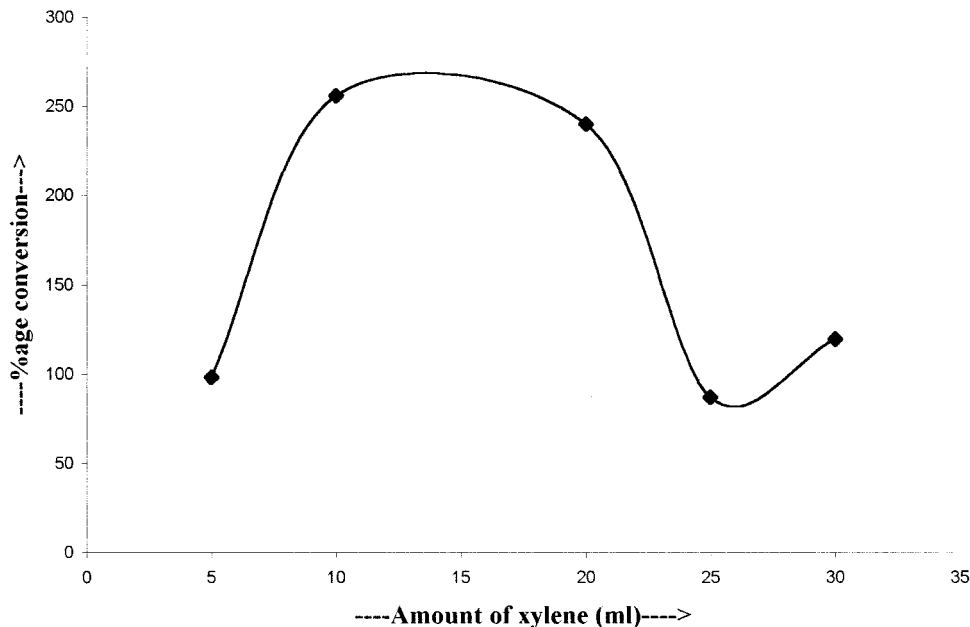
Figure 11 Effect of the variation of the amount of xylene in a water/xylene mixture on the conversion percentage of AAm to PAAm.

the conversion percentage of AAm, with various amounts of water, increases with an increase in the amount of water, and the maximum percentage of grafting (129%) and maximum conversion percentage (256%) are produced in 10 mL of water. A further increase in the amount of water reduces the percentage of grafting and the conversion percentage. However, beyond 20 mL of water, a further rise in the percentage of grafting can be observed. The decrease in the percentage of grafting and the polymer conversion beyond the optimum amount of water can be attributed to the fact that in an excess of water, the

monomer is wasted in the chain-transfer reactions. The solvent (water in this case) transfer constant of AAm,[12] being high (5.8×10^{-4}), leads to a decrease in both the percentage of grafting and the conversion percentages of AAm. Beyond 20 mL of water, a further rise in the percentage of grafting can be observed. This may be due to the fact that in a greater amount of water, the viscosity of the medium decreases; this increases the accessibility of the growing polymeric chains to the active sites and leads to a high percentage of grafting.

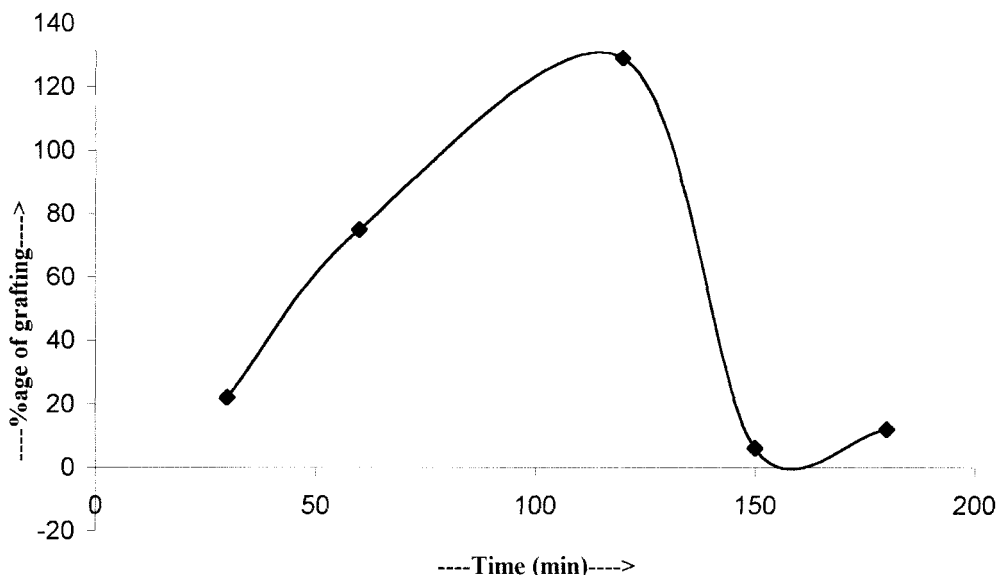Figures 10 and 11 represent the percentage of grafting

Figure 12 Effect of the variation of time on the grafting percentage of AAm onto IPP.
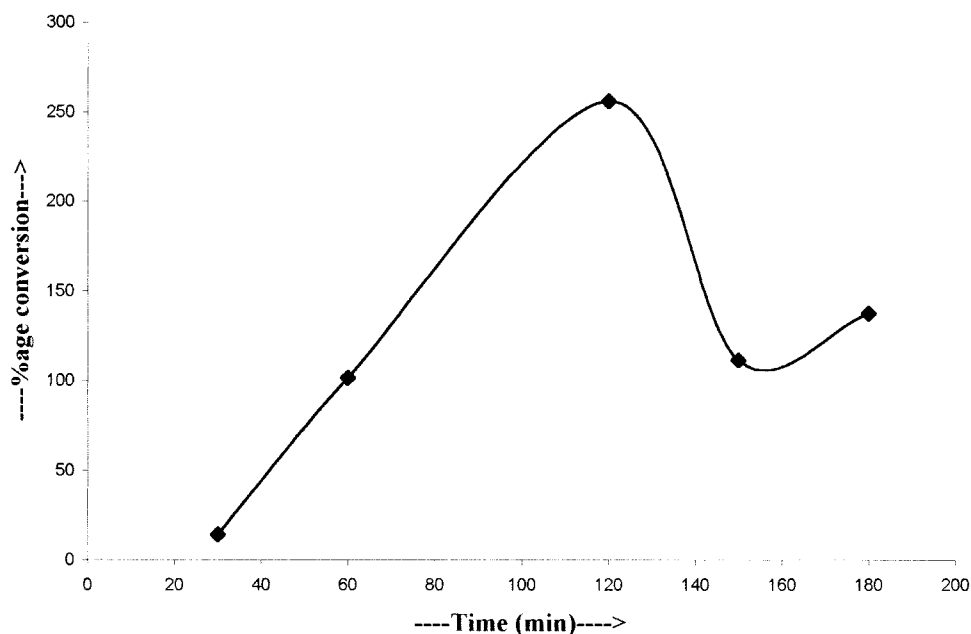
Figure 13　Effect of the variation of time on the conversion percentage of AAm to PAAm.

of AAm onto IPP and the conversion percentage of AAm to PAAm, respectively, as functions of the variations of p-xylene in a water/xylene mixture. Both the percentage of grafting and the conversion percentage increase with an increase in the amount of p-xylene; the maximum percentage of grafting (129%) and maximum conversion percentage (256%) are found in 10 mL of xylene. Beyond the optimum amount, both processes show a decrease in the percentage of grafting and conversion percentage. Furthermore, the maximum polymer formation or graft copolymer formation occurs when a 1:1 (v/v) ratio, that is, 10 mL each of water and xylene, is used in the binary solvent system. Any increase or decrease in either of the solvents reduces both the grafting and conversion percentages. This may be due to the disturbances caused in the transportation of the reactants or the products from the aqueous layer to the organic layer and vice versa.

Effect of the time of the reaction

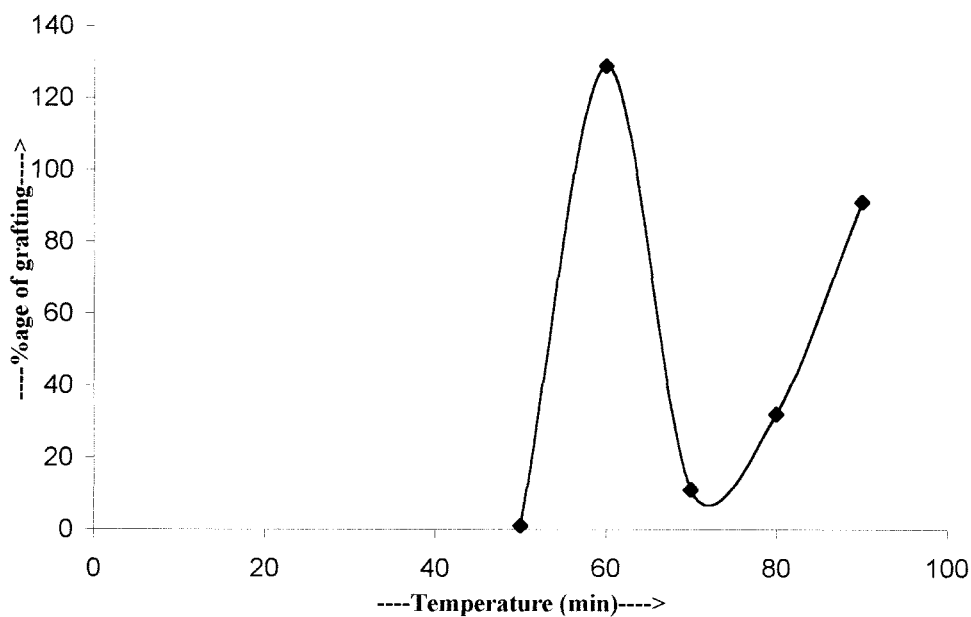Figures 12 and 13 show plots of the percentage of grafting of AAm onto IPP and the conversion percent-

Figure 14　Effect of the variation of temperature on the grafting percentage of AAm onto IPP.

TABLE IV
Optimum Conditions for the Graft Copolymerization Of AAm onto IPP by the Preirradiation Method and the Phase-Transfer-Catalyzed Method

Method	IPP	Solvent	Initiator	Temperature (°C)	Time (min)	Grafting (%)
Preirradiation	100 mg	H_2O (6 mL)	γ total dose = 7.7	100	180	176
PT-catalysed	100 mg	H_2OXylene 10:10 (mL)	[BPO] = 6.9×10^{-4} mo	60	120	129

age of AAm, respectively, as functions of the reaction time. Both the grafting percentage and conversion percentage increase with an increase in the reaction time; the maximum percentage of grafting (129%) and the maximum conversion percentage (256%) are obtained within 120 min. Then, they decrease. The decrease in the grafting or polymer conversion may be due to induced decomposition of the initiator and mutual annihilation of growing polymeric chains, which lead to preferential homopolymer formation.

Effect of the temperature

The effect of the temperature on the percentage of grafting of AAm onto IPP was studied, and the results are presented in Figure 14. The percentage of grafting increases with increasing temperature, being maximum (129%) at 60°C. A further increase in the temperature reduces the percentage of grafting. The increasing percentage of grafting with increasing temperature is due to the increased decomposition of the initiator, which leads to the formation of more free radicals and the generation of active sites on the polymeric backbone. The transportation of the monomer from the aqueous phase is also accelerated at higher temperatures, and this leads to a high percentage of grafting. A decrease in the percentage of grafting, beyond the optimum temperature, is attributable to premature termination of the growing polymeric chains and to the occurrence of chain-transfer reactions.

Comparison of the graft copolymerization of AAm onto IPP by the preirradiation method and the phase-transfer-catalyzed method

The graft copolymerization of AAm onto IPP was studied earlier by the preirradiation method; the optimum conditions for obtaining the maximum percentage of grafting were evaluated,[13] and they are presented in Table IV. The optimum conditions for the grafting of AAm onto IPP by phase-transfer catalysis are also presented in Table IV. Comparing the optimum conditions for obtaining the maximum percentage of grafting by the two methods, we can see that the maximum grafting (176%) of AAm onto IPP by the

preirradiation method is higher than that (129%) obtained by the phase-transfer-catalyzed grafting of AAm onto IPP. However, the optimum values of various reaction variables are milder for phase-transfer-catalyzed reactions. The amount of AAm (2.81×10^{-3} mol) is much lower than that (2.1×10^{-3} mol) used for grafting by the preirradiation method. The irradiation of IPP was carried out with high-energy γ-rays, with a total dose of 7.7 Mrad leading to the oxidation of the polymer. The hydroperoxidized polypropylene was treated with a monomer at a much higher temperature (100°C) for 180 min to get the maximum (176%) grafting percentage. In the phase-transfer-catalyzed grafting, the polymer IPP was treated with a monomer in the presence of a free-radical initiator (BPO) and a PTC ($Bu_4N^+Br^-$) at a low temperature (60°C) for 120 min, when the maximum percentage of grafting (129%) was obtained. Thus, PTC in a graft copolymerization reaction reduces the use of excess monomer. The milder reaction conditions, including the temperature, time of reaction, and a moderate initiator (i.e., BPO vs high-energy γ-rays), lead to a product of better quality, and the reaction proceeds smoothly with high productivity.

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