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Grafting Vinyl Monomers onto Nylon 6 Fiber. III. Graft Copolymerization of Vinyl Monomers onto Nylon 6 Fiber in Water Medium in the Presence of Fructose

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ABSTRACT

Nylon 6 fiber was grafted with various vinyl monomers e.g., methyl methacrylate (MMA), ethyl methacrylate (EMA), and n-butyl methacrylate (n-BMA), in water in the presence of fructose using a carbon arc lamp as the source of photoirradiation at 70°C and a liquor ratio of 1:26. The effects of various parameters, e.g., monomer concentration, time of grafting, and fructose concentration on grafting reactions, were studied individually for each monomer. The graft yield is greatly enhanced by increasing the monomer concentration is required for maximum grafting efficiency. The accelerating action of photopolymerization by fructose was attributed to the sensitizing action of fructose involving an energy transfer.

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INTRODUCTION

Ogiwara et al. [1] have shown that photopolymerization by UV light of various vinyl monomers (for example, methyl methacrylate (MMA), ethyl methacrylate (EMA), n-butyl methacrylate (n-BMA), styrene, acrylamide) can be accelerated by using various saccharides. They proposed a mechanism employing the saccharide as an energy transfer agent.

In the present study three different acrylic monomers (e.g., MMA, EMA, and n-BMA) were grafted onto nylon 6 fiber in the presence of a water-fructose medium.

EXPERIMENTAL

Nylon 6 fiber, acetone, MMA, and fructose are the same as mentioned in our previous paper [2]. EMA and n-BMA, as supplied by BDH Laboratories, Bombay, India, were washed with 5% sodium hydroxide solution in a separating funnel to remove the inhibitor. They were then washed repeatedly with water, dried over calcium chloride, and distilled under vacuum. Double-distilled water was used. The grafting technique using a photoirradiation source was the same as used previously [3].

RESULTS AND DISCUSSION

Preliminary experiments of grafting using water as the medium in the presence of various saccharides (e.g., glucose, sucrose, and fructose) have been attempted. It was found that satisfactory grafting takes place with fructose and hence this saccharide only was used. Earlier [2] it was shown that grafting of the vinyl monomers under consideration onto nylon 6 fiber did not take place in water medium in the absence of fructose. In the present paper the effects of various parameters (monomer concentration, time of grating, fructose concentration, etc.) on grafting have been investigated.

Effect of Monomer Concentration on Grafting

Nylon 6 fiber was grafted using a fructose concentration of 10 mmol/ L and a liquor ratio 1:26 in water medium at 70° C for 90 min using a carbon arc lamp as the UV source for four different monomer concentrations. The results are presented in Figs. 1, 2, and 3 for MMA, EMA, and n-BMA, respectively.

From the results it can be seen that with an increase in the monomer concentration both the total polymer yield (% TPY) and the graft

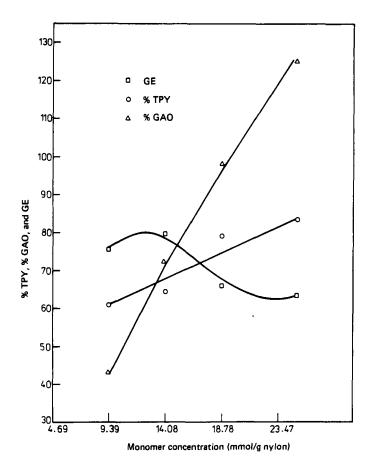
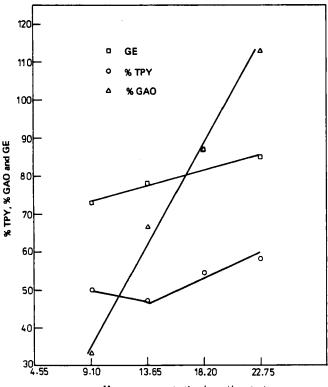


FIG. 1. Effect of monomer concentration on grafting of MMA onto nylon 6 fiber in water medium in the presence of fructose. Nylon 6 fiber, 1g; liquor ratio, 1:26; 70°C; time, 90 min, fructose concentration, 10 mmol/L: total polymer yield (% TYP) (\circ); graft add-on (%GAO) (\triangle); grafting efficiency (GE) (\Box).

add-on (% GAO) increase continuously whereas GE reaches a maximum (79.4 and 87 in the cases of MMA and EMA, respectively) and then falls. For n-BMA monomer, however, the % GAO as well as GE increases constantly and linearly as the monomer concentration increases whereas the % TPY practically remains constant. This indicates that with increase in monomer concentration grafting reaction is favored compared to homopolymer formation.

Several workers [4-6] have found a similar effect on % graft addon by an increase of monomer concentration. Other systems are



Monomer concentration (mmol/g nylon)

FIG. 2. Effect on monomer concentration on grafting of EMA onto nylon 6 fiber in water medium in the presence of fructose. Nylon 6 fiber, 1 g; liquor ratio, 1:26; time, 90 min, 70°C; fructose concentration, 10 mmol/L.

known where either grafting efficiency is good only at low monomer concentration [7] or decreases with an increase in monomer concentration [8].

Effect of Fructose Concentration on Grafting

Nylon 6 fiber was grafted with the monomers MMA, EMA, and n-BMA using a liquor ratio of 1:26 in water medium at 70°C for 90 min, varying the fructose concentration from 5 mmol to 20 mmol/L. The results are presented in Figs. 4, 5, and 6 for MMA, EMA, and n-BMA, respectively. It was shown earlier that in the absence of fructose no grafting takes place when using all three monomers. However, as the

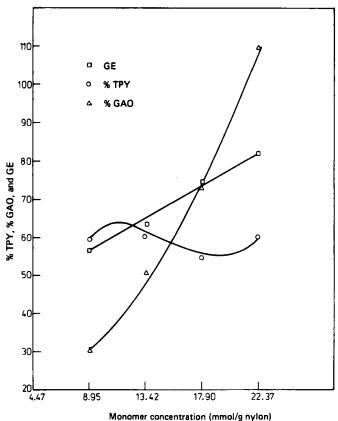


FIG. 3. Effect on monomer concentration on grafting of n-BMA onto nylon 6 fiber in water medium in the presence of fructose. Nylon fiber, 1 g; liquor ratio, 1:26; time, 90 min; 70° C; fructose concentration, 10 mmol/L.

fructose concentration increases, both the % TPY and % GAO increase up to 10 mmol/L of fructose and then falls using MMA monomer. The GE is also highest at this concentration. For EMA, however, the %TPY practically remains constant but % GAO and GE increase, reach a maximum (for 10 mmol concentration of fructose), and then fall. An optimum level of % GAO and GE is a general phenomenon [9]. In the case of n-BMA with an increase of fructose concentration, the grafting efficiency increases linearly but % GAO increases up to a fructose concentration of 15 mmol/L and then falls. % TPY also varies similar to that of % GAO. The optimum fructose concentration of fructose in this reaction seems to be 15 mmol/L.

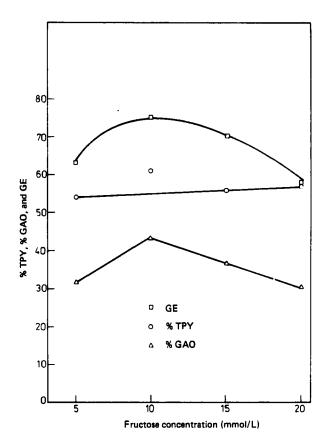
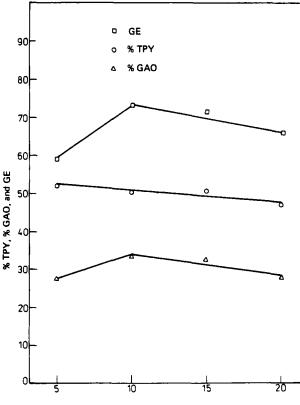


FIG. 4. Effect of fructose concentration on grafting of MMA onto nylon 6 fiber in water medium. Nylon 6 fiber, 1 g; liquor ratio, 1:26; time, 90 min; 70° C; monomer concentration, 9.39 mmol/g nylon.

Several workers [4, 10, 11] have found that there is always an optimum concentration of the initiator at which maximum grafting efficiency is achieved.

An ESR study [1] for the photopolymerization of MMA in water containing saccharide with a high-pressure Hg lamp in a hard glass tube did not show any radical formation of fructose, thus indicating that no photodecomposition of fructose takes place. In the same study, no change in reducing power of the saccharide was observed and hence the acceleration of photopolymerization of MMA in that system for fructose was attributed to the sensitizing action of fructose involving an energy transfer.

In the present work no ESR study was done but the reducing power



Fructose concentration (mmol/L)

FIG. 5. Effect of fructose concentration on grafting of EMA onto nylon 6 fiber in water medium. Nylon 6 fiber, 1 g; liquor ratio, 1:26; time, 90 min; 70° C; monomer concentration, 9.1 mmol/g nylon.

of fructose treated with a photo-source for 6 h was measured and found to be unchanged. This indicates that in the present system fructose acts only as an agent for energy transfer and does not undergo initiation at the glucosidic bond.

Effect of Time on Grafting

Nylon 6 was grafted at 70° C using a liquor ratio of 1:26 and monomer concentrations of MMA, EMA, and n-BMA of 9.39, 9.1, and 8.95 mmol/g nylon, respectively. The time was varied from 60 to 180 min. The results are presented in Figs. 7, 8, and 9. From the results it

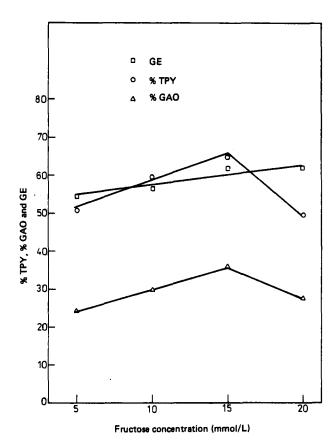


FIG. 6. Effect of fructose concentration on grafting of n-BMA onto nylon 6 fiber in water medium. Nylon 6 fiber, 1 g; monomer concentration, 8.95 mmol/g nylon; time 90 min; liquor ratio, 1:26; 70° C.

can be seen that % TPY does not change much in the case of MMA grafting. However, at 120 min there is an increase in % GAO, apparently due to a decrease in % homopolymerization. Consequently, grafting efficiency also increases in this range. The high conversion appears to affect the reaction adversely. Because the high amount of grafting has already taken place, further polymerization (hence % TPY) does not increase. A chain transfer reaction involving the homopolymer in one hand and the nylon fiber on the other can take place. If this happens, then % GAO increases, % TPY decreases, and, as a consequence, grafting efficiency increases. Chain transfer reactions involving polymers are well known and

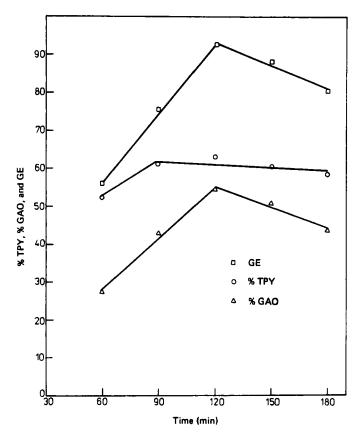


FIG. 7. Effect of time on grafting of MMA onto nylon 6 fiber in water medium in the presence of fructose. Nylon 6 fiber, 1 g; liquor ratio, 1:26; 70°C; monomer concentration, 9.39 mmol/L; fructose concentration, 10 mmol/L.

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MMA is no exception [12]. In the case of EMA grafting, however, % GAO and % TPY increase up to 120 min and thereafter they decrease. GE at this point becomes practically constant. As a large amount of grafting has already taken place, further polymerization and hence % TPY does not increase. These results are similar to those obtained in the case of MMA grafting, and it appears that a similar phenomenon might also be operating here. For n-BMA grafting it is also seen that % GAO and % TPY increase with an increase in time. The GE, however, increases, reaches a maximum (69.4), and falls. The results obtained here seem to be similar to those obtained with MMA and EMA grafting under similar conditions.

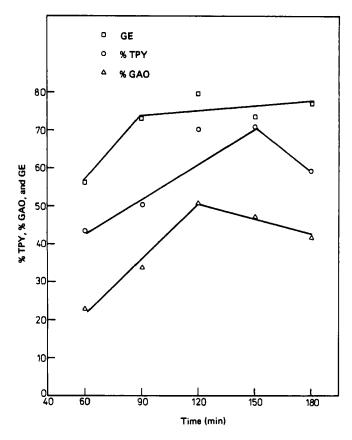


FIG. 8. Effect of time on grafting EMA onto nylon 6 fiber in water medium in the presence of fructose. Nylon 6 fiber, 1 g; liquor ratio, 1:26; fructose concentration, 10 mmol/L; monomer concentration, 9.1 mmol/g nylon; 70° C.

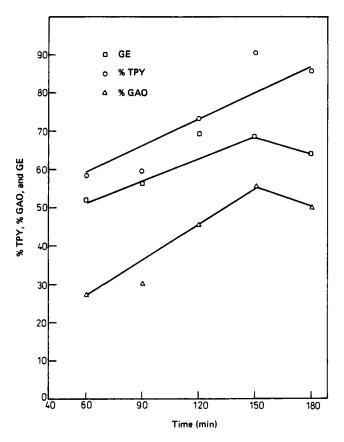


FIG. 9. Effect of time on grafting of n-BMA onto nylon 6 fiber in water medium in the presence of fructose. Nylon 6 fiber, 1 g; monomer concentration, 8.95 mmol/g nylon; liquor ratio 1:26; 70° C; fructose concentration, 10 mmol/L.

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