Characterization of Radiation Induced Ethyl Methacrylate Grafted Nylon-6 Fibers

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Synopsis

Ethyl methacrylate grafted nylon-6 fibers prepared by mutual irradiation technique was characterized to determine the modifications in its properties arising as a result of grafting. Scanning electron microscopy study provides some information about the presence of grafting. Density of fibers were found to increase constantly with percentage graft. Where as alkali solubility, moisture regain decreased with percentage graft thereby decreasing the dyeability of the fiber. However the ethyl methacrylate grafted nylon-6 fiber showed improvements in its thermal properties over the original nylon-6 fiber.

INTRODUCTION

In the recent years the gamma ray induced graft copolymerization of vinyl monomers onto nylon-6 fibers has evoked considerable interest[1-9] as it offers a versatile technique to impart desirable properties to the polymer without affecting its original properties.[10] The properties which can be modified by radiolytic grafting are dirt released properties, antistatic properties, and dyeability.

The nylon-6 fiber has not been commercially successful as a textile fiber due to its low crystalline temperature, softness, and lack of stiffness. This investigation aims at evaluating the changes in the properties of nylon-6 fibers as a result of graft copolymerizing ethyl methacrylate by mutual irradiation technique.

EXPERIMENTAL

Various ethyl methacrylate grafted nylon-6 samples were prepared with different percent graft obtained by graft copolymerization of ethyl methacrylate onto nylon-6 fibers by mutual irradiation technique with a Co-60 gamma source at the total dose of 28.4×10^{-2} Mrad as reported earlier.[4] Density of the poly(ethyl methacrylate) grafted nylon-6 fibers was determined in toluene at 30°C using a specific gravity bottle. The moisture regain was determined with vacuum desiccator. The alkali solubility studies were carried out following the procedure of Leaves et al.[11] Dyeability of fiber was carried out with the dye, Chemlene Blue SE, 2RI, and Remazol Brilliant Orange 3R. Thermal behaviour of the samples were evaluated by thermogravimetric analysis.

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RESULT AND DISCUSSION

Density

The density measurements of the poly(ethyl methacrylate) grafted nylon-6 fibers has been represented in Figure 1, which shows that the density of the grafted fiber increase with increase in percent grafting. Similar behaviour has also been reported by Mukherjee et al.[12] and Burlant et al.[13] in other systems. Such as increase in density appears quite reasonable due to an increase in density of the grafted moiety on the fiber with increase in percentage of grafting.

Moisture Regain

Moisture regain of the grafted fibers is presented in Figure 2. It was observed that an increase in percentage of grafting lowers the moisture regain ability of the nylon-6 fibers. Although grafting with ethyl methacrylate brings about opening of the structure to a certain extent still the fiber shows a decreased moisture regain which can be explained as follows. The monomer ethyl methacrylate is hydrophobic in nature and in less grafted fibers all the voids in the amorphous regions are not filled up, so fibers of low graft percentage of these monomers have higher moisture regain ability compared to more grafted fibers.

Alkali Solubility

A perusal of the results of alkali solubility as represented in Figure 3 resolved very interesting facts. It was found that an increase in percent of grafting decreases the alkali solubility of the fibers which can be explained as that with an increase in percent of grafting the resistance of the grafted fiber towards alkali. This reduction in alkali solubility after grafting suggests that the grafted

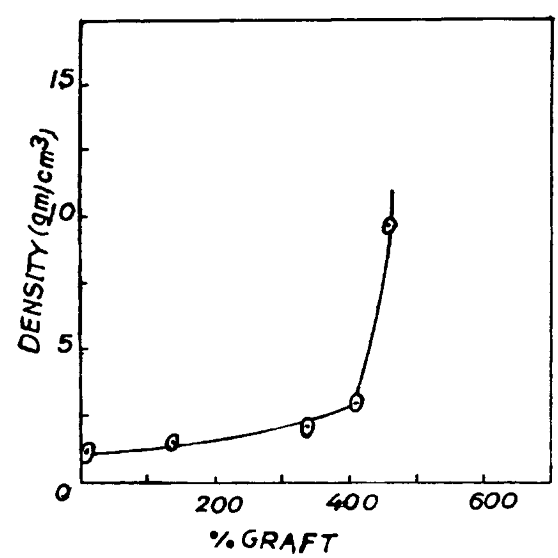

Fig. 1. Plot of graft percentage versus density.

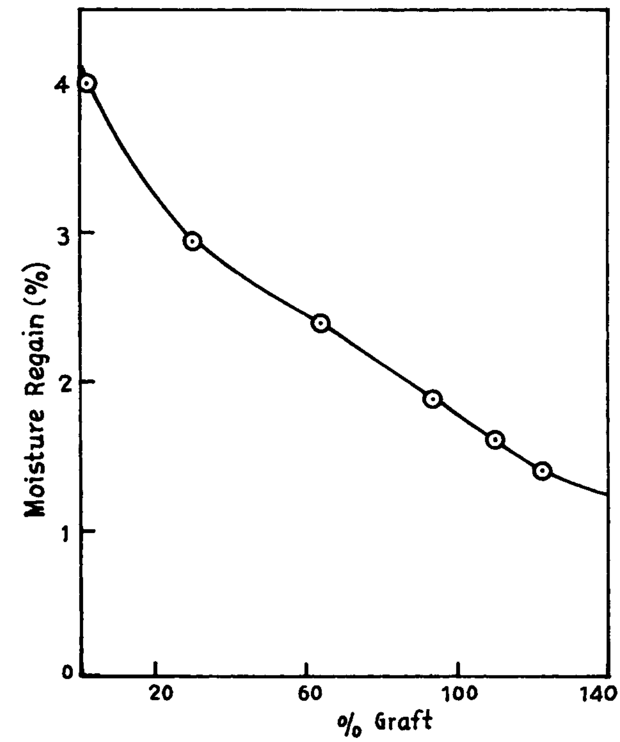

Fig. 2. Plot of graft percentage versus moisture regain.

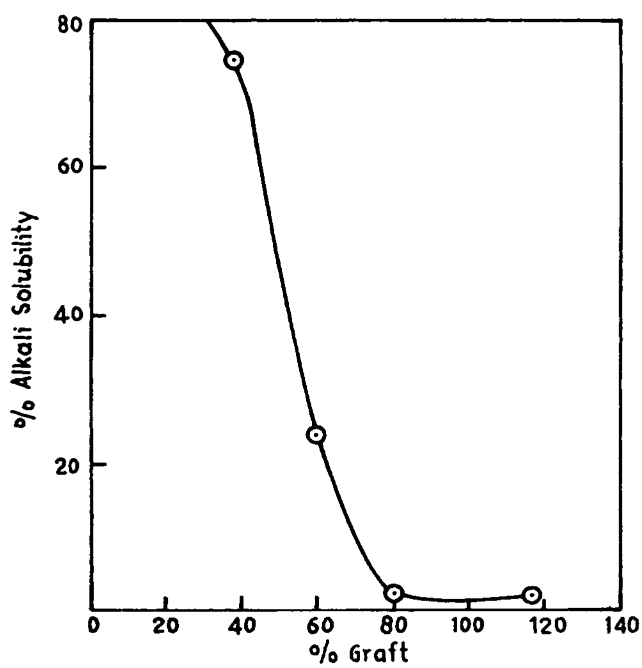

Fig. 3. Plot of graft percentage versus percentage of alkali solubility (NaOH − 10%).

polymer chains acts as diffusion barriers towards alkali penetration onto the fiber thereby protecting the polyamide chains.

Dyeability

The dye uptake ability of the grafted fibers as tested with the dye Chemlene Blue SE, 2RI. The material to liquor ratio was 1 : 200. The dye bath consists of 2% dye solution with 1 g/l trisodium phosphate maintaining a pH of 9.5. The dyeing was carried out at 85°C for 8 h. The dye uptake was determined by extracting the dye from dyed fiber with formic acid and then determining its concentration in an UV spectrophotometer at an wavelength of 400 nm.

It was observed from Figure 4 that an increase in the percent of grafting first increases as the amount of dye uptake reaches a maximum, and then with further increase in percent of grafting, dyeability of the grafted nylon-6 fiber decreased. Similar observations also show while poly(ethyl methacrylate) grafted nylon-6 fibers were dyed with the dye Remazol brilliant orange-3R dye.

This initial increase in dyeability with increase percent in grafting can be explained as follows. An increase in percent of grafting decreases the crystallinity of the fibers, that is it increases the space for accession of dye molecules. But at a higher percentage of grafting both the steric effect and the hydrophobic nature of the grafted poly(ethyl methacrylate) chains become predominant and hence together decrease the dyeability of the grafted nylon-6 fiber.

The fact that the dyeability of the grafted fibers are higher than swollen fibers is because with low graft-on percentage only a small portion of the amorphous region is being blocked by the polymer chain. But at dyeing conditions the retention of the dye between the grafted polymer chains is much higher than with native sample.

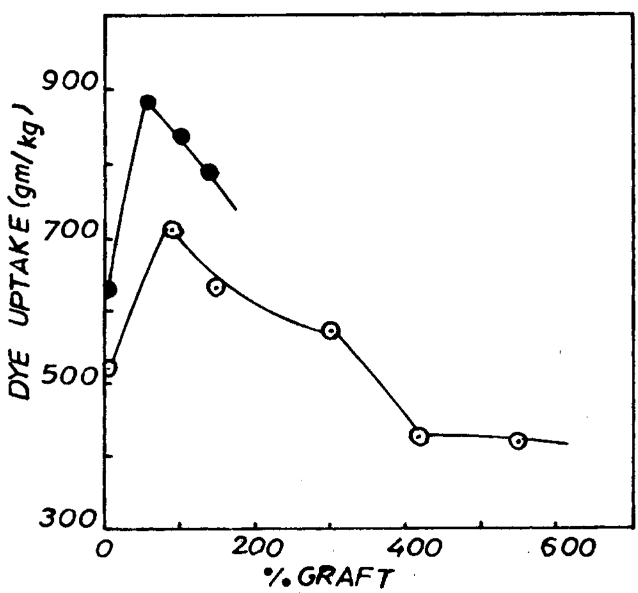

Fig. 4. Plot of graft percentage versus dye uptake (○) Chemlene Blue SE 2RI (●) Remazol Brilliant Orange 3R.

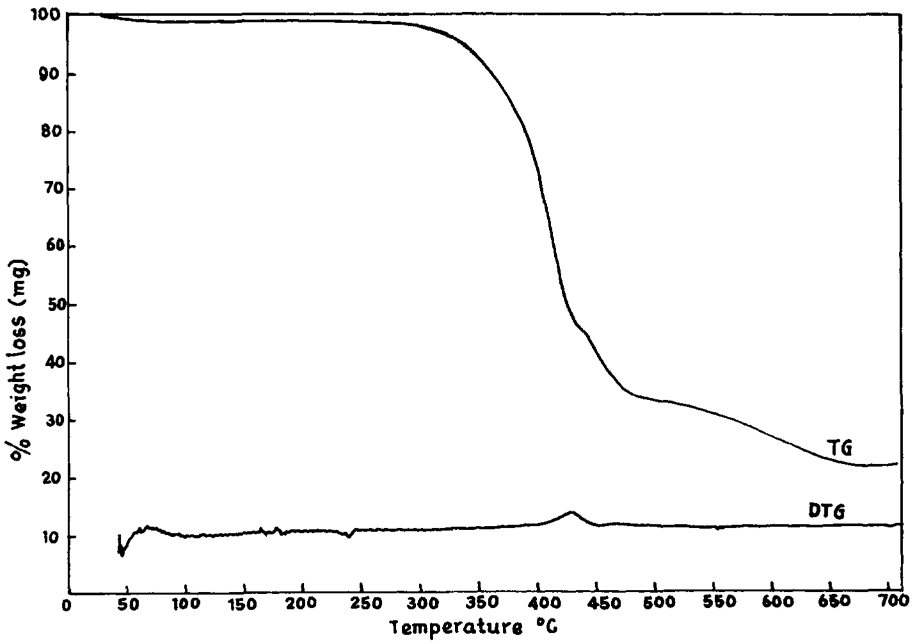

Fig. 5. TGA and DTG curve of 72% poly(ethyl methacrylate) grafted nylon-6 fiber.

In spite of all these factors all the dyed samples show very good fastness to light and washings.

Thermogravimetric Analysis

The thermogravimetric analysis was made with Du-Pont instrument at a heating rate of 10°C/min in air. The thermal behaviour of the grafted nylon-6 fiber depends mainly on two parameters such as (i) percentage of graft-on and (ii) nature of the monomer. The grafted nylon-6 fiber was found to be more resistant to melting than ungrafted fiber. The ungrafted nylon-6 fiber melts at 215°C. The TG curve of grafted nylon-6 fiber is represented in Figure 5. Showing the percentage of loss at various temperatures. TG analysis reveals that the grafted fiber undergoes 10% weight loss at about 375°C, 80% weight loss at 425°C, and 95% weight loss at 700°C. It is assumed that a small amount of the weight loss up to 375°C is due to loss of moisture or entrapped solvent present in the cavity of the grafted fiber, viewed in this way, the grafted nylon-6 fiber appears to be fairly thermostable. The rate of maximum decomposition is very fast up to 600°C when the weight loss is about 90%. The weight loss maintains a steadiness up to 425°C which is also confirmed by DTG curve.

Scanning Electron Miscroscopy

Positive evidence regarding the existence of a graft copolymer covering the fiber matrix was obtained from the photomicrographs given in Figure 6. A scanning transmission unit interfaced with Philip EM 400 transmission electron

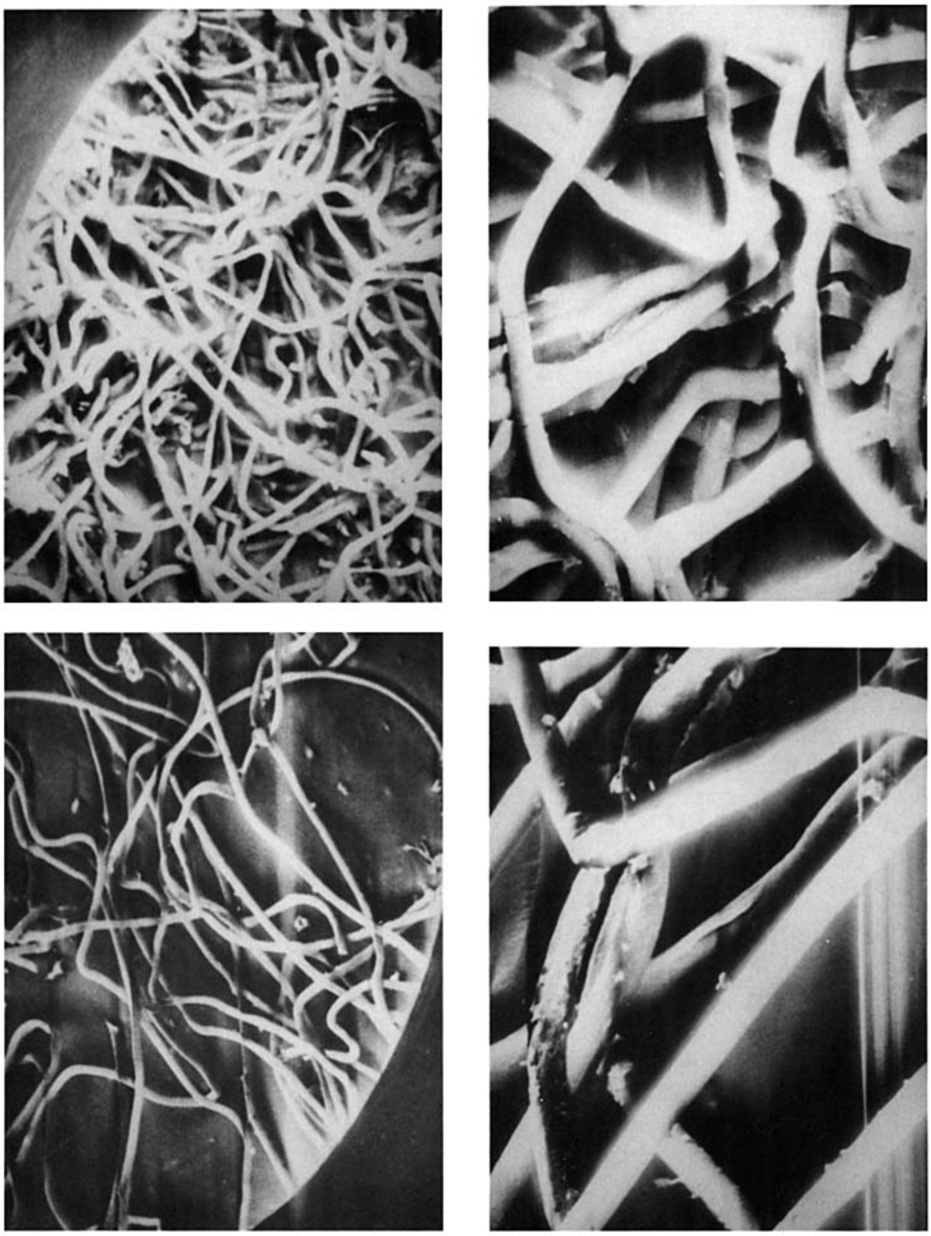

Fig. 6. Scanning electron micrographs of the fiber.

microscope was used for the purpose. The surface of the grafted fibers were examined with the scanning electron microscope at high resolution.

Grafted nylon-6 fibers with a wide range of graft percentage was used and the resulted photomicrographs were compared with the photomicrograph obtained with native nylon-6 fiber taken at identical conditions. A close examination of various photomicrographs presented in Fig. 6 clearly shows the random disorderly distribution of the fiber matrix in case of ungrafted nylon-6 fiber.

Fig. 6. *(Continued from previous page.)*

An increase in percentage of grafting increases the order in the distribution of fiber matrix. The thickening of the fiber matrix with grafted fibers indicated that the grafting has occurred throughout the fiber matrix, giving it an oval shape although it was nonuniform. With increase in percentage of grafting the packing of the fiber matrix has been widened, which indicates that the grafting has occurred in the interior of the fiber matrix as observed by the more flattened nature of the fiber matrix with increasing percentage of grafting.

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