

Removal of Cesium-134 and Cobalt-60 with Radiation-Grafted Copolymers from Their Liquid Wastes

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ABSTRACT: The introduction of new characteristics to some synthetic polymers was achieved through the γ -radiation-induced graft copolymerization of an acrylamide/vinyl acetate comonomer onto polypropylene films for the preparation of synthetic membranes. The influence of the reaction conditions on the grafting yield, such as the solvent and its composition and the inhibitor and its concentration, was investigated. An alkaline treatment was carried out for the prepared graft copolymer to improve its ion-exchange property. The grafted and treated membranes were characterized to determine the structural changes with X-ray diffraction and Fourier transform infrared spectroscopy. The

mechanical properties, swelling behavior, and electrical conductivity for the graft copolymer were studied. Improvements in the tensile strength, electrical conductivity, and hydrophilicity with grafting were observed. The KOH-treated membranes possessed greater hydrophilic properties than the untreated ones. The possibility of practical uses for these membranes in the removal of cesium-134 and cobalt-60 from their liquid wastes was also investigated. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 95: 936–945, 2005

Key words: irradiation; membranes; waste

INTRODUCTION

Cobalt-60 and cesium-134 are the most common isotopes used in different areas such as medicine, industry, and research. The wide applications of cobalt-60 and cesium-134 result in the generation of huge amounts of liquid radioactive wastes containing these isotopes.^{1,2} The direct radiation grafting of different vinyl monomers and their mixtures onto low-density polyethylene, high-density polyethylene, and polypropylene (PP) films was carried out for metal-ion separation from solutions containing a single cation or simulated medium-active waste.^{3–13}

In this study, the radiation-induced graft copolymerization of an acrylamide (AAm)/vinyl acetate (VAc) comonomer mixture onto PP films was investigated for the preparation of ion-exchange membranes that could meet the requirements of chelation of cesium-134 and cobalt-60 from their liquid wastes. The effect of the grafting conditions on the grafting yield was determined as well as the chemical treatment of the grafted amide and acetate groups for the production of different functional groups. The characterization and properties of the prepared membranes were

studied, including swelling in water, electrical conductivity, and mechanical properties.

EXPERIMENTAL

Materials

PP (30 μm thick) was produced by El-Nasr Co. for Medical Supplies (Cairo, Egypt). VAc, more than 99% pure (Merck, Germany), and reagent-grade AAm, more than 99% pure, were used as received. Other chemicals, such as solvents and inorganic salts, were reagent-grade and were used without further purification.

Graft copolymerization

The graft copolymers were prepared through the radiation grafting of an AAm/VAc binary monomer system onto PP films with cobalt-60 γ -rays at a dose rate of 0.27–1.16 Gy S^{-1} . Strips of PP were washed with methanol, dried at 50°C in a vacuum oven, weighed, and then immersed in a 30 wt % 50/50 AAm/VAc comonomer solution. The direct radiation grafting method was used in an N_2 atmosphere. The glass ampules containing the PP films and comonomer solution were then subjected to cobalt-60 γ -rays at a total dose of 20 kGy. The grafted films were removed and washed thoroughly with hot distilled water and then acetone to extract the residual monomers and the homopolymer, which may have accumulated in the

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films. The films were then dried in a vacuum oven at 50°C for 24 h and weighed. The degree of grafting was calculated by the percentage increase in the weight of the PP films after the grafting process as follows:

$$\text{Degree of grafting (\%)} = [(W_g - W_0)/W_0] \times 100$$

where W_0 and W_g are the weights of the ungrafted and grafted films, respectively.

Alkaline treatment

The grafted films were alkali-treated with a 3% KOH solution at the ambient temperature for 24 h. The membranes thus obtained were washed with distilled water several times.

Swelling measurements

The dried membranes were immersed in distilled water at approximately 25°C until equilibrium was reached (almost 24 h). The surface of the membranes was blotted with adsorbent paper to remove any attached water and was weighed quickly. This technique was described in a previous study.¹⁴ The degree of swelling of the membranes was determined as follows:

$$\text{Degree of swelling (\%)} = [(W_s - W_g)/W_g] \times 100$$

where W_g and W_s are the weights of the dry and swelled membranes, respectively.

Fourier transform infrared (FTIR) spectrophotometry analysis

IR spectra of the ungrafted and grafted treated films were recorded on a FTIR-1000 spectrophotometer (Mattson, UK) in the range of 500–4000 cm^{-1} .

X-ray diffraction (XRD) measurements

XRD patterns of the ungrafted, grafted, and grafted and treated samples were recorded on Shimadzu XD_DI diffractometer (Japan). The X-ray copper target tube was operated at 40 kV and 30 mA.

Electrical conductivity measurements

The measurement of the electrical conductivity was carried out with a Mega Ohm II (Germany) and a D812 Weiheim Multi Megaohm Meter II. The samples were inserted between two circular electrodes. Thus, through the determination of the resistance of the sample, the electrical conductivity could be calculated:

$$\sigma = (1/R)(L/A) \Omega^{-1}\text{cm}^{-1}$$

where σ is the electrical conductivity, L is the thickness of the specimen (cm), A is the cross-sectional area the disc (cm^2), and R is the ohmic resistance of the specimen (Ω).

Mechanical properties

The tensile strength and elongation at break were measured with an Instron (England) 1195. Dumbbell-shaped specimens (50 mm long and 4 mm wide with a 2.5-cm neck) were used in these tests at a crosshead speed of 50 mm/min.

Scanning electron microscopy

The surfaces and cross-sectional topography of ungrafted and grafted samples were examined with a JEOL SEM-25 scanning electron microscope. Before the examination, the membranes were dried under coated gold.

Permeability

The permeability of the membranes at different differential pressures (psi) was measured with an automated perm porometer (Ithaca, NY).

Radioactive waste treatment

Cobalt (cobalt-60) waste and cesium (cesium-134) waste were obtained from the decontamination unit at the Radiation Protection Department of the Nuclear Research Center. The activity of the liquid radioactive waste was measured with a model 800A multichannel analyzer. The grafted and treated membrane was immersed in the radioactive liquid waste, and the remaining activity was then measured after the removal of the membrane. Therefore, the uptaken radionuclide was determined as follows:

$$\text{Radionuclide Uptake (\%)} = [(I_0 - I)/I_0] \times 100$$

where I_0 and I are the initial activity and the remaining activity, respectively.

RESULTS AND DISCUSSION

Preparation of the grafted membranes

Effect of the solvent

To determine the suitable solvent for the cograftering process of AAm and VAc, we studied graft copolymerization of AAm and VAc mixtures onto PP films in different solvents (Table I). The addition of water was necessary for the grafting of hydrophilic AAm, and organic solvents were added to enhance the diffusion of the VAc monomer into the polymer matrix. The

TABLE I
Effect of the Solvent on the Graft Copolymerization of a 50/50 (w/w) AAm/VAc Mixture onto PP Films

Solvent	Degree of grafting (%)
Water/methanol (50/50 wt %)	77
Water/dioxane (50/50 wt %)	37
Water/acetone (50/50 wt %)	24.5
Water/ethyl acetate/isopropanol (35/25/10 wt %)	20.8
Water/isopropanol (50/50 wt %)	26
Acetone	23
Methanol	15
Water/ethyl acetate/carbon tetrachloride (35/25/10 wt %)	2.1

Comonomer concentration = 30 wt%; irradiation dose = 20 kGy.

grafting yield depended on the organic solvent added to the reaction mixture. A high grafting yield was observed with an H₂O/MeOH mixture in comparison with the other solvents used. This may have been due to the low rate of chain transfer to the solvent and the higher diffusion of the monomer solution into the polymer substrate. Thus, a high local concentration of the monomer around the formed active sites resulted in a high grafting yield.^{15,16}

Effect of the inhibitor

The addition of some inorganic salts to the reaction mixture was investigated in an attempt to minimize the homopolymerization and to optimize the grafting conditions. The concentration of these salts was kept constant at 0.5 wt %, and the results are given in Table II. The grafting process did not proceed successfully in the absence of an inhibitor because of the fast homopolymerization of AAm and VAc, which prevented the diffusion of more monomer molecules into the polymer matrix during radiation grafting. The addition of Mohr's salt enhanced the grafting yield to a great extent, but some homopolymer formed, and so

TABLE II
Effect of the Addition of Inorganic Salts as Inhibitors on the Grafting of an AAm/VAc Binary Mixture onto PP with 50/50 (w/w) MeOH/H₂O as a Diluent

Inorganic salt	Degree of grafting (%)
Salt-free	27
Mohr's Salt	360
FeCl ₃	286
CuSO ₄	205
CuSO ₄ + NaCl	54
LiCl	19.8

Comonomer concentration = 30% of composition 50/50 (wt %); irradiation dose = 20 kGy; salt concentration = 0.5 wt %.

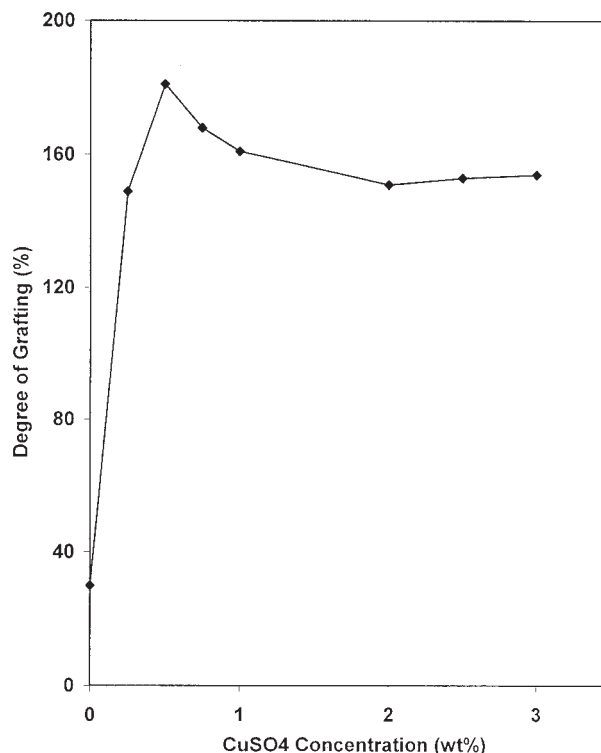


Figure 1 Effect of the inhibitor concentration on the grafting of an AAm/VAc binary mixture (50/50 w/w) onto PP in methanol/water (50/50 w/w) as a solvent. The comonomer concentration was 30 wt %, and the irradiation dose was 20 kGy.

did FeCl₃ salt. However, the presence of CuSO₄ in the reaction mixture enhanced the grafting yield to a lesser extent in comparison with that of Mohr's salt and FeCl₃, but it retarded completely the homopolymer formation, and homogeneous grafting was obtained to a reasonable degree.

The results indicated that the Cu²⁺ ion was a good scavenger for polyacrylamide (PAAm) and poly(vinyl acetate) (PVAc) macroradicals, and so the CuSO₄ salt was chosen as an inhibitor for the grafting of the AAm/VAc binary mixture onto PP films.

Effect of the CuSO₄ concentration on the grafting process

As discussed previously, a suitable inhibitor preventing the homopolymerization of the monomers and providing a reasonable degree of grafting was CuSO₄. However, Cu²⁺ could play a role in the termination of propagating chain radicals and reduce the grafting yield, and so the optimum quantity required to promote the grafting process had to be determined. Figure 1 shows the role of Cu²⁺ and its concentration in the grafting process. The maximum extent of grafting appeared at 0.5 wt % CuSO₄. Then, the graft yields slightly decreased with increasing Cu²⁺ concentra-

TABLE III
Effect of the MeOH/H₂O Composition on the Grafting Yield of AAm/VAc onto PP

MeOH/H ₂ O composition (wt %)	Degree of grafting (%)
80/20	109
70/30	200
60/40	250
50/50	300
40/60	240
30/70	226
20/80	200

Comonomer concentration = 30 wt % of composition 50/50 (wt %); CuSO₄ concentration = 0.5 wt %; irradiation dose = 20 kGy.

tion, but the grafting percentage was still higher than that of the case for which Cu²⁺ was absent. This meant that the role of Cu²⁺ in increasing the rate of grafting was greater than the termination of growing chain radicals. At high Cu²⁺ concentrations (>0.5 wt %), the rate of termination of growing chain radicals increased significantly, and the grafting percentage was reduced.

Effect of the MeOH/H₂O composition

Table III represents the grafting percentage of the AAm/VAc binary mixture as a function of the amount of water. The percentage of grafting increased with an increasing amount of water and reached its maximum at an MeOH/H₂O composition of 50/50 (w/w), beyond which it decreased. The results suggested that changing the polarity of the solvent through changes in the amount of water changed the partitioning coefficient of the monomers surrounding the growing chain radicals. The reduction of the total grafting yield at a high MeOH content may have been caused by the more favorable partitioning of VAc surrounding the polymer active sites and the reduction of the local concentration of AAm, which resulted in a decrease in the AAm grafting rate. On the other hand, if a more polar solvent were used, the favorable partitioning of nonpolar VAc into a nonpolar polymer might become more efficient, and a relatively high VAc concentration in the graft copolymer would be expected.

Characterization and properties

The characterization of the graft copolymer was performed with FTIR and XRD, which were used to clarify the morphological structures and changes that occurred because of the grafting and alkaline treatment. Some selected properties, such as the swelling behavior, electrical conductivity, and mechanical properties, were also investigated.

Structural investigation

The FTIR spectra of the grafted films showed absorption bands corresponding to carbonyl groups of AAm and VAc at 1720 and 1744 cm⁻¹, respectively. A new broad band around 3200–3363 cm⁻¹ appeared, indicating the presence of NH₂ stretching vibrations (characteristic for the structure of AAm). The alkali-treated graft copolymer showed a broad band around 3200–3600 cm⁻¹ and another peak at 1030–1100 cm⁻¹; they were assigned to OH and C—O of alcoholic groups, respectively. An absorption at 1560 cm⁻¹ was also observed, which is usually ascribed to carboxylate groups. The characteristic peak corresponding to carbonyl of the amide groups at 1744 cm⁻¹ of the alkali-treated film was somewhat smaller than that of the untreated ones, and this indicated possible hydrolysis of the remaining amide groups. The OH band at 3200 cm⁻¹ became very broad after alkaline treatment, and this suggested the possible conversion of the AAm group into carboxylic acid groups.

XRD studies

Figure 2 shows the X-ray diffractograms of blank and grafted samples of different degrees of grafting and

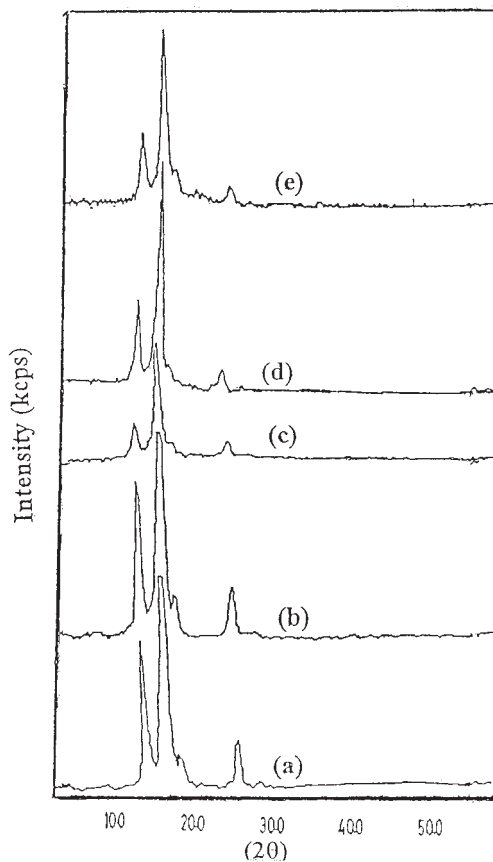


Figure 2 XRD patterns of (a) ungrafted PP, (b) 31% grafted PP, (c) 118% grafted PP, and (d,e) KOH-treated forms.

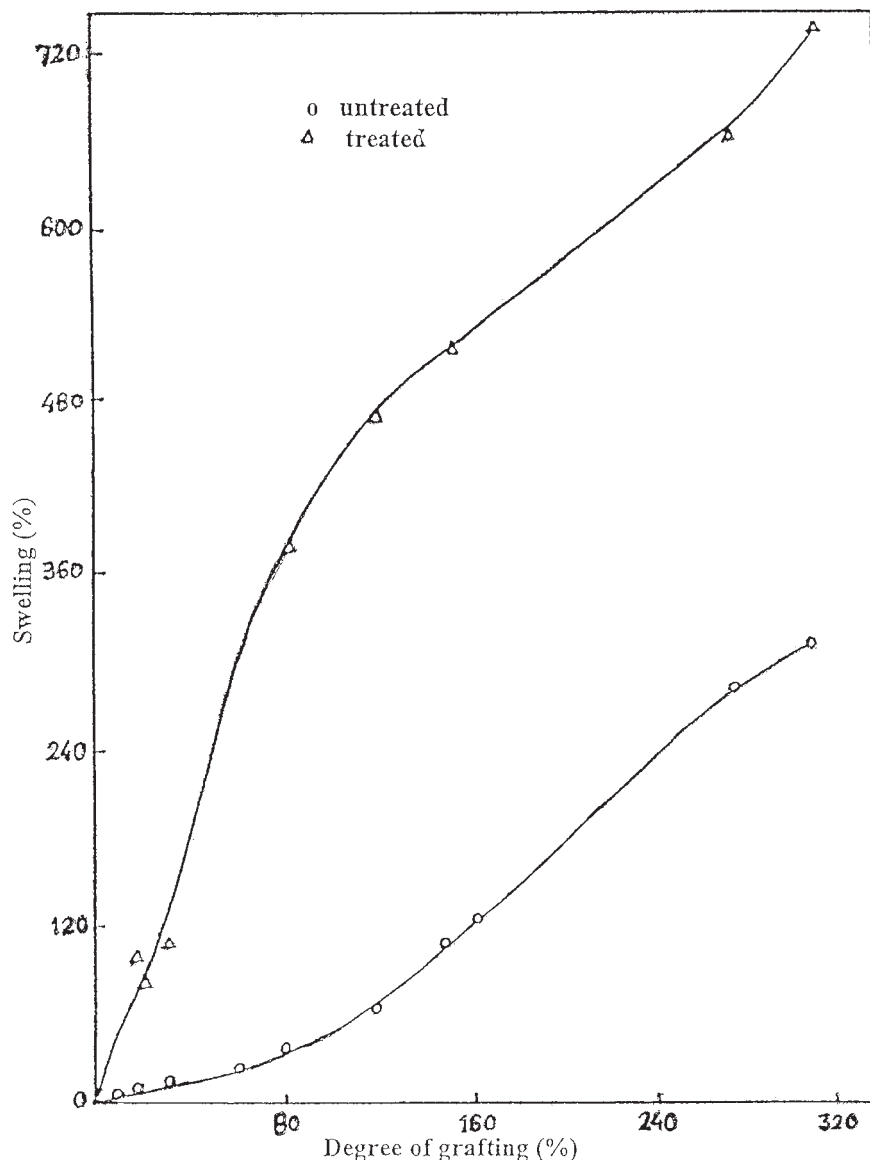


Figure 3 Effect of the membrane functional groups on the swelling of polypropylene-*g*-poly(acrylamide/vinyl acetate) and its KOH-treated form.

their KOH-treated forms. The diffraction curves of the graft copolymers did not show any new diffraction peak, and this implied the amorphous structure of these components (PAAm and PVAc). The crystalline reflections in the diffraction patterns of the graft copolymer occurred at identical diffraction angles, as in blank PP. Moreover, as the degree of grafting increased, the intensity of the crystalline diffraction peak decreased.

These results suggested that the inherent crystallinity of the backbone polymer was not employed by the graft copolymerization of VAc and AAm and that the grafting occurred only in the amorphous areas.¹⁷ The lack of intensity of the diffraction peak of the graft copolymer may have been due to partially destructive interference between X-ray beams reflected from dif-

ferent parts of the crystallites because of the increase in amorphousness with increasing grafting. Moreover, the incorporation of K^+ by the alkaline treatment of the grafted films did not affect the diffraction curve of the grafted PP copolymer, whereas it affected the intensities of the reflections because of the incorporation of the more polar groups.

Swelling behavior

Figure 3 shows the swelling percentage of PP grafted films as a function of the degree of grafting for both untreated and alkali-treated films. The swelling percentage increased with an increasing degree of grafting for both alkali-treated and untreated grafted films. However, at a given degree of grafting, the KOH-

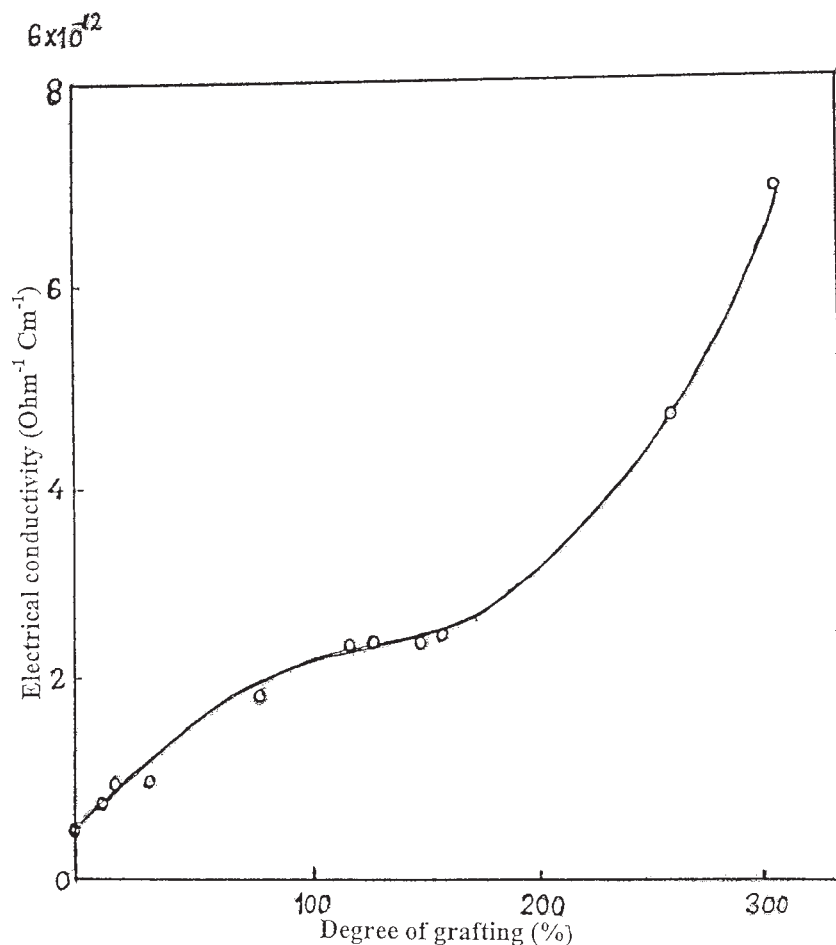


Figure 4 Electrical conductivity of polypropylene-g-poly(acrylamide/vinyl acetate) as a function of the degree of grafting.

treated films possessed a much higher swelling percentage than the untreated ones because of the improvement in the hydrophilic property of the samples with the treatment. The increase in the swelling percentage with the degree of grafting was attributed to the increase in the hydrophilicity of the graft copolymers with the increase in the number of amide groups, which were hydrophilic in nature. The alkaline treatment resulted in the hydrolysis of the poly(vinyl acetate) to poly(vinyl alcohol), and the amide groups were converted into highly solvated ionic units of the type —COOK . Adding electrolytic groups to the grafted side chains resulted in improved swelling behavior. On the other hand, the crystalline and amorphous changes in the graft copolymers were another important factor influencing the swelling percentage. The increase in the swelling percentage with an increasing grafting level may have also been due to the reduction in the crystallinity because the water molecules could easily penetrate the noncrystalline regions.

Electrical conductivity

In fact, the polymer substrate and graft chains of PAAm and PVAc were nonconductive materials. The

data given in Figure 4 show the change in the electrical properties of PP caused by the grafting process. The electrical conductivity increased as the degree of grafting increased. However, the order of magnitude of the electrical conductivity (10^{-12}) did not change even at a high degree of grafting. The change was due to the change in the polymer morphology caused by grafting with PAAm and PVAc, and the increase in the electrical conductivity was also due to the fact that the PAAm and PVAc graft chains acted as impurities.

Mechanical properties

Figure 5 shows the changes in the elongation and tensile strength at break for ungrafted and grafted PP films. The tensile strength increased with an increasing degree of grafting, but the elongation decreased. The decrease in the elongation and the improvement in the tensile strength were due to the formation of a crosslinked network structure, which increased with an increasing number of grafted chains and their increasing length. The rigidity of the graft copolymer increased with an increasing degree of grafting because of the crosslinking formation, which formed

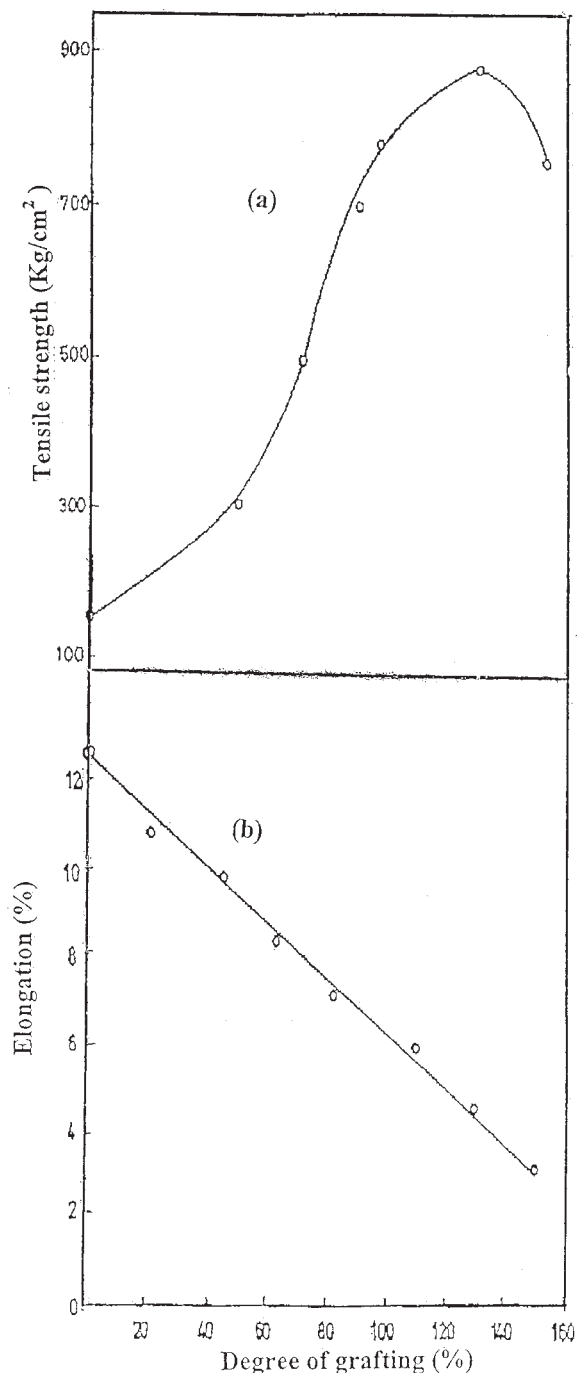


Figure 5 (a) Tensile strength and (b) elongation versus the degree of grafting for polypropylene-*g*-poly(acrylamide/vinyl acetate).

because of grafting and irradiation, which led to a restriction of the chain mobility.¹⁸

Scanning electron microscopy

An important property for the preparation of the membranes is the suitable distribution of grafted chains within the polymeric matrix. Micrographs of

sections of the ungrafted and grafted films are shown in Figure 6. A cross-sectional observation of the graft copolymer showed that, when the grafting yield was small, most of the grafted chains existed only near the surface regions of the film. With an increase in the grafting yield, the grafting regions were enlarged, and a homogeneous distribution was obtained.

It was proved that the graft polymerization took place predominantly on the polymer film surface and progressed inward through the grafted layer into the polymer matrix.

Permeability

The water permeability (expressed as the flow rate) at different pressures was measured for the prepared membranes and is shown in Figures 7 and 8. The permeability of water increased with the applied pressure. The observed permeability might have resulted from water penetration through PAAm chains and the formation of segmental gaps between the polymeric chains during membrane preparation. For a high grafting yield greater than 100%, the flow rate significantly decreased because of the formation of a crosslinked network structure, which restricted the diffusion of water. In addition, the concentration of hydrophobically grafted VAc increased as the grafting yield increased. As a result, the diffusion of water through the membrane decreased. Therefore, at a higher grafting yield, the permeability decreased, and much greater pressure was needed for water to diffuse. Thus, the membrane with 98% grafting exhibited a flow rate of 4.4 cc/s at a differential pressure of 0.48 psi, but the membrane with 190% grafting exhibited a flow rate of 0.008 cc/s at a differential pressure of 0.72 psi. The data revealed that a membrane with less than 100% grafting had higher permeability than a membrane with a higher grafting percentage.

Separation of cesium-134 and cobalt-60 from liquid waste

The possibility of the practical use of the prepared graft copolymers for the separation of cesium-134 and cobalt-60 from liquid radioactive wastes was investigated. The factors affecting the separation process, including the treatment time, degree of grafting, and temperature, were also studied.

Effect of the treatment time

The treatment time is a very important factor because of radiation safety, economics, and workers' exposure to radiation. Also, the efficiency of a membrane in waste treatment can be determined from the time required to adsorb the maximum capacity of radioisotopes by adsorption and/or chelation with its func-

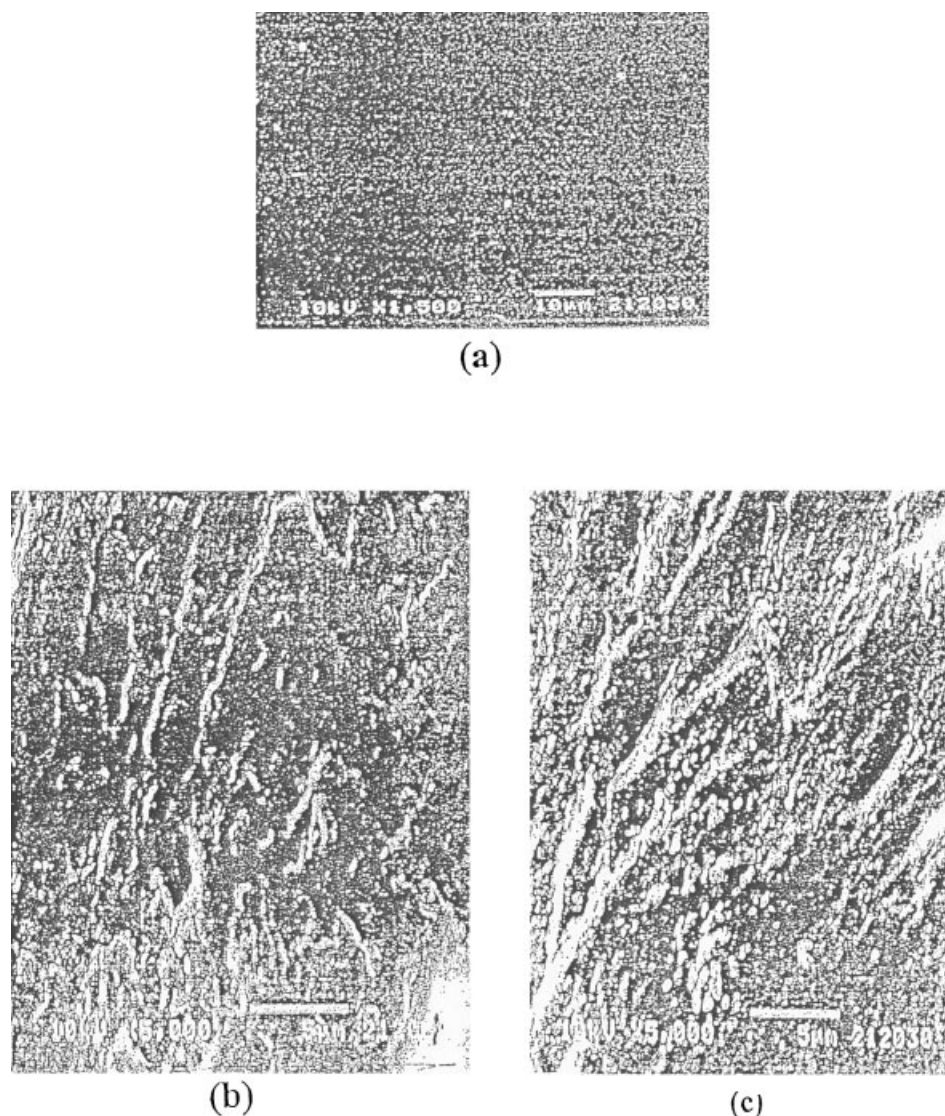


Figure 6 Section micrographs for blank PP and grafted PP: (a) blank PP, (b) 59% grafted polypropylene-g-poly(acrylamide/vinyl acetate), and (c) 159% grafted polypropylene-g-poly(acrylamide/vinyl acetate).

tional groups. The dependence of radioisotope uptake on the time for the prepared membranes is shown in Figure 9. The activity decreased (i.e., the radioisotope uptake increased) very quickly as the time increased to the maximum value within the first 30 min; then, it increased slowly and tended to level off after approximately 2 h. Moreover, under the same experimental conditions, the cesium-134 uptake was greater than that of cobalt-60 because cobalt-60 had small ionic radii and a higher charge density than cesium-134, and this led to a large hydrated radius and, therefore, low mobility; consequently, the adsorption capacity of cobalt-60 was lower than that of cesium-134. The data showed that the adsorption rate and maximum membrane capacity were higher for KOH-treated membranes than for untreated ones because of the enhanced diffusion rate of the radioisotope solution caused by the increased hydrophilicity.

Effect of the degree of grafting

The degree of grafting played an important role in determining the isotope uptake from the waste. Figure 10 shows the effect of different grafting percentages on the uptake of cesium-134 and cobalt-60 from their liquid radioactive wastes for untreated and KOH-treated PP under the same experimental conditions.

The amount of cesium-134 and cobalt-60 adsorbed by the membranes increased with an increasing degree of grafting. This behavior was observed for both untreated and KOH-treated grafted films. However, the adsorption capacity of KOH-treated membranes was higher than that of the untreated ones. These results suggested that the efficiency of these membranes was a function of the number of functional groups added to the grafted copolymer and their subsequent capacity to adsorb or chelate ions. Through

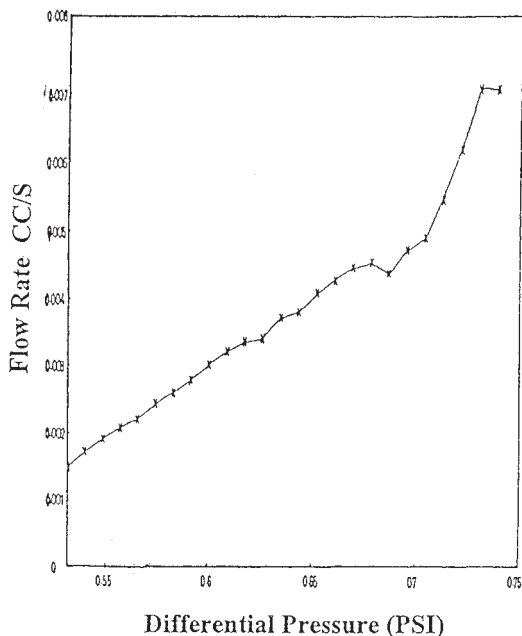


Figure 7 Permeability versus the differential pressure for polypropylene-g-poly(acrylamide/vinyl acetate) (degree of grafting = 190%).

the addition of carboxylate and hydroxyl groups via KOH treatment, the adsorption properties increased, as well as the chelating ligand derived from these groups.

Effect of the temperature

The temperature is another important factor affecting the chelation or adsorption of radioactive isotopes from waste by the membrane functional groups. Figure 11 shows the relationship between the uptake

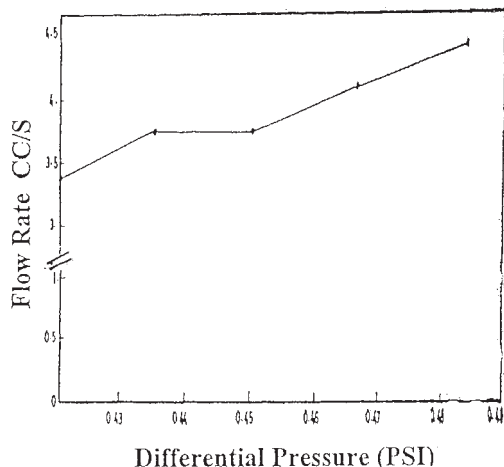


Figure 8 Permeability versus the differential pressure for polypropylene-g-poly(acrylamide/vinyl acetate) (degree of grafting = 98%).

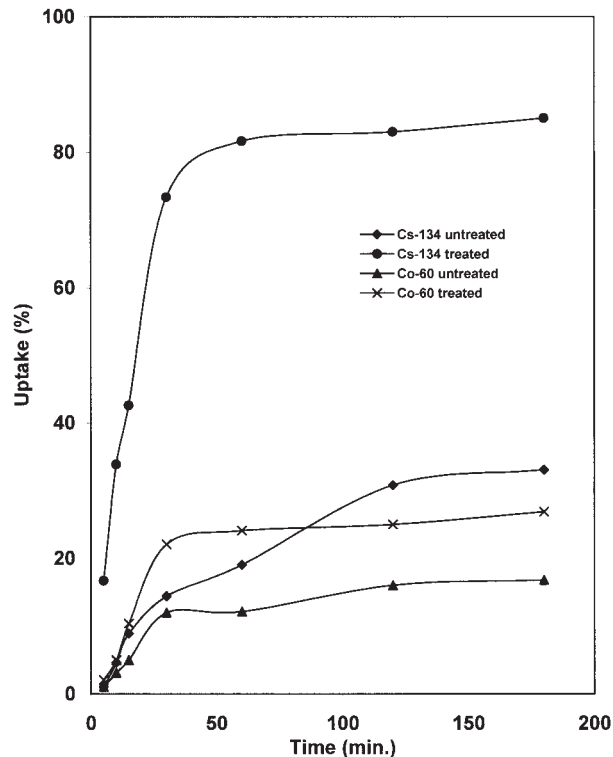


Figure 9 Effect of time on cesium-134 and cobalt-60 uptake by polypropylene-g-poly(acrylamide/vinyl acetate) and KOH-treated grafted PP (degree of grafting = 200%).

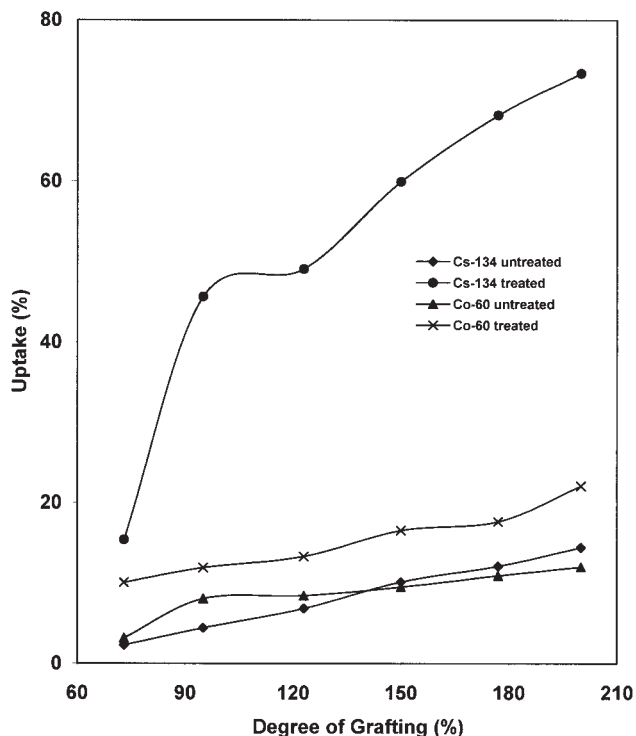


Figure 10 Effect of the degree of grafting on cesium-134 and cobalt-60 uptake by polypropylene-g-poly(acrylamide/vinyl acetate) and KOH-treated grafted PP (time = 30 min).

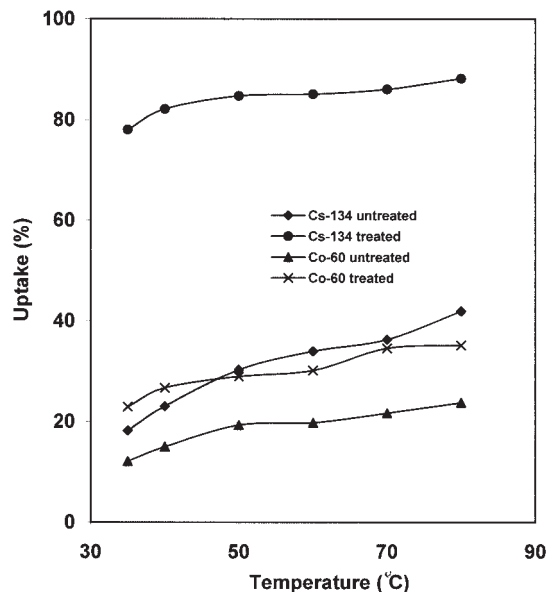


Figure 11 Effect of the temperature on cesium-134 and cobalt-60 uptake by polypropylene-g-poly(acrylamide/vinyl acetate) and KOH-treated grafted PP (degree of grafting = 200%, time = 30 min).

percentages of cesium-134 and cobalt-60 and the temperature. The uptake increased with increasing temperature for the untreated and KOH-treated grafted PP, and the maximum uptake was obtained at about 50°C. This could be attributed to the increase in the kinetic energy of the radioactive isotopes with the temperature and to the increase in the flexibility of the polymer graft chains. Consequently, the diffusion of the radioisotope solution into the membrane and the amount of radioactive isotopes taken by the membrane increased with the temperature.

CONCLUSIONS

The prepared graft copolymer possessed good adsorption properties for cesium-134 and cobalt-60, but it had

a higher affinity for cesium-134 than for cobalt-60. The maximum adsorption capacity was reached after 30 min, and this showed a high affinity toward such metal adsorption and advantages from a practical point of view. The adsorption process was affected by the temperature of the feed solution and the types of functional groups; KOH treatment enhanced the efficiency of the adsorption process. The grafted membranes possessed good mechanical strength, which may make such materials acceptable for practical use.

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