

# Synthesis and Characterization of Novel Grafted Amphoteric Poly(propylene) Fabrics

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**ABSTRACT:** Novel grafted amphoteric poly(propylene) (PP) fabrics were prepared by  $\gamma$ -irradiation, which induced grafting of methacrylic acid (MA) onto PP fabric. A subsequent reaction of PP-MA grafted chains with freshly prepared (3-chloro-2-hydroxypropyl)amine or diamine chloride compounds followed by purification yielded the corresponding amphoteric PP fabrics. The latter were characterized by elemental microanalysis for the determina-

tion of the percentage nitrogen. Fourier transform infrared and scanning electron microscopy were performed on methacrylic and amphoteric PP fabrics. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 88: 2504–2510, 2003

**Key words:** irradiation; poly(propylene) (PP); modification; functionalization of polymers; infrared spectroscopy; electron microscopy

## INTRODUCTION

Poly(propylene) (PP) is frequently used for thermoplastic materials, molding, and production of PP films at a moderate temperature because of its low heat resistance. It is difficult to dye PP fabric because of the lack of reactive functional groups in the polymer backbone. These drawbacks are successfully overcome by chemical modification of PP. The modification of PP can be achieved by graft copolymerization of various vinyl monomers using different radiation methods followed by chemical grafting techniques.

A review of the literature revealed that Chen et al.<sup>1,2</sup> grafted PP fabric with acrylic acid (AA) by preirradiation of PP fabric with  $\gamma$ -rays and electron beams in air and nitrogen gas, respectively. The effect of storage time of the preirradiated PP fabric on the degree of grafting was studied. The results indicated that the samples irradiated by  $\gamma$ -rays had a much higher degree of grafting than those treated with electron beams. Miyazaki et al.<sup>3</sup> modified PP fabric by creating hygroscopic and water repellency properties by a series of successive reactions. First, the hydrophilic groups were introduced into the nonwoven PP fabric by radiation, which induced graft copolymerization of AA or a mixture of sodium *p*-styrene sulfonate (SSS) and AA. Hygroscopic and wettability properties were obtained by treating the AA-grafted samples with sodium hydroxide solution. Next, a polyion complex was formed on the sodium acrylate surface by treating the (PP) surface with an aqueous solution of diocta-

decyl dimethylammonium bromide. Finally, the alkyl chain surface was further treated with CF<sub>4</sub> plasma to form a water-repellent surface. The structure of the modified surface was analyzed after each treatment by attenuated total reflection-Fourier transform infrared (ATR-FTIR) spectroscopy and X-ray photoelectron spectroscopy (XPS). Misra et al.<sup>4</sup> grafted methacrylic acid (MA) onto isotactic PP and studied the optimum grafting conditions. Yen et al.<sup>5</sup> investigated the copolymerization of MA, AA, and a mixture of AA and diglycol acrylate onto nonwoven PP fabric using an electron beam. The exchange adsorption properties of the ion-exchange fibers that were obtained with transition and rare earth metal ions were investigated. Several copolymers of methacrylic esters containing piperidine or dialkyl amino groups were prepared and proved to be very effective in surface dyeing of PP films and fibers.<sup>6,7</sup> Shaozao et al.<sup>8</sup> grafted 4-vinylpyridine (4VP) by preirradiation of nonwoven PP cloth. The grafts were characterized by infrared (IR) spectroscopy and scanning electron microscopy (SEM). The American Cyanamid Company<sup>9</sup> improved the dyeability of PP fibers modified with 4VP by treating the fibers with an anionic solution of dodecylsulfate (0.24–4% on weight fabric (owf)) at pH < 6 and 140–250°F. The treated fibers can be dyed with acid, basic, or vat dyes. Caldo<sup>10</sup> studied the dyeing properties of modified PP fibers by introducing amino groups. He found that the highest acid dye absorption was for the fibers containing piperazine amino groups, followed by those having an aliphatic tertiary amino moiety. Gawish et al.<sup>11</sup> grafted 2-(dimethylamino)ethyl methacrylate onto PP fabric by  $\gamma$ -irradiation. The different variable parameters affecting the grafting reaction, such as the irradiation doses, storage

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conditions, reaction time, monomer concentration, and temperature, were studied. The grafted PP was further quaternized with dimethyl sulfate or monochloroacetic acid. The grafted and quaternized fabrics were dyed with an acid dye. The moisture regain and dyeability were increased, and the mechanical properties were also determined.

The aim of the present work was the synthesis of a novel series of grafted PP fabrics based on the introduction of quaternary ammonium salt sites by a chemical modification technique. The synthesis was performed in two successive reactions. First, hydrophilic carboxylic acid groups were introduced into PP fabric by  $\gamma$ -irradiation and graft copolymerization with MA. The carboxylic acid groups that were formed on the PP fabric were allowed to react with freshly prepared solutions of (3-chloro-2-hydroxypropyl)amine, dioctylmethylamine, ethylene diamine, tetramethylethylene diamine, and cetyl tetramethylethylene diamine chloride in alkaline medium to yield the corresponding fabrics containing cationic groups that were purified by extraction with acetone and warm water. The prepared fabrics were characterized by elemental microanalysis, IR spectroscopy, and SEM. The application and properties of these amphoteric PP fabrics will be further studied in an independent paper.

## EXPERIMENTAL

Poly(propylene) fabric (100%) was obtained from Montedison Company. PP is highly isotactic and has the following specifications: warp, 238 dtex; fill, 251 dtex. MA was supplied by Merck Company and was used without further purification. Mohr's salt [ $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ ],  $\text{H}_2\text{SO}_4$ , HCL, acetone, methyl alcohol, triethylamine, tetramethylethylene diamine, ethylene diamine, dioctylmethyl amine, cetyl bromide, and epichlorohydrin were all laboratory grades chemicals.

### Technical methods

Cationic groups were introduced into PP by a series of successive reactions as follows. First, the PP-MA graft copolymer was prepared. Next, the chosen amine salts were prepared and reacted with epichlorohydrin to produce an amine hydrochloride epichlorohydrin condensate (AHE) [such as, *N*-(3-chloro-2-hydroxypropyl)amines or *N,N'*-bis(3-chloro-2-hydroxypropyl)amines] according to a previously described procedure.<sup>12,13</sup> Finally, the PP-MA grafts were condensed in alkaline medium with the previously prepared chloroquaternary ammonium compounds to produce their corresponding PP-MA ester AHE condensates.

### Preirradiation and grafting of MA onto PP

Before irradiation, PP fabric was extracted for 1 week with acetone to remove the antioxidants and ultravi-

olet stabilizers, which are inhibitors for the grafting process, and then air dried before irradiation. Irradiation was carried out in a cobalt-60 gamma accelerator. A preirradiation method was adopted. The dry fabric was exposed to an irradiation dose of 2 Mrad in the presence of air (dose rate 0.64 Mrad/h). The grafting experiments were performed in a closed Pyrex bottle, in the presence of air, in a shaking water bath. The required amounts of distilled water, Mohr's salt solutions ( $2.5 \times 10^{-3}M$ ), and  $\text{H}_2\text{SO}_4$  (0.2M)] and the MA monomer (10%) were added. The irradiated PP fabric was immersed in the monomer solution and thermostated at 95°C for 1 h. The grafted samples were thoroughly washed with methanol and distilled water to remove any traces of the homopolymer. The samples were dried, and the percentage grafting,  $G_o$ , was calculated as follows:

$$G_o (\%) = \frac{W_g - W_o}{W_o} \times 100 \quad (1)$$

where  $W_o$  and  $W_g$  are the weights of the initial and grafted weight, respectively.

### Synthesis of *N*-(3-chloro-2-hydroxypropyl)amine and *N,N'*-bis(3-chloro-2-hydroxypropyl)amine derivatives

The cationic reagents were synthesized<sup>12,13</sup> in a two-stage reaction. First, amine salts were synthesized and then the amine salt was condensed with epichlorohydrin.

#### Synthesis of amine salts

A series of mono- and diamine compounds were chosen for this study. The amine compounds used were triethylamine, dioctylmethylamine (0.1 mol), ethylene diamine, and tetramethylethylene diamine (0.2 mol). Cold hydrochloric acid (0.1 or 0.2 mol) was added in a dropwise manner to the mono- or diamine compounds, respectively, and the solution was continuously stirred in a cooling ice bath. A white crystalline precipitate was immediately formed, and the reaction was continued in the cold until complete precipitation of all the amine salt occurred.

#### Condensation of amine salts with epichlorohydrin

Epichlorohydrin (0.1 or 0.2 mol) was added in a dropwise manner with stirring to the freshly prepared amine salt, and the reaction was continued with stirring at 50°C for 1 h to yield the corresponding (3-chloro-2-hydroxypropyl)amine derivatives.

TABLE I  
Summary of Amines Hydrochloride, Amine Hydrochloride-Epichlorohydrin (AHE),  
and PP-MA-Ester-AHE Condensates

Amine Hydrochloride	Amine Hydrochloride-epichlorohydrin condensates (AHE)	PP-MA-ester-AHE Condensates
$\begin{array}{c} \text{C}_2\text{H}_5 \\   \\ \text{C}_2\text{H}_5-\text{N}^+\text{HCl} \\   \\ \text{C}_2\text{H}_5 \end{array}$ Triethylamine hydrochloride	$\begin{array}{c} \text{C}_2\text{H}_5 \\   \\ \text{Cl}-\text{CH}_2-\text{CH}-\text{CH}_2-\text{N}^+\text{C}_2\text{H}_5 \\   \quad   \\ \text{OH} \quad \text{C}_2\text{H}_5 \end{array} \quad \text{Cl}^-$ (3-Chloro-2-hydroxypropyl)triethyl ammonium chloride	$\begin{array}{c} \text{PP} \quad \quad \quad \text{C}_2\text{H}_5 \\ \quad \quad \quad   \\ \text{COOCH}_2-\text{CH}-\text{CH}_2-\text{N}^+\text{C}_2\text{H}_5 \\ \quad \quad   \quad   \\ \quad \quad \text{OH} \quad \text{C}_2\text{H}_5 \end{array} \quad \text{Cl}^-$ PP-MA-ester(2-hydroxypropyl)triethyl ammonium chloride (IVA)
$\begin{array}{c} (\text{CH}_2)_7-\text{CH}_3 \\   \\ \text{HCl} \text{N}^+-\text{CH}_3 \\   \\ (\text{CH}_2)_7-\text{CH}_3 \end{array}$ Dioctylmethyl amine hydrochloride	$\begin{array}{c} (\text{CH}_2)_7-\text{CH}_3 \\   \\ \text{Cl}-\text{CH}_2-\text{CH}-\text{CH}_2-\text{N}^+\text{CH}_3 \\   \quad   \\ \text{OH} \quad (\text{CH}_2)_7-\text{CH}_3 \end{array} \quad \text{Cl}^-$ (3-Chloro-2-hydroxypropyl)dioctylmethyl ammonium chloride	$\begin{array}{c} \text{PP} \quad \quad \quad (\text{CH}_2)_7-\text{CH}_3 \\ \quad \quad \quad   \\ \text{COOCH}_2-\text{CH}-\text{CH}_2-\text{N}^+\text{CH}_3 \\ \quad \quad   \quad   \\ \quad \quad \text{OH} \quad (\text{CH}_2)_7-\text{CH}_3 \end{array} \quad \text{Cl}^-$ PP-MA-ester(2-hydroxypropyl)dioctylmethyl ammonium chloride (IVB)
$\begin{array}{c} \text{CH}_3 \quad \quad \quad \text{CH}_3 \\   \quad \quad \quad   \\ \text{HCl} \text{N}^+(\text{CH}_2)_2\text{N}^+\text{HCl} \\   \quad \quad \quad   \\ \text{CH}_3 \quad \quad \quad \text{CH}_3 \end{array}$ Tetramethylethylene diamine dihydrochloride	$\begin{array}{c} \text{CH}_3 \quad \quad \quad \text{CH}_3 \\   \quad \quad \quad   \\ \text{Cl}-\text{CH}_2-\text{CH}-\text{CH}_2-\text{N}^+(\text{CH}_2)_2\text{N}^+\text{CH}_2-\text{CH}-\text{CH}_2\text{Cl} \\   \quad   \quad   \quad   \\ \text{OH} \quad \text{CH}_3 \quad \quad \text{CH}_3 \quad \quad \text{OH} \quad 2\text{Cl}^- \end{array}$ <i>N,N'</i> -bis(3-chloro-2-hydroxypropyl) tetramethylethylene diamine dichloride	$\begin{array}{c} \text{PP} \quad \quad \quad \text{CH}_3 \quad \quad \quad \text{CH}_3 \quad \quad \quad \text{PP} \\ \quad \quad \quad   \quad \quad \quad   \\ \text{COOCH}_2-\text{CH}-\text{CH}_2-\text{N}^+(\text{CH}_2)_2\text{N}^+\text{CH}_2-\text{CH}-\text{CH}_2\text{OH} \quad 2\text{Cl}^- \\ \quad \quad   \quad \quad   \quad \quad   \\ \quad \quad \text{OH} \quad \quad \text{CH}_3 \quad \quad \quad \text{CH}_3 \quad \quad \quad \text{OH} \end{array}$ PP-MA-ester(2-hydroxypropyl)tetramethylethylene diamine dichloride (VA)
$\begin{array}{c} \text{H} \quad \quad \quad \text{H} \\   \quad \quad \quad   \\ \text{HCl} \text{N}^+(\text{CH}_2)_2\text{N}^+\text{HCl} \\   \quad \quad \quad   \\ \text{H} \quad \quad \quad \text{H} \end{array}$ Ethylene diamine dihydrochloride	$\begin{array}{c} \text{H} \quad \quad \quad \text{H} \\   \quad \quad \quad   \\ \text{Cl}-\text{CH}_2-\text{CH}-\text{CH}_2-\text{N}^+(\text{CH}_2)_2\text{N}^+\text{CH}-\text{CH}-\text{CH}_2\text{Cl} \quad 2\text{Cl}^- \\   \quad   \quad   \quad   \\ \text{OH} \quad \text{H} \quad \quad \text{H} \quad \quad \text{OH} \end{array}$ <i>N,N'</i> -bis(3-chloro-2-hydroxypropyl)ethylene diamine dichloride	$\begin{array}{c} \text{PP} \quad \quad \quad \text{H} \quad \quad \quad \text{H} \quad \quad \quad \text{PP} \\ \quad \quad \quad   \quad \quad \quad   \\ \text{COOCH}_2-\text{CH}-\text{CH}_2-\text{N}^+(\text{CH}_2)_2\text{N}^+\text{CH}_2-\text{CH}-\text{CH}_2\text{OH} \quad 2\text{Cl}^- \\ \quad \quad   \quad \quad   \quad \quad   \\ \quad \quad \text{OH} \quad \quad \text{H} \quad \quad \quad \text{H} \quad \quad \quad \text{OH} \end{array}$ PP-MA-ester(2-hydroxypropyl)ethylene diamine dichloride (VB)
$\begin{array}{c} \text{CH}_3 \quad \quad \quad \text{CH}_3 \\   \quad \quad \quad   \\ \text{HCl} \text{N}^+(\text{CH}_2)_2\text{N}^+\text{C}_{16}\text{H}_{33}\text{Cl} \\   \quad \quad \quad   \\ \text{CH}_3 \quad \quad \quad \text{CH}_3 \end{array}$ Cetyl-chloride tetramethylethylene diamine hydrochloride	$\begin{array}{c} \text{CH}_3 \quad \quad \quad \text{CH}_3 \\   \quad \quad \quad   \\ \text{Cl}-\text{CH}_2-\text{CH}-\text{CH}_2-\text{N}^+(\text{CH}_2)_2\text{N}^+\text{C}_{16}\text{H}_{33} \quad 2\text{Cl}^- \\   \quad   \quad   \\ \text{OH} \quad \text{CH}_3 \quad \quad \text{CH}_3 \end{array}$ <i>N</i> -(3-chloro-2-hydroxypropyl)- <i>N</i> -cetyl-tetramethylethylene diamine dichloride	$\begin{array}{c} \text{PP} \quad \quad \quad \text{CH}_3 \quad \quad \quad \text{CH}_3 \\ \quad \quad \quad   \quad \quad \quad   \\ \text{COOCH}_2-\text{CH}-\text{CH}_2-\text{N}^+(\text{CH}_2)_2\text{N}^+\text{C}_{16}\text{H}_{33} \quad 2\text{Cl}^- \\ \quad \quad   \quad \quad   \\ \quad \quad \text{OH} \quad \quad \text{CH}_3 \quad \quad \quad \text{CH}_3 \end{array}$ PP-MA-ester(2-hydroxypropyl)cetyl-tetramethylethylene diamine dichloride (VC)
$\begin{array}{c} \text{H} \quad \quad \quad \text{H} \\   \quad \quad \quad   \\ \text{HCl} \text{N}^+(\text{CH}_2)_2\text{N}^+\text{C}_{16}\text{H}_{33}\text{Cl} \\   \quad \quad \quad   \\ \text{H} \quad \quad \quad \text{H} \end{array}$ cetyl-chloride ethylene diamine hydrochloride	$\begin{array}{c} \text{H} \quad \quad \quad \text{H} \\   \quad \quad \quad   \\ \text{Cl}-\text{CH}_2-\text{CH}-\text{CH}_2-\text{N}^+(\text{CH}_2)_2\text{N}^+\text{C}_{16}\text{H}_{33} \quad 2\text{Cl}^- \\   \quad   \quad   \\ \text{OH} \quad \text{H} \quad \quad \text{H} \end{array}$ <i>N</i> -(3-chloro-2-hydroxypropyl)- <i>N'</i> -cetyl-ethylene diamine dichloride	$\begin{array}{c} \text{PP} \quad \quad \quad \text{H} \quad \quad \quad \text{H} \\ \quad \quad \quad   \quad \quad \quad   \\ \text{COOCH}_2-\text{CH}-\text{CH}_2-\text{N}^+(\text{CH}_2)_2\text{N}^+\text{C}_{16}\text{H}_{33} \quad 2\text{Cl}^- \\ \quad \quad   \quad \quad   \\ \quad \quad \text{OH} \quad \quad \text{H} \quad \quad \quad \text{H} \end{array}$ PP-MA-ester(2-hydroxypropyl)cetyl-ethylene diamine dichloride (VD)

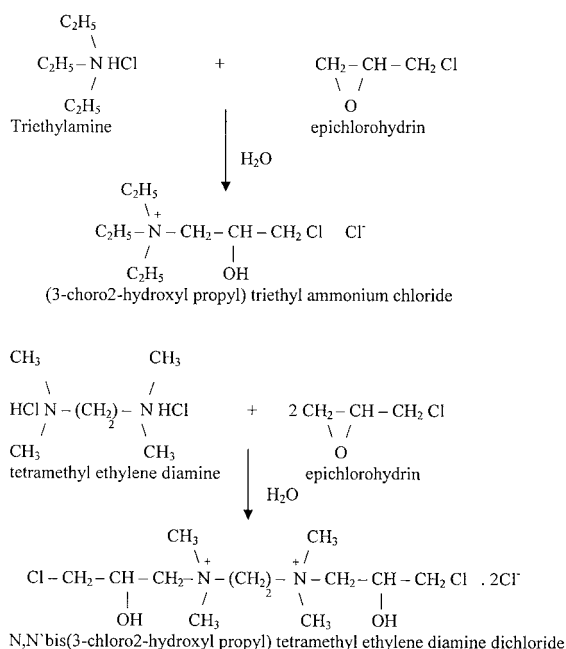
### Synthesis of *N*-(3-chloro-2-hydroxypropyl)cetyl-tetramethylethylene diamine

Tetramethylethylene diamine (0.1 mol) was mixed with cetyl bromide (0.1 mol). The reaction mixture was stirred at the reflux temperature for 5 h at which time a solid product was obtained. Cold hydrochloric acid (0.1 mol) was added with stirring to the previously prepared amine derivative that was stored on ice. The amine salt

was dissolved in 10 mL of isopropyl alcohol and 0.1 mol epichlorohydrin and refluxed for 1 h at 50°C.

### PP-MA ester of (2-hydroxypropyl)amine

The PP-MA graft copolymer of a specified graft yield was immersed in a flask containing the previously freshly prepared cationic reagents and refluxed in alkaline medium at 90°C for 1 h with continuous stir-



**Scheme 1** Condensation of amine hydrochloride salts and epichlorohydrin.

ring. The fabric was then washed with water and acetone and finally dried at room temperature and over  $\text{P}_2\text{O}_5$  in a vacuum desiccator.

The percentage weight increase due to the condensation of cationic groups onto the PP-MA graft was determined based on the dried weight as follows:

$$\% \text{ increase in weight} = \frac{W_f - W_g}{W_g} \times 100 \quad (2)$$

where  $W_f$  is the final weight after reaction and  $W_g$  is the initial graft weight. The completion of the quaternization reaction was calculated as follows:

$$\text{percentage reaction completion} = \frac{W_1}{W_2} \times 100 \quad (3)$$

where  $W_1$  is the actual weight increase due to the quaternization reaction and  $W_2$  is the theoretical weight of the alkylating agent needed for complete quaternization of the grafted MA.

The weights of grafted MA and the alkylating agent required were computed as follows:

$$\begin{aligned} \text{weight of MA} \\ = (\text{graft yield} \times \text{grafted sample weight})/100 \end{aligned} \quad (4)$$

$$\begin{aligned} \text{weight of alkylating agent} \\ = \frac{\text{MW of alkylating agent} \times \text{weight of MA}}{\text{MW of MA}} \end{aligned} \quad (5)$$

## IR spectroscopic analysis

The IR analysis was carried out with an FTIR spectrophotometer (a Jasco FTIR 300E). The samples were ground to a very fine powder, mixed with highly dried KBr powder, and pressed to transparent disks.

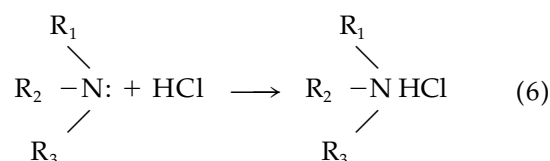
## Nitrogen analysis

Nitrogen content was estimated by the micro kjeldahl method.

## RESULTS AND DISCUSSION

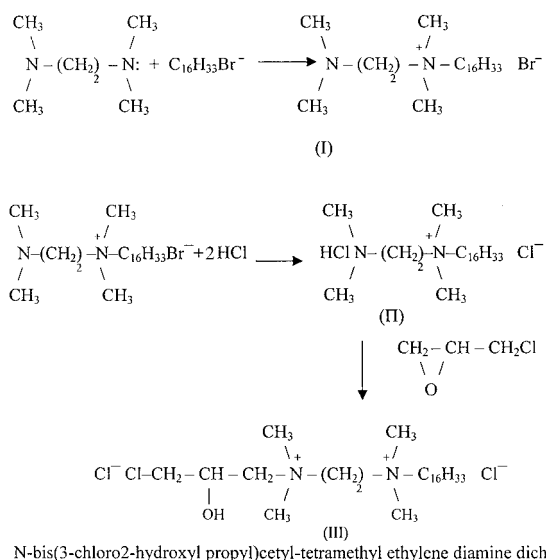
### Synthesis of amine hydrochloride salts derivatives

In the case of the monoamine compounds, the reaction takes place between equimolar amounts of the hydrochloric acid and amine in a cooling ice medium (Table I) according to the following equation:

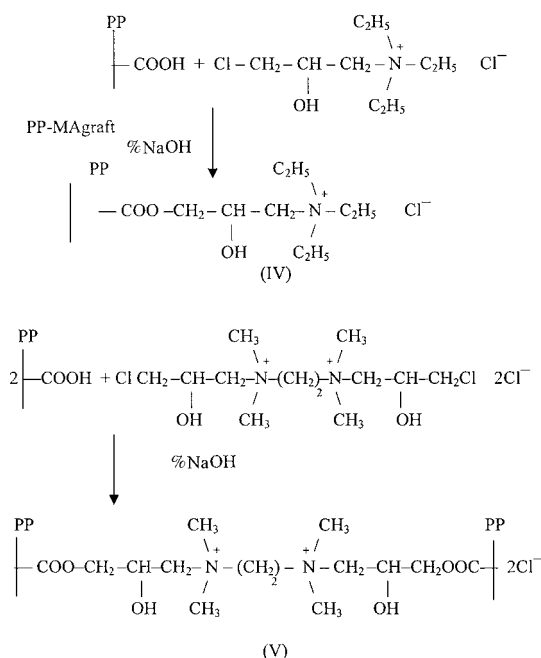


where  $\text{R}_1 = \text{R}_2 = \text{R}_3 = \text{C}_2\text{H}_5$  for triethylamine, or  $\text{R}_1 = \text{CH}_3$  and  $\text{R}_2 = \text{R}_3 = (\text{CH}_2)\text{CH}_3$  for dioctylmethyl amine.

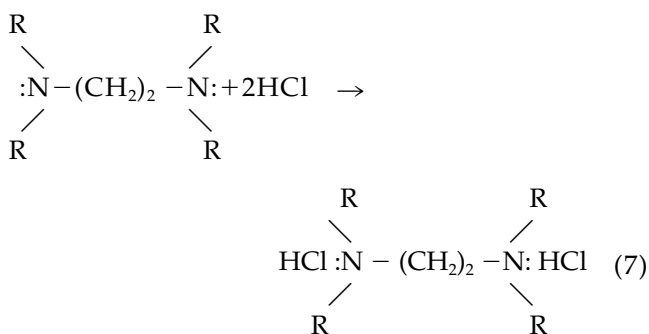
In the case of the diamine compounds, 1 mol of diamine reacts with 2 mol of hydrochloric acid as follows:



**Scheme 2** Synthesis of I, II, and III.



Scheme 3 Synthesis of IV and V.



where R = CH<sub>3</sub> for tetramethylethylenediamine, and R = H for ethylenediamine.

### Condensation of amine hydrochloride salts and epichlorohydrin

The condensation reaction takes place between equimolar amounts of amine salt and epichlorohydrin

for monoamine in the presence of water at a reflux temperature of 50°C, as shown in Scheme 1.

### Synthesis of *N*-(3-chloro-2-hydroxypropyl)cetyl-tetramethylethylenediamine dichloride

The synthesis of this compound was done in two reaction steps (see Scheme 2). First, the cetyl-tetramethylethylenediamine bromide was formed by reacting equimolar amounts of cetyl bromide and tetramethylethylenediamine at their reflux temperature until a solid product (I) was obtained. Second, the required amount of cold hydrochloride was added to I in a dropwise manner with continuous stirring to yield the hydrochloride derivative of II. The latter compound was allowed to react with 1 mol of epichlorohydrin to yield III.

### PP-MA ester of (2-hydroxypropyl)ammonium chloride

The PP-MA graft copolymers with different graft yields were allowed to react with (3-chloro-2-hydroxypropyl)amine to introduce quaternary ammonium groups into PP according to the reactions shown in Scheme 3.

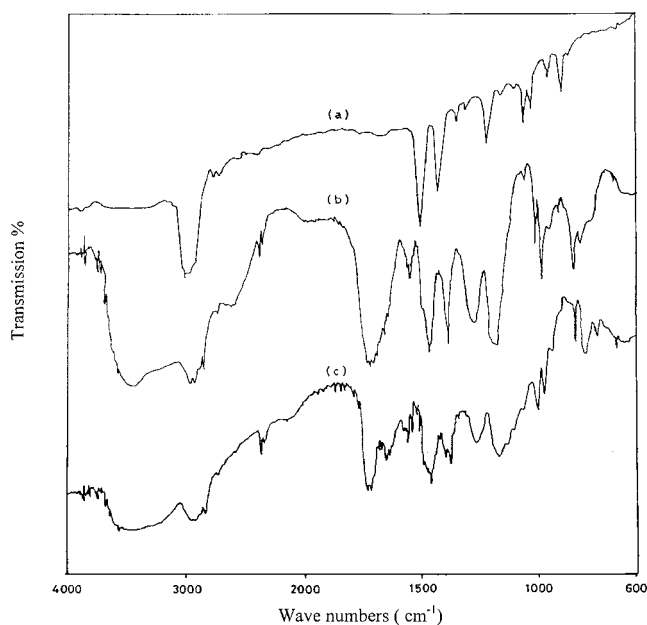
### Reaction of PP-MA grafts with the cationic agents

Condensation of MA grafts with different cationic agents (Table II) proved that the *N,N'*-bis(3-chloro-2-hydroxypropyl)tetramethylethylenediammonium dichloride (VA) and (3-chloro-2-hydroxypropyl)triethyl ammonium chloride (IVA) were the most active agents because their reactions were 61.2 and 57.7% complete, respectively. The (3-chloro-2-hydroxypropyl)dioctylmethyl ammonium chloride (IVB) reaction was 41.96% complete. The reactivities of cationic agents with the MA graft were of the following order (Table II): VA > IVA > IVB > VB > VC ~ VD.

The resulting textiles are amphoteric because of the presence of both cationic groups, resulting from the amine added to the PP-MA graft, and the presence of anionic groups, from the MA that did not graft to the PP fabric.

TABLE II  
Reaction Completion for PP-MA-Ester Condensates

PP-MA Graft %	PP-MA-Ester-AHE Condensate	Weight Increase by Cationic Agent, %	Reaction Completion %
53.5	PP-MA-ester(2-hydroxypropyl)triethyl ammonium chloride (IVA)	26.2	57.7
61.4	PP-MA-ester(2-hydroxypropyl)dioctylmethyl ammonium chloride(IVB)	27.5	41.95
60.04	PP-MA-ester bis(2-hydroxypropyl)tetramethylethylenediamine dichloride (VA)	22.16	61.2
21.5	PP-MA-ester bis(2-hydroxypropyl)ethylenediamine dichloride (VB)	12.31	37.86
15	PP-MA-ester(2-hydroxypropyl)cetyl-tetramethylethylenediamine dichloride (VC)	6.3	8.4
16.1	PP-MA-ester(2-hydroxypropyl)cetyl-ethylene diamine dichloride (VD)	5.8	8.9



**Figure 1** Infrared spectra of (a) PP fabric, (b) PP-MA grafted (38%), and (c) PP-MA-ester-AHE condensate (IVA).

### Infrared microscopic analysis

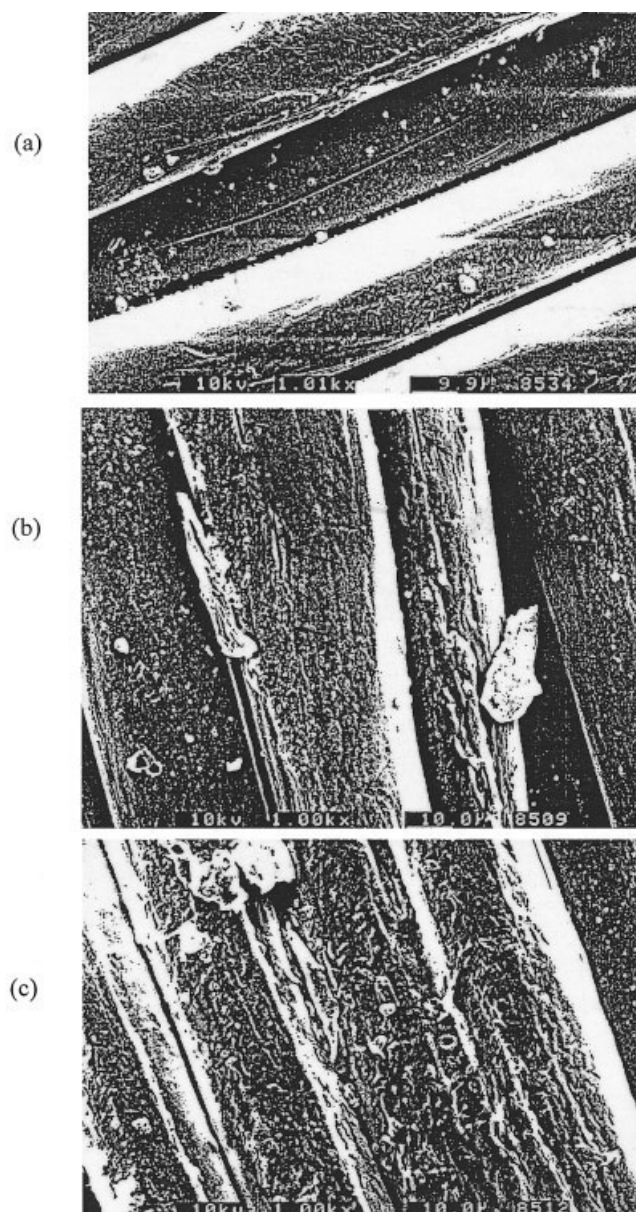
The IR<sup>14</sup> spectra of PP, PP-MA graft fabric, and PP-MA ester AHE condensates of IVA are shown in Figures 1a–1c, respectively. Pure PP shows absorption bands at 2700–3000  $\text{cm}^{-1}$ , arising from the C–H stretching of CH, CH<sub>2</sub>, and CH<sub>3</sub>, and at 1500–1400  $\text{cm}^{-1}$ , due to CH<sub>2</sub> scissors deformation and CH<sub>3</sub> asymmetric deformation.

The multiple weak bands due to the CH<sub>3</sub> wagging vibration of a normal hydrocarbon chain are at 1340–1190  $\text{cm}^{-1}$ . New bands that characterize PP-MA or PP-MA-ester-AHE condensates of fabric IVA (Fig. 1b and 1c) appeared as follows: (1) The IR spectra of graft PP-MA shows a broad OH stretch band at 3200–3600  $\text{cm}^{-1}$ , a C=O stretch of the carboxylic group at 1720  $\text{cm}^{-1}$ , and a C–O stretch at 1250  $\text{cm}^{-1}$ , indicating that MA was incorporated as a graft into PP. (2) The PP-MA-ester-AHE condensates of fabric IVA result in some major changes, with the appearance of a twin peak at 1720–1740  $\text{cm}^{-1}$ , which is attributed to the carbonyl group of the ester. The carboxylate ion group shows a strong asymmetric absorption band near 1620  $\text{cm}^{-1}$  and a weaker symmetric band near 1400  $\text{cm}^{-1}$ . An absorption band for CH<sub>2</sub>–N is at 1470–1490  $\text{cm}^{-1}$ , confirming the condensation of the quaternary ammonium salt with MA. The peaks in the region 1000–1220  $\text{cm}^{-1}$  are due to two asymmetric coupled vibrations, such as C–C(=O)–O and O–C–C for the ester group. Also, at the same regions of 1220 and 1050  $\text{cm}^{-1}$  are due to the secondary alcohol and N-alkyl group of the quaternary salt, respectively. The band at 800  $\text{cm}^{-1}$  is due to the N<sup>+</sup>–C stretching vibration

(asymmetric peak) of cationic PP, which confirms the presence of a quaternary ammonium salt group.

### Scanning electron microscopy

The scanning electron micrographs (SEM) showed a marked change compared with the original ungrafted fabric (Fig. 2). Extensive fractures are seen along the fabric, which provide more surface area for the fabric, thus increasing the amount of amine condensates. Some fragments branching from the main fabric (Fig. 2, VA) and a heavy spongy structure resulting from the grafting of MA and amine condensate (Fig. 2, VC) are also revealed.



**Figure 2** Scanning electron photomicrograph (1000 $\times$ ) of (a) original PP fabric, (b) PP-MA-AHE condensate (VA), and (c) PP-MA-AHE condensate (VC).

**TABLE III**  
**Nitrogen content of PP-MA-Ester-AHE Condensates**

PP-MA-Ester-AHE Condensate	Nitrogen, %
PP-MA-ester(2-hydroxypropyl)triethyl ammonium chloride (IVA)	2.17
PP-MA-ester(2-hydroxypropyl)dioctylmethyl ammonium chloride (IVB)	1.55
PP-MA-ester bis(2-hydroxypropyl)tetramethylethylene diamine dichloride (VA)	2.4

### Elemental microanalysis

The nitrogen content of PP-MA-ester AHE fabric, shown in Table III for some chosen fabric samples (Table II), is in the range 1.55–2.4%, thus conforming the condensation of the cationic amine agents onto PP-MA.

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