

# Photoinduced grafting of vinyl benzyl trimethyl ammonium chloride on polyester nonwoven fabric with surfactant coating and its anion-exchange properties

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**ABSTRACT**: Vinyl benzyl trimethyl ammonium chloride (VBTAC) could be efficiently and stably grafted onto polyester fiber coated with a surfactant polyester (PET) by a photoirradiation-induced graft polymerization with benzophenone as the photoinitiator without any cografting monomer required. The degree of VBTAC grafting could be controlled simply by the irradiation time and concentration of VBTAC in the monomer solution. The anion-exchange capacity (AEC) of the PET-g-VBTAC fabrics increased with increasing degree of grafting up to  $80 \pm 5\%$  and then leveled off. The maximum AEC of PET-g-VBTAC was 2.2 mequiv/g; this was similar to that of a commercial anion-exchange resin ( $2.16 \pm 0.04$  mequiv/g) and much higher than those of nylon-g-VBTAC-2-hydroxyethyl methacrylate fabrics ( $\leq 1.0$  mequiv/g) prepared with a conventional cografting system. The grafted fabric was characterized by Fourier transform infrared spectroscopy, thermogravimetric analysis, and scanning electron microscopy studies, and the sorption selectivity for anions and regeneration efficiency were estimated. The results suggest that the grafting system, in which VBTAC alone was grafted onto PET fiber coated with surfactant, was more practical and effective for the preparation of the VBTAC-containing anion exchanger, and the resulting PET-g-VBTAC fabrics could be used as an effective anion exchanger. © 2014 Wiley Periodicals, Inc. J. Appl. Polym. Sci. **2015**, *132*, 41674.

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#### INTRODUCTION

Anion-exchange resins have been widely used in laboratories and in industry for the purification for ultrapure water, the removal of environmentally important anionic contaminants from wastewater, the concentration of toxic substances for bioassay or analysis, the recovery of particular organic acids, and so on.<sup>1–4</sup> Many kinds of anion-exchange groups added onto the surfaces of matrix polymers, such as quaternary ammonium groups or primary to tertiary amino groups, produce different selectivity for anions.<sup>5,6</sup> All of the techniques used for preparation of anionexchange materials are both costly and time consuming because indirect and complex processes, including the polymerization of monomers and subsequent chemical modifications, are required for the introduction of anion-exchange functional groups into the polymer chains.<sup>7–9</sup> These processes generate a large amount of raw material waste and often require pretreatment, such as chlorination, the addition of a catalyst, and/or high temperatures; this further increases the production costs.

Attempts have been made to graft vinyl benzyl trimethyl ammonium chloride (VBTAC) onto substrate polymers either to directly introduce anion-exchange functionality<sup>10,11</sup> or to impart flame retardant properties.<sup>12,13</sup> We grafted VBTAC onto a nylon nonwoven fabric along with 2-hydroxyethyl methacrylate (HEMA) by photoirradiation and demonstrated that the grafted VBTAC allowed anion-exchange functionality on the surface of substrate without any further modification.<sup>14</sup> However, there are several problems that must be solved to make VBTAC graft polymerization more effective for the preparation of anion exchangers. One of the major problems limiting its wider application is the fact that VBTAC alone is difficult to graft onto a hydrophobic substrate because VBTAC has a highly ionizable group with a large hydration sphere incompatible with a

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**Figure 1.** Effect of the surfactant coating on the degree of grafting of VBTAC onto the PET fibers.

hydrophobic polymer; thus, the presence of a comonomer such as HEMA is essential for the grafting of VBTAC onto hydrophobic substrate polymers.<sup>10–13</sup> This cografting system is difficult to control to obtain a sufficient density of VBTAC groups in the graft chain, and this, thereby, limits the anion-exchange capacity (AEC) of the resulting product because the cografted HEMA group does not contribute to anion-exchange functionality.<sup>14</sup>

In this study, VBTAC was directly grafted onto a polyester fiber coated with a surfactant polyester (PET) without any comonomer by photoirradiation-induced graft polymerization with benzophenone (BP) as the photoinitiator to overcome the limitations of a conventional cografting system. The effects of the VBTAC concentration and photoirradiation time on the degree of grafting and AEC of the product were investigated to determine the optimal grafting conditions. The physicochemical properties, sorption selectivity for anions, and regeneration efficiency of the grafted fabric were examined.

#### **EXPERIMENTAL**

#### Materials and Reagents

The PET nonwoven fabric (80 g/m<sup>2</sup>) to be used as the substrate polymer for grafting was from Samsung Nonwoven Fabric Co. (Korea); it was coated with a nonionic surfactant based on a sorbitan alkyl ester to improve the water uptake ability of the PET nonwoven fabric. Surfactant-coated PET nonwoven fabric is widely used to produce personal hygiene items and cleaning supplies. The PET nonwoven fabric was used as received. VBTAC (Acros Organics, Belgium) and BP (Yakuri Pure Chemical Co., Japan) were used as the monomer and photoinitiator, respectively. Methanol (MeOH) and deionized water were used as solvents. All of the reagents were of higher grade and were used without further purification. Polystyrene-based PA308 resin  $[-N^+(CH_3)_3Cl^-$ , Samyang Co., Korea], which is a strong base anion-exchange resin, was used as a comparative sorbent.

#### **Photografting Procedure**

The PET fabric was cut into pieces with dimensions of 1 imes10 cm<sup>2</sup> and then dried at 60°C before grafting. The monomer solution was prepared with 80/20 v/v % water/MeOH as the solvent and 0.25-0.9M VBTAC, and 0.2 w/v % BP. One piece of PET fabric was placed in a Pyrex glass tube containing 20 mL of monomer solution, purged with nitrogen gas to eliminate oxygen, and then sealed. The tube was exposed to UV light under predetermined experimental conditions. The light source used was a 400-W high-pressure mercury lamp (Miya Electric Co., Korea), and the glass tubes were rotated and simultaneously revolved around the light source at a distance of 10 cm. After the grafting reaction, the samples were removed from the glass tubes, washed three times with hot water, then extracted with MeOH for 3 h in a Soxhlet apparatus to remove unreacted monomer and homopolymer, and dried at 60°C to a constant weight. The degree of grafting was calculated from the weight gain:

Degree of grafting (%) = 
$$\frac{100(W_1 - W_0)}{W_0}$$
. (1)

where  $W_0$  and  $W_1$  are the weights of the substrate and grafted fabric, respectively. At least three parallel experiments were carried out for each condition.

#### Characterization

The AEC (milliequivalents per gram dry fabric) of the PET fabric grafted by VBTAC (PET-g-VBTAC) was determined by the standard method with a 0.2M NH<sub>4</sub>Cl and 0.2M KNO<sub>3</sub> solution.<sup>15,16</sup> AEC was calculated with the following equation:

AEC (mequiv/g) = 
$$\frac{V_s C_s - V_{en} C_{en}}{W_1}$$
 (2)

where  $W_1$  is the weight of PET-g-VBTAC (g);  $V_s$  and  $V_{en}$  are the volumes of collected KNO<sub>3</sub> and entrained NH<sub>4</sub>Cl (L), respectively; and  $C_s$  and  $C_{en}$  are the concentrations of Cl<sup>-</sup> in collected KNO<sub>3</sub> and entrained NH<sub>4</sub>Cl (mequiv/L), respectively.

The chemical structures of the substrate PET fabric, surfactantcoated PET fabric, and PET-g-VBTAC fabric were analyzed with an IR spectrophotometer (Shimadzu IR-435, Japan) and a solid  $^{13}$ C-NMR spectrometer (Varian UnityINFINITYplus 200). The thermal decomposition behavior of the different fabrics was determined with a thermogravimetric analyzer (Mettler-Toledo TGA/DSC 1). All measurements were performed under a nitrogen atmosphere with a gas flow of 20 mL/min through the heating of the material from room temperature to 600°C at a heating rate of 10°C/min. The surface morphology of the PETg-VBTAC fabric was observed with field emission scanning electron microscopy (SEM; Hitachi S-4800, Japan).

The sorption selectivity among coexisting anions for the PET-g-VBTAC fabric was studied in batch conditions where 0.2 g of each sorbent was added to a 250-mL flask containing 100 mL of aqueous solution mixed equimolar anions. After the sorption, the solid and liquid phases were separated, and the anion concentrations in the liquid phase were measured with ion chromatography (Waters LC). The sorption capacity (q; mmol/g) was calculated with the following equation:



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Figure 2. Possible mechanism of the grafting of VBTAC onto the PET fibers by photoinduced graft copolymerization. S, singlet state; T, triplet state.

$$q = \frac{V(C_0 - C)}{W_1}$$
(3)

where  $C_0$  and C are the initial and equilibrium liquid-phase concentrations of anions (mmol/L), respectively; V is the volume of solution (L); and  $W_1$  is the dry weight of the sorbent used (g).

For the regeneration study, the PET-g-VBTAC used for the determination of AEC was regenerated by washing with a 0.2M KNO<sub>3</sub> solution once more and then reused for determination purposes. The determination and regeneration cycles were repeated five times. After each cycle, the PET-g-VBTAC was washed with deionized water and dried.

#### **RESULTS AND DISCUSSION**

#### Grafting of VBTAC onto PET and Its AEC

The effect of the surfactant coating on the grafting of VBTAC onto the PET fiber was first investigated. Figure 1 shows the degrees of grafting onto the PET fiber with or without a surfactant coating obtained with 0.7M VBTAC solution at  $60^{\circ}$ C for photoirradiation times in the range 1–4 h. As shown in Figure 1, the grafting of VBTAC onto the PET fiber was highly enhanced when the PET fiber was coated with surfactant. It is known that VBTAC is difficult to graft onto hydrophobic polymers because VBTAC has a highly ionizable group with a large hydration sphere that is incompatible with hydrophobic polymers<sup>10–13</sup> and that BP is excited to the triplet state by photoirradiation; this can extract a hydrogen atom from the hydrocarbon chain with a suitable hydrogen donor to yield free radicals capable of initiating the graft polymerization of the

monomers.<sup>17</sup> In the right of these facts, the possible reasons for the high grafting of VBTAC onto the PET fiber when the surfactant was coated are as follows: the surfactant coated may have played a role in the absorbance of the monomer in the PET fibers that may have formed a graft chain struck onto the PET fibers on photoirradiation, played a role as a hydrophilic comonomer such as HEMA that may have formed a cografting



**Figure 3.** Effect of the reaction temperature on the degree of grafting of VBTAC onto the PET fabric.





Figure 4. Effects of the irradiation time and monomer concentration on the (a) degree of grafting of VBTAC onto the PET fabric and (b) AEC of the grafted fabric.

chain struck with VBTAC onto the PET fibers on photoirradiation, and/or played a role as sites for producing free radicals that may have formed a graft chain struck onto the surfactant coating layer of the PET fiber on photoirradiation. On the basis of these possibilities and with reference to other studies,<sup>18,19</sup> the main possible procedure involved in photoinduced graft polymerization of VBTAC onto the PET fiber is shown in Figure 2.

To determine the optimum conditions for the photografting of VBTAC onto PET fiber, the effects of the photoirradiation time, reaction temperature, and concentrations of VBTAC in the monomer solution on the graft polymerization and AEC of the PET-g-VBTAC fabric were investigated. Figure 3 shows the

degrees of grafting onto the PET fiber obtained with 0.5M VBTAC, 0.2 w/v % BP, and 20 v/v % MeOH in deionized water at a photoirradiation time of 1 h at different reaction temperatures of  $50-70^{\circ}$ C. The degree of grafting increased with the reaction temperature. However, the increase in the standard deviation (n = 3) for the degree of grafting with increasing reaction temperature indicated that the reproducibility of the graft polymerization decreased with increasing reaction temperature, especially when the latter exceeded  $70^{\circ}$ C. Figure 4(a) shows the degree of grafting obtained at  $60^{\circ}$ C with 0.25-0.90M VBTAC, 0.2 w/v % BP, and 20 v/v % MeOH in deionized water and photoirradiation times in the range 1–4 h. The degree of grafting increased with the photoirradiation time and the



**Figure 5.** AEC of the grafted fabrics as a function of the degree of VBTAC grafting. AECs determined from a commercial anion-exchange resin and nylon-*g*-VBTAC–HEMA fabrics prepared with a conventional cografting system are also presented for comparison. PA308, anion exchange resin produced by Samyang Co., Korea.



Figure 6. Relative AECs of the grafted fabrics as a function of the degree of VBTAC grafting.



Figure 7. FTIR spectra of the PET, PET coated with surfactant, and PET-g-VBTAC.

concentration of VBTAC. This was explained by the fact that the number of radical sites, including the reinitiating of the polymer chain ends, formed in the grafting system increased with increasing photoirradiation time, and more monomers diffused to the PET fiber with increasing monomer concentration; this resulted in an increase in the degree of grafting.<sup>20,21</sup> The effect of the photoirradiation time on the degree of VBTAC grafting increased with the concentration of VBTAC; this indicated that the higher concentration of VBTAC was, the more VBTAC could be grafted onto the PET fiber, and a longer photoirradiation time was required to completely use the VBTAC available for graft polymerization. The AEC of the resulting PET-g-VBTAC increased with the photoirradiation time and the concentration of VBTAC, it but tended to be maximal at a certain degree of grafting and then level off [Figure 4(b)].

Figure 5 shows the effect of the degree of grafting on the AEC of the resulting PET-g-VBTAC. For comparison, the AECs determined from the commercial anion-exchange resin PA308 and nylon-g-VBTAC-HEMA prepared by a cografting system of VBTAC and HEMA<sup>14</sup> and the theoretical maximum AEC of PET-g-VBTAC are presented in Figure 5. The theoretical maximum AEC was calculated from the weight gain and the molecular mass of VBTAC; this corresponded to the VBTAC density (millimoles per gram) in the grafted polymer discussed in our previous article.<sup>14</sup>

Theoretical maximum AEC (mequiv/g) =  $\frac{1000(W_1 - W_0)}{211.74 \times W_1}$  (4)

where  $W_0$  and  $W_1$  are the weights of the substrate and grafted fabric (g) and the value 211.74 was the molecular mass of VBTAC. In contrast to the theoretical AEC, which increased logarithmically with the degree of grafting, the AEC determined for the PET-g-VBTAC fabrics increased with increasing degree of grafting up to approximately 80% and then leveled off. This



Figure 8. Solid <sup>13</sup>C-NMR spectra of the PET substrate, PET coated with surfactant, and PET-g-VBTAC.

leveling off trend for high degrees of grafting may have been due to the fact that fibers gradually increased in thickness with increasing degree of grafting; this, thereby, relatively decreased the effective surface area bearing the anion-exchange functional groups on the PET-g-VBTAC fabrics so that the chloride ions were less able to diffuse into the interior. It is also considerable that this may have been caused by excess crosslinking generation



**Figure 9.** TGA profiles of the PET, PET coated with surfactant, and PETg-VBTAC





Figure 10. SEM images of (a) PET, (b) PET coated with surfactant, and (c) PET-g-VBTAC.

at a high degree of grafting; this would reduced the anionexchange functionality of the graft chain.<sup>22</sup> From this result, the optimal degree of grafting in terms of AEC was found to be  $80 \pm 5\%$ . The maximum AEC of PET-g-VBTAC obtainable was approximately 2.2 mequiv/g; this was similar to that of the commercial anion-exchange resin (2.16 ± 0.04 mequiv/g) and much higher than those of the nylon-g-VBTAC–HEMA fabrics ( $\leq 1.0$  mequiv/g) prepared by a conventional cografting system.<sup>14</sup> In addition, Figure 6 shows the relative AEC calculated from the relative weight gain of the product times the ACE per unit weight of product:

Relative AEC (mequiv)=AEC
$$\times \frac{W_1}{W_0}$$
 (5)

where  $W_0$  and  $W_1$  are the weights of the substrate and grafted fabric (g), respectively. The theoretical line presented in Figure 6 was made with recalculated values with the theoretical maximum AEC instead of the determined AEC. We noticed that the relative AECs increased consistently with increasing degree of VBTAC grafting, although they exhibited two linear phases with different slopes. The first linear portion, which continued to have about a 90% degree of grafting, exactly corresponded with the theoretical line; this confirmed that the AEC increased in exact proportion to the degree of grafting. The second linear portion, meanwhile, was characterized by the lower slope compared to that of the theoretical line because there was no more increase in the AEC, as shown in Figure 6. Taking the productivity of the sorbent and the availability of monomer in the grafting system into consideration, we determined that the higher the degree of grafting was, the better the cost efficiency in the grafting system was.

The results obtained suggest that the single-monomer grafting system, in which VBTAC alone was directly grafted onto the

PET fiber coated with surfactant was more practical and effective than the conventional comonomer grafting system for the preparation of the VBTAC-containing anion exchanger, and the resulting PET-*g*-VBTAC fabrics could be used as an effective anion exchanger.

#### Surface Characterization

The Fourier transform infrared (FTIR) spectra obtained from original PET, substrate PET coated with surfactant, and grafted PET-g-VBTAC are presented in Figure 7. Compared with the spectrum of the original PET, different absorption peaks were observed in the spectrum of PET coated with surfactant at 1160, 1450, and 2870–2960 cm<sup>-1</sup>; these corresponded to the C—O and C—H groups of the surfactant coated on PET. After the grafting of VBTAC, several new absorption peaks appeared at 900, 1390, 1480, 1600–1640, 3020, and 3090–3700 cm<sup>-1</sup>; these corresponded to C=CH<sub>2</sub> and C=CH vinyl groups, aromatic C—H and C—C, and O—H groups of the VBTAC grafted onto PETs.

These results were verified with solid-state <sup>13</sup>C-NMR spectroscopy. As shown in Figure 8, the peak at 60 ppm, corresponding to the PET fabric, was split into triplets at 50 ppm (N—CH<sub>3</sub>) and 67 ppm (N—CH<sub>2</sub>) in the spectrum of the PET-*g*-VBTAC fabric. Furthermore, new peaks from Cl—CH<sub>3</sub> and aromatic C appeared at 38 and 145 ppm, respectively, in the PET-*g*-VBTAC spectrum. The peak at 145 ppm indicated benzene groups in the grafted VBTAC. New peaks were also observed in the PET spectrum around 12 ppm; this probably corresponded to C—CH<sub>2</sub> in the PET-coated surfactant.

The thermogravimetric analysis (TGA) study carried out with PET fabric, surfactant-coated PET fabric, and PET-g-VBTAC is shown in Figure 9. The TGA profile of the PET fabric showed



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**Figure 11.** Ion selectivity of the PET-g-VBTAC fabric and PA resin in the presence of equimolar anions.  $pH_{eq}$  equilibrium pH after adsorption;  $C_{init}$ , initial concentration of the adsorbate.

that one major weight loss started around 360°C, whereas the TGA profile of the PET fabric exhibited a two-step weight loss. The first step of weight loss, which began around 330°C and up to 400°C, may have been due to the loss of surfactant coating. The second-step weight loss starting around 400°C was due to the loss of base-material PET, which matched with the weight loss of PET in the same temperature range from 360 to 500°C. The TGA profile of PET-g-VBTAC fabric exhibited a differentstep weight loss. The initial weight loss from 40 to 120°C may have been due to the loss of water, as VBTAC was hydrophilic in nature. The next step of weight loss, which began around 170°C and went up to 270°C, was attributed to the loss of VBTAC. The last step of weight loss started around 330°C and may have been attributed to the continuous decomposition of the coated surfactant and base-material PET, as indicated by a similar temperature range for the weight losses of them of 330-500°C. The differences in the profiles of weight loss in the cases of PET, coated PET, and grafted PET are evident from Figure 9; this further confirmed the presence of surfactant coated on the PET and the grafting of VBTAC onto PET.

Figure 10 shows the SEM images of PET, PET coated with surfactant (PETs), and PETsgVBTAC with degree of grafting of 60%. The PET fiber swelled as a result of the coating of surfactant and subsequent grafting of VBTAC on the PETs fiber coated with surfactant, and showed a clear tendency of the surface becoming rougher and more heterogeneous with the coating and grafting.

#### Sorption Selectivity and Regeneration Characteristics

The important characteristics of a sorbent are the sorption selectivity, regeneration efficiency, and sorption capacity because it is the main parameters that determine the efficiency and the cost of the sorption process. The experiment for sorption selectivity was conducted with a mixture that contained 1 mM of each F<sup>-</sup>, NO<sub>3</sub><sup>-</sup>, SO<sub>4</sub><sup>2-</sup>, and  $PO_4^{3-}$  ion. The solution pH was controlled at 5–9 after the adsorption equilibrium by the addition of HCl and NaOH solutions to ascertain the effect of different equilibrium pH values on the adsorption selectivity. The results are presented in Figure 11 and compared with those obtained for PA resin under the same experimental conditions. As shown in Figure 11, the anion-adsorption selectivities of the PET-g-VBTAC fabric and PA resin for anions were pH-dependent. The selectivity order was  $SO_4^{2-} > NO_3^{-} > PO_4^{-}P > F^{-}$  at pH < 6.0 but  $SO_4^{2-} > PO_4 - P > NO_3^{-} > F^{-}$  at  $pH \ge 6.5$ ; there was little difference between the PET-g-VBTAC and PA resin, especially at pH 5.5-6.5. This may have reflected differences between the pH-dependent surface potential changes and the molecular structures for PET-g-VBTAC (fibrous material grafted with VBTAC) and PA resin (aminated styrene-DVB copolymer resin), but further studies are necessary to confirm this. Further studies on the detailed adsorption properties of the PET-g-VBTAC fabric are now in progress, and the results will be reported in a later article.

The regeneration efficiency was important in the evaluation of the use of PET-g-VBTAC as an adsorbent because the surfactantcoated PET fibers were used as the substrate polymer for grafting VBTAC, and the grafted VBTAC groups might have been easily removed from the fiber surfaces. However, the PET fibers used in the study showed no significant weight losses after a washability test with deionized water (<1%) and after a Soxhlet extraction with MeOH for 3 h ( $\leq$ 5%). To evaluate the regeneration efficiency of PET-g-VBTAC and the physical durability of the grafting layer, the AEC was repeatedly determined for PET-g-VBTAC with a degree of grafting of 85%. The PET-g-VBTAC was wrapped in a mesh sheet before use to prevent the weight loss by physical abrasion in repeated shaking and washing stages. The PET-g-VBTAC used was regenerated by repeated washing with 0.2*M* KNO<sub>3</sub> and deionized water and air-drying at 60°C. This



Figure 12. Regeneration efficiency of the PET-g-VBTAC fabric.



cycle was done five times, and the AEC and weight loss of the fabric were measured after each cycle. As shown in Figure 12, although the weight of PET-g-VBTAC decreased significantly after first and second regeneration cycle ( $\geq 2\%$ ), the total loss range was less than 4%, even after five regeneration cycles. On the other hand, the AEC of the regenerated fabric increased to about 4% greater than that of the new fabric after the first regeneration cycle; it was down to approximately 98% of that of the new fabric after five regeneration cycle; it was down to approximately 98% of that of the new fabric after that PET-g-VBTAC could be regenerated by a simple washing process without a significant reduction in the AEC or physical durability and, thus, could be used more than five times with the retention of an AEC value very similar to that of the new fabric.

#### CONCLUSIONS

VBTAC was effectively and stably grafted onto PET fiber by photoirradiation-induced graft polymerization with BP as a photoinitiator without the requirement of any cografting monomer when the PET coated with surfactant (PET) was used as the substrate for graft polymerization. The degree of grafting systematically increased with increasing photoirradiation time and concentration of VBTAC in the monomer solution. In contrast to the conventional cografting system, furthermore, the degree of VBTAC grafting could be easily controlled with changes in these two parameters. The AEC of the resulting PET-g-VBTAC fabrics increased with increasing degree of grafting up to approximately 80% and then leveled off. The maximum AEC of PET-g-VBTAC was found to be 2.2 mequiv/g; this was similar to that of the commercial anion-exchange resin  $(2.16 \pm 0.04 \text{ mequiv/g})$  but much higher than those of the nylon-g-VBTAC-HEMA fabrics  $(\leq 1.0 \text{ mequiv/g})$  prepared by the conventional cografting system. The sorption selectivity of the PET-g-VBTAC fabric in the presence of equimolar amounts of anions was found to be in the following order:  $SO_4^{2-} > PO_4 - P \ge NO_3^{-} > F^{-}$  at pH > 6.0. The repeated sorption-desorption results demonstrated a good regeneration and physical durability of the PET-g-VBTAC fabric. The results obtained from this investigation clearly suggest that the single-monomer grafting system, in which the PET fiber coated with surfactant was used as the substrate polymer for the grafting of VBTAC, is more practical and effective for the preparation of VBTAC-containing anion exchangers.

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