

Preparation and Characterization of a Novel Strong-Base Anion Exchange Fiber

Jing Wang,^{1,2} Xiang Hua Lv,³ Liang Zhao,^{1,2} Baofeng Sha,¹ Dong Zhao,^{1,2} Chengshen Zhu³

¹Institute of Chemistry, Henan Academy of Sciences, Zhengzhou 450002, People's Republic of China

²High & New Technology Research Center, Henan Academy of Sciences, Zhengzhou 450002, People's Republic of China

³School of Materials Science and Engineering, Zhengzhou University, Zhengzhou 450052, People's Republic of China

Correspondence to: L. Zhao (E-mail: wangj12@163.com)

ABSTRACT: A novel strong-base anion exchange fiber (QAPAN) was prepared in this study by preamination of a commercial poly(acrylonitrile) fiber followed by its subsequent grafting of glycidyl trimethylammonium chloride (GTA). The grafting conditions were optimized by conducting a L_{18} (3^7) orthogonal test; and properties of the prepared QAPAN were investigated by multiple analyses. Results showed that the optimal reaction conditions were GTA dosage 4 mL, temperature 60°C, reaction time 6 h, and volume ratio of water to dimethyl sulfoxide (DMSO) 0.5 : 0.5, among which volume ratio of water to DMSO was deemed as the most important factor on the QAPAN preparation. The prepared QAPAN had excellent fibrous morphology and fine mechanical stability, making its further processing feasible. Acid–base titration tests showed that the QAPAN had a total exchange capacity (TEC) of 6.34 mmol/g and a strong base capacity (SBC) of 1.34 mmol/g. Analyses of Fourier transform infrared spectroscopy and X-ray photoelectron spectroscopy further verified that the quaternary ammonium group was successfully grafted onto the QAPAN. This study provided a novel strong-base anion exchange fiber as well as a simple and repeatable method for its preparation. © 2012 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* 000: 000–000, 2012

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INTRODUCTION

Ion exchange fiber is a fibrous ion exchange material that can be used in various forms such as filaments, nonwoven fabrics, threads, and cloths.¹ Due to their large effective specific surface area and small mass transfer distance, ion exchange fibers provide much faster reaction kinetic and higher separation efficiency than traditional materials in ion exchange processes.^{2,3}

Strong-base anion exchange fibers have become one hot issue among the fiber materials in recent years. The presence of quaternary ammonium group in their structure endows this type of fibers with perfect ion exchange capabilities in a wide pH range.⁴ Thus, they are more appropriate for the practical aquatic systems. Widely studied fields for application of the strong-base anion exchange fibers include biomaterial separation,⁵ ion chromatographic separation,^{6,7} water and air purification,^{8,9} element recovery and regeneration, and so forth.

Currently, the most popular method for preparation of the strong-base anion exchange fibers uses a three-step procedure, which includes grafting of styrene onto a natural or synthetic fiber matrix, chloromethylation of the styrene by chloromethyl

ether, and amination of the chloromethyl styrene by ternary amine. Previous literature demonstrated that when using polypropylene (PP) or poly(tetrafluoroethylene) (PTFE) as the fiber matrix, the prepared fibers would have a strong base capacity (SBC) as high as 0.95–2.80 or 6.08 mmol/g.^{10,11} However, because of the complicated process and severe toxic reagents (like chloromethyl ether)¹² of this method, its application is substantially inhibited. Other simpler methods are also developed using more environmental-friendly reagents. For example, Kim *et al.*¹³ synthesized a strong-base anion exchange fiber with SBC 1.7 mmol/g by directly grafting glycidyl methacrylate (GMA) onto a PP trunk polymer followed by amination of the grafted GMA. Matsumoto *et al.*¹⁴ prepared a strong-base anion exchange fiber with SBC 0.78 mmol/g by quaternization of poly(4-vinylpyridine) fabrics after its copolymerization with polystyrene.

In this research, a novel strong-base anion exchange fiber (QAPAN) was prepared by a simple two-step method, which included preamination of a commercial poly(acrylonitrile) (PAN) fiber followed by its subsequent grafting of glycidyl trimethylammonium chloride (GTA). The GTA grafting conditions were optimized by orthogonal tests with reagent dosage,

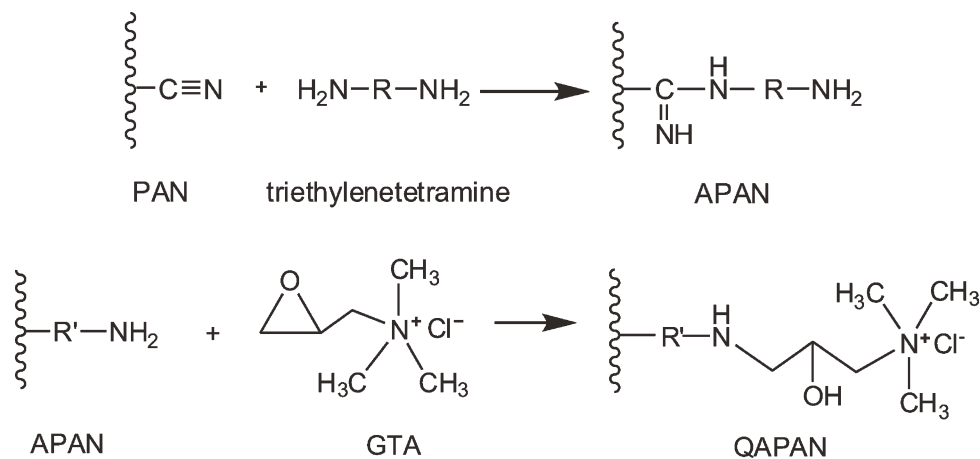


Figure 1. Preparation route for the novel strong-base anion exchange fiber (QAPAN).

temperature, reaction time, and solvent composition, and so forth as the influential factors. Properties of the prepared fibers were characterized by acid–base titration, Fourier transform infrared spectroscopy (FTIR), X-ray photoelectron spectroscopy (XPS), analyses of tensile strength, and microscope photographs.

EXPERIMENTAL

Materials

The matrix for preparation of the novel QAPAN fiber was industrially produced PAN fiber (length: 2–10 cm, linear density: 1–5 dtex, and content of acrylonitrile $\geq 90\%$) purchased from Shanghai Petrochemical (Shanghai, China). Prior to use, the fiber was washed thoroughly by deionized (DI) water and then oven-dried at 65°C until constant weight.

Triethylenetetramine (analytical grade, Tianjin Kermel Chemical Reagent, Tianjin, China) and GTA (70% aqueous solution, SA-CHEM Wuxi, Wuxi, China) were used as the amination and quaternization reagents, respectively. Dimethyl sulfoxide (DMSO, chemical grade, Shanghai Supply Station of Chemical Reagents, Shanghai, China), DI water, or their mixtures were used as the solvents in the quaternization reaction. Spectroscopic grade KBr was purchased from Sinopharm Chemical Reagent (Shanghai, China) for FTIR analysis. All other chemicals were of analytical grade and used as received.

Preparation Method

The strong-base anion exchange fiber (QAPAN) was prepared through a simple two-step method according to Figure 1. Briefly, 2.0 g of PAN fiber was immersed in 80 mL of triethylenetetramine and heated in an oil bath at $80\text{--}140^\circ\text{C}$ for 2–10 h. The resultant fiber was assigned to aminated poly(acrylonitrile) (APAN) fiber after being washed with copious DI water and dried in an air oven. Then, 1.0 g of APAN fiber was immersed in a mixed solution of GTA, DI water, and DMSO under manually stirring conditions. After reaction at $20\text{--}70^\circ\text{C}$ for 2–10 h, the fiber (QAPAN) was separated, repeatedly washed with DI water, dried in an air oven until constant weight, and then stored in a desiccator prior to use. The weight gain rate (W_t , %) was calculated using eq. (1).

$$W_t = \frac{W_1 - W_0}{W_0} \times 100\% \quad (1)$$

where W_0 and W_1 (g) are weight of fibers before and after amination or quaternization reactions, respectively.

Characterization Methods

Determination of Ion Exchange Capacity. Both total exchange capacity (TEC, indicating content of all base groups) and strong base capacity (SBC, indicating content of strong base groups) were determined.¹⁵ To obtain TEC values, about 0.1 g of fiber was equilibrated with 50 mL of 0.1 mol/L HCl solution for 12 h. The initial and equilibrated concentrations of HCl were determined by titration with standard 0.1 mol/L NaOH solutions. TEC (mmol/g) was calculated using eq. (2).

$$\text{TEC} = \frac{5 \times (V_0 - V)}{W} \times C \quad (2)$$

where V_0 and V (mL) are volumes of standard 0.1M NaOH solution consumed by initial and equilibrated HCl, respectively, C (mol/L) is concentration of standard NaOH solution, and W (g) is dry mass of the tested fiber.

To obtain SBC values, about 0.1 g of fiber in their OH^- forms was mixed with 50 mL of 0.5 mol/L Na_2SO_4 solution. After 12 h of equilibrium, 10 mL of supernatant was sampled and titrated by 0.1 mol/L HCl standard solution. SBC (mmol/g) was calculated using eq. (3).

$$\text{SBC} = \frac{5 \times V}{W} \times C \quad (3)$$

where V (mL) is volume of HCl standard solution for titration, C (mol/L) is concentration of standard HCl solution, and W (g) is dry mass of the tested fiber.

FTIR Spectroscopy. The fiber samples were cut into small pieces and then thoroughly grinded with KBr in an agate mortar. After being compressed in a powder sheeter (FW-4A, Tianjin Tuopu Instrument, Tianjin, China), the infrared spectra were

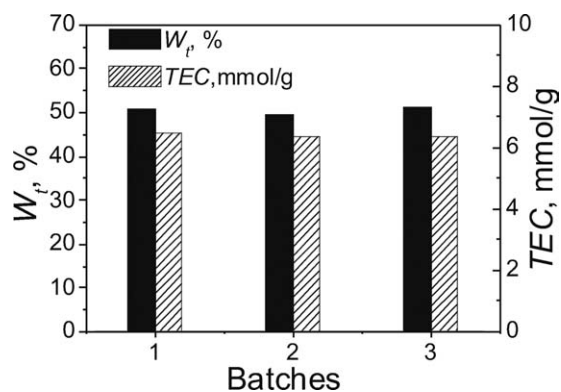


Figure 2. Correlation of weight gain rate (W_t , %) and TEC (mmol/g) for the three batches of APAN fibers prepared under the same optimal conditions: 2.0 g of PAN fibers versus 80 mL of triethylenetetramine, temperature 120°C, reaction time 6 h.

obtained in 400–4000 cm^{-1} using a Thermo Nicolet IR 200 instrument (Thermo Electron, Waltham, MA).

Microscopic Photograph. Surface morphology of the PAN, APAN, and QAPAN fibers were observed from a 59XC microscope (Shanghai Instrument, Shanghai, China). To determine their diameters, 15 stochastic filaments for each sample were tested, and the average values were calculated and reported.

Tensile Strength. A Labthink XLW (L) PC autotensile tester (Labthink Instruments, Jinan, China) was used to test tensile breaking strength of the fiber samples. For each sample, 20 stochastic filaments were tested, and the average value was calculated and reported.

XPS. XPS spectra for the QAPAN fiber were recorded using a Kratos Axis Ultra delay-line detector (DLD) spectrometer (Manchester, UK) using a monochromated Al-K α X-ray source ($h\nu = 1486.6$ eV). Both survey and high-resolution spectra were collected and calibrated to the binding energy (BE) of C $_{1s}$ at 284.6 eV. XPS peak 4.1 software was used to peak-fit the calibrated high-resolution spectra.¹⁶

RESULTS AND DISCUSSION

Preparation of APAN

Preparation conditions for the APAN fiber were optimized through a series of preliminary laboratory experiments. Results showed that the optimal reaction conditions were temperature 120°C and reaction time 6 h. Under such conditions, three batches of APAN fiber were prepared and their W_t (%) and TEC (mmol/g) were tested (Figure 2). It could be seen that both W_t and TEC for the three batches of the APAN fiber varied in a very narrow range. Their average values were 50.43 (± 0.93)% and 6.40 (± 0.07) mmol/g, respectively, significantly higher than our reported values (i.e., $W_t = 33.5\%$, TEC = 4.95 mmol/g).¹⁷ Furthermore, as compared with the 24–30 h of reaction time in previous literature,^{18–20} a much shorter reaction time (i.e., 6 h) was applied in this study, because the temperature was increased from 95 to 120°C. The above results indicated that the preparation process for APAN fiber could be well repeated, and the reaction conditions were very easy to control.

Preparation of QAPAN

Optimal conditions for QAPAN preparation were evaluated through orthogonal tests in this study. According to the preliminary tests, seven influential factors were studied. They included four single factors (*A*: GTA dosage, *B*: temperature, *C*: reaction time, and *D*: solvent composition, i.e., volume ratio of DI to DMSO) and three interacting factors (indicating the combined effects between the above single factors, i.e., *AB*, *AC*, and *BC*). Three levels were chosen for each factor, and W_t and SBC were used as the two responses. After reaction, 18 fiber samples (Q-1 to Q-18) were prepared according to L $_{18}$ (3^7) orthogonal experimental table (Table I). Results from the tests are listed and analyzed in Table I and Figure 3, respectively.

It was evident from Table I and Figure 3A that there was a positive linear relationship between the two responses W_t (16–48%) and SBC (1.1–1.6 mmol/g) under the studied conditions. Among the seven samples with SBC values above 1.4 mmol/g (i.e., Q-3, -7, -8, -9, -12, -14, -17), samples Q-3, -9, -12, and -17 had much higher W_t values than the other three, and their fibrous morphology was significantly destroyed. This demonstrated that some side reactions might occur during preparation of the above four samples. These side reactions were of no use for the graft of strong-base groups, but they could make the fibers gain more weight and destroy their fibrous structures. Therefore, conditions for samples Q-7, -8, and -14 were deemed as the most appropriate for preparation of QAPAN fibers.

To further optimize the preparation conditions, a detailed discussion of k_i and R values, which are widely used in the range analysis of orthogonal tests,^{21,22} was conducted. The calculated k_i and R values are listed in Table I, where k_i is the average of the data at i level of one factor and R is the difference between the maximal and minimal value of k_i . The R values represented the effects of various factors on the preparation of QAPAN (Figure 3B), whereas the k_i values reflected the effects of various levels (Figure 3C). It was obvious from Figure 2B that R and R' for the three interacting factors were all very low, demonstrating there were minor combining effects between the studied single factors. Among the four single factors, solvent composition (*D*, i.e. DI/DMSO) obtained the highest R and R' values, indicating that it was the most important factor in preparation of the QAPAN. Temperature (*B*) also obtained the highest R value, however, its corresponding R' value was much lower than that of solvent composition. This suggested that temperature variance was less influential for the graft of strong-base groups than the fiber weight gain. GTA dosage (*A*) obtained a similar R' value with temperature, though its R' value was much lower. Among the four single factors, reaction time (*C*) was deemed as the least influential due to its lowest R' value.

After determination of the priority sequence of the factors (i.e., $D > B > A > C$), the preparation conditions were then optimized by choosing the most appropriate levels of each factors according to the above priority sequence. Factor *D* was of course the first to be considered. As shown in Figure 2C, both W_t and SBC increased significantly with the increase of DMSO content. So the level three (D_3 , i.e., DI/DMSO = 0.5 : 0.5) was selected as the optimal solvent composition. For temperature (*B*), the largest SBC was obtained from the level two, though

Table I. Optimal Conditions for QAPAN Preparation Determined by Analyses of L_{18} (3^7) Orthogonal Tests

Samples	Factors							Responses	
	A (mL)	B (°C)	AB	C (h)	AC	BC	D	W_t (%)	SBC (mmol/g)
Q-1	4	50		6			1 : 0	16.4	1.19
Q-2	4	60		8			0.75 : 0.25	26.4	1.36
Q-3	4	70		10			0.5 : 0.5	46.2	1.48
Q-4	5	50		8			0.5 : 0.5	28.1	1.37
Q-5	5	60		10			1 : 0	26.0	1.30
Q-6	5	70		6			0.75 : 0.25	35.3	1.24
Q-7	6	50		6			0.5 : 0.5	31.8	1.47
Q-8	6	60		8			1 : 0	32.0	1.42
Q-9	6	70		10			0.75 : 0.25	46.2	1.48
Q-10	4	50		10			1 : 0	19.8	1.24
Q-11	4	60		6			0.75 : 0.25	21.3	1.26
Q-12	4	70		8			0.5 : 0.5	42.8	1.48
Q-13	5	50		10			0.75 : 0.25	28.2	1.20
Q-14	5	60		6			0.5 : 0.5	34.3	1.48
Q-15	5	70		8			1 : 0	29.1	1.11
Q-16	6	50		8			0.75 : 0.25	28.5	1.17
Q-17	6	60		10			0.5 : 0.5	47.9	1.54
Q-18	6	70		6			1 : 0	30.8	1.36
k_1^a	28.8	25.5	31.5	28.3	33.8	32.4	25.7		
k_2	30.2	31.3	31.0	31.2	30.9	31.7	31.0		
k_3	36.2	38.4	32.7	35.7	30.5	31.1	38.5		
R^b	7.4	12.9	1.7	7.4	3.3	1.3	12.8		
k_1^c	1.35	1.29	1.34	1.35	1.36	1.35	1.27		
k_2	1.30	1.41	1.38	1.32	1.38	1.33	1.32		
k_3	1.41	1.36	1.34	1.39	1.32	1.38	1.47		
R^d	0.11	0.12	0.04	0.07	0.07	0.06	0.20		

^a $k_i = (\sum W_t \text{ of each factor at } i \text{ level})/6, i = 1-3.$

^b $R = \max k_i - \min k_i.$

^c $k_i' = (\sum \text{SBC of each factor at } i \text{ level})/6, i=1-3.$

^d $R' = \max k_i' - \min k_i'.$

W_t increased significantly with the temperature increase. This demonstrated that a further increase of temperature had no advantage for the graft of strong-base groups. Contrarily, it would trigger more side reactions and thus destroy the fibrous structure. Therefore, a moderate temperature (B_2 , i.e., temperature = 60°C) was more appropriate. Comparing with the above two factors, GTA dosage (A) and reaction time (C) were much less influential in the QAPAN preparation. To save reagents and time, A_1 and C_1 were chose as the optimized levels for factor A and C , respectively.

Based on the above orthogonal experiments, the optimal reaction conditions for preparation of QAPAN fiber were obtained as $A_1B_2C_1D_3$ (i.e., GTA dosage 4 mL, temperature 60°C, reaction time 6 h, and DI/DMSO 0.5 : 0.5). Under such optimal conditions, the preparation was magnified ($\times 5$), and the process was repeated to investigate reproducibility of the experiments. Results showed that the prepared QAPAN had an average W_t of 29.9(± 0.3)%, SBC of 1.34 (± 0) mmol/g, and TEC of 6.34 (± 0.04) mmol/g. The minor data deviation among the several

batches of samples demonstrated that the reaction conditions for preparation of the QAPAN were very easy to control, and the procedure could be well repeated.

Characteristics of QAPAN Prepared by the Optimized Conditions

FTIR Spectra. Figure 4 showed FTIR spectra of the (a) PAN, (b) APAN, and (c) QAPAN fibers. By comparing spectra between PAN and APAN fibers, it could be seen that the peak at 2243 cm^{-1} ($\text{C}\equiv\text{N}$ stretching in nitrile group) was significantly reduced after the amination reaction. Simultaneously, two new bands appeared at 1645 and 1602 cm^{-1} , which could be designated as the $\text{C}=\text{O}$ group in amide and the $\text{N}-\text{H}$ group in amine, respectively.¹⁷⁻²⁰ These changes of the above bands demonstrated that amino group has been successfully introduced in the original PAN structure, probably through amination reaction with the nitrile group. After the quaternization reaction, another two new bands appeared at 918 and 968 cm^{-1} . They were deemed as the characteristic absorption peak of quaternary ammonium group

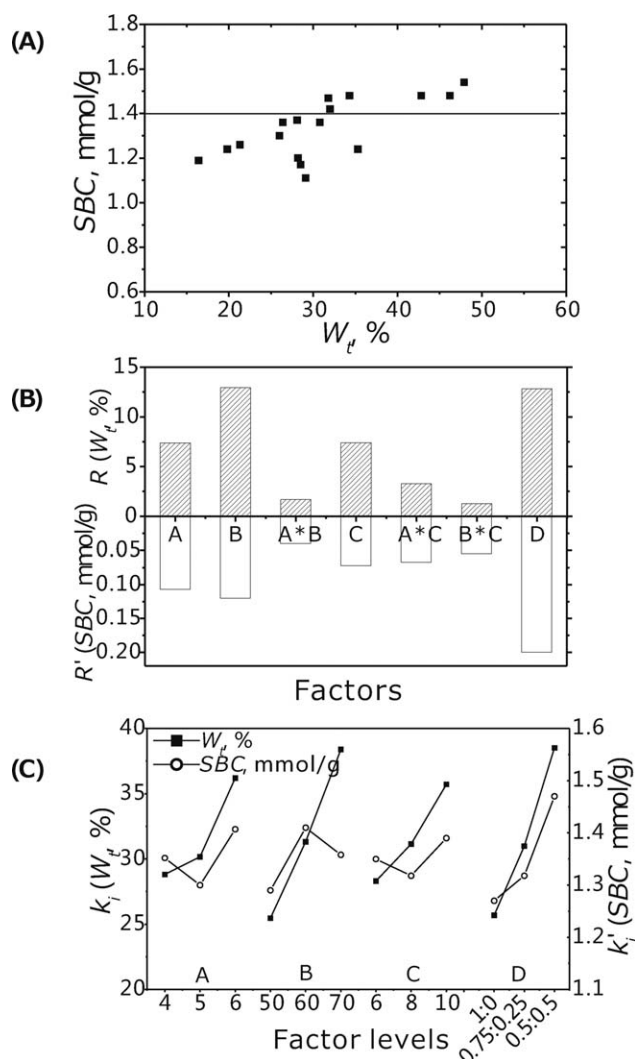


Figure 3. (A) Relationship between weight gain rate (W_t , %) and strong base capacity (SBC, mmol/g) for the QAPAN. (B) Effects of the seven studied factors on W_t and SBC. (C) Variation of W_t and SBC with three levels of the four primary factors. The studied factors includes four single factors (A: GTA dosage, B: temperature, C: reaction time, and D: volume ratio of DI to DMSO, i.e., DI/DMSO) and three interacting factors (AB, AC, and BC indicating the combined effects between GTA dosage and temperature, GTA dosage and reaction time, and temperature and reaction time, respectively). For each factor, three levels are studied.

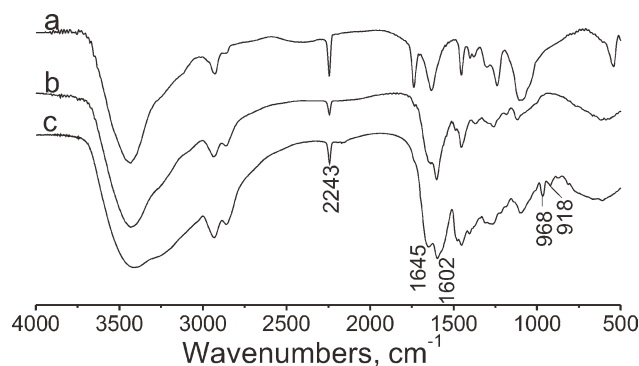


Figure 4. FTIR spectra of PAN, APAN, and QAPAN fibers.

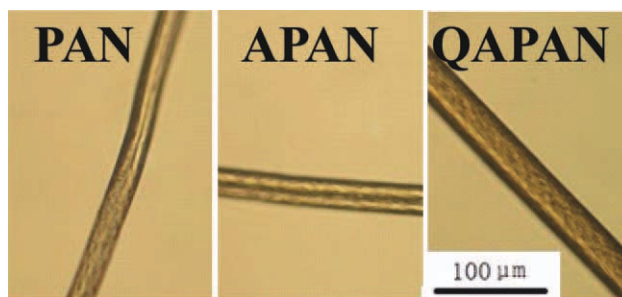


Figure 5. Microscopic photographs of PAN, APAN, and QAPAN fibers. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://www.wileyonlinelibrary.com).]

in reference,²³ indicating that quaternary ammonium group has been successfully grafted onto the APAN fiber.

Microscopic Photograph. Morphology of the (a) PAN, (b) APAN, and (c) QAPAN fibers were investigated by optical microscopic photographs as shown in Figure 5. In general, the fibers all had a characteristic fibrous morphology with a very smooth surface. After the two-step reactions, the fibers became much thicker than the starting material. Further measurement demonstrated that the original PAN fiber had an average diameter of $17.2 (\pm 2.2) \mu\text{m}$, which was increased up to $22.0 (\pm 1.7) \mu\text{m}$ and $25.1 (\pm 2.4) \mu\text{m}$ after the amination and quaternization reactions, respectively. Results from microscopic photographs indicated that the fibers retained their morphology and shape well during the modification reactions though their diameters were greatly increased. The increased diameter was suggested to be due to the contribution of the grafted side chains.^{24,25}

Tensile Strength. A remarkable advantage for fiber materials is that they can be applied in various forms such as filaments, non-woven fabrics, threads, and cloths. Therefore, it is important to keep their fine mechanical properties during chemical modification. Figure 6 shows that the APAN and QAPAN fibers had an average tensile strength of 6.12 and 5.07 cN, respectively, which were 20.1% and 33.8% lower than the original PAN fiber. This demonstrated that the amination and quaternization reactions would substantially decrease the mechanical properties of the fibers. However, the decrease of tensile strength is acceptable, and the prepared fibers are still suitable for their further processing. Yao et al.²⁶ and

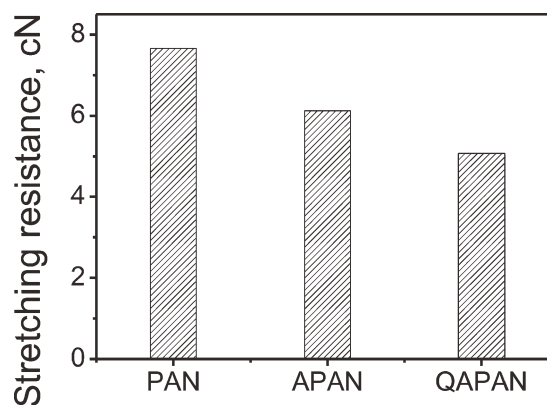


Figure 6. Stretching resistances of PAN, APAN, and QAPAN fibers.

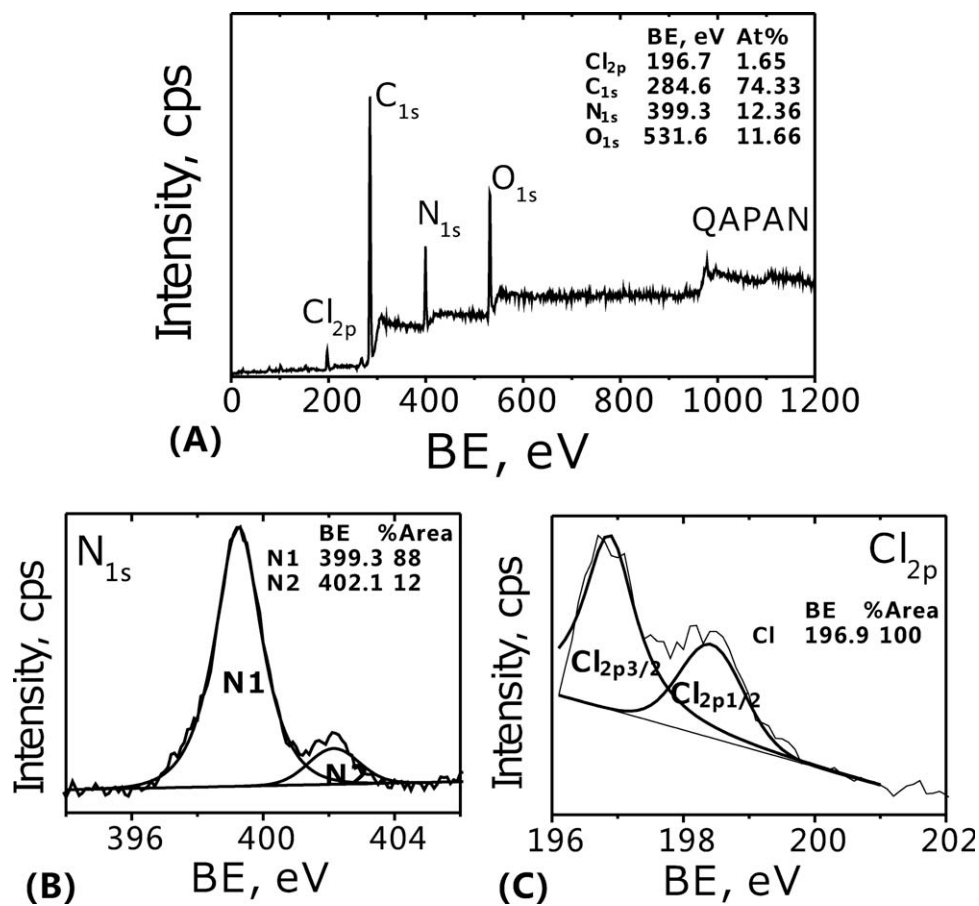


Figure 7. XPS spectra of (A) survey scan and high-resolution scan of (B) N_{1s} and (C) Cl_{2p} for QAPAN fibers. BE in eV; At%, atomic concentration in %.

Ma et al.²⁷ also reported that surface modification might have adverse effect on the tensile strength of polyurethane fibrous membranes and PP fibers. However, Zhang et al.¹¹ observed an increase in the break strength when preparing a strong-base anion exchange fiber through a four-step method from poly(tetrafluoroethylene)

fibers. The different results were suggested to be due to the different starting materials and the different preparation procedures.

XPS Spectra. XPS spectra of survey scan and high-resolution scan for the key elements on the QAPAN surface were studied

Table II. Comparison of Properties and Preparation Methods of Various Strong-Base Anion Exchange Fibers

Anion exchange fiber	Starting material	TEC (mmol/g)	SBC (mmol/g)	Cr(VI) uptake (mg/g)	Preparation method	Ref.
AIEF	Polytetrafluoroethylene	6.08	6.08	214	Four-step: irradiation, styrene grafting, chloromethylation, amination	11
FIBAN A-1	PP	0.95-2.80	0.95-2.80	-	Three-step: styrene grafting, chloromethylation, amination	10
AEF	Polystyrene-block-poly(4-vinylpyridine)	-	0.78	-	Three-step: electrospray deposition, heat treatment, quaternization	14
modified PU	Polyurethane	-	-	-	Three-step: plasma pretreatment, UV-induced 4-vinylpyridine grafting, quaternization	26
APP-g-GMA	PP	1.7	1.7	-	Two-step: GMA grafting, amination	13
QAPAN	Polyacrylonitrile	6.34	1.34	248	Two-step: amination, quaternization	This study

to gain insights into compositions of the novel QAPAN fiber. As shown in the survey spectra in Figure 7A, the fiber comprised mainly of elements C (74.33 atomic %), N (12.36 atomic %), O (11.66 atomic %), and Cl (1.65 atomic %). High-resolution spectra (Figure 7B) showed that N_{1s} could be fitted into two major peaks (N1 and N2) with BE fixed at 399.3 and 402.1 eV. According to the previous reference,²⁸ these peaks could be assigned to nonquaternized nitrogen and quaternized nitrogen, respectively. Calculation of peak area demonstrated that the quaternized nitrogen was about 12% of the total nitrogen, suggesting that only part of amino group on APAN fiber were converted to the strong base group during the quaternization reaction. Cl_{2p} high-resolution spectra (Figure 7C) was fitted with one doublet-peak with $Cl_{2p3/2}$ situated at 196.9 eV and $Cl_{2p1/2}$ at 198.4 eV, which could be designated as chloride anions.¹⁶ It was interesting to note that the atomic concentration of Cl (i.e., 1.65 atomic %) was nearly similar to that of quaternized nitrogen (i.e., 12.36 atomic % \times 12% = 1.48 atomic %). The presence of chloride anions in nearly 1 : 1 atomic ratio of quaternized nitrogen suggested that there were some positive charges in the QAPAN structures that needed to be neutralized and the positive charges were originated primarily from the grafted quaternary ammonium group on the QAPAN fiber.

Comparisons with the State-of-the-Art Fibers

Preparation and properties of the QAPAN were compared with several state-of-the-art strong-base anion exchange fibers in Table II. It can be seen that the preparation procedure of the QAPAN comprises only two steps, which is much simpler than the previously reported three- to four-step methods. The prepared QAPAN consists not only of strong quaternary ammonium group but also of a significant amount of weak amino groups (as demonstrated from its high TEC). This makes the novel fiber a combination of the advantages of the two groups and makes it more feasible for practical application under complex conditions. Preliminary adsorption studies demonstrated that the maximum uptake of aqueous Cr(VI) by the novel QAPAN was 248 mg/g, which is comparable with the previously reported value (i.e., 214 mg/g) for strong basic AIEF fibers.

CONCLUSIONS

This work presented the preparation and characterization of a novel strong-base anion exchange fiber (QAPAN), via preamination of a commercial PAN fiber followed by its subsequent grafting of GTA. The optimal reaction conditions obtained from orthogonal tests were GTA dosage 4 mL, reaction temperature 60°C, time 6 h, and solvent ratio of DI to DMSO (DI/DMSO) 0.5 : 0.5. QAPAN fiber prepared under the magnified ($\times 5$) conditions had excellent fibrous morphology, fine mechanical stability, SBC 1.34 mmol/g, and TEC 6.34 mmol/g. The method developed in this study for preparation of the strong-base anion exchange fiber is relatively simple, repeatable, and easy for scale-up. The prepared QAPAN has a wide potential application in water treatment, elemental extraction, elemental recovery, and so forth.

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