Adsorption Behavior of Monomers and Formation of Conducting Polymers on Polyester Fibers

Yaping Zhao, Zaisheng Cai, Zhaoyi Zhou, Xiaolan Fu

Key Laboratory of Science and Technology of Eco-Textile, Ministry of Education, College of Chemistry, Chemical Engineering and Biotechnology, Donghua University, Shanghai 201620, China

ABSTRACT: *In situ* adsorption of monomers on fibers plays a key role in fabricating highly conductive polyaniline (PANI)-based textiles by two-stage oxidation polymerization. Experiments were conducted in aniline monomer and hydrochloric acid solution with the variables such as contact time, initial concentration, and temperature, which can enhance the equilibrium adsorption capacity to aniline of poly(ethylene terephthalate) (PET) fibers. Equilibrium data were fit well by a Henry partition-type isotherm equation. It was found that the kinetics of the adsorption of aniline onto PET fibers at different operating conditions was best described by the pseudo-second-order model. The rate parameters of the intraparticle diffusion model for adsorption were also evaluated and compared to identify the adsorption mechanisms. The monomer exhaustion increased with increasing the temperature. The value of electrical surface resistance of conductive textiles about 3.2 kΩ was obtained when the padder squeeze step was introduced, and the molar ratio of 0.6 between the oxidant concentration and the exhausted concentration of monomers at the adsorption equilibrium was applied. Scanning electron micrographs of PANI/PET composite surfaces were observed, conforming that smooth films were produced by surface polymerization of aniline monomers adsorbed previously on fibers. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 119: 662–669, 2011

Key words: monomers; adsorption; thermodynamics; conducting polymers; polyesters

INTRODUCTION

Electrically conductive composite fabrics prepared by coating thin layers of conductive polymers, such as polypyrrole (PPY), polyaniline (PANI), and polythiophene (PTP), on the fiber surface of fabrics and yarns recently have been proposed for sensors, EMI shielding, electrostatic discharge, heating elements, dust and germ-free clothing, and conducting materials applications. PANI is one of the most promising candidates because of low price of monomer, easy synthesis, and environmental and chemical stability.[1–4] Synthesis of PANI–textile composites has been realized by electrochemical and chemical polymerization. PANI in the fibers or films produced by electrochemical method usually showed poor mechanical properties, which limit its industrial appli-

cation. Recently, most studies have been focused on *in situ* chemical polymerization of organic monomers to create conductive coatings on the surface of textiles such as nonwoven fabric, nylon 6, cotton, silk, and polyester fabric, enabling combination of flexibility of traditional textiles with the electrical conductivity of conducting polymers.[5–8]

PANI-based textiles can be fabricated chemically through a two-step process, sorption of aniline by the fiber and successive oxidation polymerization.[9–11] Oxidation conditions, the second stage, have been reported by some groups, and two methods were carried out, which were addition of oxidation solution dropwise into monomer bath and immersing fibers with monomers adsorbed into the oxidation bath reaction.[12–14] There was usually a lot of loose sedimentation deposited in the solution by taking the former method. Obviously, it can be seen that the latter way was practical from the angles of economic and environmental standpoints. However, no matter oxidation way was taken, it is crucial for the design of homogenous and highly conductive layers on textile substrates that a massive aniline monomers can be adsorbed and diffuse into fibers during the sorption process. Monomer adsorption is mainly controlled by conditions such as initial concentration, adsorption temperature, contact time, and sorption property of textile substrates. Therefore, investigation on sorption

behavior of aniline on fabrics is the vital foundation for the development of polymerization techniques to improve the quality of PANI-based conductive fabrics.

This study was concerned with the effects of conditions on the adsorption of polyester fabrics with monomers for the fabrication of conducting layers on PET textile substrates by oxidative polymerization. The kinetic properties and adsorption mechanism of aniline on PET fibers were investigated. In addition, the consequent oxidative polymerization of aniline on the fiber surface was studied in this article.

EXPERIMENTAL

Materials

Polyester fabric (plain weave, 87 ± 2 g/m^2, 25 ends/cm, and 18 picks/cm) was used, rinsed thoroughly with water, and air-dried before use. Aniline (Sinopharm Chemical Reagent, Shanghai, China) was purified by vacuum distillation. Hydrochloric acid and ammonium peroxydisulfate were analytical grade and used as received.

Adsorption studies

To determine the adsorption capacity of the PET fibers, the initial concentration of aniline in hydrochloric solution at the molar ratio aniline/HCl 1 : 1 with a pH close to 7.0 was changed between 0.1 and 0.5 mol/L at different temperatures between 313 and 368 K. The ratio of the monomer solution to fabric (liquor ratio) was 50 : 1. The amount of adsorbed aniline was determined with the expression (1) by measuring the initial and final concentrations of aniline within the adsorption medium at 231 nm using a UV–vis spectrophotometer (Hitachi UV-3100).

$$C_f = \frac{(C_0 - C_s)V \times 1000}{m},\qquad(1)$$

where C_f is the amount of aniline adsorbed onto the unit of mass of the PET fibers (mmol/g); C_0 and C_s are the concentrations of aniline in the initial solution and in the aqueous phase after the adsorption treatment for a certain period of time (mol/L), respectively; V is the volume of the adsorption solution used (L); and m is the amount of PET fibers used (g).

Kinetic experiments were carried out by continuously measuring the change of absorbance with time for the monomer solutions in contact with the PET fibers. Equilibration experiments were carried out by agitating a series of bottles containing aniline solutions of different initial concentrations (0.1–0.5 mol/L) with PET fibers at temperatures (313–368 K) for 360 min, which is more than sufficient time to reach equilibrium.

Preparation of PANI-coated PET fabrics

A laboratory procedure was used to produce PANI-coated PET fabrics. The PET fabrics were first soaked into the aniline and hydrochloric acid solution for some time. Successive polymerization was then initiated by taking the PET fabrics out to dip in the oxidant solution containing oxidant (ammonium persulfate, APS) and hydrochloric acid solution for a short time. The dipped fabric was taken out and immediately squeezed by a padder (Rapid Labortex, Taiwan) with a nip pressure 1.40 kg/cm^2. Then, the padded substrate was placed in sealed containers and cooled to 273 K using the ice water bath to allow the polymerization to continue for 2 h. Finally, the composite fabric was rinsed with ethanol and distilled water for several times and then vacuum dried.

Evaluation methods

The monomer exhaustion was estimated according to the eq. (2)

$$\%E = \left(1 - \frac{C_0}{C_e}\right) \times 100,\qquad(2)$$

where C_0 and C_e are the concentrations of aniline in solution at the initial and equilibrium time (mol/L), respectively.

Surface morphology of the fabrics was examined by scanning electron microscope (SEM, JSM-5600LV, operating at 15 kV). The surface electrical resistance (ρ_s) of the PANI/PET-conducting composite fabric was carried out by a two-probe method by placing copper strips at specific distance and measuring the resistance with a multimeter and calculated according to the eq. (3)

$$\rho_s = \frac{RW}{L},\qquad(3)$$

where W is the width (cm), L is the length of the fabric sample (cm), and R is the resistance of the fabric sample according to the multimeter (Ω).

RESULTS AND DISCUSSION

Adsorption kinetics of aniline monomers toward PET fibers

Experiments were performed under different conditions to follow the adsorption kinetics of monomers toward polyester fibers.

Effect of contact time on the amount of monomers adsorbed

Figure 1 shows the effect of contact time on the amount of aniline adsorbed for the different initial concentrations of 0.1, 0.2, 0.3, 0.4, and 0.5 mol/L at 353 K. All these curves present a two-step adsorption phenomenon. At the first step, the amount of aniline adsorbed C_f (mmol/g) increased rapidly with the increase of contact time from 0 to 30 min. More than 90% of the equilibrium adsorption capacity for aniline occurred within 30 min followed by the second stage. Figure 1 indicates that the adsorption capacity became constant, and when the adsorption reached saturation, adsorption and desorption arrived at an adsorption equilibrium state after 120 min.

Effect of initial concentration on the amount of monomers adsorbed

As shown in Figure 1, the larger the initial aniline concentration, the higher the rate of adsorption, and the initial concentration of aniline had no effect on time to reach equilibrium. Figure 2 relates the amount of aniline adsorbed at equilibrium C_{fe} (mmol/g) versus initial concentration of aniline under different solution temperatures from 333 to 368 K and the linear increasing relationship between the initial monomer concentration and the adsorption amount of aniline at equilibrium on PET fibers. All of the correlation coefficients of linear fitting reached 0.999.

Effect of temperature on the amount of monomers adsorbed

From the slopes of the fitting line increasing with the increase of solution temperature in Figure 2, it
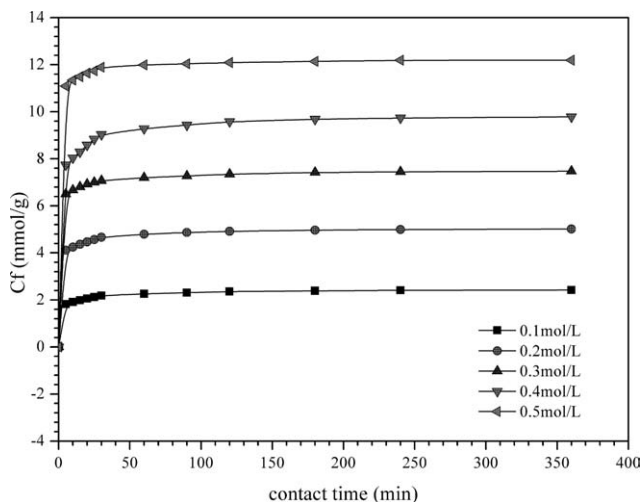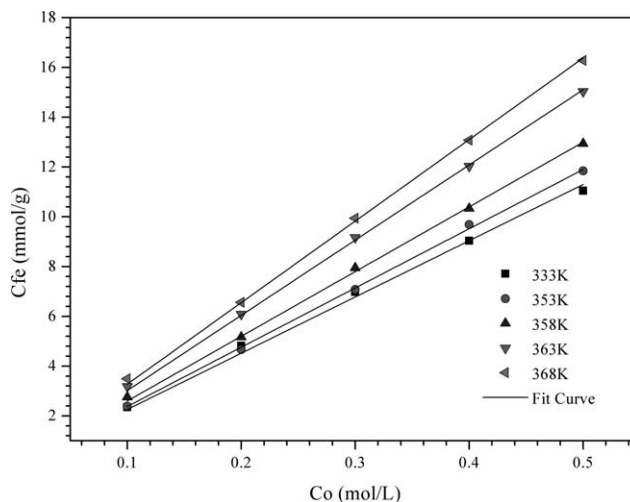

Figure 2 The adsorption amount of aniline at equilibrium versus initial concentration.

was clear that temperature was an important factor affecting the adsorption capacity of the fibers. From the data in Figure 3, it was clear that the uptake of monomers increased relatively slowly starting at 313–348 K. The increase in the monomers uptake became quite sharp above glass transition temperature of the polyester fibers (T_g= 347.5 K). This can be explained by the free volume theory.[15,16] Below T_g the fiber molecules are immobilized in the amorphous regions with few holes large enough offered to the sorption and diffusion of aniline molecules. However, when the temperature exceeds T_g, polymer chain molecules become mobile, and the amount of free volume increases with temperature as molecular motion increases with more active centers formed to interact with the sorbate molecules.
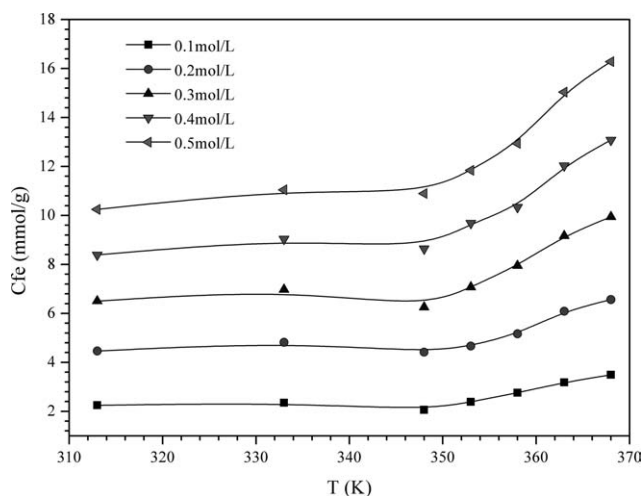

Figure 1 The adsorption of aniline toward PET fibers with different initial concentrations at 353 K.

Figure 3 The adsorption of aniline toward PET fibers under different temperatures.
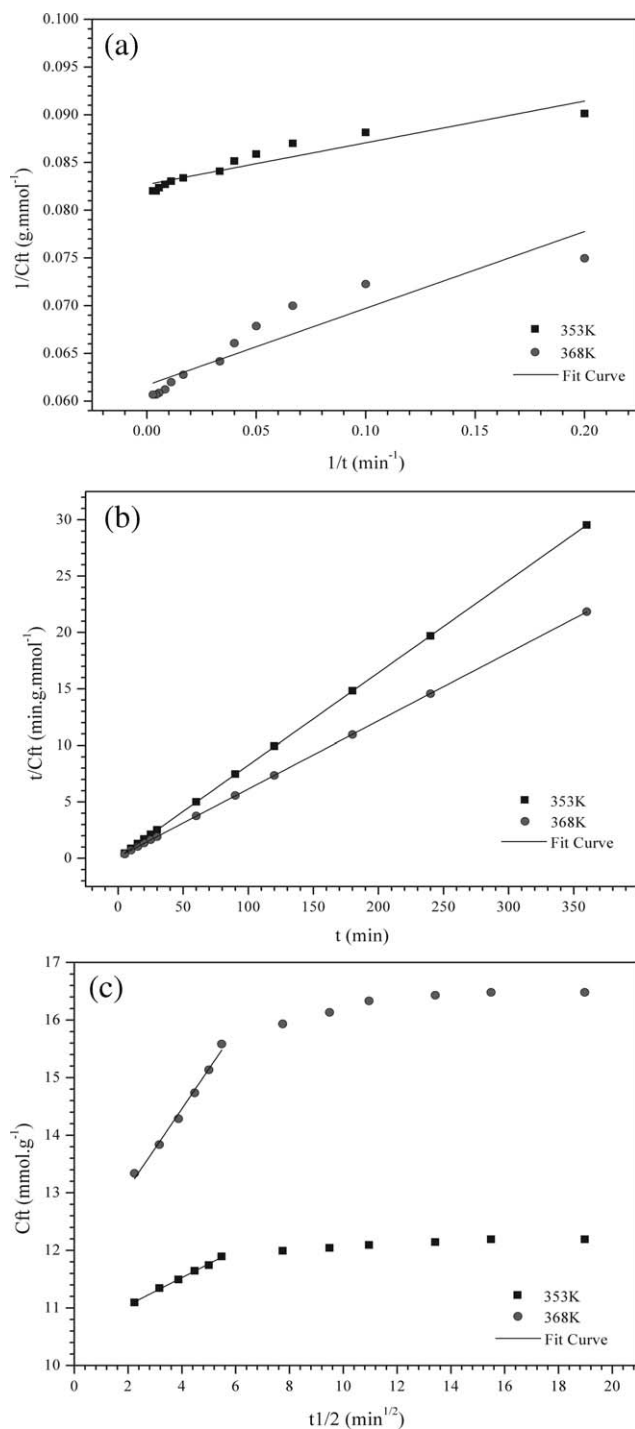
Figure 4 The fitting of experiment data to the various kinetics models. (a) Pseudo-first-order model, (b) pseudo-second-order model, and (c) intraparticle diffusion model.

Adsorption kinetic parameters

The pseudo-first-order, pseudo-second-order, and an intraparticle diffusion equations were applied to model the kinetics of aniline adsorption onto PET fibers. The pseudo-first-order rate expression[17] is presented as:

$$\frac{1}{C_{ft}} = \left(\frac{k_1}{C_{fe}}\right)\left(\frac{1}{t}\right) + \frac{1}{C_{fe}}, \tag{4}$$

the pseudo-second-order kinetic model[18] is given as:

$$\frac{t}{C_{ft}} = \frac{1}{k_2 C_{fe}{}^2} + \frac{t}{C_{fe}}, \tag{5}$$

and the intraparticle diffusion equation[18,19] can be written by the following:

$$C_{ft} = k_p t^{1/2} + C_p, \tag{6}$$

where C_{fe} is the amount of aniline adsorbed per unit mass of adsorbent at equilibrium (mmol/g), C_{ft} is the amount of aniline adsorbed at different times (mmol/g), k_1, k_2, and k_p are the rate constants of pseudo-first-order model, pseudo-second-order model, and the intraparticle diffusion model [mmol/(g min)], respectively, and C_p is the intercept for the intraparticle diffusion equation.

The straight-line plots of $1/C_{ft}$ versus $1/t$ for the pseudo-first-order reaction [Fig. 4(a)] and t/C_{ft} against t for the pseudo-second-order reaction [Fig. 4(b)] have been tested to obtain the rate parameters. The kinetic rate constants of first-order adsorption (k_1), the rate constants of second-order adsorption (k_2), and intraparticle diffusion rate constants (k_p) in Table I were calculated. It was seen that experimental data fitted pseudo-second-order equation rather than pseudo-first-order equation. The correlation coefficient for the second-order kinetic model is greater than 0.9999, indicating the applicability of this kinetic equation and the second-order nature of the adsorption process of aniline on PET fibers. The pseudo-second-order kinetic model can be used to accurately predict the amount of monomer adsorbed at different contact times and at equilibrium.

The values of the adsorbed aniline onto polyester fabric, C_{fe2}, increased from 12.2220 mmol/g at 353 K to 16.5948 mmol/g at 368 K (Table I), indicating that a high temperature favors aniline adsorption on polyester fibers. Comparing the k_2 constants at 353 and 368 K, it is known that the higher the temperature the slower is the rate of adsorption, which benefits the uniform sorption of aniline on fibers.

In addition to the adsorption of molecules on the sites of the sorbent material, the diffusion of the molecules in the boundary layer around the particle and in the porosity of the sorbent can be the limiting processes.[20,21] The first-order and pseudo-second-order models cannot identify the diffusion mechanism, and the kinetic results were then subjected to analyze by the intraparticle diffusion model for diffusion mechanism. If intraparticle diffusion is rate limited, then plots of C_{fe} versus square root of time, $t^{1/2}$, would result in a linear relationship.[22]

	Pseudo-first-order model			Pseudo-second-order model			Intraparticle diffusion model		
T(K)	k_1 (1/min)	C_{fe1} (mmol/g)	R_1^2	$k_2 \times 10^5$ [g/(mmol min)]	C_{fe2} (mmol/g)	R_2^2	k_p [mg/(g min$^{1/2}$)]	C_p	R_p^2
353	0.5287	12.0934	0.8829	0.0856	12.2220	0.9999	0.2393	10.5699	0.9966
368	1.3046	16.2127	0.8541	0.0285	16.5948	0.9999	0.6890	11.7055	0.9884

Figure 4(c) exhibits an initial portion due to an external surface adsorption or instantaneous adsorption and a linear portion followed by a plateau to equilibrium. Predominantly, aniline adsorption took place rapidly by external mass transfer followed by intraparticle diffusion. The slope of the corresponding linear region was defined as the intraparticle diffusion parameter k_p [mg/(g min$^{1/2}$)], and the calculated k_p values at 353 and 368 K were found to be 0.2393 mg/(g min$^{1/2}$) with an R^2 value of 0.9962 and 0.6890 mg/(g min$^{1/2}$) with an R^2 value of 0.9884 (Table I).

It was found that the linear plots in Figure 4(c) did not pass through the origin, indicating that intraparticle diffusion was not the sole rate-limiting step and other processes including the diffusion in the boundary layer may control the rate of adsorption.[23] Table I also shows that the higher the temperature, the larger was the value of intercept, C_p, which can give an idea about the boundary layer thickness, implying that the boundary layer effect at high temperature would become a little greater.[17] The final equilibrium adsorption process occurred after the intraparticle diffusion of aniline into the fibers.

Adsorption thermodynamics of monomers on PET fibers

To construct relationship between the amount of monomer adsorptions and the equilibrium concentrations, three models for equilibrium description of sorption behavior were selected in this study. The linear form of these three models, the Henry, Langmuir, and Freundlich isotherms, can be expressed as eqs. (7), (8), and (9), respectively:

$$C_{fe} = K_H C_{se} \tag{7}$$

$$\frac{1}{C_{fe}} = \frac{1}{n_L} + \frac{1}{K_L n_L C_{se}} \tag{8}$$

$$\ln C_{fe} = \ln K_F + n_F \ln C_{se}, \tag{9}$$

where C_{fe} and C_{se} are the equilibrium concentrations of aniline in respective phases of the PET fiber and the solution, K_H, K_L, and K_F are the absorption con-

stants in the Henry type, the Langmuir type, and the Freundlich type, respectively, n_L is the absorption saturation value in Langmuir type, and n_F denotes the degrees of deviation from isotherm linearity in Freundlich type. The linear fitting results of experimental data are presented in Figure 5(a–c). As seen from Figure 5 and Table II, all of the three adsorption isotherms were all suitable to be applied in predicting the adsorption of monomers on polyester fibers with high correlation coefficients ($R^2 > 0.99$). On the basis of the R^2 values shown in Table II, it was noted that compared with the Freundlich and Langmuir isotherms, the linear isotherm obeying Henry's law, which can be considered as a limit case of Langmuir and Freundlich types,[24,25] provided a best fit for the observed sorption data over the tested concentration range with correlation coefficients (R^2) larger than 0.998 in all cases. This implied that the sorption mechanism for monomers dissolving in amorphous fiber region and the solvate theory of solutions was applicable at below 368 K, which commonly represented the dyeing mechanism between disperse dye and PET fibers.[26,27] In addition, as shown in Figure 5(a), the slope of the straight line of Henry-type isotherm, which can give the value of the partition coefficient K, increased with the temperature, implying that higher solution temperature exerts a positive effect on the thermodynamic adsorption.

Influence of temperature on monomer exhaustion

When adsorption equilibrium reached, no more aniline monomer can go on to the fibers and some left in the waste water as a pollutant. Although anilines are inexpensive commercial reagents, it is wise to improve the adsorption capacity of fibers to aniline and to estimate the amount of aniline in fibers quantitatively as possible for economic and environmental reasons. The dyebath exhaustion percentage (%) of the dyed fiber is usually determined after dyeing process is completed. "Monomer exhaustion (%)" was introduced in this article to express the degree of aniline transfer from monomer solution to fibers. Experimental data showed that aniline exhaustion was regardless of initial concentration, and, as shown in Figure 6, this value increased with increasing the temperature and can be maintained at 65–70% at 368 K.
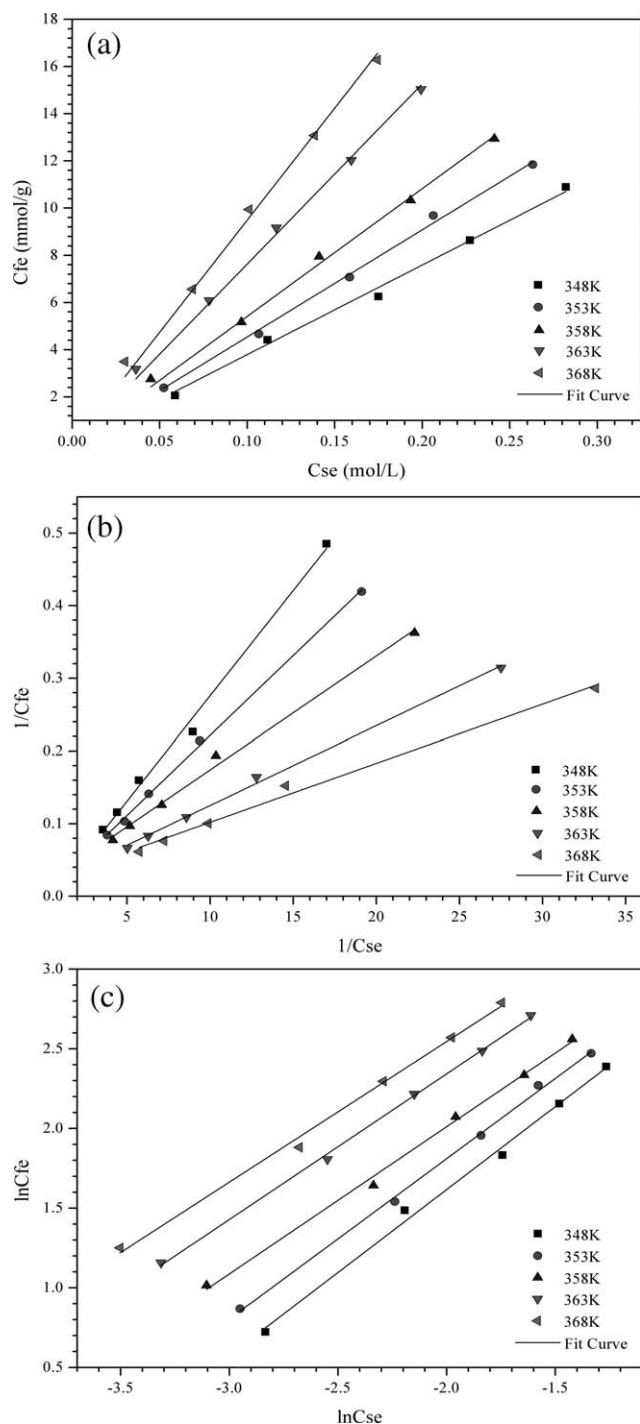
Figure 5 The adsorption isotherm fits of experimental data using three models: (a) Henry model, (b) Langmuir model, and (c) Freundlich model.

In situ formation of PANI on PET fibers

Aniline, as a plasticizer for PET, can promote structural modifications in this polymer, and the absorption of aniline has great important influence on the structure of PET fibers, resulting in the conductivity of the final composite.[28] Besides, the oxidation polymerization of aniline molecules inside fiber

TABLE II
Correlative Coefficient (R^2) for Three Adsorption Models

T (K)	Henry model	Langmuir model	Freundlich model
348	0.9989	0.9934	0.9936
353	0.9994	0.9974	0.9979
358	0.9992	0.9938	0.9958
363	0.9993	0.9968	0.9984
368	0.9988	0.9903	0.9947

substrates, that is the chain growth of polyaniline, also has a significant influence on the order and crystallinity of conductive polymers. A number of articles have discussed the mechanism of *in situ* PANI film formation[29,30] and polymerization conditions[31,32] only based on the solid substrates immersed in the reaction mixture during the oxidation of aniline. Limited attention has been paid to the oxidation research after the stage of aniline sorption, and the better understanding of the film formation mechanism is likely to improve the quality of conductive PANI coating on the surface of fiber substrates. As the previous research results shown, PANI chains grow in oxidized pernigraniline form during the consequent polymerization, and after the oxidant is consumed, pernigraniline is reduced by the residual aniline molecular to emeraldine base, which has been supported by some spectral studies.[33,34]

In this experiment, the aniline-adsorbed fabric was dipped into the oxidant bath and taken out immediately. It should be noticed that in our trial, as a result of no addition of aniline molecule in aqueous medium provided to grow PANI chain, the oxidant conditions, especially the oxidant concentration, must be controlled according to the amount of monomer adsorbed on fibers. On the assumption that the ratio of the oxidant solution to fabric (liquor ratio) and that of the monomer solution to fabric equaled, the optimal molar ratio between initial
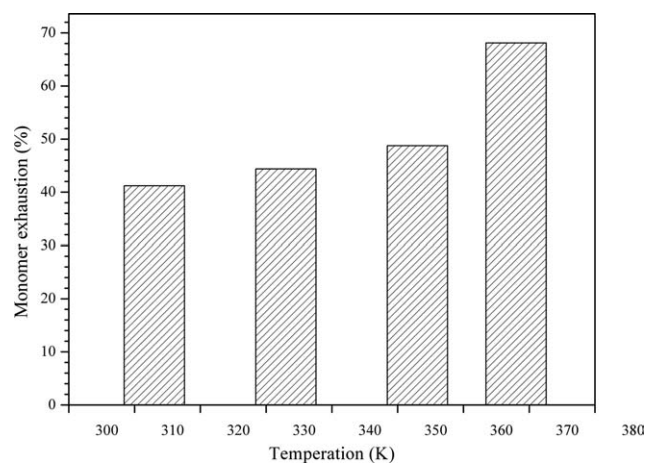

Figure 6 The monomer exhaustion at different temperatures (aniline concentration = 0.5mol/L).
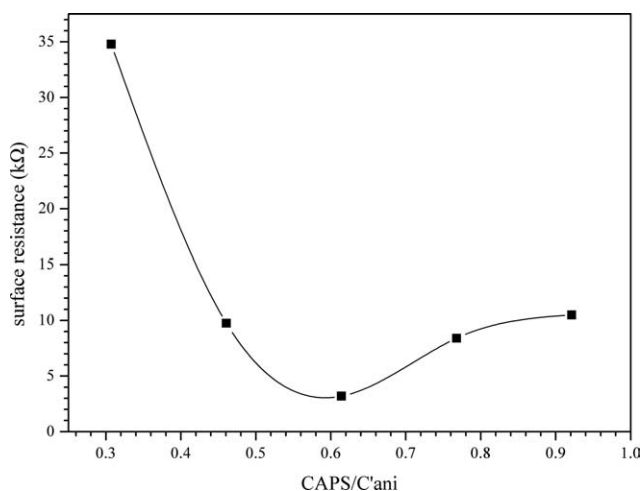
Figure 7 The change of surface resistance with the ratio of C_{APS} to C'_{ani} (aniline concentration = 0.5 mol/L; adsorption temperature = 368 K).

concentration of APS, C_{APS} (mol/L), and the exhausted concentration of aniline at the adsorption equilibrium, C'_{ani} (mol/L), $C'_{ani} = C_0 - C_e$, where C_0 and C_e are the concentrations of aniline in solution at the initial and equilibrium time (mol/L), is determined in Figure 7. It was clear that the electrical surface resistance of PANI/PET textile decreases with the ratio of C_{APS} to C'_{ani} and reached a minimum value at 0.6, where the lower value of electrical surface resistance reached about 3.2 kΩ, and then tended to increase. It presumably can be caused by the aniline depletion in the reaction zone and the formation of overoxidative PANI at high oxidant concentrations, leading to a loss of conductivity.[13]

In addition, at the early stage of polymerization, aniline monomers adsorbed on fibers were oxidized primarily to aniline cation radicals under the acidic condition. Then, the cation radicals interacted with the aniline molecules or cation radicals adsorbed on the surrounding surfaces leading to the successive film growth composed of PANI macromolecules anchored to polyester fibers, while the polymerization in the bulk gave rise to a PANI precipitate. The effect can be explained by the heterogeneous catalysis of the PANI chain initiation afforded by a surface. Electron micrographs (SEM) in Figure 8 demonstrate the pristine nature of the chemically grown polymeric films. There was an even distributed and dense deposition generated on the surface of PET fabrics [Fig. 8(a,b)], which was squeezed by two-roll padder after taken out of the oxidation bath. As Figure 8(a) shown, the film exhibited uniformly globular PANI particles of ∼ 500–1000 nm diameter and a compact and cohesive structure [Fig. 8(b)], which contributes to the conductivity. It can be clearly seen in Figure 8(c) that massive quan-

tities of the loose sediment emerged on the fibers that was not squeezed. These phenomena were due to the fact that a padder push can promote the uniform dispersion of the oxidant in the inner of fiber as well as the interstice between fibers, diminishing the yield of PANI precipitate in an aqueous medium at the liquid/solid interface effectively.
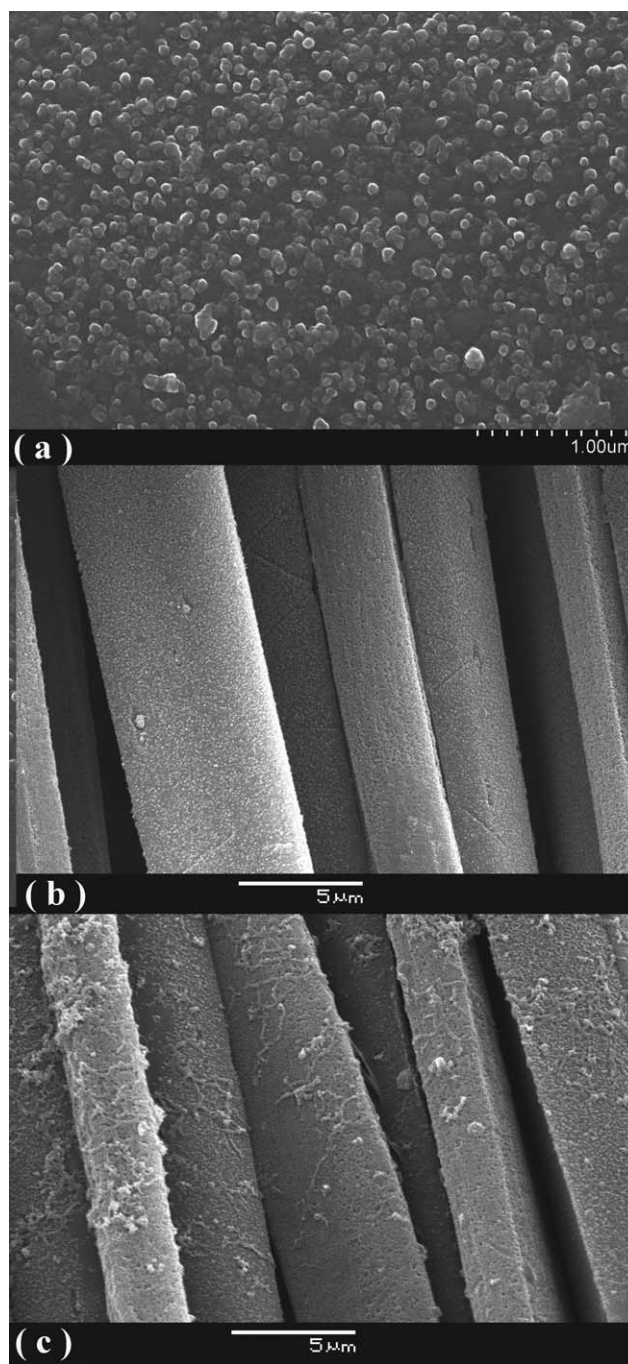

Figure 8 SEM micrographs of the composite fabric (aniline concentration = 0.5 mol/L; adsorption temperature = 368 K; APS concentration = 0.2 mol/L) (a) with squeezed (high magnification), (b) with squeezed (low magnification), and (c) without squeezed.

CONCLUSIONS

Conductive PANI-based textiles can be fabricated by two-step process, involving monomer sorption and consequent oxidative polymerization on fibers. Effects on the former stage of monomer adsorption on polyester fibers were discussed in this article. The adsorption kinetics of aniline corresponding to polyester fabric obeys preferably the pseudo-second-order kinetic, which provides the best correlation of the data. The adsorption process was found to be of a complex nature consisting of both external transfers at the earlier stages and intraparticle diffusion at the later stages. The correlation coefficient determined for linear transformation of isotherm analysis, the Henry isotherm model, which fits the experimental data reasonably well, was found to provide the best prediction for the adsorption of aniline. The monomer exhaustion increased with the increase of the adsorption temperature with regardless of initial concentration and can be maintained at 65–70% at 368 K. The lower electrical surface resistance of composite fabric, the value of which reached up to 3.2 kΩ, was obtained when the optimal molar ratio of 0.6 between the oxidant concentration and the exhausted concentration of monomers at the adsorption equilibrium was applied. SEM studies reveal a uniform and dense PANI film formed on the PET surface by the oxidation of aniline molecules previously adsorbed on fibers not the aniline monomers in bulk.

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