

Scalable Sulfonate-Coated Cotton Fibers as Facile Recyclable and Biodegradable Adsorbents for Highly Efficient Removal of Cationic Dyes

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Abstract

Adsorbents with superior adsorption capacity and facile recyclability are viewed as promising materials for dye wastewater treatment. In this work, a novel sulfonate decorated cotton fiber as a biodegradable and recyclable adsorbent was fabricated for highly efficient removal of cationic dyes. Herein, the poly(sodium p-styrenesulfonate-co-N-methylol acrylamide) (P(SSNa-co-NMAM)) with SSNa units as adsorption sites and NMAM units as thermal-crosslinking points was synthesized for modification of cotton fibers in a large scale at high temperature (160 °C). The various characterization investigations confirmed the successful construction of the P(SSNa-co-NMAM) coated cotton fibers (PCF). As expected, the as-obtained adsorbent presented outstanding adsorption performance toward cationic dyes in the both static and dynamic states, even in the synthetic effluent. The adsorption processes of cationic dyes onto the PCF were well fitted by the Langmuir isotherm model and Pseudo-second-order kinetics, respectively. The thermodynamics study showed that the adsorption reaction of the cationic dyes onto PCF was a spontaneous and endothermic process. The maximum adsorption capacities of PCF toward MEB, RhB and MG were 3976.10, 2879.80 and 3071.55 mg/g, respectively. The responsible adsorption of dyes onto the PCF was electrostatic interaction. Moreover, the adsorption capacity of PCF toward cationic dyes was slightly influenced by pH value of solution, because of the stable feature of sulfonate moiety in acid and alkali. In addition, the as-prepared PCF exhibited satisfactory recyclability and reusability. Given the aforementioned results, the as-obtained PCF is a promising adsorbent with great potential for practical application in the dye-contaminated wastewater remediation.

1. Introduction

Synthetic dyes are extensively employed in many fields to impart color including textile, leather tanning, cosmetics, and printing industries (Han et al. 2009; Janaki et al. 2013; Abdi et al. 2017; Guerra et al. 2017). It is reported that more than 100,000 commercially available dyes following the rate of 7×10^5 tons per year are generated and approximately two percent of the dyes in industrial effluents are let out into aqueous systems (Abdi et al. 2017). Notably, majority of these dyes are toxic, teratogenic and even carcinogenic, resulting in a serious threat to aquatic living organisms and human health (Janaki et al. 2013; Hu et al. 2021). To date, various methods such as adsorption (Ma et al. 2019; Zhao et al. 2021a), photocatalysis (Zou et al. 2021), filtration (Han and Wu 2019), and oxidation (Priyadharshini, Aravind Hosimin et al. 2018) have been developed for treatment of dye contaminated wastewater. Among them, the adsorption is viewed as one of the most promising methods, due to its features of the low cost, high effectiveness and easy operation (Zhou et al. 2017; Zhao et al. 2021a). Now, developing efficient and eco-friendly adsorbents for removal of dye pollutants is key point for wastewater treatment.

With the development of polymer chemistry, numerous functional polymers have been designed and prepared as adsorbents for dye removal (Ma et al. 2019; Zhao et al. 2020; Yang et al. 2021b). Compared to traditional adsorbents such as activated carbon (Özhan et al. 2014), clay (Veli and Alyüz 2007) and zeolite (Sivalingam et al. 2019), these polymers presented advantages in high adsorption capacity, facile processability and multi-functional features et.al (Zhao et al. 2020; Yang et al. 2021b). Recently, sulfonate

group has been widely utilized to fabricate various negatively charged adsorbents to remove cationic dyes (Xu et al. 2018; Xiang et al. 2019; Zhao et al. 2020). It can bind dyes through the strong interaction between the dye molecules and the sulfonic groups. Moreover, different from other charged groups like carboxyl, the sulfonic group is less impacts in harsh conditions (Jin et al. 2020). Therefore, sulfonate based adsorbents are promising candidates for cationic dye removal. Wei's group fabricated sulfonate functionalized nanodiamonds for ultrafast removal of methylene blue (MEB) with high efficiency (Lei et al. 2020). Hong et.al used a simple method for constructing sulfonate functionalized graphene as adsorbent with outstanding capacity and green regeneration for cationic dye removal (Hong et al. 2021). However, such sulfonate based adsorbents faced troubles in reclamation difficulty, cumbersome preparation routes and high cost, which significantly hinder their practical applications. Fortunately, Zhao's group facilely prepared sulfonate groups decorated nanofibrous membranes and polyurethane sponges with excellent reusability (Xu et al. 2018; Jin et al. 2020). Nevertheless, the adsorption capacity of these adsorbents was sacrificed due to the decrease of the amount of sulfonate groups. Moreover, the reported sulfonate based adsorbents were non-biodegradability and may cause secondary pollution to the environment (Qi et al. 2019). Therefore, how to the facile fabrication and large-scale production of novel sulfonate based adsorbents with high adsorption capacity, low cost, satisfactory recyclability and good biodegradability is still highly desirable.

Cotton fiber is mainly composed of cellulose, which is the most abundant renewable and biodegradable natural biopolymer (Tserki et al. 2003). Moreover, cotton fibers can be produced in large-scale with low cost (Nabi Saheb and Jog 1999; Tserki et al. 2003). Most importantly, lots of wasted cotton fibers are generated during in processing. Therefore, how to impart these cotton fibers with higher additional value is meaningful. Recently, in view of the unique advantages of the cotton fibers, numbers biosorbents derived from cotton fibers were developed for dyes removal (Xiong et al. 2014; Yang et al. 2021b; Zou et al. 2021; Krishnamoorthi et al. 2022). For example, Chen's group utilized both cationic monomer and anionic monomer to modify the cotton fibers to form an efficient biosorbent for dyes removal (Xiong et al. 2014). Krishnamoorthi and co-workers prepared a biodegradable caffeic acid/chitosan polymer coated cotton fiber as an adsorbent for dye wastewater treatment (Krishnamoorthi et al. 2022). In addition, our group was scalable preparation of CO₂-reposeive cotton fibers for removal of anionic dyes with ultrafast and selective features (Yang et al. 2021b). Inspiration from these successful works, the scalable development of sulfonate decorated cotton fibers as adsorbents with highly efficient performance for cationic dye removal is expected.

In this work, sulfonate decorated cotton fiber as a novel biosorbent is fabricated via a simple approach for application in the treatment of dye wastewater (Scheme 1). First of all, poly(sodium p-styrenesulfonate-co-N-methylol acrylamide) containing thermo-crosslinking moiety is prepared. Subsequently, the as-prepared polymer was coated on the cotton fibrous surface via thermo-crosslinking to form the targeted adsorbent. The adsorption properties of the as-prepared adsorbent towards dyes are studied in the both static and dynamic states. The adsorption kinetics, isotherms, and mechanism of cationic dyes by the obtained adsorbent are investigated. The effects of the initial pH, temperature and

ionic strength on the adsorption performance of adsorbent are systematically analyzed. The dye adsorption behavior of the as-fabricated adsorbent in the synthetic effluent is investigated. Finally, the recyclability of the as-prepared adsorbent is also evaluated.

2. Experimental Section

2.1. Materials

Cotton fibers were purchased from Suining Kangda Sanitary Materials Co. LTD, China. Sodium p-styrenesulfonate (SSNa, $\geq 90\%$) was obtained from Aladdin Co., Ltd. (Shanghai, China). Methylene blue (MEB, $\geq 98\%$), rhodamine B (RhB, $\geq 99\%$), malachite Green (MG, $\geq 99\%$), methyl orange (MO, $\geq 98\%$), naphthol green B (NGB, $\geq 99\%$), congo red (CR, $\geq 98\%$) and *N*-Methylol acrylamide (NMAM, $\geq 98\%$), were purchased from Macklin Biochemical Co., Ltd, Shanghai, China. Potassium persulfate ($K_2S_2O_8$, $\geq 98\%$), sodium chloride (NaCl, $\geq 99.5\%$), potassium carbonate (K_2CO_3 , $\geq 99.8\%$), dipotassium hydrogenphosphate (K_2HPO_4 , $\geq 98\%$), calcium chloride ($CaCl_2$, $\geq 99\%$), magnesium sulfate heptahydrate ($MgSO_4 \cdot 7H_2O$, $\geq 99\%$) and ethanol (CH_3CH_2OH , 99.7%) were acquired from Chengdu Kelong Chemical Co., Ltd.. The deionized (DI) water used in this study was prepared in ultrapure water obtained from Shanghai Li Chen Bangxi Instrument Technology Co., Ltd. (Shanghai, China).

2.2. Synthesis of P(SSNa-co- NMAM)

The poly(sodium p-styrenesulfonate-*co-N*-methylol acrylamide) (P(SSNa-*co*-NMAM)) copolymer was prepared by the method of a free radical polymerization. Typically, SSNa (2 g, 9.670 mmol), NMAM (0.109 g, 1.078 mmol), and $K_2S_2O_8$ as a initiator (0.0844 g, 0.312 mmol) were added into 20 mL of DI water to obtain a homogeneous solution. Next, the mixture was purged nitrogen for 20 min to remove oxygen before putting the flask into pre-heating oil bath at 70°C. After 16h reaction, the mixture was diluted with DI water and washed repeatedly with excess ethanol and added to DI water three times, and then vacuum dried at 40°C for 20 h to get the targeted copolymer, the yield was 93.1%.

2.3. Preparation of P(SSNa-*co*-NMAM)-coated cotton fibers

After successful preparation of the P(SSNa *co* NMAM)NMAM), a series of cotton fibers with different masses of P(SSNa *co* NMAM) were fabricated by the simple impregnation method. Herein, taking the P (SSNa *co* NMAM) solution with 5.0 wt% of concentration as an example, first of all, a dip-coating solution was formed via dissolution of a certain amount of P(SSNa-*co*-NMAM) in 20 mL of DI water .Subsequently, 160 mg of the cotton fibers was dipped into the above mentioned solution for 20 min, and then the immersed cotton fiber was treated at 160 °C for 60 min to form the stable P(SSNa-*co*-NMAM) layer on the cotton fibrous surfaces due to the thermo-crosslinking reaction. Finally, the uncross-linked P(SSNa-*co*-NMAM) on the coated cotton fiber surfaces were removed by excess DI water and vacuum dried at 80°C for 12 h, named as PCF-5. Additionally, the other P(SSNa-*co*-NMAM) coated cotton fibers (PCFs) were also synthesized with varying concentrations of polymeric solutions (1.0, 3.0, and 7.0 wt %) for comparison, which were denoted as PCF-1, PCF-3, and PCF-7, respectively.

2.4. Characterization

¹H NMR was conducted on a Bruker AV III HD 400 MHz NMR in D₂O at room temperature. The Fourier transform infrared spectra (FTIR) were operated by a Bruker Vertex 70 spectrometer about a wavenumber range of the 500-4000 cm⁻¹. X-ray photoelectron spectroscopy (XPS, Kratos AXIS Supra apparatus) was adopted to detect the surface chemical elements of samples. The morphologies of the samples were observed by field-emission scanning electron microscope (SEM, JSM-7500F). The instrument was operated at an acceleration voltage of 15.0 kV after gold spraying process. The UV-Vis spectrometer (Beijing Purkinje General Instrument Co., Ltd.) was used to measure the dye solution's absorbance.

2.5. Adsorption experiments

Herein, the batch adsorption experiments were done by varying parameters including initial dye concentration, solution pH, contact time, ionic strength, and temperature to study the adsorption behaviors of the PCFs towards cationic dyes. About 10 mg of the adsorbent was putted into the 20 mL of dye solutions with different initial concentrations (200-2400 mg/L) to investigate the adsorption performance. The experimental contact time ranged from 10 min to 60 min. The pH values of the dye solution were adjusted in presence of 0.01 mol/L HCl and 0.01 mol/L NaOH solutions. The effect of ionic strength for adsorption of dye was evaluated by varying concentrations of NaCl solution ranging from 0.0 mol/L to 0.6 mol/L. The adsorption thermodynamic studies were performed with temperature from 298 K to 318 K. After dye adsorption equilibrium, the adsorbent was filtered off and the final concentration of dyes in the aqueous solution was determined using a UV-Vis spectrophotometer. The batch adsorption experiments were performed three times and the corresponding average values were given. Herein, the adsorption capacity (Q_t) of cationic dyes uptake by PCFs was calculated using the following equation (Zhao et al. 2021a):

$$Q_t = \frac{V(C_0 - C_t)}{m} \quad (1)$$

Where C_0 and C_t are the initial dye concentration and the dye concentration in the solution at a certain time (t), V is the volume of the solution, and m is the mass of adsorbent (g).

2.6. Recyclability

The adsorbent's recyclability was evaluated by the adsorption-desorption process of the spent adsorbent from batch adsorption studies. The as-prepared PCF was placed into 50 mL of 50 mg/L MEB solution for 60 min. For the desorption and regeneration, the MEB adsorbed PCF was performed via immersing it into 0.1 mol/L HCl solution for desorption, then washed with excess DI water and vacuum dried at 70 °C for 8 h to yield regenerated PCF. Finally, the regenerated PCF was used for following cycles.

3. Results And Discussion

3.1. Preparation and Characterization

Herein, the P(SSNa-*co*-NMAM) was synthesized by free radical polymerization. In the P(SSNa-*co*-NMAM), SSNa contains sulfonic acid moieties as active sites for cationic dye adsorption (Goswami and Phukan 2017), and the function of NMAM units are thermo-crosslinking points (Yildiz et al. 2001). The successful preparation of the P(SSNa-*co*-NMAM) was demonstrated by the FT-IR and ¹H NMR tests. In Figure 1a, the characteristic adsorption bands of sulfonic acid groups at 1007 cm⁻¹ and 1035 cm⁻¹ can be observed on corresponding FTIR spectra. Further, the chemical composition of the synthesized copolymer can be confirmed by ¹H NMR spectrum (Figure S1). It suggested that approximately 9.8 mol% of NMAM units in the copolymer after calculating the integral areas between the peak at 6.5 ppm from the SSNa units and the broad peak at 0.6 ~ 2.2 ppm from the backbone of P(SSNa-*co*-NMAM), which was close to the NMAM feeding ratio of 10 mol%. These results of ¹H NMR further confirmed the target P(SSNa-*co*-NMAM) was successfully synthesized. Subsequently, a cotton fiber with P(SSNa-*co*-NMAM)-coating was fabricated by sample thermo-crosslinking. Herein, NMAM was employed as the thermo-crosslinker to anchor P(SSNa-*co*-NMAM) on the cotton fibrous surface due to N-methylol moieties highly efficient reaction with -OH groups from the cellulose and themselves at high temperature (Figure S2) (Yin et al. 2011).

To explore the chemical structures of as-prepared P(SSNa-*co*-NMAM) coated cotton fibers, the FTIR measurement was employed. The results were shown in Figure 1a. From the spectrum of PCFs, the adsorption bands at 1497 cm⁻¹ and 1640 cm⁻¹ were attributed to the skeletal vibration of the aromatic ring (C=C) from SSNa units, and the characteristic adsorption bands at 1007 cm⁻¹ and 1035 cm⁻¹ belonged to the sulfonic sodium groups from SSNa units. Meanwhile, the characteristic adsorption band at 833 cm⁻¹ and 1408 cm⁻¹ were assigned to C=O stretching and the C-N stretching vibration of the secondary amide for NMAM units. Therefore, these results imply successful formation of P(SSNa-*co*-NMAM) polymeric layers on the cotton fibrous surfaces. As expected, the intensity of these characteristic absorption bands from the P(SSNa-*co*-NMAM) increased when cotton fibers were introduced into the higher concentration coating solution (Figure 1a), further indicating that the coated polymer amount on the cotton fibers increased as polymeric solution concentration increased. Moreover, the mass change of the PCF before and after treatment were summarized in Table S1, which suggested the coated weight of the P(SSNa-*co*-NMAM) on the cotton fibers.

Afterward, the surface chemical composition of the PCF was analyzed by the XPS. As shown in Figure 1b, the main peaks at 288.5, 535.5 and 981.5 eV in cotton fibers were ascribed to the binding energy of C 1s, O 1s and O (A), respectively. However, new peaks appeared at 1075.5 eV, 500.6 eV, 233.4 eV, 172.5 eV and 402.5 eV can be observed after immobilization of P(SSNa-*co*-NMAM) on the cotton fibers, which were assigned to the S 2s, S 2p, Na 1s, Na (A) and N 1s, respectively.

Next, the micromorphology of the PCFs was visualized through SEM measurement, and the corresponding SEM images were presented in Figure 2. As shown in Figure 2a-c, the pristine cotton fiber has a smooth surface. However, the P(SSNa-*co*-NMAM) coated cotton fibrous surfaces could clearly observe polymeric layer (Figure 2d-f). Moreover, the SEM images can verify that as-prepared samples with

different loading amounts of the P(SSNa-co-NMAM) could be easily adjusted by the concentration of the polymeric solution (Figure S3). However, the concentration of the polymeric solution was 7%, the intact polymeric layer could be formed among the cotton fibers, which would significantly decrease the specific surface area of the samples. Furthermore, the energy-dispersive spectroscopy (EDS) of the sample was performed. The corresponding EDS spectrum (Figure 2g) and EDS mapping images (Figure 2h-k) were presented. The elemental mapping demonstrated a homogeneous distribution of Na and S elements derived from SSNa units on the PCF, indicating that P(SSNa-co-NMAM) are homogeneously coated on the entire cotton fibrous matrix. Until now, it can be concluded from the FTIR, XPS and SEM results that the P(SSNa-co-NMAM) coated cotton fibers were successfully prepared.

3.2. Adsorption isotherm

Next, the adsorption capacities of the as-prepared PCFs were investigated. As depicted in Figure S5, the adsorption capacity of the PCFs toward cationic dyes significantly increased with increment of the coated mass of P(SSNa-co-NMAM) on the cotton fibers. Notably, the adsorption capacity of the PCF-7 only slightly enhanced compared with that of PCF-5. The reason can be attributed to that some polymeric films were formed on the PCF-7 surface, resulting in decrease of its specific surface area compared to that of PCF-5. As a result, the active sites on the PCF-7 without significantly increased to adsorb dyes compared with that of PCF-5, implying the optimum composition ratio of the PCF-5. Thus, the PCF-5 was selected for the following experiments.

The excellent adsorption capacities are significant for absorbents which affect the practical application of materials. Hence, for investigation of adsorption isotherms of the PCF-5 toward cationic dyes, MEB, RhB, and MG were employed as the cationic dye models, and Figure S6 displays chemical structures of the dyes. As presented in Figure 3a, it could be observed that the adsorption capacity of the PCF-5 toward MEB showed an escalation tendency at the beginning of dye concentration. However, the plateau region appeared when a certain equilibrium concentration was attained. Meanwhile, the adsorption isotherms of PCF-5 toward MEB, RhB, and MG were presented in Figure 3b, which displayed the maximum adsorption capacities of 3976.10 mg/g for MEB, 2879.80 mg/g for RhB and 3071.55 mg/g for MG respectively. The difference of Q_m values may be ascribed to the varying chemical structures of dyes resulting in the different interactions between the sulfonate moieties of PCF-5 and dye molecules (Zhan et al. 2021).

Furthermore, the adsorption isotherm is beneficial to reveal the mechanism of cationic dyes onto the PCF-5. Herein, the adsorption data were fitted by the Langmuir and Freundlich empirical isotherm models. The Langmuir model proposes a monolayer sorption onto a homogeneous surface and there is no interaction between the adsorbed particles, while the Freundlich model assumes that multilayer adsorption occurs resulting in the heterogeneous adsorption systems (Song et al. 2020). The linear form of abovementioned two isotherm models are able to be described in the following equations (Zhao et al. 2021a):

The Langmuir model:

$$C_e/q_e = 1/q_m K_L + C_e/q_m \quad (4)$$

The Freundlich model:

$$\ln q_e = \ln K_F + 1/n * \ln C_e \quad (5)$$

In the two equations, q_e (mg/g) is absorption capacity (mg/g) at equilibrium and q_m (mg/g) is the maximum adsorption, C_e (mg/L) is the concentration of the dye solution at equilibrium; K_L (L/mg) is the Langmuir isotherm constant related to the free energy of adsorption; K_F (mg/g) and n are the constant of the Freundlich model and heterogeneity factor, respectively. The Langmuir and Freundlich fitting plots of PCF-5 toward MEB, RhB and MG dyes were presented in Figure 3c-d. The correlation coefficients (R^2) and model parameters were exhibited in Table 1. By the judgment on the values of the R^2 , the Langmuir model was more suitable in elucidating the adsorption process of cationic dyes onto the PCF-5 in comparison with Freundlich isotherm model, indicating that monolayer adsorption occurs. Noteworthy, the dye adsorption capacity of PCF-5 is more competitive compared with numerous similar adsorbents (Table 2), which further confirms that the PCF-5 may be a promising adsorbent and can be used in wastewater treatment.

Table 1 The results of adsorption isotherms of cationic dyes onto PCF-5

dyes	Langmuir			Freundlich		
	K_L (L/mg)	Q_m (mg/g)	R^2	K_F (L/mg)	n	R^2
MEB	0.020588	3965.10	0.99356	101.83	1.793	0.86035
RhB	0.010344	2897.80	0.9983	79.13	1.955	0.90247
MG	0.006268	3074.55	0.95121	6.64	0.959	0.85878

Table 2 The adsorption performance of the PCF-5 comparison of other adsorbents.

Adsorbent	absorbate	$^a q_{\max}$ (mg/g)	Ref
PP-PDA	$^b \text{MEB}$	434.8	(Zhan et al. 2021)
Sulfonate-grafted CMPs	MEB	1650	(Zhao et al. 2020)
PSS-GR	MEB	811	(Hong et al. 2021)
EA-4-6 NFM	MEB	2257.9	(Xu et al. 2019)
PSBMA-NaSS	MEB	760	(Xiang et al. 2019)
ND-SO ₃ H	MEB	200.8	(Lei et al. 2020)
PSSNa/PMMA	MEB	56	(Zhang et al. 2016)
PCF-5	MEB	3976.2	this work
QPVA/TEOS hybrid membrane	$^c \text{RhB}$	34.16	(Zhang et al. 2017)
H ₂ O ₂ -modified OBDCA	RhB	50	(Zhao et al. 2021b)
PCF-5	RhB	2879.8	this work
Modified Irvingia gabonensis	$^d \text{MG}$	250	(Abdi et al. 2019)
Bentonite	MG	178.6	(Bulut et al. 2008)
PCF-5	MG	3071.6	this work

* $^a q_{\max}$ is the maximum adsorption capacity. Cationic dye: $^b \text{MEB}$, methylene blue; $^c \text{RhB}$, rhodamine B; $^d \text{MG}$, malachite green.

3.3. Adsorption kinetics

In order to better inquire into the adsorption behaviors of the PCF-5, the effect of contact time on the adsorption of cationic dyes on adsorbent was investigated. In Figure 4a, it was clearly observed that the adsorption equilibrium time of dyes increased with the increment of molecular weight of dyes. This result can be explained that dye with higher molecular weight has weaker thermal motion ability and greater steric hindrance, thus it is not easy to reach the adsorption site (Hussain et al. 2022). Generally, the PCF-5 toward cationic dyes exhibited rapid adsorption rate, especially in the first 10 min, due to the presence of abundant active adsorption sites, and then gradually reached to adsorption equilibriums within 50 min. Selecting MEB as an example, a rapid adsorption rate of the PCF-5 was verified by the UV-vis spectra. As we can see from Figure 4b, as the prolongation of adsorption time the absorbance intensity rapidly disappeared, suggesting the rapidly decreased MEB concentrations in the solution within the short time, the corresponding optical picture also implying this phenomenon (Figure 4c).

Adsorption kinetic studies provide important information to reveal the mechanism of the dye adsorption process. Herein, the adsorption processes of PCF-5 toward MEB, RhB and MG were described via the pseudo-first-order (Eq. (2)) and pseudo-second-order (Eq. (3)) kinetic models, respectively, as expressed in the following equations (Yang et al. 2021a):

The pseudo-first-order model:

$$\ln (q_e - q_t) = \ln q_e - k_1/2.303 t \quad (2)$$

The pseudo-second-order model:

$$t/q_t = 1/k_2q_e^2 + t/q_e \quad (3)$$

Where q_e is the amount of adsorbed dye (mg/g) at equilibrium state; q_t is the adsorption amount at time t (min); k_1 (min^{-1}) and k_2 (g/mg/min) denote the rate constant of the pseudo-first-order and the pseudo-second-order adsorption kinetics, respectively. The related kinetic model parameters were listed in Table 3. It could be found that the correlation coefficients (R^2) of the pseudo-first-order kinetic model were very closer 1 than that of the the pseudo-second-order model. Moreover, $q_{e,cal}$ calculated by pseudo-second-order model was much closer to the experimental $q_{e,exp}$ as compared with the pseudo-first-order model. These results proved that the adsorption toward MEB of the PCF-5 obeys the pseudo-second-order model. Hence, these kinetic results verified that the chemical interaction between the cationic dye molecules and the sulfonic acid groups of the PCF-5 promoted the adsorption process.

Table 3 The parameters of kinetics for adsorption of cationic dyes onto PCF-5

dyes	Pseudo-first-order			Pseudo-second-order		
	k_1 (min^{-1})	$q_{e1, cal}$ (mg/g)	R^2	k_2 (g/mg min^{-1})	$q_{e2, cal}$ (mg/g)	R^2
MEB	0.1107	76.70	0.99283	0.001453	198.98	0.8768
RhB	0.1223	101.26	0.99699	0.00072	166.85	0.8337
MG	0.0785	93.22	0.99266	0.000459	171.37	0.9627

3.4. Effect of pH value

The pH value of the water sample acts a very important role in this adsorption system. The changes of the ionic or neutrality state of the target compounds and the charge of the adsorbent with pH would promote or inhibit the interaction between the adsorbates and the adsorbents, and thus affect the adsorption performance (Zhang et al. 2013; Zhao et al. 2015). In Figure 5a, the removal rate of the PCF-5 slightly decreased at pH=2 that because of the protonation of the sulfonic acid group in a highly acidic

environment. However, the PCF-5 presented an insignificant effect of pH=2-11 on the adsorption performance. These results demonstrated that the PCF-5 could maintain stable adsorption capability in a wide range of pH.

3.5. Effect of ionic strength

Ions can be found commonly in industry wastewater including textile and aquaculture wastewater. Therefore, the existence of salts in wastewater may affect the ionic strength between the adsorbent with the dye molecules (Yang et al. 2021b). Thus, the effect of ions strength was examined by addition of NaCl into the MEB solution. The result shown that the removal efficiency of PCF-5 was significantly inhibited when the salt concentration increased from 0 to 0.6 mol/L (Figure 5b). This phenomenon was probably attributed to the salt ions (Na^+) competition with MEB molecules to occupy the active adsorptive site with sulfonic acid groups from the PCF-5. Hence, the increase of salt concentration in MEB molecules will lead to the enhancement of shielding effect of salt ions on positively charged MEB molecules, thus reducing the removal efficiency of adsorbent.. Above phenomenon can be concluded that electrostatic interaction is a significant derive force in the adsorption process of cationic dyes onto the PCFs.

3.6. Effect of Temperature and Adsorption Thermodynamic Analysis

Another important parameter in adsorption process is the efficacy of temperature on the adsorption rate. Herein, the effect of temperature on the adsorption of dyes on the PCF-5 was investigated under different temperatures (298, 308, and 318 K). As depicted in Figure 6a, the removal rate of PCF-5 toward cationic dyes increased with the increment of temperature. It would be considered that the increment of the temperature facilitates the movement of cationic dye molecules and provides additional energy to enhance the interactions between the cationic dye molecules and the adsorption sites of the PCF-5.

To explore the spontaneous completion of the adsorption process and further to look into the adsorption mechanism of the PCF-5. The thermodynamic parameters of dyes adsorption onto PCF-5, including Gibbs free energy (ΔG_0), entropy (ΔS_0), and enthalpy (ΔH_0), were calculated by the following equations (Lei et al. 2020):

$$\Delta G^\circ = -RT \ln K_c \quad (2)$$

$$\ln K_c = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT} \quad (3)$$

where R ($8.314 \text{ J mol}^{-1} \text{ K}^{-1}$) is the universal gas constant, T (K) and K_c (dimensionless) are the absolute temperature and the adsorption equilibrium constant, respectively. ΔG^0 could be figured out by Eq. (3), whereas the ΔH^0 and ΔS^0 are calculated from the slope and intercept of the plot of $\ln K_c$ versus $1/T$ (Figure 6b), respectively. The corresponding parameters were listed in Table 4. The negative values of ΔG°

confirmed that the adsorption process toward MEB, RhB, and MG onto the PCF-5 was spontaneous nature, indicating that the efficiency of adsorption is more desirable at higher temperatures. Additionally, considering the positive values of ΔH° for MEB onto PCF-5 revealed that an endothermic reaction was in the adsorption process (Far et al. 2020). Furthermore, the fact that the positive value of ΔS° also revealed a randomness increase on the solid-liquid interface between PCF-5 and dye solution. These analyses were consistent with the result that higher temperatures could promote the sorption process of cationic dyes on the PCF-5.

Table 4. Thermodynamic parameters for the adsorption of PCF-5

dyes	T(K)	ΔG° (KJ/mol)	ΔH° (KJ/mol)	ΔS° (KJ/mol K)
MEB	298	-8.207	0.685	0.062
	308	-1.187		
	318	-1.421		
RhB	298	-6.025	0.288	0.035
	308	-7.752		
	318	-8.903		
MG	298	-3.607	0.235	0.024
	308	-4.886		
	318	-5.698		

3.7. Selective Adsorption and Dynamic Filtration Performance

Based on the negatively-charged property owing to the sulfonic acid groups ($-\text{SO}_3\text{H}$), the as-prepared PCF-5 was supposed to be beneficial to adsorb cationic dyes due to the electrostatic interaction (Zhou et al. 2018; Zheng et al. 2020). Three cationic dyes (MEB, MG and RhB) and three anionic dyes (MO, CR and NGB) were selected to study the adsorption performance of PCF-5, and Figure S6 shown their corresponding structures. The PCF-5 exhibited an excellent adsorption capacity for MEB, MG and RhB dyes, compared with that of MO, CR and NGB dyes (Figure S7). Based on these conclusions, we further explored the selective adsorption performance of the PCF. As observed from Figure 7a, the color of the MEB/MO mixture solution switched from dark green to yellow after being fully adsorbed by PCF-5. Moreover, the UV-vis spectra suggested that the peak at 664 nm of MEB almost disappeared after the adsorption, while the peaks at 464 nm of MO was only a slight decrease, verifying that MEB was selectively adsorbed from the MEB/MO mixture dyes (Figure 7b).

Further, the PCF-5 was expected to have a highly efficient wastewater purification through the filtration process. Therefore, the dynamic filtration performance of the PCF-5 toward dyes was investigated. As shown in Figure S8, the 20 mL of dark blue MEB solution can be purified by PCF-5 membrane with a gravity-driven force. Besides, the dynamic selective separation measurement of the PCF-5 toward mixed dyes can also be carried out. As we can see, MEB could be successfully separated by PCF-5 from MEB/MO mixture dyes during the filtration–separation process (Figure 7c). Meanwhile, the completely disappearing of the adsorption peak from MEB in UV–vis spectrum (Figure 7d) after dynamic filtration further proved that the cationic dyes can be highly effectively adsorbed onto the PCF-5 from the mixture under dynamic state owing to the high adsorption efficiency of the PCF-5. Therefore, aforementioned adsorption experiments demonstrated that the PCF-5 possessed outstanding adsorption capacity toward cationic dyes.

3.8. Adsorption mechanism

To gain a deeper understanding on the adsorption behavior of the PCFs toward cationic dyes, adsorption mechanism is discussed in detail. It is not only for potential practical application but also for further exploitation of advanced adsorbents.

As mentioned above, the adsorption process of the PCFs conformed to the Langmuir model and pseudo-second-order kinetic model indicated that was considered to greatly involve the chemical interaction between cationic dyes and PCFs. Taking MEB as an example, the potential interactions between PCFs and MEB were comprehensively investigated. Firstly, the effect of ionic strength of the solution revealed that the adsorption capacity of the PCFs toward cationic dyes significantly decreased with increasing ionic strength, implying that the main driving force of MEB adsorption onto PCFs is the electrostatic interaction (Xu et al. 2018). Furthermore, the π – π interaction might have a role in the MEB adsorption since both the MEB molecules and the PCFs have the aromatic rings (Lei et al. 2020). Thus, these possible interactions between the PCFs and cationic dyes were illustrated in Figure 8. In short, the cationic dyes adsorption of PCFs could be summarized that the major mechanism for the adsorption of dyes is electrostatic interaction, while the π – π interaction also contribution of another adsorption mechanism.

3.9. Adsorption performance in simulated dye effluent

The practical potentials of as-proposed PCF-5 was evaluated for treating a simulated dye effluent with containing multiple components (Chen et al. 2020). Table 5 listed the ingredients of the simulated dye effluent. The adsorption performance of PCF-5 on simulated dye effluent was demonstrated by the UV-vis spectra and corresponding photographs (Figure 9). In Figure 9a, the removal efficiency of PCF-5 could reach to 99.9% in the simulated dye effluent. Furthermore, Figure 9b shown that the original color of the simulated dye effluent changes from purple blue to transparency after treatment by the PCF-5. These results can further manifest that the PCF-5 showed excellent adsorption performance in contaminated wastewater remediation.

Table 5 The composition of the simulated dye effluent

Compound	λ_{\max} (nm)	Concentration (mg L ⁻¹)
MEB	664	20
RhB	554	20
MG	618	20
NaCl		10
K ₂ CO ₃		10
K ₂ HPO ₄		10
CaCl ₂		10

3.10. Recyclability

There is no doubt that the recyclability of adsorbents which is directly related to the cost-effectiveness of an adsorption process is also important indicators for its practical applications. In this work, the recyclability of the PCF-5 was evaluated via using successive cycles of adsorption-desorption. As presented in Figure 10, PCF-5 exhibited a quite stable property with a nearly constant removal efficiency during the recycling experiments. Its removal efficiency still maintained above 90% after 5 successive cycles. Meanwhile, the SEM image of the regenerated PCF-5 was presented in Figure S9a. It displayed that the cotton fibrous surfaces still coated by the P(SSNa-co-NMAM) copolymer well even after 5 successive cycles. Moreover, the S, Na and N peaks of the regenerated PCF-5 still can be obviously observed in the XPS spectra (Figure S9b). Hence, from the results of the cyclic adsorption experiments and the characterization of regenerated PCF-5, it can be concluded that the PCF-5 possessed outstanding durable feature.

4. Conclusion

In summary, a novel P(SSNa-co-NMAM) coated cotton fiber was fabricated by a simple thermo-crosslinking and successfully applied them for the adsorption removal of cationic dyes from wastewater. The maximum adsorption capacity of the as-prepared PCFs for cationic dyes was 3965.1 mg/g for MEB, 2897.8 mg/g for RhB and 3074.5 mg/g for MG, respectively. Most importantly, the PCFs exhibited the efficiently separation capability toward cationic dyes from the mixture under the both static and dynamic conditions. Interestingly, the PCFs could highly adsorb the cationic dyes from simulated wastewater. Furthermore, the PCFs exhibited excellent reusability, since the removal efficiency of the as-prepared PCFs could be maintained even above 90% after 5 cycles. Given the combined advantages of low cost, high adsorption capacity, selected adsorption and excellent durability of the PCFs, we can foresee that PCFs would become an alternative adsorbent to the commercial adsorbents for wastewater treatment or other environmental remediation applications.

Declarations

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Author contributions

Linhua Li: Conceptualization, Methodology, Investigation, Writing - original draft. **Baojie Dou:** Investigation, Visualization, Validation. **Jianwu Lan:** Supervision, Project administration, Funding, Writing - review & editing. **Jiaojiao Shang:** Software, Data curation, Visualization. **Yafang Wang:** Software, Data curation. **Jincheng Yu:** Validation. **Erhui Ren:** Data curation, acquisition. **Shaojian Lin:** Conceptualization, Investigation, Supervision, Writing - review & editing, Funding.

Ethics declarations

Conflict of interest

All authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Human and animal rights

This manuscript does not contain any studies with human participants or animals performed by any of the authors.

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Figures

Figure 1

a) FTIR spectra and **b)** XPS diagram of the samples.

Figure 2

SEM images of the cotton fibers **a–c)** and PCF-5 **d–f)** at various magnifications; EDS spectrum **g)** and element mappings **h–j)** of the PCF-5.

Figure 3

Effect of initial MEB concentration on the adsorption of MEB on PCF-5 **a)**; adsorption isotherms **b)**, the fitting plots of Langmuir model **c)**, and the fitting plots of Freundlich model **d)** for MEB, MG and RhB uptake by the PCF-5 (PCF-5 dose=10 mg; concentration of dye solution =200-2400 mg/L; volume of sample=20 mL).

Figure 4

Effect of contact time on the adsorption of MEB, MG, and RhB onto PCF-5 **a)**; UV-vis spectra showing the variation of the MEB concentration **b)**; The kinetics

models for adsorption of MEB, RhB, and MG onto PCF-5, respectively: pseudo-first-order models (c) and pseudo-second-order models (d). (PCF-5 dose=10 mg; concentration of dye solution =100 mg/L; volume of sample=20 mL).

Figure 5

Effects of pH (a) and NaCl concentration (b) on the removal efficiency of the PCF-5 toward MEB. (PCF-5 dose=10 mg; concentration of dye solution=100 mg/L; volume of sample=20 mL)

Figure 6

Effect of temperature on the removal rates of the MEB, MG and RhB onto PCF-5 (a) (PCF-5 dose=10 mg; concentration of dye solution =500 mg/L; volume of sample=20 mL); Van't Hoff plots of $\ln K_c$ vs $1/T$ for the adsorption of MEB, MG and RhB (b).

Figure 7

The photographs and the related UV-vis spectra of the static adsorption (a, b) and dynamic filtration (c, d) experiments of the PCF-5 for MEB and MO.

Figure 8

Possible adsorption mechanism of the MEB onto the PCFs.

Figure 9

the UV-vis spectra (a) and the related photographs (b) of the simulated dye effluent before and after adsorption by the PCF-5 (PCF-5 dose=10 mg; concentration of dye solution=100 mg/L; volume of sample=20 mL).

Figure 10

The removal efficiency of the regenerated PCF-5 toward MEB after 5 successive adsorption-desorption cycles. (PCF-5 dose=50 mg; concentration of dye solution=100 mg/L; volume of sample=50 mL).

Supplementary Files

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