

# Modification of activated carbon via grafting polyethyleneimine to remove amaranth from water

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**Abstract** A new adsorbent was fabricated via grafting polyethyleneimine (PEI) on activated carbon (AC) to remove anionic amaranth from water. X-ray photoelectron spectroscopy and Fourier transform infrared spectroscopy were used to characterize the grafting process of PEI. The adsorption behavior of the anionic amaranth (AM) on the modified AC is further evaluated by adjusting pH, the modified AC dosage, contact time and the initial AM concentration. AM adsorption was dramatically dependent of solution pH and the optimum pH is 1.08. The adsorption rate was very high within 20 min and equilibrium was achieved at 180 min. The maximum adsorption amount of AM on the modified AC is 72.68 mg/g at pH 1.08. Compared with the unmodified AC, the adsorption efficiency on the modified AC was dramatically increased from 29.9 to

97%. An electrostatic interaction mechanism was proposed in the acidic solution. The adsorption isotherms were consistent with the Langmuir model and the obtained kinetic data were in accordance with a pseudo-second order adsorption behavior for the modified AC.

**Keywords** Activated carbon · Modify · Adsorption · Dyes · Mechanism

## Introduction

Currently, amaranth (AM) is extensively used in food, textile, wood and leather industries. Large amount of wastewater containing AM is discharged into bodies of water (Gupta et al. 2012a; Gupta and Nayak 2012). The wastewater is harmful because AM causes serious health problems to humans and animals, such as cancer, anaphylaxis and respiratory problems (Gupta et al. 2012c, 2013). Thus, it is necessary to remove AM from wastewater.

Several methods have been used to remove AM from wastewaters, such as ion exchange, flocculation, degradation, adsorption and membrane filtration (Akil et al. 2015; Mohammadtaghi et al. 2014; Gupta et al. 2011b, 2012b, 2015; Saleh and Gupta 2011, 2012a, b, 2014; Mittal et al. 2010a; Saravanan et al. 2013a, b, c, d, e, f, 2015a, b, c, d, 2016). Among these methods, solid-phase adsorption is very convenient and effective. The solid-phase adsorbents include activated carbon (AC), alumina and polymer, etc. (Gupta et al. 2011a; Gupta and Saleh 2013; Khani et al. 2010; Karthikeyan et al. 2012; Saravanan et al. 2014a, b, c; Saleh and Gupta 2012c). AC is becoming increasingly popular in dealing with the dye-bearing wastewater due to the advantages of easy operation, cost-effectiveness and high efficiency (Mohd et al. 2010; Gupta et al. 1998; Devaraj et al. 2016; Rajendran et al. 2016; Mittal et al.

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2009a, 2010b; Jain et al. 2003). However, most of the researches focused on commercial activated carbons. The commercial AC still has some shortcomings, such as weak hydrophilic property, relatively low sorption capacity and low selectivity (Rafatullah et al. 2013; Gupta 2004; Ahmaruzzaman and Gupta 2011). Therefore, some researches modified the surface of AC by introducing polymer matrix to further improve the sorption capacity and selectivity. The reactive black 5 was removed by AC modified with cationic surfactant (Heibati et al. 2014). AC functionalized with cationic surfactant was one of the most powerful adsorbent for the dye removal. The cationic surfactant was directly attached on AC via cationic exchange (Mohammadi et al. 2011; Mittal et al. 2009b). Recently, more studies have focused on modifying AC via electrostatic adsorption. However, electrostatic interaction is easily destroyed by other factors, such as pH. So, it is necessary to overcome the disadvantages via surface grafting.

In this study, we modified AC with polyethyleneimine (PEI) via surface grafting to remove AM from water. The effect of pH, the modified AC dosage, the initial concentration and the contact time were evaluated. Adsorption kinetics and isotherms were also studied.

## Materials

Commercially granular AC was used as the starting material. Branched polyethyleneimine (PEI) and amaranth (AM) were obtained from Aladdin Chemical Reagent Co., Ltd.  $\text{HNO}_3$ , anhydrous tetrahydrofuran (THF) and thionyl chloride were purchased from Sinopharm Chemical Reagent Co., Ltd. Deionized water was utilized for preparing all solutions throughout the experiments.

## Modification of activated carbon

8 g of AC was first oxidized by 150 mL of concentrated  $\text{HNO}_3$  (69 wt%  $\text{HNO}_3$ ) at 85 °C for 30 h. After filtration, the solid was washed with deionized water five times and dried in a vacuum oven to give AC-COOH. In the second step, 5.6 g of the oxidized AC was treated with 80 mL concentrated thionyl chloride at 75 °C for 48 h. The mixture was separated by filtration. THF was used to wash the solid. Subsequently the solid was dried in a vacuum oven to give AC-COCl (3.5 g). Finally, 3.5 g of the chlorinated carbon was refluxed for 48 h in anhydrous THF solution containing branched polyethyleneimine (1.2 mg/mL). After filtration, the obtained solid was washed with deionized water and dried under vacuum, defined to AC-PEI (4.1 g).

## Adsorption experiments

Adsorption experiments were implemented on a shaker with a vibrating speed of 240 rpm, using 15 mL centrifugal

tube. The effects of the parameter including pH, the modified AC dosage, the contact time and the initial AM concentration on the removal were evaluated. After adsorbed, the samples were filtered and the filtrate was analyzed at 520 nm by a UV-2401 spectrophotometer (Japan). The adsorption amounts ( $Q$ ) and efficiency ( $R$ ) of AM on the modified AC were obtained from the following equations:

$$Q = \frac{V(C_b - C_a)}{m}, \quad (1)$$

$$R = \frac{(C_b - C_a)}{C_b} \times 100\%, \quad (2)$$

where  $m$  (g) represents the dosage of AC-PEI and  $V$  (L) is the volume of AM solution.  $C_b$  and  $C_a$  (mg/L) are the concentration of AM before and after adsorption, respectively. All experiments were repeated three times for data analysis.

## Characterization

X-ray photoelectron spectroscopy (XPS) was recorded using an ESCALab220I-XL (VG Scientific, UK). The binding energies were referenced to the C1s line at 284.6 eV from adventitious carbon. The Fourier transform infrared (FT-IR) spectroscopy was obtained between 400 and 4000  $\text{cm}^{-1}$  using a FT-IR spectrometer (Iprestige-21, Japan) with a resolution of 4  $\text{cm}^{-1}$ .

## Results and discussion

### Characterization of the modified activated carbon

The FTIR spectra of AC-COOH and AC-PEI are presented in Fig. 1. The spectra of AC-COOH showed a broad peak between 3200 and 3600  $\text{cm}^{-1}$ , coming from –OH groups. The peaks of C=O and C–O in AC-COOH appeared at

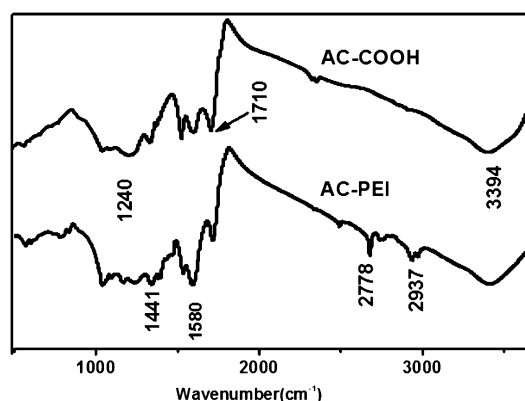
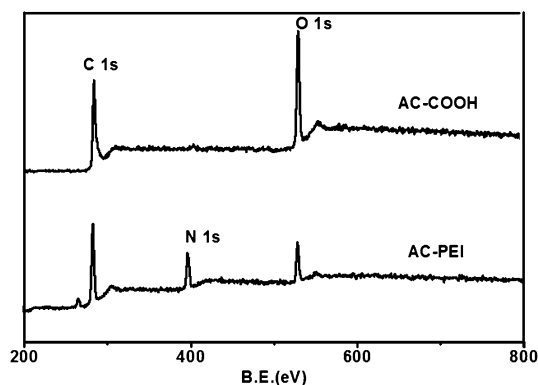


Fig. 1 FTIR spectra of AC-COOH and AC-PEI



**Fig. 2** XPS survey spectra of AC-COOH and AC-PEI

1710 and 1240  $\text{cm}^{-1}$ , respectively, which came from  $-\text{COO}-$  groups in carboxyl AC. The C–H bond vibrations at 2937 and 2778  $\text{cm}^{-1}$  appeared after modification with PEI. The successful modification of AC with PEI was also confirmed by the vibration absorptions at 1441 and 1580  $\text{cm}^{-1}$ , ascribed to the stretching vibration of C–N–H.

The surface modifications were further ascertained by XPS. The spectra of AC-COOH revealed carbon and oxygen peaks at 284.7 eV (C1s) and 530.9 eV (O1s), respectively (Fig. 2). After modification by PEI, the nitrogen peak at 398.2 eV was observed. The C1s spectra of AC-COOH and AC-PEI are shown in Fig. 3. The C1s spectra of the AC-COOH were curve-fitted to two peaks with bond energies at 284.9 and 289.4 eV for the C–C species and the O–C=O species, respectively. However, compared to AC-COOH, AC-PEI exhibited two new peaks at 288.1 and 285.8 eV corresponding to O=C–N and C–N species. These further demonstrated that PEI was grafted on the AC surface.

## Adsorption

### Effect of pH

pH is a key factor that influences adsorption process when AC-PEI was used as a sorbent. The experiments were carried

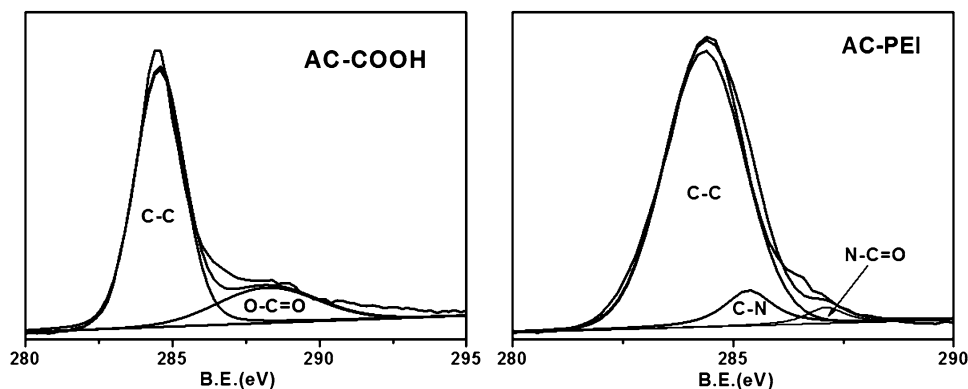
out in solutions containing 150 mg/L AM, 2 mg/mL AC-PEI with a contact time of 240 min. Figure 4a presented the influence of pH on the adsorption amount from 1.08 to 12.7 at 298 K. The adsorption amount decreased with increasing pH values. The maximum adsorption amount is 72.68 mg/g when the initial pH of AM solution is 1.08. When pH increases, the adsorption amount decreases and then presents an increasing trend in the pH value range of 11.2–12.7. This suggests that AC-PEI was high-efficiency adsorbent for AM under acidic conditions. The reason could be explained as the changes of surface charge of the adsorbent. At low pH value, the surface of AC-PEI is positively charged due to the protonation of the amine and imino groups ( $-\text{NH}_2$  and  $-\text{NH}-$ ), while the anionic AM are still negatively charged. The electrostatic attraction between the protonated adsorbent and the anionic AM enhances the adsorption capacity (Royer et al. 2009). Therefore, the mechanisms of adsorption can be considered as the electrostatic interactions between the AC-PEI surface and the AM anions. On the other hand, the adsorption efficiency of AM increased with increasing pH from 11 to 12. This was because the surface of AC-PEI was covered with  $\text{OH}-$  groups in strong basic condition. The mechanism is anion exchange in this situation. Hence, the pH of aqueous solution significantly influenced the adsorption behavior of AC-PEI for AM.

We also compared the adsorption capacity of AM on AC and AC-PEI (Fig. 4b) at pH = 1.08. After adsorbed by AC and AC-PEI, the residual concentration of AM is 117.1 and 5.5 ppm, respectively. The adsorption efficiency was dramatically increased from 29.9 to 97%. The result showed that attachment of polyethyleneimine onto activated carbon enhanced markedly the removal ratio of AM from water.

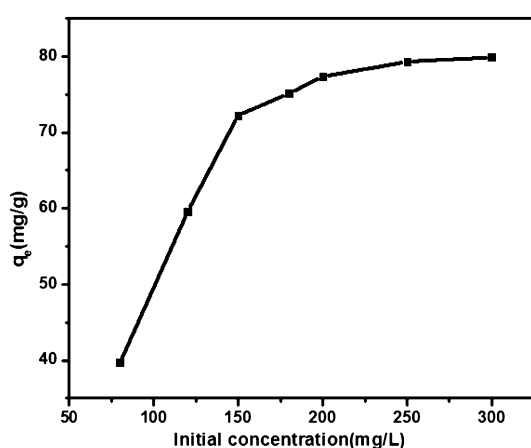
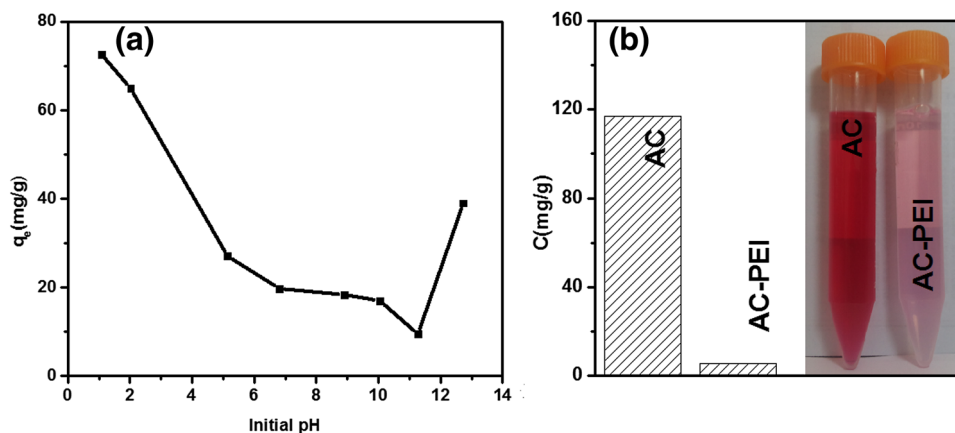
### Effect of initial AM concentration

Effect of the initial AM concentration on the adsorption performance was studied by adding AC-PEI (2 mg/mL) to different concentrations of AM solution (80, 120, 150, 180, 200, 250 and 300 mg/L) at pH 1.08. The results showed that the adsorbed amount of AM first increases with the

**Fig. 3** Peak-fitted C1s signals of AC-COOH and AC-PEI



**Fig. 4** Effects of the initial pH on the adsorption amount of AM on AC-PEI (a) and the adsorption capacity of AM on AC and AC-PEI (b)



**Fig. 5** The effect of initial AM concentrations on the adsorption

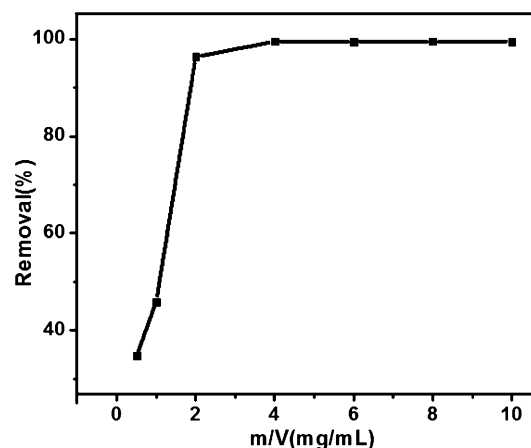
increasing initial AM concentration and then tends to level off (Fig. 5). This is attributed to the higher mass transfer driving force at high initial AM concentration, and finally the adsorption reached saturation (Özacar 2003).

#### Effect of AC-PEI dosage

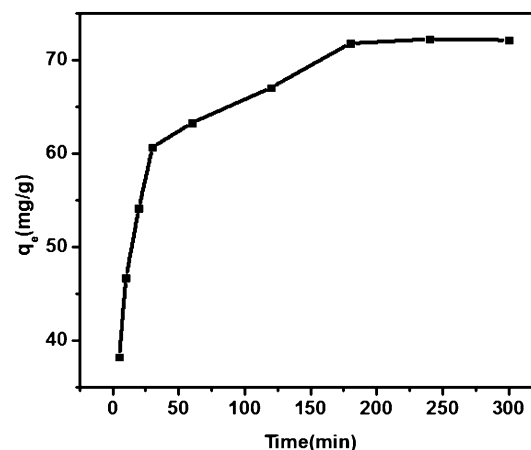
The effect of AC-PEI dosage on the adsorption was studied at initial AM concentration (150 mg/L) and pH (1.08). Figure 6 illustrated the effect of the AC-PEI dosage (0.5, 1, 2, 3, 4, 5, 6, 7, 8, 9 and 10 mg/mL) for AM adsorption. The removal percent of AM enhances from 34.72 to 99.49% with the increase of AC-PEI dosage from 0.5 to 4 mg/mL and then remains invariant at higher dosage. The increase of the removal efficiencies could be due to that the quantity of active adsorption points increased with increasing the dose of adsorbent.

#### Effects of contact time

In order to investigate the effect of contact time, 2 mg/mL AC-PEI was added to the AM solutions (150 mg/L) in



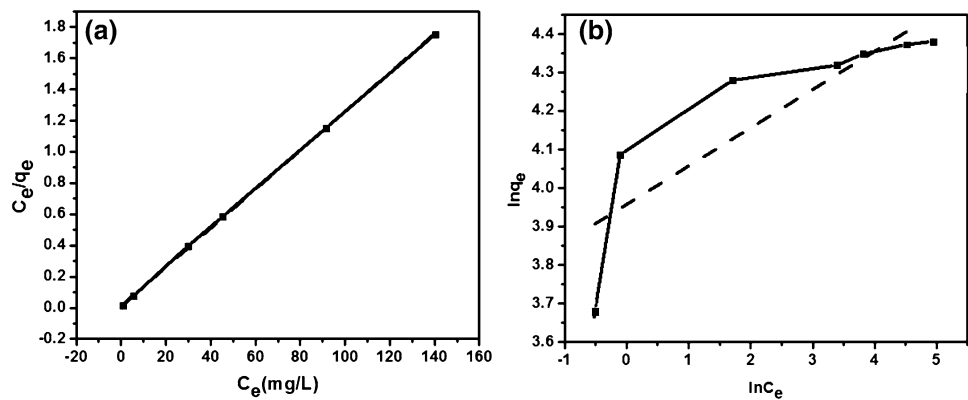
**Fig. 6** Effect of AC-PEI dosage on the AM adsorption



**Fig. 7** Effects of contact time on the adsorption amount of AM on AC-PEI

pH = 1.08 (Fig. 7). Figure 7 shows a rapid uptake of AM within the first 50 min, indicating a high affinity between the AC-PEI surfaces and AM molecules. Then, the adsorption rate decreased until reaching the equilibrium (180 min), due to the intraparticle diffusion of AM

**Fig. 8** **a** Langmuir and **b** Freundlich isotherms for the adsorption of AM onto AC-PEI



molecules. After 4 h, the AM adsorption capacity with initial AM concentrations of 150 mg/L is 72.68 mg/g. Rapid increasing of the capacity at initial contact time can be attributed to that many active adsorption sites are available; then the capacity remains invariant, which was possibly due to the mutually exclusive of the AM molecules on AC-PEI. Yao and coauthors had also observed that the removal efficiency of dye by multi-walled carbon nanotubes increased with increasing contact time and finally reached equilibrium (Yao et al. 2010).

**Adsorption isotherms**

Adsorption isotherms express the relationship between the adsorbed amount on the adsorbent and the residue concentration in solution. The isotherm data of AM on AC-PEI at pH 1.08 and 298 K were fitted by Langmuir and Freundlich models. The Langmuir model assumed that the monolayer adsorption occurs on a homogeneous surface (Yao et al. 2015). The Langmuir isotherm equation was followed:

$$\frac{C_e}{q_e} = \frac{1}{q_m} (K_e + C_e), \tag{3}$$

where  $C_e$  (mg/L),  $q_e$  (mg/g) and  $K_e$  (L/mg) are the equilibrium concentration of AM, the equilibrium adsorption capacity and the equilibrium constant, respectively.  $q_m$  (mg/g) is the maximum adsorption amount.

The Freundlich isotherm supposed that a multilayer adsorption occurs on a heterogeneous substrate. The Freundlich isotherm equation was followed:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e, \tag{4}$$

where  $K_F$  and  $n$  are Freundlich constants. Figure 8 showed the Langmuir and Freundlich isotherms.  $K_L$ ,  $q_0$ ,  $K_F$  and  $n$  were calculated from two isotherms and the results of this analysis were listed in Table 1. The square of the correlation coefficient ( $R^2$ ) are also tabulated in Table 1. The  $R^2$  (0.9998) of Langmuir model is bigger than that of

**Table 1** Isotherm parameters for AM adsorption on AC-PEI at 298 K

Isotherms	Parameters	$R^2$
Langmuir	$q_m = 80.65$ mg/g $k_L = 1.04$ L/mg	0.9998
Freundlich	$k_F = 50.57$ L/mg $n = 10.07$	0.6885

**Table 2** Comparison of adsorption capacity of AM with sorbents reported in literature

Sorbent	$q_m$ (mg/g)	References
Pomegranate peel	3.45	Ali et al. (2013)
Puffed rice	21.97	Chowdhury and Saha (2016)
<i>E. crassipes</i> roots	28.51	Guerrero-Coronilla et al. (2014)
<i>E. crassipes</i> leaves	43.10	Guerrero-Coronilla et al. (2014)
AC-PEI	72.68	This work

Freundlich model (0.6885), suggesting that Langmuir model is reliable. Moreover, a dimensionless factor, called separation factor ( $R_L$ ), associated with Langmuir isotherm is investigated to study the viability of adsorption on AC-PEI, which are expressed:

$$R_L = \frac{1}{(1 + K_L C_0)}, \tag{5}$$

where  $C_0$  (mg/L) is the initial AM concentration and  $K_L$  (L/mg) is Langmuir constant. The value of  $R_L$  indicates the type of the isotherm: irreversible ( $R_L = 0$ ), favorable ( $0 < R_L < 1$ ), linear ( $R_L = 1$ ), unfavorable ( $R_L > 1$ ). The  $R_L$  of AM adsorption onto AC-PEI is in between 0.0032 and 0.0119, showing a favorable adsorption of AM. According to Fig. 8, the maximum adsorption capacity of AM is 80.65 mg/g. The adsorption capacity of AM and other results reported in literature were listed in Table 2, which showed that the AC-PEI are an efficient sorbent. Thus the AC-PEI sorbent will show great potential in the field of adsorption of AM.

**Table 3** Kinetic models for AM adsorption on AC-PEI at 298 K

Models	Model parameters	$R^2$
Pseudo-first order	$k_1 = 0.0235 \text{ min}^{-1}$	0.9561
Pseudo-second order	$k_2 = 0.0014 \text{ mg}^{-1}/\text{min}$	0.9994
Intraparticle diffusion	$k_{i1} = 7.018 \text{ mg/g min}^{1/2}$	0.9626
	$c_1 = 23.30 \text{ mg/g}$	
	$k_{i2} = 1.1669 \text{ mg/g min}^{1/2}$	
	$c_2 = 54.28 \text{ mg/g}$	
	$k_{i3} = 0.0872 \text{ mg/g min}^{1/2}$	
	$c_3 = 70.73 \text{ mg/g}$	0.1572

### Adsorption kinetics

For further exploring the adsorption mechanisms, adsorption kinetic was evaluated at 298 K and pH 1.08. The Lagergren pseudo-first-order and pseudo-second-order kinetic models were used to study the experimental data. These two kinetic equations can be expressed as follows:

$$\ln(q_e - q_t) = \ln(q_e) - k_1 t \quad (6)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (7)$$

where  $q_e$  and  $q_t$  (mg/g) represent the adsorption amount at equilibrium and any time (h), respectively.  $k_1$  ( $\text{h}^{-1}$ ) is the pseudo-first-order model rate constant and  $k_2$  ( $\text{g}/\text{mg}/\text{h}$ ) is the rate constant of the pseudo-second-order model.

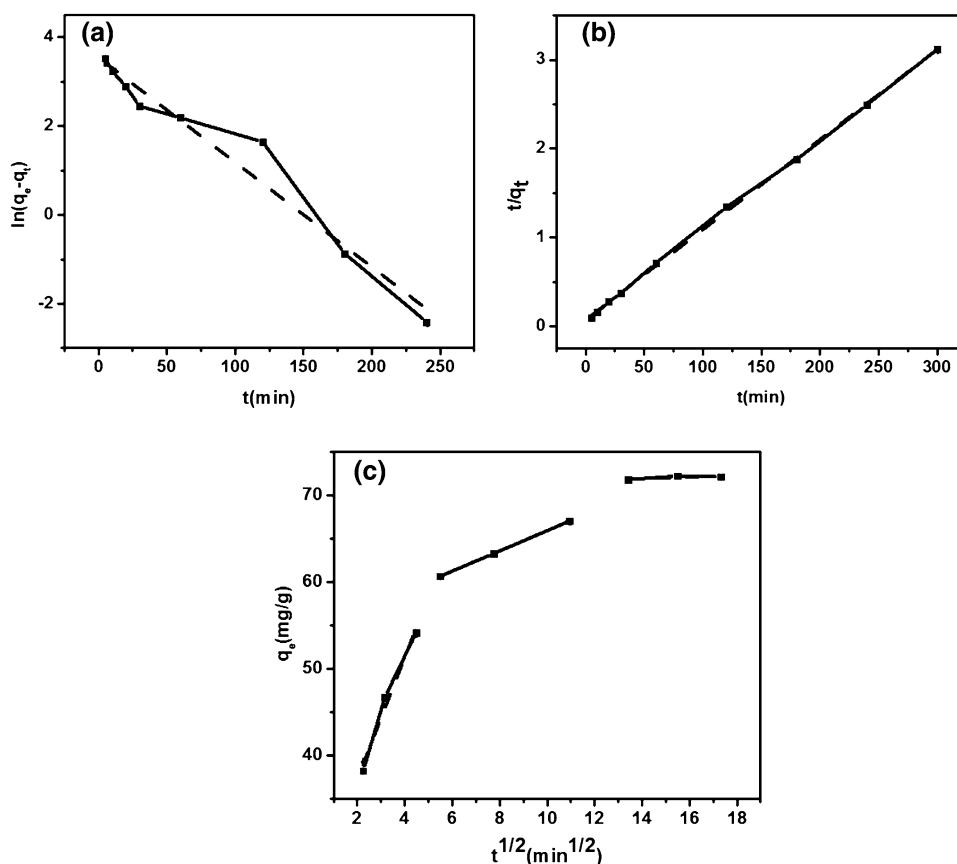
The calculated kinetic parameters and the correlation coefficients ( $R^2$ ) were presented in Table 3. The  $R^2$  (0.9994) of the pseudo-second-order model is bigger than that of the pseudo-first-order model. Therefore, the adsorption of AM onto AC-PEI follows the pseudo-second-order model and the chemisorption is the rate limiting step.

The Weber–Morris intraparticle diffusion kinetic model was also used to study the steps involved during adsorption, which is expressed as

$$q_t = k_i t^{1/2} + c, \quad (8)$$

where  $k_i$  ( $\text{mg}/\text{g}^{-1} \text{ h}^{-1/2}$ ) is the intraparticle diffusion rate constant, and  $c$  (mg/g) is a constant that gives idea about the thickness of the boundary layer. i.e., larger the value of  $C$  the greater is the boundary layer effect. Figure 9c presents the multi-linear plots. It suggests that the sorption process includes three steps. The first straight portion represents the stage of external mass transfer, and at the second step AM entered the pores of the adsorbents and were adsorbed on the internal surface of the AC-PEI and

**Fig. 9** **a** Pseudo-first-order model, **b** pseudo-second-order model, and **c** intraparticle diffusion model for the adsorption of AM onto AC-PEI





the last is attributed to the intraparticle diffusion (Liu et al. 2015).

## Conclusion

In this work, we fabricated a new adsorbent via grafting polyethyleneimine on activated carbon (AC) to enhance the selective removal of AM from water. The modified activated carbon was characterized by FTIR and XPS. The effect of parameters including pH, initial AM concentration, AC-PEI dosage and contact time were also investigated. The optimum adsorption condition is pH = 1.08 and the contact time is 180 min. The adsorption capacity of AM onto AC-PEI at 298 K is 72.68 mg/g. This is higher than the adsorption capacity of AM onto the unmodified AC. The electrostatic interaction between AC-PEI and AM is the driving motivation in the acidic solution. AM adsorption on AC-PEI was accordance with the pseudo-second-order kinetics model and the equilibrium data are well consistent with the Langmuir models. The adsorbents containing amino-group will be beneficial to the removal of anionic organic pollutants from polluted water.

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