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Adsorption behaviors of lead ion onto acetate modified activated carbon fiber

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ABSTRACT

Activated carbon fiber (ACF) was modified with acetates of sodium, potassium and lithium at concentration of 15% and tested to adsorb lead from aqueous solution. The effect of acetate treatments of ACF was studied in adsorption behavior of lead ion. Acetate treatment reduced surface area and pore volume of ACF, but the adsorption amount of lead ion on the modified ACF (MACF) was greater than that on the pristine ACF. The adsorbed lead could be fully desorbed by using 0.01 mol/L HNO_3 solution. The maximum adsorption capacity of lead ion on acetate lithium modified ACF was 165.1 mg/g and the adsorption isotherm follows Langmuir isotherm model better than the Freundlich isotherm model. The adsorption kinetic data could be described well by the Lagergren pseudo-second-order kinetic equation.

Keywords: Activated carbon fiber; Surface treatment; lead; Adsorption; Desorption

1. Introduction

Water pollution due to toxic heavy metals is a serious environmental and public health issue. The heavy metals such as lead, cadmium, copper and zinc, are among the most common pollutants found in industrial effluents. These metals are of special concern due to their toxicity and persistency properties in nature. Lead pollution often influences agricultural areas, lakes, rivers, and so on. Lead toxicity is well known to cause blood dyscrasia and kidney and nervous troubles [1]. Hence, it is necessary to remove lead from wastewater before being discharged. There are number of treatment methods for the removal of heavy metals from water and wastewater. Chemical precipitation, ion exchange, ultrafiltration, membrane techniques and adsorption are the methods used for the treatment of water and wastewater containing heavy metals. But the addition of chemicals often incurs high operational costs and may not meet strict regulatory requirements [2]. Although reverse osmosis and ion exchange methods are effective in removing such pollutants, they are expensive in the operational procedure. These factors have limited the use of methods for the removal of lead and other heavy metals from water and wastewater especially in most of developing countries [3,4].

Adsorption is considered as one of the best available technologies for removal of heavy metals from water. Various adsorbents such as silica gel, alumina clay, synthetics polymer resins and carbonaceous materials are used in adsorption method [5]. The activated carbon, either granular activated carbon (GAC), or powdered activated carbon (PAC) is the major applying for removal heavy metals adsorption [6–11]. Activated carbon fiber (ACF) is a novel and fibrous carbonaceous adsorbent. As compared with conventional granular or PACs, ACF has been widely

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used as an excellent adsorbent because of its large surface area, microporous character, and high adsorption/desorption rate [12–15]. The adsorption kinetics and capacities for ACF are 10–100 times higher than those conventional adsorbents [16,17]. It is thought that ACFs faster adsorption rate is due to its higher surface area compared to activated carbons, which arises from ACFs uniform microporous structure and graphite-like molecular characteristics [18,19]. ACF has additional advantages in terms of its hydrophobicity, resistance to both alkaline and acidic media, and structural stability under inert atmospheres at temperatures up to several hundred degrees. Nevertheless, ACF displays low adsorption selectivity toward polar and polarizable organic molecules [20]. Adsorption capacity and selectivity for specific environmental pollutants can be tailored by modifying the pore structure and surface chemical properties of adsorbents [6,21]. So, modifications of ACF through chemical functionalization can enhance it affinity for certain contaminants [22,23].

The main goal of this study lies on the modification of ACF through chemical functionalization to improve its performance as adsorbents of toxic metals in water, specifically for lead ion (Pb^{2+}) and compare the adsorption performance of three carboxylates, namely acetates of sodium, potassium and lithium modified ACFs, with the unmodified ACF. In addition, the lead adsorption equilibrium and kinetics were determined at various concentration conditions.

2. Experimental

2.1. Materials

All the chemicals used in the study are of analytical grade. All the solutions in the study were prepared using deionized water. All glassware was cleaned by rinsing with hydroxylamine hydrochloride, soaking in 10% HCl, and rinsing with deionized water.

2.2. Preparation of surface modified ACF

Viscose rayon-based carbon fiber in felt form used in this study was supplied by Zichuan Carbon Fiber Co. Ltd. (China). It was repeatedly treated with boiling deionized water until the pH of the solution remained unchanged, then dried overnight at 80°C and cooled in a desiccator.

ACF (10 g) was treated with 200 mL aqueous solution of 15% of acetates of sodium, potassium and lithium for 72 h. The impregnation was carried out at 70°C till the complete evaporation of water and then dried in oven at 100°C for 24 h to produce modified activated carbon fiber such as NaACF, KACF, LiACF, respectively. The dried material was washed with deionized water till the washing liquid became free from metal ion and then dried at 100°C.

2.3. Characterization

Textural properties of the pristine and modified ACFs were determined by nitrogen adsorption of previously dried samples at 100°C for 24 h. The specific surface area and the pore structure were evaluated from nitrogen adsorption data at 77 K (Micromeritics, ASAP 2000).

2.4. Adsorption/desorption of lead ion

The adsorption capacity of various modified ACF was determined by batch adsorption isotherms at 20°C in aqueous solution. Lead ion was hardly adsorbed onto any of the ACF samples in solutions having values of pH less than 3, at values of pH higher than 7, the adsorption experiments failed as a result of precipitation of lead hydroxide. Thus, in adsorption experiments we fixed the pH values of the initial solution at 4, 5 and 6. In several glass vials, 100 mL of solution containing various lead ion concentrations (200, 400, 600, 800, 1,000 mg/L) were contacted with 0.05 g of modified ACF (previously powdered to a particulate size of about 350 μm). The vials were placed in a water bath at 20°C for approximately 24 h, and the pH was maintained constant (± 0.10) adjusted either with dilute 0.01 mol L^{-1} HNO_3 or NaOH throughout the adsorption tests. Then, each solution was filtered with a polycarbonate membrane of 0.45 μm of pore diameter, and the concentration at equilibrium was determined. Initial and equilibrium concentrations were measured by an atomic fluorescence spectrometer (AFS) (PS Analytical Ltd., Kent, UK). The amount of lead ion adsorbed was calculated from the difference between the quantity of metal ion added to the modified ACF and the metal ion content of the supernatant with the following equation:

$$q_e = V(C_0 - C_e)/W, \tag{1}$$

where q_e is the amount adsorbed (mg/g); C_0 and C_e are the initial and equilibrium lead concentrations in the solution (mg/L), respectively; V is the solution volume (L); and W is the mass of ACF (g). The adsorption capacity of modified ACF was compared with pristine ACF.

After the adsorption equilibrium was reached, the desorption procedure was carried out. Half of the initial volume of solution in contact with the metal loaded modified ACF was substituted with an equal volume of acidic solution at pH 2 ± 0.1 (prepared by dilution of nitric acid in deionized water) to attain a new equilibrium for 24 h. Then, the solutions were

Table 1
Microstructure of pristine ACF and modified ACFs

Sample	BET surface area (m^2 g^{-1})	Micropore volume (cm^3 g^{-1})	Total pore volume (cm^3 g^{-1})	Average pore diameter (nm)
Pristine ACF	976.3 ± 1.53	0.3835	0.464	2.316
LiACF	856.3 ± 1.38	0.3246	0.407	2.363
NaACF	803.6 ± 0.83	0.3189	0.398	2.453
KACF	795.9 ± 2.01	0.3135	0.391	2.513

filtered through polycarbonate membranes of 0.45 μm, and the concentration of metal was measured by AFS. The mass of metal desorbed by modified ACF was computed by a mass balance.

2.5. Kinetic study

The adsorption kinetic study was performed for lead in solution at initial pH 6.0 and room temperature (20 ± 1°C). Several glass vials were used to hold 100 mL lead solution of known initial concentration and 0.05 g of adsorbent at initial pH 6.0, and shaken at 180 r/min for a duration ranging from 0 to 480 min. At certain period of time, each vial was removed from the shaker, and the solution was filtered through a polycarbonate membrane of 0.45 μm pore diameter to measure the lead concentration.

2.6. Effect of solution pH

To determine the influence of pH on lead ion adsorption, experiments were performed at various initial pH. Lead ion was hardly adsorbed onto any of the ACF samples in solutions having values of pH less than 3. At values of pH higher than 7, the adsorption experiments failed as a result of precipitation of lead hydroxide. Thus the solution initial pH values in this study ranged between 3.5 and 6.5. Initial concentration of 1,000 mg/L of lead ion and 0.50 g of modified ACF per 1,000 mL of solution were used. The suspensions were stirred for 240 min.

All the experiments in this study were triplicate. The results presented were the mean values with a total error of less than 5%.

3. Results and discussion

3.1. Characterization of adsorbents

Information about pore volume and BET surface area of adsorbents were summarized in Table 1. As shown in Table 1, the surface area and pore volume of ACF were reduced after treatment with acetates. On the other hand, the average pore diameter was slightly increased. This may be due to several factors such as the increase of oxygen-containing functional groups which are attributed to the block of the micropores and large molecules of residual humic-type compounds, electrostatic repulsion of surface probe molecules and erosion of carbon by acetates [24]. It was noted that the reduction of surface area of LiACF was smaller than the others, while the total pore volumes of all three modified ACFs were almost same, this may be ascribed to that Li^+ ion is smaller than Na^+ and K^+ ions. In this study, the adsorption capacity of the surface modified ACF was compared to that of the pristine ACF.

3.2. Adsorption amounts of lead ion on various acetate-modified ACFs

The experimental results depicted in Fig. 1 indicated that lead ion adsorption was in the order: LiACF (163.5 mg/g)>KACF (153.8 mg/g)>NaACF(143.3 mg/g) >ACF (130.1 mg/g). The impregnated ACF had greater adsorption capacity than the unmodified ACF. This could be explained due to a higher availability of oxygen-containing groups on impregnated ACF. It is well known that the Pb^{2+} ions in aqueous solutions adsorb more on the ACF acid sites and the major acid sites are the carboxylic, phenolic and lactonic [25]. The addition of carboxylic functional groups at the surface of the impregnated ACF led to an increase in metal ion removal because of the high affinity of the carboxylic functional group for metal ions [26]. In addition, the surface area is one of the key factors to control the adsorption efficiency of a adsorbent. The larger the surface area is, the higher the adsorption efficiency is. It was noted from Table 1 that the surface area of LiACF was larger than the other modified ACFs, while the total pore volumes of all three modified ACFs were almost same, so the adsorption efficiency of LiACF was higher than those of other types. Due to the higher adsorption efficiency of LiACF, this was used for all the other experimental studies.

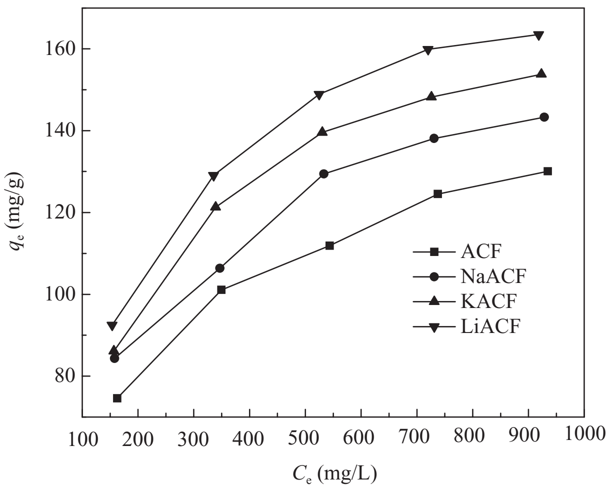

Fig. 1. Adsorption of lead ion on various acetate modified activated carbon fibers. Experimental conditions employed: pH = 6.0, agitation speed = 180 r/min, adsorbent dosage = 500 mg/L.

3.3. Effect of initial pH on adsorption of lead ion

The pH of the aqueous solution is an important controlling parameter in the adsorption process. The experimental results obtained from lead ion adsorption onto LiACF under different initial pH conditions (maintained constant throughout the adsorption tests) were shown in Fig. 2. LiACF dosage (500 mg/L), lead ion (1,000 mg/L) and agitation speed (180 r/min) were kept constant in all experiments. The lead ion adsorbed by LiACF was sensitive to initial pH variation over the examined range of 3.5–6.5. The adsorption amount increased with increasing initial pH to reach a maximum at pH 6.0. At initial pH <6.0, H^+ ions competed with lead ions for the surface of the adsorbent, which would restrict the approach of lead ions due to the repulsion [27]. Hence, the metal removal was the lower amount presumably due to the enhanced competition of proton with lead ions for ligand binding sites and complex formation. On the other hand, the adsorptive behavior of lead ion resulted pH dependent because the pH values also affected the charge on the adsorbent surface. At pH < 6.0, lead ions can be repelled by the surface positive charges on the adsorbent due to the protonation of oxygen groups ($R{-}OH_2^+$). The adsorption of lead ions increased as the initial pH of the system increased, lead ions are attached on the surface of ACF by replacing H^+ ions of the carboxylic and phenolic ions until a maximum yield is reached. The condition of initial pH > 6.0, noted in adsorption decrease, may be attributed to precipitation of the lead ions as hydroxides [20]. For this reason, the optimal pH value was selected to be 6.0.

3.4. Adsorption isotherms

In the sorption technology, it is utmost essential to determine an adsorption, as it shows how the adsorption molecules are distributed in the liquid phase and with the adsorbent. For the design purpose, it is necessary to fit the isotherm data with the models. Langmuir and Freundlich isotherm equations are most widely used for the equilibrium study. Adsorption isotherms were determined at different pH values to evaluate the capacity of modified ACF composite adsorbent to adsorb lead ions (Fig. 3). These isotherms represented the adsorption behavior of lead ion on the adsorbent as a function of increasing aqueous lead concentration

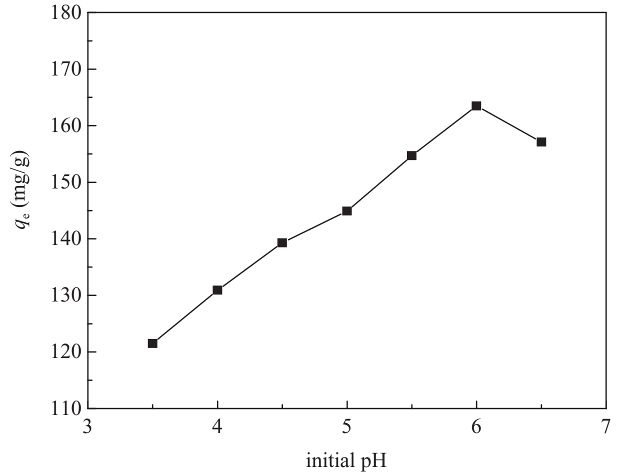

Fig. 2. Effect of initial pH on lead ion (1,000 mg/L) adsorption by LiACF adsorbent. Experimental conditions employed: agitation speed = 180 r/min, adsorbent dosage = 500 mg/L.

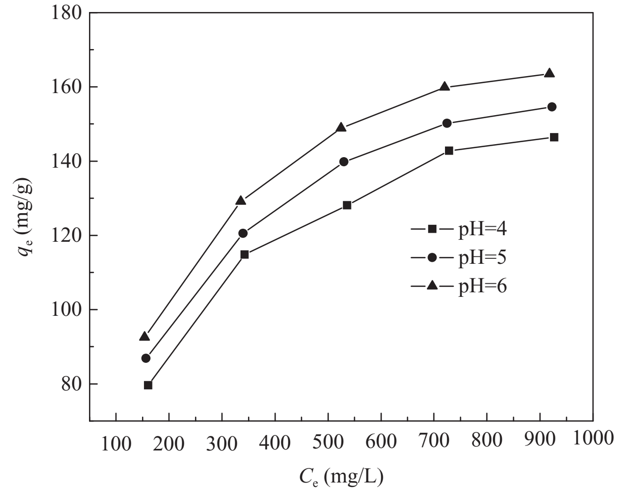

Fig. 3. Adsorption isotherm for lead ion by LiACF adsorbent. Experimental conditions employed: agitation speed = 180 r/min, adsorbent dosage = 500 mg/L.

Table 2
The parameters of Langmuir and Freudlich equation

	Langmuir equation			Freundlich equation		
Initial pH	q_m (mg g^{-1})	b (L mg^{-1})	R^2	$1/n$	K_f	R^2
4.0	178.57	5.13×10^{-3}	0.9993	0.3517	13.8830	0.9843
5.0	182.15	5.49×10^{-3}	0.9993	0.3322	16.7625	0.9864
6.0	194.93	6.61×10^{-3}	0.9997	0.3265	18.5357	0.9834

for a contact time of 24 h at initial pH = 4.0, 5.0 and 6.0. The increasing adsorption capacity observed for lead ion when the pH went up was related to the higher concentration of available carboxylic groups.

The results of lead ion adsorption on adsorbent (Fig. 3) were analyzed by using the Langmuir model to evaluate parameters associated to the adsorption behavior. The linear form of Langmuir equation at a given temperature is represented by:

$$q_e = q_m b c_e / (1 + b c_e), \tag{2}$$

where c_e is the aqueous phase ion equilibrium concentration (mg/L), q_e is the amount of lead ion sorbet onto 1 g of the considered adsorbent (mg/g), b is the adsorption constant (L/mg) related to the energy of adsorption and represents the affinity between the adsorbent and adsorbate, q_m is the maximum adsorption capacity (mg/g).

Experimental data of the adsorption isotherms of lead ions acquired at different initial pH were correlated with Langmuir model. The isotherm parameters related to the model were listed in Table 2. It could be seen that both q_m and b increased with increasing initial pH from 4.0 to 6.0. The maxima adsorption capacities (q_m) were 178.53, 182.15 and 194.93 mg/g at pH values 4, 5 and 6, respectively. High values of b were reflected in the steep initial slope of an adsorption isotherm, indicating desirable high affinity. Therefore, LiACF performed well in lead adsorption at initial pH 6.0 compared to other initial pH values examined.

The Freundlich isotherm model was also used to analyze the results of lead ion adsorption on LiACF adsorbent (Fig. 3). The Freundlich model can be expressed by the following equation:

$$q_e = k_f c_e^{1/n}, \tag{3}$$

where q_e is the amount adsorbed at equilibrium (mg/g), k_f is the Freundlich constant related to the adsorption capacity (L/g), c_e is the equilibrium concentration (mg/L) and n is the Freundlich exponent.

Experimental isotherm data acquired at different initial pH were fit with the Freundlich model and the isotherm parameters related to the model obtained from the experimental data are listed in Table 2. The data showed that the k_f constant was increased with the increase of initial pH values, at initial pH 6.0, k_f reached its corresponding maximum value, and $1/n$ value at initial pH 6.0 was smaller than that at other initial pH values. These implied that the binding capacity reached the highest value and the affinity between the adsorbent and lead ions was also higher than other initial pH values. The correlation coefficients (R^2) given in Table 2 also showed that the Langmuir equation gave a better fit than Freundlich equation to the adsorption isotherms.

3.5. Kinetic study

In order to obtain the adsorption kinetic information of lead ion on the modified ACF adsorbent, the change of lead ion concentration with adsorption time was recorded for an initial concentration of 200, 400, 600, 800, 1,000 mg/L and a fixed pH solution of 6.0, adsorbent dosage 0.50 g/L (Fig. 4). It could be seen that the adsorption process of lead ions onto ACF was slow, this might be due to that the solute has to diffuse through the macro, meso and micropores of ACF to

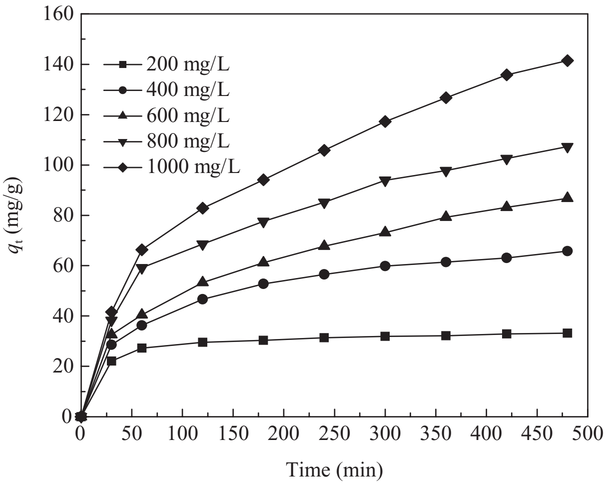

Fig. 4. Adsorption kinetics of lead ion by LiACF adsorbent. Experimental conditions employed: pH = 6.0, agitation speed = 180 r/min, adsorbent dosage = 500 mg/L.

Table 3
Kinetic parameters for lead ion adsorption by LiACF adsorbent

C_0 (mg L^{-1})	q_e (mg g^{-1})	k_2 (L mg^{-1} min^{-1})	R^2
200	34.20	15.46×10^{-4}	0.9998
400	72.36	2.291×10^{-4}	0.9987
600	100.21	1.053×10^{-4}	0.9931
800	121.36	1.004×10^{-4}	0.9952
1000	168.92	0.511×10^{-4}	0.9911

reach the active sites. It was also evident from Fig. 4 that the adsorption of lead ion increased rapidly with time as well as with the increase of the initial ion concentrations. However, the time to reach the adsorption equilibrium took longer with an increase in the concentration. The adsorption of lead ions reached to equilibrium state after 50 min reaction at initial lead ion concentration of 200 mg/L. For the initial lead ion concentration of 1,000 mg/L, the adsorption time reached to equilibrium state was more than 480 min.

To investigate the mechanism of adsorption, the Lagergren rate equation model was applied to experimental data. The pseudo-second-order kinetic equation could be derived as:

$$dq_t/dt = k_2(q_e - q_t)^2. \tag{6}$$

Separating the variables in Eq. (6) gave:

$$-d(q_e - q_t)/(q_e - q_t)^2 = k_2 dt. \tag{7}$$

Integrating both sides for the boundary conditions $t = 0$ to $t = t$ and $q_t = 0$ to $q_t = q_t$ gave the integrated rate law for a pseudo-second-order reaction,

$$1/(q_e - q_t) = 1/q_e + k_2 t. \tag{8}$$

Eq. (8) could be rearranged to obtain:

$$t/q_t = 1/(k_2 q_e^2) + t/q_e. \tag{9}$$

The kinetic constant, k_2, could be determined by plotting of t/q_t against t.

The kinetic experimental data of lead ion on the LiACF composite adsorbent was simulated by pseudo-second-order rate Eq. (9). The results are listed in Table 3.

Remarkably, the kinetic data could be described well by the pseudo-second-order rate equation with a minimum linear correlation coefficient of 0.9911 (see Table 3). It could be seen that the values of the pseudo-second-order rate constant decreased with increasing the initial lead ion concentrations.

4. Conclusions

The modification of ACF by acetates of sodium, potassium and lithium significantly improved its adsorption capacity due to its concentration of carboxylic groups, although its structure was damaged to some extent when it was modified and made this material a suitable adsorbent to remove toxic of heavy metals such as lead in aqueous solution. The adsorption capacity of modified ACF for lead ion is about 1.3 times higher than that of unmodified ACF. The overall adsorption rate was illustrated by the pseudo-second-order kinetic models. The equilibrium data obtained from this study was well presented by Langmuir model. The results demonstrated that lithium acetate modified ACF could be used as an effective adsorbent for the adsorption of lead ions from aqueous solution.

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