



Comparison of As(III) Adsorption by Nanomagnetic Fe₃O₄, Activated Carbon and Modified Activated Carbon

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Nat. Env. & Poll. Tech.
Website: www.neptjournal.com

Received: 28-02-2022
Revised: 30-03-2022
Accepted: 06-04-2022

Key Words:

Nanomagnetic Fe₃O₄
Modified adsorbent
Activated carbon
Adsorption
Arsenic pollution

ABSTRACT

As a kind of new material, nanomagnetic Fe₃O₄ (NMF) has many advantages in water and wastewater treatment. In this paper, the adsorption characteristic for arsenic(III) (As) by NMF was studied, and the adsorption was compared with the traditional adsorbent of activated carbon (AC) and modified activated carbon (MAC). The results showed that the NMF had high adsorption performance for As, and the adsorption performance of modified activated carbon and activated carbon were far lower than that of magnetic nanomagnetic Fe₃O₄. The adsorption capacity for As with NMF, MAC, and AC was 0.189 mg.g⁻¹, 0.023 mg.g⁻¹ and 0.013 mg.g⁻¹ in 0.25 mg.L⁻¹ As solution, respectively. The adsorption rate for As was different from the three adsorbents. For NMF, it needs only 10 minutes to reach an adsorption balance, while the time to balance was 20 minutes for MAC and more than 120 minutes for AC. The adsorption for As by the three adsorbents all conform to Langmuir adsorption isotherm, and the adsorption kinetics for As by the three adsorbents were in accordance with the pseudo-second-order kinetics model. The adsorption efficiency of MAC and AC was lower than nanomagnetic Fe₃O₄. The nanomagnetic Fe₃O₄ was an effective adsorbent for arsenic.

INTRODUCTION

As one of the most abundant elements, arsenic (As) is widely distributed in nature. By estimate, the annual total global arsenic emissions to the environment are as high as 22,100 tons, and the total amount of arsenic flow into the water environment is about 110 thousand tons (Varga et al. 2017, Zheng et al. 2017). The arsenic in water has mainly come from one of the emissions of agricultural and industrial wastewater, which contains excessive pesticides, herbicides, and wood preservatives, and the second was the deposition of arsenic in the atmosphere. The combustion of arsenic-bearing coal and crude oil can make a lot of arsenic-containing compound particles enter the atmospheric environment in form of dust and then enter surface water by means of sedimentation, and the dissolution of natural minerals (Jerome et al. 2015). With the development of industry and agriculture, the total annual discharge of industrial wastewater increased over decades, and the amount of arsenic entering the environment increased (Hu et al. 2019, Sun et al. 2016).

Arsenic and its compounds are carcinogenic materials to humans and other species and are considered environmental virulence metals together with cadmium, lead, mercury, and chromium (Shen et al. 2017). IARC (International Agency for Research on Cancer) and CDC (Centers for Disease Control)

identified arsenic as the first carcinogenic substance. At the same time, due to its genetic toxicity, arsenic was considered a priority control pollutant by WHO (World Health Organization) (Pari & Jalaludeen 2011, El-Moselhy et al. 2017, Kukucka et al. 2016). It had been documented that when the content of arsenic in a human body is 0.01-0.052g, it would lead to arsenic poisoning, and when the content reached 0.06-0.2g, it would lead the person to death (Zeng et al. 2017, Nandre et al. 2017, Mukherjee et al. 2017). Documents showed that many people died from arsenic poisoning and hundreds of millions of people were being at serious risk from As in many countries throughout the world, such as China, India, Vietnam, Bangladesh, etc. (Itziar et al. 2004). The removal of arsenic from water and wastewater has become the focus problem in many countries (Egbosiuba et al. 2020, Ding et al. 2017, Molinari & Argurio 2017).

Adsorption technology of activated carbon has been deemed one of the most popular technologies for pollution removal in water and wastewater treatment (Orlando et al. 2020, Kiran et al. 2020, García-Mateos et al. 2015). However, there are still many drawbacks in the adsorption of activated carbon for heavy metals, such as low selectivity, low adsorption capacity, etc. In recent years, some modification methods have been proposed to improve the adsorption

capacity and selectivity of activated carbon. For example, Yang et al. modified the ACF with nitration treatment, heat treatment, and heat treatment after nitration, and they showed that the modification changed the surface physical and chemical properties of the ACF samples, and increased the adsorption capacity of ACF (Yang et al. 2016). Shafeeyan et al. studied the surface modification of activated carbon with gaseous ammonia and showed that the adsorption capacity of activated carbon for carbon dioxide increased with the introduction of basic nitrogen functionalities into the carbon surface (Shafeeyan et al. 2010). Palomo et al. produced more condensed nitrogen structures (such as pyridinic and pyrrolic structures) in the activated carbons by oxidation and reduction treatment (Palomo et al. 2017). Jaramillo et al. showed the treatment of activated carbon with ozone combined with heat treatment could control the acidic-basic character and the strength of the carbon surface, and the treatment with ozone yielded acidic carbons, carbon dioxide, and steam activations (Jaramillo et al. 2010). Macías-García et al. modified the porous texture and superficial groups of a commercial activated carbon through chemical and thermal treatment and showed high adsorption and electro adsorption for Cu(II) ion (Macías-García et al. 2017); Chiang and Juang had shown that the surface modifications of activated carbon could improve the adsorption for carbon dioxide in general (Chiang & Juang 2017).

With the development of adsorption technology, nanometer materials have been studied widely in water and wastewater treatment (Bao et al. 2016, Sun et al. 2017). As a new nanometer material, magnetic Fe_3O_4 has been studied in water pollution treatment extensively and has received some good results (Song et al. 2015, Uheida et al. 2006, Zavareh et al. 2017, Mhamme & Samaka 2018).

To facilitate the development of adsorption technology in heavy metal removal in the environment, a comparison study of adsorption characteristics of conventional adsorbent of AC (activated carbon), MAC (modified activated carbon), and new material of nanomagnetic Fe_3O_4 for arsenic has been carried out.

MATERIALS AND METHODS

Experimental Materials

The preparation of nanomagnetic Fe_3O_4 : Nanomagnetic Fe_3O_4 (Fe_3O_4 or NMF) was prepared by the method of hydrothermal synthesis (Legod & Waal 2007). Ferrous precursor ($\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, 10g) and ferric chlorite ($\text{FeCl}_3 \cdot 7\text{H}_2\text{O}$, 20g) were dissolved in 1500ml of distilled water. To the filtered solution, 150ml of 25% NH_4OH solution was added to raise the pH to about 11-12. After aging for about 20h at room

temperature, the precipitate formed collected by filtering, washed with 500ml of distilled water, and then allowed to dry at room temperature.

The preparation of AC: The activated carbon was coconut shell granular activated carbon and the particle size was 25cm-50cm in diameter. After soaking in pure water for about 8-10h and washing repeatedly with deionized water, the activated carbon was dried to constant weight for over 24h at 105°C.

The preparation of MAC: The modification of activated carbon was performed under neutral conditions. The activated carbon (100 g) was immersed in a solution of potassium permanganate (0.1mol.L^{-1} , 500mL) and stirred slowly with a constant temperature oscillator for 10h, then the activated carbon was separated and washed repeatedly with deionized water to no color, and dried to constant weight for 24 h at 105°C.

The preparation for arsenic solution: The arsenic(III) (As) was purchased from Beijing Wanjia Biotechnology co. LTD. 1000mg.L^{-1} of As(III) reserve solution was prepared with deionized water and diluted to a certain concentration when used.

All chemicals in the study were of analytical grade.

Adsorption Experiments Method

Adsorption experiments were performed at room temperature ($20 \pm 1^\circ\text{C}$), and the pH of the water was 6.7. The adsorbent (NMF/MAC/AC) was put into 250 mL of water (containing As(III)) and shaken on a magnetic stirrer (50 RPM) for 180 min. Then the solution was filtered with a $0.45\mu\text{m}$ membrane filter and the amount of arsenic adsorbed was analyzed.

Analysis Method

The concentration of arsenic in water was analyzed by atomic fluorescence spectrometry. 1.0ml mixed solution of ascorbic acid and thiourea (10%) was added into 0.1mL water sample and stirred to blend, and then 2% of the hydrochloric acid was added to 10mL, stand still for 60min and then the content of arsenic was analyzed with non-dispersive atomic fluorescence photometer (PF6-2).

RESULTS AND DISCUSSION

Adsorption for Arsenic by NMF, AC and MAC

The study of adsorption capability for As by NMF, AC, and MAC was conducted in 250mL water with initial concentrations of 0.25mg.L^{-1} , 0.50mg.L^{-1} , 0.75mg.L^{-1} , and 1.00mg.L^{-1} , respectively. respectively. The dosage of NMF was 0.10 g, 0.31 g, and 0.62 g, and the dosage of AC and

MAC was controlled at 5 g, 10 g, and 15 g in 250 mL. The adsorption results with the three adsorbents were shown in Fig. 1, and Fig. 3.

It could be seen that the adsorbent of NMF, AC, and MAC had different adsorption capacities for As. The NMF showed high removal efficiency for As though at a low dosage. The removal rate for As could reach 75-85%, 80-90%, and 90-100% at a dosage of NMF was 0.10 g, 0.31 g, and 0.62 g, respectively. While the removal rate for As by adsorption with

the traditional adsorbent of AC was very low, and the removal rate was only 20-30%, 50-70%, and 80-90% at dosages of 5 g, 10 g, and 15 g. On the other hand, with MAC, the removal rate increased to 70-80%, 80-90%, and 80-100% at dosages of 5 g, 10 g, and 15 g. The results also showed that the adsorption of arsenic on NMF was very fast, and the adsorption could reach equilibrium in less than 10 min. The adsorption rate on AC was slow, and the arsenic removal rate increased slowly in 180 min. While the adsorption rate on MAC increased

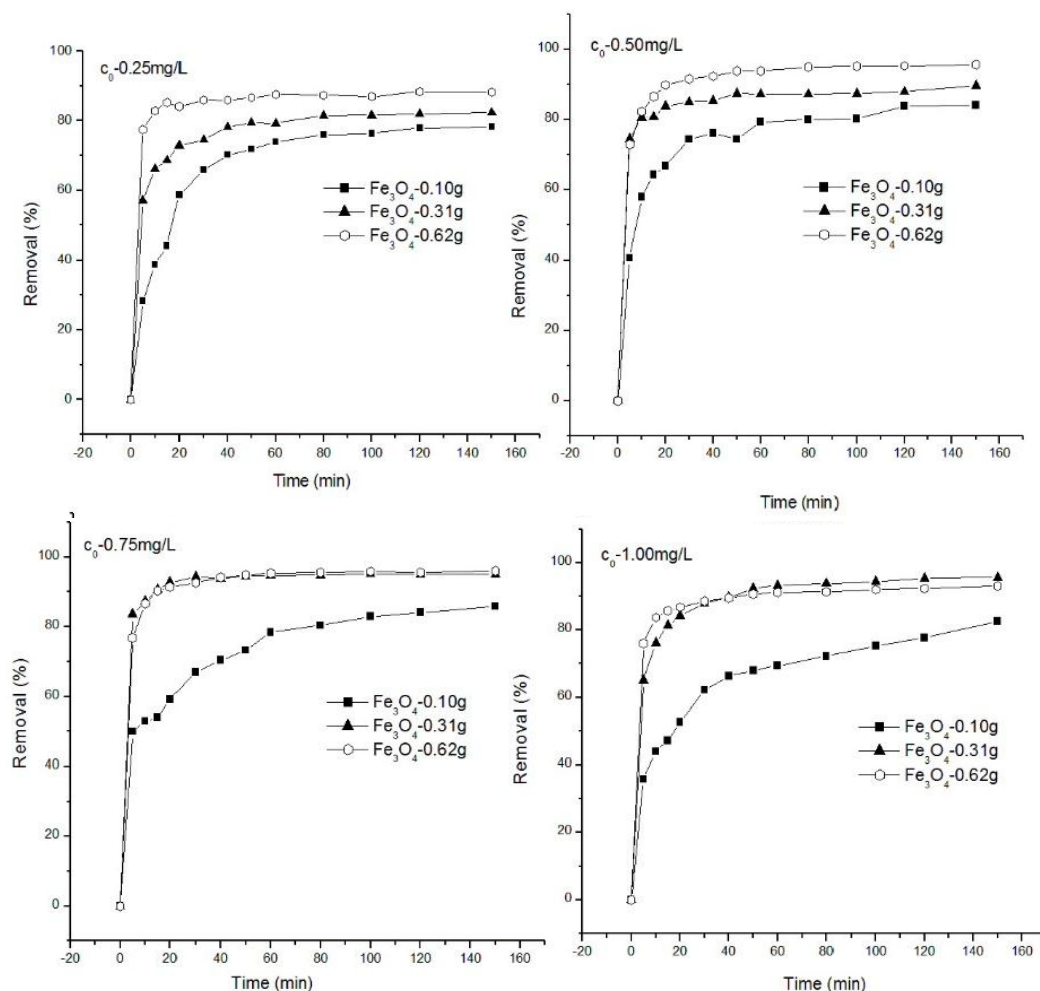


Fig.1: The adsorption for As with NMF.

Table 1: The kinetic parameters of adsorption for As.

	The first-order kinetics model				The second-order kinetics model		
	q_t (mg.g ⁻¹)	q_e (mg.g ⁻¹)	k_1 (min ⁻¹)	R ²	q_e (mg.g ⁻¹)	k_2 (g.mg.min ⁻¹)	R ²
AC	0.013	0.004	0.011	0.671	0.016	0.718	0.996
MAC	0.023	0.012	0.023	0.959	0.024	4.330	0.997
NMF	0.189	0.027	0.042	0.812	0.191	6.947	0.962

Table 2: Parameters of Langmuir Equation and Freundlich Equation.

Langmuir model				Freundlich model		
	a	K_L	R^2	n	K_F	R^2
AC	0.062	1.781	0.961	0.487	0.036	0.821
MAC	0.085	3.518	0.983	0.413	0.062	0.677
Fe ₃ O ₄	2.262	4.557	0.993	0.472	1.758	0.818

significantly, and the adsorption reached balance in 20-30 min. As a new adsorption material, nanomagnetic Fe₃O₄ has a better adsorption effect for arsenic. The traditional material of activated carbon has little adsorption efficiency for As. The adsorption capacity of AC for arsenic was improved to a great extent by the modification of activated carbon.

The study of Adsorption Kinetics and Isotherms

The adsorption kinetics study: The experiment was conducted in 250mL water with an initial concentration of arsenic of 0.25mg.L⁻¹. The dosage of AC, MAC, and NMF was 5.0g,

5.0g, and 0.10g respectively. The adsorption amount at different times was analyzed according to equation (1), and the adsorption capacity results were shown in Fig 4.

$$q_t = \frac{V(C_{0i} - C_i)}{m} \quad \dots(1)$$

In which, q_t was the adsorption amount (mg.g⁻¹); V was the volume of the water (L); C_{0i} was the initial concentration of As (mg.L⁻¹), and C_i was the concentration of As at the time of t ; m was the mass of the adsorbent (g).

The adsorption amount for As with Fe₃O₄ was much higher than that of MAC, and AC. The adsorption for As

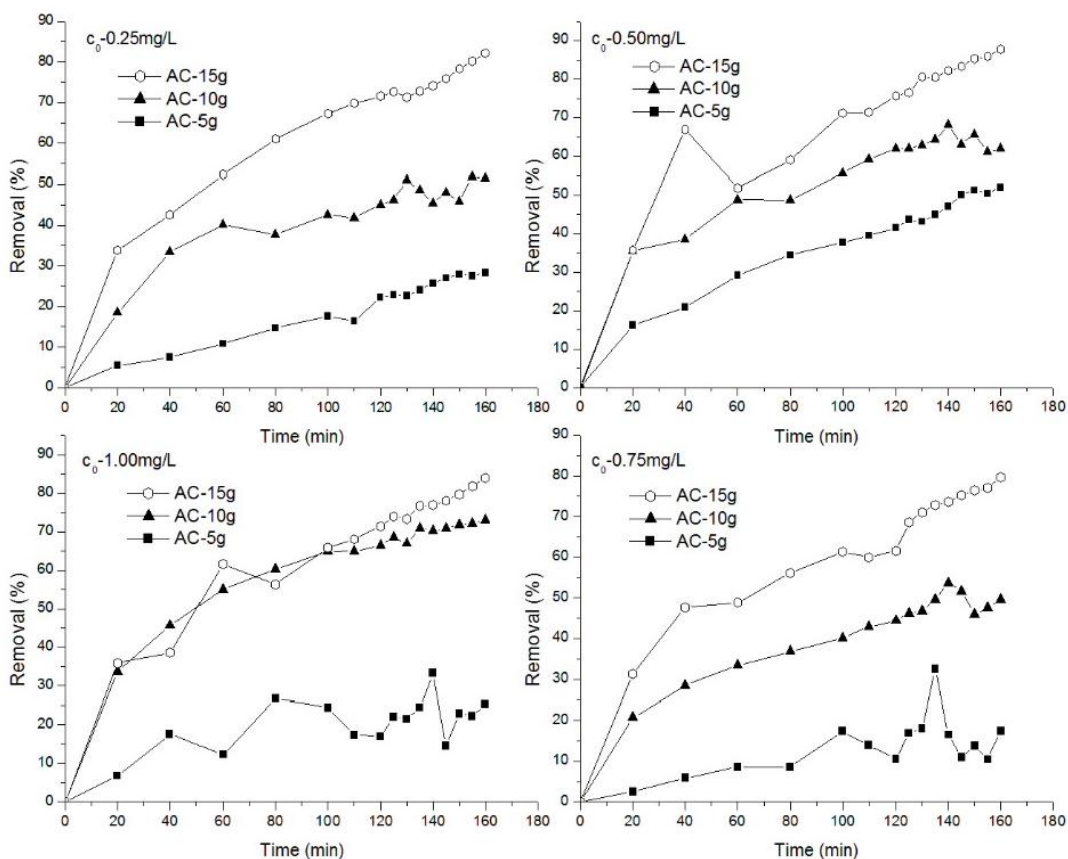


Fig.2: The adsorption for As with AC.

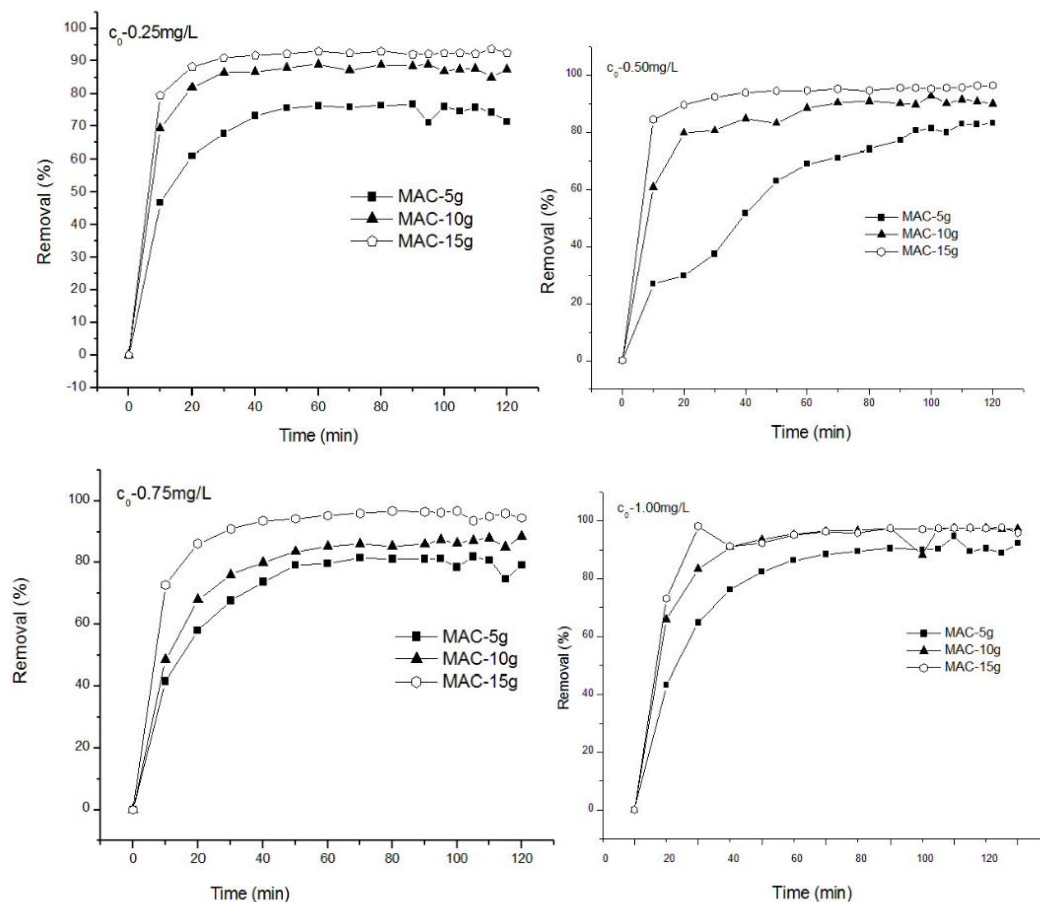


Fig.3: The adsorption for As with MAC.

with NMF reached equilibrium in about 10 min, while for MAC and AC, it needs more than 30 min.

The adsorption kinetics was fitted with a pseudo-first-order kinetics model and pseudo-second-order kinetics model according to equations (2) and (3) (Alvarez-Torrellas et al. 2016, Bao et al. 2017, Zhang et al. 2017), and the fitted results were shown in Table 1.

$$\ln(q_{e1} - q_t) = \ln q_{e1} - k_1 t \quad \dots(2)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_{e2}^2} + \frac{t}{q_{e2}} \quad \dots(3)$$

In which: q_t was the adsorption capacity at the contact time t , q_{e2} was the equilibrium adsorption capacity calculated by pseudo first and second order kinetics model, and k_2 was the rate constant of the pseudo-first and second order kinetics model respectively; R^2 was simulated correlation coefficient.

The results in Table 1 showed that the R^2 of the pseudo-second-order kinetics model of the three adsorbents was

superior to the first-order kinetics model, which indicated that the adsorption of As on the three adsorbents conforms to the pseudo-second-order kinetics model. The adsorption for As

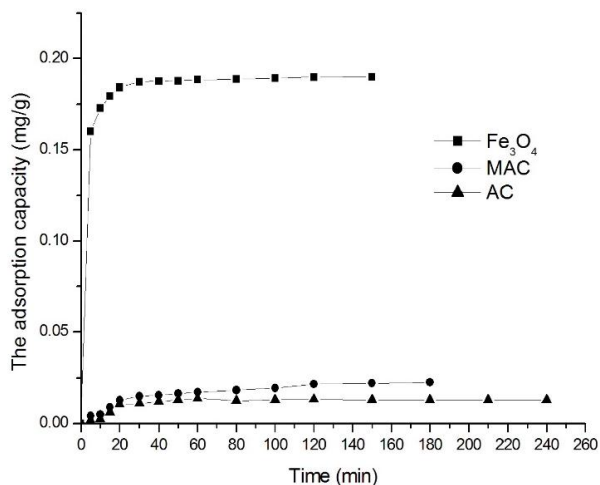


Fig. 4: Adsorption capacity with AC/MAC/NMF.

with AC, MAC, and NMF could be fitted by the pseudo-second-order kinetics model very well (Fig. 5). The rate constant with NMF was all higher than that with AC and MAC.

Adsorption isotherms for As with NMF, AC, and MAC:

The adsorption isotherms for As with the three kinds of adsorbents were analyzed in 250mL water with an initial concentration of As of 0.2 mg.L⁻¹, 0.3 mg.L⁻¹, 0.4 mg.L⁻¹, 0.5 mg.L⁻¹, 0.6 mg.L⁻¹, 1.0 mg.L⁻¹, 1.2 mg.L⁻¹, 1.5 mg.L⁻¹, 2.0 mg.L⁻¹, 3.0 mg.L⁻¹ and 5.0 mg.L⁻¹. The dosage of NMF, AC, and MAC was 0.1 g, 5.0 g, and 5.0 g respectively, and the adsorption time was 120 min. The equilibrium adsorption

capacity was calculated according to equation (1) and the results were shown in Fig. 6.

As a whole, the adsorption equilibrium capacity of As increased with the increase of the initial concentration of As. The NMF exhibited high adsorption capacity for As. The adsorption efficiency of NMF is significantly higher than that of MAC, and the efficiency of MAC is a little higher than that of AC. When the concentration of As is low, the equilibrium adsorption capacity increased rapidly with the increase of As concentration. With the increase of As concentration, more and more adsorption sites on the adsorbents' surface

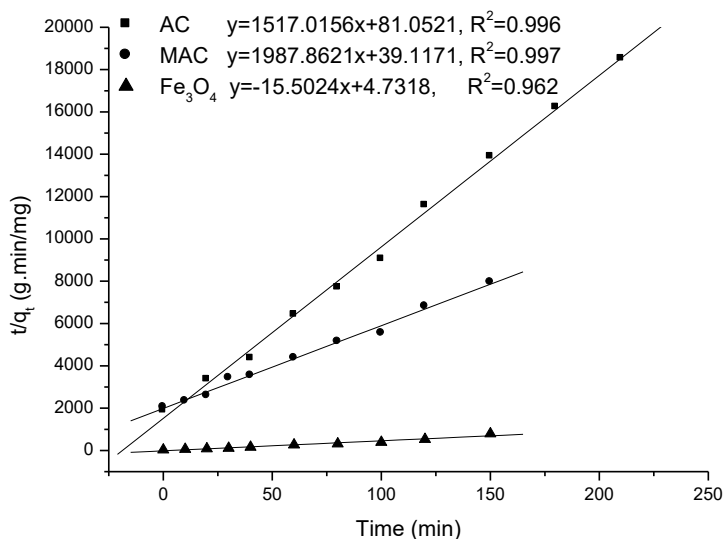


Fig. 5: The simulation results of the pseudo-second-order model.

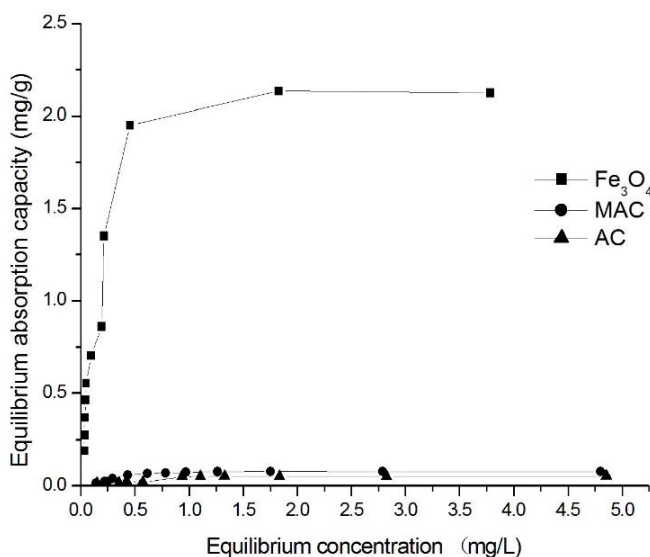


Fig. 6: Adsorption equilibrium for As with NMF, AC and MAC.

were occupied and thus reduced the adsorption capacity, and finally, the adsorption was saturated.

The adsorption equilibrium results were fitted with Langmuir adsorption isotherm (equation (4)) and Freundlich adsorption isotherm (equation (5)) (Pojananukij et al. 2017), and the results were shown in Table 2.

$$q_c = \frac{aK_L C_e}{1 + K_L C_e} \quad \dots(4)$$

$$q_c = K_F C_e^n \quad \dots(5)$$

In which, q_c (mg.g⁻¹) was the equilibrium adsorption, C_e (mg.L⁻¹) was the equilibrium concentration; a (mg.g⁻¹) represent the maximum adsorption in the Langmuir model, K_L , K_F and n were adsorption constants respectively, R^2 was the simulated correlation coefficient.

The results in Table 2 showed that the adsorption for As with the three adsorbents expresses the same characteristics, and the adsorption could be better described with the Langmuir equation (Fig.7). The result in Fig. 7 also showed that the adsorption capacity for As by NMF was much higher than that of MAC, and the MAC was higher than that of AC.

The Analysis of Surface Characteristics of NMF, AC and MAC

The groups on the surface of the adsorbent affect the properties of the adsorbent (Tom & Andreas 2017, Niu et al. 2017, Mines et al. 2017). Fig. 8 showed the FT-IR spectrum results of the three adsorbents of NMF, AC, and MAC. For AC, there was a weak absorption peak at 2,920 cm⁻¹, which was the stretching vibration of the -CH₂ or -CH₃ bond on

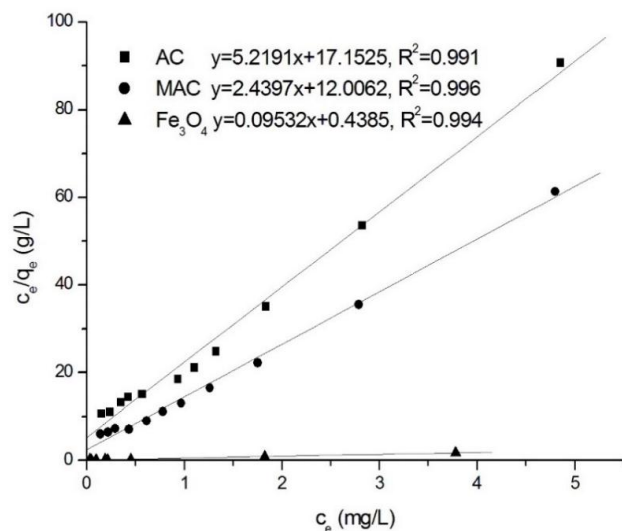


Fig.7: The simulation results of the Langmuir equation.

the surface of alkanes; the strong absorption peak appears at 1,594 cm⁻¹, which could be the stretching vibration of -C=O and -C=C bond, or the bending of the -HN group; the bending vibration of -OH contribute the adsorption peak of 1,436 cm⁻¹; the peak at 1,170 cm⁻¹ could be attributed to the stretching vibration of -C-O-C- or the -C-O stretching vibration of the chain acid anhydride. The peak at 1,031 cm⁻¹ was caused by the stretching vibration of -C-O. At around 749 cm⁻¹, it was caused by the outward vibration bending of -NH. The peak near 603 cm⁻¹ was caused by rocking of -NH (Ribeiro et al. 2015).

For MAC, the absorption peak at 3,526 cm⁻¹ was the single bridge stretching vibration of the alcohol -OH group, at 3,445 cm⁻¹ was the stretching vibration of the primary amine -HN group, and at 1,629 cm⁻¹ was the in-plane bending vibration of the alcohol -OH group. It could be seen that the modification changed the functional groups on the surface of activated carbon greatly, and the hydroxyl groups of alcohols or phenols on the surface of the modified activated carbon increased. The increase of the acidic oxygen-containing groups will adsorb more π electrons existing in a graphite state on the surface of the activated carbon, which makes the surface of the activated carbon carry more positive charges and have a stronger adsorption effect on the electro-negative arsenic ions. Therefore, the removal efficiency for As with modified activated carbon was much better than that of activated carbon.

For NMF, the adsorption peaks appear in 3,122 cm⁻¹, 3,031 cm⁻¹, 2,003 cm⁻¹, 1,761 cm⁻¹, 1,398 cm⁻¹, 1,074 cm⁻¹, 989 cm⁻¹ and 593 cm⁻¹. The absorption peaks were caused by the stretching vibration of -OH, symmetrical stretching vibration of -C≡C, the stretching vibration of aldehyde -C=O or stretching vibration of lipid -C=O, stretching vibration of -C-N or primary inward bending vibration of -OH or stretch-

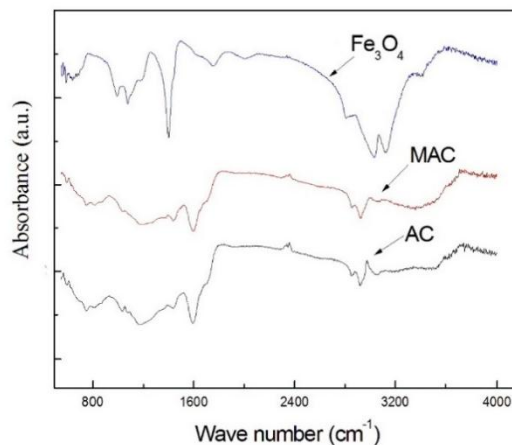


Fig. 8: FT-IR spectra.

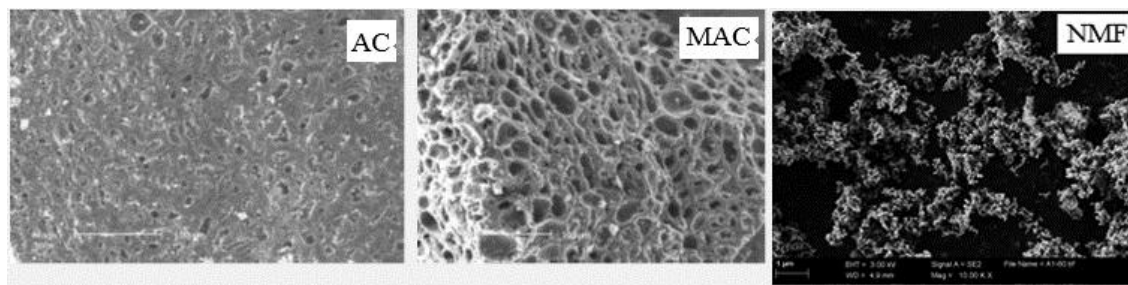


Fig. 9: SEM micrographs of NMF, AC and MAC.

ing vibration of R-NO₂, the stretching vibration of C-O-C or the stretching vibration of C-O, the outward shaking of -NH₂. The oxygen-containing functional groups on the surface of NMF enhanced the adsorption capacity. The surface groups impact the adsorption ability, and NMF showed high adsorption ability for As than AC and MAC.

The surface morphology of SEM of the three adsorbents (Fig.9) showed that the surface of AC was much smoother, and there were a few irregular pores with little mesopore and macro-pore on the surface of AC. While the surface of MAC was much rough, more macropore and mesopore could be seen. The modification process improved the surface pore characteristics and increased the adsorption capacity of activated carbon. The NMF was composed of uniform particles with particle sizes less than 50 nm, the particles were connected by a chain. This kind of chain structure of nanoparticles was conducive to the adsorption and aggregation of heavy metal ions to the surface.

CONCLUSIONS

The adsorption efficiency for arsenic by conventional adsorbent of activated carbon-modified activated carbon and new adsorbent of magnetic nano Fe₃O₄ was compared in the batch experiment. The results showed that the new nanomagnetic Fe₃O₄ had excellent adsorption properties for As. Though the modification with potassium permanganate improved the surface pore structure and improved the adsorption ability of activated carbon, the adsorption efficiency for As with MAC was still low. When the As concentration was 2.5 mg.L⁻¹, the maximum adsorption capacity for As with NMF could reach 2.262 mg.g⁻¹ with a dosage of 0.1 g NMF in 250 mL, while for MAC and AC, the maximum adsorption was only 0.0853 mg.g⁻¹ and 0.0623 mg.g⁻¹ with a dosage of 5g MAC and AC in 250 mL. The adsorption rate for As with NMF, MAC, and AC were different obviously. For NMF, it needs only 10-20 min to reach adsorption equilibrium, while for MAC and AC, the adsorption needs 100 min to reach equilibrium. The adsorption for As with the three adsorbents of NMF, AC, and

MAC followed the pseudo-second-order kinetics model and Langmuir isotherm model all.

ACKNOWLEDGEMENT

The authors thank the support of the “science and technology project of Dalian (No.2015E11SF063)”, “the Fundamental research funds for the central universities” (No. 3132016327)”, and “the Sub-project of Central Sharing Funds for using sea area (No.2013-348-7)”.

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