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Removal of tetracycline from aqueous solution by a Fe₃O₄ incorporated PAN electrospun nanofiber mat

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ABSTRACT

Pollution of antibiotics, a type of emerging contaminant, has become an issue of concern, due to their overuse in human and veterinary application, persistence in environment and great potential risk to human and animal health even at trace level. In this work, a novel adsorbent, Fe₃O₄ incorporated polyacrylonitrile nanofiber mat (Fe-NFM), was successfully fabricated via electrospinning and solvothermal method, targeting to remove tetracycline (TC), a typical class of antibiotics, from aqueous solution. Field emission scanning electron microscopy and X-ray diffraction spectroscopy were used to characterize the surface morphology and crystal structure of the Fe-NFM, and demonstrated that Fe-NFM was composed of continuous, randomly distributed uniform nanofibers with surface coating of Fe₃O₄ nanoparticles. A series of adsorption experiments were carried out to evaluate the removal efficiency of TC by the Fe-NFM. The pseudo-second-order kinetics model fitted better with the experimental data. The highest adsorption capacity was observed at initial solution pH 4 while relative high adsorption performance was obtained from initial solution pH 4 to 10. The adsorption of TC on Fe-NFM was a combination effect of both electrostatic interaction and complexation between TC and Fe-NFM. Freundlich isotherm model could better describe the adsorption isotherm. The maximum adsorption capacity calculated from Langmuir isotherm model was 315.31 mg/g. Compared to conventional nanoparticle adsorbents which have difficulties in downstream separation, the novel nanofiber mat can be simply installed as a modular compartment and easily separated from the aqueous medium, promising its huge potential in drinking and wastewater treatment for micro-pollutant removal.

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Introduction

During the past few decades, antibiotics have been extensively used in both human and animals for disease control and also to improve feed efficiency and promote growth in veterinary operation. In USA, tetracycline (TC) class was reported as the most widely used veterinary antibiotics (FDA, 2011). China has also reported the excessive usage of TC, mostly in animal feed in a 2007 nationwide survey (Hvistendahl, 2012). However, most TC administrated is poorly absorbed by human or animal, resulting as high as 80%–90% of the parent compound excreted (Hirsch et al., 1999). Subsequently, TC residues and their metabolites could enter aquatic environment through manure leaching and run-offs (Sarmah et al., 2006).

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TC has been detected in surface water and ground water in many countries (Lindsey et al., 2001; Kummerer, 2009; Watkinson et al., 2009). An investigation along Huangpu River revealed that TC was the compound with the highest concentration among 22 antibiotics analytes, ranging from 5.61 to 147.15 ng/L (Jiang et al., 2011). Noticeably, wastewater treatment plants have been identified as an important hotspot for TC release (Michael et al., 2013). The presence of TC traces in environments has promoted the development and proliferation of TC resistant genes, which could possess great hazards to human and animal health (Zhu et al., 2013). Thus, there is an urgent need to develop efficient and economical treatment techniques to remove TC from contaminated aqueous environment.

The current technologies for TC removal include biological degradation, ozonation, photocatalytic degradation, ion-exchange, membrane filtration and adsorption (Le-Minh et al., 2010). Compared to other methods, adsorption has prevailing properties such as fast removal rate, and low cost. Many adsorbents have been reported in the removal of TC, such as metal oxides (Gu and Karthikeyan, 2005; Liu et al., 2012a,b), clays (Figueroa et al., 2004; Liu et al., 2012b), resins (Zhang et al., 2014) and activated carbons (Li et al., 2009, 2010). Among them, magnetic nanoparticles (NPs) have drawn great interests in the past few years due to their high specific surface areas, large adsorption capacities and magnetic properties (Zhang et al., 2011; Dai et al., 2012; Bao et al., 2013; Zhou et al., 2012). Although those NPs could be attracted by applying external magnetic field, the separation processes are still tedious and expensive. Hence, development of macro-scale composite nanomaterials containing iron oxide which could be easily separated is highly desirable.

Owing to the high specific surface area, porosity and interconnecting channeling pore structures, one dimensional electrospun nanofibers have become popular supporters for reactive NPs. Various works have been reported for composite nanofibers application in water treatment, such as heavy metal adsorption (Taha et al., 2012), photocatalytic degradation of organic pollutants (Liu et al., 2012c) and disinfection (Qu et al., 2013). However, to our best knowledge, little work has been done on the electrospun composite nanofibers for TC removal.

In this work, a widely studied electrospinning raw material, polyacrylonitrile (PAN), is chosen as the base material due to its easy availability and high tensile strength of the as-spun nanofibers. Fe₃O₄ nanoparticle incorporated polyacrylonitrile nanofibers (Fe-NFM) was successfully prepared by electrospinning and solvothermal method. Their surface morphology and crystal structure were examined through field emission scanning electron microscopy (FESEM) and X-ray diffraction (XRD), respectively. Batch adsorption experiments, including effect of solution pH, adsorption kinetics and adsorption isotherm, were systematically conducted to evaluate the adsorption performance of Fe-NFM for TC removal.

1. Materials and methods

1.1. Materials

Tetracycline hydrochloride (USP grade) was obtained from Amresco (Solon, OH, USA) and used without further purification. PAN (molecular weight: 90,000) was purchased from Kunshan Hong Yu Plastic Co., Ltd. (Suzhou, China). Diethylene glycol (DEG), N,N-dimethylformamide (DMF), N,N-dimethylacetamide (DMAc), anhydrous ferric chloride (FeCl₃), sodium citrate (Na₃Cit) and sodium acetate (NaAc) were of analytical grade and obtained from Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China).

1.2. Preparation of PAN nanofiber mat (NFM)

The electrospinning solution was prepared by adding 2 g PAN powder to 18 g well-mixed DMF/DMAc solution (m:m, 1:1), and the mixture was continuously stirred under water bath for 2 hr by a magnetic stirrer (DF-1, Tianjin, China). The PAN/DMF/ DMAc solution was transferred to a 10 mL syringe secured with a 21-gauge needle. The flow rate was controlled by a syringe pump (TCI-IV, Veryark, Nanning, Guangxi, China) at 0.9 mL/hr. The applied voltage and distance between needle and receiving drum were kept at 12 kV and 15 cm, respectively.

1.3. Preparations of Fe₃O₄ NPs incorporated PAN nanofiber mat (Fe-NFM)

Fe-NFM was synthesized by a modified literature method (Shen et al., 2013). Briefly, 4 mmol anhydrous FeCl₃, 1.6 mmol Na₃Cit and 12.0 mmol NaAc were consecutively added into 40 mL DEG solution with continuously stirring at 80°C until a homogenous solution was formed. The solution was then transferred to a Teflon-lined stainless steel autoclave (Qiangqiang, Shanghai, China). PAN nanofiber mat were then fully immersed in the solution. The autoclaves was sealed and heated at 200°C for a series of reaction time. After cooling, Fe-NFM was retrieved from the autoclave. The Fe-NFM was washed with deionized water for several times, dried at 60°C for 24 hr, and kept in a dessicator as an adsorbent for subsequent experiments.

1.4. Characterization of Fe-NFM

FESEM (S-4800, Hitachi, Tokyo, Japan) with energy dispersive X-ray (EDAX) spectrometer was employed to examine the surface morphologies of NFM and Fe-NFM. A turbo pumped sputter coater (150 T ES, EMS, Hatfield, PA, USA) was used to coat surfaces of the samples with cadmium for 15 sec. XRD patterns were obtained on a X’Pert PRO (PANalytical, Almelo, The Netherlands) X-ray diffractometer with a Cu Kα radiation source (λ = 1.54060 Å).

1.5. Batch adsorption experiments

Batch adsorption experiments, including effect of initial solution pH, adsorption kinetics, effect of ionic strength, and adsorption isotherm, were conducted to evaluate the performance of Fe-NFM for the adsorption of TC from aqueous solution. Unless otherwise stated, all experiments were conducted in triplicate at room temperature with an adsorbent dosage of 0.5 g/L, and the bottles containing the adsorbent (Fe-NFM) and adsorbate (TC) were wrapped with aluminum foil to avoid light exposure, and shaken at a speed of 150 r/min.

In the pH effect study, 10 mg Fe-NFM was added into a 100 mL glass bottle containing 20 mL of 44 mg/L TC solution with different pH ranging from 3 to 12. Solution pH was adjusted by 0.1 mol/L HCl or 0.1 mol/L NaOH. The mixture of adsorbent and adsorbate was equilibrated, and then filtered through 0.45 μm membrane filters before analyzing the TC concentration of filtrate by a UV–vis spectrometer (TU-1810, Shanghai, China).
Beijing Puxi, China) at wavelength of 358 nm. The amount of TC adsorbed onto the Fe-NFM was calculated as follows:

\[
q_e = \frac{(C_0 - C_e)V}{m}
\]

where, \(q_e\) (mg/g) is the amount of TC adsorbed onto the Fe-NFM at equilibrium time, \(C_0\) (mg/L) and \(C_e\) (mg/L) are the concentrations of TC in the solution at the initial and equilibrium times, respectively, \(V\) (L) is the volume of TC solution, and \(m\) (g) is the weight of Fe-NFM.

In the adsorption kinetics study, 100 mg Fe-NFM was added into a 500 mL glass bottle containing 200 mL TC solution with an initial concentration of 44 mg/L, and the initial solution pH was adjusted to 5.95. The samples were drawn at predetermined time intervals and filtered through 0.45 \(\mu\)m membrane filters, and TC concentrations in the filtrates were measured. The amounts of TC adsorbed on the adsorbent at specific time were determined by the following equation:

\[
q_t = \frac{(C_0 - C_t)V}{m}
\]

where, \(q_t\) (mg/g) is the amount of TC adsorbed by the adsorbent at time \(t\), and \(C_t\) (mg/L) is the concentration of TC at time \(t\).

Similar to the kinetics study, three sets of 100 mg Fe-NFM were separately added into 200 mL TC solutions (\(C_0 = 44\) mg/L) with varying NaCl concentrations of 0.05, 0.1 and 0.5 mmol/L. The initial solution pH was adjusted to 6.00 ± 0.05. The samples were drawn, filtered through 0.45 \(\mu\)m membrane filters, and TC concentrations in the filtrates were measured. The amounts of TC adsorbed on the adsorbent at specific time were determined by the following equation:

\[
q_t = \frac{(C_0 - C_t)V}{m}
\]

where, \(q_t\) (mg/g) is the amount of TC adsorbed by the adsorbent at time \(t\), and \(C_t\) (mg/L) is the concentration of TC at time \(t\).

In the adsorption isotherm study, 10 mg Fe-NFM was added into a 100 mL glass bottle containing 20 mL TC solutions with different initial TC concentrations varying from 11 to 444 mg/L, and the initial solution pH was adjusted to 4.00 ± 0.03. Other procedures were the same as the pH effect experiments.

2. Results and discussion

2.1. Preparation of Fe-NFM

For the solvothermal synthesis of Fe\(_3\)O\(_4\) NPs, reaction time plays an important role. Preliminary adsorption experiments were conducted to determine the optimum reaction time for the solvothermal synthesis of Fe-NFM. Fig. 1 depicts the impact of solvothermal synthesis time on TC removal efficiency by the Fe-NFM. At a reaction time of 10 hr, TC removal efficiency by the Fe-NFM was only about 70%. However, when solvothermal synthesis time increased to 12 hr, more than 90% of TC was adsorbed on Fe-NFM. The highest removal efficiency (95.7%) was observed at a reaction time of 14 hr. Further extension of reaction time did not further improve TC removal, thus, 14 hr was chosen as the optimized solvothermal reaction time for the synthesis of Fe-NFM used in the subsequent studies.

2.2. Characterization of Fe-NFM

Phase structures of the as-prepared NFM, Fe-NFM and Fe\(_3\)O\(_4\) NPs, which were simultaneously formed with Fe-NFM after solvothermal treatment, were examined through XRD. Pure PAN NFM has diffraction peaks at \(2\theta = 29^\circ\) (Callister, 1997). In addition, the nanofibers surfaces have transformed from smooth to coarse due to the deposition of Fe\(_3\)O\(_4\) NPs. This randomly distributed one dimensional nanofiber structure in Fe-NFM provided it with large specific surface area and high porosity, which greatly increased the adsorption sites, and reduced mass transfer resistance. The visual appearance of nanofibers changed from white to dark brown after the deposition of Fe\(_3\)O\(_4\) NPs (Fig. 3e). The presence of Fe on NFM was further confirmed by EDAX spectrum (Fig. 4). The atomic ratio of Fe to O is 0.788 to 1, which is close to the Fe:O ratio of 0.75:1 in Fe\(_3\)O\(_4\). As NFM does not contain O, the presence of Fe\(_3\)O\(_4\) was supported by the XRD pattern of Fe\(_3\)O\(_4\) NPs and FESEM observation discussed in follows.

The surface morphologies of the as-prepared NFM and Fe-NFM were examined by FESEM. As shown in Fig. 3a–d, the nanofibers were beadless, uniform, continuous, and randomly distributed. Comparing NFM and Fe-NFM, the fiber diameters are almost the same, ranging from 400–500 nm. However, the nanofibers in Fe-NFM turn wavier. The morphology change could be attributed to increased mobility of PAN molecules at solvothermal temperature (200°C), which was above the glass transition temperature of PAN (104°C) (Callister, 1997). In addition, the nanofibers surfaces have transformed from smooth to coarse due to the deposition of Fe\(_3\)O\(_4\) NPs. This randomly distributed one dimensional nanostructure in Fe-NFM provided it with large specific surface area and high porosity, which greatly increased the adsorption sites, and reduced mass transfer resistance. The visual appearance of nanofibers changed from white to dark brown after the deposition of Fe\(_3\)O\(_4\) NPs (Fig. 3e). The presence of Fe on NFM was further confirmed by EDAX spectrum (Fig. 4).

The atomic ratio of Fe to O is 0.788 to 1, which is close to the Fe:O ratio of 0.75:1 in Fe\(_3\)O\(_4\). As NFM does not contain O, the result from EDAX analysis further proves the presence of Fe\(_3\)O\(_4\) NPs.
2.3. Effect of solution pH

The distribution of TC species in aqueous solution and the surface properties of sorbent are generally highly pH-dependant. The chemical structure of TC is shown in Fig. 5. TC is an amphoteric molecule which has three acid dissociation constants (3.30, 7.68 and 9.68) (Figueroa et al., 2004). It can exist in cationic (H3TC+) (dominant below pH 3.30), zwitterion (H2TC) (dominant between pH 3.30 and 7.68), and anionic (HTC− and TC2−) (dominant above pH 7.68) forms. It is expected that the uptake of TC by the Fe-NFM adsorbent is associated with the solution pH.

The influence of solution pH on TC adsorption by Fe-NFM was investigated via batch adsorption experiments by a series of TC solutions with varying pH levels (Fig. 6). The uptake of TC first rose to a maximum when the initial solution pH increased from 3 to 4. TC adsorption capacity was partially reduced at the initial solution pH range from 4 to 7, and then stayed relatively constant at the initial pH range from 8 to 10. As the initial solution pH was above 10, the adsorption of TC by the sorbent sharply decreased. When the initial solution pH was 12, the adsorbent was found to be not effective for TC removal. Therefore, we conclude that Fe-NFM is effective in TC removal from pH 3 to 10, thus could be widely applied to the treatment of environmental water, ranging from ground water (pH 5.5), to municipal discharged effluent (pH 6 to 7). Leaching of Fe3+ was also evaluated and indicated that when pH was above 5, the leaching was negligible. Hence, Fe-NFM would be an environmental friendly adsorbent when used at moderate pH range.

The observed adsorption behavior could be resulted from a combination of speciation of TC species, surface charge properties of Fe-NFM and the possible complexation between TC and Fe-NFM (Gu and Karthikeyan, 2005). At pH 3, due to protonation of dimethyl-ammonium group, TC dominated in cationic form, while the surface of Fe-NFM was also positively charged (point of
zero charge: 6.1). Strong repulsive force experienced between TC and Fe-NFM would be the major factor contributing to the low TC uptake. Likewise, at pH above 10, electrostatic repulsion between deprotonated TC molecules and negatively charged Fe-NFM surface appeared. Thus, at extreme pH condition, TC uptake could be dominated by the electrostatic interaction. At relative neutral pH, complexation of TC and Fe-NFM was formed and contributed to the adsorption of TC (discussed in Section 2.6). As observed in Fig. 6, the equilibrium pH increased to slightly higher values as initial pH was below 7, and vice versa when initial pH was above 7. This phenomenon could be linked with the buffering effect of the protonated or deprotonated iron oxide hydroxyls (Zheng et al., 2009). A similar effect was observed in other studies of Fe3O4 contained sorbent (Lim et al., 2009b).

2.4. Adsorption kinetics

The adsorption kinetics describes the adsorption rate of adsorbate by the sorbent, which controls the equilibrium time, and the kinetics parameters give important information for designing and modeling the adsorption process. In this study, the adsorption kinetics of TC on Fe-NFM was conducted at initial pH of 5.95, which is a moderate pH usually present in environmental water bodies (Fig. 7). It was found that the TC adsorption by the adsorbent rapidly increased during the first 10 hr, followed by a slower adsorption process, and the equilibrium was reached within 60 hr. It is proposed that the initial adsorption phase is dominated by the external mass transfer, while in the second phase rate-limiting step is intra-particle diffusion, causing a much slower adsorption rate.

Both pseudo first-order and pseudo second-order kinetic models were used to examine the rate of the adsorption process, and the model fittings are shown in Fig. 7. A linear form of pseudo-first-order kinetics can be expressed as:

\[
\log q_e - q_t = \log q_e - \frac{k_1 t}{2.303}
\]

where, \( k_1 \) (hr\(^{-1}\)) is the rate constant of pseudo first-order adsorption.

The pseudo second-order kinetic model based on the adsorption equilibrium capacity is generally determined by (Liu et al., 2013):

\[
\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}
\]

where, \( k_2 \) (g/(mg·hr)) is the rate constant of pseudo second-order adsorption. The parameters \( q_e, k_1, \) and correlation coefficient (\( R^2 \)) of pseudo-first-order are 52.52 mg/g, 0.0647 hr\(^{-1}\), and 0.9132, respectively. \( q_e, k_2, \) and \( R^2 \) of pseudo-second-order kinetic model are 54.88 mg/g, 0.120 g/(mg·hr), and 0.9354, respectively. Therefore, the pseudo second-order model fitted the experimental data better than pseudo first-order model.

2.5. Effect of ionic strength

Due to the presence of various ions in the aqueous environment, the background ionic strengths of the waters are different. It is important to evaluate the influence of background ionic strength on the adsorption of TC by the adsorbent. On the other hand, an ionic strength dependence study on the uptake of pollutant by sorbent is a useful method to investigate the underlying adsorption mechanism and to distinguish between inner- and outer-sphere surface complexes.

In order to investigate the effect of ionic strength and adsorption mechanism, the influence of ionic strength on TC adsorption by the adsorbent was studied. Clearly as shown in Fig. 8, with increasing NaCl concentration from 0 to 0.5 mol/L, the TC adsorption on Fe-NFM increased as well. No screening effect, i.e. placing Na\(^+\) or Cl\(^-\) between TC and Fe-NFM, was observed with increasing ionic strength, suggesting that TC adsorption on Fe-NFM at pH 6 was not dominantly attributed by electrostatic interaction. There was also no observation of competitive effects by Na\(^+\) and Cl\(^-\) to TC uptake, probably due...
to the low initial TC concentration (equivalent to $10^{-4}$ mol/L) and the resultant low surface coverage of TC on Fe-NFM. When Fe-NFM was dispersed in TC solution, a diffusive electric double layer could be formed by the protonation of iron oxide. With an increase in salinity level, compression of the diffusive electric double layer would happen, resulting in a reduction in repulsive force and improvement of TC adsorption (Chen et al., 2012). Hence, it is proposed that complexation between TC and hydrolyzed Fe$_3$O$_4$ from Fe-NFM surface through inner-sphere mechanism was the main contributor to the adsorption process (Gu and Karthikeyan, 2005).

### 2.6. Adsorption isotherm

The adsorption isotherm provides the most important information in understanding an adsorption process. The adsorption isotherm experiments of Fe-NFM were conducted with TC concentrations ranging from 11 to 444 mg/L at 25°C (Fig. 9). As presented in Section 2.3, the highest adsorption was found at pH 4, thus pH 4 was chosen in the isotherm experiments in order to explore the greatest potential of Fe-NFM for TC adsorption based on the maximum adsorption capacity. Both Langmuir and Freundlich isotherms were employed to analyze the experimental data (Lim et al., 2009a).

Langmuir isotherm assumes monolayer adsorption on a homogeneous surface and the maximum adsorption capacity is determined by Eq. (5):

$$q_e = \frac{q_{\text{max}} b c_e}{1 + b c_e} \tag{5}$$

where, $q_{\text{max}}$ (mg/g) and $b$ (L/mg) are the Langmuir constants.

TC adsorption was also investigated using the Freundlich isotherm (Eq. (6)).

$$q_e = k c_e^1/n \tag{6}$$

where, $k$ and $n$ are the Freundlich constants, $k$ is related to the adsorption capacity of the adsorbent, and $1/n$ is a constant known as the heterogeneity factor that is related to the surface heterogeneity.

The values of Langmuir and Freundlich constants were obtained from fitting the experimental data (Fig. 9). Experimental data fits Freundlich model much better than Langmuir model, suggesting that the uptake of TC on Fe-NFM should be multilayer adsorption process. According to Langmuir model, the maximum adsorption capacity of TC on Fe-NFM was calculated as 315.31 mg/g. Here, we compare the adsorption capacity between Fe-NFM and other recently developed magnetic adsorbents. As shown in Table 1, Fe-NFM possesses a relatively high adsorption capacity compared to many other recently reported magnetic adsorbents, demonstrating that Fe-NFM could be applied as an efficient adsorbent for TC removal.
3. Conclusions

A Fe$_3$O$_4$ NPs composite nanofiber material, Fe-NFM, was successfully prepared as an effective adsorbent for the removal of TC from aqueous environment. Fe-NFM was characterized by FESEM and XRD. The results indicated that with the solvothermal method, Fe$_3$O$_4$ NPs can be successfully incorporated to the PAN nanofibers surface. This composite nanofiber adsorbent could effectively remove TC at the initial solution pH range from 4 to 10. Electrostatic interaction and complexation between TC and Fe-NFM both played important roles in TC adsorption. The kinetic data correlated better with pseudo-second-order kinetic models than pseudo-first-order kinetic model. Freundlich isotherm model could better describe the adsorption equilibrium data, while the maximum adsorption capacity was 315.31 mg/g as obtained from Langmuir model, Freundlich isotherm model could better describe the adsorption equilibrium data, while the maximum adsorption capacity was 315.31 mg/g as obtained from Langmuir model.

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<td>German Research Center for Environmental Health, Germany</td>
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<tr>
<td>Zhejiang University, China</td>
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### Terrestrial environment

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<tbody>
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<td>Christopher Anderson</td>
<td>Massey University, New Zealand</td>
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<td>Zuzong Cai</td>
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### Atmospheric environment

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<td>Centre National de la Recherche Scientifique France</td>
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<td>Xin Yang</td>
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### Environmental biology

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<td>Sijin Liu</td>
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