Bioresource Technology 233 (2017) 159–165

Contents lists available at ScienceDirect

Bioresource Technology

journal homepage: www.elsevier.com/locate/biortech

Preparation of biochar by simultaneous carbonization, magnetization and activation for norfloxacin removal in water

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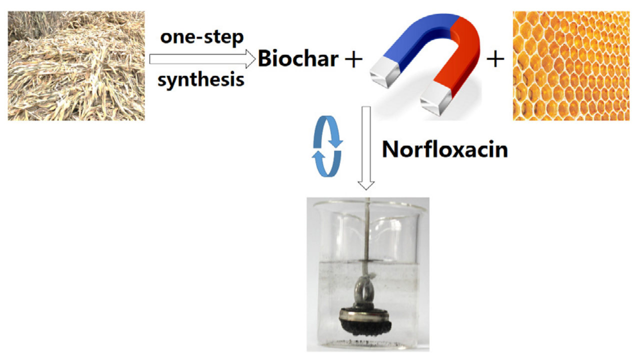
HIGHLIGHTS

- AMB can be prepared by simultaneous calcination, magnetization, and activation.
- AMB has the large surface area and can be conveniently recycled.
- AMB showed the strong adsorption of norfloxacin.
- AMB had a good adsorption effect on norfloxacin in a wide pH range.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 11 January 2017
Received in revised form 20 February 2017
Accepted 22 February 2017
Available online 27 February 2017

Keywords:
Biochar
Norfloxacin
Magnetization
Activation
Simultaneous

ABSTRACT

Activated magnetic biochar (AMB) was prepared with corn stalks, reed stalks, and willow branches by simultaneous carbonization, magnetization, and activation, and used for norfloxacin removal in water. The exploration results showed that the zeta potential was positively charged at pH 2–10. These prepared activated magnetic biochars have a large specific surface area (>700 $m^2 \cdot g^{-1}$) and pore volume (>0.3 $cm^3 \cdot g^{-1}$). The quasi-second-order kinetic adsorption equation could better describe the adsorption of NOR on AMB. The Langmuir isotherm showed the better fitting results on AMB. The AMB showed the strong adsorption of NOR, and the saturated adsorption capacity of corn activated magnetic biochar was the highest, 7.6249 $mg \cdot g^{-1}$. The adsorption of NOR on AMB was a spontaneous endothermic process. The effect of pH on the adsorption behaviors of NOR on AMB was not obvious, and AMB had a good adsorption effect on NOR in a wide pH range.

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1. Introduction

Norfloxacin is one of the quinolone antibiotics. In recent years, due to the rapid development of quinolone antibiotics, norfloxacin (NOR) has become one of the most widely used quinolone antibiotics (Zhang et al., 2013a). NOR is extensively used in human clinic and livestock breeding because it can effectively inhibit Gram-positive bacteria and Gram-negative bacteria (Zhou et al., 2016). Although the application of quinolone antibiotics have been banned in animal husbandry in the European Union and other regions (Kools et al., 2008), antibiotics such as quinolones have been widely used in livestock disease prophylaxis in many regions, especially in aquaculture (Cabello et al., 2016, 2013).

Human and animal absorption of these antibiotics is low and 60–70% of the final NOR will be discharged into the water environment through feces and urine. It is difficult to completely remove NOR from water with traditional water treatment methods (Özcan et al., 2016). Residual NOR in the water environment may enhance drug resistance and contaminate drinking water, thus threatening

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http://dx.doi.org/10.1016/j.biortech.2017.02.103
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human health (Zhou et al., 2016). Therefore, it is necessary to remove NOR from the water environment. Effective removal methods of such antibiotics include adsorption, photolysis, chemical oxidation, etc (Babić et al., 2013; Naik et al., 2009; Peng et al., 2012a,b).

Among these method, biodegradation and advanced oxidation methods have some limitations, but the application scope of the adsorption method is wide (Peng et al., 2012a,b; Wang et al., 2009). At present, the studies of the antibiotics adsorption were focused on relatively cheap biochar prepared with forestry wastes, agricultural wastes, and industrial organic wastes (Cui et al., 2016; Teixidó et al., 2011; Yao et al., 2013). Biochar has been extensively studied and widely applied in the fields of pollutant treatment and water purification because of its inherent characteristics such as porous structure, high specific surface area, large pore volume, acid and alkali corrosion resistance, and rich functional groups (Ahmad et al., 2014; Lehmann et al., 2011). However, due to the small diameter of biochar particles, it is difficult to separate the effluent from the solution after treatment (Zhang et al., 2007). A large number of pollutants are contained in the particles. If these particles are not removed in time, secondary pollution will be generated, thus affecting biochar regeneration and re-use. Therefore, the application of biochar in wastewater treatment is limited.

In the past, biochars were often separated by filtration, but the filtration method might lead to screen clogging or adsorbent loss (Zhang et al., 2007). Therefore, biochar is magnetized by introducing a magnetic medium such as iron or a cobalt compound so that the biochar can be sucked out in an external magnetic field to realize solid–liquid separation (Reddy and Lee, 2014). The carbon material loaded with Fe^{3+} salt is carbonized at high temperature and biochar can be easily prepared with γ-Fe_2O_3 as the magnetic medium (Zhang et al., 2013b; Zhu et al., 2014a). However, the magnetic medium hinders the development of the pore structure of carbon materials. Magnetic materials obtained in this method have the lower specific surface area and the pore structure is not fully developed, thus limiting the utilization of waste biomass. Activation can increase the surface area and the porous structures, form some surface oxygen-containing functional groups, and further increase the adsorption capacity of pollutants (Angın et al., 2013; Trakal et al., 2014).

In this paper, corn stalks, reed stalks, and willow branches were used to prepare biochar by simultaneous carbonization, magnetization, and activation. Activated magnetic biochar (AMB) with the high performance was prepared by simultaneous calcination, magnetization, and activation of $ZnCl_2$ and $FeCl_3$ impregnated raw materials by calcination. The magnetic composite materials obtained with this simple one-step synthesis has the large surface area and can be conveniently recycled.

Magnetization and activation are two important hotspots in biochar study (Tang et al., 2013). However, in previous many studies, magnetization methods of biochar are separated from activation methods (Bastami and Entezari, 2012; Han et al., 2015). The combined preparation method of carbonization, magnetization and activation of biochar with in one step was seldom reported.

The adsorption properties of AMB were investigated by controlling the pH, adsorption time, and the initial concentration of NOR. The adsorption mechanism was discussed based on kinetic equation fitting, adsorption isotherm fitting, and calculation of thermodynamic parameters. The study results provided a scientific basis for the application of AMB in the treatment of waste water containing NOR.

2. Materials and methods

2.1. Main reagents and solutions

Norfloxacin standard was purchased from the National Institute of Food and Drug Control (purity 99.5%), molecular formula: $C_{16}H_{18}FN_3O_3$, relative molecular mass of 319.24. All the drugs were of analytical grade.

2.2. Preparation of biochar by simultaneous carbonization, magnetization and activation

Corn stalks, reed stalks, and willow branches were acquired from Panshan County, Panjin City, Liaoning Province, China. Then stalks were washed with deionized water and dried. After crushing with plant crusher, the crushed stalks passed through a 100-mesh sieve and then collected.

1 g of $ZnCl_2$ and 2 g of $FeCl_3$ were added to 50 ml of deionized water and mixed uniformly. Then, 10 g of raw material powder was added to the mixed solution. Next, the mixed solution was placed in a constant-temperature shaker and shaken at a speed of 150 rpm for 24 h, and then dried at 80 °C.

The collected particles were put into a tubular resistance furnace. Nitrogen flow was purged into the furnace according to the flow rate of 400 $mL\cdot min^{-1}$ in order to maintain the low oxygen content in the furnace. The temperature in the furnace was increased to 500 °C according to the program-controlled heating rate of 10 $°C\cdot min^{-1}$ and then maintained for 90 min pyrolysis. After the pyrolysis process, biochars were naturally cooled to room temperature and then removed from the furnace. Then biochars were washed with deionized water to neutral pH and dried at 105 °C. Finally, Corn activated magnetic biochar (CAMB), reed activated magnetic biochar (RAMB) and willow activated magnetic biochar (WAMB) were obtained.

2.3. Adsorption experiments

2.3.1. Adsorption kinetics experiments

The NOR solution of 10.0 $mg\cdot L^{-1}$ was prepared, and 25.0 mL of the solution was placed in a centrifuge tube containing 0.1g of corn activated magnetic biochar (CAMB), reed activated magnetic biochar (RAMB) and willow activated magnetic biochar (WAMB). The centrifuge tubes were put on the shaking table under the conditions of 25 °C and 150 $r\cdot min^{-1}$. The tubes were taken out respectively after 10 min, 30 min, 1 h, 2 h, 4 h, 8 h, 16 h and 24 h. Then the solution in the tubes was filtered through the membrane with the pore size of 0.45 μm and the concentration of NOR in the filtered solution from each tube was measured.

2.3.2. Adsorption isotherm experiments

The NOR solution of 1, 2, 3, 5, 6, 8 and 10 $mg\cdot L^{-1}$were prepared, and 25 mL of different concentrations of NOR solution were accurately weighed into a centrifuge tube containing 0.1g of CAMB, RAMB and WAMB respectively.

The centrifuge tubes were placed in a constant temperature shaker and shaken for 24 h at 15, 25, 35 °C (288.15, 298.15, 308.15 K) and 150 $r\cdot min^{-1}$, respectively. Then the solution in the tubes was filtered through the membrane with the pore size of 0.45 μm and the concentration of NOR in the filtered solution from each tube was measured.

2.3.3. Effects of pH on AMB adsorption

The NOR solution of 10 $mg\cdot L^{-1}$ was prepared, and 25 mL of the solution was placed in a centrifuge tube containing 0.1g of corn activated magnetic biochar (CAMB), reed activated magnetic biochar (RAMB) and willow activated magnetic biochar (WAMB). The pH of the solution was adjusted with NaOH and HCl, so that the initial pH of the solution was in the range of 2–10.

The centrifuge tubes were placed in a constant temperature shaker and shaken for 24 h at 25 °C and 150 $r\cdot min^{-1}$.Then the solution in the tubes was filtered through the membrane with the pore

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