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Original

Fixed bed adsorption of tetracycline on a mesoporous activated carbon: Experimental study and neuro-fuzzy modeling

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Abstract

This study investigates the use of synthesized mesoporous carbon in the fixed bed adsorption, as a promising process, to eliminate tetracycline from wastewater. In order to study the adsorptive capability of adsorbent, particles were embedded in a laboratory-scale Pyrex glass tube. An increase in initial concentration and decrease in bed height and flow rate led to the higher adsorption capacity. The highest bed capacity of 76.97 mg g^{-1} was obtained using 4 cm bed depth, 4 mL min^{-1} and 50 mg L^{-1} influent concentration. The initial part of breakthrough curve perfectly matched the Adams–Bohart model at all experimental conditions. However, it was anticipated that Yoon–Nelson model could predict the whole curve acceptably, the results showed an inaccurate fitting. Therefore, the adaptive neuro-fuzzy inference system (ANFIS) was used to predict the breakthrough curve using data series of adsorption experiments. This model indicated a good statistical prediction in terms of relative errors.
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Keywords: Apricot shell; Tetracycline; Column adsorption; Machine learning; Neuro-fuzzy

1. Introduction

Only three percent of the water found on the earth's surface is freshwater and a large amount of that is locked up in glaciers, ice caps and permanent snow. The remaining freshwater is in groundwater, lakes and rivers which is used for agriculture, industry and so on. But regrettably, such activities can contaminate freshwater by a large variety of potentially perilous substances. The most important risk from the presence of these pollutants in surface water is the long-term harmful consequences on various ecosystems, including acute and chronic toxicity (González-Pleiter et al., 2013). In spite of successful effect of antibiotics in veterinary and human medicine treating bacterial and protozoan infections and as additives in animal foodstuffs, their residues in the water can cause antimicrobial resistance genes (ARGs), even at relatively low concentrations (Xu et al., 2016). The transformation and migration of these ARGs in the ecosystem is potentially more harmful than the antibiotic residues in the environment (Chen, Zheng, Zhou, & Zhao, 2017). According to the WHO projection, they will cause dying of 10 million people annually by 2050; consequently this threatening pollutant is considered as one of the most important health concerns of the current century (He et al., 2016). The concentrations for antibiotic residue in the hospital and pharmaceutical manufacturing wastewater have been reported usually up to 100 to 500 mg L^{-1} (Larsson, de Pedro, & Paxeus, 2007).

Adsorption by porous materials has been widely considered to be the most promising and robust method in eliminating antibiotics from wastewater due to its low cost, simplicity, fastness, high efficiency as well as environmental friendliness (Marzbali, Mir, Pazoki, Pourjamshidian, & Tabeshnia, 2017). Furthermore, producing low-cost adsorbent by agricultural waste as a raw material, makes adsorption an operational process (Martins et al., 2015). The continuous fixed bed mode peruses the practical application of the adsorption process. Moreover, several advantages like simple operation, high yield and easy scale up from a laboratory-scale distinguish this process from batch mode adsorption (Oguz, 2013).

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Common methods in chemical processes usually have limitations of complexity and nonlinearity in their systems. Among different techniques proposed to these processes, adaptive neuro-fuzzy inference system (ANFIS) is highly recommended; this is not only because it does not need governing chemical and physical laws of processes, but also due to this fact that once it is developed and trained, ANFIS model can easily predict the output of system for further designation of chemical processes (Cole, Powell, & Edgar, 2012; Pirdashti, Curteanu, Kamangar, Hassim, & Khatami, 2013; Shacham & Brauner, 2008). The neuro-fuzzy approach has recently been developed to predict Pb(II) adsorption by ostrich bone (Amiri, Abedi-Koupai, Eslamian, Mousavi, & Hasheminejad, 2013), methane adsorption on zeolites (Rezaei, Rahmati, & Modarress, 2015) and Cu(II) removal from Leachate (Turan & Ozgonenel, 2013).

Consequently, this work aims to study tetracycline removal from aqueous solution by synthesized apricot shell-activated carbon in a continuous adsorption system. We have investigated the effects of flow rate, influent concentration and bed depth on tetracycline uptake by the produced adsorbent in a laboratory scale fixed-bed column. In addition, a model based on ANFIS system has been designed to predict tetracycline adsorption as a function of empirical parameters.

2. Experimental

2.1. Materials

Phosphoric acid with a purity of 85% was purchased from the Merck Company. The precursor, apricot nut shell from Iranian local fruit, was chosen as a raw material for production of adsorbent. Initially the shells were cleaned and washed with hot distilled water in order to remove dust-like impurities and then were dried in air oven at 80 °C for 24 h. Subsequently, the shells were ground in a mill and sieved to the desired particle size (≈ 500 μm). The sample kept in a closed bottle for further experiments. Hydrochloride salt of tetracycline (>97%) used in this study was purchased from Sinadaro Pharmaceutical Company (Tehran, Iran) and was used without further purification. The properties of tetracycline hydrochloride are listed in Table 1. All drug stock solutions were prepared by dissolving accurately weighed drug in high-purity water for needed concentrations.

2.2. Adsorbent preparation and characterization

The nanoporous activated carbon was prepared from the carbonaceous material as follows: Firstly, the raw material (5 g) was immersed into the prepared phosphoric acid at a 1:1 impregnation weight ratio under magnetic stirring for 24 h, and then the mixture was dried in oven at 100 °C for 2 h. The dried mixture was placed in an electrical furnace with a length of 25 cm and a diameter of 3 cm, which was heated at the rate of 7 °C min^{-1} until it reached 400 °C, and then maintained for 90 min under nitrogen flow of 200 mL min^{-1}. After the activation, the sample was cooled down to room temperature and was washed several times with hot distilled water until the filtrated solution reached pH 6.5. This was done to remove the remaining phosphoric acid and also other inorganic species that might be formed during the process (Martins et al., 2015). Finally, the sample was placed in an air oven (at 80 °C for 24 h) for complete drying. The comprehensive description of production stages was presented in our previous study (Marzbali, Esmaieli, Abolghasemi, & Marzbali, 2016). The activated carbon yield was calculated based on following equation:

$$yield(\%) = \frac{W_{AC}}{W_{AS}} \quad (1)$$

where W_{AC} (g) is the dry weight of final activated carbon and W_{AS} (g) is the dry weight of apricot stone.

The produced adsorbent was characterized by several analyzes. The Brunauer–Emmett–Teller (BET) method was applied to study the surface area and porosity of activated carbon. This analysis was carried out by N_2 adsorption–desorption isotherms at 77 K using Quadra Chrome adsorption instrument. The surface area was calculated by linear part of BET plot in the relative pressure ranged between 0.05 and 0.25. Moreover, the microscopic features of the raw material and derived adsorbent were studied by the Field Emission Scanning Electron Microscopy (FE-SEM).

2.3. Fixed bed column experiments

The fixed-bed mode is more similar to practical wastewater treatment and presents useful adsorption properties of a newly developed adsorbent. The fixed bed adsorption experiments were carried out in a laboratory-scale glass column with an inner diameter of 5 mm and 20 cm of length. The prepared adsorbent was packed in the column, with glass wool layer at both ends of the bed to ensure a good liquid distribution. It should be noted that tetracycline adsorption by glass wool is inconsiderable. The drug stock solution was pumped into the bed in an upward direction using a peristaltic pump (Fig. 1). The experiments were done at different bed heights (2–6 cm), influent tetracycline concentrations (20–80 mg L^{-1}) and influent flow rates (4–8 mL min^{-1}). The pH of inlet solution was set at 5, as an optimum value, which was obtained in our previous study (Marzbali et al., 2016). After beginning the process, the effluent sample was collected at regular time intervals, centrifuged at 3000 rpm for 1 h and then analyzed to obtain effluent concentration. For analysis, several specified concentrations of

Table 1
Characteristics of tetracycline.

Compound	Chemical formula	Color	Water solubility[a] (g L^{-1})	Molecular weight[a] (g mol^{-1})
TC·HCl	$C_{22}H_{24}N_2O_8 \cdot HCl$	Yellow crystalline powder	50 @ 72 °F	480.8955

[a] Data from CAMEO Chemicals (http://cameochemicals.noaa.gov/chemical/18230).

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