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Investigation of effective parameters on adsorption of amoxicillin from aqueous medium onto activated carbon

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ABSTRACT

In this study, the adsorption of amoxicillin onto activated carbon was investigated. The effect of particle size and the effluent flow rate was discussed as well as the kinetics and isotherm of adsorption equilibrium. The isotherm equilibrium studies showed that the Langmuir model was appropriate for describing the adsorption equilibrium of amoxicillin onto the activated carbon. Furthermore, the kinetics of adsorption fit the pseudo-second-order model while the highest adsorption amount occurred at pH = 5. Moreover, the change of particle size from 600 microns to 125 microns resulted in increasing the adsorption amount of 102 mg/g to 225 mg/g. Furthermore, the breakthrough curves indicated that the controlling mechanism of mass transfer was intra-particle diffusion. Also, by reducing the length of the bed from 6.8 to 3.4 cm, the breakpoint time decreased from 3.2 hours to 54 minutes at 300 ppm initial concentration. Eventually, the breakpoint time increased from 2 minutes to 55 minutes by decreasing the average particle diameter from 840 to 250 microns.

1. Introduction

Access to safe potable water is an essential requirement for society. The discharging of pollutants into the environment inflicts harmful effects on human health, crops, and natural ecosystems. This has led to a reduction in the discharge of such substances into the environment [1]. Antibiotics have been considered as emerging pharmaceutical contaminants in the aquatic environment, even at very low concentrations due to their adverse effects on human and animal life [2]. In general, bio-degradation and chemical oxidation, as well as physical methods, have been proposed for removing these emerging pollutants from aqueous mediums [3]. Since conventional biological techniques have low removal efficiency and chemical oxidation is often complex and expensive, physical methods, including membrane and adsorption, appear to be the most appropriate options [4]. According to various reports, microfiltration and ultrafiltration are not effective in the removal of pollutants measuring 100 to 1000 times smaller than the pores [5]. However, nanofiltration and reverse osmosis have shown an appropriate capacity for removing pharmaceuticals from wastewater [6]. However, it should be noted that membrane processes generally require high pressure and have high operating costs that limit their usage [7]. Adsorption can be considered as an efficient method among physical methods to remove organic compounds from aqueous medium [8,9]. This process is considered a superior method for the separation of toxic organic compounds from contaminated water on solid adsorbents because of its simplicity in design and operation. However, the main disadvantages include the regeneration of the adsorbent and its selectivity [10,11]. Activated carbon (AC) is the most widely used adsorbent for removing organic contaminants from wastewater due to its excellent properties such as high porosity and high specific surface area, as well as good adsorption capacity and cheap source availability [12]. Amoxicillin is one of the most frequently used oral antibiotics, which belongs to the penicillin group of drugs and is one of the beta-lactams. The consumption of water contaminated by these antibiotics may lead to

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acute poisoning [13]. Hence, its release into the environment should be limited, especially from hospital wastewater sources. Several papers have investigated the capacity of the adsorption of amoxicillin onto activated carbon powder [12,14]. Lima et al. (2018) synthesized activated carbons from the capsules of Para cashew for the removal of amoxicillin in simulated hospital effluents; they reported an excellent removal of up to 98% [15]. Saucier et al. (2017) investigated the removal of amoxicillin and paracetamol from synthetic wastewater by using magnetic activated carbon beads. They synthesized two different adsorbents by the pyrolytic method using a mixture of iron(III)/cobalt(II) benzoates and iron(III)/cobalt(II) oxalates; both adsorbents had a high removal capability for simulated hospital effluents [16]. In another work, Limousy et al. (2017) used chemically activated carbon particles derived from olive stone for the removal of amoxicillin from the aqueous solution; they reported that 93% of the amoxicillin was removed at 20 °C with the initial concentration of 25 mg/L [17]. The majority of the previous researches focused on the surface modification of AC for improving the adsorption capacity. However, the impact of process parameters, such as the particle size of the granular AC and the feed flow rate to adsorbent loading ratio, which are very important in the adsorption column design have not been intensely studied. Therefore, this study aims to investigate the important parameters of the column adsorption process, which are essential in the scale-up and designing of commercial treatment plants for pharmaceutical wastewaters.

2. Materials and methods

2.1. Materials

The Amoxicillin was obtained from the Daana Pharmaceutical Company (Tabriz, Iran) in analytical purity and was used without further purification. The chemical structure of amoxicillin is shown in Figure 1.

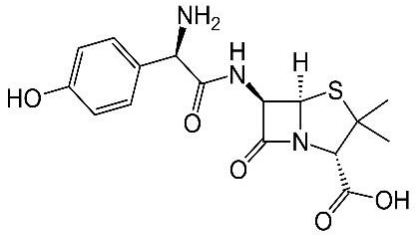

Fig. 1. Chemical structure of amoxicillin [18].

The water contaminated with 500 ppm of amoxicillin was prepared by dissolving 0.5 g of amoxicillin powder in 1 L of deionized water. Hence, the different concentrations of the solutions were obtained by diluting this solution. The granular activated carbon (GAC) used in this study was provided by Scharlau (Spain). Five different average particle sizes of 125, 250, 400, 600, and 840 µm of GAC were prepared by crushing and sieving. In all the experiments, the concentration of amoxicillin was determined using a UV-vis spectrophotometer (Jenway-6705, UK) at the wavelength of 230 nm.

2.2. Kinetic experiments

The kinetic experiments were carried out by stirring 1000 mL of the synthetic amoxicillin solution with the initial concentration of 100 ppm in contact with 0.2 g of fine granular activated carbon (average diameter < 125 µm) using a magnetic stirrer at a speed of 500 rpm. At different time intervals, a sample of 2 mL was drawn, filtered, and then analyzed by the UV-vis spectrophotometer to determine its concentration. For all the experiments, the temperature was kept at 25±2 °C.

2.3. Adsorption equilibrium experiments

In this section, the adsorption isotherms, the effect of solution pH, and the influence of adsorbent particle size on the adsorption of amoxicillin onto the powder activated carbon (PAC) were investigated. Each case followed a different procedure, which is discussed below. To perform the adsorption equilibrium experiments in a batch system, 50 mL of amoxicillin solution, ranging from 30 to 100 ppm (by 10 ppm steps), was placed in contact with 10 mg of the adsorbent in a beaker. The pH of the solution in all the experiments was adjusted to 7 by the addition of trace amounts of NaOH or HCl. The suspensions were then stirred on a magnetic stirrer at 500 rpm for 20 h at 298 K. After achieving adsorption equilibrium, the suspensions were filtered through a 0.45 µm filter, and finally, the equilibrium concentration of the solution was determined. The schematic presentation of the described system is shown in Figure 2(a). In addition, 10 mg of PAC was added to 50 mL of amoxicillin solution with a fixed concentration of 100 ppm to evaluate the effect of pH on the adsorption of amoxicillin; six different pH levels, ranging from 2 to 8, were prepared by the addition of a trace amount of HCl or NaOH. The above-mentioned procedure was followed to gather the equilibrium data for these six samples. Furthermore, to investigate the effect of the particle size on the adsorption efficiency and the equilibrium time, the adsorbent pellets were chosen in three different diameters of 125, 250, 600 µm. The initial concentrations, adsorbent dosages, and the conditions of operation were the same as the previous section, and the solution pH was adjusted to 7.

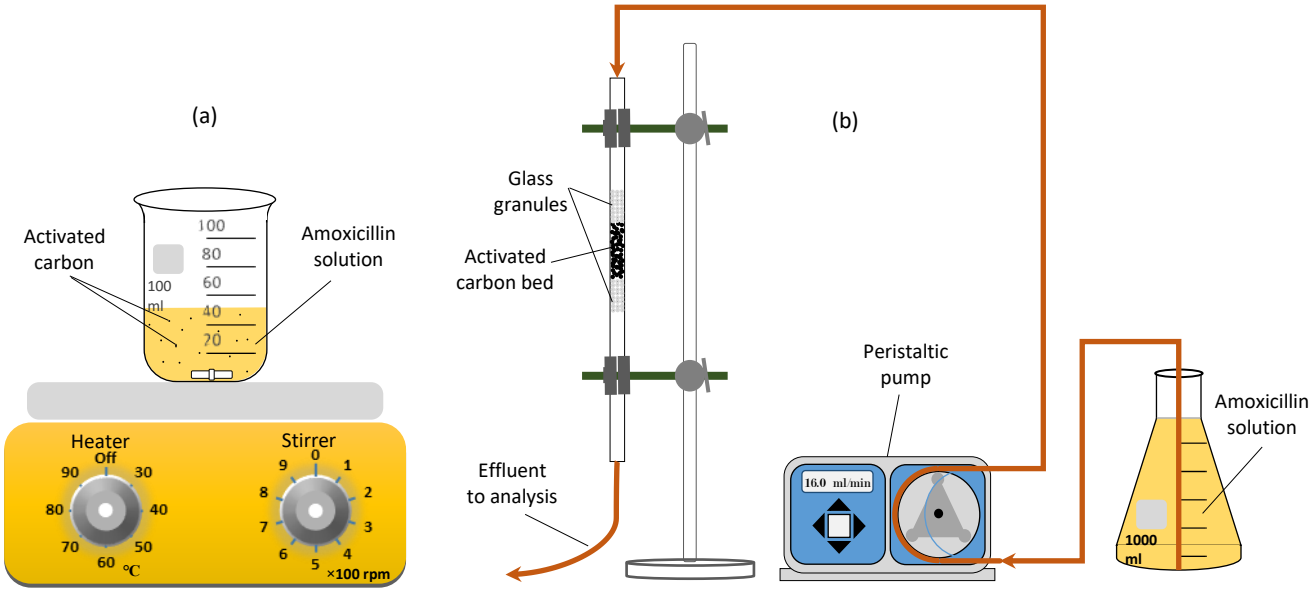

Fig. 2. Schematic representation of the experimental setup for the adsorption system: (a) batch and (b) fixed-bed.

2.4. Fixed-bed adsorption

A fixed-bed adsorption system was set up to explore the effect of process parameters such as particle size, breakthrough time, and bed length, the adsorption column used in this study had a length of 30 cm and an inner diameter of 12 mm. The column was packed with the granular activated carbon, while a certain amount of glass granules were placed at the bottom of the column to serve as support material and prevent any discharge of the adsorbent. Also, a layer of glass granules was placed on top of the adsorbent bed to act as a distributor and to avoid channeling. The influent was pumped to the column using a peristaltic pump. Figure 2(b) shows the schematic of the experimental setup. The fixed-bed experiments were conducted to investigate the effect of initial concentration, bed length, adsorbent particle size, and effluent flow rate on breakthrough curves. The contaminated solutions were prepared with initial concentrations of 100, 200, and 300 ppm to study the impact of the initial concentration of amoxicillin in a column adsorption process. Each solution was fed to the adsorbent bed with a 3.4 cm length that contained 1 g of adsorbent particles with the average size of 250 µm. The inflow rate was adjusted to 4 mL/min, and samples were taken at different times from the output stream; then, the effluent concentrations were determined using the UV-vis spectrophotometer. The process continued until the amoxicillin concentration at the outlet reached its initial concentration. The effect of the bed length on the performance of adsorption was evaluated by placing 1 and 2 g of adsorbent into the column to form beds with a length of 3.4 and 6.8 cm, respectively. Then, the contaminated solution with a fixed concentration of 300 ppm and the flow rate of 4 mL/min was fed to the bed, and the effluent solutions were sampled at consecutive times. To study the effect of adsorbent particle size on the performance of the column adsorption, the activated carbon particles used were three different sizes: 250, 400, and 840 µm. In all three tests, 1 g of adsorbent was placed in the column, and the initial concentration of the influent and flow rate were adjusted to 300 ppm and 4 $mL.min^{-1}$, respectively. The samples were taken at consecutive times until the effluent concentration reached its influent concentration. GAC with a diameter of 250 microns and an amount of 1 g (bed length of 3.4 cm) at the flow rates of 4 and 16 mL/min was used for evaluating the effect of the effluent flow rate.

3. Results and discussion

3.1. Adsorbent characterization

The specific surface area of the adsorbent was measured as 1082 m^2/g via BET analysis using Quanta chrome ChemBET 3000 equipment. Additionally, infrared spectroscopy analysis (FTIR) was carried out to determine the functional groups on the surface of the activated carbon. As shown in Figure 3, the sample has sharp peaks at some wave numbers. The wave number of 3753 represents the O—H bonds of the phenolic group [19]. The wave numbers of 2887, 1726, 1469, and 1112 represent the C—H bond, nitrogen-containing carbonyl group (amide), carboxyl functional group, and C—O stretching, respectively [8,20].

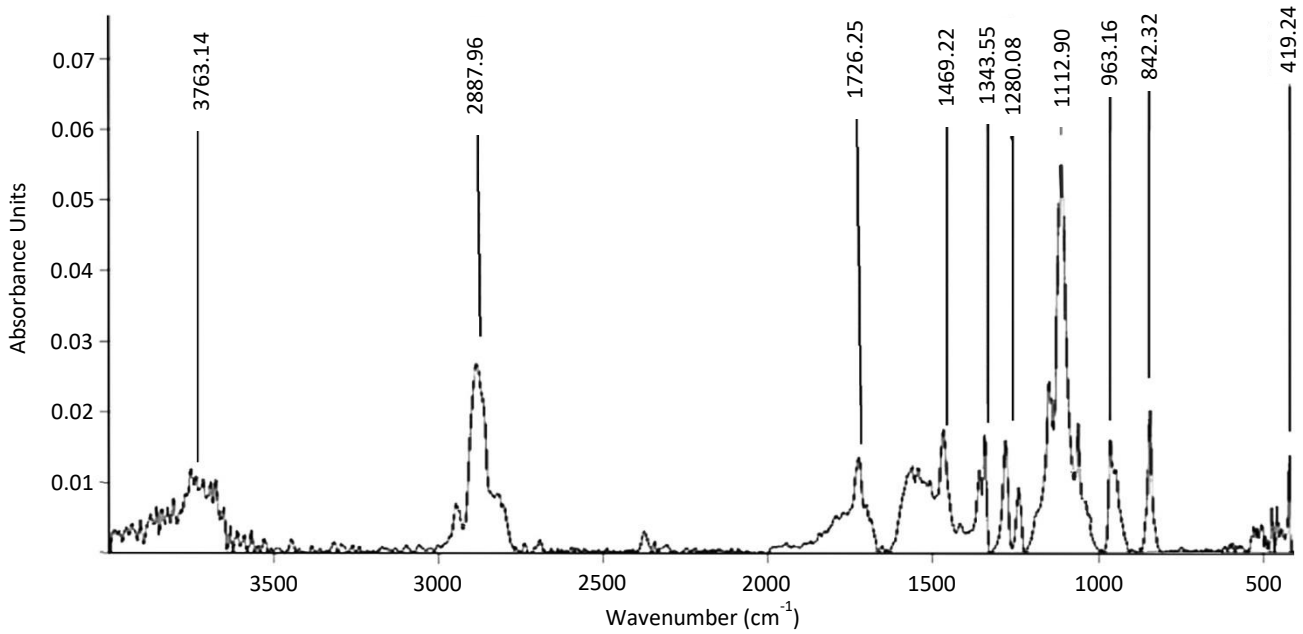

Fig. 3. FTIR analysis of the used activated carbon.

3.2. Batch adsorption results

3.2.1. Adsorption equilibrium and kinetics

After achieving equilibrium, the adsorbed amount (q_e) was calculated by Equation (1):

$$q_e = \frac{V(C_0 - C_e)}{m} \quad (1)$$

where V is the volume of the solution, C_0 and C_e are the initial and equilibrium concentrations of adsorbate, respectively, and m represents the mass of the adsorbent.

Table 1. Equations and parameters of adsorption equilibrium isotherms [21].

Isotherm name /Equations	Parameter	Value
Langmuir	q_0	312.5
$q_e = q_0 \frac{K_l C_e}{1 + K_l C_e}$	K_l	0.08
$R_l = \frac{1}{1 + K_l C_0}$	R_L	0.29
	R^2	0.996
Freundlich	K_f	53.01
$q_e = K_f C_e^{1/n}$	1/n	0.412
	R^2	0.942
Dubinin-Radushkevich	q_m	234.4
$\ln q_e = \ln q_m - B\varepsilon^2$	B	9×10^{-6}
$\varepsilon = RT\ln(1+1/C_e)$		
$E_a = \sqrt{1/2B}$	E_a	235.7

q_0: Max. amount of adsorption, K_l: Langmuir constant, R_L: Separation factor, K_f: Adsorption constant, $1/n$: Adsorption intensity, q_m: Amount of adsorbed feed, B: Dubinin-Radushkevich constant, and E_a: Mean adsorption energy.

The Langmuir, Freundlich, and Dubinin-Radushkevich (D-R) isotherm models were employed to fit the experimental equilibrium adsorption data [21]. The equations and parameters of the adsorption isotherms are given in Table 1. Moreover, Figure 4 shows a comparison of the experimental data with the studied isotherms. Based on the comparison of the R^2 values, the Langmuir model may be the most suitable isotherm model for the regression of the amoxicillin adsorption data. Three conventional models were applied to investigate the kinetic of adsorption of amoxicillin onto PAC: pseudo-first-order, pseudo-second-order, and intra-particle diffusion [22]. The models were fitted to the experimental data by the linearization of equations, and the parameters of each model were determined. According to the results presented in Table 2 and Figure 5, the pseudo-second-order kinetic model best describes the adsorption of amoxicillin onto activated carbon.

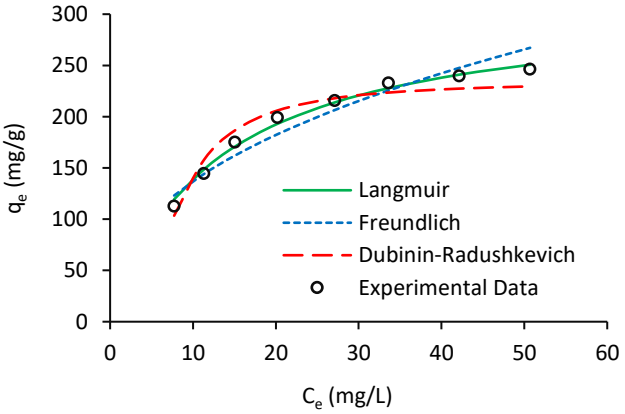

Fig. 4. Comparison of different isotherms for adsorption of amoxicillin onto PAC at 298 K.

Table 2. Equations and parameters of kinetic models [22].

Kinetic model / Equation	Parameter	Value
Pseudo-first-order $\log(q_{e1}-q_t)=\log q_{e1}-k_1 t$	k_1	409×10^{-4}
	q_{e1}	216
	R^2	0.978
Pseudo-second-order $\frac{t}{q_t}=\frac{1}{k_2 q_{e2}^2}+\frac{1}{q_{e2}}t$	k_2	209×10^{-6}
	q_{e2}	249.3
	R^2	0.999
Intra-particle diffusion $q_t=K_{id}t^{1/2}+C$	K_{id}	11.93
	C	82.84
	R^2	0.803

k_1: first order rate constant, k_2: second order rate constant, q_t: adsorbed amount at time t, q_{e1}: first order equilibrium amount q_{e2}: second order equilibrium amount, K_{id}: intra-particle rate

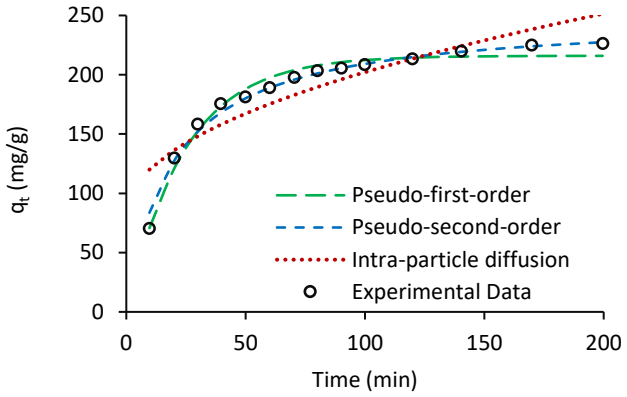

Fig. 5. Comparison of the experimental data with different kinetic models for the adsorption of amoxicillin onto PAC at 298 K.

3.2.2. The effect of pH

Figure 6 presents the effect of pH on the equilibrium adsorption of amoxicillin on activated carbon under a certain condition. It is clear that by increasing the pH value from 2 to 5, the amount of equilibrium adsorption of amoxicillin changes from 197 to 272 mg/g, but with a further increase in pH from 5 to 8, the equilibrium adsorption capacity decreases to 176 mg/g. The maximum adsorption occurs at a pH = 5, and this is the optimum pH for the adsorption of amoxicillin onto the activated carbon. This phenomenon can be explained by the fact that an amoxicillin molecule can get a positive or negative charge, or it can be neutral based on the pH of the solution, and the adsorption of amoxicillin is affected by this factor. Amoxicillin has three different functional groups: carboxyl functional group with pK_a = 2.4, amine functional group with pK_a = 4.7 and phenol functional group with pK_a = 9.6 [14,23]. Also, activated carbon has a positive surface charge in pH < pH_{pzc} and a negative surface charge in pH > pH_{pzc}. The pH_{pzc} refers to the pH that the surface has no net electric charge. The pH_{pzc} of the present activated carbon is calculated to be 4.8 by the pH titration procedure [24]. At a pH < 5, the carboxyl functional group (-COOH) in the amoxicillin group is converted to carboxylate ($-COO^-$). Accordingly, adsorption occurs due to electrostatic forces between the amoxicillin molecules (anion) and the activated carbon surface (cation). However, at pH > 5, adsorption decreases due to the increase of OH^- ions in the solution and competition between the hydroxide and carboxylate ions.

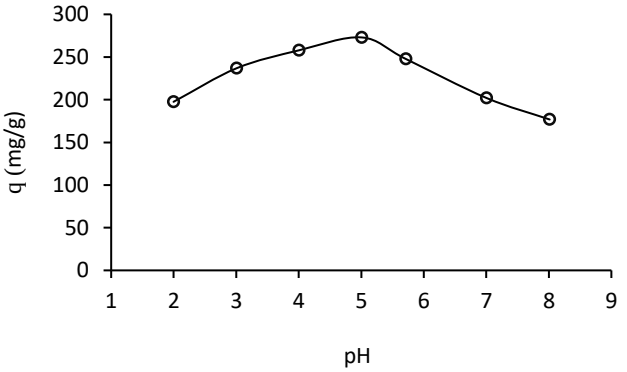

Fig. 6. The effect of pH on adsorption amount with the initial concentration of 100 ppm, the adsorbent concentration of 0.2 g/L, and the particle diameter of 125 microns.

3.2.3. Particle size effect

In industrial applications, the adsorbent is used in a granular form to reduce the pressure drop in the adsorption column. For a granular adsorbent, intra-particle diffusion cannot be negligible because of considerable diffusion paths inside the particle. Therefore, evaluating the effect of the adsorbent particle size on the adsorption rate and process efficiency is an important subject. Figure 7 illustrates the adsorption trend at different particle sizes as time progresses. As can be seen in Figure 7, by decreasing the particle size, the adsorption occurs at a faster rate such that with a decrease in the particle size from 600 to 125 µm, the adsorbed amount increases from 102 to 225 mg/g. In other words, the rate of adsorption is higher for particles with a smaller size, and equilibrium can be achieved faster. In fact, by increasing the particle size, the total length of the diffusion path inside the pores of the adsorbent particle increases and under these conditions, the adsorption process occurs more slowly. It can be concluded that the particle size is a very effective parameter in the design of such adsorption units and cannot be ignored.

3.3. Fixed-bed adsorption results

3.3.1. The effect of initial concentration

Three different concentrations were analyzed to investigate the effect of the initial concentration of amoxicillin. Figure 8 shows the breakthrough curves for three different initial concentrations. As is shown in this

figure, by increasing the initial concentration from 100 to 300 ppm, the breakpoint time is reduced from 140 minutes to 51 minutes, and the saturation time of the bed decreases from 8.24 to 7.10 hours. It should be noted that the breakpoint time and saturation time refer to the times that C/C_0 reaches 0.05 and 0.95, respectively. Furthermore, Figure 8 demonstrates that the curves are steep at the beginning and have long tails to reach saturation. This development can be explained by the fact that the solid located over the mass transfer zone front is almost free of adsorbate, which causes a high mass transfer rate while the long tails occur due to slow intra-particle diffusion; this trend of breakpoint is typical for the case of intra-particle diffusion control. A similar trend is reported by Sotelo et al. [25] for the adsorption of caffeine from the aqueous solution onto activated carbon.

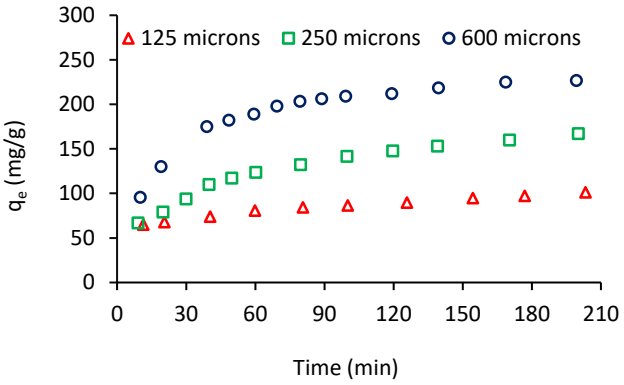

Fig. 7. The effect of particle size on the adsorbed amount at different times with the initial concentration of 100 ppm, adsorbent concentration of 0.2 g/L.

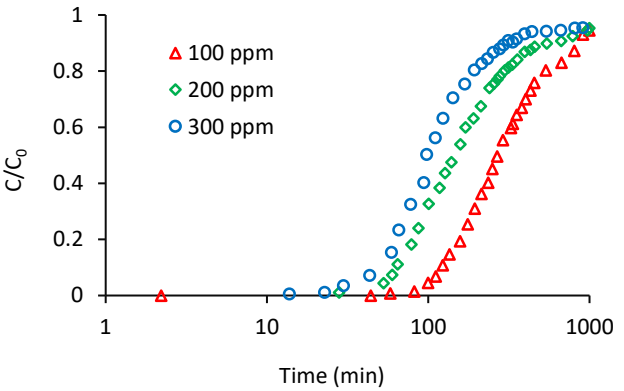

Fig. 8. The effect of initial concentration on the breakthrough curves (particle diameter = 250 µm, bed length = 3.4 cm, flow rate = 4 mL/min).

3.3.2. Bed length effect

The bed length is one of the essential parameters in the adsorption in fixed-bed systems. Two different bed lengths were chosen to study the influence of the bed length. It is clear from Figure 9 that by reducing the length of the bed from 6.8 to 3.4 cm, the breakpoint time decreases from 3.2h to 54 minutes; also, the saturation time is reduced from 14.4 to 12.1 hours. It is worth mentioning that for a system with favorable isotherms like the present system (see Figure 4), the concentration profile in the mass transfer zone acquires a characteristic shape and does not change as the mass transfer zone moves down to the end of the bed [24]. Thus, the breakthrough shapes in Figure 9 are almost identical, as it was expected.

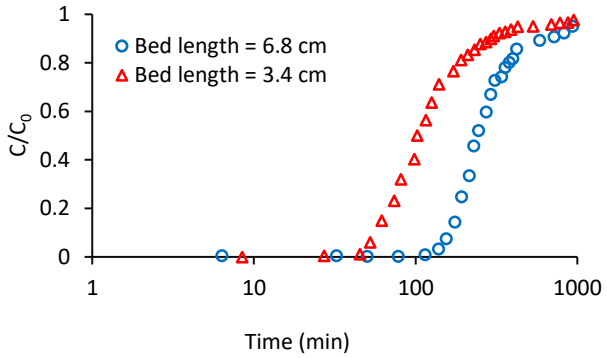

Fig. 9. The effect of bed length on the breakthrough curves (particle diameter = 250 µm, initial concentration = 300 ppm, and flow rate = 4 mL/min).

3.3.3. The effect of particle size

As mentioned in section 2.4, three different particle diameters of GAC were used to investigate the effect of particle size on the adsorption process in the fixed-bed system. Figure 10 shows the breakthrough curves for three different adsorbent particles. It can be observed that the breakthrough curves become wider with increasing the particle size, and thus the breakpoint time is reduced. Consequently, by increasing the adsorbent particle diameter from 250 to 840 µm, the breakpoint time is reduced from 55 minutes to 2 minutes. In general, by increasing the particle size, the diffusion path for the adsorbate into the adsorbent particle increases and causes higher internal mass transfer resistance inside the particle, i.e., the mass transfer coefficient decreases.

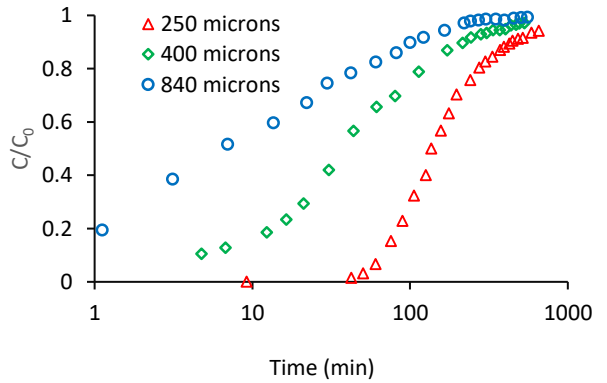

Fig. 10. The effect of particle diameter on the breakthrough curves (initial concentration = 300 ppm, bed length = 3.4 cm, and flow rate = 4 mL/min).

3.3.4. The effect of effluent flow rate

The flow rate of effluent is also one of the important parameters in the efficiency of the adsorption column. Figure 11 presents the breakthrough curves for two different flow rates: 4 and 32 mL/min. There is a decrease in the breakpoint time and saturation time of the bed from 140 to 59 min and 24.8 to 8.5 h, respectively. The justification for this observation is that by increasing the flow rate, the external mass transfer coefficient and the overall mass transfer coefficient are raised, and consequently, the adsorption rate increases. So the adsorbent is saturated earlier, and then the breakthrough and bed saturation occurs in less time for a higher flow rate.

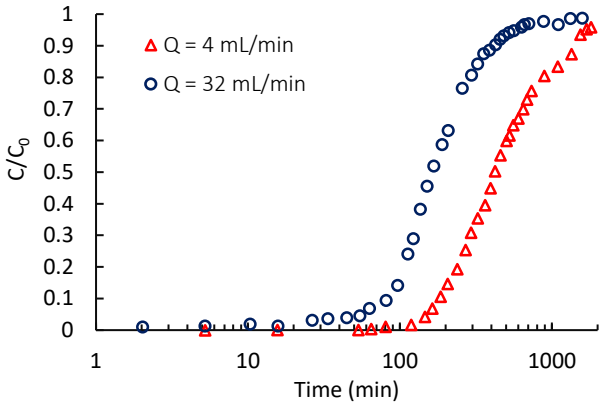

Fig. 11. Breakthrough curves for two different flow rates of 4 and 16 mL/min (initial concentration = 300 ppm, particle diameter = 250 µm, and bed length = 3.4 cm).

4. Conclusions

In this study, the adsorption process of amoxicillin from aqueous medium onto activated carbon in batch and fixed-bed models was investigated. In the batch mode, the equilibrium and kinetic parameters were calculated for the system, and the effect of some parameters like pH and particle size were examined as well. Isotherm studies showed that the Langmuir model was an appropriate model for describing the adsorption equilibrium. Furthermore, the kinetics of adsorption fit the pseudo-second-order model better than the pseudo-first-order and intra-particle diffusion models. The result showed that the highest adsorption amount occurred at pH = 5. Furthermore, decreasing the particle size increased the adsorption rate. The shapes of the breakthrough curves obtained by fixed-bed adsorption indicated that the controlling mechanism of mass transfer was the intra-particle diffusion. In addition, the effects of initial concentration, bed length, and particle size in the fixed-bed operation were evaluated. The results demonstrated that the increase of initial concentration and particle size and the decrease in bed length, decreased the breakpoint time.

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