

Studies of Adsorption Equilibria and Kinetics of Amoxicillin from Simulated Wastewater using Activated Carbon and Natural Bentonite

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Abstract

Activated carbon and natural bentonite, which is available abundant in Pacitan, East Java, Indonesia, have been used as adsorbents to remove amoxicillin from its synthetic waste. The aim of this study was to investigate the potential use of both activated carbon and natural bentonite to adsorb amoxicillin. Equilibrium and kinetic of amoxicillin adsorption onto activated carbon and natural bentonite have been determined in batch studies. Isotherm adsorption experiments were carried out at three different temperatures, i.e 303.15, 313.15 and 323.15 K. Langmuir and Freundlich isotherm models were used to describe the equilibrium data. Langmuir model fits the experimental data well for both adsorbents. The pseudo-first-order, pseudo-second-order and intra-particle diffusion models were used to evaluate the adsorption kinetics. The adsorption kinetic data used natural bentonite fitted well with pseudo-first-order, and activated carbon with pseudo-second-order. Intra-particle diffusion involves in the adsorption mechanism, but it was not the rate-controlling step.

Keywords: Amoxicillin, kinetic, isotherm, adsorption, activated carbon, bentonite.

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1. Introduction

Antibiotics are specially designed to control bacteria in humans and animals and are routinely used as human and veterinary medicine [1]. Amoxicillin is one of the most important commercial antibiotic due to its high bacteria resistance and large spectrum against a wide variety of microorganisms. It consists of two fundamental parts that contain β -lactame inner and the side chain called D-hydroxyphenylglycine [2]. Amoxicillin wastes cause unpleasant odor, skin disorder, and may cause microbial resistance among pathogen organisms or the death of microorganism which are effective in wastewater treatment. The resistant bacteria may cause disease that cannot be treated by conventional antibiotics [1]. For those reasons, amoxicillin waste need to be treated before disposed to the environment.

There are many methods for the removal of amoxicillin from sewage water, such as membrane process, ion exchange, biological degradation, and adsorption using various kind of adsorbents [1,3,4]. Adsorption process is proven to be an effective process for the removal of various pollutants from its aqueous solutions because adsorption process can remove pollutant in wide range of concentrations [5,6]. Adsorption processes involve the movement of adsorbate molecules in aqueous solution toward active sites in adsorbent pores, so they may be controlled by mass transfer effects (external and intraparticle diffusion) and the rate of adsorption on the active sites of the adsorbent [5]. To improve the efficiency of the adsorption processes, it is essential to develop the more effective and cheaper adsorbents with higher adsorption capacities [7,8].

Activated carbon is known as the most widely and effectively used adsorbent since it has high adsorption

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capacity [9,10]. The adsorption capacity of activated carbon depends not only on its surface area, but also on its internal pore structure, surface characteristic, and the presence of functional groups on pore surface. Internal pore structure and surface characteristic play an important role in adsorption processes [10]. Due to the relatively high cost of activated carbons there have been attempt to utilize low cost adsorbents.

Natural clay, such as bentonite, may be an alternative adsorbent because of their abundance in most continents of the world and its low cost [9,11]. Bentonite is mainly composed of montmorillonite, consists of layers of two tetrahedral silica sheets sandwiching one octahedral alumina sheet. Due to the isomorphous substitution of the silicon ions by aluminum or ferric cations in the tetrahedral sheets, and the aluminium ions by magnesium or ferrous cations in the octahedral sheets, bentonite has net negative charges on its layer lattice. The chemical nature and pore structure of bentonites generally determine their sorption ability [11,12].

In this study, activated carbon (supplied by PT. Lautan Luas, Tbk.) and natural bentonite from Pacitan, East Java, Indonesia, were used as adsorbents. The aim of this study was to investigate the potential use of both activated carbon and natural bentonite to adsorb amoxicillin. Laboratory batch kinetic and isotherm studies were conducted to evaluate the adsorption capacity of both adsorbents. The effects of temperature and particle size of the adsorbent were also studied.

2. Experimental

2.1. Material

Adsorbent. Adsorbents used in this study were activated carbon and natural bentonite. Activated carbon obtained from PT. Lautan Luas Tbk., the characteristics of this carbon were provided by the manufacturer (Table 1). The bentonite used in this study was analyzed using Atomic Absorption Flame Emission Spectrophotometer (Shimadzu AA-6200) and the result can be seen in Table 2. This processed form bentonite was supplied by CV. Mitra Buana Perkasa Indonesia and used without any further treatment.. Both adsorbents were first dried and sieved to obtain particle sizes of 100, 120 and 140 mesh, then washed with distilled water several times to remove water-soluble impurities. The washed samples were dried in a vacuum drying oven at 378 K and 2 in Hg vacuum for 24 hours and then placed in a desiccator for further use in the adsorption experiments.

Adsorbate. Amoxicillin trihydrate was supplied by PT. Coronet Crown Indonesia It has a molecular weight of 419.46. This material was directly used without any further treatment.

2.2. Experiment

2.2.1. Adsorption equilibrium

A preliminary adsorption experiment was conducted to determine the time required to reach equilibrium condition using bentonite and activated carbon as adsorbents. Samples were taken every certain time interval and the concentration of amoxicillin was analyzed using spectrophotometer (GENESYS 10V). The product analysis showed that equilibrium conditions were reached in 4 hours for bentonite and 12 hours for activated carbon. Adsorption equilibrium studies were conducted using static technique. Equilibrium data were obtained by adding 0.01-1.7 g of bentonite into a series of 100 mL conical flasks each filled with 50 mL of amoxicillin solution with initial concentration of 300 mg/L. The conical flasks were then placed in a thermostatic shaker bath (Mettler Type WB-14 equipped with a SV 1422 temperature controller) and were shaken at 120 rpm for 4 hours. The above procedure was then repeated using activated carbon as an adsorbent. The mixture of activated carbon and amoxicillin solutions were shaken for 12 hours. During adsorption studies the system were kept constant at three different temperatures (303.15, 313.15 and 323.15 K).

After equilibrium time had reached, the solutions were centrifuged (MLW T.51.1) at 2500 rpm for 1 min, and the clarified supernatant solutions were carefully decanted and analyzed for amoxicillin concentration.

The amount of amoxicillin adsorbed at equilibrium is calculated based on the following equation:

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

where q_e (mg/g) is the amount of amoxicillin adsorbed in adsorbent at equilibrium, C_o and C_e (mg/L) are the initial and equilibrium concentration of amoxicillin solutions, respectively, m (g) is the amount of adsorbent, V (L) is the volume of solution.

2.2.2. Adsorption kinetics

Adsorption kinetic study was carried out in order to test the relationship between contact time and amoxicillin uptake. A series of 250 mL amoxicillin solution with concentration of 300 mg/L was filled into three-necks round bottom flasks. The adsorption kinetic experiments were conducted in a thermostatic shaker bath at 303.15 K and 120 rpm. Adsorbents (bentonite or activated carbon), with different particle sizes (100, 120, and 140 mesh) were added to the amoxicillin solutions. During the experiment, the aqueous samples were periodically taken at certain time intervals, and the concentration of amoxicillin solutions were analyzed. using spectrophotometer (GENESYS 10V).

Description	
Raw material	Coconut shell
Specific surface area	1,200 m ² /g
Bulk density	0.5 g/cm ³
Moisture content	5 %
Ash content	5 %
Iodine number	1,000 mg/g
pH value	9

Parameter	Composition %
SiO ₂	47.8965
Al ₂ O ₃	39.119
Fe ₂ O ₃	3.44
CaO	0.15
MgO	0.45
K ₂ O	0.24
Na ₂ O	0.47
MnO ₂	0.03
Cr ₂ O ₃	0.00
CuO	0.0045
Moisture content	8.2

3. Results and Discussion

3.1. Adsorption equilibrium studies

The analysis and design of adsorption separation processes requires the relevant adsorption equilibria, which is the most important piece of information in understanding an adsorption process [10]. It is also important for designing an adsorption system. The adsorption equilibrium indicates how the adsorbate molecules distribute between the liquid phase (solution) and the solid phase (adsorbent) when the adsorption process reaches an equilibrium state [13]. To describe the adsorption equilibrium data of amoxicillin on both adsorbents, activated carbon and bentonite, Langmuir and Freundlich isotherm models were used. All fitted parameters were obtained from optimization using Sigma Plot 9.0 software.

3.1.1. Langmuir isotherm

The Langmuir adsorption assumes that adsorption occurs at specific homogeneous sites within the adsorbent by monolayer adsorption onto a surface containing a finite number of adsorption of uniform strategies of adsorption with no transmigration of adsorbate in the plane of the surface [13,14]. Therefore, saturation is reached after a certain time, beyond which no further sorption can take place [8]. The Langmuir equation is applicable to homogeneous sorption, where the sorption of each sorbate molecule onto the surface has equal sorption activation energy [15].

The Langmuir equation has the following form [16]:

$$q_e = q_{\max} \frac{K_L \cdot C_e}{1 + K_L \cdot C_e} \quad (2)$$

where q_e (mg/g) is the solid phase adsorbate concentration at equilibrium, C_e (mg/L) is the equilibrium concentration of adsorbate in solution, q_{\max} (mg/g) is the maximum adsorption capacity corresponding to complete monolayer coverage on the surface and K_L (L/mg) is the Langmuir constant related to the heat of adsorption [14,16].

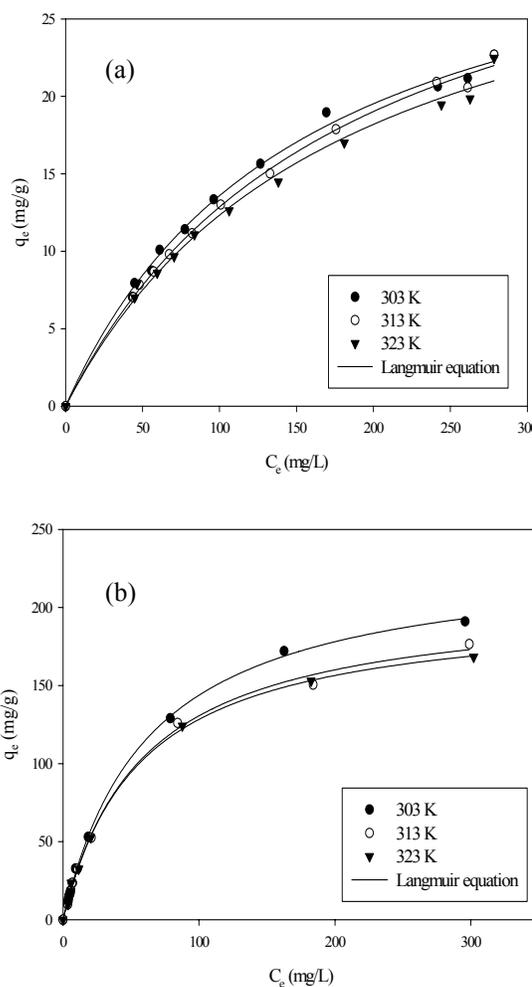


Figure 1. Langmuir adsorption isotherm of Amoxicillin using (a) Bentonite; (b) Activated Carbon.

Fig. 1 depicts the Langmuir adsorption isotherm of amoxicillin on bentonite and activated carbon. Conformation of the experimental data into Langmuir isotherm model as shown in Fig. 1 indicates the

homogeneous nature of bentonite or activated carbon surface, which means each amoxicillin molecule on bentonite or activated carbon has equal adsorption activation energy [13]. The results also demonstrate the formation of monolayer coverage of amoxicillin molecule at the outer surface of bentonite or activated carbon. The optimal parameters from the fitting of Langmuir equation with the experimental data are given in Table 3.

Temperature has an important role in adsorption, and generally it has a negative influence on the amount adsorbed [17]. The adsorption of the organic compounds is an exothermic process and the physical bonding between the organic compounds and the active sites of the adsorbent will weaken as the temperature increases. The solubility of amoxicillin increased with temperature. The interaction force between amoxicillin and water molecules become stronger than amoxicillin and adsorbent, consequently amoxicillin is more difficult to be adsorb onto the adsorbent. Both of these features are consistent with the order of Langmuir adsorption capacity as seen in Table 3.

The heat of adsorption can be estimated using van Hoff equation, which relates the Langmuir equilibrium constant K_L to the temperature:

$$K_L = K_o \exp\left(\frac{-E}{RT}\right) \quad (3)$$

where E (J/mol) is the activation energy of adsorption and K_o (L/mg) is the adsorption equilibrium coefficient. The gas constant R is equal to 8.314 J/mol K and T (K) is the temperature of the solution. The activation energy indicates the type of adsorption. Activation energy which is ranged between 5-40 kJ/mol corresponds to a physisorption mechanism and 40-800 kJ/mol suggests a chemisorption mechanism [10]. The relation between the Langmuir equilibrium constant and $1/T$ is given in Fig. 2. The value of K_o and E obtained from eq. (3) are 1.9627 L/g and 15.3953 kJ/mol for bentonite ($R^2 = 0.9989$), and 0.097 L/g and 5.5094 kJ/mol for activated carbon ($R^2 = 0.9992$). The value of E obtained in this study indicating that the adsorption of amoxicillin using bentonite and activated carbon are assigned to a physisorption.

The essential characteristics of the Langmuir equation can also be expressed in term of a dimensionless separation factor, R_L , defined as [16]:

$$R_L = \frac{1}{1 + K_L.C_o} \quad (4)$$

where C_o (mg/L) is the highest initial solute concentration and K_L (L/mg) is the Langmuir constant. The value of R_L (0.155-0.4398) were in the range of 0-1 at all temperatures studied which confirm the favorable uptake of the amoxicillin (Table 4) [16].

Table 4. Parameters R_L indicated the shape of isotherm [16]

Value of R_L	Type of isotherm
$R_L > 1$	Unfavorable
$R_L = 1$	Linear
$0 < R_L < 1$	Favorable
$R_L = 0$	Irreversible

3.1.2. Freundlich isotherm

The Freundlich equation is an empirical equation, which assumes a heterogeneous adsorption surface, where the adsorption sites have different energies of adsorption and are not equally available [14,16]. This equation describes reversible adsorption and is not restricted to the formation of monolayer.

The Freundlich isotherm equation has the following form:

$$q_e = K_F.C_e^{1/n} \quad (5)$$

where K_F (mg/g)(L/mg)^{1/n} is Freundlich constant which can be defined as the adsorption or distribution coefficient and represents the adsorption capacity, the quantity of adsorbate adsorbed onto adsorbent for a unit equilibrium concentration. $1/n$ characterizes the heterogeneity of the system [10,16].

Table 3. Parameters of Langmuir Equation

Temp. (K)	Bentonite				Activated Carbon			
	q_{max} (mg/g)	K_L (L/mg)	R^2	R_L	q_{max} (mg/g)	K_L (L/mg)	R^2	R_L
303.15	44.6621	0.0044	0.9971	0.4453	233.775	0.0160	0.9994	0.1566
313.15	36.5188	0.0054	0.9960	0.3954	206.671	0.0170	0.9978	0.1487
323.15	32.6781	0.0064	0.9915	0.3556	200.431	0.0178	0.9962	0.1430

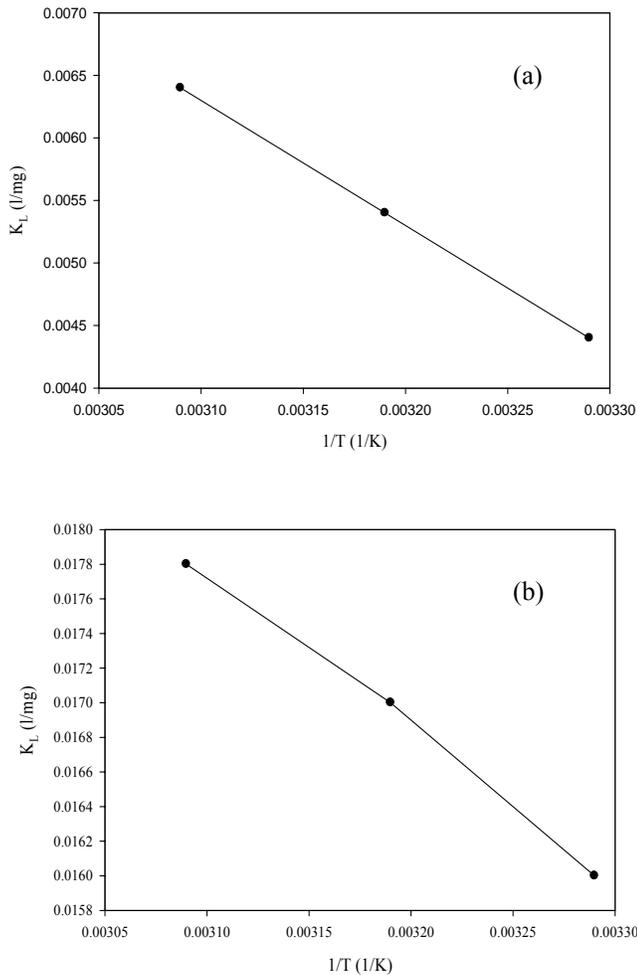


Figure 2. Plot K_L vs. $1/T$ for Amoxicillin adsorption using (a) Bentonite; (b) Activated Carbon.

The Freundlich equation predicts that the concentration of amoxicillin on bentonite or activated carbon will increase as long as there is an increased in the concentration of amoxicillin in the solution, which means that there is no maximum capacity of amoxicillin on bentonite or activated carbon. Fig. 3 depicts the Freundlich adsorption isotherm of amoxicillin on bentonite and activated carbon. The optimal parameters from the fitting of Freundlich equation with the experimental data are given in Table 5. It can be seen from Table 5 that activated carbon has higher capacity of amoxicillin adsorption according to the value of K_F . The value of $1/n$ constants from Table 5 are ranging between 0 and 1, indicate favorable adsorption

The $1/n$ value is also a measure of the adsorption intensity or surface heterogeneity, it is becoming more heterogeneous as its value gets closer to zero. A value for $1/n$ below one indicates a normal Freundlich isotherm while $1/n$ above one is indicative of cooperative adsorption [13].

By comparing Fig.1 and Fig. 3 and the value of R^2 in Table 3 and Table 5, it can be seen that Langmuir isotherm model is better in describing the adsorption of amoxicillin using activated carbon or bentonite.

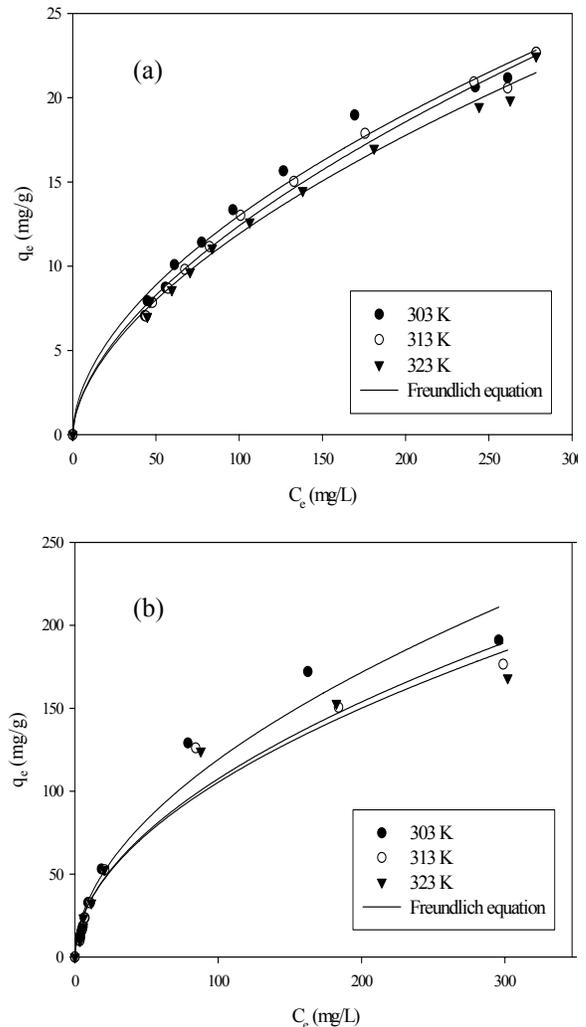


Figure 3. Freundlich adsorption isotherm of Amoxicillin using (a) Bentonite; (b) Activated Carbon.

Temperature (K)	Bentonite			Activated Carbon		
	$1/n$	K_F (mg/g).(L/mg) ^{1/n}	R^2	$1/n$	K_F (mg/g).(L/mg) ^{1/n}	R^2
303.15	0.5409	1.0334	0.9872	0.5101	10.4875	0.9658
313.15	0.5824	0.8482	0.9844	0.5173	9.9164	0.9700
323.15	0.5758	0.8404	0.9842	0.5275	9.0523	0.9653

3.2. Adsorption kinetic studies

In order to investigate the mechanism of amoxicillin adsorption and potential rate controlling steps such as mass transport and chemical reaction processes, kinetic models have been used to test experimental data. These kinetic models include the pseudo-first-order equation, pseudo-second-order equation, and intraparticle-diffusion equation.

Fig. 4 and 5 show that the value of q_e increases gradually with the decreasing of adsorbent particle size as expected because if the particle size of adsorbents (bentonite and activated carbon) decreases, more active adsorption sites are available on the surface of adsorbents particle and attract more amoxicillin to their surface [14].

3.2.1. Pseudo-first-order kinetic model

The pseudo-first-order kinetic model was derived based on the adsorption capacity at anytime. This model has been widely used and was suggested by Lagergren [10] for the adsorption of solid/liquid and its equation is given as follows:

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \quad (6)$$

where q_e and q_t (mg/g) the solid phase adsorbate concentration at equilibrium and at time t (min), respectively, and k_1 (min^{-1}) is the rate constant for the pseudo-first-order model. Integrating Eq. (6) for the boundary condition $t = 0 - t$ and $q_t = 0 - q_t$ gives:

$$q_t = q_e(1 - \exp(-k_1 t)) \quad (7)$$

The kinetic experimental data and the pseudo-first order model fit of amoxicillin adsorption on bentonite and activated carbon of different particle sizes are showed in Figure 4(a) and 5(a).

The pseudo-first order parameters k_1 and q_e were determined using Sigma Plot 9.0 software and summarized in Table 6.

3.2.2. Pseudo-second-order kinetic model

Another kinetic model derived based on the adsorption capacity at anytime is pseudo-second-order model, which has the following form [18]:

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \quad (8)$$

where k_2 ($\text{g}/\text{mg}\cdot\text{min}$) is the rate constant of pseudo-second-order model. Integrating Eq. (8) for the boundary condition $t = 0 - t$ and $q_t = 0 - q_t$ gives

$$q_t = q_e \left(\frac{q_e k_2 t}{1 + q_e k_2 t} \right) \quad (9)$$

The kinetic experimental data and the second order model fit of amoxicillin adsorption on bentonite and activated carbon of different particle sizes are showed in Figure 4(b) and 5(b). The pseudo-second order parameters and the correlation coefficient, R^2 are shown in Table 6.

From the values of R^2 of both models for bentonite in Table 6 show that pseudo-first order model gives better correlation than the pseudo-second order. Furthermore the values of q_e obtained from the pseudo-first order model agree with experimental values. This result confirms that the physisorption might be the controlling mechanism for adsorption of amoxicillin on bentonite.

The experimental data of activated carbon fits better with the pseudo-second model (Fig. 5). It can be seen from the higher R^2 and the agreement of the experimental and calculated q_e values of the pseudo-second model (Table 6). Activated carbon has several functional groups on its surface. They contributed as acid and base functional group. The adsorption mechanism might be include interaction between base functional group and amoxicillin chemically [19].

Table 6. Parameters of kinetic models for adsorption amoxicillin using bentonite and activated carbon

Adsorbent	Particle size	q_e (exp) (mg/g)	Pseudo-first order			Pseudo-second order			Intra-particle diffusion		
			k_1 (min^{-1})	q_e (mg/g)	R^2	$k_2 \cdot 10^4$ (g/mg) (min^{-1})	q_e (mg/g)	R^2	k_p ($\text{mg}\cdot\text{g}^{-1}\cdot\text{minute}^{-1/2}$)	C	R^2
Bentonite	$^{+80}/_{-100}$	6.4900	0.0198	6.5115	0.9995	27.823	7.7321	0.9854	0.7020	-0.5025	0.9841
	$^{+100}/_{-120}$	6.9700	0.0206	6.9696	0.9992	26.707	8.4103	0.9863	0.6712	-0.3781	0.9843
	$^{+120}/_{-140}$	7.2700	0.0223	7.2632	0.9990	25.085	8.8010	0.9866	0.6197	-0.1374	0.9836
Activated carbon	$^{+80}/_{-100}$	24.890	0.0052	23.0104	0.9799	2.5042	24.7580	0.9931	1.2861	0.0100	0.9999
	$^{+100}/_{-120}$	25.920	0.0054	24.3051	0.9799	2.0579	25.7547	0.9928	0.8389	4.8736	0.9827
	$^{+120}/_{-140}$	26.690	0.0060	25.3785	0.9801	1.7435	26.9574	0.9927	0.5588	10.3370	0.9768

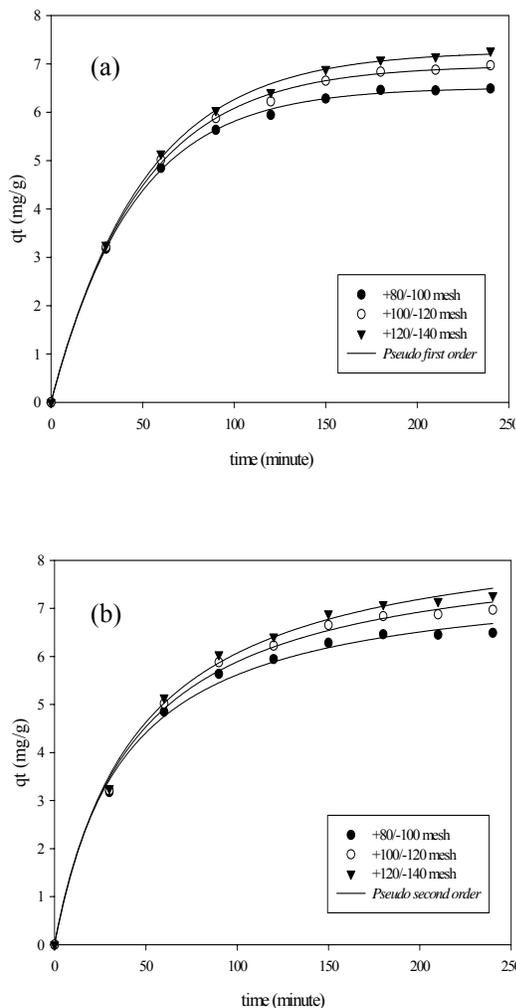


Figure 4. Amoxicillin kinetic adsorption using bentonite (a) pseudo-first-order; (b) pseudo-second-order.

3.2.3. Intraparticle diffusion

The kinetic data can also be analyzed by an intra-particle diffusion kinetic model, formulated as [12]:

$$q_t = k_p t^{1/2} + C \quad (10)$$

where k_p ($\text{mg.g}^{-1}.\text{minute}^{-1/2}$) is the intra-particle diffusion rate constant and C is the intercept of the plot of q_t versus $t^{1/2}$. If this linear plot passes through the origin, then intraparticle diffusion is the rate-controlling step.

The plots of intra-particle diffusion do not pass through the origin. They present multilinearity, indicating three steps take place, as shown in Fig. 6 and 7.

At the first, sharper portion may be considered as an external surface adsorption or faster adsorption stage, the second portion describes the gradual adsorption stage, where the intra-particle diffusion is rate-controlled. The third portion is attributed to the final equilibrium stage, where intra-particle diffusion starts to slow down due to

the extremely low adsorbate concentrations in the solution [12]. The gradual adsorption may be controlled by intra-particle diffusion. Straight lines as shown in Fig. 6 and 7 indicate that intra-particle diffusion is involved in the amoxicillin adsorption using both bentonite and activated carbon, but it was not the rate-controlling-step [4,12,16].

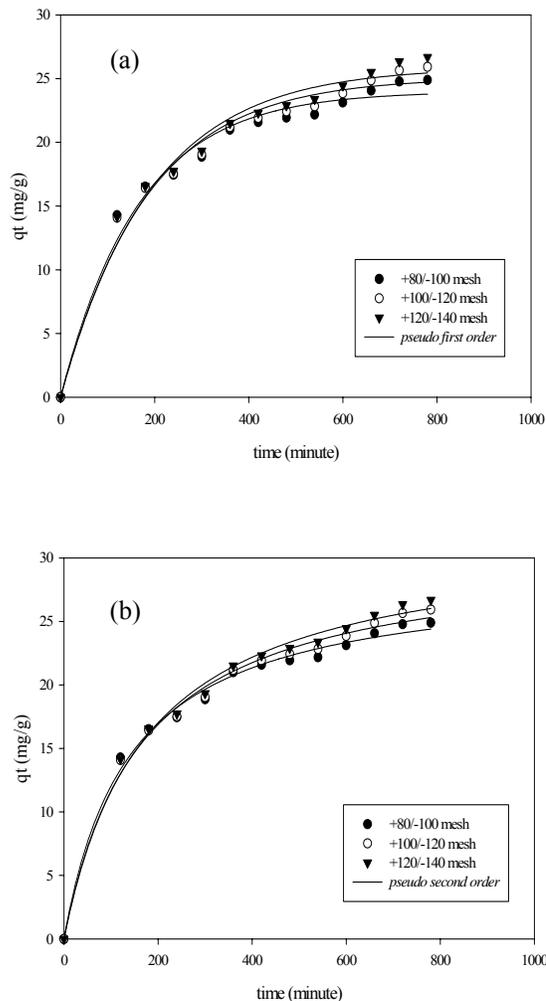


Figure 5. Amoxicillin kinetic adsorption using activated carbon (a) pseudo-first-order; (b) pseudo-second-order.

At the first adsorption step, there is no significant difference in amoxicillin concentration for the three particle sizes because amoxicillin diffuses from bulk solution into adsorbents surface. Fig. 6 shows that bentonite particle size affects adsorption mechanism in the second and third portions of the intra-particle diffusion model, which indicates that physisorption plays an important role in the amoxicillin adsorption on bentonite. While amoxicillin adsorption onto activated carbon includes the interaction between the base group at its surface and amoxicillin so the influence of particle size is more at

third portion of intra-particle diffusion model as seen in Fig. 7.

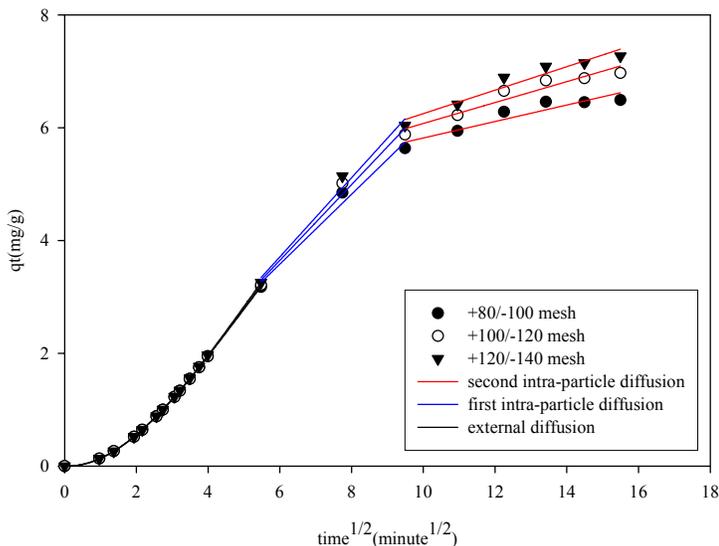


Figure 6. Amoxicillin kinetic adsorption using bentonite with intra-particle model.

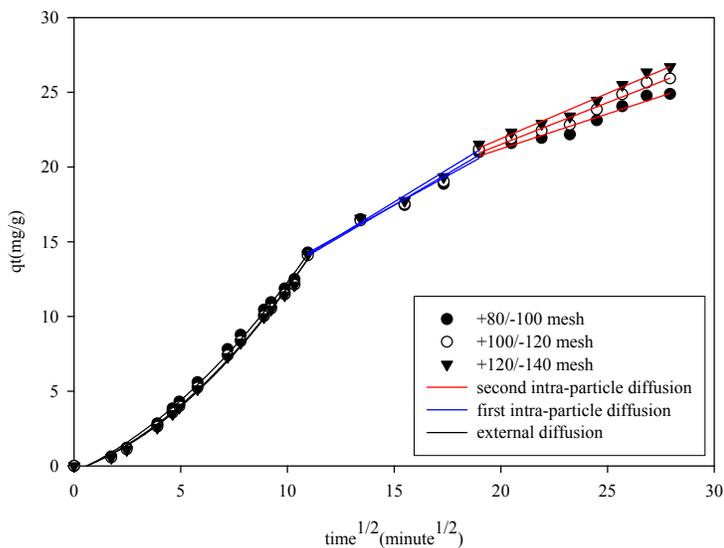


Figure 7. Amoxicillin kinetic adsorption using activated carbon with intra-particle model.

4. Conclusions

Amoxicillin can be removed by adsorption using natural bentonite and activated carbon. The equilibrium data for both bentonite and activated carbon followed Langmuir

model. Activated carbon has higher adsorption capacity than bentonite. The adsorption kinetics of amoxicillin using bentonite can be well described by pseudo-first-order, while activated carbon by pseudo-second-order. It is also found that the kinetic of sorption process also fitted well intraparticle diffusion model, but diffusion was not the only rate-controlling step.

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