Boron Removal from Aqueous Solution Using Curcumin-impregnated Activated Carbon
(Penyingkiran Boron daripada Larutan Akuas Menggunakan Karbon Teraktif Terkandung Kurkumin)

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ABSTRACT

This study was conducted to investigate the batch and fixed-bed adsorption properties of boron on curcumin-impregnated activated carbon (Cur-AC). The maximum boron removal was obtained at pH5.5 and 120 min of contact time. Langmuir and Freundlich isotherm models were applied and it was determined that the experimental data conformed to both models. The Langmuir maximum adsorption capacities for Cur-AC (5.00 mg/g) and regenerated Cur-AC (3.61 mg/g) were obviously higher than the capacity for bare activated carbon (0.59 mg/g). Kinetic studies indicated the adsorption of boron conformed to the intra-particle model. The highest boron removal in fixed-bed column adsorption was achieved up to 99% for the first 5 min at an inlet concentration of 890 mg/L and a flow rate of 8.0 mL/min. Thomas and the Yoon-Nelson models gave better fit to the experimental data. Cur-AC can be reused after elution processes with slightly lower adsorption capacity.

Keywords: Adsorption; boron; curcumin; fixed bed column; turmeric

ABSTRAK

Kajian ini telah dijalankan untuk menentukan ciri-ciri penjerapan boron secara kelompok dan secara turus lapisan tetap terhadap karbon teraktif terubahsuai dengan kurkumin (Cur-AC). Penyingkiran maksimum boron telah diperoleh pada pH5.5 dan masa sentuhan selama 120 min. Hasil kajian ini mendapati bahawa data uji kaji ini telah mematuhi kedua-dua model isoterma penjerapan Langmuir dan Freundlich. Kapasiti penjerapan maksimum Langmuir untuk Cur-AC (5.00 mg/g) dan Cur-AC regenerasi (3.61 mg/g) adalah lebih tinggi berbanding kapasiti penjerapan bagi karbon teraktif yang tidak diubah suai (0.59 mg/g). Kinetik kajian menunjukkan penjerapan boron mematuhi model intra-partikel. Penyingkiran boron secara turus penjerapan telah mencapai sehingga 99% untuk 5 min pertama (kepekatan influen 890 mg/L dan kadar aliran sebanyak 8.0 mL/min). Hasil kajian menunjukkan data uji kaji lebih mematuhi Model Thomas dan Yoon-Nelson dan Cur-AC boleh digunakan semula selepas proses regenerasi dengan kapasiti penjerapan yang lebih rendah.

Kata kunci: Boron; kunyit; kurkumin; penjerapan; turus lapisan tetap

INTRODUCTION

Boron is widely distributed in the environment from natural or anthropogenic sources and can be found mainly in the form of boric acid or borate salts. The main boron sources, whose presence is detected in surface waters, are urban wastes rich in detergents and cleaning products; industrial wastes, which can come from a wide range of different activities and several chemical products used in agriculture (Halim et al. 2012). Boron has virulence for reproduction and causes disease of the nervous system. In humans, the sign of acute toxicity includes nausea, vomiting, diarrhea, dermatitis and lethargy (Cengeloglu et al. 2007, 2008). Therefore, the removal of hazardous boron from wastewater is important for environment control. The European Union has classified boron as a pollutant of drinking water in national and international drinking water directives (Polat et al. 2004). The concentration of boron in the drinking water as recommended by the World Health

Organization (WHO) should be below 0.3 mg boron/L (Halim et al. 2012). There is a legislation requirement by Malaysia Department of Environment (DOE) to reduce boron concentration to below 1 and 4 mg/L for Effluent Discharge Standards A and B, respectively (Chong et al. 2009).

Physiochemical and biological treatments have been used widely to remove boron from wastewater. There are several physicochemical treatments, including using ion exchange (Simonnot et al. 2000), adsorption (Nor Hasleda Mamat et al. 2011), reverse osmosis (Nadav et al. 2005), membrane filtration (Mottez et al. 1998), electrodialysis (Kabay et al. 2008) and electrocoagulation (Halim 2012). As stated in the literature, conventional sedimentation and biological treatment remove boron inefficiently (Bouguerra et al. 2008; Şahin 2002). Boron is classified as highly soluble in water (Yılmaz et al. 2005). Conventional and biological treatment methods are not able to remove it

from aqueous solutions; however, some high-cost methods have been devised such as ion exchange, electrodialysis and sedimentation. In literature, there are many studies carried out to remove boron from both wastewater of industries producing boron and other industries that use boron compounds. In one of these studies, boron was removed by batch adsorption methods using a carbon-mineral composite adsorbent.

Various researchers have studied the applications of surface-modified activated carbon (AC) in wastewater treatment (Çelik et al. 2008; Chen et al. 2007; Owlad et al. 2010). Surface impregnation is one of the most important chemical modifications. The impregnation optimizes the existing properties of AC, giving a synergism between the chemicals and the carbon (Chen et al. 2007). Boron removal from aqueous solutions by carbon impregnated with salicylic acid has been studied in a batch system (Çelik et al. 2008).

Curcumin-impregnated activated carbon (Cur-AC) as a new modified activated carbon is expected to overcome the difficulty of removing boron from wastewater treatment. Curcumin is an orange-yellow pigment found in the rhizome of the turmeric plant (*Curcuma longa*). It has the ability to form the orange complex with boron in aqueous conditions. It reacts with boric acid by forming a red compound known as rosocyanine (APHA et al. 2005). The aim of this study was to investigate the boron removal in aqueous solutions based on batch and column adsorption test using Cur-AC as an adsorbent.

MATERIALS AND METHODS

ADSORBENT PREPARATION

Bare-AC was prepared by using commercial coconut shell activated carbon (particle size of AC, 1.00 – 3.35 mm; surface area, 900 m^2/g). Curcumin powder was extracted from *Curcuma longa* L. or turmeric that was obtained from Merck, Germany. About 500 g of AC was shaked with 1 L of 5000 mg/L curcumin solution for 2 h at 25°C, pH 11-12 and 200 rpm of stirring rate to prepare Cur-AC. These adsorbent media were thoroughly washed with distilled water and dried in an oven at 60°C for 24 h.

BATCH ADSORPTION EXPERIMENT

Optimum conditions of the batch adsorption experiment were determined using 2 g of AC and 50 mL of model boron solution in 250 mL conical flask and sealed with Parafilm. Model boron solution was prepared from boric acid at 1000 mg/L concentration. The batch experiment was conducted at an ambient temperature and the optimum conditions of all pertinent factors, such as pH, agitation speed and contact time were determined. The effects of pH on boron removal efficiency were studied by varying the pH of solution from 2 to 13 using a concentrated sulfuric acid and sodium hydroxide solution. The batch experiment was performed using an orbital shaker (Protech, model 719) at a constant shaking speed of 200 rpm. Finally, these solutions were filtered using filter paper with a 0.45 µm pore size and the boron concentration was determined by carmine methods (APHA et al. 2005). In this method, boron was determined by its reaction with carminic acid in the presence of sulfuric acid to produce reddish to bluish color. The amount of color is directly proportional to the boron concentration. Test results are measured using spectrophotometer at 605 nm.

Adsorption isotherm tests were also carried out in the reaction mixture containing of 50 mL of boron solution (900 mg/L) by varying the mass of adsorbent (0.2 g - 15 g). A kinetic study was performed by shaking a series of 50 mL of boron solution with a known initial concentration (1200 mg/L) with 2 g of composite adsorbent in a 250 mL conical flask at room temperature, optimum pH of 5.5 and constant shaking speed of 200 rpm. The conical flask was collected at specific time intervals (5, 10, 15, 30, 45, 60, 90, 120 and 150 min) and then the sample was filtered using filter paper with a 0.45 µm pore size.

FIXED-BED ADSORPTION EXPERIMENT

A fixed-bed adsorption experiment was conducted using a column of 6.4 cm diameter and 36 cm length. The column was packed with 227.02 g of Cur-AC between two supporting layers of pre-equilibrated glass wool. The column was charged with boron synthetic wastewater in the up-flow mode at a volumetric flow rate of 8 mL/min using a Masterflex peristaltic pump. Samples were collected at various time intervals and analysed for boron using carmine methods (APHA et al. 2005). This column experiment was triplicated to obtain repeatable results.

DESORPTION

The exhausted column was subjected to desorption to regenerate the Cur-AC using an acidic elution solution (0.1 M hydrochloric acid) (Ozturk & Kavak 2005). To regenerate the column, the elution solution was pumped through the adsorbent in the up-flow mode at a volumetric flow rate of 8 mL/min. The breakthrough solution was then collected for determination of boron concentration. Finally, it was thoroughly washed with distilled water and ready to be reuse as a regenerated-AC.

RESULTS AND DISCUSSION

EFFECT OF INITIAL pH

The initial pH of wastewater plays an important role in a batch absorption experiment (Martín-Esteban et al. 1996). Wastewater acidity influences the binding sites of metal to the adsorption surfaces (Özacar & Şengil 2005). The effect of pH on the boron adsorption was investigated in order to obtain the optimum pH for maximum boron removal. The effect of pH was investigated in the range of 2.0–13.0. Figure 1 shows that the boron removal was affected by the pH. The maximum boron removal efficiency was obtained
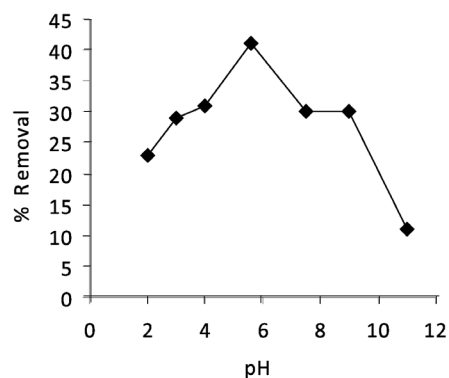
FIGURE 1. Removal of boron at different initial pH values at 25°C (initial concentration 1000 mg/L, volume of boron solution 50 mL, weight of adsorbent 2 g and shaking speed 200 rpm)

at a slightly acidic range (pH5.5). It is obviously different from previous finding that state optimum pH for maximum boron removal at a slightly alkaline range (pH8) (Chong et al. 2009). In this study, boron in borates form reacted with curcumin (in enol form) on the AC surface preferentially in an acidic solution containing hydrochloric or sulfuric acid to form a red boron-chelate complex (rosocyanine) (Spicer & Strickland 1952) as shown in Figure 2.

EFFECT OF CONTACT TIME

Kinetic rate of Cur-AC for boron removal from boron solution was carried out at pH5.5 and a temperature of 25°C (Figure 3). Stirring of activated carbon in boron solution is important because it maintains the activated carbon in suspension, offering the maximum surface to boron adsorption. Starting from the results obtained, the amount of boron adsorbed increases with contact time and attains equilibrium at 120 min for Cur-AC for initial concentration of 1000 mg/L at 25°C. This value for contact time is accepted for the following tests. The amount of boron adsorbed increased with increasing contact time and remained nearly constant after the equilibrium time (Figure 3).
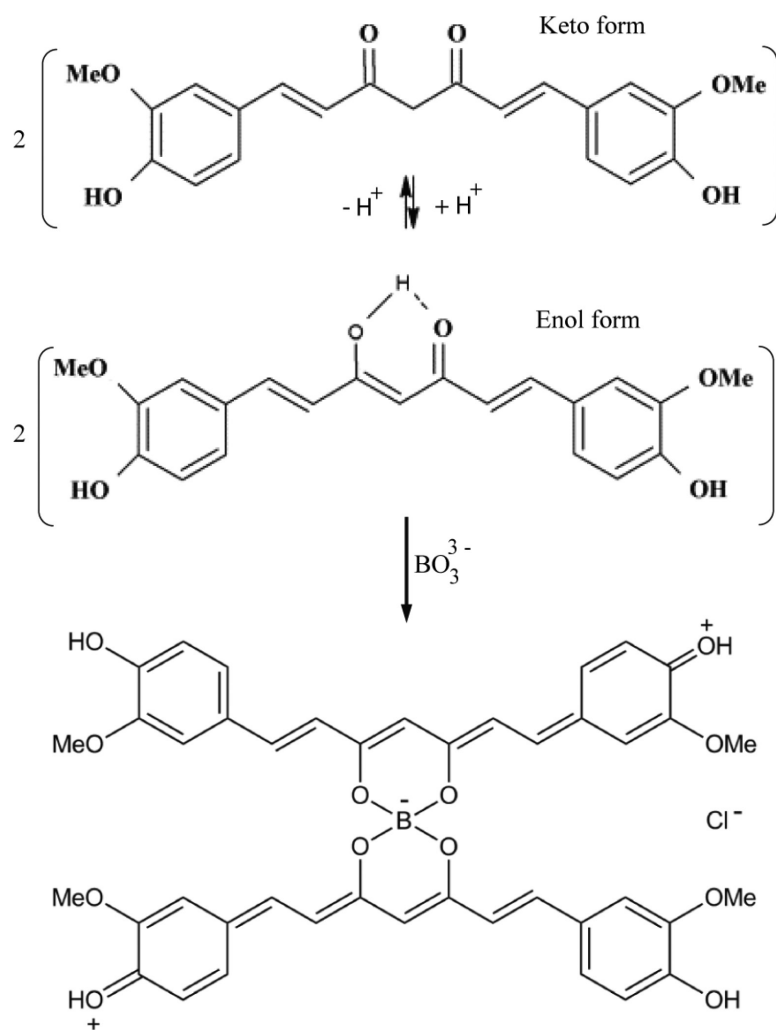

FIGURE 2. Reaction of boron and curcumin to form boron-chelate complex (rosocyanine)
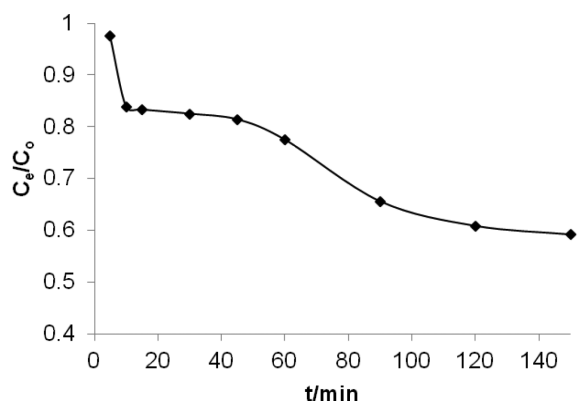
FIGURE 3. Kinetic rate of Cur-AC for boron removal from aqueous solution (initial concentration 1000 mg/L, volume of boron solution 50 mL, weight of adsorbent 2 g, shaking speed 200 rpm and at pH5.5)
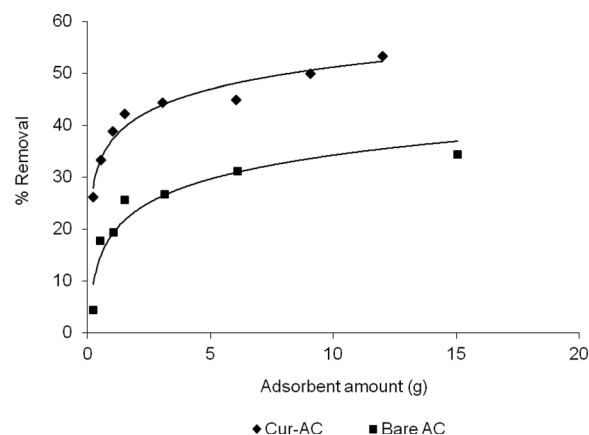

FIGURE 4. Effect of adsorbent dose of Cur-AC and bare AC on the removal of boron from aqueous solution (initial boron concentration was 1000 mg/L)

EFFECT OF ADSORBENT DOSE

The removal of boron was increased as the adsorbent dose increased because of the increase in the total available surface area of the adsorbent particles (Figure 4). For the initial concentration of 1000 mg/L, the uptake of boron reached up to 55% for a Cur-AC compared with 35% for bare AC. The effectiveness of boron removal as shown by the Cur-AC is the impact of curcumin impregnation on the surface of AC.

ADSORPTION ISOTHERMS

Adsorption isotherms are essential for the description of how adsorbate concentration will interact with adsorbent media and they are useful to optimize the use of media as adsorbents. Therefore, empirical equations (Langmuir and Freundlich isotherm models) are important for adsorption data interpretation and predictions. Both Langmuir and Freundlich models were used for the evaluation of experimental results. The Langmuir model assumes only one solute molecule per site and a fixed number of sites. The Langmuir isotherm relates q_e (milligrams of adsorbate adsorbed per gram of adsorbent media) and C_e (the equilibrium adsorbate concentration in solution), as shown in (1):

$$q_e = \frac{QbC_e}{(1+bC_e)}. \tag{1}$$

The Langmuir equation can be described by the linearized form,

$$\frac{1}{q_e} = \frac{1}{Q} + \frac{1}{QbC_e}, \tag{2}$$

where Q is maximum adsorption capacity in mg/g and b is the Langmuir constant.

The boron maximum adsorption capacity (Q) that was derived from the Langmuir plot for Cur-AC was 5.005 mg/g, obviously higher than that for bare AC (0.5923 mg/g). Cur-AC also indicated its renewable properties when maximum adsorption capacity (Q) for regenerated Cur-AC was slightly lower (3.6153 mg/g), as shown in Table 1.

The Freundlich isotherm assumes that the uptakes of adsorbate occur on a heterogeneous surface by multilayer adsorption and the amount of adsorbate adsorbed increases infinitely with an increase in concentration. The Freundlich equation is given as,

$$q_e = K_F C_e^{1/n}, \tag{3}$$

TABLE 1. Isotherm constants comparison between Cur-AC, bare AC and regenerated Cur-AC

Isotherms	Cur-AC	Bare AC	Regenerated Cur-AC
Langmuir			
R^2	0.9847	0.8442	0.8990
Q (mg/g)	5.0050	0.5923	3.6153
b	0.0014	0.0013	0.0007
Freundlich			
R^2	0.9985	0.9255	0.6604
n	0.1704	0.0922	0.4042
K_F (mg/g)(mg/L)^n	1.35×10^{-15}	1.05×10^{-30}	4.07×10^{-7}

where K_F is a rough indicator of the adsorption capacity and $1/n$ is the adsorption intensity. A linear form of the Freundlich expression will yield the constants K_F and $1/n$.

$$\log q_e = \log K_F + \frac{1}{n} \log C_e. \tag{4}$$

Therefore, K_F and $1/n$ can be determined from the intercept and slope of the linear plot of $\log q_e$ versus $\log C_e$. The magnitude of the exponent $1/n$ gives an indication of the favorability of adsorption (Chiou & Li 2003). Adsorption isotherms obey Freundlich isotherm for Cur-AC and bare AC whereas Langmuir isotherm controls the adsorption of boron for regenerated Cur-AC.

KINETICS AND MECHANISMS OF BORON ADSORPTION

To examine the controlling mechanisms of the adsorption process, four kinetic models were tested to fit the experimental data points: pseudo–first order, pseudo–second order, Elovich and intra-particle diffusion. Mathematical expressions of these models are given in (5) – (8), respectively (Chien & Clayton 1980; Lagergren 1898; McKay et al. 1999; Weber & Morris 1963).

The pseudo-first-order kinetic equation is,

$$\ln(q_e - q_t) = \ln q_e - k_1 t, \tag{5}$$

where k_1 is the rate constant (min^{-1}) of pseudo-first-order model, q_e and q_t are adsorption capacity (mg/g) at

equilibrium and at time t respectively. The plot of $\ln(q_e-q_t)$ versus t gives, k_1 and q_e values as shown in Figure 5(a).

Pseudo-second-order kinetic model was also applied to analyze kinetic adsorption for liquid solution kinetic adsorption in the following linear form (Weber & Morris 1963),

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}(t), \tag{6}$$

where k_2 is the rate constant of pseudo-second-order model $(\text{g}/(\text{min mg}))$ and q_t and q_e are the amount of adsorbate adsorbed at time t and at equilibrium (mg/g), respectively. The values of constants $(k_2$ and $q_e)$ can be obtained from the intercept and slope of the linear plot of t/qt versus t (Figure 5(b)).

The linear form of Elovich equation can be simplified as (Bohart & Adams 1920);

$$q_t = \ln(\alpha\beta) + \frac{1}{\beta} \ln t, \tag{7}$$

where α is the initial rate (mg/g min) and the parameter β is related to the extent of surface coverage and activation energy for chemisorptions (g/mg) (Bohart & Adams 1920; Thomas 1944). The value of constant $(\alpha, \beta,)$ can be obtained from the intercept and slope of the linear plot of q_t versus $\ln t$ (Figure 5(c)).

The intra-particle diffusion kinetic model explains about the nature of the adsorption process. If a plot of the linearized form of the intra-particle diffusion model
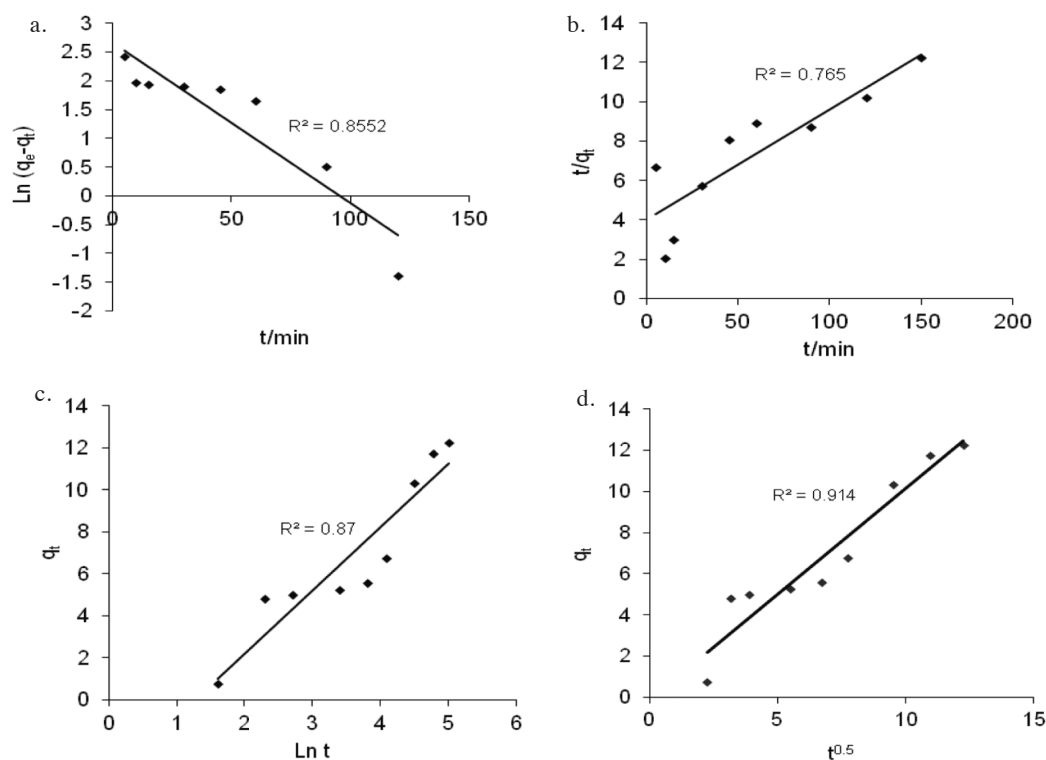

FIGURE 5. Plots of pseudo first-order (a), pseudo second-order (b), Elovich (c) and intra-particle diffusion kinetic models (d) for boron removal using Cur-AC as adsorbent

passes through the origin, the process is affected by only intra-particle diffusion; otherwise, adsorptive processes other than intra-particle diffusion might occur (Özacar et al. 2008).

Determination of rate parameter for intra-particle diffusion uses the following equation (McKay et al. 1999);

$$q_t = k_i t^{0.5} + c, \qquad (8)$$

where C is the intercept and k_i is the intra-particle diffusion rate constant (mg/g min$^{-1/2}$). The plot may present multi linearity, indicating that three steps take place (Figure 5(d)). The first, sharper portion is attributed to the diffusion of adsorbate through the solution to the external surface of adsorbent or the boundary layer diffusion of solute molecules. The second portion describes the gradual adsorption stage, where intra-particle diffusion is rate limiting (Yoon & Nelson 1984).

The study showed that the adsorption kinetics of boron adsorption on Cur-AC is in compliance with the intra-particle model, as it gives the highest R^2 ($R^2 = 0.914$) as compared with other models (Table 2). However, Elovich and pseudo–first-order kinetics models also show a good correlation. The adsorption kinetics of Cur-AC was more likely to follow the pseudo–first-order and intra-particle models. This suggests that the adsorption kinetics of boron was controlled by the physical adsorption and diffusion mechanisms that occur at the surfaces and pores of Cur-AC. As can be observed in Figure 3, the curves show an initial portion with a high slope and a second portion where the development tends to become constant. This is a typical development pattern in adsorption processes where, as the

TABLE 2. Constant values of pseudo–first-order, pseudo–second-order, Elovich and intra-particle kinetics models for boron adsorption on Cur-AC

Kinetic models	Constants
Pseudo 1st order	
R^2	0.855
k_1 (min^{-1})	0.027
q_e (mg/g)	14.426
Pseudo 2nd order	
R^2	0.765
k_2 (min^{-1})	0.00084
q_e (mg/g)	18.182
Elovich	
R^2	0.870
α (mg/g.min)	10.729
β (g/mg)	0.3326
q_e (mg/g)	52.682
Intra-particle	
R^2	0.914
K_i (mg/g.min$^{1/2}$)	1.024
c	0.102
q_e (mg/g)	8.034

process continues, the solute diffusion towards the sorbent is slower due to a decrease in its concentration.

FIXED-BED COLUMN ADSORPTION

Adsorption breakthrough curves (Figure 6) were obtained using influent boron concentration, C_o of 890 mg/L and flow rate at 8 mL/min. The highest boron removal in fixed-bed column adsorption was achieved up to 99% for the first 5 min for both fresh and regenerated Cur-AC. Statistical analysis (t-test) indicated that there were no significant different in boron removal between fresh and regenerated Cur-AC. It's indicated that regenerated Cur-AC was able to be reused after regeneration process without reduction of boron removal performance. Thomas, Bohart-Adams and Yoon-Nelson mathematical models have been applied in this study for the evaluation of efficiency and applicability of column models for large-scale operation (Bohart & Adams 1920; Thomas 1944; Yoon & Nelson 1984). Thomas developed a model for adsorption processes in which external and internal diffusion limitations are not present (Thomas 1944). The linearized form of the Thomas model can be expressed as:

$$\ln\left(\frac{C_o}{C_t} - 1\right) = \frac{k_T q_o m_c}{Q} - kTCot, \qquad (10)$$

where k_T is the Thomas rate constant (mL/mg.min), q_o is the equilibrium adsorption capacity (mg/g) of boron uptake, Co is the influent boron concentration (mg/L), Ct is the effluent boron concentration at time t (mg/L), m_c is the mass of adsorbent (g), Q is the inlet flow rate (mL/min) and t is the flow time (min). The value of Co/Ct is the ratio of inlet to outlet boron concentrations. A linear plot of ln [(Co/Ct) – 1] against time (t) was drawn to determine the values of q_o and k_T from the interception point and slope of the plot, respectively. The equilibrium adsorption capacity for Cur-AC was found to be higher than that for bare AC and regenerated Cur-AC, as well (Table 3). Regression coefficient values (R^2) indicate that data fit reasonably well for the Thomas model.
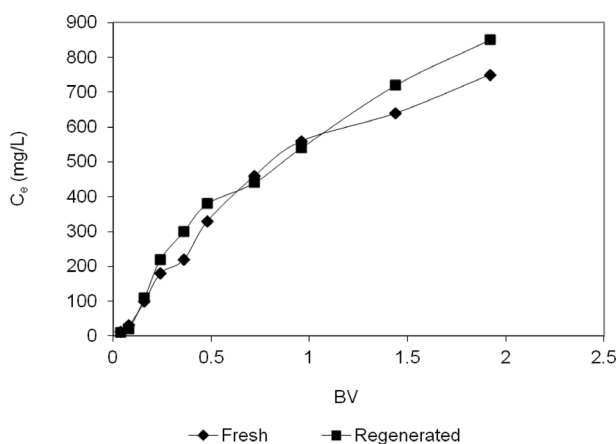

FIGURE 6. Fixed-bed column adsorption for boron removal using fresh and regenerated Cur-AC

Yoon and Nelson (1984) developed a model to describe the adsorption behaviour in the continuous column adsorption. The linearized form of the Yoon-Nelson model is given as:

$$\ln\left(\frac{C}{C_o - C}\right) = k_{YN}t - t_{0.5}k_{YN},$$ (11)

where k_{YN} is the rate velocity constant (L/min) and $t_{0.5}$ is the time (min) required for 50% adsorbate breakthrough. A linear plot of ln $[Ct/(Co - Ct)]$ against sampling time (t) was used to determine the values of k_{YN} and t from the slope and intercept of the plot. According to the Yoon-Nelson model, the amount of boron being adsorbed in a fixed bed is half of the total boron entering an adsorption bed within $2t_{0.5}$ period. For a given bed (Sivakumar & Palanisamy 2009),

$$q_{oYN} = \frac{q_{total}}{X} = \frac{\frac{1}{2}C_o\left[\left(\frac{Q}{100} \times 2t_{0.5}\right)\right]}{X} = \frac{C_oQt_{0.5}}{1000X}.$$ (12)

From (12) above, the adsorption capacity, q_{oYN} (in mg/g) varies as a function of inlet boron concentration (C_o/mg/L), flow rate (Q/mL/min), weight of adsorbent (X/g), and 50% breakthrough time ($t_{0.5}$). Table 3 indicates that q_{oYN} for Cur-AC and regenerated Cur-AC was 2.24 mg/g and 2.42 mg/g, respectively. It was slightly higher compare with bare AC that only 1.97 mg/g.

Generally, the Bohart-Adams model is used to describe the initial part of the breakthrough curve. The expression is given as:

$$\ln \frac{C_t}{C_o} = k_{AB}C_ot - k_{AB}N_o\frac{Z}{F},$$ (13)

where Co is the influent boron concentration (mg/L), Ct is the effluent boron concentration (mg/L), k_{AB} is the kinetic constant (mL/mg.min), F is the linear velocity (flow rate/column section area, cm/min), Z is the bed depth of the column (cm) and N_o is the saturation concentration (mg/mL). A linear plot of ln (Ct/Co) against time (t) was drawn and values of k_{AB} and N_o were determined from the slope and interception point of the plot, respectively. Table 3 lists the values of k_{AB}, N_o and R^2 for Cur-AC, regenerated Cur-AC and bare AC. All the R^2 values are less than 0.9, which indicates that the data do not fit into the model perfectly compared with the Thomas and Yoon-Nelson models. Therefore, the Thomas and Yoon-Nelson models can be used to describe the behavior of the adsorption of boron in a continuous column using Cur-AC rather than Bohart-Adams model.

CONCLUSION

Boron removal by Cur-AC is optimal at pH5.5 and at 120 min of contact time. The adsorption of boron onto Cur-AC and bare AC can be described better by Freundlich isotherm. The maximum adsorption capacity of boron for Cur-AC was significantly higher than that for bare AC. From the kinetic studies, the experimental results showed that the adsorption of boron conformed to the intra-particle model. The experimental data showed a better fit to the Thomas and Yoon-Nelson adsorption models. The Cur-AC can be regenerated using 0.1 M HCl solution to be reused with slightly lower adsorption capacity.

TABLE 3. Constants for fixed-bed models for boron adsorption on Cur-AC

Fixed-bed models	Cur-AC	Regenerated Cur-AC	Bare AC
Bohart-Adams			
k_{AB} (L/mg.min)	2.09×10^{-5}	7.2×10^{-5}	5.43×10^{-5}
N_o (mg/L)	311.62	270.44	364.46
R^2	0.8099	0.8145	0.8921
Thomas			
k_t (L/mg.min)	1.93×10^{-5}	2.76×10^{-5}	7.21×10^{-5}
q_o (mg/g)	1.70	1.54	1.44
R^2	0.9484	0.8366	0.9688
Yoon-Nelson			
k_{YN} (t/min)	0.0212	0.0198	0.0566
q_{oYN} (mg/g)	2.24	2.42	1.97
$t_{0.5}$ (min)	88.64	92.42	75.34
R^2	0.9668	0.9652	0.9282

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