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Mohammad Taghi Ghaneian[a], Ghader Ghanizadeh[b], Mohammad Tahghighi Haji Alizadeh[c], Mohammad Hasan Ehrampoush[a] & Farhan Mohd Said[d]

[a] Department of Environmental Health Engineering, Faculty of Public Health, Shahid Sadoughi University of Medical Sciences, Yazd, Iran

[b] Health Research Center and School of Health, Baqiyatallah University of Medical Sciences, Tehran, Iran

[c] Department of Environmental Health Engineering, International Campus of Shahid Sadoughi University of Medical Sciences, Yazd, Iran

[d] Faculty of Chemical and Natural Resources Engineering, University Malaysia Pahang, Pahang, Malaysia

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[a] *Department of Environmental Health Engineering, Faculty of Public Health, Shahid Sadoughi University of Medical Sciences, Yazd, Iran;* [b] *Health Research Center and School of Health, Baqiyatallah University of Medical Sciences, Tehran, Iran;* [c] *Department of Environmental Health Engineering, International Campus of Shahid Sadoughi University of Medical Sciences, Yazd, Iran;* [d] *Faculty of Chemical and Natural Resources Engineering, University Malaysia Pahang, Pahang, Malaysia*

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Pyrolysis of fresh sheep bone led to the formation of bone charcoal (BC). The structural characteristics of BC and surface area were determined by X-ray diffraction (XRD) and scanning electron microscopy (SEM). N_2 gas adsorption–desorption was analysed by Brunauer–Emmett–Teller isotherm model. The prepared BC was used as an effective sorbent for the removal of phosphate from aqueous solutions. The effect of major parameters, including initial phosphorous concentration, sorbent dosage, pH and temperature, was investigated in this study. Furthermore, adsorption isotherms and kinetics were evaluated. BC was an effective sorbent in phosphate removal from aqueous solution especially in phosphate concentration between 2 and 100 mg/L. The maximum amount of sorption capacity was 30.21 mg/g, which was obtained with 100 mg/L as the initial phosphate concentration and 0.2 g as the sorbent dosage. Best reported pH in this study is 4; in higher pH, adsorption rate decreased dramatically. By increasing the temperature from 20 to 40°C sorption capacity increased; this phenomenon described that adsorption is endothermic. Equilibrium data were analysed by Langmuir, Freundlich and Temkin isotherms. Pseudo first- and second-order and Elovich models were used to determine the kinetics of adsorption in this study. Collected data highly fitted with Freundlich isotherms and pseudo second-order kinetics. Achieved results have shown well the potentiality for the BC to be utilized as a natural sorbent to remove phosphorous from water and wastewater.

Keywords: adsorption; bone charcoal; phosphorous; isotherm; kinetic

1. Introduction

In contemporary societies, due to social and industrial improvement, wastewater production in large scale is an inevitable consequence. Untreated wastewater is the most hazardous compound to human populations and their environment. Wastewater treatment is one of the primitive demands of societies to prevent lethal diseases.[1] Phosphorous and nitrogen are common elements in wastewater, which have significant effect on eutrophication phenomena in water bodies.[2] Major components of phosphorus include the form of organic phosphates (e.g. detergents) and inorganic phosphates (*ortho* and polyphosphates).[3] Controlling phosphorus discharge from municipal and industrial wastewater treatment plants is a key factor in preventing eutrophication of surface waters. The new rule from Environmental Protection Agency requires that all existing wastewater treatment plants discharging in excess of 150 pounds of total phosphorus per month to surface waters meet below 1 mg-P/L effluent limit.[4,5] With regard to phosphorous hazards in aquatic environment, there is no doubt that phosphorous removal is so notable and need specific consideration during wastewater treatment to reach this goal. Physicochemical treatment methods and biological nutrient removal are two common methods for the removal of phosphate. These processes essentially transfer phosphate from the liquid to the sludge phase that needs to be hauled and disposed of elsewhere and also need more concentration on mechanism.[6] Complete phosphate removal is unattainable by these methods due to thermodynamic and kinetic limitations.[7] One of the most popular and economic methods for phosphorous removal in these days is the adsorption method. In these years, utilizing natural sorbent and adsorption method is becoming more popular in research scale. Adsorption mechanism is one of the techniques that would be comparatively more useful and economical for phosphates removal. In adsorption, the separation mechanism involves attracting one or more solutes in a liquid or gas mixing to the surface of a solid sorbent where they are held by intermolecular forces.[8,9] There are different kinds of sorbents that are utilized for phosphate elimination from aqueous solution such as fly ash,[10] wastewater sludge,[11] goethite [12] and red mud.[13] Bone charcoal (BC) is one of the low-cost natural sorbent, which has been utilized for pol-

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*Corresponding author. Email: farhan_msaid@yahoo.co.uk lution removal from aqueous solution in recent years.[14] Phosphorous adsorption on to BC and its isotherms and kinetics were analysed, respectively, in this research.

2. Materials and methods

2.1. Reagents and instruments

Phosphate solution (100 mg/L) was prepared with K_2HPO_4 (Merck Company). All other chemicals (NaOH, HCl, $(NH_4)_6Mo_7O_{24}.4H_2O$, H_2SO_4 and $SnCl_2$) were analytically grade reagents, which were used without further purification and prepared in laboratory condition.[15] Surface structure of BC was evaluated through scanning electronic microscopy (SEM) images (Model XL30 Philips). Specific surface area of BC was calculated via Brunauer–Emmett–Teller (BET) and Barrett–Joyner–Halenda (BJH) models. Data analysis for the calculation of $\mathrm{BET}_{\mathrm{Surface\ area}}$ and $\mathrm{BJH}_{\mathrm{Surface\ area}}$ (m^2/g), pore volume and porosity were carried out by Belsorb software (Version 5). The pH values were measured using a pH meter (Mettler Co., USA). Final concentration of the phosphate was measured using UV/Visible model (cecil-1011) spectrophotometer at 690 nm wavelength by molybdenum blue method.[15–17]

2.2. Bone charcoal preparations

The BC sample used in this study was synthesized from fresh sheep bone in laboratory condition. Fresh sheep bone was collected from butchery shop in Tehran, Iran. Fresh sheep bone was washed with boiled water to separate organic residuals and dried in oven with 100–110°C for 24 h. Pyrolysis of bones was performed by electrical furnace at 400°C for 2.5 h. Later, BC was soaked in 0.01 M HCl for 2 h to dissolve and remove any remaining impurities on BC surface and structure. Then, pulverize BC with ASTM standard sieves in the range of 0.1–0.5 mm.[18] X-ray diffraction method (XRD) and energy dispersive analysis X-ray (EDAX) spectra were utilized for evaluating BC ingredients. To calculate the $\mathrm{pH}_{\mathrm{Zpc}}$ of BC, 0.5 g of sorbent was suspended in 0.01 M sodium chloride solution after primary pH measurement for 48 h as contact time on shaker. Final pH also measured after suspensions were centrifuged. The Iodine number of sorbent with regard to ASTM and D4607-94 was determined by titration method with sodium thiosulphate as the titrant.

2.3. Adsorption experiments

All experiments in this study were fulfilled by batch technique. Batch experiments were carried out in different doses of adsorbent, initial phosphate concentration, pH and temperature. The initial concentration of phosphate was in the range of 2–100 mg/L. The doses of adsorbent also differed between 0.2 and 2.0 g/L. The pH of the solution was adjusted with HCl or NaOH (0.1 M) and measured using a pH meter (Mettler Co.). The effect of temperature on phosphate adsorption was investigated in temperatures between 20 and 40°C. The phosphate solution (100 mL) of desired initial concentration, pH, adsorbent dosage and temperature was taken in Al-covered glass vials and agitated in a shaker model (GFL 3017), 150 rpm for 24 h. At the end of each test, the suspensions were centrifuged for 7 min at 1500 rpm.

The amount of adsorbed phosphate at equilibrium on adsorbent surface, q_e, was calculated from the mass balance equation as

$$q_e = \frac{V}{M} \times (C_0 - C_e) \tag{1}$$

where q_e is the adsorption capacity (mg/g), V is the volume of solution (L), C_0 is the initial concentration of phosphate (mg/L), C_e is the equilibrium concentration (mg/L) and M is the weight of adsorbent (g). All tests were carried out with 150 rpm as the agitation speed and the average of three replicates experiments was reported.

Isotherms of phosphate adsorption on BC were evaluated with Langmuir, Freundlich and Temkin isotherm models to determine adsorption process for the description of solid–liquid sorption systems.[19,20] To determine the best isotherm model, compare their linear regression analysis and also correlation coefficient (R^2).

Linear form of Freundlich isotherm was determined with plot of log C_e versus log q_e, which is presented in the following equation:

$$\log q_e = \log k + \frac{1}{n} \log C_e \tag{2}$$

where q_e and C_e are the parameters that are described in Equation (1). k and n are constants that are related to the adsorption capacity and the adsorption intensity, respectively.

Linear form of Langmuir isotherm is calculated as

$$\frac{1}{q_e} = \frac{1}{q_{\max} \times bC_e} + \frac{1}{q_{\max}} \tag{3}$$

where $q_{\max}$ is described as the maximum amount of pollutant adsorption (mg/g) and also b is the constant that indicates adsorption equilibrium (L/mg). Langmuir isotherm is determined with the plot of $(1/q_e)$ versus $(1/C_e)$.[21] Linear equation Temkin isotherm is represented as

$$q_e = B \ln K_t + B \ln C_e \tag{4}$$

where $B = (RT/b)$, R is the universal gas constant (J/mol K), T indicates absolute temperature in K and b is also the adsorption equilibrium constant (L/mg). K_t and B constants are being determined by plotting q_e versus ln C_e.[21]

Pseudo first- and second-order and also Elovich kinetic models were used to determine adsorption kinetic process in this study. Lagergren equation was used to evaluate data with first-order model that is indicated as

$$\frac{dq_t}{d_t} = k_1(q_e - q_t) \tag{5}$$

Integrated form of the above equation can be shown as

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.203}t \tag{6}$$

where q_e and q_t are the amounts of phosphate per unit weight of BC (mg/g), respectively, at equilibrium time and time t. k_1 is the constant rate whose values are calculated from the plot of log $(q_e - q_t)$ versus t.

Pseudo second-order equation is also described as

$$\frac{dq_t}{d_t} = k_1(q_e - q_t)^2 \tag{7}$$

The linear and integrated form of the above equation is

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t \tag{8}$$

where q_e and q_t are the amount of adsorbed phosphate, respectively, at equilibrium time and at the time t (mg/g). k_2 (g/mg min) is the constant rate of equation and can be determined with a plot of (t/q_t) versus t. [21,22]

Elovich model is the last model, which is used for describing the adsorption process. Although this model was first used to study gas on solids chemisorption, it has been successfully improved to study adsorption from aqueous solution.

The Elovich equation is introduced as

$$\frac{dq_t}{d_t} = \alpha e^{-\beta q_t} \tag{9}$$

Integration of Equation (9) in the same boundary condition as pseudo first- and second-order equations leads to the following equation:

$$q_t = \frac{1}{\beta}\ln(\alpha\beta) + \frac{1}{\beta}\ln t \tag{10}$$

where α (mg/g min) is called as the initial sorption rate and the parameter β (g/mg) is also dependent on the surface coverage and chemisorption activation energy.[23,24]

3. Results and discussion

3.1. *Characterization of BC*

The morphology of the sample was examined by a scanning electron microscopy (SEM). The SEM image of the synthesized sample is shown in Figure 1. The results show the high porosity on the charcoal surface. EDAX results (Figure 2) showed that calcium and phosphorous are the major components which formed BC structure. The PXRD pattern of the prepared BC is shown in Figure 3. It shows that the main phosphorus phase is the hydroxyapatite (BC consists of 76% CaHAP ($Ca_{10}(PO_4)_6(OH)_2$). BET and BJH surface areas of entire sorbent are 138.35 and 111.92 m^2/g, respectively. Cheung et al. mentioned that BET surface area of synthesized BC was 100 m^2/g [25] and Ghanizadeh and Asgari also reported that BET of BC was 92.5 m^2/g [21]. Comparison of BC BET and activated carbon shows that activated carbon BET value is much more (1477 m^2/g [26]) than BC. Iodine number of BC was 119.26 mg/g (Table 1). Iodine number of synthesized BC was reported as 98 mg/g by Abe et al. [27]

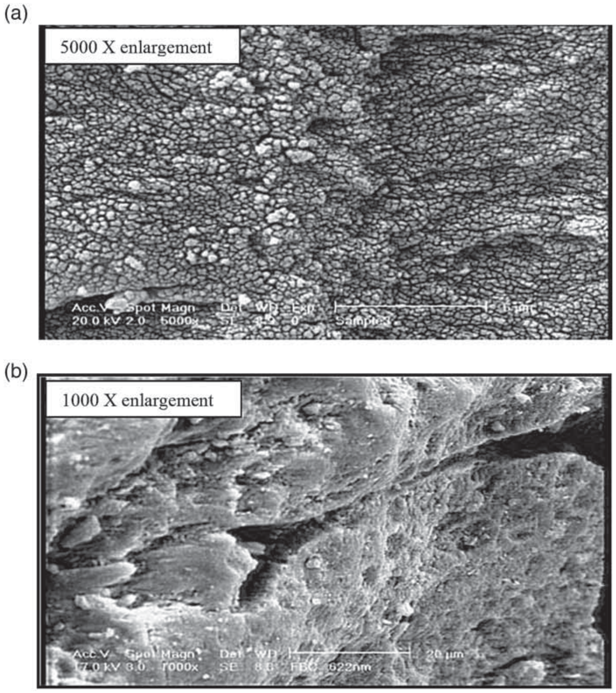

Figure 1. SEM surface image of BC: (a) 5000 × enlargement and (b) 1000 × enlargement.

3.2. *Effect of initial phosphate concentration*

The effect of initial phosphate concentrations on BC adsorption was investigated. A known amount of BC was soaked in six different phosphate concentrations separately, at different contact times of 10, 30 and 120 min. The results show that the amount of phosphate adsorbed on BC increased with the increase in initial phosphate concentration (Figure 4) and more ions are attracted to the BC surface from solution through diffusion mechanism till they reach saturation. This mechanism was studied by Wang et al. during phosphate biosorption.[28] The slope of the curve is linear and ascending (Figure 4). The amount of curve slope in 120-min contact time is higher than other contact times. Also in this contact time, q_e increased from 0.23 mg/g in 2 mg/L initial phosphate concentration up to 4.74 mg/g in 100 mg/L

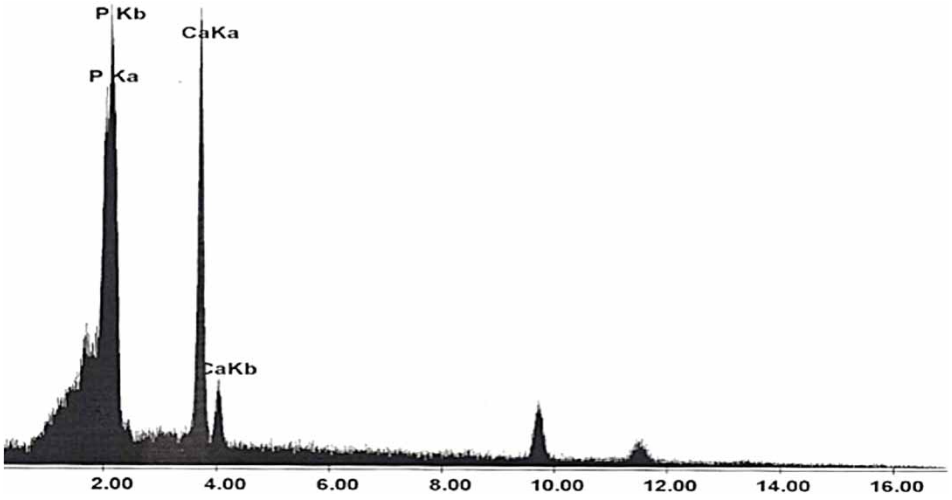

Figure 2. EDAX of BC.

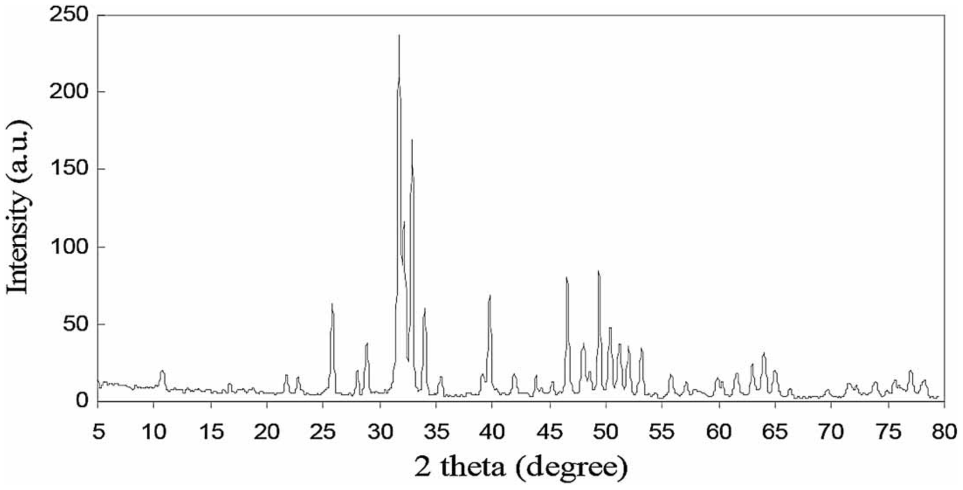

Figure 3. The XRD pattern of the synthesized BC.

Table 1. Charactristics of synthesized bone charcoal.

pH_{ZPC}	$BET_{surface\ area}$ (m^2/g)	$BJH_{surface\ area}$ (m^2/g)	Iodine number (mg/g)	Pore volume (cm^3/g)	Pore diameter (nm)
7.8	138.35	111.92	119.26	0.302	8.73

phosphate concentration. The similar effect has been widely studied by other researchers.[24–29]

3.3. *Effect of adsorbent dosage*

Sorption rate is a significant factor that has a influence on sorption equilibrium. To determine the effect of sorbent dosage on q_e, adsorption tests were examined with various amounts of BC (0.2, 0.5, 0.8, 1, 1.5 and 2.0 g/L) at different initial phosphate concentrations. The examination was performed at 48 h. Figure 5 shows that the adsorption capacity of BC decreased as the dosage increased. At BC doses higher than 1.5 g/L, curve reaches saturation and the highest amount of q_e was observed at 0.2 g/L dose and 100 mg/L initial phosphate concentration. At low sorbent dosage more amount of pollutant could be attracted to sorbent surface.[24–30] But the BC adsorption capacity was decreased by increasing the BC dosage. This decrease could be related to the use of surface area as the unsaturated form.[21]

3.4. *Effect of pH*

According to Figure 6, pH variations affect the efficiency of phosphate removal and the efficiency increased in lower pH. Our experiments showed that the pH_{ZPC} of BC is 7.8, it reveals that in pH below than the pH_{ZPC}, the surface charge of adsorbent is positive, and at pH above pH_{ZPC} surface,

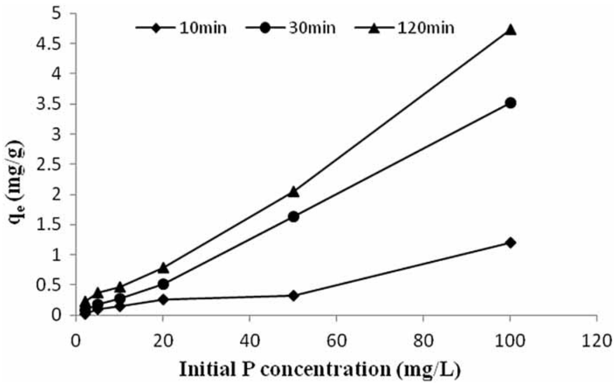

Figure 4. Effect of initial phosphate concentration (mg/L) at several contact times and 2 g/L sorbent dosage on q_e (mg/g).

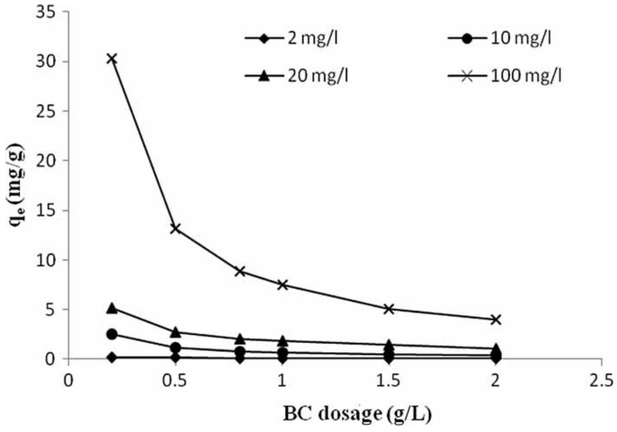

Figure 5. Effect of BC dosage (g/L) and initial phosphorous dosage on q_e (mg/g).

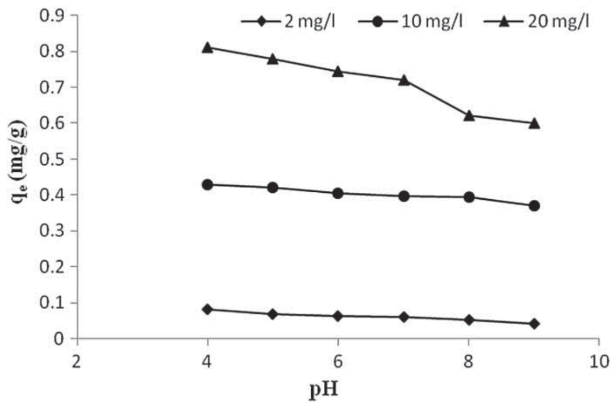

Figure 6. Effect of pH at several initial phosphate concentrations and 2 g/L sorbent dosage on q_e (mg/g).

the charge will be negative.[31] At pH above pH_{ZPC}, the hydroxyl group of BC surface could be deprotonated as follows [21]:

$$\text{HAP} - \text{OH} \rightarrow \text{HAPO}^- + \text{H}^+$$

The surface charge of BC shifts to negative, which leads to decrease in phosphate adsorption. Ou et al. [32] reported that pH values of solution have dramatic effect on phosphate removal efficiency; adsorption efficiency increase with decreasing pH. This result occurred due to the repulsion between sorbent and pollutants in solution.[32] Similar result was found by Xiong and Mahmood [33] who mentioned that at pH_{ZPC} 6.5, the amount of adsorbed phosphate increased from pH 4–6. However, at higher pH (above pH 6), the adsorbed phosphate decreased.[33]

3.5. *Effect of temperature*

The effect of temperature on phosphate adsorption by BC was studied. Phosphate adsorption capacity (q_e) slightly increased with increase in temperature from 20 to 40°C (Figure 7). The results show that the adsorption mechanism is an endothermic phenomenon; thus, the rate of phosphate adsorption increased with increase in temperature. The q_e increased slightly in linear form. The highest amount of q_e was obtained at 40°C with 10 mg/L initial phosphate concentration. Many adsorption researches reported that phosphate adsorption on several sorbents is an endothermic process.[24,34,35] The endothermic adsorptions are considered as chemical adsorption in which the adsorbate undergoes chemical interaction with the adsorbent.[36] Therefore, the adsorption of phosphate on the BC can be considered as a chemisorption.

3.6. *Adsorption isotherms*

The study of adsorption isotherms in sorption process is one of the significant concern to optimize system performance. Langmuir, Freundlich and Temkin are the three proper models that are used to analyse isotherms data. It is significant to determine the most appropriate correlation for the equilibrium curve. Adsorption isotherms studies were performed in different initial phosphate concentrations (2, 5, 10, 20, 50

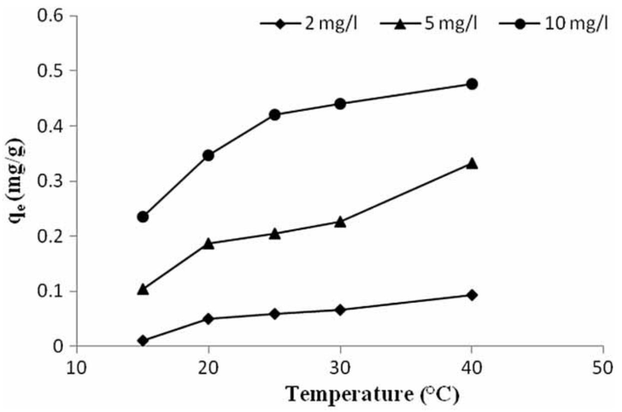

Figure 7. Effect of temperature in several initial phosphate concentrations and 2 g/L sorbent dosage on q_e (mg/g).

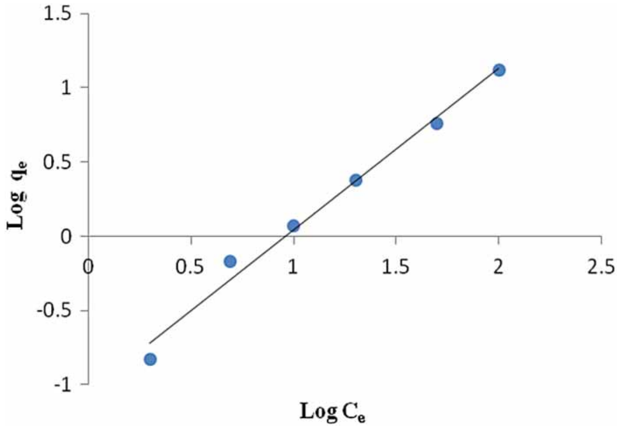

Figure 8. Langmuir isotherm model in 21°C, 10 mg/L initial phosphate concentration and 0.2 g/L sorbent dosage.

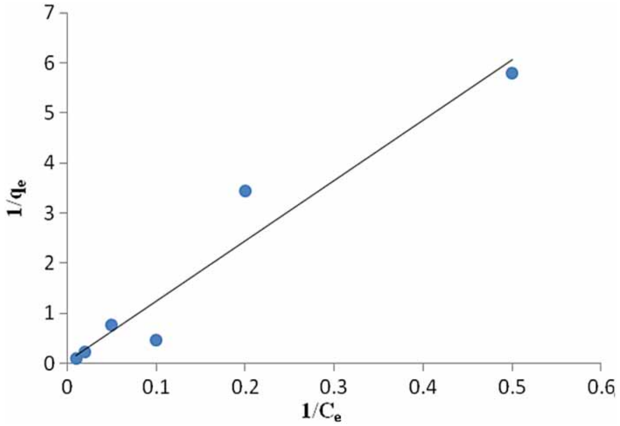

Figure 9. Freundlich isotherm model in 21°C, 10 mg/L initial phosphate concentration and 0.2 g/L sorbent dosage.

and 100 mg/L), 48 h as the contact time in order to reach equilibrium, 0.2 g BC as the sorbent dosage and at 21°C.

Freundlich isotherm indicated the adsorption on heterogeneous surfaces of sorbent and multilayer sorption process (Figure 8). Langmuir isotherm (Figure 9) indicated adsorption on monolayer surface of sorbent.[37] The sorbent surfaces reach to complete coverage by pollutant in 24 h. After 24 h, no significant changes are shown on the amount of adsorbed pollutant and desorbed pollutant. This effect was also reported in Saha study.[38] The Freundlich plot (Figure 8) supported this study. In several studies, Freundlich model was reported as a proper isotherm to describe adsorption mechanism of phosphate on different sorbents.[39,40]

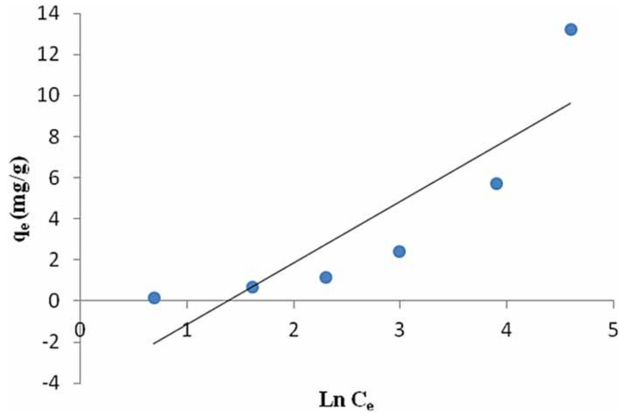

Figure 10. Temkin isotherm model in 21°C, 10 mg/L initial phosphate concentration and 0.2 g/L sorbent dosage.

Freundlich equation represents the highest fit ($R^2 = 0.9873$) (Figure 8 and Table 2). k_f and n were determined from line equation which resulted to 0.32 and 1.11, respectively. In Freundlich model, the higher the k_f and n imply higher binding capacity and affinity between the sorbent and phosphate ions.[41] Among these models, Temkin equation indicated the bad fitness ($R^2 = 0.7641$) (Figure 10 and Table 2).

3.7. *Adsorption kinetics*

Kinetic models have been used to determine several factors such as controlling chemical reactions, diffusion control and mass transfer mechanism. Pseudo first-, second-order and Elovich models were used in this study to describe the kinetic mechanism of phosphate adsorption on BC surface.

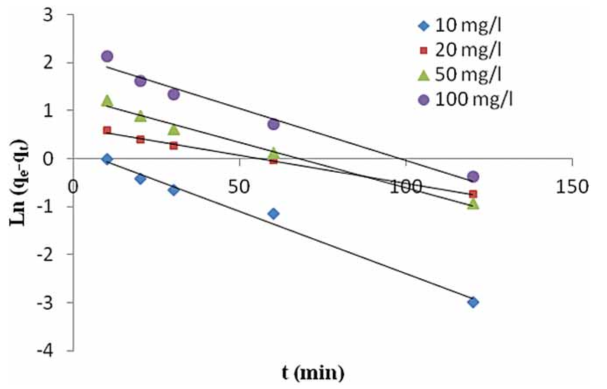

Figure 11. Pseudo first-order kinetic of phosphate adsorption in several initial concentrations.

Table 2. Parameters of Langmuir, Freundlich and Temkin isotherm models of phosphate adsorption.

Langmuir constants			Freundlich constants			Temkin constants		
$q_{\max}$ (mg/g^{-1})	b (L mg^{-1})	R^2	k_f (L g^{-1})	n	R^2	k_t (L mg^{-1})	B	R^2
30.2	0.0367	0.9371	0.32	1.11	0.9873	0.92	5.31	0.7641

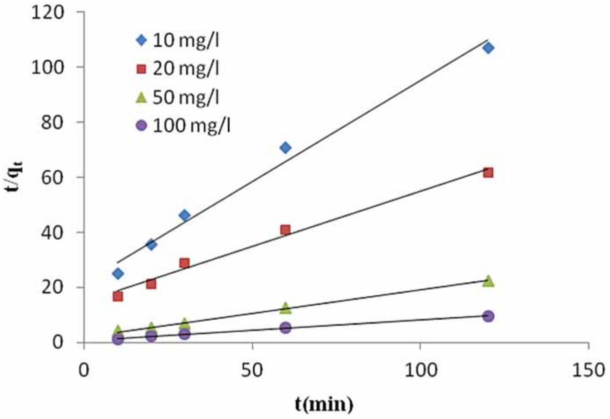

Figure 12. Pseudo second-order kinetic of phosphate adsorption in several initial concentrations.

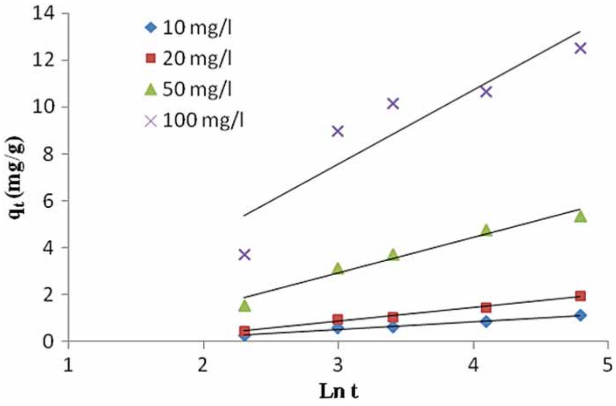

Figure 13. Elovich model correlation of phosphate adsorption in several initial concentrations.

Thus, kinetic studies of adsorption contribute to beneficial information for economical instrument and batch design. A plot of ln $(q_e - q_t)$ versus t (Figure 11) was used to determine the pseudo first-order kinetic models. Pseudo second-order kinetics and Elovich model correlation are shown in Figures 12 and 13, respectively. Both k_1 and k_2 were calculated to predict line equation. Kinetic parameters of pseudo first-order, second-order and Elovich model are summarized in Table 3. Regression coefficient and compatibly of experimental data with theoretical were considered in this study to reach the best kinetic model in order to process prediction. Although both pseudo first and second order indicated high R^2, pseudo second-order kinetics had the highest R^2. The experimental and theoretical data represented well correlation. Pseudo second-order kinetics has been introduced as the best descriptive model not only in this research but also in many different adsorption processes, which have been done by several researchers. Adsorption of phosphate on dolomite,[42] surface-modified zeolite, hydrotalcite and activated alumina [43] and iron-zirconium binary oxide [44] are the examples of phosphate adsorption studies, which follow-up pseudo second-order kinetics.

4. Conclusion

Besides several sorbents that are utilized for phosphate removal from aqueous solution, BC has a great potential in phosphate adsorption from water and wastewater effluent. Because of high surface area, low-cost preparation method and also acting as an environment friendly compound, this material could be used as an effective sorbent for phosphate removal in water and wastewater industry. Surface area of BC was evaluated by BET and BJH methods, which showed that the sorbent has high porous structure. According to the pH_{zpc} of BC, the sorbent has the potential for the removal of negative charge pollutant in acidic solutions. BC exhibited remarkable speed of phosphate uptake. More than 90% of final adsorption capacity was performed in 120 min. Due to observed results, the amount of phosphate in effluent could be decreased up to 0.5 mg/L. There is a good agreement between equilibrium data in this study and Freundlich isotherm that suggested adsorption mechanism is a multilayer process on the surface of sorbent. Pseudo second-order kinetics is the best model to describe phosphate adsorption on BC. This model described chemisorption and partial ion exchange process. Completion studies could be established in large scale.

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Table 3. Pseudo first- and second-order and Elovich kinetics parameters.

Elovich model			Pseudo second-order kinetic parameters				Pseudo first-order kinetic parameters				Initial phosphate concentration
R^2	β	A	R^2	K_1 (min^{-1})	q_e, pred. (mg/g $^{-1}$)	q_e exp. (mg/g $^{-1}$)	R^2	K_2 (g mg^{-1} min $^{-1}$)	q_e, pred. (mg/g $^{-1}$)	q_e exp. (mg/g^{-1})	C_0 (mg/L)
0.98	0.44	0.32	0.98	9.3×10^{-3}	1.11	1.17	0.98	0.618	0.992	1.17	10
0.98	0.86	0.57	0.99	0.016	4.83	2.42	0.98	0.219	2.86	2.42	20
0.96	1.60	1.51	0.99	0.044	7.42	5.73	0.99	0.055	9.62	5.73	50
0.83	1.88	3.15	0.99	0.1	15.54	13.2	0.97	0.021	18.34	13.2	100

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