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Removal of chromium(VI) from aqueous solution using iron orthophosphate that is synthesized by different methods

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ABSTRACT

The adsorption of chromium(VI) from aqueous solutions was studied on iron orthophosphate synthesized by solution, solution combustion, and solid state method. The properties of samples were determined by X-ray powder diffraction, nitrogen adsorption, and scanning electron microscope. The adsorption of chromium(VI) on solution combustion synthesized iron orthophosphate was found to be maximum in comparison to the other synthesis approaches. The equilibrium data for the adsorption of chromium were analyzed in Langmuir and Freundlich isotherm models. The adsorption process could be described by pseudo-second-order kinetic model. Iron orthophosphate was the most active at pH 2. The maximum adsorption capacity for iron orthophosphate synthesized by solution combustion, solid state, and solution method are 21, 13.2, and 12.1 mg/g at 100 ppm chromium concentration with 1 g/L adsorbent dose for 2 h. Iron orthophosphate is cheap and easy to operate for adsorption of hexavalent chromium(VI) from wastewater.

Keywords: Chromium(VI); Adsorption; Iron phosphate

1. Introduction

In recent years, the pollution of heavy metals has received increased attention because of the effects in ecosystem, agriculture, and public health. Chromium is one of the important toxic heavy metals which exist different oxidation states. Hexavalent chromium forms chromate (CrO_4^{2-}) and dichromate ($Cr_2O_7^{2-}$) which is more soluble and widely used in many fields of industrial process [1]. Its applications include chromate pigments in dyes, paints, and plastics; chromates used as anticorrosive agents to surface coatings; and chromic acid electroplated onto metal parts to provide protective coating [2].

For removing chromium(VI) from wastewater, different methods include precipitation [3], electrochemical precipitation [4], chemical activation [5], photoreduction [6], electrocoagulation [7], cementation [8], ion exchange [9], osmosis [10], solvent extraction [11], filtration [12], distillation and evaporation, etc. were investigated. These methods require high energy, expensive equipments and large quantities of chemicals and most of them have disadvantages like toxic byproduct formation and incomplete removal. Adsorption method is the most popular method which is widely used for removal of pollutants from water because of simplicity and low cost [13]. This technique

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also finds a solution for the problem of byproducts with using efficient adsorbent [14]. The most effective adsorbent is active carbon due to its high capacity of adsorption in spite of the high cost production [15–17]. A variety of materials such as activated slag [18], carbon nanotubes [19,20], clinoptilolite [21], aluminosilicate [22], hydroxyapatite [23], lignin [24] and sawdust [25], etc. have been investigated. To develop the limitations of low adsorption capacity, there is need to explore low cost adsorbents [26].

Iron phosphate is an important material because of advanced application such as catalysis [27], ion exchange, electrochemical and magnetic material [28]. It has been also used as an adsorbent for some metals [29–31]. Lenoble et al. [32] investigated the removal of arsenic using iron phosphate. Yin et al. [33] studied the removal of mercury(II) and silver(I) using amorphous iron(III) phosphate. Zhang et al. [34] used iron phosphate for the removal of chromium(III).

The present study undertakes the adsorption capacity of iron orthophosphate which is prepared by different synthesis approaches for the removal of Cr (VI) from aqueous systems. The effects of pH, contact time, amount of adsorbent, and concentration of solution were optimized for each product. The equilibrium results were modeled by both the Langmuir and Freundlich isotherms. The effect of synthesis method and surface properties were also compared and discussed.

2. Experimental

Iron(III) nitrate nonahydrate (Merck), diammonium hydrogen phosphate (Riedel), and glycine (Merck) were used for adsorbent preparation. Chromium(VI) solution was prepared from potassium dichromate (Sigma) and diphenylcarbazide(Sigma) was used as a complexing agent. All solutions were prepared with deionized water which had a specific conductivity of 18 mS. All chemicals were analytical grade and used without further purification.

2.1. Synthesis of adsorbent

Iron(III) phosphate was synthesized by solution combustion method, solution method, and solid state synthesis.

The iron phosphate was prepared by solution combustion method as reported in previous work [35]; 4.04 g $Fe(NO_3)_3.9H_2O$, 1.32 g $(NH_4)_2HPO_4$, and about 1.25 g glycine were mixed and dissolved in deionized water. The combustion mixture was dehydrated at 110˚C and the remaining gel-like residue was then introduced in a muffle furnace preheated at 500˚C.

Solution synthesis of iron phosphate was synthesized from a mixture of $Fe(NO_3)_3.9H_2O$ and $(NH_4)_2HPO_4$. Appropriate amount of iron nitrate and diammonium hydrogen phosphate (1:1 mole ratio) were dissolved in water, mixed, and evaporated. Then, the mixture was calcined at 550˚C for 6 h.

One mmol $Fe(NO_3)_3.9H_2O$ and 1 mmol $(NH_4)_2HPO_4$ were mixed in porcelain crucible for solid state synthesis. The mixture was heated step-by-step. First, temperature was raised up to 300˚C. After being held for 2 h at 300˚C, the samples were crushed and blended. Then, temperature was raised up to 650˚C with an increase in 5˚C per minute and the product was calcined at this temperature for 6 h.

2.2. Characterization of adsorbent

X-ray diffractometer was carried out using Rigaku DMAX-2200 with Cu Kα radiation to obtain phase identification. BET (Branauer, Emmett and Teller) surface area measurements were recorded using Quantachrome Quadrasorb SI Automated Surface Area Analyzer with nitrogen as the adsorption gas. The pore volume and average pore diameter were determined by BJH (Barrett, Joyner, Halenda) model on adsorption. The morphology of the powder was recorded by SEM using Quanta 200 FEG scanning electron microscopy.

2.3. Adsorption studies

Chromium solution was prepared by dissolving $K_2Cr_2O_7$ in deionized water. The Cr(VI) solution used in the experiments was prepared by diluting 300 mg/L potassium dichromate solution into deionized water to obtain the desired Cr(VI) concentration. Solution pH was adjusted to the required value with 0.1 mol/L H_2SO_4 and 0.1 mol/L NaOH. Ten milligram adsorbent was dispersed in 10 mL aqueous solution of Cr(VI) and stirred with magnetic stirrer at 200 rpm agitation speed, allowing sufficient contact time for adsorption equilibrium. The adsorbent were separated with filter paper and the concentrations of chromium were measured using UV–vis spectrophotometer at 540 nm, according to the diphenylcarbazide method [36]. Adsorption isotherm study was carried out with different initial concentrations of chromium(VI) from 50 to 300 mg/L with the adsorbent dosage at 1 g/L. The effect of time and pH were studied at room temperature with a chromium(VI) concentration of 10 mg/L and an adsorbent dosage 1 g/L.

The experiments were performed in replicates of three, and the samples were analyzed in replicates of three as well. The amount of metal ions adsorbed q (mg/g adsorbent) was obtained as follow Eq. (1):

$$q = \frac{[(C_o - C)V]}{m} \tag{1}$$

where C_o and C are the initial and equilibrium concentrations (mg/L), respectively; V is the volume of the aqueous phase (L); and m is the mass of the adsorbent used (g).

3. Results and discussion

3.1. Characterization of iron phosphate

The XRD pattern of the samples is shown in Fig. 1. The iron phosphate catalyst exhibited mainly quartz phase presented by peaks at 20.28° and 25.88° (ICDD 29-0715). The results show that the products prepared by different methods were structurally identical.

The morphology of the iron orthophosphate samples was studied by SEM and SEM images are shown in Fig. 2. These images prove that surface properties were changed by synthesis method. The products prepared by SCS indicate the formation of surface like foam, the products prepared by SSS indicate the formation of surface like balls, and the products prepared by SM indicate the formation of surface like plates.

The BET surface area and pore size of products which are indicated in the nitrogen adsorption–desorption isotherm and are shown in Table 1 are in constant change as the synthesis method changes. Pore sizes distributed in the range between 2 and 8 nm, indicating the presence of pores in the mesoporous range.

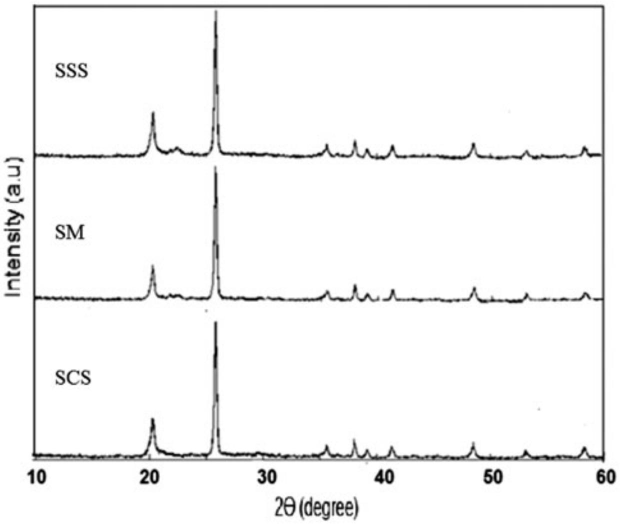

Fig. 1. XRD patterns of iron orthophosphates prepared by different methods. (SCS: solution combustion synthesis; SSS: solid state synthesis; and SM: solution method).

3.2. Chromium adsorption

The adsorption is one of the most effective and convenient methods for the removal of chromium(VI) from wastewater. The effect of chromium solution pH, contact time, and initial chromium concentration were studied in a batch experiment. Influences of these parameters were investigated by varying anyone of the process parameters and holding the other parameters constant.

3.2.1. Effect of pH

The effect of initial pH of the solution was shown in Fig. 3. The role of hydrogen ion concentration was observed at different pH range of 2–10. The experiments were at the constant initial metal and adsorbent concentrations of 100 and 1 g/L, respectively. The contact time was fixed to 120 min for all the experiments. The results show that increasing in pH resulted decreasing in metal adsorption capacity, because of the anionic nature of the hexavalent chromium. At low pH, the surface carries positive charge and can readily absorb the anion of Cr(VI). At $\text{pH} < 5$, Cr(VI) existed as $Cr_2O_7^{-2}$; At $\text{pH} > 8$, Cr(VI) existed as CrO_4^{2-}; when the pH was between 5 and 8, Cr(VI) existed in two or more forms. A pH value of 2 is selected as the best condition for this reaction condition. Solution combustion synthesis of iron orthophosphate has the maximum capacity for Cr(VI) removal.

3.2.2. Effect of contact time

Contact time is an important parameter for economical wastewater treatment [37]. The effect of contact time on the adsorption process was shown in Fig. 4. It was evident that time has significant influence on the adsorption of chromium(VI). From figure, it shows that the removal rate of Cr(VI) increased with increasing in contact time. Its growth rate continuously slowed down because of nonavailability of active sites on the adsorbent, and after 120 min, the removal rate did not change any more.

3.2.3. Effect of initial chromium concentration

The influence of initial chromium concentration on the adsorption capacity of the removal of chromium

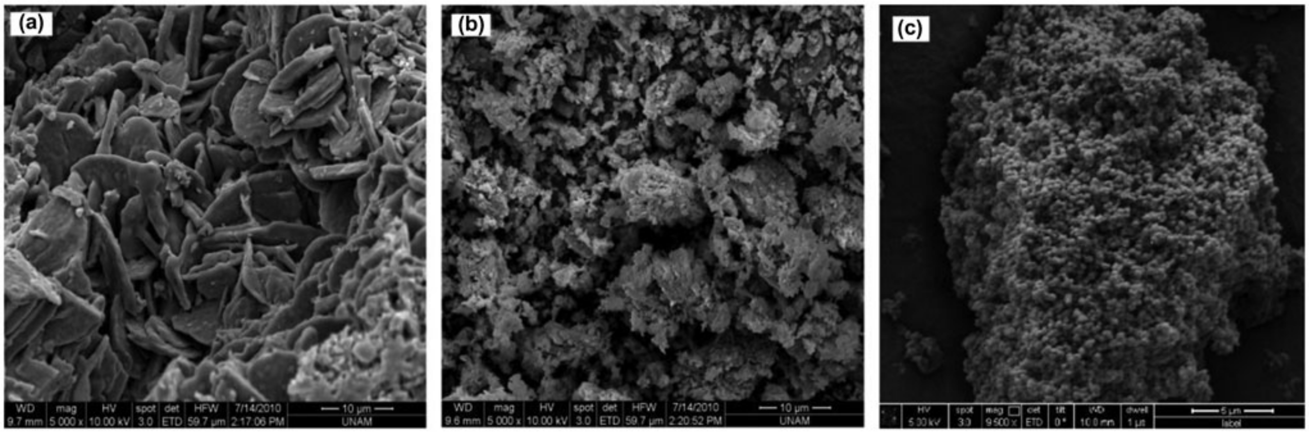

Fig. 2. SEM images of iron orthophosphates prepared by different methods (a) SM: solution method; (b) SCS: solution combustion synthesis; and (c) SSS: solid state synthesis.

Table 1
Surface area and pore size of products prepared by different methods

Sample	Surface area (m^2/g) (BET)	Pore size (Å) (BJH)
$FePO_4$ (SCS)	11.768	18.76
$FePO_4$ (SSS)	7.358	73.24
$FePO_4$ (SM)	5.918	76.13

Notes: SCS: solution combustion synthesis; SSS: solid state synthesis; SM: solution method.

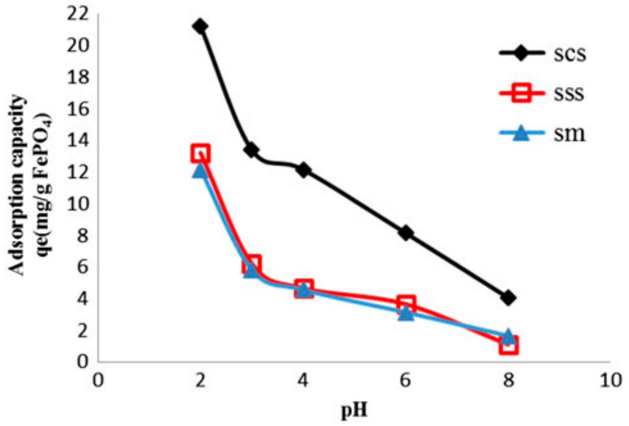

Fig. 3. Adsorption isotherm for Cr(VI) removal at different pH values.

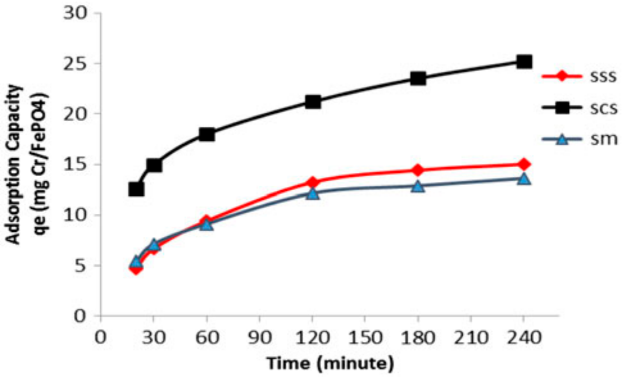

Fig. 4. Influence of contact time on Cr(VI) removal.

(VI) is shown in Fig. 5. Three adsorbent has been systematically investigated by varying the initial concentration between 50 and 300 mg/L. Their pH were adjusted to 2 and kept oscillating for 2 h at room temperature. Adsorption capacity was found to increase with increasing the initial metal concentration. It may be due to the interaction between the metal ions and the active sites of the adsorbent and fraction of surface coverage increased subsequently.

3.3. Adsorption isotherms

Adsorption isotherms are very important in order to design adsorption processes. The adsorption process of chromium(VI) was tested with Langmuir and Freundlich isotherm models. Langmuir isotherms

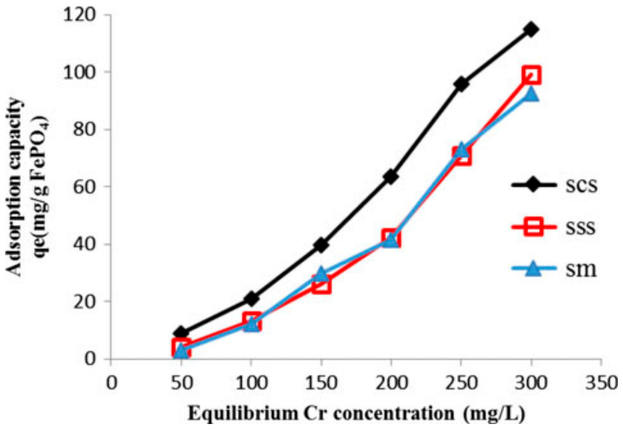

Fig. 5. Influence of initial chromium concentration on Cr(VI) removal.

were chosen to determine the maximum adsorption capacity corresponding to the complete monolayer coverage on iron orthophosphate. Langmuir equation is expressed by the following Eq. (2):

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

where q_e is the amount of metal ions adsorbed per unit mass of adsorbent (mg/g), C_e is the equilibrium concentration of the solute in the bulk solution (mg/L), and Q and b are the Langmuir constants related to maximum adsorption capacity and free energy of adsorption. The Langmuir parameters can be used to predict the affinity between the sorbate and sorbent. To investigate the favorability of the adsorption process, the separation factor (R_L) was calculated. R_L is defined by following Eq. (3):

$$R_L = \frac{1}{1 + bC_i} \tag{3}$$

where C_i is the initial concentration of solution (mg/L) and b is the Langmuir constant (g/L). The value of separation factor, R_L, indicates the nature of the adsorption process as $R_L > 1$ Unfavorable, $R_L = 1$ Linear, $0 < R_L < 1$ Favorable, and $R_L = 0$ Irreversible [38]. The values found between 0 and 1 confirm that the ongoing adsorption process is much more favorable on iron orthophosphate.

Freundlich isotherm was used to estimate the adsorption intensity of the chromium(VI) on adsorbent surface. Freundlich equation is given in Eq. (4):

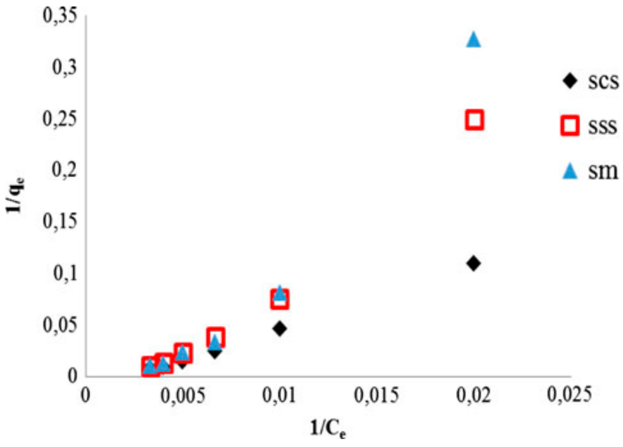

Fig. 6. Langmuir isotherm for adsorption of chromium(VI).

$$\ln q_e = \ln K + \frac{1}{n} \ln C_e \tag{4}$$

where K and n are indicators of adsorption capacity and adsorption intensity, respectively. For $n = 1$, the partition between the two phases is independent of the concentration. The situation $n < 1$ is the most common and corresponds to a normal L-type Langmuir isotherm, while $n > 1$ indicates the cooperative sorption, which involves strong interactions between molecules of adsorbate [39]. Langmuir and Freundlich adsorption isotherm were shown in Figs. 6 and 7. Adsorption isotherm model parameters are presented in Table 2.

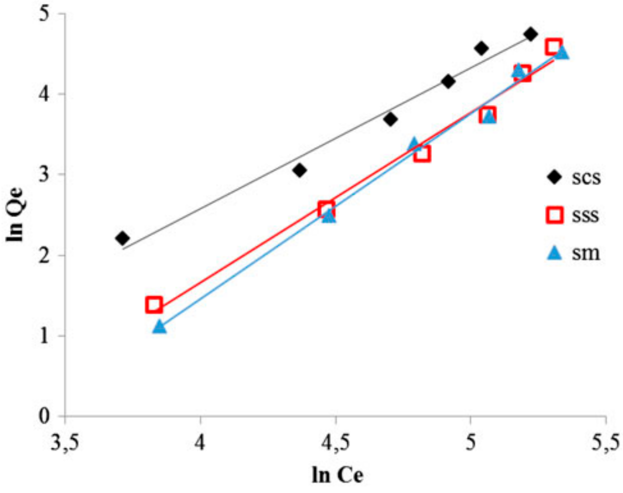

Fig. 7. Freundlich isotherm for adsorption of chromium(VI).

Table 2
Langmuir and Freundlich adsorption model constants for Cr(VI) adsorption

	Langmuir parameters				Freundlich parameters		
Sample	Q (mg/g)	b (L/mg)	R_L	R^2	K	n	R^2
$FePO_4$(SCS)	69.44	0.0023	0.813	0.9986	406.38	0.4826	0.9864
$FePO_4$(SSS)	39.53	0.0025	0.800	0.9946	621.29	0.4928	0.9903
$FePO_4$(SM)	34.13	0.0027	0.787	0.9676	2830.2	0.4252	0.9971

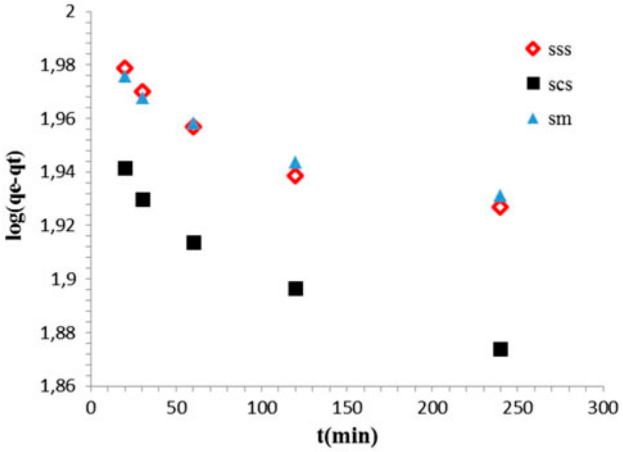

Fig. 8. Plot of the pseudo-first-order adsorption kinetics of chromium(VI).

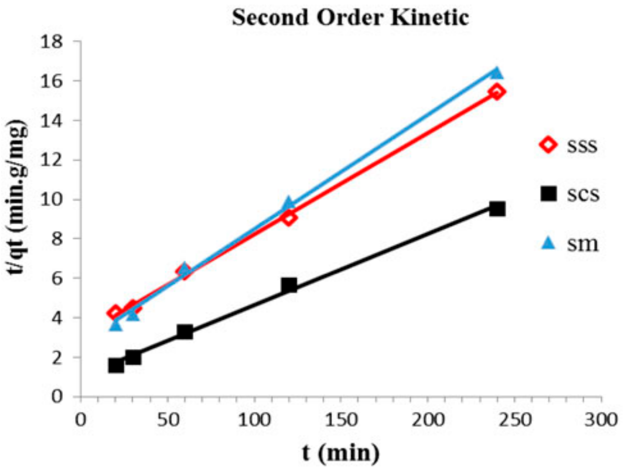

Fig. 9. Plot of the pseudo-second-order adsorption kinetics of chromium(VI).

3.4. Adsorption kinetics

To understand the mechanism of solid/liquid adsorption, pseudo-first-order and pseudo-second-order models were employed. The conformity between experimental data and the model predicted values was expressed by correlation coefficients (R^2 values close or equal to 1).

Pseudo-first-order kinetic model is given as Eq. (5);

$$\log(q_e - q_t) = \log q_e - \frac{k}{2.303} t \tag{5}$$

where q_e and q_t are amounts of chromium adsorbed at equilibrium and time t (mg/g), k is equilibrium rate constant of pseudo-first order (min^{-1}). Fig. 8 shows plot of linearization form of pseudo-first-order model. A comparison of the results is given in Table 3. The correlation coefficients for the pseudo-first-order kinetic model obtained for all adsorbents were low. This suggests that this adsorption system is not a pseudo-first-order reaction.

Pseudo-second-order model is given as Eq. (6);

$$\frac{t}{q_t} = \frac{1}{h} + \frac{t}{q_e} \tag{6}$$

where q_e and q_t are amounts of chromium adsorbed at equilibrium and time t (mg/g), the initial sorption rate

Table 3
Comparison of pseudo-first- and pseudo-second-order adsorption

	Pseudo-first order			Pseudo-second order			
Sample	q_e	k	R^2	h	q_e	k	R^2
$FePO_4$(SCS)	86.696	6.9×10^{-4}	0.929	0.964	27.778	1.25×10^{-3}	0.9958
$FePO_4$(SSS)	94.471	4.6×10^{-4}	0.883	0.323	19.455	8.53×10^{-4}	0.9988
$FePO_4$(SM)	94.016	4.6×10^{-4}	0.913	0.369	17.301	1.23×10^{-3}	0.9971

$h = kq_e^2$ (mg/g min), where k is the pseudo-second-order rate constant (g/mg min) [40]. A relatively high R^2 value indicates that the model successfully describes the kinetics of adsorption. Fig. 9 shows plot of pseudo-second-order model. A comparison of the results is given in Table 3. The correlation coefficients for pseudo-second-order kinetic model obtained were greater than 0.995 for all adsorbents. These indicate that the adsorption system studied belongs to the pseudo-second-order kinetic model.

4. Conclusions

The present work was carried out keeping in view the presence of toxic metals in industrial waste. Adsorption is a very effective technique and iron orthophosphate has a good adsorption effect for the removal of chromium from wastewater. The experimental results show that the best conditions for the treatment of Cr(VI) from wastewater are pH 2 and 120 min adsorption time at room temperature. The adsorption process of Cr(VI) can be described by Langmuir isotherm and Freundlich isotherms model. The separation factor shows that the iron orthophosphate is an efficient adsorbent and can be used for the adsorption of chromium(VI) metal ion from aqueous solution. The suitability of pseudo-first- and pseudo-second-order equation for adsorption of Cr(VI) onto iron orthophosphate is also discussed. The kinetic studies showed that the pseudo-second-order model was fitted well to the adsorption data under selected condition. Iron orthophosphate synthesized by different techniques may be used due to low cost and effective adsorbent for the recovery of chromium ion from wastewater. Solution combustion synthesis iron orthophosphate is the most efficient adsorbent because of surface area. Adsorption process is mainly a surface phenomenon and adsorption efficiency can significantly be affected by surface area. The experiment has been performed for three times and it has been observed that the adsorbents are reusable.

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