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KINETICS AND ISOTHERM STUDIES OF HEXAVALENT CHROMIUM ADSORPTION FROM WATER USING BONE CHARCOAL

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ABSTRACT

Bone charcoal (BC) prepared from cow bone is used as an adsorbent for the removal of hexavalent chromium from water in this study. The effects of Cr (VI) concentration, pH, contact time and the adsorbent dosage were investigated. The equilibrium data were fitted to Langmuir, Freundlich, Temkin, Dubinin–Radushkevich, Koble-Corigon and Redlich-Peterson isotherm models. Apart from R^2 , four error functions were used to validate the isotherm and kinetics data. The experimental adsorption isotherm complied with Freundlich and Koble-Corigon equation models ($R^2 > 0.99$) and the amount of adsorption (q_e) was 2.2 mg/g. The elevation of BC dose led to a decrease in q_e and increasing the pH led to the decreasing of Cr (VI) adsorption. The kinetic studies revealed that the adsorption of Cr (VI) complied with the pseudo-first-order kinetic ($R^2 > 0.95$). Analysis of data with Dubinin–Radushkevich isotherm showed that the energy of Cr (VI) adsorption process onto BC was 4.36 kJ/mol, which implies that the adsorption of Cr (VI) onto BC is physical in nature.

KEYWORDS:

Adsorption, Chromium, Water treatment, Bone charcoal

1. INTRODUCTION

Chromium is one of the most important heavy metals whose anions can be found in water as tri and hexavalent oxidation states. Although Cr (III) plays an important role in the control of blood sugar levels, however, at high concentrations its damages are similar to Cr (VI). It appears that both states of this element to be toxic to human; however, the hexavalent form of Cr is 500 times more hazardous than the trivalent form [1, 2]. This form of Cr has several toxic effects on human health such as epithelia and skin

irritation, lung cancer, kidney, liver and gastric damages, dermatitis asthma, nasal septum and penomonitis [3, 4].

Since the carcinogenic characteristics of this pollutant are confirmed, the responsible organizations such as the World Health Organization (WHO) and Environmental Protection Agency (EPA) improved their standards for drinking water. So, based on WHO set, the maximum concentration level goal of Cr for drinking water should not be more than 0.05 mg/l [5]. One of the most important Cr (VI) discharge to aqueous environments is derived from industrial waste streams such as pigment manufacturing, wood preservation, leather tanning and waste disposal sites leakage [6, 7]. Due to the health effects, the permissible concentration of hexavalent chromium for the discharge on surface water is considered to be 0.1 mg/l. Removal of the excess level of this pollutant from water and waste streams should be carried out by the appropriate techniques [8].

At present several methods are available which are applicable for the removal of Cr (VI) from water [9]. Many of these techniques have their own drawbacks and suffer from their constraints. However, adsorption is the most popular, promising and versatile technique for the treatment of contaminated waters and thus, it has gained much attention of the researches [10, 11]. Various sorbents such as bagasse fly ash, activated slag, cross-linked chitosan, activated carbon, tamarined hull and polymer coated silica gel have been used for the elimination of the Cr (VI) from water and waste streams [8, 11]. Some of the adsorbents are expensive and this constraint can be improved with the proper selection and application of inexpensive adsorbent materials [12]. However, in recent years, scientists have been employing low-cost materials such as local organic and inorganic sorbents for the elimination of pollutants from water [13].

This study investigated the purification of Cr (VI) contaminated water with bone charcoal. We described the adsorption isotherm studies with the relevant experimental data tested with Freundlich, Langmuir, Temkin and Dubinin-Radushkevich, Koble-Corigon and Redlich-Peter-

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son equations. The adsorption kinetics was tested with Lagergren models and adsorption energy was calculated with Dubinin-Radushkevich model.

2. MATERIALS AND METHODS

2.1. Preparation and Characterization of Bone charcoal

Bone charcoal (BC) was prepared and characterized according to the procedure described previously [14]. The fresh bone from cattle and sheep was crushed into pieces of 10–15 cm in length, rinsed three times with water and boiled at least three times in water for 2 h to remove fat and residual protein pieces. The bone was dried at 100–110 °C overnight and cooled in a desiccator. Pyrolyses of bone was performed in a rectangular furnace that was externally heated by electric power sources. The pyrolysis was carried out at 400 °C for 2 h so that the heating rate was 35 °k/min. The solid brownish black yield was transported to a desiccator and cooled at room temperature and pulverized by standard ASTM sieves with a 18-35 range mesh (0.5-1 mm) [14-16]. The chemical composition of BC was analyzed using X-ray fluorescence (Philips, Model X PER MPD). The results of these analyses are shown in Table 1. Other characteristics of BC are described in our previous study [14]. In the summary, BC is not uniformly porous and the size of pores are less than 20 nm, micro-pore sorbent with Brunauer-Emmett-Teller (BET) specific surface area 100 m²/g and Barrett-Joyner-Halenda (BJH) specific surface area 116 m²/g was determined by N₂ gas adsorption-desorption. The porosity of BC was determined through the conventional adsorption of nitrogen gas at 77 K and 91.43 KP pressure [9]. Analysis of data for calculation of BET_{Surface area} and BJH_{Surface area} (m²/g), pore volume (cm³/g) and porosity was carried out by Belsorb software (Ver.5) (data not shown).

2.2. Reagents and measurement method

All chemicals and reagents were of analytical grade that were used without further purification and purchased from Merck Co., Germany. All reagents were prepared in

double distilled water. The stock solution of Cr (VI) was prepared (500 µg/ml) by dissolving K₂Cr₂O₇ in 1.0 L of distilled water. The test solutions were prepared by successive dilution of stock solution to obtain the desired concentrations in the range of 1-2 mg/l. The diphenylcarbazide procedure used for the determination of Cr (VI) at 540 nm wavelength by using Visible- Cecil-1011 spectrophotometer which is the standard colorimetric method for the examination of Cr(VI) in aqueous solutions [17-19]. The pH of solutions was measured using a pH meter (Wagteck Mi 151) and adjusted using 0.1 M HNO₃ and 0.1 M NaOH solution. pH-meter was standardized with standard buffers before every measurement.

2.3. Adsorption Experiments

For convenient evaluation of the effective parameters, the adsorption of Cr (VI) within BC was performed with batch procedure (Table 2). These tests were carried out in a 250 ml high-density polyethylene bottles. The bottles were stirred with a shaker (GFL 3017) at 150 rpm at room temperature. The adsorbent was then separated by centrifugation (Eppendorf 5810R) and the residual hexavalent chromium was determined. The pH of solutions was measured using a pH meter (Wagteck Mi 151). The pH of solutions was adjusted at the beginning of the experiments and controlled afterwards and no significant pH variation was observed during each experiment. All of the adsorption experiments were carried out at 25±0.5 °C and the average of three replicates experiments was reported. The chromium removal efficiency (RE) and equilibrium adsorption capacity were calculated from Eqs. (1) and (2).

$$RE = \left(\frac{C_{ini} - C_t}{C_{ini}} \right) \times 100 \quad (1)$$

$$q_e = \frac{V}{M} \times (C_{ini} - C_t) \quad (2)$$

where, C_{ini} and C_t are the initial and concentration at time t of Cr (VI) (mg/l); q_e is equilibrium Cr (VI) concentration on adsorbent (mg/g), V is the volume of solution (L), M is the mass of BC sample used (g) [20, 21]. Adsorption

TABLE 1 - Chemical composition of BC (%mass)

CaO	P ₂ O ₅	MgO	Zn	Na ₂ O	SiO ₂	LOI ^a	Others	Total
92.896	2.954	0.672	0.664	0.6	0.095	2.06	0.059	100

^a Loss on ignition

TABLE 2 - Experimental conditions for chromium removal by BC

Experiment	Operational conditions			
	pH	Chromium concentration (mg/L)	BC dose (g/L)	Contact time (min)
Influence of pH solution	3-10	1.5	0.5	60
Influence of BC concentration	3	1.5	0.1 -1	60
Influence of chromium concentration and time	3	1-2	0.5	30-150
Adsorption kinetic of different concentrations	3	1-2	0.5	1440
Equilibrium tests	3	1-5	0.5	1440

isotherm studies and relevance experimental data were tested with two and three-parameter isotherms. Apart from correlation coefficient (R^2), the validity of adsorption isotherm and its goodness-of-fit was evaluated with the Marquardt's percent standard deviation (MPSD) and the hybrid error function (HYBRID) which can be described as:

$$MPSD = 100 \sqrt{\frac{1}{N-P} \sum_{i=1}^N \left(\frac{q_{ei}^{\text{exp}} - q_{ei}^{\text{cal}}}{q_{ei}^{\text{exp}}} \right)^2} \quad (3)$$

$$HYBRID = \frac{100}{N-P} \sum_{i=1}^N \left[\frac{(q_{ei}^{\text{exp}} - q_{ei}^{\text{cal}})^2}{q_{ei}^{\text{exp}}} \right] \quad (4)$$

where q_{ei}^{exp} is the observation from the batch experiment i , q_{ei}^{cal} is the estimated from the isotherm for corresponding q_{ei}^{exp} , N is the number of observations in experimental isotherm and P is the number of parameters in regression model. The smaller MPSD and HYBRID values reveal more accurate estimation of q_e value [22,14]. Also in kinetics studies apart from the correlation coefficient (R^2), the validity of kinetic models was measured by the normalized standard deviation (NSD), and average relative error (ARE) which can be defined as:

$$NSD = 100 \sqrt{\frac{1}{N-1} \sum_{i=1}^N \left[\frac{(q_t^{\text{exp}} - q_t^{\text{cal}})}{q_t^{\text{exp}}} \right]^2} \quad (5)$$

$$ARE = \frac{100}{N} \sum_{i=1}^N \left| \frac{(q_e^{\text{exp}} - q_e^{\text{cal}})}{q_e^{\text{exp}}} \right|_i \quad (6)$$

where q_t^{exp} and q_t^{cal} (mg/g) are experimental and calculated Cr (VI) adsorbed on BC at time t and N is the number of measurements made. The smaller NSD and ARE values indicate more accurate estimation of q_t values [14, 22].

3. RESULTS AND DISCUSSION

3.1. Equilibrium adsorption isotherms and kinetics of adsorption of various chromium concentrations

The adsorption isotherms express the specific relationship between the concentration of sorbate and its amount adsorbed onto sorbent surface at the given experimental conditions and the equilibrium concentration of sorbate in the liquid phase. Therefore, analysis of the isotherm data would be done to determine the maximum capacity of sorbent and to develop an equation for designing column purposes. So, in order to describe the chromium uptake capacity and its adsorption behavior onto BC, isotherm data obtained were fitted by four "two-parameter isotherm" including Langmuir, Freundlich, Dubinin-Radushkevich and Temkin and two "three-parameter isotherm" including Redlich-Peterson and Koble-Carrigan [13, 22]. In the case of three-parameter isotherms, the isotherms constants (ARP and P) were obtained by maximizing of R^2 value using a

trial and error method with using of MAPLE (Ver. 15) and MATLAB Microsoft, which was optimized after 14 iterations. Tables 3 and 4 indicated the parameters of isotherms by linear regression methods and the calculated adsorption isotherm parameters for Cr (VI) adsorption onto BC, respectively. As demonstrated in Table 3, adsorption of Cr (VI) within BC comply with both two and three-parameter isotherm models among which Freundlich and Koble-Corrigan models turned out to be extremely satisfactory with the highest values of $R^2 > 0.99$ and lowest MPSD and HYBRID values compared to the other adsorption models. The Freundlich model is an empirical method for adsorbents with heterogeneous adsorbing surfaces. The results from energy dispersive analysis X-ray (EDAX) and X-ray diffraction (XRD) analysis showed that the main compound of BC is carbon and hydroxyapatite (data not shown). This compound may randomly distribute on the surface of BC that may introduce heterogeneity onto the surface [23]. It is known that $1/n$ in Freundlich model is the adsorption intensity and the values of $1/n$ are laying between 0 and 1 confirmed the favorable conditions for adsorption. The $1/n$ of the Freundlich isotherm in this study was obtained 0.2. In addition, Koble-Corrigan model is generally applied with heterogeneous adsorption surface [22]. Therefore, these results suggest that surface of BC for adsorption of chromium is heterogeneous and produces a favorable adsorption condition. Dubinin-Radushkevich (D-R) isotherm usually used to distinguish between physical and chemical adsorption [24]. In this regard, experimental data were further interpreted by D-R isotherm. Analysis of data with D-R isotherm showed that the energy of Cr (VI) adsorption process onto BC was 4.36 kJ/mol. Based on literature for adsorption type and energy of adsorption, this range of energy is lower than the typical range of bonding energy for chemisorption (8-16 kJ/mol), and demonstrates that in the adsorption of Cr (VI) onto BC physisorption plays a significant role [7]. The kinetic adsorption data is probably the most important factor in adsorption system design, to determine the adsorbate uptake rate and the time required to attain equilibrium for industrial applications. Thus, to provide this data for Cr (VI) adsorption onto BC, two of the most used kinetic models (pseudo first-order and pseudo second-order) was evaluated. The linear mathematical representations and calculated rate constants of these models are given in Table 4. The adsorption rate constant for pseudo-first and pseudo-second-order models was calculated from the slope of the linear plot of $\ln(q_e - q_t)$ versus t and between t/q_t against t , respectively. q_e is the amount of Cr (VI) adsorbed (mg/g) at equilibrium and q_t is the amount of adsorption (mg/g) at the time t [25, 26, 27]. Calculated rate constants of kinetic models are shown in Table 5. Referring to Table 5 and comparison of the R^2 , NSD and ARE values of these models suggest that the equilibrium adsorption of hexavalent chromium onto BC could be best described with the pseudo-first order kinetic model. In addition, in this model, the calculated equilibrium adsorption capacities (q_e^{cal}) are very close to the experimental adsorption capacities.



TABLE 3 - Linearized expressions of adsorption isotherms [11, 19, 20]

Isotherm	Linearized form	Plot	Parameters
Langmuir	$\frac{C_e}{q_e} = \frac{1}{b \cdot q_{max}} + \frac{C_e}{q_{max}}$	$\frac{C_e}{q_e}$ vs C_e	q_{max} (slope ⁻¹) b(Slope/intercept)
Freundlich	$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e$	$\ln q_e$ vs $\ln C_e$	$K_f = \exp(\text{intercept})$ n= slope
Temkin	$q_e = B \ln K_T + B \ln C_e$	q_e vs $\ln C_e$	B= slope $K_T = B \times \exp(\text{intercept})$
Dubinin- Radushkovich	$\ln q_e = \ln q_{max} - D \epsilon^2$	$\ln q_e$ vs ϵ^2	$q_{max} = \exp(\text{intercept})$ D= -slope
Redlich-Peterson	$\ln[(A_{RP} \times \frac{C_e}{q_e}) - 1] = g \ln C_e + \ln B_{RP}$	$\ln[(A_{RP} \times \frac{C_e}{q_e}) - 1]$ vs $\ln C_e$	g= slope $B_{RP} = \exp(\text{intercept})$ A_{RP}^*
Koble-Corrigan	$(\frac{1}{q_e}) = (\frac{1}{A_{KC} \times C_e^p}) + (\frac{B_{KC}}{A_{KC}})$	$\frac{1}{q_e}$ vs $\frac{1}{C_e^p}$	$A_{KC} = \text{slope}^{-1}$ $B_{KC} = (\text{intercept/slope})$ p^*

*Optimized with trial and error

TABLE 4 - Calculated isotherm parameters for Cr (VI) adsorption onto BC

Isotherm	R ²	MPSD	HYBRID	Parameter
Langmuir	0.996	54.3	17.9	$q_{max} = 2.2 \text{ mg/g}$ $b = 3.53 \text{ (L/mg)}$
Freundlich	0.998	3.98	0.062	$K_f = 2.44 \text{ (mg g}^{-1}) \text{ (L mg}^{-1})^{1/n}$ n=0.2
Temkin	0.988	21.5	1.49	B= 0.33 $K_T = 1 \text{ (L/mg)}$
Dubinin- Radushkovich	0.83	45.84	7.77	$q_{max} = 1.054 \text{ mg/g}$ D= 0.0263
Redlich-Peterson	0.985	10.0266	0.4	g= 0.3 $B_{RP} = 4.694 \text{ (L/mg)}^g$ $A_{RP}^* = 17.7024 \text{ (mg/g) (L/mg)}$
Koble-Corrigan	0.999	4.225	0.053	$A_{KC} = 18.19 \text{ (mg/g) (L/mg)}^p$ $B_{KC} = 13.33 \text{ (L/mg)}^p$ $p^* = 1.227$

TABLE 5 - Mathematical representations and calculated rate constants of kinetic models

First -order kinetics $\log(q_e - q_t) = \log q_e - \frac{K_1}{2.303} t$	C ₀	K ₁ (1/min)	q _e Exp.(mg/g)	q _e Cal.(mg/g)	R ²	ARE	NSD
		1	0.004	1.99	2	0.98	
	1.5	0.008	1.98	1.99	0.97		
	2	0.006	1.95	1.96	0.95		
						6.55	21.15
Second-order kinetics $\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t$	C ₀	K ₂ (g/mg/min)	q _e Exp.(mg/g)	q _e Cal.(mg/g)	R ²	ARE	NSD
	1	0.003	1.99	1.88	0.95		
	1.5	0.004	1.9	1.89	0.93		
	2	0.005	1.4	1.99	0.92		
						10.9	78.27

3.2. Effects of BC dose

Although adsorption is a promising and versatile process for water and wastewater purification, however, in some cases high cost of adsorbents may be recognized as the biggest barrier to apply it on mass scale. Thus, from economic point of view the optimization of adsorbent dose

and the best required mass of sorbent for scale-up and designing of large scale equipment is necessary. The influence of BC on chromium adsorption was investigated in a range of 0.1-1 g/100ml under the operational conditions specified in Table 2. The results of percentage removal of chromium at various BC doses are presented in

Fig. 1. As shown in Fig. 1 increasing BC dosage from 0.1 to 0.7 g/ 100 ml had a positive effect on Cr (VI) removal efficiency and residual concentration of pollutant on water body however, the elevation of BC dosage beyond this value was not effective on Cr (VI) adsorption. Based on this result, it can be concluded that 0.7 g/100 ml of BC is the optimum mass of this sorbent for Cr (VI) elimination from water that led to residual concentration lower than 0.05 mg/l as the maximum allowable concentration value recommended by WHO. Although, increasing BC dosage led to enhanced removal and low residual of Cr (VI); however, higher dosage of BC led to decreased Cr (VI) adsorption onto BC (mg/g). This phenomenon can be explained by many factors, which can be attributed to the adsorbent dose. At the start, some of the active adsorption sites may remain unsaturated and as dosage of BC is increased, this can lead to unfavorable use of sorbent, resulting in reduction of the ratio of Cr (VI) adsorbed to BC (mg/g). The second reason is that at the higher masses of BC, adsorbent particles may become agglomerated and lead to decrease in the available surface for sorption [24].

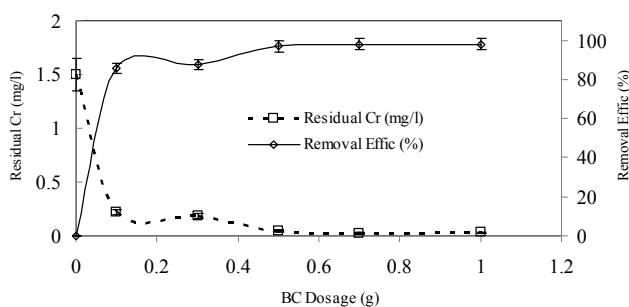


FIGURE 1 - Effect of BC dosage on Cr (VI) adsorption

3.3. Effect of contact time and Cr (VI) concentration

In this work, influence of different initial chromium concentration (1-2 mg/L) on adsorption process was investigated under the condition given in Table 2. Figs. 2 and 3, represent the results of the effects of contact time and Cr (VI) concentration on the adsorption. As illustrated in Fig 3, it can be concluded that the equilibrium was reached at 150 min. It is clear that increasing the contact time beyond the 150 min had not significant impact on adsorption performance. In addition, Fig. 3 implies that adsorption led to saturation that displays the possible monolayer sorption of Cr (VI) onto BC. As shown in Fig. 3, increasing initial Cr (VI) concentration from 1 up to 2 mg/l led to decrease of uptake efficiency from 99% to 70% and increased residual concentration from 0.01 mg/l to 0.6 mg/l which implies that sorption capacity is dependent on preliminary Cr (VI) concentration. Since in a fixed dosage of sorbent and identified concentrations of adsorbate, sufficient vacant site of adsorbent is available for pollutant adsorption, increase of adsorbate concentration led to limited ratio of vacant sites of adsorbent surface to Cr (VI) concentration, so that a decrease in percentage adsorption corresponding to an increase in initial adsorbate concentration occurred.

Increasing of q_e (mg/g) with elevation of initial Cr (VI) concentration may be related to driving forces needed to overcome the resistances of pollutants migration from the aqueous solutions to the BC surface. Similar results were found in previous studies [27]. Comparison of Cr (VI) uptake capacity by the adsorbent used in this study and those reported elsewhere is shown in Table 6. It can be concluded that the sorption capacity of the BC is still higher than some of the available ones in the literature.

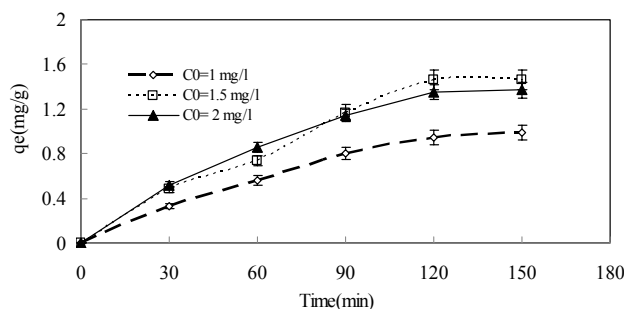


FIGURE 2 - Effect of Contact time on Cr (VI) adsorption

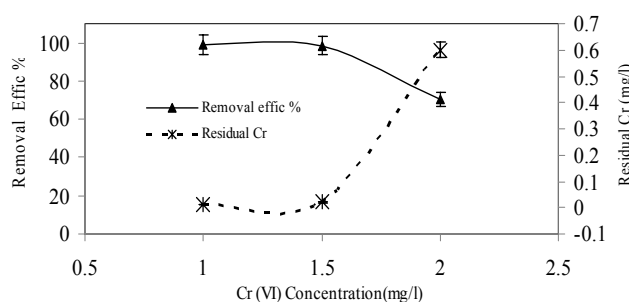


FIGURE 3 - Adsorption of Cr(VI) in different concentration

TABLE 6 - Capacity of different sorbent for Cr (VI) adsorption

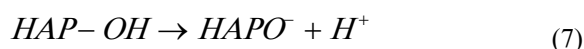
Adsorbents	q_e (mg/g)	Ref.
Activated Carbon	1.6	[29]
Rice husks	0.6	[30]
Saw dust	1.5	[30]
Coir pith	0.2	[30]
Clay-Chitosan	1.30	[31]
Polyvinyl Pyrol-coated gel	1	[4]
Soya cake	0.0028	[32]
Bone Charcoal	2.2	This study

3.4. Effect of pH and mechanism of adsorption

Since the pH of the aqueous solution has a key role in adsorption process, the effect of this parameter on the elimination of Cr (VI) by BC was examined in a range of 3-10 under the conditions provided in Table 2. The means of three replicate experiments are shown in Fig. 4. As shown in Fig. 4, it is clear that the adsorption of Cr (VI) is highly influenced by the solution pH.

As illustrated in Fig. 4, Cr (VI) removal efficiency onto BC decreases from 98.5% to 55% and residual concentra-

tion of Cr (VI) increased from 0.07 to 0.18 mg/l, when the pH solution elevated from 3 to 10. Maximum chromium adsorption at pH 3 can be explained by the evaluation influence of pH solution both on the surface charge of the BC particles and the dominate species of chromium ions in solution (7). This phenomenon can be explained according to the pH_{ZPC} of the BC and nature of Cr (VI) in solutions. Based on the previous experiments the pH_{ZPC} of the BC was about 8.3 [28]. This means that the BC surface was negatively charged at solution pHs above 8.3 and chromium ions were repelled by the BC surface resulting in the reduction of Cr (VI) adsorption. At pH higher than pH_{ZPC} , the surface of BC can be deprotonated in the following reaction [14]:



Therefore, in the elevated range of pH, the surface charge of BC is negative and at pH values below the pH_{ZPC} of BC, the surface of BC was positively charged, causing better Cr ions adsorption through the electrostatic attraction. Other than pH_{ZPC} the nature of Cr ions in solutions is very important in their adsorption which is dependent on the solution's pH. In acidic and basic solutions, $HCrO_4^-$, CrO_4^{2-} and CrO_7^{2-} are predominant, respectively [7,8]. Since the pH_{ZPC} of BC was determined to be 8.3, the surface charge of the BC was negative above this point of pH and only anionic species could be adsorbed on to BC below of its pH_{ZPC} and adsorption of anions forms of Cr (VI) was not suspected beyond pH 8.3 as the negative charges were predominant on BC surface. Thus, increasing of pH from 3 to higher than pH_{ZPC} led to decreasing of Cr (VI) adsorption onto BC.

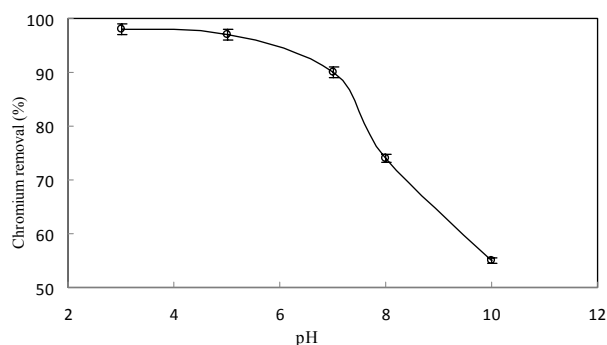


FIGURE 4 - Adsorption efficiency of Cr (VI) Vs pH adsorption of Cr (VI)

4. CONCLUSION

In the present work, Bone charcoal, as a low-cost and efficient adsorbent, was tested for the removal of Cr (VI) from aqueous solution. The adsorption process is pH dependent and the optimum pH was 3.0. The kinetic studies showed that the adsorption data were fitted well to the pseudo-first order model with high interrelation coefficient ($R^2 > 0.95$) and smaller the normalized standard de-

viation (NSD) and average relative error (ARE). Furthermore, the isotherm equilibrium studies confirmed that the Freundlich and Koble-Corrigan forms are best-fitted models for the adsorption process of Cr (VI) by BC. Adsorption capacity of Cr (VI) was 2.2 mg/g, and optimal dosage of BC was 0.7 g /L. A result from analysis of data with Dubinin-Radushkevich isotherm showed that adsorption of Cr (VI) onto BC is physical in nature. Although, we found several aspects of Cr (IV) adsorption onto BC; however, further studies will be required to scale-up and optimize process variables.

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