

## Adsorption of chromium and copper in aqueous solutions using tea residue

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**ABSTRACT:** In this study, adsorption of copper and chromium was investigated by residue of brewed tea (Tea Waste) from aqueous solutions at various values of pH. It was shown that adsorbent dose, copper and chromium ion concentrations in such solutions influence the degree of these heavy metal ions' obviolation. The adsorption level of the prepared solutions was measured by visible spectrophotometer. The tea residue adsorbed copper (II) and chromium (VI) ions at initial solution pH by 25 % and 3 %, respectively. During the experiments the peak adsorption occurred in hydrated copper nitrate aqueous solution at pH range of 5-6. Likewise the maximum adsorption appeared in potassium chromate aqueous solution at pH range of 2-3. In addition, tea residue adsorbed about 60 mg/g of copper (II) ion at pH=5, while chromium adsorption was registered at about 19 mg/g at pH=2. The data obtained at the equilibrium state, was compared with Langmuir and Freundlich models. Results showed that regarding the kinetics of adsorption, the uptake of copper (II) and chromium (VI) ions by tea residue was comparatively faster, with the adsorption process exhaustion completed within the first 20 min of the experiments. Furthermore, results revealed that adsorption data concerning the kinetic phase is closely correlated with a pseudo-second order model with  $R^2 > 0.99$  for copper (II) and chromium (VI) ions

**Keywords:** Adsorption kinetics; Heavy metal; Isotherms; Langmuir and Freundlich models

## INTRODUCTION

Industrial wastes in aqueous solutions are complicated contaminants containing heavy metals, mostly lead, copper, nickel and chromium, which if not properly dealt with, cause serious environmental pollution imposing costly clearing operations (Babu and Ramakrishnan, 2003). These heavy metals are poisonous not only to human, but also to aquatic community (Corbbitt, 1999; Tunali *et al.*, 2006; Mahvi, 2008; Zvinowanda *et al.*, 2009; Rafaat *et al.*, 2010). There are different methods of removing heavy metals from wastewater (Babel and Opiso, 2007). Considering their potential hazard for human health and agricultural products, effective methods of remediation have been developed (Nouri *et al.*, 2008). Among different adsorption methods, precipitation following coagulation has been widely used, though during this process large volume of sludge is also produced (Palmer, 1995). Moreover, various methods, such as

filtration, ultra filtration, microfiltration, and active carbon are developed for efficient adsorption process, which irrespective of their effectiveness are quite expensive (Ricordel, 2001; Pesavento, 2003; Qadeer and Akhtar, 2005; Chen *et al.*, 2010). A large number of artificially made and natural adsorbents are identified, including rubber used in tires (Kadrivelu *et al.*, 2001), clay (Mohan *et al.*, 2001), kyanite and silica gel (Ajmal *et al.*, 2001), bone char and crab shell (Cheung *et al.*, 2001), animal bone (Banat *et al.*, 2000), nut shell (Vaughan *et al.*, 2001), saw dust (Yu *et al.*, 2001; Argun *et al.*, 2007; Vinodhini and Das, 2010), sugar-beet pulp (Senthilkumaar *et al.*, 2000; Pehlivan *et al.*, 2006), waste tea leaves (Al-Asheh *et al.*, 2000), chitosan beads and human hair (Mohan *et al.*, 2001), vermiculite or zeolite (Pandey *et al.*, 2010), IR93 (Ricou-Hoeffer *et al.*, 2001; Dizadji *et al.*, 2009) and fly ash extracted from burned coal (Lin, 2001; Mohan *et al.*, 2001; Ricou-Hoeffer Lecuyer *et al.*, 2001). Recent studies indicated that bio-adsorbent, composed of cellulose material, has excellent

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adsorption properties in aqueous solutions. Borage, citrus skin (Ajmal, 2001), grated wood (Mohan *et al.*, 2001; Malakootian, *et al.*, 2008), stones from plum, apricot, cherry and dates (EL-Hendawi *et al.*, 2001) are examples of such bio-adsorbent materials. Tea waste, a widely available and cheap bio-adsorbent in many countries, has exhibited reasonable adsorption of heavy metal ions in aqueous solutions (Cay *et al.*, 2004; Amarasingha and Williams, 2007; Nouri *et al.*, 2009). In this paper, the adsorption of Cu and Cr ions from aqueous solutions by tea residue (the residue of the tea after being brewed), namely the Lahijan variety cultivated in the Northern regions of Iran, was investigated. The study was done at the fluid mechanic laboratory in Tehran from October 2009 to July 2010.

## MATERIALS AND METHODS

### *Adsorbent preparation*

Natural Lahijan tea, produced by Ata Factory of Lahijan in Iran, was initially brewed and then the tea residue was washed with hot distilled water until the water became colorless. Then the tea residue was then dried out in a dryer for 2 h at 130 °C and ultimately grounded to the appropriate size.

### *Wastewater solution preparation*

Wastewater solution was prepared by dissolving precisely measured quantities of copper nitrate ( $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ ) and potassium chromate ( $\text{K}_2\text{CrO}_4$ ) in distilled water to obtain various concentrations of heavy metal ions in aqueous solutions.

### *Batch adsorption tests*

Batch adsorption tests were conducted by mixing known quantities of tea residue prepared as indicated above and 20 mL of solutions of known heavy metal concentrations. Heavy metal ion concentrations for  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  were within the range of 0.1 to 0.5 Molar, and for  $\text{K}_2\text{CrO}_4$  were within the range of 0.001 to 0.007 g/L in the respective solutions. A mixer was used to mix the mixture at the speed of 180 rpm for 20 min. Samples were filtered using a centrifuge at the speed of 4000 rpm for 10 min. Adsorption of heavy metal ions onto tea residue in the aqueous solutions was measured using a spectrophotometer (T80 PG Instrument) at 620 nm (blue color) and 430 nm (yellow color). Series of experiments were conducted to determine the effect of adsorbent dose and initial heavy metal ion concentration with regards to the behavior of adsorption process. A fixed

ambient temperature of 25 °C was maintained during all the experiments.

### *Effect of pH on adsorption*

Effect of pH on Cu (II) and Cr (VI) adsorption was studied by mixing 1g of adsorbent in 10 mL of known concentrations of the reference heavy metal ions at various pH values ranging from 1 to 9.

Through the controlled application of NaOH to the  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  solution and HCl to the  $\text{K}_2\text{CrO}_4$  solution, the pH of the test solutions was set at 5 and 2, respectively.

### *Isotherm experiments*

Equilibrium isotherm experiments were performed by mixing 1 g of tea residue in 20 mL of the  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  solutions for the range of initial heavy metal concentration of 0.1 to 0.4 Molar. The test solution was then mixed for 90 min using a mixer. Test samples were filtered using a centrifuge system to obtain clear solutions. Similarly, the same procedure was followed for the  $\text{K}_2\text{CrO}_4$  solutions but for a range of initial heavy metal concentration of 0.001 to 0.007 g/L and a tea residue quantity of 0.5 g, with the solution being mixed for 70 min.

## RESULTS AND DISCUSSION

### *Effect of adsorbent dose*

The adsorbing property of tea residue was tested at various concentrations of Cu (II) and Cr (VI). The adsorbate's intended concentrations in the aqueous solution were obtained by dissolving 0.5, 1, and 1.5 g of adsorbent in 20 mL of solution in a manner described above. Referring to Fig. 1, the dosage increase of tea residue from 0.5 to 1.5 g enhances the Cu ion uptake of tea residue from 20 % to 60 %. The same trend was observed when Cr (VI) solution was tested (Fig. 2). It can be observed that in all cases, equilibrium reached at lower values of q (heavy metal ion adsorbed per adsorbent unit weight).

### *Effect of initial metal ion concentration*

The adsorption percentage of Cu (II) and Cr (VI) ions in the so-prepared solutions for various initial concentrations at given pH values was measured and presented in Figs. 3, 4, 5 and 6. The results showed the percentage of heavy metal ion removal was higher for lower concentrations of Cu (II) and Cr (VI). It can be concluded that within the limitations defined in



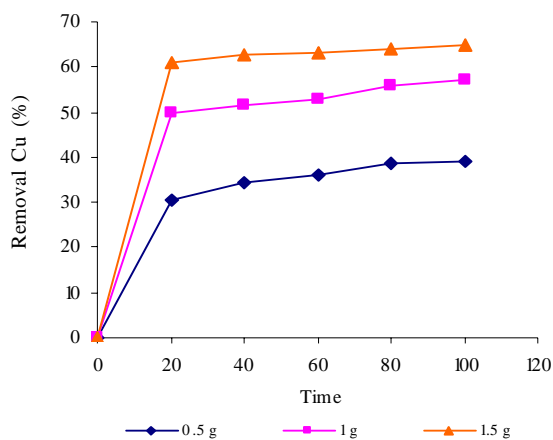


Fig. 1: Effect of adsorbent dose on adsorption of Cu (II) (20 mL Cu (NO<sub>3</sub>)<sub>2</sub>, 3H<sub>2</sub>O (0.2 Molar) and pH=2)

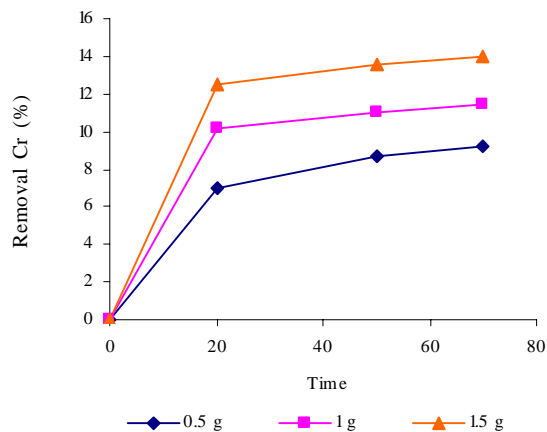


Fig. 2: Effect of adsorbent dose on adsorption of Cr (VI) (20 mL K<sub>2</sub>CrO<sub>4</sub> (0.00347g/L) and pH=2)

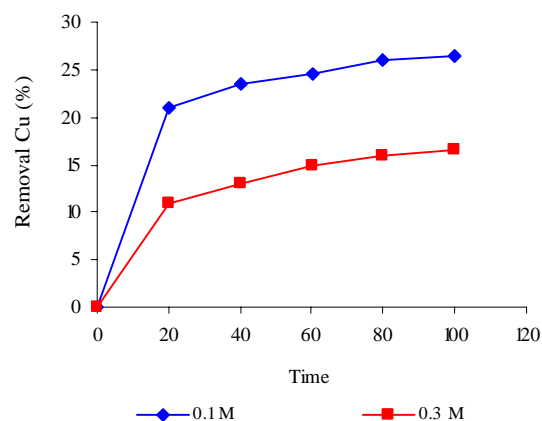


Fig. 3: Adsorption of two concentrations of Cu (II) ion (20 mL Cu (NO<sub>3</sub>)<sub>2</sub>, 3H<sub>2</sub>O + 1 g tea residue and pH=real)

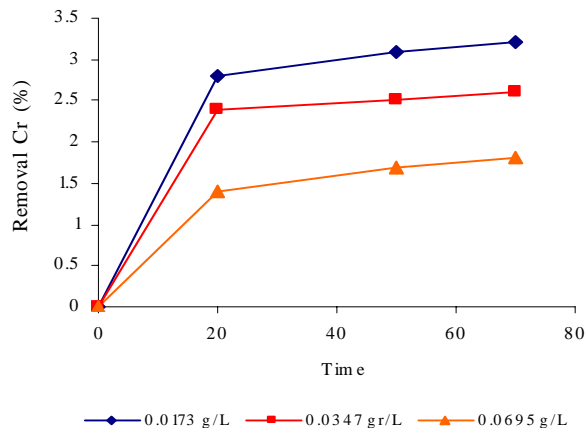


Fig. 4: Adsorption of different concentrations of Cr (VI) ion (20 mL K<sub>2</sub>CrO<sub>4</sub> + 0.5 g tea residue and pH=real)

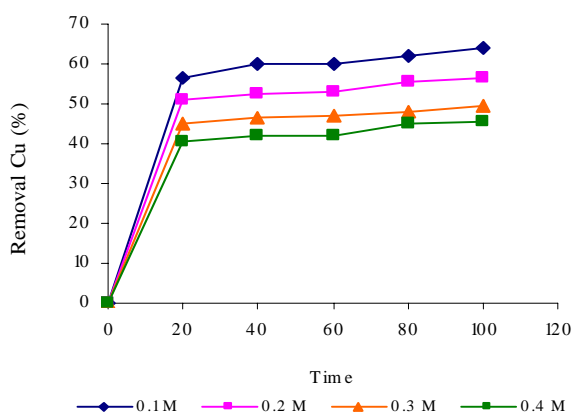


Fig. 5: Adsorption of different concentrations of Cu (II) ion (20 mL Cu (NO<sub>3</sub>)<sub>2</sub>.3H<sub>2</sub>O + 1g tea residue and pH=5)

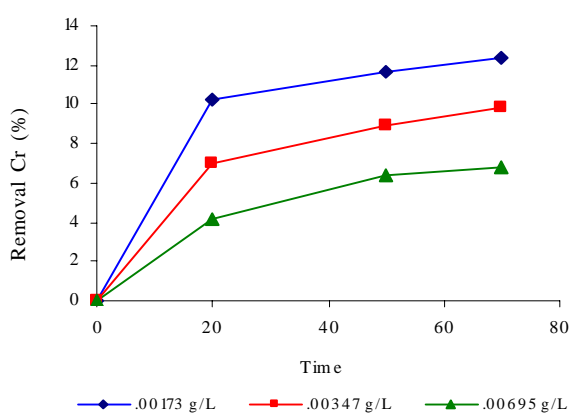


Fig. 6: Adsorption of different concentrations of Cr (VI) ion (20 mL K<sub>2</sub>CrO<sub>4</sub> + 0.5 g tea residue and pH=2)



the experiments, the adsorption property of the tea residue decreases by heavy metal ion concentration increasing.

#### Adsorption isotherms

There are several models to describe adsorption isotherm relationship (Seader and Henly, 2006). The Langmuir model (Kim *et al.*, 2004), which is based on the fact that adsorption is independent from interaction between adsorbate molecules, was used for adsorption of gases onto solids. In such cases, adsorption is maximized when the adsorbing surface is fully covered by a single layer of adsorbent.

Using Langmuir model, adsorption isotherm is expressed by the following relationship:

$$1/q_e = 1/(bq_0C_0) + 1/q_0 \quad (1)$$

Also, according to Freundlich model (Chang *et al.*, 2003) it is as follow:

$$\log q_e = \log k + (1/n) \log C_e \quad (2)$$

$$\text{Removal (\%)} = (C_0 - C_e) / C_0 \quad (3)$$

Where  $q_e$  is the amount of solute adsorbed per adsorbent unit weight at equilibrium state;  
 $q_0$  is the amount of solute adsorbed per unit adsorbent unit weight when all available sites are covered;  
 $C_0$  is the initial liquid phase;  
 $C_e$  is the residual liquid phase at equilibrium;  
 $b$  is the adsorption coefficient;  
 And  $k$  is the adsorption capacity and  $n$  is adsorption intensity.

Adsorption isotherms for Cu (II) and Cr (VI) are shown in Figs.7 and 8. Also, Table 1 was tabulated according to above indicated models. According to Freundlich and Langmuir models, the following equations can be introduced as:

$$q_e = (C_0 - C_e)V/W \quad (4)$$

Where  $V$  is the volume (mL);  $W$  is the adsorbent weight (g) and  $q_e$  is the quantity of solute adsorbed per adsorbent unit weight.

Figs.7 and 8 show the adsorption isotherms for Cu (II) and Cr (VI) onto tea residue at 25 °C. There is a relation between hydration enthalpy and interaction of solute and adsorbent. Hydration enthalpy is the energy that allows the detachment of  $H_2O$  molecules from cations and facilitates the interaction of heavy metal ions with the functional groups on tea residue. The more the cation is hydrated, the less it is attached to the adsorbent (Ricordel *et al.*, 2001; Martin-Dupoint *et al.*, 2002). Comparative data regarding the adsorption properties of other bio-adsorbent materials is presented in Table 2.

#### Influence of pH on adsorption

Influence of the given solution's pH on the adsorption property is indicated in Fig. 9. It is observed that adsorption of Cu (II) and Cr (VI) is maximized when the pH range maintained at 5-6 and 2-3, respectively. This could be explained by adsorbent surface charge change at different pH values. At a low pH value, the cations compete with the  $H^+$  ions, which present in the solution for the active sites, thus a lower adsorption results. At high pH values, adsorbent pocesses to a

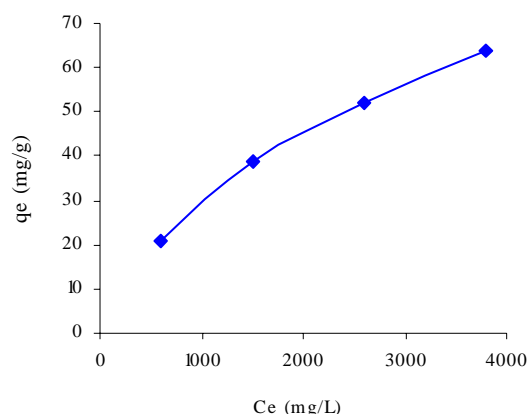


Fig. 7: Adsorption isotherms for Cu (II) ion at 25 °C

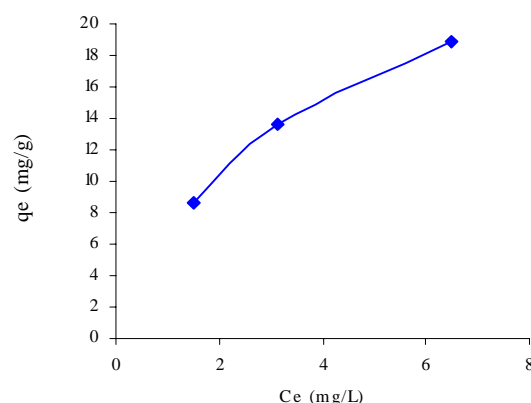


Fig. 8: Adsorption isotherms for Cr (VI) ion at 25 °C



Table 1: Langmuir and Freundlich isotherm constants for adsorption of Cu (II) and Cr (VI) ions by tea residue at 25 °C

Metal	Freundlich constants			Langmuir constants		
	K	1/n	R <sup>2</sup>	q <sub>0</sub>	B	R <sup>2</sup>
Cu (II)	0.5428	0.5772	0.996	59.523	0.83169	0.9417
Cr (VI)	7.0129	0.5419	0.9911	29.76	0.27	1

Table 2: Summary of modified plant as adsorbents for the removal of Cu (II) and Cr (VI) ions from aqueous solution

Removal ions	Adsorbent	Modifying agent(s)	Q <sub>max</sub> (mg/g)	References
Cu (II)	Sugar beet pulp	Hydrochloric acid	0.15	(Pehlivan <i>et al.</i> , 2006)
Cu (II)	Saw dust (Oak tree)	Hydrochloric acid	3.60	(Argun <i>et al.</i> , 2007)
Cr (VI)	Saw dust (Oak tree)	Hydrochloric acid	1.74	(Argun <i>et al.</i> , 2007)
Cr (VI)	Groundnut husk	Sulfuric acid	11.4	(Dubey and Gopal, 2006)

higher negative charge, thereby the higher attraction for the cations occurs. For higher pH values, the cations may form complexes which ultimately precipitate; therefore, for very high pH ranges, any cited separation may not be solely attributed to the adsorption process itself (Gaikwad, 2004; Sciban *et al.*, 2006).

#### Adsorption kinetics

Adsorption kinetics reveals the uptake rate of the solute and its residence time. Results showed a rapid adsorption rate of Cu (II) and Cr (VI) followed by slower adsorption rate by tea residue. The driving force for adsorption was higher at the beginning, since the concentration difference between the bulk solution and the solid-liquid interface was initially larger. The initial rate of adsorption was diminished by elapse of time,

partly due to the saturation and slower diffusion of the solute into the adsorbent. There are several adsorption kinetics models that can be helpful in understanding the adsorption kinetics, particularly the limits imposed on the adsorption rate. The most widely used model is the Lagergren pseudo first-order model (Ho and McKay, 1999; Preetha and Viruthagiri, 2005) introduced below:

$$dq/dt = k_1 (q_e - q) \quad (5)$$

$$\log (q_e - q) = \log (q_e) - (k_1 t)/2.303 \quad (6)$$

A pseudo-second order model, which is presented by the following equations, was also proposed (Yang and Al-Duri, 2005):

$$dq/dt = k_2 (q_e - q)^2 \quad (7)$$

$$t/q = t/q_e + 1/(k_2 q_e^2) \quad (8)$$

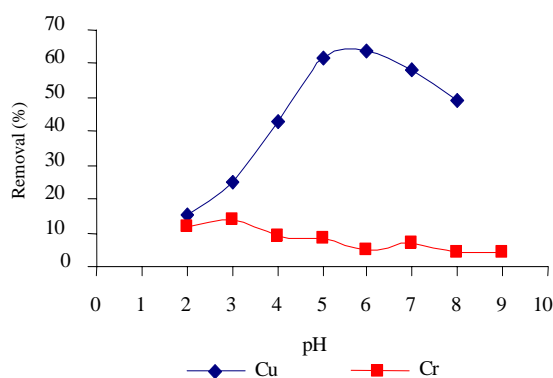


Fig. 9: Influence of pH on adsorption of Cu (II) and Cr (VI) ions onto tea residue (20 mL Cu (NO<sub>3</sub>)<sub>2</sub> · 3H<sub>2</sub>O + 1 g tea residue and 20 mL K<sub>2</sub>CrO<sub>4</sub> + 0.5 g tea residue, T= 25 °C)

Where  $q$  is the amount of metal adsorbed per unit weight of adsorbent (mg/g) at time  $t$ ;  
 $q_e$  is the amount of metal adsorbed per unit weight of adsorbent (mg/g) at equilibrium;  
 $k_1$  and  $k_2$  are the adsorption rate constants;  
 And  $h$  is the initial adsorption rate (mg/g/min) equal to  $k_1 q_e$  and  $k_2 q_e^2$ .

In this study, the respective results were fitted to pseudo- second order model and are shown in Figs.10 and 11. The kinetics parameters were calculated and presented in Tables 3 and 4. The initial adsorption rate ( $h$ ), increased by the increase of Cu (II) initial solution concentration. In addition, the rate constants decreased with the solution concentration increasing. Here again,



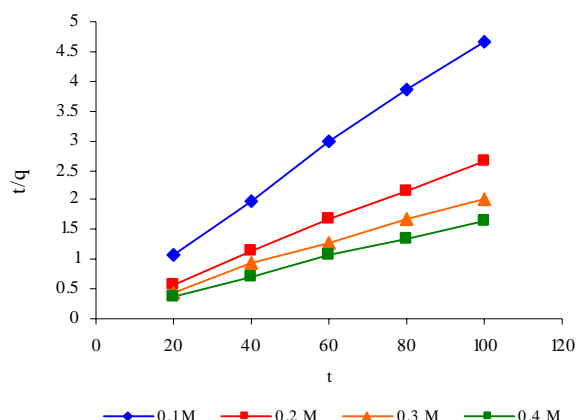


Fig. 10: Pseudo-second order model for adsorption of Cu (II) ion by tea residue mixed with 20 mL of solution at 25 °C

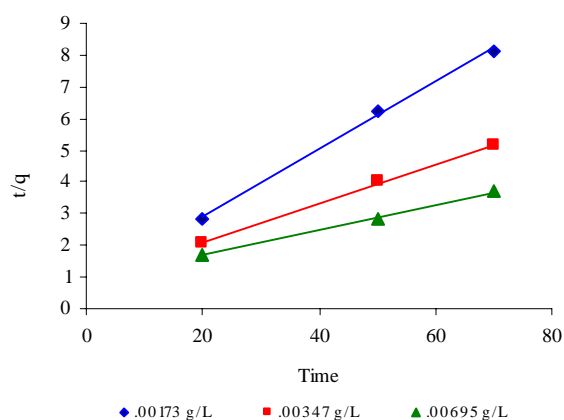


Fig. 11: Pseudo-second order model for adsorption of Cr (VI) ion by tea residue mixed with 20 mL of solution at 25 °C

Table 3: Second order kinetic parameters for adsorption of Cu (II) ion by tea residue

Metal	Initial solution concentration (mg/g)	Initial adsorption rate $h$ (mg/g/min)	Rate constant $k_2$ (g/mg/min)	Amount adsorbed experimental $q_{exp}$ (mg/g)	Amount adsorbed predicted, $q_{pre}$ (mg/g)	$R^2$
Cu (II)	0.1	5.3908	0.01116	21.38	21.9	0.998
Cu (II)	0.2	9.708	0.0063	37.833	39.06	0.998
Cu (II)	0.3	8.888	0.0033	49.63	51.87	0.994
Cu (II)	0.4	13.495	0.00341	60.827	62.89	0.997

Table 4: Second order kinetic parameters for adsorption of Cr (VI) ion by tea residue

Metal	Initial solution concentration (mg/g)	Initial adsorption rate $h$ (mg/g/min)	Rate constant $k_2$ (g/mg/min)	Amount adsorbed experimental $q_{exp}$ (mg/g)	Amount adsorbed predicted, $q_{pre}$ (mg/g)	$R^2$
Cr (VI)	0.00173	1.349	0.015	8.58	9.43	0.9984
Cr (VI)	0.00367	1.362	0.006	13.6	14.7	0.9974
Cr (VI)	0.006951	1.410	0.003	18.9	20.40	0.9974

the driving force for the adsorption was the difference between the heavy metal ion concentration in the solution and that on the solid-liquid interface, which is evidently larger at higher concentrations of heavy metal ions. Hence, in compliance with the observed results, at higher concentrations of the solutions, higher initial adsorption rates prevailed.

## CONCLUSION

Tea residue is a cheap and readily available natural waste which can be used for obviation of Cu (II) and Cr (VI) ions from wastewater. In adsorption kinetics, the initial rate of adsorption is detrimental with respect

to time. Regarding the kinetics of adsorption and according to the obtained results, the utilized pseudo-second order model provided a good approximation for the kinetics of Cu (II) and Cr (VI) ion adsorption onto tea residue. It was indicated that the equilibrium isotherms were attained after 100 min. Maximum adsorption of Cu (II) and Cr (VI) ions were registered at pH values of 5 and 2, respectively. It was also indicated that the adsorption rate and the adsorption capacity were influenced by the adsorbent's initial dose concentration in the aqueous solution. According to the results, it can be deduced that the physical and chemical properties of the adsorbent, such as surface



area and surface charge, play a dominant role in adsorption characteristics of bio-adsorbent materials, in particular tea residue. It is notable that the surface properties of the waste bio-adsorbent could be improved by various thermal and chemical treatments applied to the adsorbent prior to the process of adsorption (Vaughan *et al.*, 2001; Kumar *et al.*, 2006; Sciban *et al.*, 2006; Atafar *et al.*, 2010). Comparative results revealed that tea residue provides a better adsorbing property in comparison with other treated bio-adsorbent materials indicated in this paper as well as similar documented research.

Considering the fact that tea residue is a widely available bio-adsorbent material in Iran, further investigations concerning various applications of tea residue for off-take of harmful heavy metal ions from industrial wastewater streams, deserves much more rightful attention. It must also be recognized that the utilization of tea residue would also contribute to the so-called green biotechnology which is a fast growing area of applied scientific research.

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