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Removal of Cr(VI) and Cu(II) ions from aqueous solution by rice husk ash—column studies

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ABSTRACT

In this present study, the technical feasibility of rice husk ash for adsorptive removal of Cr (VI) and Cu(II) is examined in packed bed from aqueous solutions. The effects of different operating parameters such as height of the packed bed column (5–15 cm), flow rate of aqueous metal ion solution (10–30 ml min^{-1}), and initial metal ion concentration (10–30 mg L^{-1}) were studied. Capacity of the bed to absorb metal ions was found to increase with an increase in bed height, decrease in flow rate, and metal ion concentration. Adams–Bohart, Yoon–Nelson, and Thomas models were applied to the experimental data for the prediction of breakthrough point and also to determine the model parameters to characterize the column. The Yoon–Nelson model and Thomas model were applicable for Cr(VI) removal, and for Cu(II), Thomas model was suited.

Keywords: Adsorptive removal; Column study; Yoon–Nelson model; Thomas model; Adams–Bohart model

1. Introduction

Cr(VI) and Cu(II) presence in water bodies are of concern due to their toxicity. Cr(VI) is found to be present in mining, cement, steel, paint, and tannery industry, whereas Cu(II) is found to be present in mining, brass manufacture, petroleum refining, and electroplating industries. Cr(VI) and Cu(II) have been reported to be toxic to animals and humans, and they are known to be carcinogenic in nature [1]. Copper is an essential micronutrient element of several proteins and enzymes. Deficiency of copper causes demineralization of bones, anemia, fragility of arteries, discoloration of skin and hair. However, high concentrations (above 0.05 mg L^{-1}) in drinking water may lead to accumulation of copper in liver and lenticular nucleus of brain and may cause hepatic cirrhosis and brain necrosis [2].

Excessive accumulation of Cr(VI) in humans may cause ulcerations, dermatitis, and allergic skin reactions. The permissible limit of Cu(II) in drinking water is 1.5 mg L^{-1} and the permissible limit of Cu(II) in industrial effluent is 3 mg L^{-1} [3]. The concentration of Cr(VI) in industrial waste water remains in the range from 0.5 to 270 mg L^{-1} [4]. According to EPA the maximum tolerance limit of Cr(VI) in drinking water is 0.1 mg L^{-1} [5].

Large quantities of Cr(VI) and Cu(II) are exposed to the environment by the untreated disposal of industrial wastewater. This wastewater can be treated by different removal technologies to attain the permissible limit of them. The common effective technologies

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for the removal of heavy metal from wastewater are membrane separation, electrochemical precipitation, ion exchange, reverses osmosis, ultrafiltration, and adsorption [6,7]. A major drawback of precipitation process is sludge formation and its disposal. Ion exchange is considered a better alternative but its operating cost is high. All other techniques have higher operating cost. Among these techniques, the adsorption method is efficient, simple, cost-effective, and extensively adopted. A wide variety of agriculture and industrial waste such as rice husk, coconut shell, sawdust, shells of wheat, shells of rice, wheat straw, fly ash, red mud, and clarified sludge can be used as low-cost adsorbent.

Metal ions, Cr(VI) and Cu(II), were chosen for the adsorption studies in continuous column operation with regard to their wide use in industry and also their potential pollution impact. Cr(VI) from electroplating and similar industries was reported to be toxic and also carcinogenic and also bio-accumulated into flora and fauna, thus creating ecological problems [8]. In the copper-cleaning, copper plating, and metal processing industries, Cu(II) concentrations approach 100–120 mg L^{-1}; this value is very high in relation to the water quality [9]. Mazumder et al. [2] reported the treatment of effluent from rinsing tank of electroplating plant (chrome plating and bronze plating) from an industrial establishment in Kolkata, India, where the wastewater contain chromium 8.6–24.8 mg L^{-1} and copper 5.0–6.5 mg L^{-1} from the chrome and bronze platting unit, respectively. In this study, rice husk ash was used as the low-cost adsorbent for the removal of Cr(VI) and Cu(II) ions from aqueous solution in continuous flow. Initially, the effects of operating parameter are investigated and modeling is carried out using well-known models.

2. Experimental

2.1. Preparation of adsorbent

Initially, rice husk was collected from a local rice mill near Kolkata, West Bengal, India, and consequently, rice husk was washed with distilled water and then sun dried for 3–4 d. Then, the dried rice husk was burned 550°C in a furnace to prepare ash. The ash was sieved to pass through a −44 + 52 mesh (250–350 μm) screen. After sieving, the rice husk ash was kept by in an air tight container.

2.2. Metal solution

1,000 mg L^{-1} of Cu(II) solution was obtained by dissolving 3.929 g of copper(II) sulfate pentahydrate ($CuSO_4{\cdot}5H_2O$) in double distilled water. Similarly, 1,000 mg L^{-1} of Cr(VI) solution was obtained by dissolving 2.83 g of potassium dichromate ($K_2Cr_2O_7$) in double distilled water. Finally, stock solution of Cu(II) and Cr(VI) was diluted to obtain required standard solution containing 5, 10, 15, 20 mg L^{-1} of Cu(II) and Cr(VI). Throughout the experiment, the glass wares were washed with 20% nitric acid followed by washing with the distilled water and then the glass wares were dried in oven at 383 K.

2.3. Reagents and instrument used

All the reagents used in this study were of analytical grade and obtained from E. Merck Limited, Mumbai, India. pH meter (Multi 340i, WTW, German) was used to measure pH of the solution. UV-Spectrophotometer (Dr 5000, HACH, USA) was used to determine the Cr(VI) and Atomic Absorption Spectrophotometer [AA 240 VARIAN, Australia] was used to determine Cu(II) content in standard and treated solution after adsorption experiment.

2.4. Analysis of metal ions

The concentration of chromium(VI) ions in the standard and treated solutions after adsorption was determined using a UV-spectrophotometer (Dr 5000, HACH, USA) using 1,5 diphenyl-carbazide as the complexing agent as recommended [10]. The absorbance of the purple colored solution was read at 540 nm after 10 min. The calibration curve was prepared first with known strengths of dilute Cr(VI) solutions prepared from stock solutions. Cu(II) ion before and after the adsorption was measured using atomic absorption spectrophotometer [AA 240 VARIAN, Australia].

2.5. Adsorption experiments

The effect of pH (2–8) on the adsorptive removal of the metal ion is carried out in batch mode. The column experimental set up consists of 1.8 cm diameter glass column and 50 cm height. Three such columns, in parallel, homogeneously packed with rice husk ash. The schematic diagram of the experimental setup is shown in Fig. 1. The continuous down flow was maintained using a multichannel peristaltic pump (Cole-Parmer, model-7535-04, USA). A constant head was maintained above the bed to avoid channeling of the liquid. The effects of different flow rates, bed heights, and initial metal ion concentrations on breakthrough curves were studied. Initial pH of the metal

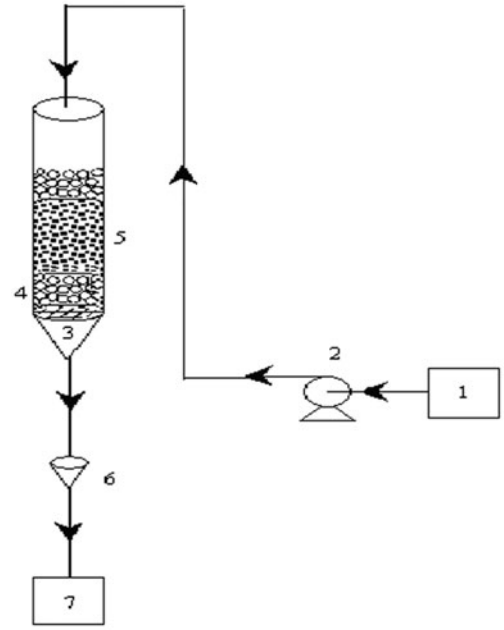

Fig. 1. Schematic diagram of experimental set up of column study.
Notes: (1) reservoir tank with metal solution; (2) peristaltic pumps; (3) porous sheet; (4) glass beads; (5) adsorbents; (6) filter; (7) treated effluent storage.

ion solutions was adjusted by adding 0.1 N HCl. Samples were collected for analysis from the bottom of the columns at desired interval of time.

3. Results and discussion

3.1 Characterization of the adsorbent

Scanning electron microscopic image of the adsorbent was carried out to see its surface structure. SEM images of adsorbent are represented in Fig. 2. From the SEM images, it can be clearly seen that the surface is irregular in nature. The surface structure of rice husk ash was analyzed using an X-ray diffractometer (Model No. XRD 3000P, Seifert, Germany) and shown in Fig. 3. Composition of rice husk ash is shown in Table 1. The physical characteristics of the rice husk ash are shown in Table 2. The point of zero charge was determined by solid addition method [11].

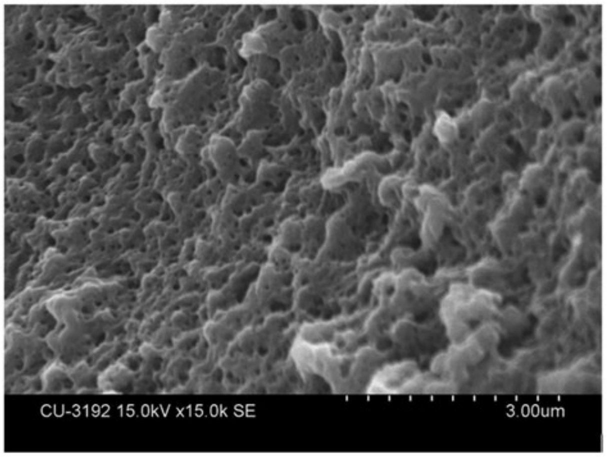

Fig. 2. Scanning electron micrograph (SEM) of rice husk ash.

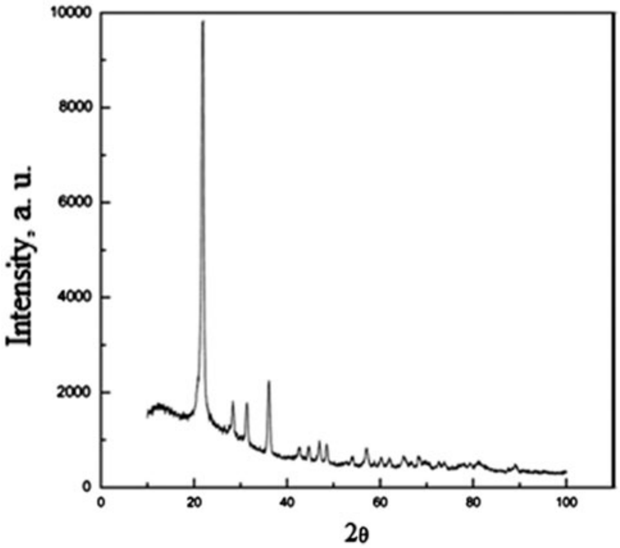

Fig. 3. XRD study of rice husk ash.

3.2. Effect of initial pH

To investigate the effect of pH, batch adsorption studies were conducted in the pH range of 2–8.The effect of optimum pH had an important role in metal ions adsorption. Cr(VI) exists in different forms in aqueous solution, and the stability of this form is dependent on the pH of the solution. The percentage removal of Cr(VI) reached a maximum value at pH 2. The $HCrO_4^-$ form is more stable between pH 1.0 and 4.0. The H_2CrO_4 form is stable in low pH range of the solution but its concentration decreases with increasing pH of the aqueous solution. The CrO_4^- form of Cr(VI) is more stable in higher pH range of the solution. Hence, increasing the pH of the Cr(VI) solution will shift the concentration of $HCrO_4^-$ to the another forms CrO_4^- and $Cr_2O_7^{2-}$. At low pH of metal solution, the adsorbent surface surrounded by the hydronium ions which enhance the adsorption of

Table 1
Chemical composition of rice husk ash

Constituent	Fe_2O_3	Al_2O_3	CaO	MgO	SiO_2	Na_2O	K_2O
Percentage weight %	0.6	0.3	1.4	0.5	84.3	0.4	0.2

Table 2
Characterization of the adsorbent

Characteristics	Blackberry leaves
Particle size (μm)	250–350
Mean diameter (m)	3.02×10^{-4}
Surface area ($m^2\ g^{-1}$)	57.5
Bulk density ($kg\ m^{-3}$)	0.96
Point of zero charge, pH_{ZPC}	8.5

Cr(VI) on the adsorbent. Hence, in the figure it is seen that there is a steep rise in adsorption which indicates maximum amount of removal of Cr(VI) when the pH decreases to 2. Similar results were obtained by other researchers [6].

At very low pH, no significant amount of Cu(II) removal occurred. At low pH, the adsorbent surface is covered by hydronium ions; hence, Cu(II) has to compete with hydronium ions for the active sites of the adsorbent. The maximum amount of Cu(II) adsorption on the adsorbent occurred at pH 6. Similar results were obtained by other researchers [10].

3.3. Influence of the flow rate

The influence of the sample flow rate on the adsorption of Cr(VI) and Cu(II) was examined at the flow rate of 10, 20 and 30 mL min^{-1}. The breakthrough curves are shown in Figs. 4 and 5, respectively. This graph is a plot of dimensionless ratio of concentration (C_t/C_0) vs. time. From this graph, it was shown that breakthrough occurred more rapidly as the flow rate increases. At lower flow rate, breakthrough occurred more slowly than at higher flow rate. At higher flow rate, the metal ion concentration in the effluent was high. This behavior occurred because at higher flow rate, the solvent had less contact time with the solute in the column. At lower flow rate, the solvent had more time to contact with the column and this behavior produced higher removal of metal ions in column [12,13]. It was found that best adsorption capacity was found at 10 ml min^{-1} flow rate for the removal of Cr (VI) and Cu(II), respectively.

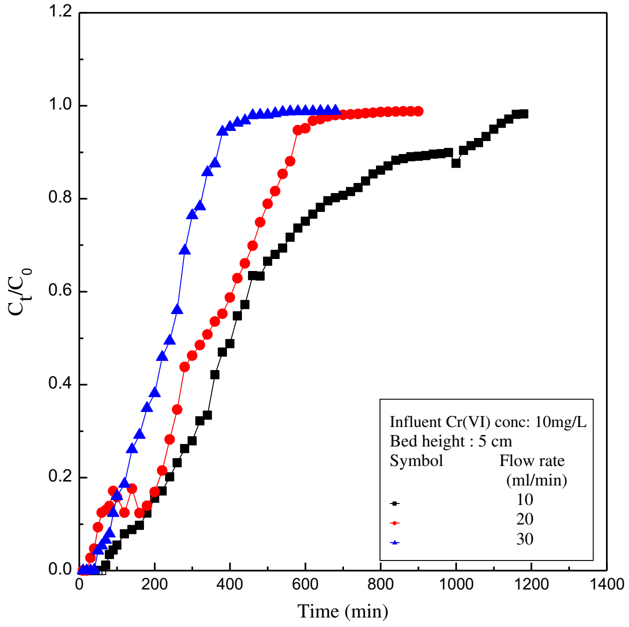

Fig. 4. Effect of flow rate on the adsorption of Cr(VI) at constant bed height and influent metal ion concentration.

3.4. Influence of influent concentration of metal ions

The influent metal ion concentration has an important role on breakthrough curve. The breakthrough curve is shown in Figs. 6 and 7 for Cr(VI) and Cu(II) removal, respectively. From this graph, it was shown that adsorption capacity of the packed bed increased with the decrease in metal ion concentration [14]. While at higher influent metal ion concentration, the adsorption capacity of packed bed decreased. At very high influent metal ion concentration, adsorption capacity decreased due to saturation of active saturation surfaces. At lower influent metal ion concentration, the adsorption capacity increased because the metal ions would react with active binding sites and this behavior produced maximum adsorption. From the breakthrough curve shown in Figs. 6 and 7, it was found that adsorption capacity increased at low Cr(VI) and Cu(II) concentration, respectively.

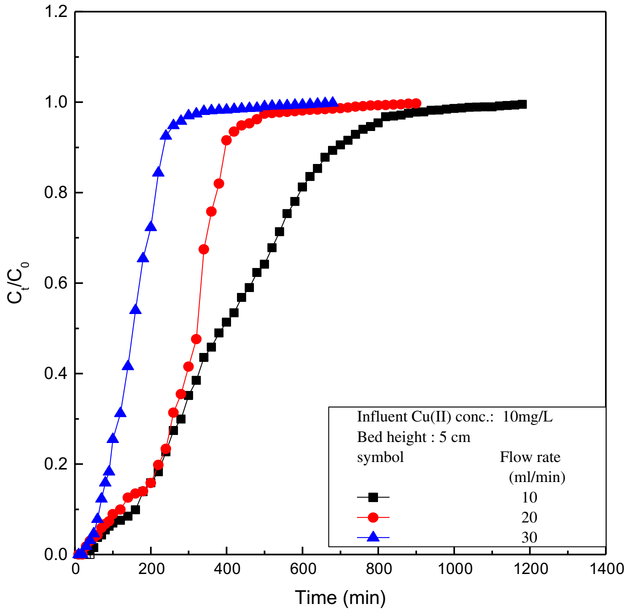

Fig. 5. Effect of flow rate on the adsorption of Cu(II) at constant bed height and influent metal ion concentration.

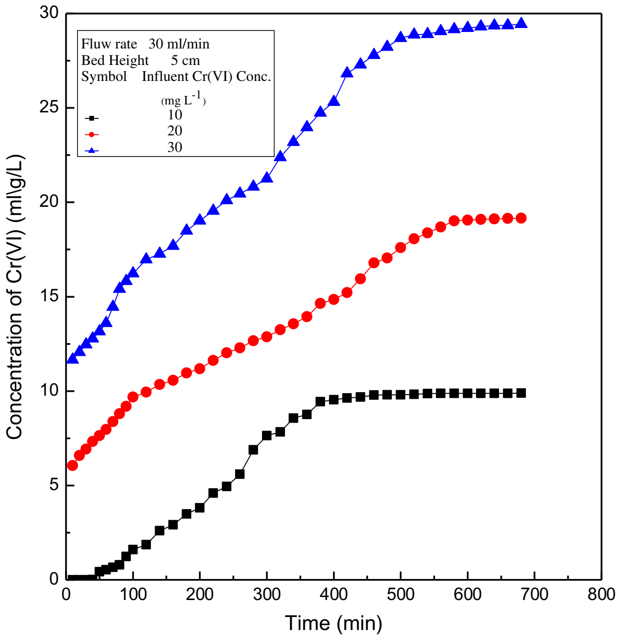

Fig. 6. Effect of influent metal ions concentration for the removal of Cr(VI) at constant bed height and flow rate.

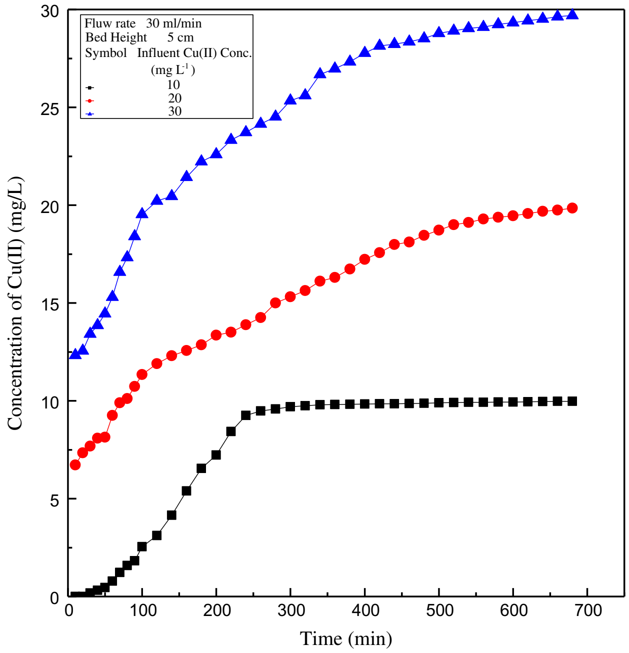

Fig. 7. Effect of influent metal ions concentration for the removal of Cu(II) at constant bed height and flow rate.

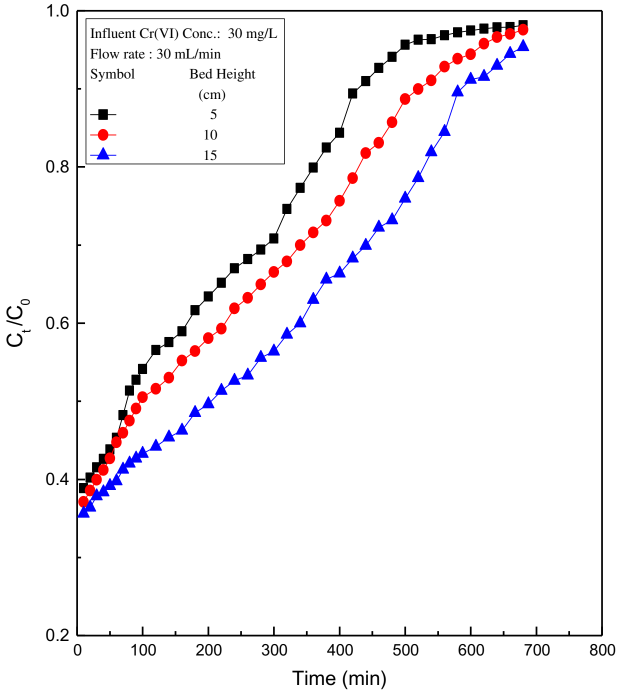

Fig. 8. Effect of bed height of column for the removal of Cr(VI) at constant flow rate and influent metal ion concentration.

3.5. Influence of bed height in packed column

To investigate the influence of bed height on the metal adsorption process, the column studies were examined at bed height of 5, 10, and 15 cm. The breakthrough curve is shown in Figs. 8 and 9. From this graph, it was shown that the breakthrough occurred more rapidly at lower bed height. At higher bed height, breakthrough occurred slowly than the lower

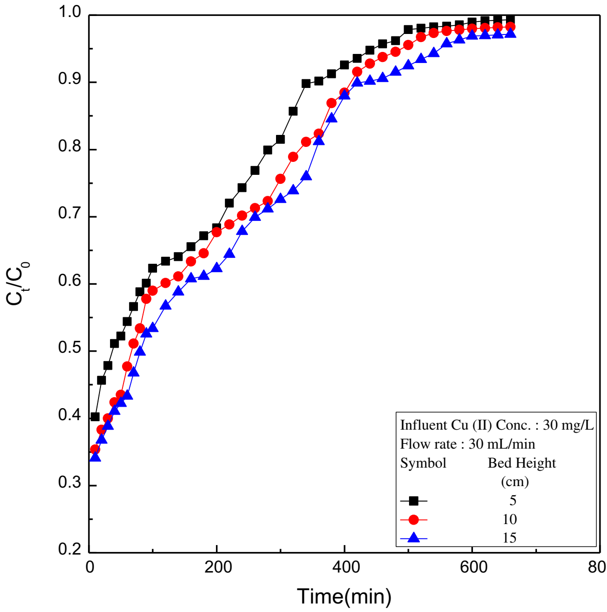

Fig. 9. Effect of bed height of column for the removal of Cu(II) at constant flow rate and influent metal ion concentration.

bed height because at higher bed height aqueous solution got more binding sites of the adsorbent and this behavior produced higher removal of metal ions in the column [15,16]. Maximum removal of Cr(VI) and Cu(II) occurred at bed height of 15 cm.

3.6. Adsorption study of Cr(VI) and Cu(II) on rice husk ash

The effect of adsorption capacity of rice husk ash for the removal of Cr(VI) and Cu(II) was examined. The breakthrough curve was shown in Fig. 10. From the graph, it was seen that the breakthrough curve was more steeper for Cu(II) ions than Cr(VI) ions which inferred that adsorption of Cr(VI) was higher than Cu(II) at a constant bed height and flow rate.

3.7. Modeling of column study

Maximum adsorption capacity of the adsorbent in a fixed bed column is calculated by following equations:

$$q_e = \frac{v}{1000w} \int_{0}^{t_{\text{total}}} (C_0 - C_t)\, dt \tag{1}$$

$$q_{e,\max} = \frac{q_{\text{total}}}{w} \tag{2}$$

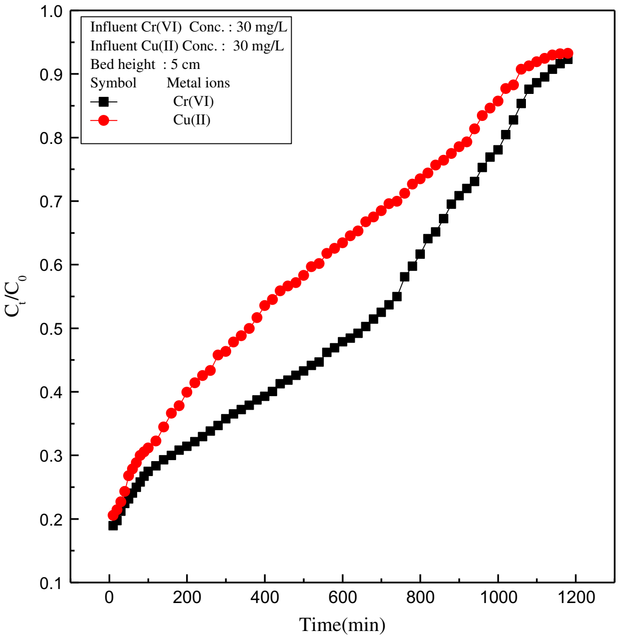

Fig. 10. Comparison of adsorptive removal of Cr(VI) and Cu(II).

3.7.1. Adams–Bohart model [17]

The data obtained from the column studies were investigated to calculate the adsorption capacity and adsorption rate constant developed by Bohart and Adams. The Bohart and Adams model is widely used in column studies. The expression given by Bohart and Adams for an adsorption column is given as follows:

$$\ln\left(\frac{C_t}{C_0}\right) = k_{AB} C_0 t - k_{AB} N_0 \left(\frac{z}{v}\right) \tag{3}$$

The values of adsorption capacity N_0 and kinetic constant k_{AB} can be obtained from the intercept and slope of the linear plot of $\ln(C_t/C_0)$ vs. time. The values of k_{AB}, N_0 and other correlation coefficients are tabulated in Table 3 for Cr(VI) and Cu(II) removal, respectively. From the tabulated data, it is seen that the rate constant (k_{AB}) and adsorption capacity (N_0) are dependent on flow rate and metal ion concentration. It is observed that the value of rate constant (k_{AB}) increases with the increasing flow rate.

3.7.2. Yoon–Nelson model [18]

Yoon–Nelson model is based on the assumption that the rate of decrease in the probability of

Table 3
Bohar–Adams model parameters

Flow rate, v (ml min^{-1})	Influent concentration, C_0 (mg L^{-1})	Bed height (cm)	Rate constant, k_{AB} (ml min^{-1} mg^{-1})	Saturation concentration, N_0 (g L^{-1})	Correlation coefficient, R^2
For Cr(VI) removal					
10	10	5	0.000876	8.92	0.8302
20	10	5	0.00066	17.11	0.7832
30	10	5	0.00181	14.47	0.6051
10	20	5	0.000304	16.61	0.8462
20	20	5	0.000305	33.55	0.8819
30	20	5	0.000205	34.60	0.9623
10	30	5	0.0000433	68.27	0.9429
20	30	5	0.00009	62.89	0.9267
30	30	5	0.00013	44.45	0.9236
10	30	10	0.0000873	36.71	0.8948
20	30	10	0.00009	46.66	0.9693
30	30	10	0.0001133	53.44	0.92365
10	30	15	0.0000883	27.53	0.9172
20	30	15	0.0000763	39.79	0.9633
30	30	15	0.0000553	36.60	0.9801
For Cu(II) removal					
10	10	5	0.00088	8.51	0.9341
20	10	5	0.00095	15.34	0.941
30	10	5	0.00282	9.38	0.9129
10	20	5	0.00035	13.72	0.8324
20	20	5	0.00025	37.88	0.8963
30	20	5	0.00015	41.52	0.9801
10	30	5	0.00006	43.51	0.9363
20	30	5	0.0001	55.96	0.8986
30	30	5	0.00016	32.26	0.9307
10	30	10	0.00001	28.86	0.8629
20	30	10	0.0001	42.72	0.9308
30	30	10	0.00006	50.19	0.9658
10	30	15	0.0001217	18.49	0.89416
20	30	15	0.0000907	33.71	0.88669
30	30	15	0.000059	37.69	0.89216

adsorption for each adsorbate molecule depends on the adsorbate adsorption. The expression given by Yoon–Nelson for column studies is given as follows:

$$\frac{C_t}{C_0 - C_t} = \exp(k_{YN}t - k_{YN}\tau) \tag{4}$$

The values of τ and k_{YN} can be obtained from the intercept and slope of the linear plot of $\ln(C_t/C_0 - C_t)$ vs. time. The relationship between the sampling time and breakthrough fraction was investigated at different influent concentration and flow rate. Linear regression results and the values of R^2 are tabulated in Table 4 for Cr(VI) and Cu(II) removal, respectively. It was observed that the time required for 50% breakthrough (τ) decreased with increasing flow rate. From Table 4, it is seen that the value of rate constant k_{YN} increases with the increasing flow rate and the value of τ decreases with the increasing flow rate for Cr(VI) removal through rice husk ash. But it is observed that the value of τ does not decrease with the increasing flow for Cu(II) removal through rice husk ash. Hence, it can be concluded that the Yoon–Nelson model is appropriate to

Table 4
Yoon–Nelson model parameters

Flow rate, v (ml min^{-1})	Influent concentration, C_0 (mg L^{-1})	Bed height (cm)	Time, τ (min)	Rate constant, k_{YN} (min^{-1})	Correlation coefficient, R^2
For Cr(VI) removal					
10	10	5	384.5538	0.0105	0.9248
20	10	5	337.587	0.0086	0.8258
30	10	5	211.102	0.0214	0.6884
10	20	5	316.4177	0.0079	0.9007
20	20	5	104.7704	0.0061	0.976
30	20	5	118.85	0.0071	0.9769
10	30	5	623.261	0.0019	0.9683
20	30	5	318.25	0.0080	0.9303
30	30	5	79.6755	0.0071	0.9396
10	30	10	1,014.3437	0.0032	0.9397
20	30	10	566.8648	0.0037	0.9878
30	30	10	171.4827	0.0029	0.9365
10	30	15	1,198.64	0.0031	0.9541
20	30	15	723.863	0.0030	0.9850
30	30	15	200.392	0.0029	0.9887
For Cu(II) removal					
10	10	5	362.8691	0.0107	0.9636
20	10	5	330.4135	0.0113	0.9608
30	10	5	139.5022	0.03293	0.9462
10	20	5	251.6705	0.00941	0.8900
20	20	5	293.7375	0.00766	0.9413
30	20	5	96.8257	0.00637	0.9878
10	30	5	335.4972	0.00362	0.9645
20	30	5	240.1722	0.00476	0.9326
30	30	5	59.7003	0.01038	0.9421
10	30	10	5.647	0.000835	0.9810
20	30	10	64.054	0.0011	0.9836
30	30	10	200.3703	0.00189	0.9889
10	30	15	59.7003	0.0103	0.9421
20	30	15	335.497	0.00362	0.9644
30	30	15	300.828	0.00476	0.9326

describe the column operation of Cr(VI) removal than the Cu(II) removal through the continuous column operation.

3.7.3. Thomas model [19]

Thomas model is a widely used model which explains the fixed bed column performance. Thomas model assumes minimum axial dispersion of adsorbate within the adsorbent. The mathematical form of Thomas model is given as follows:

$$\frac{C_t}{C_0} = \frac{1}{1 + \exp\left[\left(\frac{k_{Th} q_e x}{Q}\right) - k_{Th} C_0 t\right]} \tag{5}$$

The linear form of the Thomas model is given as follows:

$$\ln\left(\frac{C_t}{C_0} - 1\right) = \left(\frac{k_{Th} q_e x}{Q}\right) - k_{Th} C_0 t \tag{6}$$

The calculated Thomas model parameters are listed in Table 5 for Cr(VI) and Cu(II), respectively. The values of correlation coefficient are listed in Table 5. The result indicated that they were all good fits with correlation coefficient (R^2). From this table, it is seen that the value of calculated $q_{e,\max}$ from Thomas model is higher than the value of experimental $q_{e,\max}$. It is observed that the calculated $q_{e,\max}$ increases as the adsorbent dose increases but the value of K_{Th}

Table 5
Thomas model parameters

Flow rate, v (ml min^{-1})	Influent concentration, C_0 (mg L^{-1})	Bed height (cm)	k_{Th} (ml min^{-1} mg^{-1})	$q_{e,\max(\mathrm{cal})}$ (mg g^{-1})	$q_{e,\max(\mathrm{exp})}$ (mg g^{-1})	Correlation coefficient, R^2
For Cr(VI) removal						
10	10	5	1.05	6.15	3.2492	0.9248
20	10	5	0.862	10.717	5.3095	0.8258
30	10	5	2.14	10.080	5.7788	0.6884
10	20	5	0.395	10.0452	5.7003	0.9007
20	20	5	0.4045	20.2067	10.6857	0.9303
30	20	5	0.3565	11.2722	6.4368	0.9769
10	30	5	0.0663	29.8148	15.6360	0.9683
20	30	5	0.1437	25.7277	14.1708	0.9532
30	30	5	0.2383	9.95444	4.8632	0.9397
10	30	10	0.1	27.185	13.5253	0.9396
20	30	10	0.143	26.3533	15.6734	0.9879
30	30	10	0.205	12.1206	7.0685	0.9365
10	30	15	0.103	19.087	10.562	0.9541
20	30	15	0.123	23.5925	12.376	0.9850
30	30	15	0.096	9.7	5.095	0.9875
For Cu(II) removal						
10	10	5	1.7	3.6253	3.0792	0.9636
20	10	5	1.13	10.5730	5.3252	0.9608
30	10	5	3.29	6.6490	3.896	0.9462
10	20	5	0.45	8.3534	4.6133	0.8900
20	20	5	0.383	18.9039	10.6943	0.9413
30	20	5	0.3185	9.2214	4.7419	0.9878
10	30	5	0.1207	15.9760	8.7454	0.9645
20	30	5	0.1587	22.8735	13.2133	0.9327
30	30	5	0.346	8.5286	4.654	0.9421
10	30	10	0.1393	16.4687	8.9589	0.9137
20	30	10	0.137	23.9253	12.8134	0.9643
30	30	10	0.1513	11.6041	6.3617	0.9780
10	30	15	0.146	12.375	7.026	0.9346
20	30	15	0.12	19.7425	10.772	0.9344
30	30	15	0.10	11.819	6.646	0.9163

decreases for the same. Moreover it is seen that the value of $q_{e,\max}$ decreases as the flow rate increases. Hence, it can be concluded that the experimental data are appropriate according to the theoretical concept and Thomas model is the best fit model to describe the column operation.

4. Conclusions

Continuous column studies have been carried out for the removal of Cr(VI) and Cu(II) ions using rice husk ash as adsorbent.

(1) This study shows the adsorption capacity of rice husk ash for the removal of Cr(VI) and Cu(II) from the aqueous solution. From the experimental results, it is seen that the capacity of adsorbent is very high for the removal of Cr(VI) and Cu(II). This inexpensive adsorbent finally produces a cost-effective treatment through this column study.

(2) The capacity of the adsorbent depends on the pH and flow rate of the metal solution, influent metal ion concentration, and the bed height of the packed column. The removal process is effective at pH 2 for Cr(VI) removal and at pH 6 for Cu(II) removal.

(3) Maximum amount of metal removals is seen in lower flow rate. As the flow rate increases, the breakthrough curve becomes steeper and the breakthrough time decreases. This occurs due to the less contact time of metal ions with the adsorbent.

(4) Adsorption capacity of the packed column increases at a decreasing rate of metal ion concentration for a fixed bed height. Moreover, the adsorption capacity increases with increase in the adsorbent dose.
(5) Adams–Bohart model, Yoon–Nelson model, and Thomas model are used to analyze the experimental data. Thomas model and Yoon–Nelson model fit well with the experimental data to describe the column performance but unsatisfactory performance of the column study obtains from Adams–Bohart model for Cr(VI) removal. From the experimental data it is seen that Thomas model is the best fit model for Cu(II) removal.

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