

# Removal of potentially toxic elements from aqueous solutions and industrial wastewater using activated carbon

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## ABSTRACT

Water contamination with potentially toxic elements (PTEs) has become one of the key issues in recent years that threatens human health and ecological systems. The present study is aimed at removing PTEs like cadmium (Cd), chromium (Cr), copper (Cu) and lead (Pb) from aqueous solutions and industrial wastewater using activated carbon (AC) as an adsorbent through different batch and column experiments. Results demonstrated that the removal of PTEs from aqueous solutions was highly pH dependent, except for Cr, and the maximum removal (>78%) was recorded at pH 6.0. However, maximum Cr removal (82.8%) was observed at pH 3.0. The adsorption reached equilibrium after 60 min with 2 g of adsorbent. Coefficient ( $R^2$ ) values suggested by the Langmuir isotherm model were 0.97, 0.96, 0.93 and 0.95 for Cd, Cr, Cu and Pb, respectively, indicating the fit to this model. In column experiments, the maximum removal of PTEs was observed at an adsorbent bed height of 20 cm with the optimal flow rate of 3.56 mL/min. Furthermore, PTEs removal by AC was observed in the order of Cu > Cd > Pb > Cr. Findings from this study suggest that AC could be used as a promising adsorbent for simultaneously removing several PTEs from wastewaters.

**Key words** | activated carbon, adsorptive removal, aqueous solution, industrial wastewater, potentially toxic elements

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## INTRODUCTION

Environmental pollution is one of the most severe dilemmas nowadays, especially with rapid industrialization and urbanization releasing a high quantity of potentially toxic elements (PTEs) into the aquatic environment. Among the toxic compounds found in wastewater, PTEs are of great significance due to their high toxicity even at low concentrations (Cheng *et al.* 2016). PTEs are known as eco-toxicological hazardous elements, and their chronic toxicities and accumulative capabilities in living organisms have been of great interest in recent years (Cay *et al.* 2004). Wastewater from various industrial units like glass production, paint industries, mining operations and hospitals generates PTEs such as chromium (Cr), copper (Cu),

cadmium (Cd), manganese (Mn), lead (Pb), nickel (Ni) and zinc (Zn) (Khan *et al.* 2009). PTEs can cause several ecological problems because of their toxicity, long persistence, bio-accumulation and bio-amplification in the food chain, and therefore are considered as toxic elements for aquatic flora and fauna and even for human beings at low concentrations (Tajar *et al.* 2009).

PTEs enter into the human body via drinking contaminated water, ingestion of food and inhalation of air. PTEs increase the acidity of the blood. As a result, blood draws calcium (Ca) from the bones to restore the blood pH. This phenomenon causes brittle bones (osteoporosis), which is commonly found in children and aged people.

Sequestration of these metals from industrial wastewater is essential because they seriously damage the environment (Fiol *et al.* 2006). The removal of these toxic PTEs can be done by various conventional methods including filtration, chemical precipitation, bio-sorption, complexation, chemical oxidation/reduction, electrochemical treatment, coagulation or flocculation and membrane technology (Camarillo *et al.* 2012). However, every technology has its own limitations such as incomplete removal, higher cost due to high energy demand, production of poisonous intermediate chemicals and generation of toxic sludge.

In recent years, various technologies have been employed to make use of economical and accessible organic wastes such as coconut shell, sawdust, (peanut shell, rice hull and apricot stone) (Patnuka *et al.* 2008), for the purposive removal of PTEs from aqueous solution or industrial wastewater. Adsorption is a widely acceptable treatment technique with numerous advantages and is practiced extensively in chemical as well as process industries, both for waste gas and for adsorption of PTEs from wastewater treatment (Gupta & Saleh 2013). In addition, this technique normally generates little sludge for further disposal. Therefore, the use of activated carbon (AC) in adsorption has been widely adopted for the adsorption of various PTEs from wastewater (Sarin & Pant 2006). However, no study has been done on applying AC for the simultaneous removal of PTEs from aqueous solutions and industrial wastewater so far. AC is a black, carbonaceous material like granular or powder charcoal and has highly developed porosity, internal surface area, and relatively high mechanical strength (Sahu *et al.* 2010). AC is more effectively used for the removal of PTEs from industrial wastewater and household water due to its excellent adsorption characteristics including its large surface area and larger number of pores (Sirianuntapiboon & Ungkprasatcha 2007).

Commercial AC is widely used in industrial applications for pollutant adsorption and catalytic purposes. In recent years, toxic metallic species and other organic contaminants have been removed both from liquid and gaseous phases using AC (AL-Othman *et al.* 2012). The widespread uses of AC are due to its higher efficiency in the adsorption process (Roosta *et al.* 2013). Due to its excellent adsorbent properties, AC has been enormously efficacious as a remediation solution for wastewater treatment and its recovery. In the present study, an effort has been made to study the adsorption of metal ions onto commercially available AC. The adsorption of PTEs onto AC is influenced by different working parameters such as pH, initial concentration, contact

time, column bed height, and flow rate, because adsorption of metal ions onto AC is highly dependent on these experimental conditions and the adsorbent dose (Bernard *et al.* 2013).

Thus, in this study, an effort has been made to remove various PTEs including Cd, Cr, Cu and Pb from aqueous solution as well as from industrial wastewater through batch and column experiments using AC as an adsorbent. In addition, different experimental conditions were set to identify their effects and investigate the optimum removal performance of AC.

## METHODS AND MATERIALS

### Activated carbon

Commercial AC employed in this study was obtained from Norit Nederland BV Amersfoort. The particle size of this AC ranged from 1 to 3 mm. All experiments were conducted in the laboratory of the National Centre of Excellence in Geology, University of Peshawar (Pakistan). Industrial wastewater samples were collected from the main drain receiving wastewater from all individual industrial units.

### Batch experiments

Adsorption studies were performed using the batch technique with different concentrations (5, 10 and 20 mg/L) of PTEs in aqueous solutions prepared from their respective salts (CdCl<sub>2</sub>, K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, CuSO<sub>4</sub> and Pb (NO<sub>3</sub>)<sub>2</sub>). For this purpose, different adsorbent doses (0.5, 1.0, 1.5 and 2 g) and 100 mL of aqueous PTE solution at different concentrations (5–20 mg/L) were taken in an Erlenmeyer flask and shaken at 300 rpm for different contact times (5, 10, 30 and 60 min), while the pH was maintained in the range of 2–8. The pH was adjusted using 0.1 M HCl or NaOH. The temperature was kept constant at 30 °C (Dwivedi *et al.* 2008). The same procedure was also followed for industrial wastewater. The amount of PTEs adsorbed at different conditions was calculated using Equation (1).

$$q_e = \frac{C_i - C_e}{W} \times V \quad (1)$$

where  $q_e$  (mg/g) is the amount of PTEs adsorbed;  $C_i$  and  $C_e$  are initial and final equilibrium concentrations (mg/L), respectively.  $V$  is the volume (L) of aqueous solution and  $W$  is the weight (g) of the adsorbent (Açıkıldız *et al.*

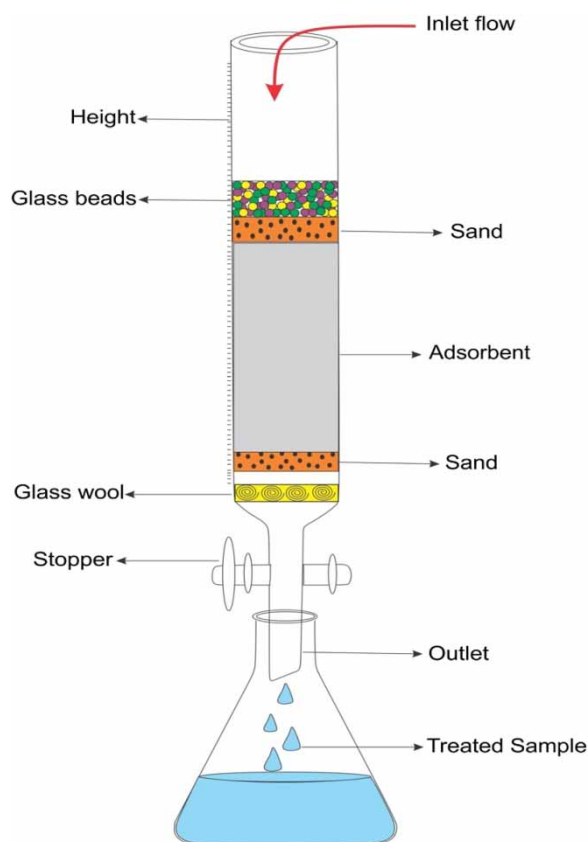
2014). PTE removal in percent (% MR) was calculated by the following equation (Equation (2)).

$$(\%MR) = \frac{C_i - C_e}{C_i} \times 100 \quad (2)$$

The concentration of PTEs both before and after the treatment was determined by atomic adsorption spectrometer (Dwivedi *et al.* 2008). The obtained adsorption data were modeled using the Langmuir isotherm model (Equation (3)), as follows:

$$\frac{C_e}{q_e} = \frac{1}{KQ_{\max}} + \frac{C_e}{Q_{\max}} \quad (3)$$

where K (1/mg) is the Langmuir adsorption constant, which is the monolayer adsorption capacity, and  $Q_{\max}$  is the maximum adsorption capacity in mg/g. These values can be obtained by plotting  $C_e/q_e$  against  $q_e$  (Shama & Gad 2010). The correlation coefficient  $R^2$  has been used to get the best fit linear equation (Freundlich 1906).



**Figure 1** | Diagrammatic presentation of the column used for sorption experiments.

## Column experiments

A glass column with an internal diameter of 2.8 cm and height of 40 cm was used for the column adsorption experiment, as shown in Figure 1. Glass wool was used at the bottom of the column for the purpose of supporting the column and preventing blockage of the outlet. Cleaned sand was placed above and below the materials of the column for equal water dissipation and to control adsorbent washout with water. To maintain uniform flow of the metal solution into the column, glass beads of 1.5 mm diameter were placed to achieve 2 cm height. AC was added to the column up to the desired heights of 5, 10, 15 and 20 cm. Aqueous solutions with different initial concentrations (5.0, 10 and 20 mg/L) were allowed from the top of the column at a controlled flow of 5 mL/min (Figure 1). Water samples were collected at different time intervals (5, 10, 30 and 60 min) after passing through the column (Dwivedi *et al.* 2008). The same technique was also used for removal of PTEs from industrial wastewater.

## RESULTS AND DISCUSSION

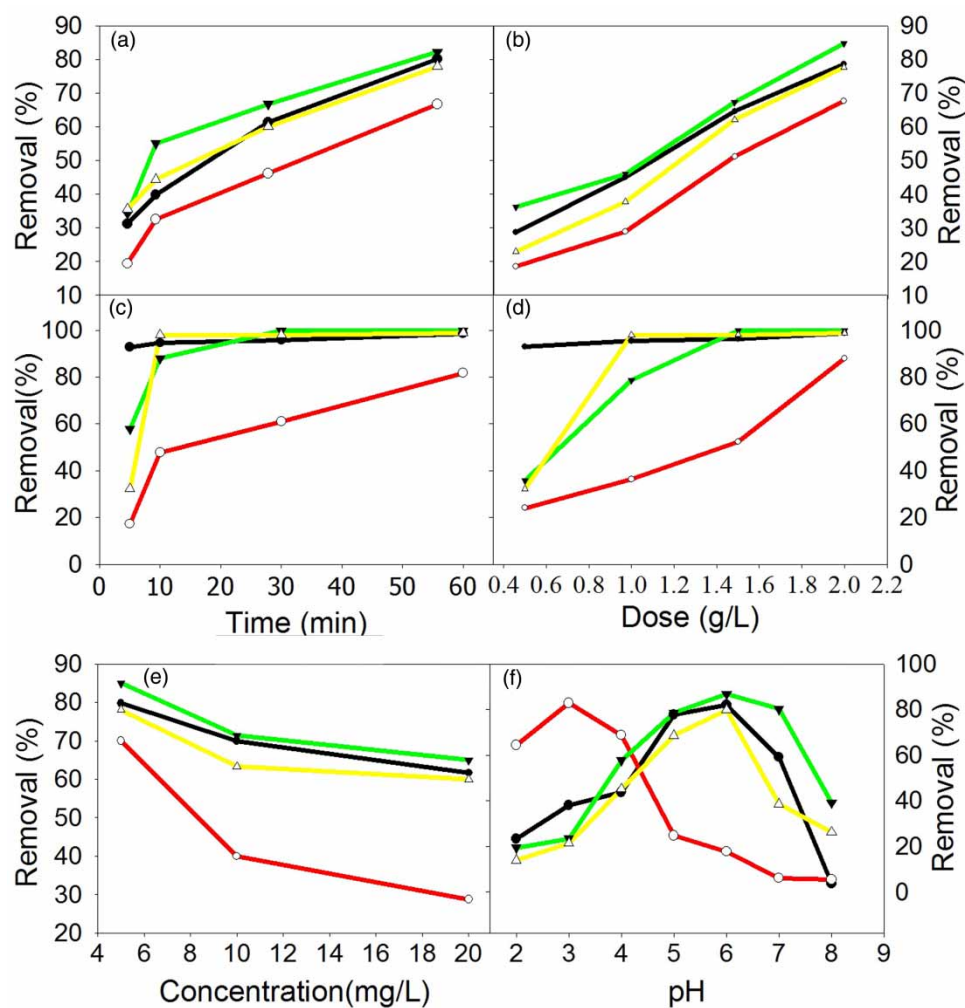
### Batch adsorption experiments

#### Effect of contact time

Table S1 (available with the online version of this paper) presents the results of different contact time intervals (5, 10, 30 and 60 min) for PTE removal. The results indicated that the percent adsorption increased with increasing contact time and the maximum adsorptions of 80.2, 66.8, 82.2 and 78.0% for Cd, Cr, Cu and Pb, respectively, were achieved from aqueous solutions under the present experimental conditions. In addition, 98.4, 87.6, 99.9 and 99.9% removal of Cd, Cr, Cu and Pb, respectively, was recorded for wastewater at a contact time of 60 min (Figure 2(a) and 2(c)), and the detailed results of all the treatment times for both aqueous solutions and wastewater are shown in Table S1. The maximum  $q_e$  (mg/g) values were 0.20, 0.17, 0.20 and 0.19 mg/g recorded for Cd, Cr, Cu and Pb, respectively, in aqueous solution, while 0.002, 0.14, 0.08 and 0.14 mg/g for Cd, Cr, Cu and Pb, were recorded respectively for wastewater under the studied experimental conditions (Table S1).

#### Effect of adsorbent dose

The accessibility and availability of adsorption sites is governed by the adsorbent dosage used (Rafeah *et al.* 2009).



**Figure 2** | Removal (%) of PTEs using AC at 30 °C under different experiment conditions; (a) and (c) effect of contact time, (b) and (d) effect of adsorbent dose, (e) effect of initial concentration, and (f) effect of solution pH.

The removal of PTEs by AC was also studied at different adsorbent doses (0.5, 1, 1.5 and 2.0 g). The results indicated that the percentage adsorption of metals increased with increasing the adsorbent dose. The maximum adsorption of 78.6, 62.7, 84.8, and 71.3% for Cd, Cr, Cu and Pb, respectively, was recorded for an adsorbent dose of 2.0 g, as shown in Figure 2(b) and 2(d). The results of all PTEs removal performance with different adsorbent dosages are presented in Table 1. In addition, the removals of Cd, Cr, Cu and Pb from wastewater were observed to be about 98.8, 87.8, 99.9 and 98.7%, respectively, with an adsorbent dose of 2.0 g (Table 1), and after this stage the adsorption became almost constant. The maximum  $q_e$  (mg/g) for Cd, Cr, Cu and Pb was 0.19, 0.16, 0.21 and 0.18 mg/g from aqueous solutions and 0.24, 0.08, 0.04 and 0.07 mg/g from wastewater, respectively (Table 1). The increase in percent removal of

metal ions that occurred with increasing adsorbent doses could be linked to an increase in availability of adsorbent surface area as well as adsorption sites. After that, with further increase of adsorbent dosage, no significant adsorption efficiency was observed and this could be because of the overcrowding of adsorbent particles, which causes overlapping of adsorption sites (Abbas *et al.* 2013).

#### Effect of initial ion concentrations

The removal efficiency of metals decreased with increasing initial concentrations of metal, as shown in Figure 2(e). Removal efficiencies (%) were found to decrease in the ranges of 79.8–61.7, 70.0–28.8, 85.0–65.1 and 78.0–60.0% for Cd, Cr, Cu and Pb, respectively, as shown in Table 2. But the metals uptake  $q_e$  (mg/g) were found to increase to

**Table 1** | Effects of adsorbent dose on removal of PTEs (%) from aqueous solutions and industrial wastewater

AC dose (g/L)	Aqueous solution				Industrial wastewater			
	Influent concentration (mg/L)	Effluent concentration (mg/L)	Removal efficiency (%)	qe (mg/g)	Influent concentration (mg/L)	Effluent concentration (mg/L)	Removal efficiency (%)	qe (mg/g)
<b>Cd</b>								
0.5	5	3.56	28.6	0.28	0.026	0.002	93.0	0.37
1	5	2.74	45.1	0.22	0.026	0.001	95.4	0.48
1.5	5	1.77	64.6	0.21	0.026	0.001	96.1	0.59
2	5	1.06	78.6	0.19	0.026	0.001	98.8	0.24
<b>Cr</b>								
0.5	5	4.07	18.6	0.18	1.79	1.360	24.0	0.09
1	5	3.55	29.0	0.14	1.79	1.140	36.3	0.06
1.5	5	2.44	51.2	0.17	1.79	0.850	52.4	0.06
2	5	1.86	62.7	0.16	1.79	0.220	87.8	0.08
<b>Cu</b>								
0.5	5	3.19	36.2	0.36	0.81	0.521	35.7	0.06
1	5	2.70	46.0	0.23	0.81	0.173	78.6	0.06
1.5	5	1.63	67.4	0.22	0.81	0.001	99.0	0.05
2	5	0.76	84.8	0.21	0.81	0.001	99.9	0.04
<b>Pb</b>								
0.5	5	3.85	23.0	0.23	1.48	1.002	32.2	0.09
1	5	3.11	37.8	0.19	1.48	0.030	97.9	0.14
1.5	5	1.89	62.2	0.21	1.48	0.029	98.0	0.09
2	5	1.43	71.3	0.18	1.48	0.019	98.7	0.07

**Table 2** | Effect of initial concentrations on removal of PTEs (%) from aqueous solutions

Influent concentration (mg/L)	Effluent concentration (mg/L)	Removal efficiency (%)	qe (mg/g)
<b>Cd</b>			
5	1.0	79.8	0.11
10	3.0	69.9	0.34
20	7.6	61.7	0.61
<b>Cr</b>			
5	1.5	70.0	0.17
10	6.0	39.9	0.11
20	14.2	28.8	0.29
<b>Cu</b>			
5	0.75	85.0	0.21
10	2.86	71.4	0.35
20	7.0	65.1	0.65
<b>Pb</b>			
5	1.09	78.0	0.19
10	3.67	63.3	0.31
20	8.0	60.0	0.60

0.61, 0.29, 0.65 and 0.60 mg/g for Cd, Cr, Cu and Pb respectively, from 0.11, 0.17, 0.21 and 0.19 mg/g with the increase in concentration from 5 to 20 mg/L (Table 2). This decrease in metals removal efficiency is due to the fact that at low concentrations the ratio of adsorbent surface active sites to the total metals increases, and therefore all metals interacting with the adsorbent can be easily removed (Karthikeyan *et al.* 2005).

### Effect of pH

Water pH is important operating parameter that considerably affects the metals' adsorption process of AC (Acharya *et al.* 2009). Results indicated that the adsorption of different PTEs increased with increasing pH values of aqueous solutions from 2 to 6, except Cr (Figure 2(f) and presented in Table S2 (available with the online version of this paper). The removal of Cr was observed to be higher (82.8%) at pH 3 compared to other studied pH values. In the aquatic system, Cr exists in three ionic forms ( $\text{HCrO}_4^-$ ,  $\text{Cr}_2\text{O}_7^{2-}$ , and  $\text{CrO}_4^{2-}$ ), and the aqueous pH is responsible for their stability. At lower pH, Cr exists in

its  $\text{HCrO}_4$  form, which is attracted by the highly protonated adsorbent surface due to the electrostatic attraction between the positively charged surface of the adsorbent and the negative chromate ions (Liu *et al.* 2014). On the other hand, the adsorption of the remaining three metals, Cd, Cu and Pb, was low in the acidic medium, and this may be due to competition between metals and  $\text{H}^+$  or  $\text{H}_3\text{O}^+$  ions for the same negatively charged sites on the adsorbent found in the solution (Tajar *et al.* 2009). Similarly, the adsorption of metals increased by increasing the solution pH from 2 to 6 and reached optimal efficacy of 82.0, 86.8 and 79.8% for Cd, Cu and Pb, respectively at pH 6.0 (Figure 2(f)). However, the maximum  $q_e$  (mg/g) was 0.20, 0.04, 0.22 and 0.19 for Cd, Cr, Cu and Pb, respectively at aqueous solution pH 6 (Table S2). There is a gradual decrease in the percent removal when the pH of the solution increases from 6 to 8 due to the electrostatic repulsion between metals and AC at higher pH (Liu *et al.* 2014).

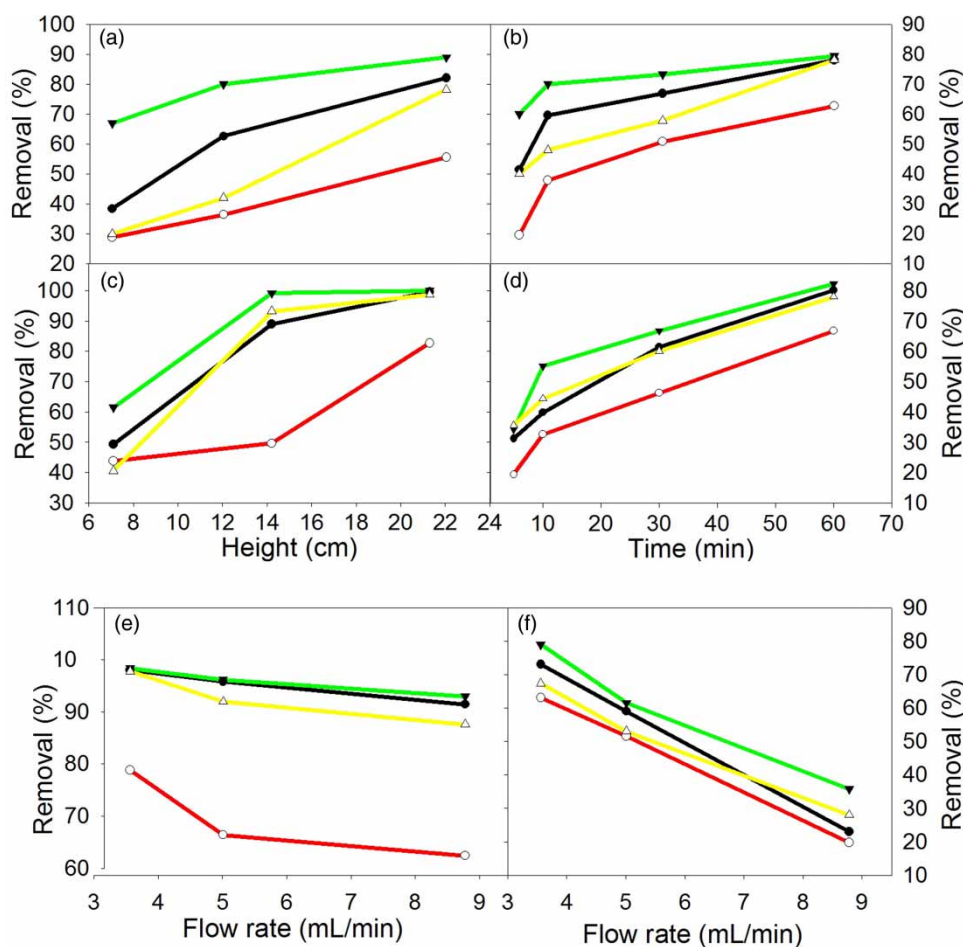
### Isotherm study

The Langmuir isotherm model was applied to experimental data and the analyzed results showed that the  $R^2$  values for Cd, Cr, Cu and Pb were 0.96, 0.96, 0.93 and 0.95, respectively for aqueous solutions and 0.99, 0.86, 0.98 and 0.99, respectively for wastewater, which were closely related to Langmuir model parameters in other reported studies (Shrestha *et al.* 2013). The maximum adsorption capacity  $Q_{\text{max}}$  calculated from Langmuir was 15.5, 74.3, 14.3 and 30.4 mg/g for Cd, Cr, Cu and Pb, respectively, as presented in Table S3 (available with the online version of this paper).

### Column adsorption experiments

#### Effect of adsorbent bed height

Adsorption of the metals was studied taking different adsorbent heights (5.0, 10 and 20 cm) at a constant flow rate of



**Figure 3** | Removal (%) of PTEs by AC at 30 °C in column experiments under the experimental conditions; (a) and (c) effect of column height, (b) and (d) effect of contact time, and (e) and (f) effect of flow rate in column experiments.

5 mL/min of the adsorbate. Figure 3(a) and 3(c) present the results of different PTE removals at various adsorbent bed heights. It can be seen that by increasing the column height from 5 to 20 cm the adsorption rate increased. The maximum removal was 82.0, 55.6, 89.1, and 78.2% for Cd, Cr, Cu and Pb, respectively from aqueous solutions and 99.6, 82.7, 98.8 and 98.7% for wastewater at a bed height of 20 cm (Table S4, available with the online version of this paper). The percent removal of different metals were observed in the order of Cu > Cd > Pb > Cr.

### Effect of contact time

Results indicated that the adsorption increased with increasing contact time. The adsorption rates were 78.0, 62.7, 79.4 and 78.4% for Cr, Cd, Cu and Pb, respectively, for aqueous solutions. In addition, Cd, Cr, Cu and Pb removal of 98.1, 92.9, 99.9 and 99.6%, respectively, was achieved at a contact time of 60 min, as shown in Figure 3(b) and 3(d). The details of removal efficiencies at all the contact times in both aqueous solutions and industrial wastewater are given in Table S4. The reason for the higher removal after longer contact time is the availability of metallic ions to adsorb onto the surface of the adsorbent.

### Effect of flow rate

PTE removal efficiency using AC in column experiments was also investigated by applying different flow rates (3.56, 5.01 and 8.78 mL/min). A flow rate of 3.56 mL/min was found to be most suitable for the adsorption of all selected metals and observed to be 98.0, 78.8, 98.4 and 97.8% for aqueous solutions, and 73.0, 63.2, 79.0 and 67.3% for Cd, Cr, Cu and Pb, respectively, for industrial wastewater, as shown in Figure 3(e) and 3(f). Results indicated that by increasing the flow rate, the percent adsorption decreases steadily, as presented in Table 3. The reduction of metal uptake is due to the availability of insufficient contact time of the adsorbent with metal ions in the solution.

## CONCLUSIONS

In this study, AC was employed to remove different PTEs from aqueous solutions as well as industrial wastewater under various experimental conditions. Results revealed that Cu showed better removal from both batch and column experiments compared to Cd, Cr and Pb in all the treatments. Maximum adsorption of Cd, Cu and Pb was recorded at pH 6, while Cr showed maximum adsorption

**Table 3** | Effect of different flow rates on removal of PTEs (%) via AC for aqueous solution and wastewater

Flow rates (mL/min)	Aqueous solution			Industrial wastewater		
	Influent concentration (mg/L)	Effluent concentration (mg/L)	Removal efficiency (%)	Influent concentration (mg/L)	Effluent concentration (mg/L)	Removal efficiency (%)
<b>Cd</b>						
3.56	5	0.10	98.0	0.026	0.0007	73.0
5.01	5	0.21	95.8	0.026	0.001	59.0
8.78	5	0.33	91.4	0.026	0.020	23.0
<b>Cr</b>						
3.56	5	1.01	78.8	1.79	0.66	63.1
5.01	5	1.68	66.4	1.79	0.69	51.5
8.78	5	1.88	62.4	1.79	0.90	19.7
<b>Cu</b>						
3.56	5	0.08	98.4	0.81	0.52	79.0
5.01	5	0.29	96.2	0.81	0.39	61.5
8.78	5	0.51	93.0	0.81	0.17	35.8
<b>Pb</b>						
3.56	5	0.11	97.8	1.5	0.49	67.3
5.01	5	0.40	92.0	1.5	0.57	53.0
8.78	5	0.62	87.6	1.5	1.08	28.0

at pH 3. Results demonstrated that by increasing the adsorbent dosage and height of the column, the removal efficiency increased and maximum removal occurred at a dose of 2 g with a column height of 20 cm. In addition, the Langmuir isotherm model better represented the results and the adsorption process was found to be spontaneous. Findings from this study suggested that AC could be a promising adsorbent for the effective removal of PTEs from aqueous solutions and industrial wastewaters.

## ACKNOWLEDGEMENTS

The authors acknowledge the financial support of the Higher Education Commission (HEC) Islamabad, Pakistan and University of Peshawar, Pakistan.

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First received 12 June 2016; accepted in revised form 16 February 2017. Available online 3 March 2017