

Utilization of Fly and Coarse Ash for Removal of Heavy Metal Ions from Contaminated Water

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Abstract

Rapid and unsustainable industrialization has caused many environmental problems that led to rise in wastewater contamination, which is one of the major environmental concern. This study attempts to access the main pollutants found in the wastewater. Heavy metals were found to be the major pollutants in contaminated water. Four types of ashes simple fly ash, simple coarse ash, acid activated fly ash and acid activated coarse ash were evaluated for the adsorption of Cadmium (Cd), Chromium (Cr), and Copper (Cu) from contaminated water in single contaminant system. Adsorption of these heavy metals was increased by increasing the contact time until the equilibrium attained. The adsorption sequence for the heavy metal removal was acid activated fly ash > fly ash > acid activated coarse ash > simple coarse ash. Pseudo first order kinetic model could best fit for the adsorption of Cd, Cr, and Cu. The effect of pH on the adsorption of Cadmium (Cd), Chromium (Cr), and Copper (Cu) was also studied. It was observed that the removal of cadmium and copper was maximum at basic pH while the removal of chromium was maximum at acidic pH. Results indicated that maximum adsorption was recorded on acid-activated fly ash for Cd (92.12%), Cr (85.22%), and Cu (64.01%) with the variation of time and pH. Our results suggest that the acid activated fly ash could be the best adsorbent out of these for the removal of cadmium, chromium, and copper because of its larger surface area and smaller particle size as well as the acid activation increase its adsorption efficacy due to the presence of electrostatic interaction.

Keywords: Heavy Metals; Wastewater; Adsorption; Fly Ash; Coarse Ash

Abbreviations

FA: Fly Ash; CFA: Coal Fly Ash; AAS: Atomic Absorption Spectrophotometer; AAFA: Acid Activated Fly Ash; AACA: Acid Activated Coarse Ash; CA: Coarse Ash; CD: Current Densities

Introduction

Removal of heavy metals is being carried out by different sources such as precipitation, ion exchange, membrane filtration etc. Adsorption, along with the selection of an appropriate adsorbent, may be an efficient method for removing heavy metals from wastewater [1]. While activated carbon, alumina, silica, and ferric oxide are suggested adsorbents with high metal adsorption capability, they are costly and difficult to remove from wastewater after use. As a result, in recent years, there has been a massive increase in research towards developing low-cost alternatives to these adsor-

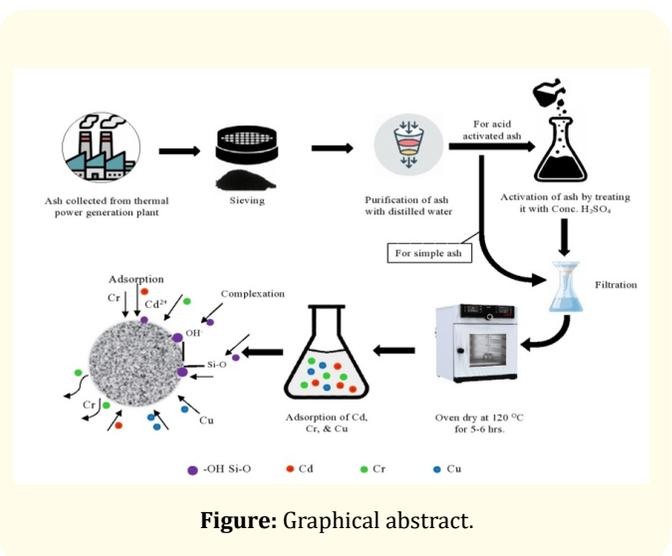


Figure: Graphical abstract.

bents using a range of carbonaceous and mineral precursors.

Almost 121 million tons of coal fly ash (CFA) per annum, produces by coal-fired power plants [2]. More than 500 million tons were produced in China during 2015 [3]. Many studies have been devoted to the reuse of FA to protect the environment, architecture, agriculture, ceramics and the rehabilitation of beneficial components [4]. Few studies have been conducted [5,6] on the possible use of coal fly ash in wastewater treatments, but such papers concentrate mainly on the adsorption of pollutants from fly ash, which were an enormous amount of available industrial waste created in coal-fired energy plants during burning.

Fly ash was used mainly as building materials such as asphalt, concrete, bricks and roads [7]. Fly ash is a possible adsorbent due to its excess availability and low economic cost [8]. Furthermore, it is beneficial due to its hydrophobicity, is environmentally safe, and very easy to decompose [9]. Fly ash is alkaline and shows pH from 10 to 13. It is spread through the water. The surface of FA with elevated pH values can be neglected by means of electrostatic adsorption and precipitation to separate heavy metal ions from water [10].

In testing the impact of the initial metal concentration on adsorption capability, FA found to be efficient adsorbent for Cd and Cu. Due to its core nature and adsorption capability, FA neutralized the metal solution as its initial metal concentration was reduced [11]. Because the potential of raw fly ash to adsorb was very small, several chemical and physical techniques were developed to enable fly ash to increase its adsorption capability, such as microwave, acid-FA [13], salt-FA [12] alkali-FA [7].

Activated fly ash has been used in contaminated water [13,14], to treat toxic substances that have been infected by dyes, metal ions [15], or Volatile Organic Compounds [16]. Chemically adapted FA has a strong ability to remove waste water toxins. The retention efficiencies in CDs are 91 - 99%, and 90 - 97%, respectively, for As, Cd, Cu, Mn, Al, Ni, Hg, Fe, Pb and Zn, ions from aqueous solution. The CFA is used for thioglycolic acid and 2-mercaptoethanol, respectively. This is because of mercaptans' chemical properties (presence of OH and SH groups for coordination with metal ions).

Coarse and fly ash is an effective strategy for heavy metals removal i.e. cadmium (Cd), copper (Cu), and chromium (Cr) from wastewater. Heavy metals like Cd, Cr, and Cu can be removed by using acid-activated fly and coarse ash as adsorbents, it is, therefore, essential to evaluate the removal efficiency of acid-activated fly and coarse ash towards the heavy metals (i.e. Cd, Cr, and Cu).

Aim of the Study

The current study was aimed to evaluate the adsorption capacity of acid-activated fly ash and coarse ash for the elimination of Cd, Cr, and Cu ions.

Materials and Methods

A batch experiment was conducted at the environmental biogeochemistry laboratory at ISES, the University of Agriculture, Faisalabad to check the response of fly and coarse ash towards the removal of three selected heavy metals cadmium (Cd), copper (Cu), and chromium (Cr) from contaminated water. Heavy metals were artificially introduced in water for contaminated sample preparation. Stock solutions of chromium, copper, and cadmium were prepared by dissolving a known concentration of potassium dichromate ($K_2Cr_2O_7$), copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$), and cadmium chloride ($CdCl_2$), respectively. After that, the sub stock solutions were prepared from these stocks, and working solutions prepared for further analysis. For the experimental purpose, the fly ash and coarse ash was taken from thermal power plants depending on crop residues burning. Four types of ashes were used as a treatment, fly ash, coarse ash, acid-activated fly ash, and acid-activated coarse ash.

Preparation of acid-activated fly ash and coarse ash

The fly ash (FA) which is used in this study was collected from thermal power plants depending on crop residues burning and was collected manually in different polythene bags and taken to the environmental biogeochemistry laboratory for further studies. The fly and coarse ash were purified with distilled water and then dried at 110°C for 5h in an oven. Activation of the fly and coarse ash was done by treating it with concentrated H_2SO_4 (98 percent w/w) in a ratio of 1:1 weight and is kept in an oven maintained for 24 hours at a temperature of 150°C. Finally, the free acid was washed away with distilled water.

In order to prepare 2M H_2SO_4 solution in 100 mL, 9.8g of H_2SO_4 was added in 100 mL of distilled water (2 M H_2SO_4 in 100 mL). Thereafter, 90g of fly and coarse ash was transferred through a screen size (160), and the latter was sieved. Fly ash under ultrasonic operation was combined with a 2-molar H_2SO_4 solution for 30 minutes. The mixture was filtered and washed with excess water and dried at 120°C, then the mixture was cooled at ambient temperature, and recycled. Modified fly ash passes through 160 mesh size sieve and was prepared [17].

Adsorption studies

The effects of different parameters like contact time and initial

pH were presented for determining the adsorption efficacy of all types of ashes described earlier.

Twenty mL of contaminated samples of Cd, Cr, and Cu were poured in 50 mL volumetric flask than an amount of 25 mg of each adsorbent i.e. fly ash, acid-activated fly ash, coarse ash, and acid-activated coarse ash was added in samples in each flask. The samples were stirred using the mechanical shaker at room temperature. To observe the equilibrium time for Cd, Cu, and Cr ions adsorption varying contact time like 0, 1, 2, 3, 4, 5, 6, and 7 hours were studied. Later on, the samples were filtered by using syringe filters (0.22 nm pore size) and the concentration of the elements was analyzed by using atomic absorption spectrophotometer (AAS) model Hitachi polarized zeeman AAS Z-8200 Japan.

The next experiment was performed to evaluate the impact of pH on adsorption efficiency. For this purpose, 20 mL of contaminated solution was poured into the glass bottles, and pH was adjusted at 3, 5, 7, and 9. 0.1 N HCl and 0.1 N NaOH were used to adjust the pH. 25 mg of the adsorbent dose was also added in the same glass bottles and placed on a shaker for 6 hours. After 6 hours filter solution by using syringe filters (0.22 nm pore size) and pH was measured again by using a pH meter. The samples were run on AAS to analyze the absorption capacity of fly ash at different pH scales.

Statistical analysis

The results were discussed by using mean \pm SD. All the Statistical analysis was performed using MS Excel 2010, version office Xp. The heavy metals concentrations were subjected to equilibrium isotherm models and kinetic models (pseudo-first-order, pseudo-second-order) were also applied using statistical functions of MS Excel 2010 and Sigma Plot version 10 [18].

Results and Discussions

Effect of contact time

The amount of cadmium (a), chromium (b), and copper (c) adsorbed on the surface of the acid-activated fly ash, fly ash, acid-activated coarse ash and coarse ash as a function of contact times shown in Figure 1. The contact time was varied from 0 to 7 hours for all three heavy metals and the rate of the applied adsorbent was 25 mg. The concentration of heavy metal in the filtrate was measured by using atomic absorption spectrophotometer (Hitachi Polarized Zeeman AAS, Z-8200, Japan). The removal of heavy metals increased with the increase in contact time available for the adsorption process. It was noted that the removal efficiency of Cd after 1 hour was only 21.70%, 17.07%, 16.38%, 8.81% for acid-activated fly ash, fly ash, acid-activated coarse ash, and coarse ash, respectively and after 6 hours the removal efficiency of Cd was increased

to 96.58%, 94.47%, 89.14%, and 81.14% for acid-activated fly ash, fly ash, acid-activated coarse ash, and coarse ash, respectively. After 7 hours slight increase in the removal efficiency of Cd was detected. The removal efficiency was slightly increased to 99.73%, 97.40%, 91.40%, 83.06% for acid-activated fly ash, fly ash, acid-activated coarse ash, and coarse ash, respectively. It was noted that the removal efficiency of Cr after 1 hour was 12.82%, 10.67%, 8.81%, and 5.37% for acid-activated fly ash, fly ash, acid-activated coarse ash, and coarse ash, respectively and after 6 hours the removal efficiency of Cr was increased to 88.74%, 81.14%, 77.28%, and 61.01% for acid-activated fly ash, fly ash, acid-activated coarse ash, and coarse ash, respectively. While in case of Cu, the removal efficiency after 1 hour was 6.44%, 4.65%, 2.04%, 0.24% and after 6 hours the amount of Cu adsorbed was increased to 85.66%, 75.15%, 66.42%, and 51.1% for acid-activated fly ash, fly ash, acid-activated coarse ash, and coarse ash, respectively. It was observed that the equilibrium was attained in Cd, Cr, and Cu after 6 hours as no considerable increase in removal efficiency was detected at the contact time of 7 hours. The surface area controls the removal capacity of solid particles [19].

It was noted that the adsorption of heavy metal onto the surface of different types of ashes depends upon the type of heavy metal; similar types of results were also presented by [20]. The difference in the amount adsorbed or the heavy metal uptake by ash can be explained in terms of the difference in the ionic size of the metal ions [21]. It was evident that the acid-activated fly ash has the best adsorption capacity among all types of adsorbents used followed by simple fly ash, acid-activated coarse ash, and the coarse ash had the lowest adsorption capacity. It may be because of the fact that the fly ash has a smaller particle size as compared to coarse ash and hence have a larger surface area available for the metal ion adsorption. As the contact time increased, the sites of the ash start gradually occupied by the heavy metals and it continues to do so until the adsorption reached equilibrium [22]. It was observed that the adsorption of Cd was much faster as compared to the adsorption of Cr and Cu. It was also observed that among all three heavy metals Cd shows the best adsorption while Cu was the least adsorbed heavy metal.

Adsorption kinetics

The adsorption kinetics depends on the physical and chemical properties of the adsorbent as well as on the mass transfer process [23]. The pseudo-first-order equation which is generally expressed as [24]:

$$\ln(C_0/C_t) = kt$$

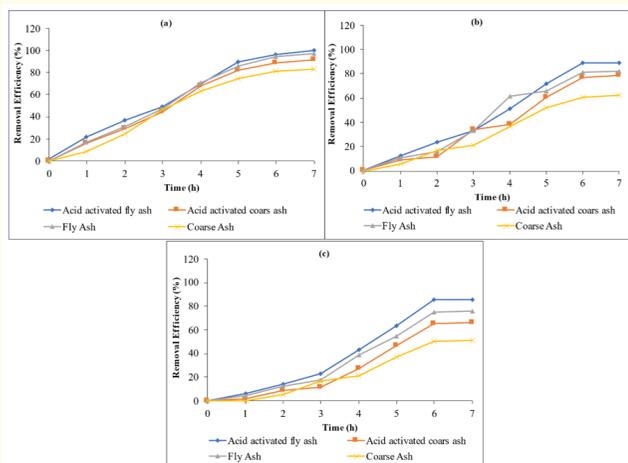


Figure 1: Comparison between acid-activated fly ash, acid-activated coarse ash, fly ash, and coarse ash on Cd (a), Cr (b), and Cu (c) adsorption depending on contact time. The horizontal lines represent the adsorption capacity of heavy metals over an interval of time (0, 1, 2, 3, 4, 5, 6, and 7 hours).

Where, C_0 (mg L^{-1}) is the initial concentration of heavy metal and C_t (mg L^{-1}) is the concentration of heavy metal at time t (h), respectively, and k_1 is the rate constant for pseudo-first-order reaction (h^{-1}). The plot of $\ln(C_0/C_t)$ versus t describes the pseudo-second-order model and the values of q_e and k_1 were determined from the slopes and intercepts of the plot.

The pseudo-second-order equation is generally expressed as [25]:

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2$$

Where q_e and qt is the sorption capacity at equilibrium and time t (mg g^{-1}), respectively, k_2 is the rate constant of the pseudo-second-order sorption ($\text{gmg}^{-1} \text{h}^{-1}$). The plot of t/qt versus t describes the pseudo-second-order model and the values of q_e and k_2 were determined from the slopes and intercepts of the plot.

All the kinetic parameters, the first-order rate constant (k_1), the second-order rate constant (k_2), calculated adsorption capacity (q_e), and regression coefficients (R^2) were investigated and the resulted values are described in table 1. It was suggested that adsorption of Cd, Cr, and Cu follows pseudo-first-order kinetics as the values of R^2 obtained from pseudo-first-order reactions are larger as compare to the values obtained from pseudo-second-order reac-

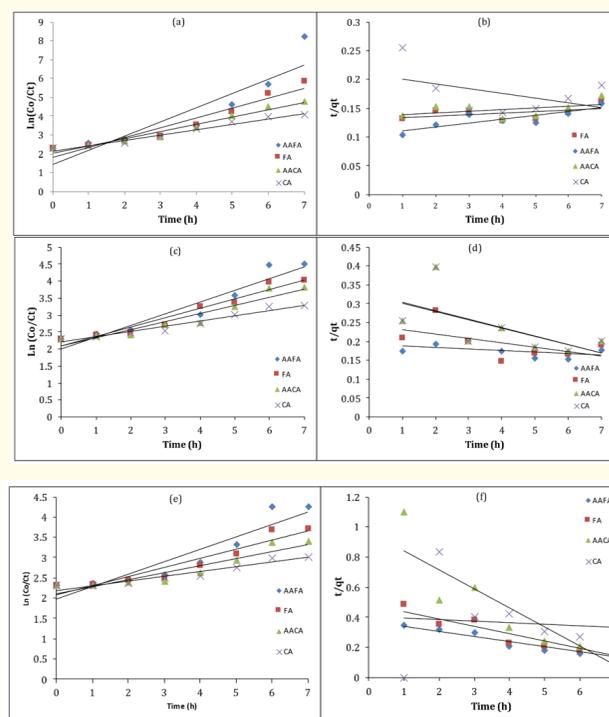


Figure 2: Lagergren's ($\ln C_0/C_t$) and Ho-McKay (t/qt) plots for heavy metal adsorption on various types of ashes. First-order kinetics for Cd (a). Second-order kinetics for Cd (b). First-order kinetics for Cr (c). Second-order kinetics plot for Cr (d). First-order kinetics plot for Cu (e). Second-order kinetics plot for Cu (f) against time (h).

tion and previous studies have suggested the same [26]. So, pseudo-first-order was taken as the best-fit equations for Cr adsorption. Al-othman and co workers reported the similar type of results [27].

Effect of pH

It is evident that pH plays a vital role in the uptake of metal ions, as it determines the surface charges of the adsorbent. It was observed that the adsorption of metals on the surface of the ashes was highly dependent on the pH of the solution. Figure 3a-3c shows the uptake of cadmium, chromium, and copper on the surface of the fly ash and coarse ash as a function of pH.

The pH varied from 3, 5, 7, and 9 for all three heavy metals and the rate of the applied adsorbent was 25 mg. The heavy metal adsorption on the surface of the adsorbent showed a different dependence on pH for all the heavy metals studied. It was observed that the amount of heavy metals adsorbed increased as the pH of the

Metal	Adsorbents	Pseudo-first-order model			Pseudo-second-order model		
		q_e (mg g ⁻¹)	K_1 (h ⁻¹)	R ²	q_e (mg g ⁻¹)	K_2 (g mg ⁻¹ h ⁻¹)	R ²
Cd	Acid activated fly ash	1.452	0.75	0.824	0.104	0.006	0.717
	Fly ash	1.830	0.521	0.926	0.130	0.002	0.245
	Acid activated coarse ash	2.048	0.382	0.960	0.135	0.003	0.232
	Coarse ash	2.157	0.285	0.977	0.209	0.008	0.198
Cr	Acid activated fly ash	1.997	0.344	0.899	0.193	0.004	0.278
	Fly ash	2.103	0.275	0.946	0.241	0.011	0.319
	Acid activated coarse ash	2.094	0.240	0.914	0.324	0.022	0.395
	Coarse ash	2.202	0.157	0.958	0.325	0.022	0.396
Cu	Acid activated fly ash	1.968	0.005	0.870	0.375	0.033	0.865
	Fly ash	2.076	0.003	0.895	0.484	0.048	0.823
	Acid activated coarse ash	2.108	0.002	0.880	0.967	0.126	0.740
	Coarse ash	2.190	0.001	0.927	0.407	0.011	0.001

Table 1: Pseudo-first-order and pseudo-second-order model parameters for the adsorption of Cd, Cr, and Cu.

solution increases in the case of copper and cadmium while the amount of chromium ion adsorbed decreased with the increase in pH of the solution, in short, the copper and cadmium has a better removal efficiency at basic pH while the chromium effectively removes in the acidic condition.

It was noted that the removal efficiency of Cd at pH 3 was 85.41%, 83.41%, 81.43% and 80.12% for acid-activated fly ash, fly ash, acid-activated coarse ash, and coarse ash, respectively and at pH 5 the removal efficiency of Cd was 88.41%, 86.41%, 85.41% and 84.12% for acid-activated fly ash, fly ash, acid-activated coarse ash, and coarse ash, respectively. At pH 7, the removal percentage was increased to 92.12%, 90.99%, 90.12% and 88.75% for acid-activated fly ash, fly ash, acid-activated coarse ash, and coarse ash, respectively. The maximum removal efficiency for Cd was observed at pH 9. The graphs shown that the removal efficiency at pH 9 was 98.75%, 96.84%, 95.92% and 94.32% for acid-activated fly ash, fly ash, acid-activated coarse ash, and coarse ash, respectively.

The pH effect for fly ash adsorption of Cd can be calculated based on the complex composition of the water oxides present in fly ash. In an acidic solution, a positive charge forms on the surface of fly ash oxides. The reduced adsorption amount depends on the surface charge thereby produced is not appropriate for Cd adsorption, which results in the chloride ion being readily identified with

the surface of the fly ash. In an alkaline medium, the surface of fly ash is negatively charged and hence facilitates the adsorption of Cd.

It was noted that the removal efficiency of Cr at pH 3 was 85.22%, 67.39%, 48.64% and 39.90% for acid-activated fly ash, fly ash, acid-activated coarse ash, and coarse ash, respectively and at pH 5 the removal efficiency was observed 82.75%, 63.74%, 45.72% and 35.12% for acid-activated fly ash, fly ash, acid-activated coarse ash, and coarse ash, respectively. At pH 7, the removal efficiency of Cr was decreased to 69.74%, 52.12%, 32.76% and 24.12% for acid-activated fly ash, fly ash, acid-activated coarse ash, and coarse ash, respectively. The lowest removal efficiency was absorbed at pH 9 with the percentage of 53.26%, 33.77%, 22.19% and 14.35% for acid-activated fly ash, fly ash, acid-activated coarse ash, and coarse ash, respectively. The difference in the adsorption of Cr with different pH could be described by the role of silica for the adsorption of Cr on fly ash. When pH was low positively charged silica surface occurs and at high pH, a negatively charged surface prevails. The adsorption of anions such as Cr ion is much stronger in acidic pH than in alkaline pH.

While in the case of Cu the removal efficiency at pH 3 was 45.78%, 22.43%, 12.94% and 6.18%, and at pH 5 the removal efficiency was 49.12%, 29.41%, 19.42% and 10.43% for acid-activated fly ash, fly ash, acid-activated coarse ash, and coarse ash, respectively. At pH

7 the removal efficiency of Cu adsorbed was increased to 57.12%, 40.32%, 25.12% and 19.42% for acid-activated fly ash, fly ash, acid-activated coarse ash and coarse ash, respectively. The maximum removal efficiency was observed at pH 9, where the percentage was 64.01%, 47.35%, 25.76% and 25.76% for acid-activated fly ash, fly ash, acid-activated coarse ash, and coarse ash, respectively. With the increase in pH of the solution from 3 to 9 the extent of removal increases. At low pH the surface of the fly ash was positive and there was a creation of the complex $[\text{Cu}(\text{OH})_4]^{2-}$, the complex formed will be adsorbed on the fly ash surface. At higher pH the surface of the adsorbent was negative and there was an increase in the adsorption of Cu ions by the fly ash. The increase in metal ions adsorption by fly ash at higher pH values may be attributed to calcium contents that provide alkalinity in the system raising the pH to strongly alkaline values and facilitating the adsorption of cations by fly ash [21].

It was noted that the pH of the solution increases as we add the ash into it because of its alkaline nature. As suggested by [28] that heavy metal ion's adsorption at the solid-solution interface is not governed by the free metal concentration, but by the much stronger adsorbed hydroxo, sulfato, carbonato, and other metal species. It is also suggested that the enhanced removal of heavy metal is greatly attributed to the presence of hydroxide ions. The presence of one hydroxyl ion would alter one adsorbed cupric ion to be more readily accommodated in these surface lattice which follows further adsorption [29].

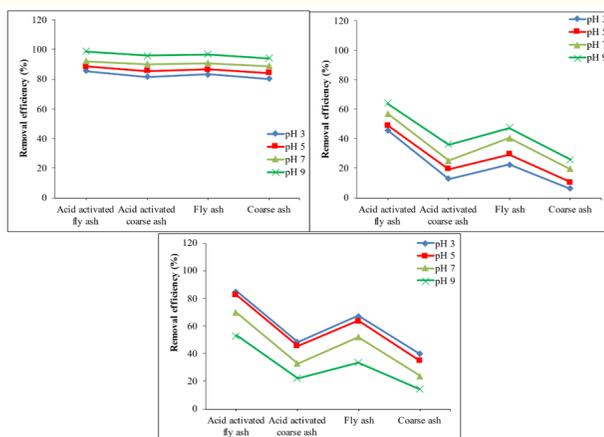


Figure 3: Effect of pH on Cd (a), Cr (b), and Cu (c) removal by adsorption mechanism. The vertical line represents the amount of Cd adsorbed over different treatments (acid-activated fly ash, acid-activated coarse ash, fly ash, coarse ash).

Conclusion

Our study revealed the application of ash as a low-cost adsorbent for the removal of Cd, Cr, and Cu ions from wastewater. The kinetic study shows that cadmium, chromium and copper adsorption was reached to the maximum level at 6 hours. It was observed that acid-activated fly ash has a better removal efficiency for all heavy metals studied followed by fly ash, acid-activated coarse ash, and coarse ash, respectively with optimum dose of 25 mg/ 100 mL. The data was tested with both pseudo first order and second order kinetic models and results suggested that the adsorption of Cd, Cr, and Cu follows the pseudo-first-order kinetic model. Chromium removal was significantly increased at acidic pH. Cadmium and copper removal was maximum at alkaline pH. The removal efficiency for cadmium was 92.12%, for chromium was 85.22% and for copper was 64.01%. It is suggested that acid-activated fly ash can be used as the most suitable strategy for heavy metals removal from wastewater and reduce environmental pollution in the future. Further should be focused temperature studies and adoptability on large scale.

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