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Adsorptive removal of chromium(VI) using Cu/Fe impregnated activated carbon prepared from solid sludge

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ABSTRACT

Chromium(VI) can be introduced to the environment from different industrial activities. This study focuses on the removal of chromium(VI) using activated carbon. In this work, solid sludge obtained from a treatment plant in Nizwa, Oman was used to prepare the activated carbon. The preparation was done following two processes. The first one was pyrolysis of dried solid sludge for 2 h at a temperature of 700°C under nitrogen gas flow of 150 mL/min. The second process was physiochemical activation using potassium hydroxide with an impregnation ratio of 1:1 under a mixture of nitrogen and carbon dioxide gases with a flow of 150 mL/min for 2 h. The morphology and chemical composition of the prepared carbons were characterized using scanning electron microscopy-energy-dispersive infrared spectroscopy. The prepared carbons were used for Chromium(IV) removal and the removal was performed at different dosages, metal concentrations, and pH at a temperature of 30°C for 6 h. Chromium levels were analyzed using flame atomic absorption spectroscopy (FAAS). The highest removal of chromium(VI) was found to be 23% at 1.5 g of activated carbon (AC) dosage and pH 3. The influence of impregnating the prepared activated carbon with Fe(III) and Cu(II) metals on Chromium removal was investigated. The treated activated carbon with copper achieved the highest removal efficiency of 94.5% at pH 3. Therefore, addition of Cu(II) metal to sludge AC is efficient in enhancing the removal of chromium(VI).

Keywords: Adsorption; Chromium(VI); Activated carbon; Sludge; Removal

1. Introduction

Wastewater is produced by combination of industrial, domestic, commercial, agricultural uses, storm water and sewer water [1–3]. It contains various physical, chemical, and biological pollutants that depends on the original source. A wide range of pollutants such as organic, inorganic hazardous, gasses and vapors can be found in wastewater [4]. The organics include halogenated organic compound, natural organic matter, compound with phenol group, nitro and amino compound, dyes, pesticides, drugs and toxins and miscellaneous organic compound. The reported inorganics content in wastewater are copper, chromium, selenium, nickel, cadmium, mercury, zinc, arsenic, lead, gold, silver, iron, tin, molybdenum, cobalt, manganese, and aluminum [5]. Therefore, wastewater treatment is necessary before discharging to water bodies. Various physical and chemical treatment methods have been used such as biological degradation, chemical precipitation, ion exchange, adsorption, reverse osmosis, flocculation, and coagulation [4].

Heavy metals such as chromium, lead, vanadium, arsenic, cadmium, and mercury are harmful to the environment

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and human health [6]. Usually, heavy metals discharged in municipal wastewater to be carried by water, while those in industrial effluents can be caused by different process such as catalysts, instrumentation leaks and corrosion product [7,8]. Chromium is one of these heavy metals that can be in the industrial discharges [9]. The two most stable species of chromium are the trivalent (Cr(III)) and hexavalent (Cr(VI)) species [10]. Chromium(VI) is a highly toxic metal compared to chromium(III). There are different industrial sources of chromium(VI) which come from cooling tower, leather tuning, plating, electroplating, rinse water and anodizing baths, chemical process, etc. [9]. According to World Health Organization (WHO) the allowable limit for Cr(VI) in drinking water is 0.05 mg/L [8,11,12]. It can cause various cancer disease, skin, and stomach irritation. At high exposure, can lead to liver damage, dermatitis, kidney circulation, nerve tissue damage, death in high concentration and exposure [9]. Therefore, the removal or reduction of chromium(VI) to chromium(III) is a key process to treat or eliminate chromium(VI) from contaminated water and wastewater [12]. Different methods that have been used for heavy metals removal from wastewater including adsorption [13], ion exchange [8,14], membrane filtration [15], and electrocoagulation [16]. Because it is simple process, high removal efficiency and low treatment costs, adsorption is widely used to remove Cr(VI) from wastewater compared to other methods [14].

Adsorption using activated carbon is used extensively for gold, air, water purification, sewage treatment, gas respirators and medicine as well in the removal of heavy metals [17]. Activated carbon can be produced from different materials such as rice husk, coconut husk, wood, bamboo, coir, coal, petroleum pitch and lignite [9,18]. These materials might contain inorganic material (lignin, cellulose, sugars) and the rest is silica [7,19]. Activated carbon can be prepared by two steps. In the first step, the raw material is carbonized at 800°C temperature in presence of inert gas [20]. The second step is the activation of the carbonized product which is carried out in atmospheric conditions then under air or CO_2 flow at 800°C–900°C [7]. The resulting activated carbon properties will be different depending on the carbonaceous materials, the activating agent used and the conditions of the previous two steps [19,21]. The produced activated carbon is characterized by carbon density, total surface area, adsorptive capacity and particle size distribution [22]. Activated carbons have relatively high pore volumes and surface areas [22]. Due to this they have effective adsorptive capacity or removal efficiency of heavy metals, biological oxygen demand, chemical oxygen demand, color from wastewater phenol number (index of carbons ability to remove odor) [19]. Regeneration of saturated activated carbon is important to reduce the operational and product sewage. For regeneration activated carbon there are several methods including steam, thermal and chemical regeneration [23]. There are different factors that was found to contribute significantly to Cr(VI) adsorption such as pH, adsorbent dose, metal concentration, the size of adsorbent particles [14,18].

Sewage sludge is produced at huge amounts in Oman. Part of it is used as fertilizers while the most is remained unused. This sewage sludge can be converted to activated carbon that can be used for heavy metal removal [1,21]. The aim of this study is to prepare activated carbon from sewage solid sludge collected from one of the sewage treatment station in Oman and use it to remove chromium(VI) from wastewater. Hence the objectives of this study are the preparation of activated carbon from sludge using chemical/physical activation, optimize the removal of chromium(VI) and improve this removal by using Cu or Fe metals impregnation.

2. Material and method

2.1. Materials and chemicals

Chromium standard, HCl, KOH, DDW, carbolite reactor, scanning electron microscopy (SEM), energy-dispersive infrared spectroscopy (EDX). Sodium hydroxide, potassium dichromate, sulfuric acid, ethanol, ethylene glycol, ferric nitrate, $CuCl_2$, and deionized distilled water. Activated carbon prepared from sludge.

2.2. Adsorbate: chromium(VI)

A stock solution of chromium(VI) was prepared (1,000 ppm) by dissolving 2.827 g of $K_2Cr_2O_7$ salt in 1,000 mL of distilled water. The desired concentrations ranging from 0.25 to 10 mg/L were made by diluting the stock solution with distilled water.

2.3. Preparation of activated carbon using physiochemical activation method

The raw materials of sludge were collected from Nizwa treatment plant, Oman. About 90.056 g of sample was collected and dried for 24 h in oven at 105°C. Then, it was grinded to powder and stored for further use. Approximately 100 g of sludge powder was carbonized using an electric furnace (carbolite reactor). The sample was heated at 700°C with a rate of 10°C/min under inert atmosphere for 2 h. The carbonized sample was chemically activated by potassium hydroxide at a 1:1 w/w ratio. It was then dried overnight in an oven under 100°C. After drying it was placed in a horizontal tube furnace under high purity nitrogen with flow rate of 150 mL/min till it reached 700°C. The N_2 gas was switched to CO_2 and the physical activation was completed in 2 h. The activated carbon was washed with HCl and then with DDW until reached neutral pH 7. After that it was dried, grinded, and sieved with size 40–120 mm.

2.4. Removal of chromium(VI) using the prepared sludge activated carbon

Adsorption technique is applied for purification and removal of toxic compounds from wastewater. In this study, the removal of chromium(VI) using activated carbon produced from solid sludge was investigated. The effect of dosage and concentration and catalyst addition on chromium removal were reported. The analysis of the treated samples was done using atomic absorption spectroscopy (AAS). Calibration curve was used to determine the concentration of chromium in the samples in contact with the activated carbon. The concentration of the calibration

standards (1–15 mg/L) were prepared from $K_2Cr_2O_7$ salt within the range of the FAAS (Model Thermo iCE 3000 series) instrument (Yang et al. [24]). Calibration standards preparation was done by taking 0.1, 0.2, 0.4, 0.5 and 0.75 mL from 1,000 ppm Cr. Then, they were transferred into 50 mL volumetric flasks and filled up to the mark with 1% HNO_3 trace metal grade.

The prepared activated carbon was used for chromium(VI) removal. The removal experiment of chromium(VI) using different masses of sludge activated carbon (AC) was maintained at conditions (shaking speed = 170 rpm, T = 30°C, pH = 3, C = 10 mg/L and t = 6 h). Chromium levels in each solution was measured using flame atomic absorption spectroscopy (FAAS). Removal efficiency and adsorption capacity were calculated using the following equations [24];

$$\text{Removal efficiency}(\%) = \left[\frac{C_0 - C_e}{C_0}\right] \times 100\% \quad (1)$$

$$q_e = \left[\frac{C_0 - C_e}{m}\right] \times V \quad (2)$$

where C_0 and C_e (mg/L) are the concentrations of Cr(VI) at initial and equilibrium in the solution respectively, q_e is the adsorption capacity (mg/L), V is the volume of the solution (L) and m is the mass of AC used (g).

2.4.1. Effect of dosage

The influence of sample dosage on chromium removal was investigated. This factor was studied by keeping other factors constant. The dosage effect was investigated by the following procedures. First, 10 ppm stock solution was prepared and transferred into a beaker, then H_2SO_4 (2M) was added to adjust the pH to 3. Different masses of AC (0.05, 0.1, 0.2, 0.3, 0.4, 0.5, 1.0 and 1.5) g were weighted and transferred into 500 mL conical flasks. Then, 50 mL of the 10 ppm (pH = 3) standard was added to each conical flask. All comical flasks were kept in a shaking incubator at the following conditions: shaking speed = 170 rpm, T = 30°C, pH = 3 and t = 6 h. After 6 h the solutions were taken and filtered. Standards and samples were measured by AAS [24].

2.4.2. Effect of concentration

This experiment was done to study the effect of increasing the concentration on the percentage removal. A series of chromium standards (0.25, 0.5, 1, 2, 4, 5, and 10 mg/L) were prepared from 1,000 ppm chromium stock solution in 100 mL volumetric flasks and filled with distilled water. The pH of each standard was then controlled to pH 3. Then, 0.5 g of the AC was added to each conical flask containing 50 mL the prepared concentrations. The remaining 50 mL of each prepared standards was poured without addition of AC into another 500 mL conical flasks. All conical flasks were put in the shaking incubator at conditions (shaking speed = 170 rpm, T = 30°C, pH = 3 and t = 6 h). After 6 h all solutions were filtered and analyzed for chromium levels using AAS [24].

2.4.3. Effect of pH

The influence of pH on the percentage removal was examined. A series of chromium standards (pH 2, 3, 4, 5, 6, and 7) were controlled. The concentration of each standard equal 10 ppm was prepared from 1,000 ppm chromium stock solution in 100 mL volumetric flasks and filled with distilled water. Then, 0.5 g of the AC was added to each conical flask containing 50 mL of the prepared solution. The remaining 50 mL of each prepared standards was poured without addition of AC into another 100 mL conical flasks. All conical flasks were kept in the shaking water bath at conditions (shaking speed = 170 rpm, T = 30°C, conc. = 10 ppm and t = 6 h). After 6 h, all solutions were filtered and analyzed for chromium levels using AAS [24].

2.5. Activated carbon treated with iron and copper

2.5.1. Synthesis of AC/Fe

In this experiment 10% hydrated ferric nitrate was added to AC by weighing 7.0 g of AC and dispersed in 150 mL of DDW, 100 mL of ethanol was added and sonicated for 5 h. Then, 20 mL ethylene glycol was added followed by shaking for 2 h. Next, 12.53 mL solution containing 5.06 g of ferric nitrate was added dropwise to make 1 M solution. After that, the pH was maintained at 8–9 pH, followed by heating at 80°C for 6 h with stirring. Finally, precipitate was filtered, washed with DDW and dried at 110°C overnight [12].

2.5.2. Synthesis of AC/Cu

The prepared activated carbon was treated with copper. 3.3625 g of $CuCl_2$ was added to 50 mL of DDW in 100 mL conical flask. This solution was poured in 4 g sludge AC, then left for 6 h under sonication. After that, product was filtered and dried at 90°C for 48 h [25].

2.5.3. Chromium(VI) adsorption using AC/Fe and AC/Cu

This experiment was used to investigate effect of adding iron and copper to the activated carbon as catalyst in chromium(VI) removal. The pH was controlled to pH 3 for several 10 mg/L Cr(IV) standards. Then, 0.5 and 1.0 g of the sludge AC/Fe was added to 100 mL conical flask of each of the prepared solution. All conical flasks were kept in the shaking water bath at conditions (shaking speed = 170 rpm, T = 30°C, conc. = 10 ppm and t = 6 h). After 6 h all solutions were filtered and analyzed for chromium levels using AAS [26]. Similar procedure was followed with using sludge AC/Cu.

2.5.4. Leaching of Fe and Cu after adsorption process

Treated standards with AC and AC/Cu sample from sludge with (1 g, pH3 and 10 ppm Cr(VI)) were analyzed for Fe and Cu levels [27].

3. Results and discussion

Chromium(VI) is one of the heavy metals that are harmful to the environment and human health. This paper is aiming to test the effectiveness of activated carbon prepared from sewage sludge for Cr removal. To achieve this aim, activated carbon was prepared using physical and chemical activation. The influence of experimental parameters such as adsorbent dosage, adsorbate concentration and pH on Cr removal was examined

Moreover, the results of the addition of other ligands (iron or cupper) to improve the efficiency of activated carbon removal for chromium will be detailed.

3.1. SEM-EDX results of the prepared activated carbon

Different methods are used to remove chromium from waste such as ion exchange, chemical reduction and precipitation, reverse osmosis, and adsorption using activated carbon [28]. Conventional methods are limited because of high costs and long treatment times; however, adsorption is simple and cost effective. Adsorption using activated carbon prepared from waste materials is an effective technology because of its well-developed porous texture and high surface area. Conversion of waste sludge to an adsorbent is considered a challenge for many researchers due to its association with human waste [29]. In this paper, activated carbon was prepared from waste sludge to address the issue of waste accumulation.

Fig. 1 shows SEM images of the surface morphology of the prepared activated carbon. The chemical composition of the prepared activated carbon was done by energy-dispersive X-ray spectrometer (EDXS) as it is shown in Fig. 1. The activation is clearly shown in the image with the highest peak is for carbon which shows 79.8%. The carbon atom has highest percentage which is 79.8%, then the oxygen element with 15.4%. Other elements such as Si, K, Al, Cl, Mg are of very small percentage (4.9%). These elements can be from the original sludge.

3.2. Removal of chromium(VI) using the prepared sludge activated carbon

3.2.1. Effect of adsorbent dose on adsorption

Fig. 2 displays the adsorption results for adsorbent dose of 0.05g/50 mL to 1.5 g/50 mL. The removal of 10 mg/L chromium(VI) was studied at pH = 3 with different dosage of activated carbon (AC).

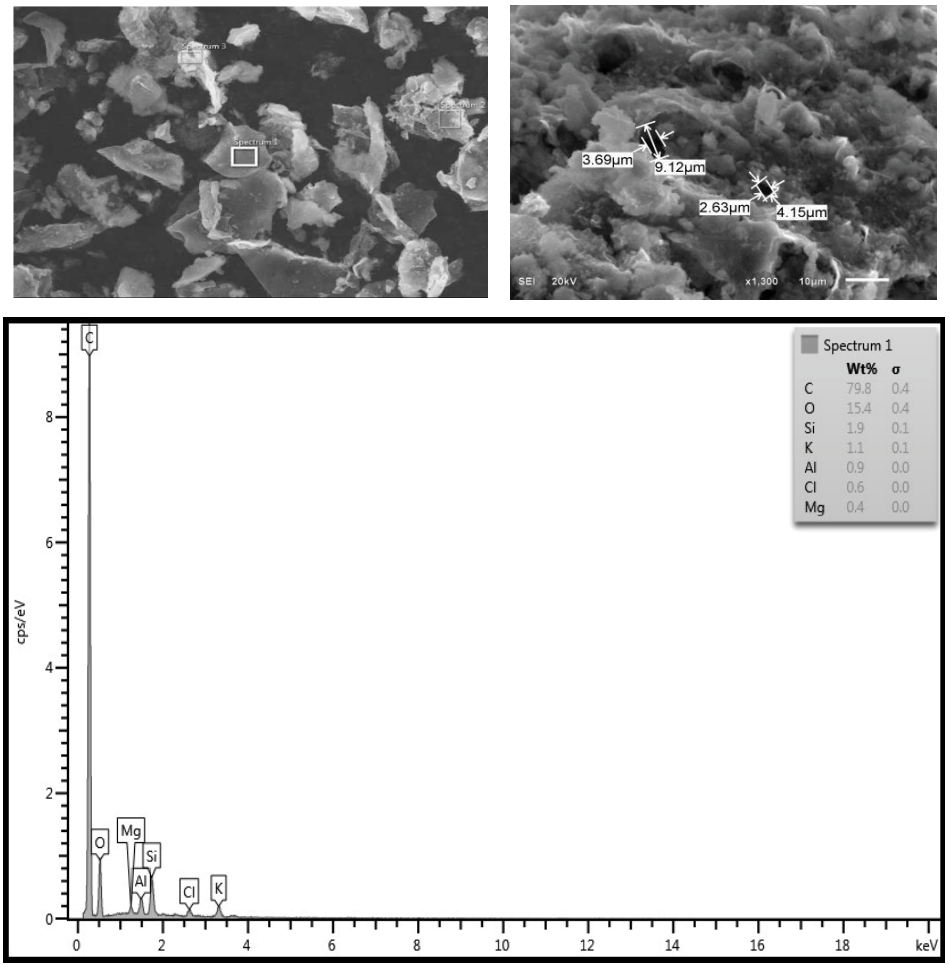

Fig. 1. SEM and EDX images of the prepared activated carbon (AC).

Fig. 2 shows that the removal has increased from 5% to 23% when the dosage was increased from 0.05 to 1.5 g respectively. This can be directly linked to the availability increasing adsorbent dose active sites leading to an increase in the effective surface area resulting from the increase of adsorbent dose [24,30]. Adsorption takes place via mass transfer by which substances are transferred from wastewater by adsorption on the solid surface. It permits a large surface area for adsorption which is then bonded by chemical and physical interactions [31].

The equivalent (mg/g) of chromium with initial concentration 10 mg/L at pH = 3 was studied at different dosage. Fig. 3 shows that the equivalent at 0.05 g was 0.40 mg/g. When a mass of 0.4 g of AC was added the equivalent was 0.19 mg/g. The addition of 1.5 g AC showed the lowest equivalent 0.07 mg/g. indirect relationship between AC dosage and percentage of equivalent of chromium was observed in Fig. 3. Hence, when the mass of AC increases the equivalent of chromium decreases according to Eq. (1) [24].

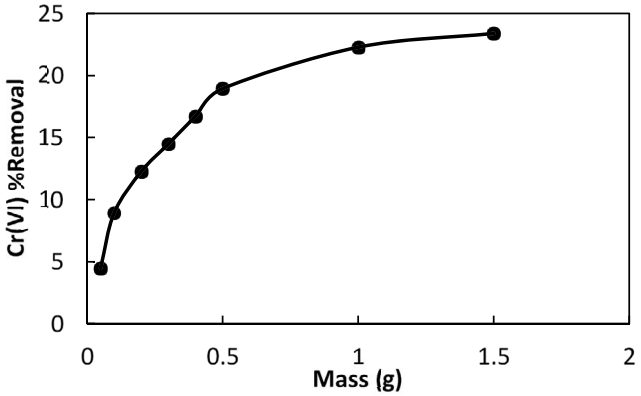

Fig. 2. Effect of dosage on the percentage of Cr(VI) removal. Conditions: shaking speed = 170 rpm, T = 30°C, pH = 3, C = 10 mg/L and t = 6 h.

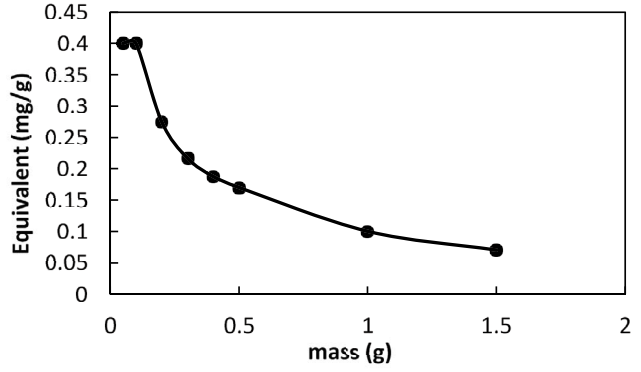

Fig. 3. Effect of adsorbent dose on metal uptake (q_e) of Cr(VI) (shaking speed: 170 rpm, temperature: 30°C, pH 3, initial conc.: 10 mg/L and time: 6 h).

3.2.2. Effect of initial concentration of chromium(VI)

The effect of initial concentration of chromium(VI) on the percentage removal by the prepared sludge activated carbon is illustrated in Fig. 4. It is clear from the figure that the removal increased with increasing chromium(VI) concentration. The maximum removal of 80% was achieved for the initial chromium concentration of 10 mg/L. In Labied et al. [32] study, the maximum Cr removal of 90% was also observed at an initial concentration of 10 mg/L at pH 2 and adsorption time of 2 h. However, the adsorption efficiency was observed to decrease after this concentration, and it reached 78% at initial concentration of 50 mg/L.

Table 1 shows a comparison between this study and two other studies used activated carbon for the removal of Cr(IV). The two studies used different raw materials of activated carbon including shaddock peels [9] and longan seeds [24]. According to the table mentioned above, raw material used to prepare the activated carbon affected the adsorbent metal removal efficiency. The solid sludge activated carbon (1.5 g) showed a higher removal efficiency (78.89%) with lower equivalent (0.79 mg/g) compared to longan seeds. This can be due to the differences in deferent factors such as porosity, surface area, condition of preparation and condition of process [24].

3.2.3. Effect of solution pH

The uptake of heavy metals from wastewater is controlled by the pH. It strongly influences the degree of

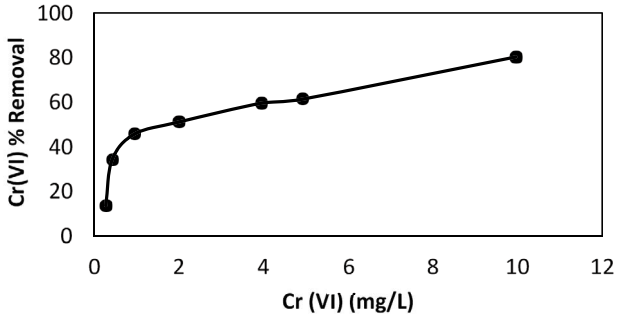

Fig. 4. Effect of concentration on the percentage of removal of Cr(VI). Conditions: shaking speed = 170 rpm, T = 30°C, pH = 3, m = 1.5 g and t = 6 h.

Table 1
Comparing removal % with different types of AC

Raw material of AC	Equivalent (mg/g)	C_0 (mg/L)	pH	Removal %	References
Solid sludge	0.79	10	3	78.89	This project
Shaddock peels	9.95	50	2	99.2	[9]
Longan seed	35.02	100	3	62.5	[24]

ionization of adsorbate and the surface charge of the adsorbent during reaction.

The removal of 10 ppm chromium(VI) by sludge activated carbon at different initial metal pH was studied. The pH of the adsorption medium was varied between 2 to 7 and other adsorption parameters were kept constant at shaking speed = 170 rpm, T = 30°C, m = 0.5 g and t = 6 h). The maximum removal was observed at pH 3. Yang et al. [24] reported similar results where they also got maximum adsorption of Cr(VI) by longan seed activated carbon at pH 3. After pH 3, there was a sharp change in adsorption of Cr(VI) because of the competitive adsorption between chromate ions and hydroxyl ions [24].

3.3. Removal of chromium(VI) using sludge S-AC/Fe and S-AC/Cu

To enhance the adsorbent activity, activated carbon was impregnated with iron and copper. It has been reported that activated carbon loaded with copper and iron is effective for removal of different contaminants from aqueous solution [32]. For this reason the prepared sludge activated carbon was impregnated with copper and Iron for the purpose of enhancing chromium(VI) removal. Fig. 5 shows that the modification of sludge activated carbon (0.5 g) using copper resulted in enhancing the Cr(VI) removal to 94% at pH 3. The high Cr (IV) removal with carbon modified with copper is attributed to the increase of carbon stability due to formation of strong metal-carbon complexes that could be as Cu^{+}/Cu^{+2} or as metal oxides at the surface. The presence of these complexes has been reported to increase the positive charge density of the surface [32]. The presence of these complexes results in the electrostatic attractions between positively charged surface and the negatively charged chromium ions. The improved sorbent efficiency for contaminant removal using metal-doped activated carbon has been reported in other studies [32]. However, in this study the Cr(VI) removal decreased sharply to 2.7% with impregnation of iron.

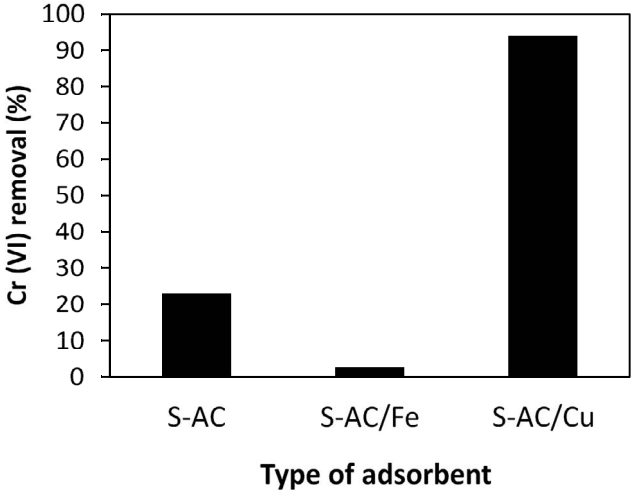

Fig. 5. Percentage removal of Cr(VI) using the modified activated carbons.

Table 2 shows the effect of adding Cu(II) metal to the prepared sludge activated carbon in order to enhance Cr(VI) removal in comparison with other studies that used the activated carbon without metals. This study shows that treating sludge AC with Cu(II) resulted in increasing the % removal from 23% to 94.57% at pH 3. Malwade et al. [33] used *Leucaena leucocephala* for preparing AC and 54% removal of Cr(VI) was observed at pH 4 and 100 ppm initial concentration. Moreover, Yang et al. [26] used Cassava Sludge AC and reported 60% removal of Cr(VI) with initial concentration of 10 ppm Cr(VI) and pH 4 [26]. As can be seen from Table 2 Cu(II) impregnated AC is showing better efficiency in Cr(VI) removal compared to other studies as it can increase the stability of the formation of metal-AC complexes.

3.4. Leaching of Fe and Cu after adsorption process

Leaching experiment was done to test the possible leaching of the catalysts from the activated carbon to the treated water sample. After adsorption process using S-AC/Fe it was found that solution has very low levels of iron (0.09 ppm). This mean that there is very minimal leaching of Fe from AC to solution after adsorption process [26].

The same experiment was done for the S-AC/Cu. After adsorption process using AC/Cu sludge, 0.01 ppm Cu was found in the treated water after adsorption. This mean that there is less leaching of Cu from AC to solution after adsorption so the AC/Cu should be washed after synthesis [27].

4. Conclusion

This study focuses on the removal of chromium(VI) using sludge activated carbon. The results showed the removal of 10 mg/L chromium at different dosage. The removal of chromium was studied at different concentrations. There was a direct proportional between AC dosage and percentage removal of chromium. The removal of chromium was studied at different pH. The % removal was high at pH 3. The removal of Cr is always greater at high dosage of AC and high concentration of Cr. The maximum removal of Cr from wastewater can be achieved by using

Table 2
Comparing Cr(VI) removal % with different types of activated carbon

Raw material of activated carbon	Heavy metal	Catalyst	C_0 (mg/L)	pH	Removal %	References
Solid sludge	Cr(VI)	Cu(II)	10	3	94.57	This project
Leucaena leucocephala	Cr(VI)	–	100	4	54	[33]
Cassava sludge	Cr(VI)	–	10	4	60	[26]

the following conditions; shaking speed = 170 rpm, T = 30°C, pH = 3, m = 0.5 g and t = 6 h. To enhance the Cr removal efficiency, the sludge AC adsorbent was impregnated with Fe(III) and Cu(II). The chromium(VI) removal increased to 94.57% with treating the activated carbon with Cu(II). However, the highest removal of sludge AC/Fe was found to be 3.53%. Therefore, sludge AC impregnated with Cu(II) is an efficient adsorbent for chromium(VI) removal.

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