Characterization & Batch Sorption Study for Chromium (VI) Removal from Aqueous Solutions by Activated Carbon Adsorbent Prepared from Indigenous Sugarcane Bagasse

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Abstract

Chromium being a carcinogenic element present in drinking water in the less developed areas in the poor countries contributes to many infectious diseases. The removal of chromium traces from water needs to have an easy and efficient way for poor countries. Concerning this, a low-cost industrial bio-adsorbent based on bagasse (the sugar industry waste) is prepared and characterized for Cr (VI) removal from aqueous solutions. Preparation of the adsorbent is performed by carbonization and steam activation of sugarcane bagasse (SCB). The FTIR spectra and the morphology of the adsorbent before and after Cr (VI) removal was studied using FTIR and SEM. All the experiments were carried out in a batch process with laboratory-prepared samples to study the effects of pH, adsorbent dose, adsorbate concentration, shaking time and shaking speed. It was observed that the highest removal efficiency was achieved at pH=2, adsorbent dose=0.75 g, adsorbate concentration=60 mg/L, shaking speed=150 rpm, and shaking time=20 minutes. These results suggest that this bio-adsorbent can provide a simple, effective, and cheap method for removing Cr (VI) ions from effluents and water resources.

Keywords—Aqueous solution, sugarcane bagasse, activated carbon, sorption

1 Introduction

THE quality of water is largely affected by the effluent from industries containing toxic heavy metals. Cr is one of the heavy metals that occur in two forms, i.e., trivalent and hexavalent [1]. The compounds of chromium are widely used in magnetic tapes, finishing of metals, electroplating, wood protection, pigments, tanning of leather, manufacturing of chemicals, electrical-electronic equipment, textile dyes, antirust agents in water cooling, and catalysis etc. [1-3]. The effects of Cr (VI) are very harmful including the disorder of liver, skin, kidney, respiratory organ, bronchitis, perforation of nasal septum and ulcer formation[4-6].

Growing public concern about the risks associated with Cr (VI) contaminated water has led to strict international regulations. US Environmental Protec-

tion Agency and World Health Organization (WHO) recommended Cr concentration in drinking water below 50 $\mu\text{g/L}$. Pakistan has also adopted the same guidelines. In Pakistan, the industries such as leather tanning, metal finishing, chrome plating, textile, fabrication, cement, dye, and pigment generate abundant quantities of effluent with high Cr concentrations from 0.5 to 270 mg/L [3][7]. Therefore, it is essential that before discharge, these industries decrease Cr (VI) contents up to the level of acceptance. However, high costs of operating wastewater treatment systems along with the financial constraints make it one of the major problems, especially in developing countries. The traditional methods used for heavy metals removal from water and wastewater include oxidation, precipitation, electrolytic removal, membrane filtration, flotation, solvent extraction, bio reduction, ion exchange and sorption [4][8-10]. The financial problems faced by the industry needs to be investigated to find a cheap and environmentally friendly method. Sorption methods are cost-effective and safe to use. Activated Carbon

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(AC) possesses certain properties that make it adsorptive in nature. The material and the method of activation for AC preparation determine the surface functional groups. The pore structure can also be refined by the activation process. The large surface area up to $2000\text{ m}^2/\text{g}$ is provided by micropore, mesopore and ultra-micropore [1]. An unreasonable cost of AC limits its commercial use in developing countries. There are certain benefits of the biosorption such as the possibility of metal recovery, low-cost, minimum chemical or biological regeneration of sludge, high efficiency, etc. [11][12].

Waste materials like a grape waste, rice husk, fungal biomass, distillery sludge green algae, maple sawdust, hazelnut shell, algal bloom residue derived activated carbon, wood, rice straw-based carbon, coconut tree sawdust, almond nutshell and tea leaf waste have been studied for Cr (VI) removal [1][4][9], [13-18]. There are various functional groups attached to these materials. Some of these functional groups are aldehyde, aliphatic, alkene, hydroxyl, amide, aromatic nitro, silicate, sulphonate etc. [19][2]. FTIR studies indicated that the above functional groups are responsible for Cr (VI) sorption [6][20]. Since different materials identified in earlier studies are not easily available in Pakistan, therefore, the purpose of this study is to identify a readily available low-cost natural resources that could be used as bio-sorbent, especially for Cr (VI) removal from aqueous solutions.

In our local context, we selected SCB which is widely available in Pakistan. Pakistan produces about 16.6 million tons of SCB annually, which is used as fuel for boilers, and as raw material for the manufacture of pulp paper and boards [21]. Earlier, Gomes et. al. [15] carried out a study on chromium removal from aqueous solutions using SCB and rice husk. They conducted the batch sorption experiments to investigate the effect of different parameters, i.e., pH, adsorbent dose, and contact time.

Cronje et. al. [3] also carried out a study on the optimization of Cr (VI) removal employing developing AC from SCB. The SCB used in the study was treated with zinc chloride. Similarly, there are various other studies available in the literature, but these studies are limited as far as the optimization of the removal efficiency by variation in certain parameters are concerned. In the present study activated SCB prepared by carbonization and steam activation (SCB) was used for Cr (VI) removal from aqueous solutions. The Cr (VI) ion sorption experiments were conducted at batch scale, and in the influence of different parameters was investigated. Characterization and detailed morphological study were also carried out

2 Material & Methods

The following sections describe the material and methods used in this study.

2.1 Activated Granular Activated Carbon

Sugar Cane Bagasse (SCB) was collected from Matiari Sugar Mill Limited in Matiari district, which is located about 30 km from the study area of Mehran UET, Jamshoro, Sindh, Pakistan. This waste was utilized for activated carbon (AC) preparation to investigate Cr (VI) removal. Initially, SCB was ground and sieved up to the range of size from 4.0-1.7 mm. Bagasse was mixed with sugarcane molasses in a 1:0.5 w/v ratio. The homogenized sample was compressed using Simplot Mounting Press (model 20-1320) at a pressure of 5000 psi to form pellets of 2.5 cm diameter. The pellets were placed in a tubular reactor and pyrolyzed in Nitrogen (N_2) atmosphere at 700°C for 1 hour. The sample was then cooled overnight in the furnace under an inert atmosphere. Later, the sample was activated with carbon and carbonized for 45 min at 850°C in N_2 atmosphere. The carbonized carbon was washed using 0.1N HCl to remove ash and other adhering material. Carbon was washed dried and stored. Figure 1 shows the process of preparing pellets from the industrial sugar cane bagasse.

2.2 Standard Solution Preparation

Cr (VI) stock solution (1000 mg/L) was prepared by dissolving potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) in one litre of deionized water. To obtain initial Cr (VI) concentrations of 60 to 100 mg/L, the deionized water was added to the stock solution and diluted. The solution pH was adjusted from 2-10 using 0.5N HCl and 0.5N NaOH respectively. All analytical grade chemicals were used in this study.

2.3 Adsorbent Characterization

Analytical analysis of developed sorbent loaded with activated carbon was carried out through FTIR for observing sorption before and after the experiment. The spectral range was $4000\text{--}600\text{ cm}^{-1}$. The scanning electron microscope (SEM) characterization was carried out using Jeol JSM 6380 to observe the surface texture before and after activation, and after sorption. To measure porosity, the sample was placed into a Penetrometer and the Quanta-chrome Autocan-33 Model mercury intrusion porosimeter. For the measurement of the pore volumes between the ranges from 6.0000 to $0.0062\text{ }\mu\text{m}$ radius, the Penetrometer was filled with mercury and the system pressure increased gradually

Fig. 1: Process of preparing pellets from the industrial sugar cane bagasse

up to 1900 Kg/cm^3 . The calculation of the pore radius was done using the Washburn Equation [22]. For measuring the surface area, Quanta-chrome NOVA 1200 Model sorp-tometer was used. The system was heated for the removal of moisture and atmospheric gases. N_2 was introduced both in liquid and gas phase, and the temperature was gradually reduced to 30°C . The adsorption features of arsenic onto ICPG were considered under equilibrium and dynamic conditions.

2.4 Batch Sorption Experiments

The batch sorption experiments of Cr (VI) ion removal by activated SCB were carried out at ambient conditions of temperature. The solution (100 ml) containing 60-100 mg/l Cr (VI), 0.75 g of activated SCB was added, and pH was adjusted to 2 with 0.1 M HCl-KCl buffer (5 ml). The contents were mechanically shaken at 150 rpm for 20 minutes and filtered with filter paper (Whatman filter paper No. 42). The solution was analyzed on Variant Spectr AA 20 Plus using an air-acetylene flame. Triplicate analysis ($n=3$) was carried out with integration, and delay time of 3 seconds. The removal efficiency, i.e., sorption capacity of the developed adsorbent was calculated by using Equation 1.

$$\% \text{Sorption} = (C_0 - C_f / C_0) \times 100 \quad (1)$$

Where C_0 is the initial known concentration of metal, C_f is remaining (final) concentration of metal ion in the solution (mg/L) [13]. At an equilibrium time, the metal uptake (q_e) was estimated by using Equation 2,

$$w(q_e - q_0) = v(C_0 - C_f) \quad (2)$$

Where $q_0 = 0$, Eq. (2) will become:

$$q_e = v(C_0 - C_f) \times 100w \quad (3)$$

where q_e represents the adsorbed amount of metal i.e. Cr (VI), and C_e is the remaining (final) metal ion concentration (mg/L) at equilibrium time and these are

Characteristics	Bagasse	Activated bagasse
Specific surface area (m^2/g)	8.01	1537
Average pore diameter (nm)	5.01	38.7
Porosity (%)	23.22	64.42
Total pore volume (ml/g)	0.234	0.561

TABLE 1: Proprtirs of prepared activated carbon

measured directly by Varian Spectr AA20 Plus at the wavelength of 357.9 nm, v (ml) is the aqueous solution volume, and w is the weight (g) of the adsorbent.

3 Results & Discussion

This section presents the results of this study.

3.1 Characterization of Activated Carbon

In our study, the properties of activated carbon before and after activation were determined and the results are shown in Table 1. Analysis of results shows that the steam activation removed volatile impurities, creating a highly developed active porous surface, as well as increased the pore diameter, pore volume and specific surface area. The FTIR spectra of the adsorbents before and after Cr (VI) sorption indicated the number of peaks showing the functional groups. Figure 2a-2b exhibits several peaks. The FTIR spectra of the adsorbent before and after Cr (VI) removal is shown in Figure 2a-2b. The spectra were recorded with attenuated total reflectance accessory within $4000\text{-}600 \text{ cm}^{-1}$. The results of the absorption peaks with possible assignments are given in Table 2. The band frequency around 3707 cm^{-1} indicates the presence of hydroxyl ($-\text{OH}$) group and its substantial shift from 3707 to 3400 cm^{-1} reveals the Cr binding with ($-\text{OH}$) group. Similar assignments have been reported by Anand Kumar et. al. [19] for Bael fruit.

Fig. 2: Fourier transform infrared (FTIR) spectrum of activated bagasse

Adsorbent	Activated bagasse	
	before Cr (VI) binding	after Cr (VI) binding
O-H	3707	3400
-NH	3210	3227
C-H	3017	2970
-NH ₃	2261	2517
C=O	1525	1559
-CH ₃	1442	1396
-CH ₂	1351	1339
-CO	–	1020
-OCH ₃	–	–
Bending vibrations	994	
	838	650
	711	

TABLE 2: FTIR spectra of adsorbent before and after Cr (VI) sorption

When activated carbon was loaded with Cr (Figure 2b), the N-H absorption peak shifted from 3210 to 3227 cm^{-1} , which shows that this functional group was involved in metal binding. In C-H stretching, the vibration was observed within 3017-2950 cm^{-1} . Same observations have been reported for Bael fruit from 2924 to 2913 cm^{-1} [19]. The band 2261 and 2517 cm^{-1} are assigned to stretching vibrations of $-\text{NH}_3$ groups. This was due to the treatment of activated carbon with hydrochloric acid. The signal observed at 1525 and 1559 cm^{-1} could also be assigned to $\text{C}=\text{O}$ vibrations. Additional peaks at 1442 and 1351 cm^{-1} are assigned to CH_3 and CH_2 which shifted to 1396 and 1339 cm^{-1} respectively. Additional peaks below 1000 cm^{-1} are due to vibrations of aromatic

compounds. Here sorption may be because of physical sorption, ionic exchange, complexation with functional groups, chemical reaction with surface sites, and surface precipitations. The porosity and surface texture of the prepared activated sample was recorded by the scanning electron microscope. In Figure 3a, SEM image of SCB, prior to activation is displayed showing the absence of pores or voids. The SEM of an activated sample in Figure 3b reveals well-developed pores like capillaries open at one or both ends.

3.2 Optimization of Developed Sorbent

The developed sorbent loaded with activated carbon was further tested by investigating different parameters to achieve an optimized performance. Therefore, the effect of different parameters including dosage of sorbent, pH, shaking speed and time and sorbate initial conditions were studied.

3.2.1 Effect of pH on the Sorption Capacity of Activated Carbon

Solution pH is essential to study because it influences metal sorption. Chromium ions exist in the form of HCrO_4^- at very low pH, while at higher pH up to 6, different forms such as $\text{Cr}_2\text{O}_7^{2-}$, $\text{Cr}_2\text{O}_4^{2-}$ and $\text{Cr}_3\text{O}_{10}^{2-}$ coexist, of which HCrO_4^- predominates. Equilibrium shifted from HCrO_4^- to CrO_4^{2-} and $\text{Cr}_2\text{O}_7^{2-}$ as the pH increases [9]. At lower values of pH, due to greater attractive forces, the hydronium ions would be surrounded on the adsorbent surface, thereby increasing Cr (VI) contact with binding positions of an adsorbent. The overall surface charge on the adsorbents turned out to be negative and sorption decreased due to the rise of pH [23].

At increased pH (≥ 5), there is a gradual reduction in the degree of protonation of the surface which

Fig. 3: SEM images of sugar cane bagasse (SCB): (a) untreated bagasse, (b) activated bagasse, (c) carbon activated bagasse

Fig. 4: Effect of pH on percentage sorption of Cr (VI)

Fig. 5: Impact of adsorbent dosage on percentage sorption of Cr (VI)

decreases the sorption. At pH=2, maximum sorption was achieved, therefore, further experiments were performed at pH=2. In this study, initially, the percentage sorption was studied at various pH values (2–10) at unit intervals using activated carbon 0.75g/100 ml. Initial Cr (VI) concentration of 100 mg/l was shaken for 20 minutes at 150 RPM. It was observed that the sorption of Cr (VI) increased in acidic conditions at pH=5 to 2. At pH=2, highest removal 75.21% was obtained as shown in Figure 4. These results are in agreement with the results of similar investigations carried out earlier [12].

3.2.2 Impact of Adsorbent Dosage on Sorption Efficiency of Activated Carbon

The impact of the adsorbent dose was investigated for the sorption of Cr (VI) by changing the adsorbent fraction (0.25-1.25g) using 100 mg/l Cr (VI) solution at the optimized condition. It was observed from Figure 5 that the sorption of Cr (VI) increased from 54.9% to 76% with an increase of adsorbent dose from 0.25 to 0.75 g/100 ml solution. Further increasing the adsorbent dose shows that the rate of sorption remained constant, which probably caused sorption

sites overlapping provided by the higher dosage of adsorbent [24].

3.2.3 Effect of Shaking Time on the Sorption Capacity of Activated Carbon

The effect of shaking time on Cr (VI) sorption was studied by varying the contact time from 5 to 40 minutes with a 5-minute interval. Rotary shaker was used for shaking the sample at 150 rpm with adsorbent dose 0.75 g/100ml. It can be observed in Figure 6 that with an increase in agitation time, the sorption capacity increased until equilibrium was obtained after 20 minutes. The amount of Cr (VI) adsorbed increased up to 86.66% within 20 minutes. Hence, for further experiments, the optimum shaking time of 20 minutes was selected. In the present study, the optimum shaking time (equilibrium time of 20 minutes) is less when compared with other adsorbents, i.e., coal: 45 minutes [25][26]; grape waste: 60 minutes [13], rice husk: 120 minutes[9], and Bael fruit: 240 minutes [19].

Fig. 6: Effect of shaking time on percentage sorption of Cr (VI)

Fig. 8: Effect of sorbate on percentage sorption of Cr (VI)

Fig. 7: Effect of shaking speed on percentage sorption of Cr (VI)

3.2.4 Effect of Shaking Speed on the Sorption Capacity of Activated Carbon

Experiments were conducted with various shaking speeds (90-200 rpm) at an interval of 20 rpm at optimized conditions as shown in Figure 7. Shaking at an appropriate speed makes the binding site available and provides intimate contact between the adsorbent and adsorbate. Agitation at low speed does not facilitate proper contact of metal ions and active site. Instead, the adsorbent settles at the bottom, rather disperses in the solution. High-speed agitation reduced the metal sorption process. Maximum sorption of 88.88% was achieved at 150 rpm, making all the surface binding sites readily available for Cr sorption.

3.2.5 Effect of Sorbate Concentration on the Sorption Capacity of Activated Carbon

The effect of the initial concentration of Cr (VI) varying from 60 to 100 mg/L was examined on the percentage sorption removal with 0.75g/100 ml of solvent. The sorption percentage increased from 80% to 98% as the metal ion concentration decreased,

as shown in Figure 8. While there is an availability of limited surfaces in case of higher concentrations, hence, it reduces the percentage sorption of Cr (VI). This trend is the same as reported in various literature [2][4][8]. The developed sorbent is found very effective for Cr(VI) removal from aqueous solution, however, there are not too many studies conducted on Cr(VI) removal through batch studies. The same efficiency range has been reported by some other studies for arsenic removal from groundwater including [27][28].

4 Conclusion

The indigenous adsorbent developed from SCB has shown a substantial potential for Cr (VI) removal from dilute solutions. The sorption capacity was found impressive. During the parametric study of sorbent, the maximum sorption 98% of chromium was achieved by optimization of different operational, chemical, and physical parameters. It was observed that sorption is dependent on various parameters such as pH, carbon dose, shaking time, speed, and concentration of the metal ion. Furthermore, the analytical investigations, i.e., FTIR and SEM have endorsed the sorption results as the variations were significant for the before and after samples. These techniques also confirmed the presence of Cr (VI) ions on the carbon due to the activation process which significantly increased the removal efficiency of the adsorbent. The development sorbent can be applied for industrial and commercial applications for the compliance of local and international set-regulation for effluent treatment standard. Furthermore, this study was a batch scale which can be extended to a continuous or semi-continuous scale with local and regional government support.

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