Chemical Modification of Banana Peels and Banana Pseudostemfor the Adsorptive Removal of Chromium (VI) from Aqueous Solution

Sujata Adhikari[1], Krishna Subedi[1], Sabin Dhungana[1], Ram Lochan Aryal[1],Hari Paudyal[1], Kedar Nath Ghimire[1], Bhoj Raj Poudel[1,2, *], Megh Raj Pokhrel[1]

[1]Central Department of Chemistry, Institute of Science and Technology, Tribhuvan University, Kirtipur, Kathmandu, Nepal

[2]Department of Chemistry, Tri-Chandra Multiple Campus, Tribhuvan University, Kathmandu, Nepal

*Corresponding E-mail: chembrpoudel@gmail.com

Abstract:

The removal of Cr(VI) from the water was studied using the raw and modified banana peels and pseudostem wastes as bio-adsorbent under the batch adsorption technique at room temperature. The chemical modification of raw adsorbents was performed by treating with conc. sulphuric acid. Characterizations were performed by FTIR, XRD, and SEM analyses. The raw form of adsorbents showed insignificant adsorption compared to the charred form. Therefore, the further process was carried out using modified adsorbents only. The maximum adsorption capacity for charred banana peel (CBP) and charred banana pseudostem (CBS)was found to be maximum at optimum equilibrium pH 2. The adsorption data fitted well with the Langmuir adsorption model and followed pseudo-second-order kinetics. Therefore, owing to high efficiency and low cost with maximum removal percentage, thus prepared adsorbents are expected to be used satisfactorily in the adsorption of Cr(VI) from aqueous solutions.

Keywords: *Adsorption; Characterization; Chromium(VI); Isotherm; Kinetics*

Introduction

Chromium is the earth's 21st most abundant element and the sixth most abundant transition metal. It is one of the top 16 heavy metals which has a destructive effect on human health. Chromium exists usually in both the hexavalent chromate Cr(VI) and trivalent chromite Cr(III) forms in contaminated water streams [1]. The toxicity of chromium depends upon its oxidation state. Cr(III) is relatively insoluble and required by microorganisms as an essential trace metal nutrient, while Cr(VI) is regarded to have great concern because of its toxicity [2].

In a solution, the Cr(VI) exists in various forms depending upon the pH, such as chromate $(Cr_2O_4)^{2-}$, hydro-chromate $(HCrO_4)^-$ or dichromate $(Cr_2O_7)^{2-}$

[2,3]. The sources of chromium are industrial as well as laboratory effluents [4]. The WHO recommendation limit for the Cr(VI) contamination in aqueous sources is only 0.05 mg/L [5]. The toxic implications of chromium in the environment have made the US Environmental Protection Agency (USEPA) set the level of Chromium in drinking water to 0.1 mg/L [6]. But the industrial and mining effluents are found to contain a much higher concentration of chromium as compared to the permissible limit. Cr(VI) can cause serious health problems like respiratory infections, weakness in the immune system, liver and skin problems, lung cancer, and death. Inhalation of hexavalent chromium can affect badly the larynx, pharynx, and lungs, and bioaccumulation of Cr(VI) in essential organs could damage metabolic functions

and have carcinogenic, mutagenic, and teratogenic impacts on living beings [7,8]

Several treatment technologies have been evolved to immobilize Cr(VI) from aqueous environments including, the conventional chemical process like chromium hydroxide precipitation along with other methods such as ion exchange, reduction, electrochemical precipitation, solvent extraction, membrane separation, evaporation, foam separation, electro-dialysis and so on[9]. The continuing increase in the variety and amount of hazardous chemicals in effluents make conventional separation methods inefficient and sometimes even ineffective and inadequate. Therefore, compared to other techniques, adsorption is the most favorable process and the preferred one because of its high efficiency, low price, and non-hazardous technique of complete removal of metal ions even at its low concentrations [10].

Several researchers have used varieties of agricultural wastes for the preparation of low-cost adsorbents from cheaper and readily available materials for the removal of toxic metal and non-metal ions from water [11,12]. For the removal of heavy metals from water, such as Cr(VI), Cu(II), Pb(II), and Zn(II) [13], as well as Pb(II) [14], Cd(II) [15], and Cr(VI) [16], raw and modified banana peels have been used in a number of studies. However, no prior reports on Cr(VI) biosorption using banana peels and banana pseudostem wastes by treating with Conc. H_2SO_4 have been found. In this work, a novel material for Cr(VI) biosorption has been prepared after charring banana peels and banana pseudostem. Among many agricultural wastes used as an adsorbent, banana waste has been significant because various parts of banana can be utilized such as banana fruit, peels, trunks, pseudostems, leaves, and piths. It contains carbon-rich organic compounds such as cellulose, hemicellulose, chlorophyll pigments, pectin, lignin that constitute fibers with good mechanical properties, and some other low molecular weight compounds [17]. The pectin substances are complex heteropolysaccharides containing galacturonic acid, arabinose, galactose, and rhamnose as the major sugar constituents; the carboxyl groups of galacturonic acid

allow pectin substances to strongly bind metal cations in an aqueous solution [18].

Chemical modification is the most frequently used technique for the preparation of efficient adsorbentsfrom raw biomass. In the present study, it has been attempted to chemically modify the raw adsorbents treated with the concentrated sulphuric acid solution for the adsorptive removal of Cr(VI) from the aqueous solution.

Materials and Methods

Chemicals

Reagents of analytical grade were used; further purification was not done to them. A 1000 mg/L potassium dichromate stock solution was made in a 0.1N nitric acid solution. 1000 µg $HCrO_4^-$ in the form of Cr(VI) are included in 1 mL of stock potassium dichromate solution (VI). To makeworking solutions, dilutions in 0.1N nitric acid were performed. To keep the pH constant, a 0.1 M NaOH and 0.1 M HCl solution was utilized.

Preparation of adsorbents

Crude samples of banana peels and banana pseudostem, collected from the local agricultural farm of the Chitwan district of Nepal, were cut into pieces and thoroughly cleaned. The samples were dried in sunlight and grounded into powdered form by a mechanical grinder (mill) and sieved through 212 µm pore size sieving device. Thus, prepared powered adsorbents were stored in an airtight container, and designated as raw banana peel (RBP) and raw banana pseudostem (RBS) and applied for further adsorption experiments.

100 g of RBP and RBS both were treated with 200 mL of concentrated sulphuric acid and allowed to soak for 2 days at room temperature for complete reaction. Later, it was washed with distilled water several times till neutrality. Finally, it was dried in a hot air oven at 80 °C for 6-8 hours and sieved through a 212 µm pore-sized sieving device. Thus, prepared adsorbents were charred banana peel (CBP) and charred banana pseudostem (CBS) adsorbents respectively.

Adsorption experiment

The amount of Cr(VI) adsorbed at equilibrium was determined from the formula below:

$$q_e = (C_i - C_e)\,V/W \qquad (1)$$

Where C_i and C_e are the concentrations of Cr(VI) in mg/L at initial and equilibrium states respectively. V is the volume of Cr(VI) used in a liter (L) and W is the weight of adsorbent used in gram (g). Similarly, the percentage removal of metal ions was calculated from the following formula:

$$\text{Percentage Removal}\,(A\%) = \frac{C_i - C_e}{C_i} \times \qquad (2)$$

Characterization of adsorbent

The surface functional groups were analyzed using FTIR spectroscopy on IR AFFINITY -1 Shimadzu (Shimadzu, Kyoto, Japan) spectrometer in the wavenumber range of 400 – 4000 cm^{-1}. The crystallinity of the bio-adsorbent was examined using X-ray diffraction (XRD) patterns by an X-ray Diffractometer (Rigaku Co., Japan) with Cu Kα (λ = 1.54056 Å) radiation. The surface morphological characteristics were investigated using scanning electron microscopy (FE-SEM; JEOL, JSM-6701F, Japan). The spectrophotometric analysis was carried out by using WPAS UV-Vis spectrophotometer of model 2306 (Electronics India) and with the help of Deluxe pH meter MODEL ME-963P (Max Electronics, India).

Results and Discussion

Characterization of Adsorbent

The SEM images of RBP and RBS showed that the fibers are stuck together due to the presence of lignin, pectin, and other viscous compounds that are changed to more porous structures with different shapes and sizes in CBP and CBS. This may be due to the treatment with conc. sulphuric acid may be due to the cleavage of polymeric materials of raw adsorbents converted into smaller particles with large surface areas [19]. Similarly, the shape of the adsorbent surface was shifted from a smaller and irregular pattern before sorption to a more complex agglomerated, much smoother, and shining pattern after Cr-loading which could be due to the filling of void spaces with Cr(VI) anions by interaction with the Cr-contaminated water [20]. These surface properties can be a factor providing an increase in the
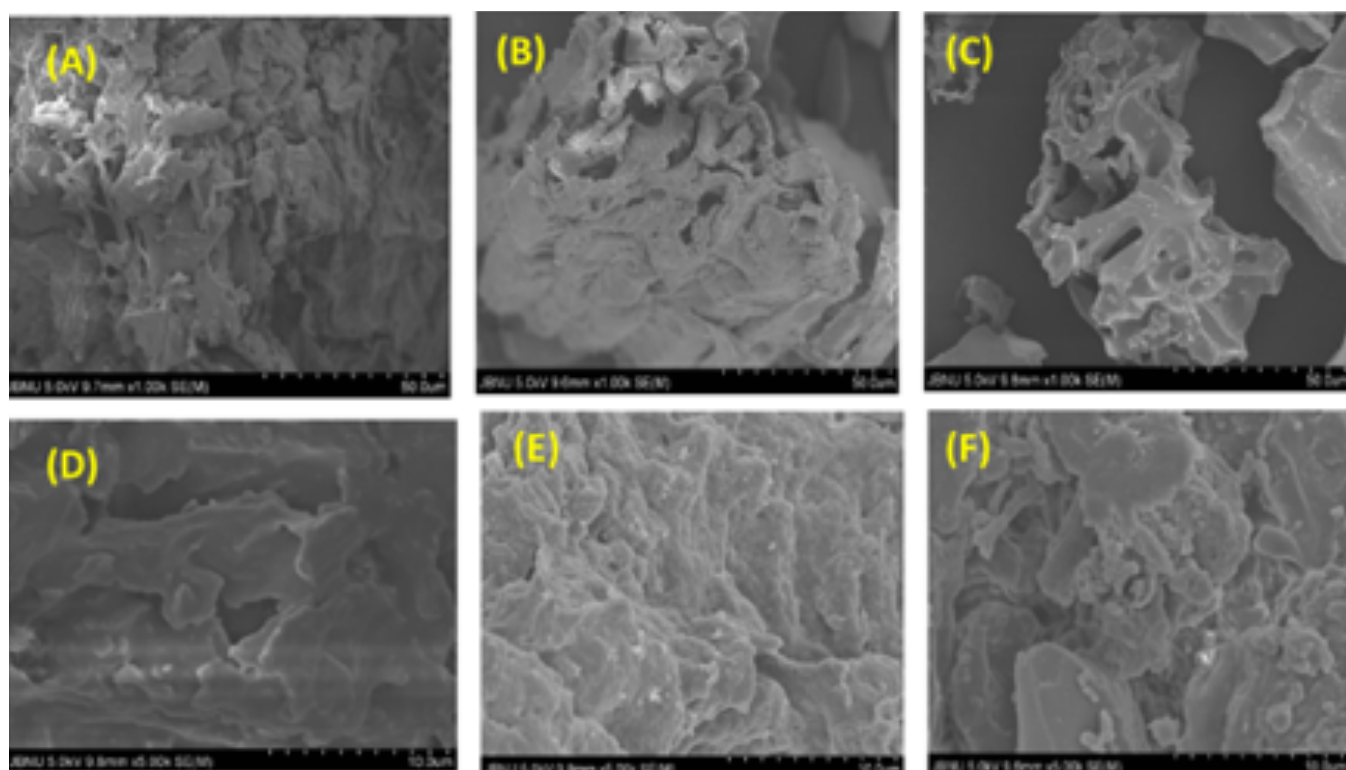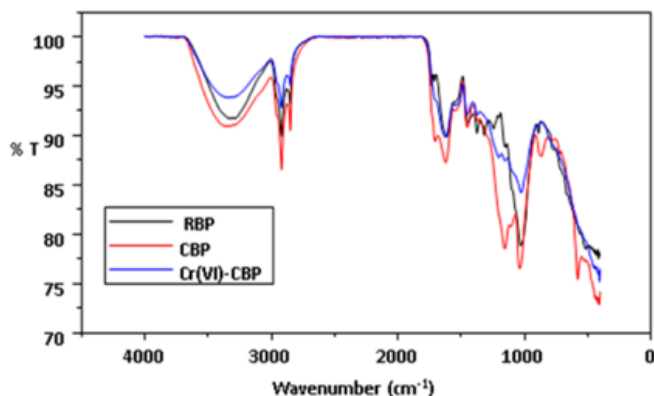

Figure 1. *SEM images of A) RBP B) CBP C) Cr(VI)-CBP D) RBS E) CBS and F) Cr(VI)-CB*

total surface area. In addition, the pore structures of the adsorbents could reduce the diffusional resistance and facilitate mass transfer because of their high internal surface area [21].

Figure 2. *FTIR spectrum of RBP, CBP, and Cr(VI)-CBP adsorbents.*

The spectral data of FTIR of all adsorbents RBP, CBP, and Cr(VI)-CBP are shown in Figure 2. The FTIR spectra of RBP appeared similar to the observations of Memon et al. 2008. The broad peak at 3360 to 3700 cm^{-1} was observed due to the stretching vibration of hydroxyl groups (OH) that existed in cellulose, hemicellulose, and lignin [22]. This peak in the case of CBP is wider than RBP which may be due to the ring-opening of the cellulose molecule and the large number of OH groups present in it. The spectra displayed the major bands at 3265 cm^{-1}, 2920 cm^{-1}, 1651 cm^{-1}, 1589 cm$^{-1,}$ and 1478 cm^{-1} which are assigned to be OH stretching, C-H stretching, C=O stretching in carboxylic acid or ester, C=C stretching and C-O-C stretching vibration of ether respectively which have reduced intensity in case of CBP. FTIR spectra of Cr-adsorbed CBP didn't show any sharp peak rather it disappears [23]. Therefore, this observation indicated the significance of available oxygen in hydroxyl and carbonyl groups, which has a profound effect on the surface characteristics of the banana peel [24]. The functional groups play an important role in the adsorption of the metal ions by donating electrons to heavy metal ions.
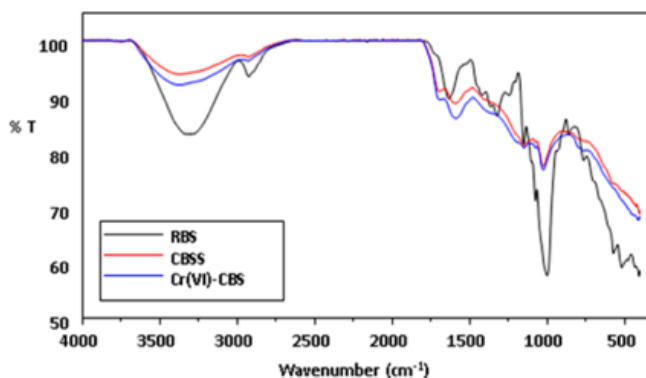
Figure 3. *FTIR spectrum of RBS, CBS, and Cr(VI)-CBS adsorbents.*

The FTIR spectra of RBS, CBS, and Cr(VI)-CBS are shown in Figure 3. The absorption bands at 3325 cm^{-1} to 3376 cm^{-1} can be assigned to stretching vibrations and other polymeric associations of hydroxylic groups. The symmetric stretching at 2925 cm^{-1} is associated with -CH$_2$ groups present in polysaccharides in RBS [25]. Thus, FTIR data interpreted that RBS contains several functional groups having cellulose, hemicellulose, and lignin suggesting an aromatic character that created a suitable environment for interaction between adsorbent and metal ion [26]. The FTIR spectra of treated adsorbent i.e., CBS and Cr(VI)-CBS did not show any significant changes, suggesting only a small reduction of organic compounds of the adsorbent.

The XRD analysis was used to find out the nature of the adsorbent whether it is crystalline or amorphous in nature.
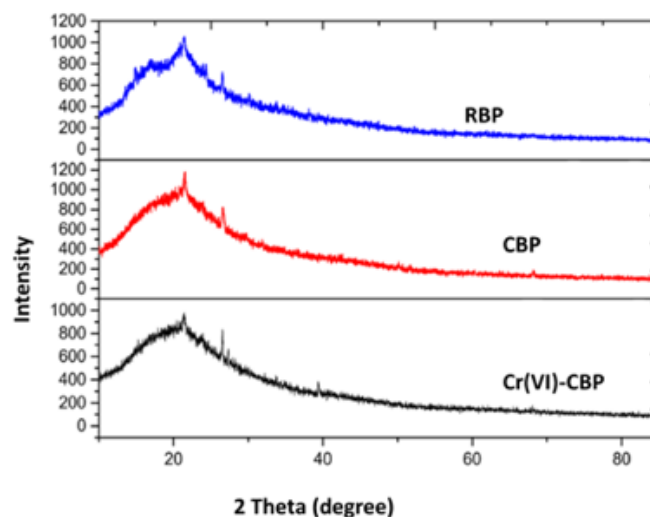
Figure 4. *XRD pattern of RBP, CBP, and Cr(VI)- CBP.*

From Figure 4, it can be seen that minor peaks can be observed at an angle of 16.6°, 21.44°, and 26.4° representing the typical characteristics of peak positions of cellulose. Similarly, other peaks and humps in RBP suggest they tend to be an amorphous phase too. The XRD spectra of CBP show after chemical activation, the peak intensity becomes lower and broader, reflecting a decrease in crystallinity. Therefore, the observation suggests that the carbon content present in CBP may be mainly amorphous in texture [27]. This result can be interpreted as amorphous materials showing better absorbance due to their high surface area and the greater number of active sites [28].
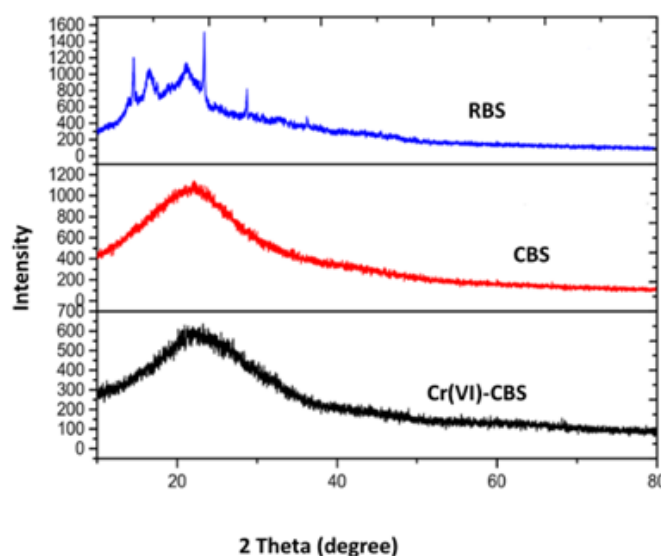
**Figure 5.** *XRD pattern of RBS, CBS, and Cr(VI)- CBS.*

As can be seen from this figure above, the XRD spectrum of RBS consists of peaks at 2θ equal to 14°, 17.14°& 22.1° which are the typical characteristics of peaks positions of cellulose with other diffused peaks [29].Also, the results show that RBS contains many ordered as well as disordered regions whichimply that RBS consists of cellulose with both amorphous as well as crystalline regions. Moreover, after the chemical treatment of raw adsorbents to CBS, the strong sharp peaks got destroyed and a new broad hump can be seen as in Figure 5. The amorphous nature of CBS can be ascribed to the progressive removal of hemicellulose, lignin, and other amorphous non-cellulosic compounds induced by the chemical treatment (charring) due to ring-opening reaction.

Lastly, the reduction in the intensity and broader hump of spectra is observed for Cr(VI)- CBS [29].

Effect of pH for Adsorption of Cr(VI)

To evaluate the influence of pH on the adsorption process, batch adsorption experiments on pH studies were carried out at different initial pH ranging from 1 to 8.

Figure 5 shows the effect of pH on the adsorption of Cr(VI) onto all the four used adsorbents (CBP,RBP, RBS, and CBS) at an initial concentration of 20 ppm Cr(VI) solution at room temperature. From the experiment, it is revealed that the adsorption of Cr(VI) was found to be effective at pH 1 and 2 for all four adsorbents, and the adsorption capacity of Cr(VI) is decreased with the increase in pH. Previous studies reported in the literature suggested that at low pH with the increase in proton, there occurs a reduction of Cr(VI) ions to Cr(III) rather than being adsorbed to the adsorbent [30]. Therefore, from this fact, and following the literature survey, it is concluded that pH = 2 is the best optimum pH value for the maximum removal efficiency or adsorption of Cr(VI) ions [31,32]. However, the raw form of adsorbents didn't show an appreciable adsorption percentage regarding modified ones. Therefore, due to quite low (negligible) Cr(VI) adsorption by RBP and RBS, further studies were carried out using modified adsorbents only i.e. CBP and CBS.
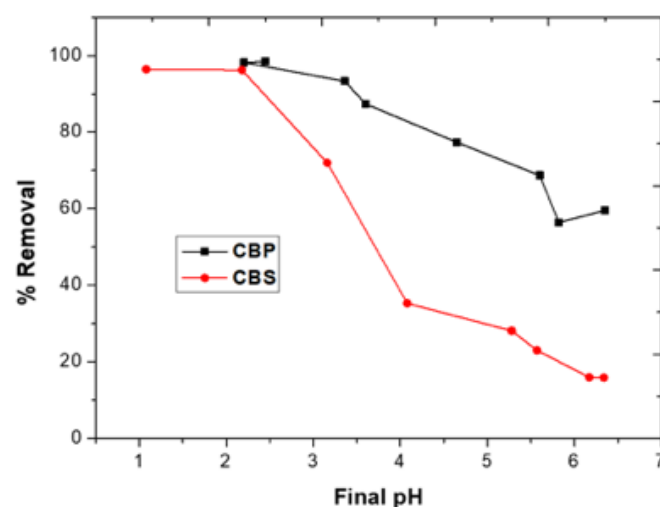
Figure 6. *Effect of pH on the adsorption of Cr(VI) onto CBP and CBS(Conditions: Initial Cr(VI) concentration = 20 mg/L, Total volume of sample = 20 mL, Shaking time = 24 hours, Adsorbent dose = 25 mg)*

Adsorption Isotherm

The equilibrium relationships between adsorbent and adsorbate are best explained by the adsorption isotherm. To evaluate the best-fit isotherm model, the experimental data obtained were analyzed with both the Langmuir and Freundlich adsorption models.

The empirical formulation of the Langmuir model assumes monolayer adsorption (the adsorbed layer is one molecule in thickness), with adsorption only occurring at a fixed number of definite localized sites, that are identical and equivalent, with no lateral interaction and steric hindrance between the adsorbed molecules, even on the adjacent sites [33].

$$\frac{c_e}{q_e} = \frac{1}{q_m K_L} + \frac{c_e}{q_m} \tag{3}$$

Where, q_e is the amount adsorbed at equilibrium (mg/g), c_e is the equilibrium concentration of adsorbate in (mg/L), q_m is the maximum adsorption capacity (mg/g) and 'k_L' is the Langmuir adsorption constant (L/mg). When c_e/q_e is plotted against c_e, then a straight-line having a slope $1/q_m$ and an intercept $1/q_m k_L$ is obtained. From this graph, the value of q_m and 'k_L' can be determined.

Freundlich model can be applied to multilayer adsorption in heterogeneous systems, especially for organic compounds or highly interactive species on activated carbon describing the non-ideal and reversible adsorption, not restricted to the formation of a monolayer.

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \tag{4}$$

where q_e is the amount adsorbed (mg/g), c_e is the equilibrium concentration of the adsorbent (mg/L), K_F and n are the Freundlich equilibrium coefficients, considered to be relative indicators of adsorption capacity and adsorption intensity.
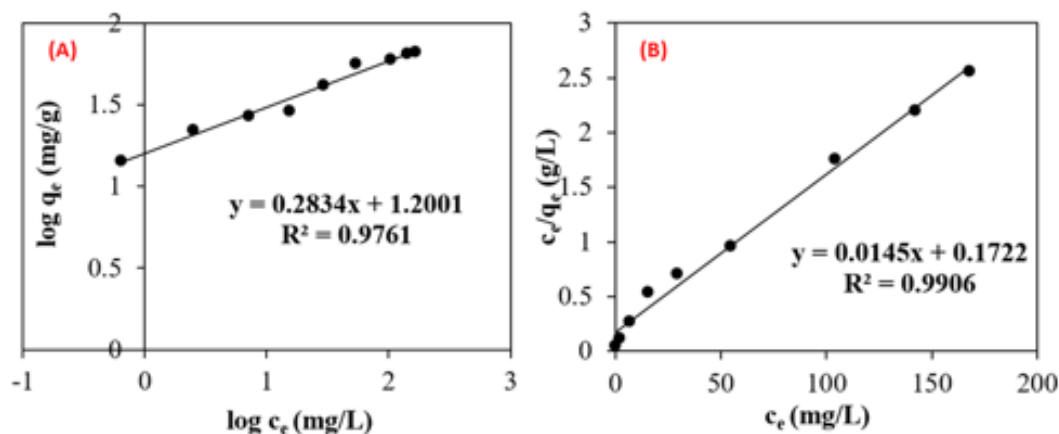

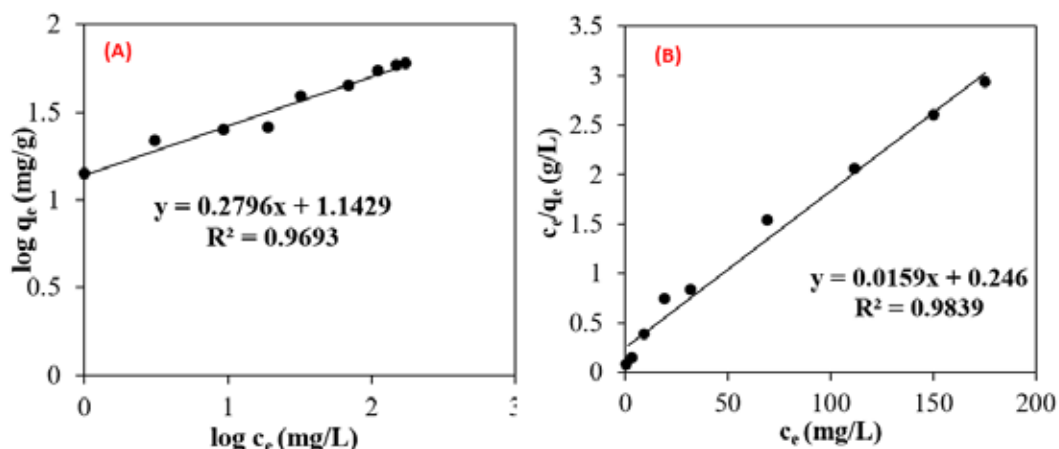**Figure 7.** *A) Freundlich isotherm and B) Langmuir isotherm for adsorption of Cr(VI) onto CBP.*

Figure 8. *A) Freundlich isotherm and B) Langmuir isotherm for adsorption (f Cr(VI) onto CBS.*

***Table 1.** Langmuir and Freundlich parameters for adsorption of Cr(VI)*

Adsorbents	Expt. Maxima q_m (mg/g)	Langmuir Isotherm			Freundlich Isotherm		
		q_m (mg/g)	K_L (L/mg)	R^2	K_F (mg/g)	$1/n$	R^2
CBP	65.70	68.96	0.085	0.99	15.85	0.283	0.97
CBS	59.76	62.89	0.064	0.98	13.89	0.279	0.96

A linear plot between log q_e and log c_e gives an intercept and a slope, which were used to estimate the value of K and $1/n$ respectively. The value of n ranges between 0 and 1 which is the measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero [33].

The isotherm parameters for the biosorption of Cr(VI) with their respective correlation coefficients are tabulated in the Table 1.

Hence, the adsorption model is best fitted by the Langmuir isotherm model. This indicates that the surface has homogeneous binding sites, equivalent sorption energy, and no interaction between adsorbed species. It further explains that in this model, once the site is filled no further adsorption takes place at that site and the surface eventually reaches a saturation point where the maximum adsorption of the surface is achieved [34].

Adsorption Kinetics

Adsorption kinetics provides valuable information about the reaction pathways and mechanism of the reaction. Kinetic models are generally studied to understand the sorbent-sorbate interactions and to describe the adsorption rate concerning the equilibration time [35].

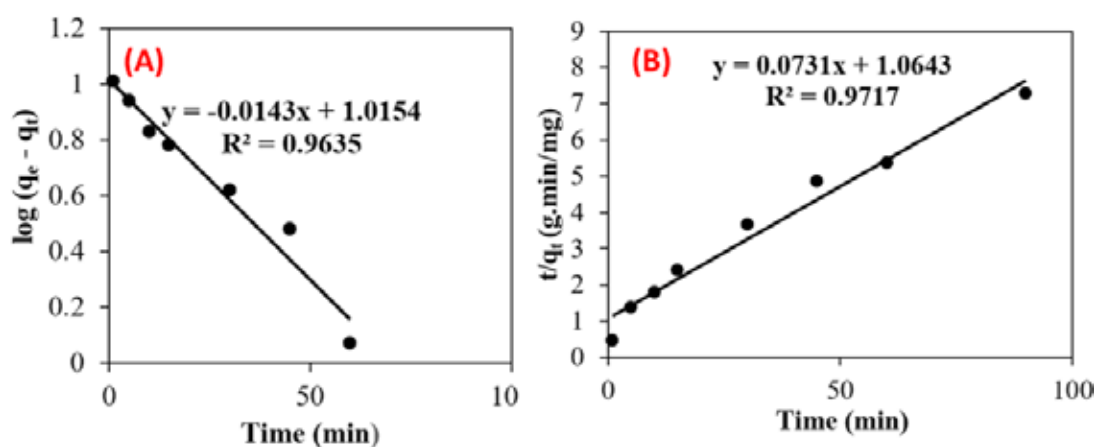The pseudo-first-order kinetic model is represented in the equation as:

$$\log(q_e - q_t) = \log q_e - \frac{K_1 t}{2.303} \quad (5)$$

Where q_e and q_t are the amounts of metal ions adsorbed at equilibrium (mg/g) and at a time 't' (min) respectively. K_1 is the rate constant of pseudo-first-order reaction (min^{-1}). The plot of log ($q_e - q_t$) versus time gives a straight line with slope ($-K_1/2.303$) and intercept (log q_e) from which the values of K_1 and q_e can be calculated respectively [36].

The pseudo-second-order adsorption kinetics is expressed as:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \quad (6)$$

Where q_e and q_t are the adsorption capacity at equilibrium (mg/g) and at a time 't' (min) respectively.

***Figure 9.** A) Pseudo-first-order and B) Pseudo-second-order kinetic models for adsorption of Cr(VI) onto CBP.*
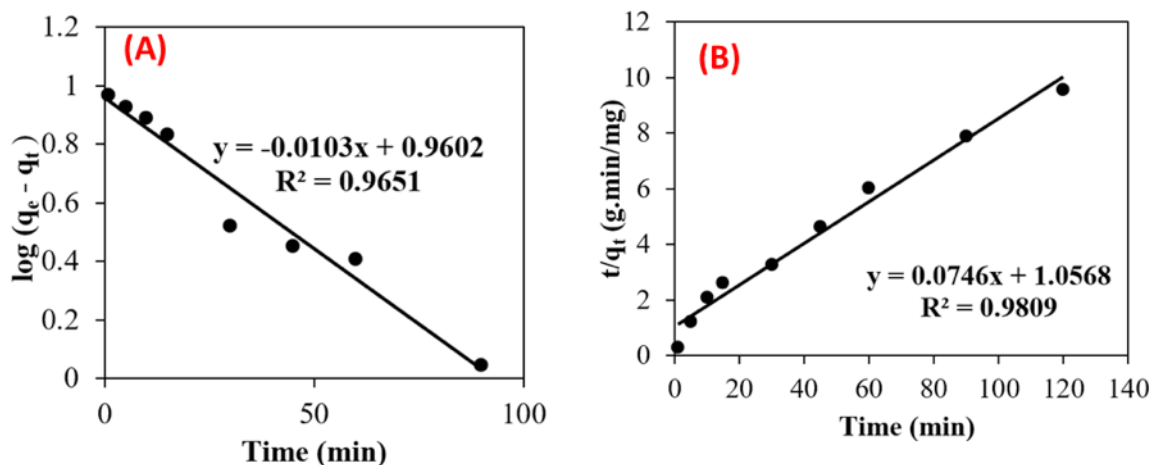
Figure 10. *A) Pseudo-first-order and B) Pseudo-second-order kinetic models for adsorption of Cr(VI) onto CBS*

K_2 is the rate constant of pseudo-second-order reaction (g/mg.min). The values of q_e and K_2 can be determined experimentally with the help of slope and intercept obtained from the linear plot of t/q_t versus time (t) [36].

The kinetic parameters for the biosorption of Cr(VI) with their respective correlation coefficients are tabulated in Table 2.

From the data represented in Table 2, it can be concluded that the pseudo-second-order kinetic model is the best fit kinetic model suitable for modeling the biosorption of Cr(VI). This model is more likely to predict the kinetic behavior of biosorption with chemical sorption being the rate-controlling step [37].

Conclusion

The present study has shown that low-cost adsorbents of banana peel and banana pseudostem can be used as one of the best adsorbents for the removal of Cr(VI) by the adsorption process. From the characterization techniques, the adsorbents were found to have carboxyl and hydroxyls as major functional groups involved in adsorption (FTIR), porous structure (SEM) and amorphous in nature (XRD), therefore evaluated to

be better for the adsorption of chromium. It was found that the adsorbents show the maximum adsorption capacity at pH 2.The value of maximum adsorption capacity q_m was observed to be 65.70 mg/g and 59.75 mg/g for CBP and CBS respectively. The optimum contact time for the adsorbents was reported to be 90 min and 120 min for CBP and CBS respectively. The isotherm studies showed that the experimental data were best fitted for the Langmuir isotherm model. Similarly, in the case of kinetic studies, the experiments followed pseudo-second-order kinetics. Therefore, based upon the present research work, it can be concluded that the modified form of agro-waste (banana peel and banana pseudostem) can be better used as an effective adsorbent for the adsorptive removal of chromium(VI) from aqueous solution.

Table 2. *Kinetic parameters determined for the adsorption of Cr(VI)*

Adsorbents	Expt. q_e (mg/g)	Pseudo-first-order kinetic model			Pseudo-second-order kinetic model		
		K_1 (min^{-1})	q_e (mg/g)	R^2	K_2 (g/mg.min)	q_e (mg/g)	R^2
CBP	12.35	0.032	10.36	0.96	0.005	13.67	0.97
CBS	12.54	0.023	9.12	0.96	0.005	13.40	0.98

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