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Adsorptive Removal of Cr(VI) from Aqueous Solution by Sugarcane Biomass

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Abstract

The potential and effectiveness of activated carbon derived from carbonization of sugarcane bagasse (Saccarhum officianrum) for adsorptive removal of Cr(VI) was examined by adsorption technique. Activated carbon was prepared by subjecting the raw sugar cane bagasse to chemical modification using concentrated sulphuric acid in 500 g/L of ratio of adsorbent weight to volume of concentrated sulphuric acid (H_2SO_4). Boehm method was used to estimate the oxygen containing functional groups. The acidic functional groups, specific surface area and adsorptive capacity all greatly increased with chemical modification. The batch removal of Cr(VI) from aqueous solution was investigated. The influence of pH, initial concentration of metal ion and contact time were also investigated. The maximum adsorption capacity of Cr(VI) was found to be 131.68 mg/g at optimum pH of 1 at the laboratory temperature respectively. The equilibrium time for Cr(VI) were found to be 180 minutes respectively. Kinetics of adsorption was found to follow pseudo-second order model. Both Langmuir and Freundlich adsorption isotherm could be used to describe adsorption isotherm but the Langmuir isotherm was found to be in good agreement with experimental data.

Keywords: Sugarcane bagasse, adsorption technique, toxicity and chemical modification.

Introduction

The environmental pollution due to the development in technology is one of the most important problems of this century. Heavy metals like chromium (Cr), lead (Pb), mercury (Hg), arsenic (As), cadmium (Cd), copper (Cu), aluminium (Al), and nickel (Ni) in waste water are hazardous to the environment. These metals cannot be degraded or readily detoxified biologically and have tendency to accumulate in living materials. Besides, heavy metals discharge in the wastewater can be toxic to aquatic life and render natural waters unsuitable for human consumption[1]. The Cr(VI) is toxic to animals and plants. The tolerable limit of Cr(VI) for drinking water is 0.05 mg/L[2].

Chromium is priority metal pollutant introduced into water bodies from many industrial processes such as tanning, metal processing, paint manufacturing, steel fabrication and agricultural run-off. Chromium is also used in explosive ceramic and photography. Chromium occurs in the aquatic environment as both trivalent and hexavalent states. Hexavalent chromium, which is primarily present in the form of chromate (CrO_4^{--}) and dichromate ($Cr_2O_7^{--}$) ions possess significantly higher levels of toxicity than the other valency states[3]. The toxicity of Cr(VI) is well documented and it is considered a hazard to health of man and animals. The various compounds of chromium are found to be both corrosive to flesh and carcinogenic. The presence of Cr(VI) in the aquatic environment at high concentration is lethal to marine species[4].

Khan *et al*. studied the adsorption of cadmium on sugarcane bagasse from aqueous solution. The efficiency of adsorption was found to depend on the initial metal ion concentration, pH and contact time. Sugarcane bagasse an agricultural residue from sugar refining industries had been shown to have a great potential as a low cost adsorbent for Cd(II) removal and could be applied for waste water management strategies. Both Langmuir and Freundlich adsorption isotherms describe the sorption behaviour. The adsorption data followed pseudo-second order kinetics[5].Hamadi *et al*. studied the adsorption kinetics for the removal of Cr(VI) from aqueous solution by using tyre and saw dust. They found that the removal was favourable at low pH, with maximum removal at pH 2. They found both adsorbents were effective to remove Cr(VI) from solution. The sorption kinetics was found to follow pseudo-second order model[6].Basyal and Ozdin studied the biosorption of Cr(VI) and Al(III) on Chrysemonas Luteola TEM05. They found the greatest capacity for Al(III) 55.2 mg/g and for Cr(VI) 3.0 mg/g at pH 5 and pH 4 respectively. The biosorption equilibrium was described by Langmuir and Freundlich isotherm. Freundlich isotherm fitted well for both ions. The kinetic data followed pseudo-second order model[7].

Bhattarai studied the adsorptive behaviour of chromium on raw rice husk and modified rice husk. The adsorptive behaviour was pH dependent. He found the maximum adsorptive capacity of modified rice husk for Cr(VI) was 190 mg/g at pH 1 which was much higher in comparison to raw rice husk. The adsorption data was best described by Langmuir isotherm and pseudo-second order kinetics[8]. Gupta *et al.* reported an adsorption studies on the removal of hexavalent chromium from aqueous solution using a low cost fertilizer industry waste material.

The waste was chemically treated, activated, characterized, and used for the adsorption of chromium. The work involves batch experiments to investigate the effect of contact time, pH, temperature, concentration, and adsorbent dose on the extent of adsorption by carbon slurry. The maximum adsorption was found at 70 min, 2.0 pH, 4.0 g/L dose, and 303 K temperature. Maximum adsorption capacity (15.24 mg/g) of Cr(VI) on carbon slurry was observed at 100 mg/L initial Cr(VI) concentration.

Langmuir and Freundlich adsorption isotherm models were applied to analyze adsorption data, and both were found to be applicable to this adsorption system, in terms of relatively high regression values. Thermodynamic parameters showed that the adsorption of Cr(VI) onto carbon slurry was feasible, spontaneous, and exothermic under the studied conditions. Kinetics of adsorption was found to follow the pseudo-second-order rate equation[9]. Kamila and Santhi investigated the equilibrium and kinetic study of the dynamics of the adsorption of single and mixture of reactive dyes on the chemically cross linked ontophosphoric ricinus communis. The multi system of the reactive dyes system onto the phosphoric assisted epicarp of ricinus communis as a low cost adsorbent used for this study[10]. Sankaramakishan *et al.* prepared the novel xanthated chitosan with CS_2 in alkaline media and used to remove Cr(VI) from waste water. They found the maximum uptake of Cr(VI) to be 71 mg/g which was significantly more than plain chitosan 49 mg/g. The adsorption behaviour was pH dependent[11]. There have also been published papers by various research group[12-15].

The objective of the present work is to prepare low cost adsorbent from agricultural waste sugarcane bagasse and to investigate the adsorption capacity of the adsorbent for the sorption of Cr(VI) from aqueous solution.

Material an d Methods

Chemicals: All chemicals $K_2Cr_2O_7$, H_2SO_4, HNO_3, NaOH and 1, 5 Diphenylcarbazideemployed were of reagent grade. Laboratory distilled water was used for the preparation of all solutions.

Preparation of adsorbent form raw sugarcane bagasse (RSCB): Raw sugarcane bagasse (RSCB) in the present research was collected from a fruit-seller located at Panga, Kirtipur. This raw bagasse was cleaned with distilled water and dried in sunlight for 3 days and grinded in mechanical grinder at Central Department of Chemistry, Kirtipur. The powdered waste was again dried in sunlight for 3 days and crushed very finely in the grain crusher. The finely powdered raw adsorbent was packed in an air tight bottle.

When raw sugarcane bagasse (RSCB) is treated with conc. H_2SO_4 at laboratory temperature, it produces an adsorbent having high surface area and high degree of micro porosity. For the process of chemical modification, the RSCB was mixed in 500g/L ratios with conc. H_2SO_4 and allowed to soak for 24 hours at room temperature to ensure complete reaction. The excess of acid was removed by washing it with distilled water until pH of the filtrate was neutral and then it was dried at 100^0C for 8 hours in an oven and sieved to the particle of size 225 µm. The adsorbent is designated as MSCB.

Preparation of stock potassium dichromate solution: A stock solution of Cr(VI) (1000 µg/ml) was prepared by dissolving 2.828 g of potassium dichromate crystal in a 1000 ml volumetric flask in 0.1N nitric acid solution. From this stock potassium dichromate solution, working solution of lower concentration was prepared by dilution method in 0.1 N nitric acid solutions. 0, 0.5, 1, 1.5, 2, 2.5, 3, 3.5 and 4 ml of working Cr (VI) solution of 5 mg/L were taken in a different 25 ml volumetric flask and was acidified with 1 ml of 5 N H_2SO_4 in each volumetric flask and shaken well. Then 1 ml of 0.25% DPCI solution was added to each volumetric flask and shaken well. The volume was then made up to the mark by adding distilled water.

After 15 minutes the absorption spectra of red-violet colored Cr(VI)-diphenylcarbazide complex was recorded in Perkin-Elmer spectrophotometer against reagent blank. At λ_{max} 540 nm absorbance of all the solutions were measured against reagent blank using the same spectrophotometer.

The effect of contact time was studied at room temperature at optimum pH. The equilibrium time of the adsorbent for Cr(VI) were investigated at their respective pH values by taking 25 ml of 20 mg/L concentration of Cr(VI) solution in 125 ml reagent bottles with 25 mg adsorbent dose. The mixtures were shaken in a mechanical shaker for 10, 20, 30, 40, 50, 60, 80, 100, 120 mins., 3 hr, 4 hr, 6 hr, 10 hr, 12 hr and 24 hr respectively. The metal ion concentration before and after adsorption were determined by spectrophotometer.

The percentage removal or removal efficiency is given by equation (1)

$$\text{Removal\%} = \frac{c_i - c_e}{c_i} \times 100\% \qquad (1)$$

where, c_i = Initial concentration of chromium, c_e = Final concentration of chromium

The amount of adsorbate adsorbed in mg/g at equilibrium is determined by the equation (2)

$$q = \frac{(c_i - c_e)}{W} \times V \text{ mg/g} \qquad (2)$$

Where:V = Volume of adsorbate solution in liter, W = Weight of adsorbent in g, c_i = Initial concentration of chromium mg/l, c_e = Final concentration of chromium in mg/l.

Adsorption isotherms: The adsorption isotherms for the adsorption of Cr (VI) on MSCB and RSCB are shown in figure 1. The adsorption of Cr(VI) on respective adsorbents increases with increase in time and attained an equilibrium value after some time. Experimentally it was found that concentration ranging from 10 mg/L to 400 mg/L the amount of Cr(VI) adsorbed increases from 7.92 to 131.68 mg/g in case of MSCB and 3.09 to 27.01 mg/g in case of RSCB for 25 mg/L solution at optimum pH 1.0 at lab temperature. The obtained results revealed that the adsorption capabilities of MSCB were found to be increased greatly than RSCB, due to chemical modification.

The Langmuir equation applied for adsorption equilibrium is as follows[16]

$$\frac{c_e}{q_e} = \frac{1}{q_m b} + \frac{c_e}{q_m} \qquad (3)$$

where c_e the equilibrium concentration (mg/L), q_e the amount of adsorbed under equilibrium (mg/L), q_m the theoretical maximum adsorption capacity corresponding to complete monolayer coverage (mg/g) and b is the adsorption constant (L/mg) related to the enthalpy of adsorption, q_m and b are determined from the slope and intercept of the plot of c_e/q_e against c_e.

The Freundlich isotherm assumes that the uptakes of metal ions occur on a heterogeneous surface by multilayer adsorption and that the amount of adsorbate adsorbed increases infinitely with an increase in concentration. It can be expressed in the linear form as follows[17]

$$\log q_e = \log K + 1/n \log c_e \qquad (4)$$

where, q_e is the amount of metal ion adsorbed per gram of adsorbent at equilibrium (mg/g), c_e is equilibrium concentration of the adsorbent (mg/L), K and n are constants which are considered to be the relative indicators of adsorption capacity and adsorption intensity, 1/n is characteristics constant for adsorption system (mg/L) i.e. it shows intensity of adsorption. The value of 1/n lies between 0 and 1 indicates the favorable adsorption of metal ions. The plot of log q_e versus log c_e should give a straight line with slope equal to 1/n and the intercept on the y-axis equal to log K.

Boehm Titration: In order to quantify surface functional group of sugarcane bagasse modified charcoal, Boehm titration procedure was employed. For this 250 mg of adsorbent was agitated with 50 ml of 0.02 N bases namely, Na_2CO_3, $NaHCO_3$, NaOH and 0.02 N HCl as acidic reagent in mechanical shaker for 48 hours. The mixture was then filtered and 5 ml of this filtrate was titrated with 0.02N HCl or NaOH using phenolphthalein or methyl orange indicator. On the basis of acid or base consumed by the definite weight of charcoal, the amount of oxygen containing functional groups per gram charcoal was calculated following the Boehm's procedure. The result obtained from the Boehm's titration regarding the amount of acidic/basic functional group is presented in table 1.

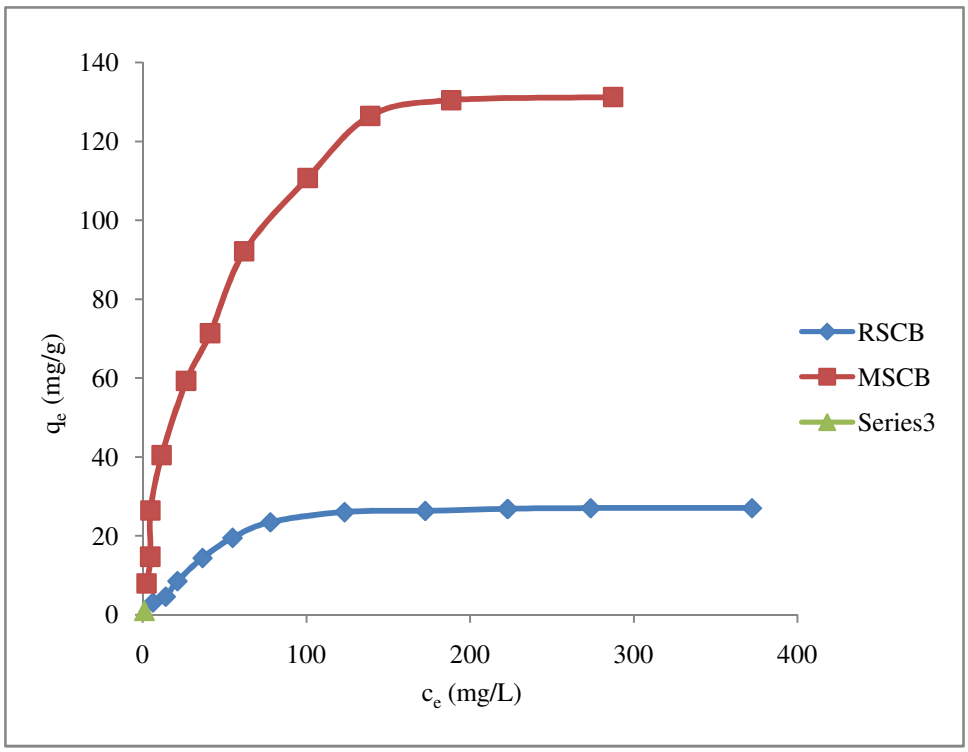

Figure-1
Adsorption isotherm for adsorption of Cr(VI) onto RSCB and MSCB

From the above table it evident that the oxygen containing (acidic) functional groups increases significantly on treating the raw charcoal with sulphuric acid at moderate temperature. It has been found that the acidic and basic surface sites co-exist simultaneously but the concentration of basic sites decrease with increase in acidic character of the surface[18].

Surface Area Determination: The surface area of sugarcane bagasse was determined by chemical adsorption technique. For the determination of surface area, 0.5 g of raw and modified sugarcane bagasse was agitated with 2.5 ml of different concentration of acetic acid for 120 mins. The final concentration of acetic acid was determined by titrating with 0.1 N standardized NaOH solutions.

The calculated values for RSCB and MSCB from the aforementioned technique appeared to be 49 m^2/g and 357 m^2/g, respectively, suggesting that the material after acid treatment have shown larger surface sites. This much surface area could have been developed after acid treatment onto the surface functional groups as determined by the Boehm titration shown in table 1.

Calibration Curve for Cr(VI) Complex with DPCI: Extensive literature reviews indicate that in the absence of acid there is no formation of colored complex between Cr(VI) and DPCI. 1 ml of 5 N H_2SO_4 solution was added in 25 ml final solution as the acid has positive impact in the formation of intense red-violet color of Cr(VI)-diphenylcarbazide. The nature of the absorption spectra and λ_{max} obtained at 540 nm match with the reported values[8].The Adherence to Beer-Lambert's was tested by reacting aliquots of standard solution containing 0 to 1.4 ppm of Cr(VI) in final volume of 25 ml following the procedure of determination of λ_{max}. The absorbance of each solution prepared as shown in table 2, which was monitored it λ_{max} at 540 nm. A calibration curve for the Cr (VI))-diphenylcarbazide was drawn as shown in figure 2. The plot was found to be linear. From the plot it is clear that the proposed method obeys Beer-Lambert's law from 0 to 1.4 ppm of Cr(VI) solution.

Table-1
Results of Boehm's titration

S.N.	Adsorbents	Functional groups milliequivalent per gram (meq/g)			
		Carboxyl	Lactones	Phenols	Basic
I	MSCB	1.5	0.4	0.6	0.3
II	RSCB	0.5	0.1	0.3	0.8

Table-2
Determination of calibration curve for Cr(VI)

S.N.	i	ii	iii	iv	v	vi	vii	viii
Concentration (ppm)	0	0.2	0.4	0.6	0.8	1.0	1.2	1.4
Absorbance	0	0.089	0.195	0.303	0.446	0.551	0.649	0.762

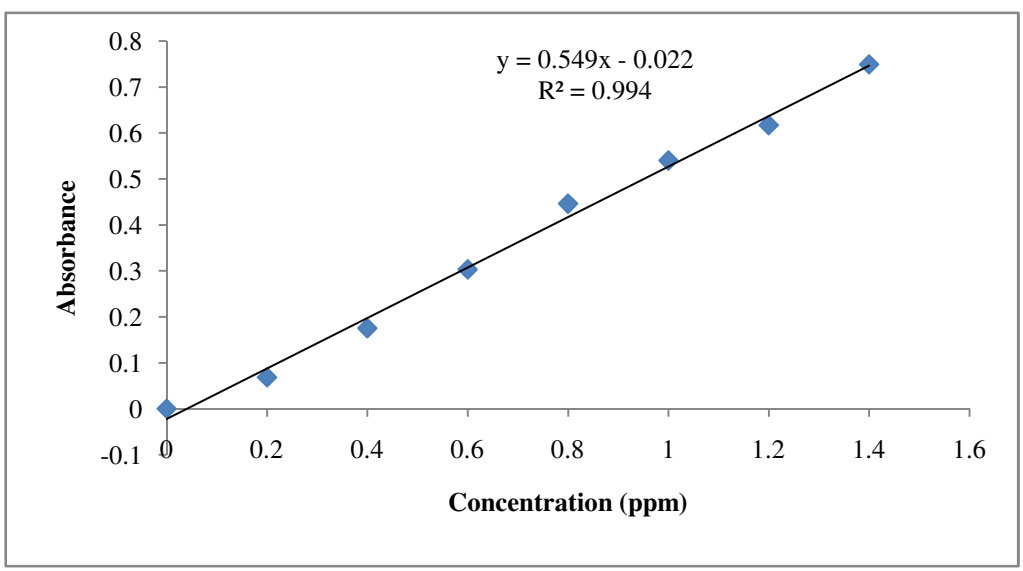

Figure-2
Calibration curve for determination of Cr(VI)

Results and Discussion

Batch pH Studies: Effect of pH on Cr(VI) adsorption: Figure 3 shows the effect of pH on the adsorption of Cr(VI) on RSCB and MSCB at initial concentration of 25 mg/L at lab temperature. The percentage removal of Cr (VI) by MSCB decreased from nearly 95% to 11% when the pH of the solution was increased from 1 to 6. Similarly in case of RSCB the removal percentage decreases from about 35% to 4% when pH is increased from 1 to 6. This shows that adsorption was maximum at pH 1 and adsorption was insignificant at pH 6. Thus the optimal pH for the adsorption of Cr (VI) on these adsorbent was found to be 1[8].

It has been reported that biomaterials like sugarcane bagasse removes Cr(VI) from aqueous solution through anionic adsorption and partial reduction in to Cr(III)[19, 20].

Figure 4 represent the Langmuir isotherm for the adsorption of Cr(VI) respectively on RSCB and MSCB. Adsorption of Cr(VI) give the linear relationship with Langmuir isotherm. Langmuir parameters are determined from the slopes and intercepts of their respective plot. Similarly Freundlich parameters are also determined and these values are given in table 3.

From table 3 it is clear that the value of 1/n is less than unity in respective charcoals which indicates adsorption isotherm is favourable for removal of metal ions. From the table it is also clear that the correlation coefficient (R^2) in case of Langmuir adsorption isotherm is greater than Freundlich adsorption isotherm which indicates that adsorption process fits more closely to Langmuir adsorption isotherm model.

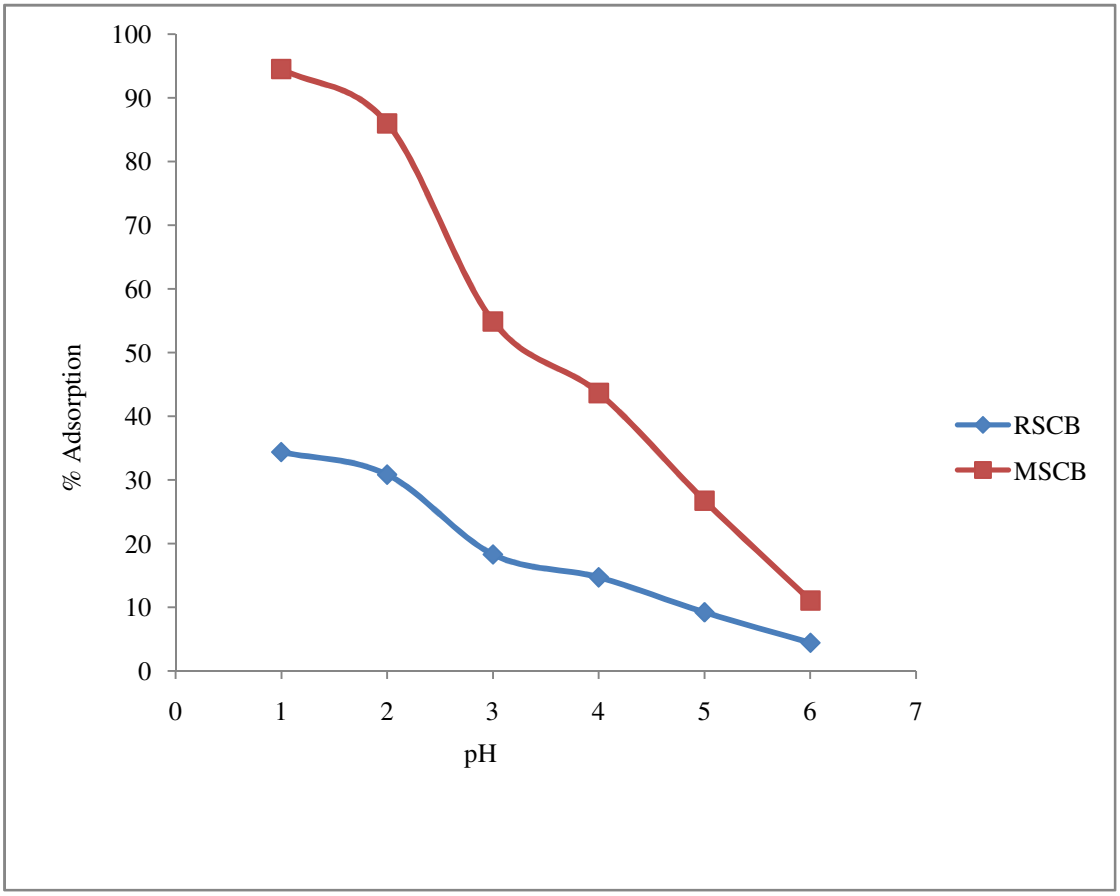

Figure-3
Effect of pH on removal of Cr(VI) by RSCB and MSCB

Table-3
Langmuir and Freundlich parameters for the adsorption of Cr(VI) on MSCB and RSCB

S.N.	Adsorbents	q_m(exp) (mg/g)	Langmuir Isotherm			Freundlich Isotherm		
			q_m(mg/g)	b(L/mg)	R^2	K(mg/g)	1/n	R^2
i	MSCB	131.68	140	0.036	0.977	8.09	0.541	0.91
ii	RSCB	27.01	33.33	0.018	0.974	1.48	0.552	0.903

Batch Kinetics Study: Adsorption kinetics: Figure 5 represent the pseudo-second order kinetic models for the adsorption of Cr(VI) on respective adsorbents. The sorption of Cr(VI) on the respective adsorbents were observed from 10 min to 24 hours. Initially the adsorption capacity was found to be optimal due to the presence of large number of active sites. After the achievements of active saturation condition in adsorption of Cr(VI) the sorption of the metals become constant. For Cr(VI) the saturation condition obtained after 120 mins and 180 mins.

The values of correlation coefficients and graphical plots clearly showed that the pseudo-second order kinetic model best describes the kinetic data with higher R^2 values than the other models. The amount of adsorption q_e (mg/g) of Cr(VI) on the respective adsorbents obtained from the experiments were close to the qe (mg/g) value obtained from the pseudo-second order plot. Thus it is concluded that the adsorption of Cr(VI) followed the pseudo-second order kinetic model. The best fitting of kinetic data to pseudo-second order implies that both Cr(VI) adsorption occur in a monolayer fashion and adsorption pattern occurs through chemical adsorption or ion exchange. The value of correlation coefficient and rate constants for Cr(VI) are shown in table 4.

Table-4
Pseudo-second order rate constant for the adsorption of Cr(VI) onto MSCB and RSCB

S.N.	Adsorbents	Pseudo-second order		
		K_2(g/mg.min)	h(mg/g.min)	R^2
1	MSCB	3.76×10^{-4}	0.275	0.95
2	RSCB	9.06×10^{-4}	0.11	0.974

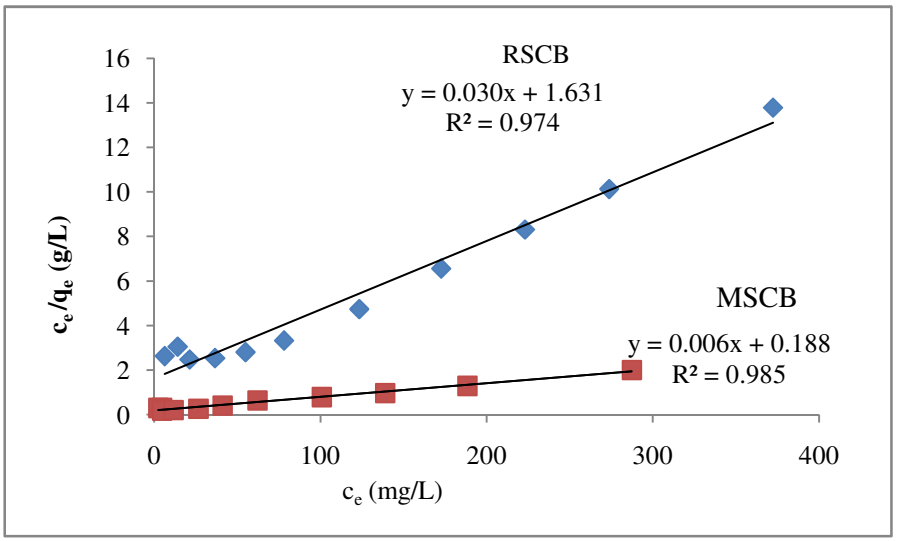

Figure-4
Langmuir isotherm plot for adsorption of Cr(VI) onto RSCB and MSCB

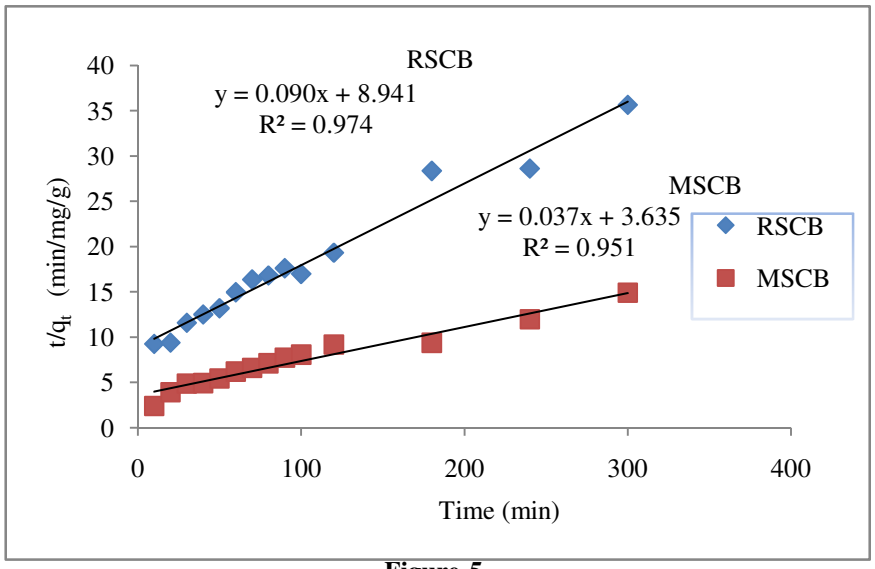

Figure-5
Pseudo-second order kinetic plot for adsorption of Cr(VI) onto RSCB and MSCB

Scanning Electron Microscope (SEM) Image of the Charcoal: The SEM images of raw and modified charcoal are shown in figures 6 and 7 respectively. The morphological studies clearly revealed that the adsorbent is amorphous and highly porous in nature. It was found that there were holes and caves like openings on the surface of adsorbent which would have more surface area available for adsorption than the raw charcoal.

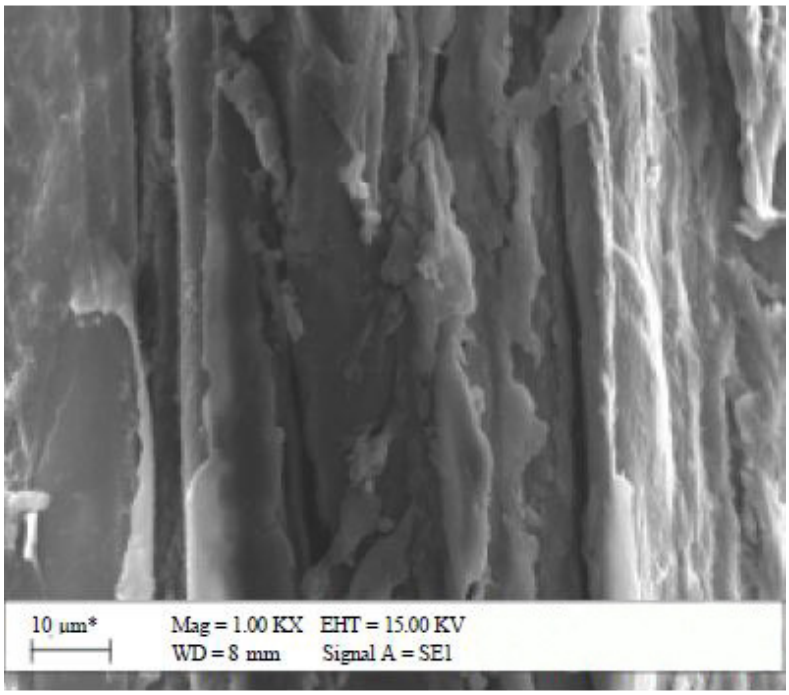

Figure- 6
SEM image of RSCB

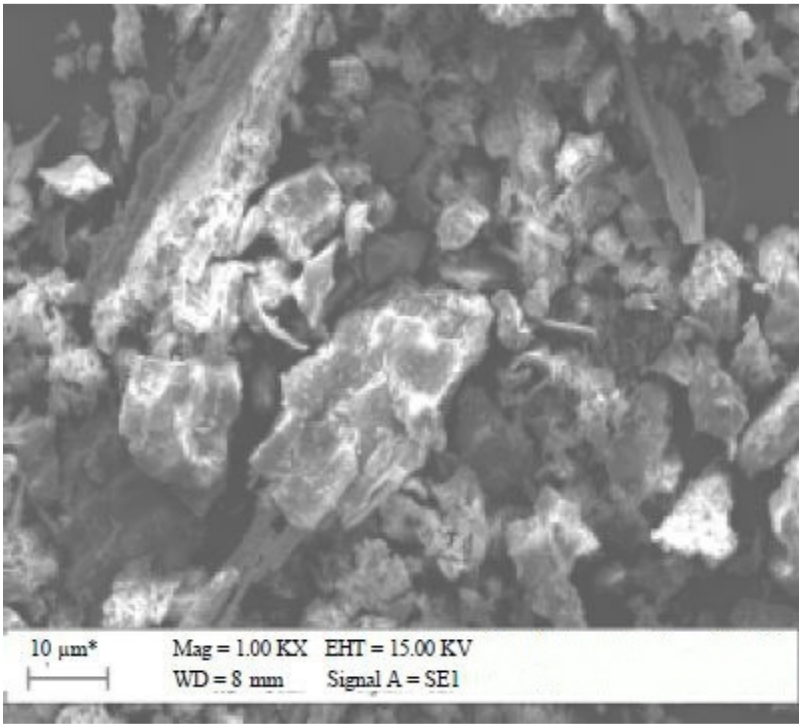

Figure-7
SEM image of MSCB

Spectra Analysis: Infrared Spectroscopy (IR) Analysis: The IR spectra of raw and modified sugarcane bagasse are shown in figure 8. The IR spectra provide fingerprints of different functional groups present in the adsorbent surface. The mechanism for the adsorption for heavy metals by sugarcane bagasse is linked to the role played by the essential stretching functional groups like hydroxyl (-OH), carboxylic (-COOH), carbonyl (C=O) and other aromatic and phenolic groups[21].The spectra show that, bands at 1370 cm^{-1} and 1440 cm^{-1} are associated to C-O stretching O-H bending modes of phenols and carboxylic acids. Bands nearly at 3000cm^{-1} is associated –OH group of phenol, alcohol and carboxylic groups. The IR spectra of raw and modified sugarcane bagasse adsorbent were analyzed using Perkin-Elmer infrared spectrophotometer in nujol medium. The IR spectra of the adsorbent were taken from NAST, Khumaltar, Kathmandu, Nepal.

Conclusion

Sugarcane bagasse, which is abundantly accessible low-cost agricultural waste in Nepal, can be easily converted to carbonaceous material by chemical treatment with concentrated H_2SO_4. This treated sugarcane bagasse can act just like commercial activated carbon for the metal removal by adsorption phenomena. The surface area of the sugarcane bagasse charcoal after chemical treatment had increased greatly and was found to be 357 m^2/g.

The chemically modified sugarcane bagasse charcoal has been found to be an efficient adsorbent for the removal of Cr(VI) from the aqueous solution than using the untreated raw sugarcane bagasse. The adsorption behaviour was dependent on the pH of the system, initial concentration of the adsorbate and the contact time as well.

From the study, it was observed that the maximum adsorption of Cr(VI) on to these adsorbents took place at optimal pH 1.0. The maximum adsorption capacity of Cr(VI) on modified sugarcane bagasse charcoal and raw sugarcane bagasse charcoal was found to be 131.68 mg/g and 27.01 mg/g respectively at their optimal pH 1.0. Both the Langmuir and Freundlich adsorption isotherms were tested to describe the adsorption process. It is concluded that Langmuir adsorption isotherm model is found to be more applicable than Freundlich adsorption isotherm for the better description on the adsorption behaviour of Cr(VI) metal ions.

The equilibrium time for Cr(VI) is found to be 180 min. The experimental data were fitted with pseudo first-order, pseudo second-order and the second order model. It was found that pseudo second order model provides a best descriptive to experimental data.

Characterization of adsorbent was performed by determining surface area of the charcoal, SEM image, IR spectra analysis and performing Boehm titration. All of these characterizing parameters support well for the efficacious adsorption capacity for Cr(VI) metal ions from aqueous system on modified sugarcane bagasse than raw sugarcane bagasse. Thus sugarcane biomass (*Saccarhum officianrum*) can be very effectively used to remove heavy metals from aqueous system and the effectiveness of this biomass can be enhanced by chemical modification.

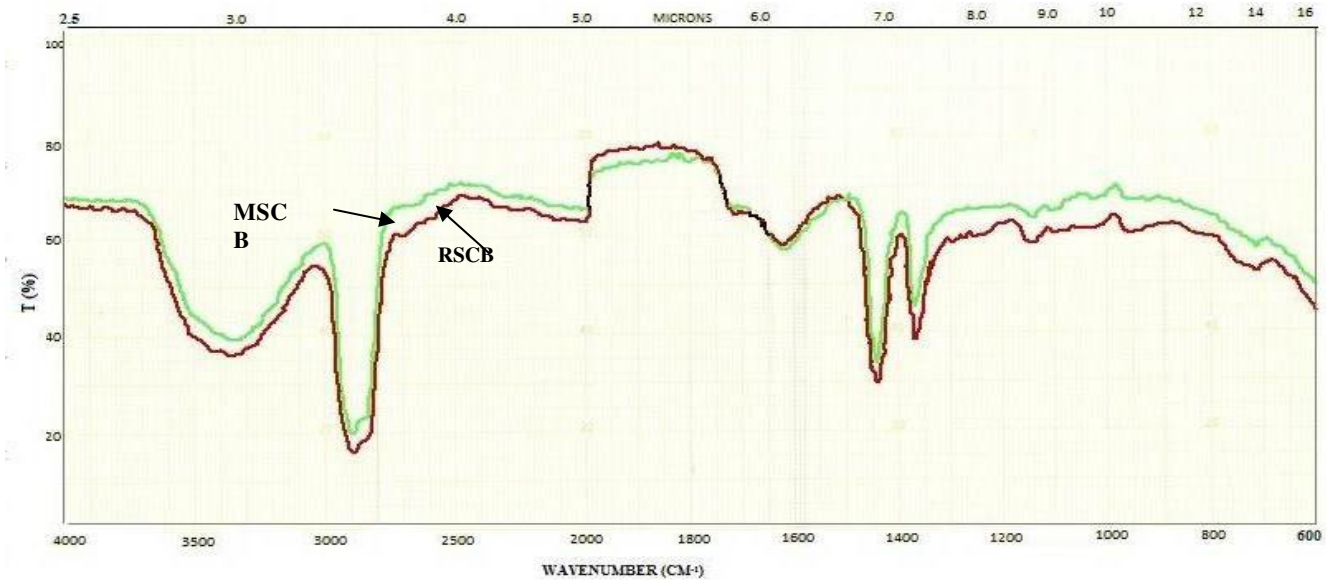

Figure-8
IR spectra of RSCB and MSCB

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