

Chromium(VI) Removal Using Biosorbents Derived from *Moringa Oleifera*

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Abstract

The increasing influx of heavy metals into water bodies from industrial and domestic activities is of the global concern because of their well-documented negative effects on human and ecosystem health. *Moringa oleifera* is low cost and environmental friendly material to remove the Cr(VI) from aqueous solution. This study is carried out with the objectives to optimize the feasibility condition of contact time, biosorbents dosage and pH range in removing Cr(VI) by using *Moringa oleifera* seed powder. The removal characteristics of Cr(VI) by *Moringa oleifera* was analyzed using UV-Visible spectrophotometer. The optimum percentage removal of Chromium achieved at 120 min at pH 5. The initial metal ion concentration and effect of temperature, Freundlich and Langmuir adsorption isotherm model and kinetics were studied.

Keywords: Environmental; Biosorbents; *Moringa oleifera*; Adsorbent dosage

Introduction

Many coagulants are widely used in conventional water treatment processes based on their chemical characteristics. These coagulants are classified into inorganic, synthetic organic polymers and natural coagulants. Coagulation is the most essential process in the treatment of both turbid surface and industrial wastewaters. Coagulation-flocculation is one of the most important physicochemical treatment steps in wastewater treatment to reduce the turbidity of wastewater [1,2]. Due to the lack of proper water treatment systems in these rural or under developed communities the best immediate option is to use simple and relatively cost effective point of use technologies such as coagulation [3-5]. Chromium(VI) is highly toxic and soluble in water and it is a strong oxidizing agent that causes severe damage to cell membranes. The most commonly used primary chemical coagulants, namely alum (AlCl_3), ferric chloride (FeCl_3) and polyaluminium chloride (PAC). The disadvantages associated with these coagulants such as ineffectiveness in low temperature water relatively high procurement cost, detrimental effects on human health, production of large sludge volumes and the fact that they significantly affect pH of treated water. There is strong evidence linking aluminium based coagulants to the development of Alzheimer's disease in human beings [6]. In addition to these problem chemicals used for water treatment in developing countries constitute a high percentage of annual running expenditure of water treatment companies. The cost of these chemicals in other industrial applications. They are so many methods for removal of Chromium from ground water. The methods are oxidation with chlorine and potassium permanganate treatment with limestone, liquid-liquid extraction, ion exchange, chemical precipitation, bioremediation, use of activated carbon and other filtering materials [7-13].

Moringa oleifera as a coagulant agent provided significant results which justify it as an alternative coagulant in the process of coagulation/flocculation of produced water (which is the waste that has the higher volume during the production and exploration of oil [14-16]. *Moringa oleifera* seed acid extract of natural polyelectrolyte very effective as a coagulant for removal of fluoride from water. *Moringa oleifera* is the best alternative coagulant that can replace aluminium sulphate [17,18]. In rural areas of the Sudan in particular, the powder seeds of *Moringa oleifera* are traditionally utilized for water purification because of their

strong coagulating properties for sedimentation of suspended mud and turbidity. Many parts of the tree are used as traditional medicines, the seeds contain up to 40% by weight of quality edible oil (greater than 80% unsaturated fatty acid content) and the seeds (and oil free press cake) yield proteins capable of acting as effective coagulants in water and wastewater treatment [19]. The active coagulants of the *Moringa oleifera* seeds have been determined to be cationic peptides of relatively low molecular weight (6-16 kda) with an isoelectric pH value of 10. In terms of water treatment technologies *Moringa oleifera* seed in diverse extracted and purified forms has proved to be effective at removing suspended material, generate reduced sludge volumes in comparison to alum soften hard water and act as effective adsorber of cadmium. The use of plant material as natural coagulants to clarify turbidity of wastewater is of common practice [20,21]. Since ancient times powdered roasted grains of *Zea mays* were used by solider in Peru as a means of settling impurities in the 16th and 17th century. In India ancient writings refer to the use of the seeds of the Nirmali tree *strychnos potatorum* as a clarifier. The sap of tuna cactus (*Opuntia ficus indica*) is widely used in chili as water purifying agent. Similarly dried beans (*Vicia faba*) and peach seeds (*Percica vulgaris*) are widely used for these purposes in Bolivia and Peru. *Moringa oleifera* seed act as a natural adsorbents and antimicrobial agent. Its seed contain 1% active polyelectrolyte's that neutralize the negative charged colloid in the dirty water. This protein can therefore be a nontoxic natural polypeptide. In the present study the effect of dosage, effect of initial chromium ion concentration, effect of pH, effect of contact time and effect of temperature, Freundlich and Langmuir adsorption isotherm model, Kinetics were studied. *Moringa oleifera* seed powder is environmental friendly method. *Moringa oleifera* seed powder used in water treatment it produce large amount of sludge it is used as biofertilizer or biocompost.

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Materials and Methods

Materials

All chemicals used were of analytical grade and supplied by Merck (purity >99%). The stock solution of Cr(VI) was prepared at a concentration of 1000 mg/L from potassium dichromate using double distilled water. The pH adjustments were made using 0.1 M NaOH and 0.1 M HCl.

Preparation of *Moringa oleifera* seed powder: Good quality dry seeds of *Moringa oleifera* pods that were collected from Tindivanam. The dried *Moringa oleifera* seeds coat and wings were removed manually followed by the grinding of the seeds into a fine powder using a domestic blender then sieving the grind powder through 250 μ m sieve [1].

Equilibrium experiments: Equilibrium studies at different metal ions concentrations were investigated using the batch adsorption process as described earlier. Batch adsorption studies were conducted in a set of 250 ml glass stopper Erlenmeyer flasks containing appropriate dose (0.5 g) of adsorbent and 50 ml of metal ion solution. This mixture solution was agitated at a speed of 500 rpm in a magnetic shaker until the equilibrium was attained. After equilibrium, supernatant was filtered and the equilibrium concentration of Cr(VI) was analyzed, respectively, using UV-Visible spectrophotometer. The percentage metal ion removal (R) was calculated using the following equations:

$$\% \text{ Removal of Cr(VI)} = \frac{C_i - C_e}{C_i} \times 100 \quad (1)$$

Where C_0 and C_e are the initial and equilibrium concentrations of the metal ion solution (mg/L), V is the volume of the solution (L), and M is the mass of the adsorbent used (g). The obtained data were fitted into adsorption isotherms, Lagergren's first-order, Pseudo-second-order Models.

Results and Discussion

Effect of adsorbent dosage

The relation between the percentage removals of chromium with adsorbent dosage is shown in Figure 1. From this curve it is clear that the percentage removal of chromium increased with the increase of adsorbent dosage the similar result were found that other researchers. The initial chromium concentrations were set at 5 ppm and pH of 5. The *Moringa oleifera* seed powder was varied between 0.1 g/l to 0.5 g/l. For coagulant dosage of 0.5 g/l achieved percentage reduction of chromium was more than 85%. This result can be explained as when the adsorption dose reached a certain rate the adsorption site was used up, hence with reduced tendency of the particles to adsorb any more ions to its surface. So removal rate of heavy metal ions no longer increased. The similar results were found by other researchers [22-24].

Effect of contact time

The optimum contact time was determined based on the percentage efficiencies. The removal of Cr(VI) from solution as a function of time is presented in Figure 2. It can be seen that the adsorbed amount of metal ions increased by increasing contact time and reached equilibrium at 120 min. Further experiments were carried out as 120 min for contact time. This result may be due to the use of vacant adsorption sites on the adsorbent surface. Large number of vacant adsorption sites were available for initial stage of sorption. After a lapse in time the remaining vacant surface sites were occupied due to repulsive forces between the solute molecules on the adsorbent surface and the bulk phase. The

similar results were obtained by other researcher [25]. At this point the amount of metal being adsorbed onto the biosorbents were in a state of dynamic equilibrium with the amount of adsorb from the biosorbents.

Effect of pH

The effect of pH on the removal of chromium is shown in the Figure 3. It is evident that for *Moringa oleifera* seed powder the percentage removal of chromium is almost 85% at pH 5. When the basic pH reached 10-13 where a high degree of the precipitation of metal ion can be expected in the insoluble forms. The percentage removal of chromium ions increased at acidic pH values are present H^+ and Cr^{3+} ions compete for the active sites of adsorption. The increasing in $pH > 8$ which has been estimated to occur metal hydroxide. Further experiments were carried out at pH 5. The similar results have been reported by other researchers. So pH 5 was considered as optimum condition and was used for further study [26].

Effect of initial metal ion concentration

The effect of Cr(VI) concentration from 5 to 10 mg/l on the adsorption process has been investigated using 0.5 g of *Moringa oleifera* seed powder. The percentage removal is achieved at 88% for 5 mg/l at contact time of 120 min. The percentage removal was found to decrease exponentially at high chromium concentrations lack of available active sites is required. Similar results have been reported in previous studies [26]. *Moringa oleifera* seed powder adsorption and interparticle bridging takes place to remove Cr(VI) from synthetic aqueous solutions (Figure 4).

Effect of temperature

The effect of temperature on Cr(VI) adsorption was studied. The experiment were conducted with Cr(VI) concentration of 5 ppm and a *Moringa oleifera* dosage of 0.250 g at pH 5 for 2 hrs. The temperature varies from 25°C to 100°C, indicating that the adsorption of Cr(VI) onto *Moringa oleifera* is an endothermic process. The adsorption capacity of Cr(VI) was increased by increasing the temperature (Figure 5).

Adsorption isotherms

The adsorption studies were conducted at fixed initial concentration of heavymetals by varying adsorbent dosage. The equilibrium data obtained were analysed in the light of Langmuir and Freundlich isotherms. Adsorption isotherm was assayed by studying the adsorption of Cr(VI) with wide concentration range in aqueous solutions onto *Moringa oleifera* seed adsorbent. The amount adsorbed of metal ion onto carbon adsorbent (mg/g) was represented against the

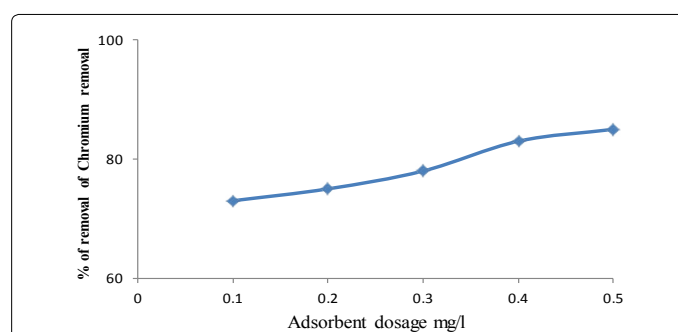


Figure 1: Effect of *Moringa oleifera* seed powder dosage. Experimental conditions: Initial Chromium solution concentration- 5 ppm, *Moringa oleifera* seed powder dosage- 0.1 to 0.5 g.

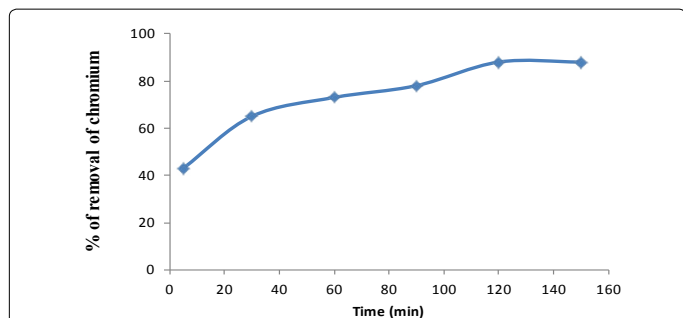


Figure 2: Effect of contact time. Experimental conditions: Initial Chromium solution concentration- 5 ppm, *Moringa oleifera* seed powder dosage- 0.5 g, Time- 30 to 120 min.

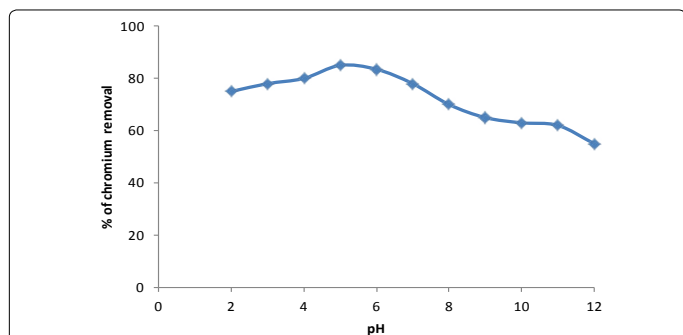


Figure 3: Effect of pH. Experimental conditions: Initial Chromium solution concentration- 5 ppm, *Moringa oleifera* seed powder dosage- 0.5 g, Time- 120 min, pH 2-12.

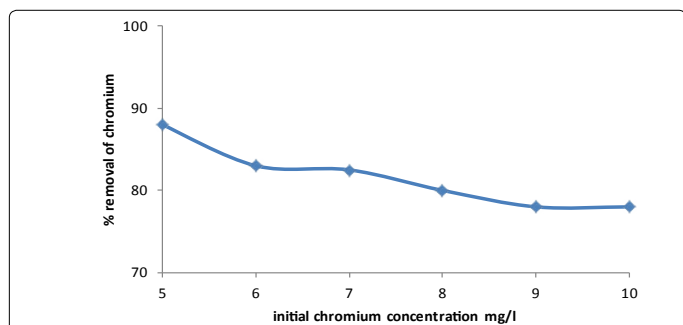


Figure 4: Effect initial chromium concentration. Experimental conditions: Initial Chromium solution concentration- 2 to 10 ppm, *Moringa oleifera* seed powder dosage- 0.5 g, Time- 120 min, pH 5.

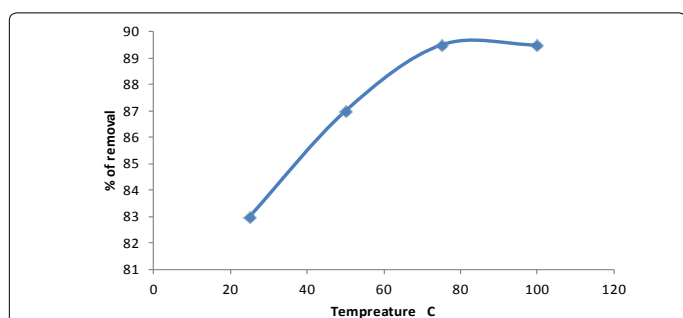


Figure 5: Effect of Temperature. Experimental conditions: Initial Chromium solution concentration- 5 ppm, *Moringa oleifera* seed powder dosage- 0.5 g, Time- 120 min, pH 5, Temperature 20 to 100°C.

equilibrium concentration of metal ion in aqueous solution. Langmuir isotherm model is based on the assumption that maximum adsorption corresponds to a saturated monolayer of adsorbate molecules on the adsorbent surface that the energy of adsorption is constant and that there is no transmigration of adsorbate in the plane of the surface. Adsorption isotherm was obtained by shaking the adsorbent of fixed doses and the adsorbate solution containing varied concentrations of metal ion for 2 h. The Langmuir isotherm model represents the equilibrium distribution of metal ions between the solid and liquid phases. The following equation can be used to describe adsorption isotherm according to Langmuir:

$$\frac{C_e}{Q_e} = \frac{1}{Q_m K_L} + \frac{C_e}{Q_m} \quad (2)$$

Where C_e is the equilibrium concentration (mg L^{-1}), Q_e is the amount adsorbed at equilibrium (mg g^{-1}), Q_m (mg g^{-1}) and K_L (L mg^{-1}) are Langmuir constants related to adsorption capacity and energy of adsorption respectively. Figure 6 shows the linear plot of C_e/Q_e vs. C_e for *Moringa oleifera* powder. The values of Q_m and K_L were determined from slope and intercept of the linear plot of C_e/Q_e vs. C_e . The experimental data and the correlation coefficients (R^2) values of *Moringa oleifera* seed powder was 0.947 indicates the applicability of the Langmuir isotherm model. The essential feature of Langmuir isotherm can be expressed by means of dimensionless constant referred to as the separation factor or equilibrium parameter, R_L which is defined by the following equation [24]:

$$R_L = \frac{1}{1 + K_L C_i} \quad (3)$$

Where C_i is the initial metal ion concentration (mg L^{-1}). The value of separation factor R_L , indicates the nature of the adsorption process as given below:

RL value	Nature of adsorption process
$RL > 1$	Unfavorable
$RL = 1$	Linear
$0 < RL < 1$	Favorable
$RL = 0$	Irreversible

The values of R_L values calculated for this study are given in Table 2. The adsorption process will be favorable if the R_L values lie between 0 and 1. The R_L values given in Table 2, very well lie in this range and hence the adsorption process was favorable [25].

The Freundlich model was chosen to estimate the adsorption intensity of the metal ions on the carbon adsorbent surface. The Freundlich equation is presented as:

$$Q_e = K_F C_e^{\frac{1}{n}} \quad (4)$$

This expression can be linearized to give the following equation:

$$\log Q_e = \log K_F + \frac{1}{n} \log C_e \quad (5)$$

Where K_f (mg/g) and n are Freundlich constants incorporating all factors affecting the adsorption process such as adsorption capacity and intensity of adsorption. These constants are determined from the intercept and slope of linear plot of $\log q_e$ versus $\log C_e$, (Figure 7). Although the correlation coefficients are greater than 95%, they do

Freundlich isotherm			
Adsorbent	1/n	K _F (mg g ⁻¹)	R ²
<i>Moringa oleifera</i>	0.439	9.015	0.963
Langmuir isotherm			
Adsorbent	Q _m (mg g ⁻¹)	K _L (L mg ⁻¹)	R ²
<i>Moringa oleifera</i>	11.36	0.88	0.9475
Tempkin isotherm			
Adsorbent	β	K _T (mg L ⁻¹)	R ²
<i>Moringa oleifera</i>	5.3598	4.829	0.9349

Table 1: Isotherm parameters for the adsorption of total iron at 30°C.

Cr(VI) concentration (mg L ⁻¹)	The R _L value
5	0.2272
6	0.1893
7	0.1623
8	0.1420
9	0.1262
10	0.1136

Table 2: The values of R_L values.

not correlate the data as well as the Langmuir isotherm, which has consistently higher correlation coefficients.

Tempkin isotherm model

Tempkin isotherm contains a factor that explicitly takes into account adsorbing species-adsorbate interactions. This isotherm assumes that the heat of adsorption of all molecules in the layer decreases linearly with coverage due to adsorbate-adsorbate interaction and adsorption is characterized by a uniform distribution of binding energies, up to some maximum binding energy. Tempkin isotherm has generally been used in the linearized and rearranged form as following: [26,27]

$$Q_e = \hat{a} \ln K_T + \hat{a} \ln C_e \quad (6)$$

Where, K_T is an equilibrium constant of binding corresponding to the maximum energy of binding (mg L⁻¹) and the β is related to the heat of adsorption. Figure 8 shows a plot of Q_e versus ln C_e, which enables the determination of the isotherm constants K_T and β. The values of K_T, β and correlation coefficient, R² for Tempkin isotherm model are given in Table 1 [28,29].

Adsorption kinetics

In order to investigate the mechanism of adsorption onto adsorbent, two kinetic models were studied: Lagergren's first-order, Pseudo-second-order.

Lagergren's first-order kinetic model: The pseudo-first-order kinetic model of Lagergren is more suitable for lower concentration of solute and its linear form is:

$$\log(Q_e - Q_t) = \log Q_e - \frac{k_1}{2.303} t \quad (7)$$

Approximately linear fits were observed for all pH and concentration indicating that sorption reaction can be approximate to first order reversible reaction model. Where, Q_t (mg g⁻¹) is the amount of adsorbate adsorbed at time t (min); Q_e (mg g⁻¹) is the adsorption capacity in the equilibrium; k₁ (min⁻¹) is the rate constant of pseudo-first-order model.

The values of k₁ and Q_e for *Moringa oleifera* adsorbents were determined from the plot of log(Q_e-Q_t) vs. time (Figure 9) [30,31].

Pseudo-second-order kinetic model: Adsorption kinetics was

explained by the pseudo-second-order model expressed as following linear equation:

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{t}{Q_e} \quad (8)$$

Where, k₂ is the second order rate constant (g mg⁻¹ min⁻¹). The values of k₂ for removal of chromium by *Moringa oleifera* adsorbents were calculated from the slopes of the respective linear plots of t/Q_t vs.t (Figure 10). The correlation coefficients, R² were 0.920 for *Moringa oleifera* respectively suggest a strong relationship between the parameters and also explain that the process follows pseudo second order kinetics. Pseudo second order model better fitted to pseudo second order model [32].

Scanning electron microscope

The main features of the SEM are an electron source which

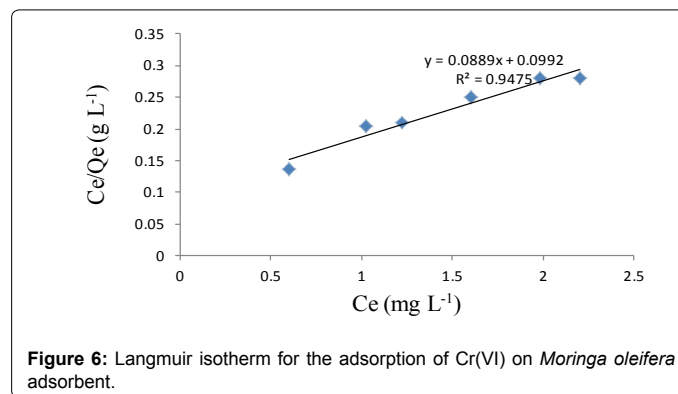


Figure 6: Langmuir isotherm for the adsorption of Cr(VI) on *Moringa oleifera* adsorbent.

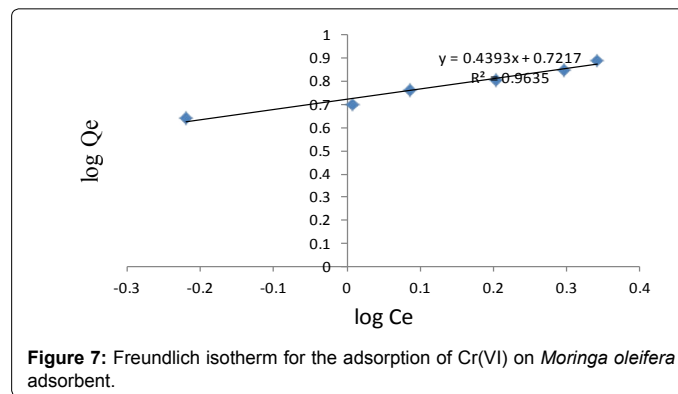


Figure 7: Freundlich isotherm for the adsorption of Cr(VI) on *Moringa oleifera* adsorbent.

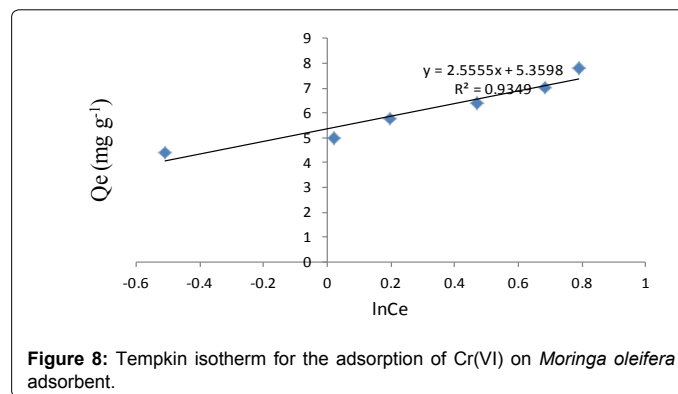


Figure 8: Tempkin isotherm for the adsorption of Cr(VI) on *Moringa oleifera* adsorbent.

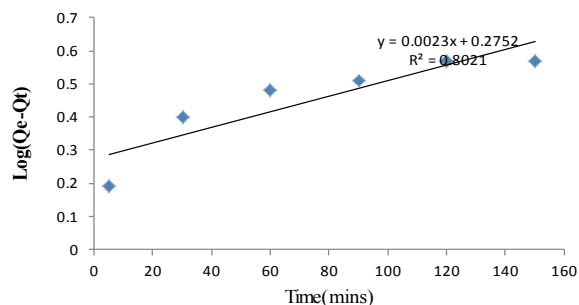


Figure 9: Lagergren first-order-kinetic model of Cr(VI) adsorption onto 1 g L⁻¹ adsorbent, initial Cr(VI) concentration 5 mg L⁻¹, pH 5.

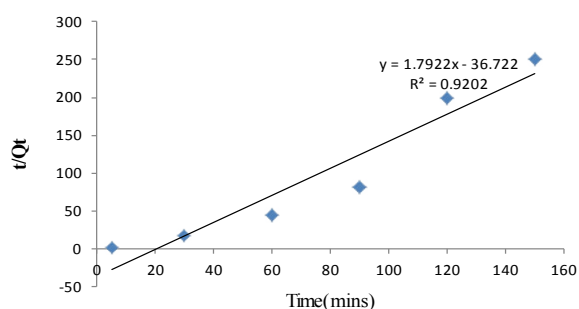


Figure 10: Pseudo-second-order kinetic model for the adsorption of Cr(VI) onto 1 g L⁻¹ of adsorbent with initial Cr(VI) concentration of 5 mg L⁻¹ at pH 5.

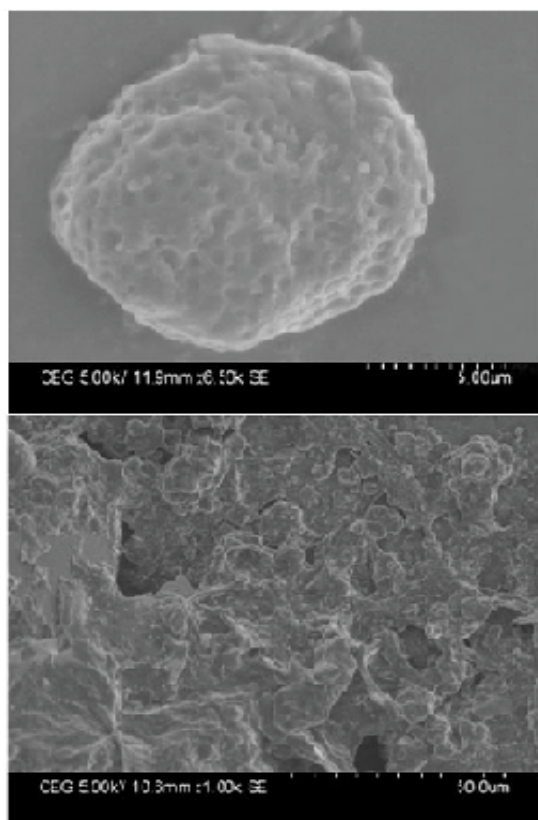


Figure 11: Scanning electron microscopy of (a) before and (b) after adsorption of chromium (VI).

provides the electrons that interact with the material to be examined an arrangement of metal apertures, magnetic lenses and scanning coils or deflectors plates that confines focuses and turns the beam of electrons into a thin and focused monochromatic beam which is accelerated towards the sample and which irradiates the specimen in a raster fashion. *Moringa oleifera* as such seed powder very fine particles size to the order of a millimeter or less and that there are pores within the particles of varying size. Using single *Moringa oleifera* as a coagulant the flocks formed with porous structure. After adsorption of *Moringa oleifera* seed powder chainlike and spherical structure confirms Cr(VI) onto the *Moringa oleifera* seed powder is shown in Figure 11.

Conclusions

In order to investigate *Moringa oleifera* is an effective adsorbent for the removal of Cr(VI) from aqueous solution. The adsorption is metal ion concentration is pH dependent. Batch experiments were conducted to study the effect of time, pH, temperature, initial concentration, adsorbent dosage and kinetic data showed good correlation with pseudo second order model. Maximum chromium removal 85% achieved at 120 min contact time, pH and 0.5 g of adsorbent dosage. Chemical treatment method used for the coagulation experiments it produces a secondary sludge. Natural coagulants are environmentally friendly material for removal of chromium from aqueous solution. The sludge left over after treatment it is used as biofertilizer or biocompost. The surface morphology structure of before and after adsorption of chromium(VI) were determined.

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