# Removal of chromium (VI) in synthetic wastewater by using immobilized Sugarcane bagasse (*Saccharum officinarum L.*) onto alginate Polvinyl glutaraldehyde

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***Abstract*: Alginate Polvinyl glutaraldehyde was used to immobilized biosorbent Sugarcane bagasse (*Saccharum officinarum L.*) . Immobilization enhances sorption chromium. The influence of several operating experimental parameters such as solution pH, biosorbent dose concentration, initial chromium (VI) concentration, contact time, temperature and agitation rate determined in the experiment were effective in determining the efficiency of chromium (VI) onto banana peels (L.). Langmuir adsorption isotherm, Freundlich adsorption isotherm, Dubinin-Kaganer-Radushkevich (DKR) adsorption isotherm and Temkin adsorption isotherm were tested in batch equilibrium studies. For kinetics studies, Pseudo-first-order model, Pseudo-second-order model, Elovich model and Weber and Morris intra-particle diffusion model were applied to the experimental data and followed by thermodynamic study. The results showed that immobilized waste biosorbent was a low-cost promising sorbent for sequester of chromium (VI) ions from wastewater.**

***Keywords*: Chromium (VI), Sugarcane bagasse (*Saccharum officinarum L.*), Alginate Polvinyl glutaraldehyde, adsorption isotherm, adsorption kinetics, thermodynamic study**

## Introduction

Today's one of the environmental challenge is the excessive use of heavy metals for industrial and domestic activities which it contaminates the ground and surface water therefore, it is important to eliminate these elements from water and wastewater before discharging into the environment (Zhang et al;2010,Balarak et al;2016, Naghizadehi et al; 2014). Cr(VI) containing waste water discharged from various industries including mining, tanning, cement, production of steel and other metal alloys, electroplating operations, photographic material and corrosive painting industries (Gupta et al;2010, YaoQ etal al;2010). The carcinogenic, mutagenic and toxic of Cr(VI) can negatively affect on the life cycle; thus, there is significant need to remove this element for the elimination of this element (Rao et al;2010,Balarak;2014, Shen Y S;2010). Heavy metals can be removed from industrial wastewater using physico-chemical treatment technologies such as precipitation, ion exchange, electrochemical processes and membrane processes (Abdul Hussain et qal;2005,Ye J et al;2010,Hang et al 2010). However, these technologies are expensive .Therefore, it is required to produce an easy, effective, economic and eco-friendly technique for wastewater treatment Veglio and Beolchini, 1997; Volesky, 2001). Biosorption is a cost- effective and eco-friendly technique for removal of heavy metals. Biosorption may be defined as removal of substances from solution by biological material (Gadd, 1992; Bhalerao, 2011).In the present research work, Sugarcane bagasse (*Saccharum officinarum L.*) is an agro-industrial based waste material. The present research describes the increased application and efficiency of Sugarcane bagasse (*Saccharum officinarum L.*) biomass when immobilized for the biosorption of chromium (VI) from wastewater. The study was extended with the objective for estimation and calculation of various parameters affecting the biosorption such as solution pH, biosorbent dose concentration, initial concentration of heavy metals, contact time, temperature and agitation rate. Adsorption isotherms model and kinetics models was employed to understand the probable biosorption mechanism. . Thermodynamic study was also carrying out to estimate the standard free change ($\Delta G^0$ ), standard enthalpy change ($\Delta H^0$ ) and standard entropy change ($\Delta S^0$ ).

## MATERIALS AND METHODS

Chemical and reagent: All the chemicals and reagents used were of analytical reagent (AR) grade. Double distilled water was used for all experimental work including the preparation of metal solutions. The desired pH of the metal ion solution was adjusted with the help of dilute hydrochloric acid and sodium hydroxide.

### *Preparation of chromium (VI) solution:*

The stock solution of 1000 ppm of chromium (VI) was prepared by dissolving 0.7072 g of potassium dichromate (K Cr O ) in 250 ml of double distilled water

### *Preparartion of biosorbent*

The Sugarcane bagasse (*Saccharum offcinarum* L.) was collected locally and washed with several times with distilled water to remove the surface adhered particles, dirt, other unwanted material and water soluble impurities and water was squeezed out. The washed biosorbent was then dried at $50^0$C overnight and grounded in a mechanical grinder to form a powder. The powder was sieved and a size fraction in the range of 100-200 µm will be used in all the experiments. This powder was soaked (20 g/l) in 0.1 M nitric acid for 1 hour. The mixture was filtered and the powder residue was washed with distilled water, several times to remove any acid contents. This filtered biomass was first dried at room temperature and then dried in an oven at 105ºC for 1-2 hrs. For further use, the dried biomass was stored in air tighten plastic bottle to protect it from moisture.

***Immobilization of the biosorbent:***

2g of Glutaraldehyde was take in a beaker with 50ml of water and melted it ,if required added a little water more till it melted completely. 2.0 g anhydrous calcium chloride in 100ml distilled water was made. In another beaker add Glutaraldehyde, 10ml of Polyvinyl alcohol, 0.5g of powdered sugarcane bagasseand finally added calcium alginate solution and mixed it well This mixture was extruded as droplet in 2% solution of calcium chloride through a glass nozzle (1.0 cm length and 2.5-3mm internal diameter) and the solution was stirred to avoid clumping of immobilized beads. Beads were formed of approximately 3-3.5mm diameter. The beads were allow curing for 1 hour at 4ºC in the same solution and then washed thoroughly with distilled water. After that it was stored in refrigerator for further use as biosorbent.

***Instrumentation:***

The pH of the solution was measured by digital pH meter (EQUIP-TRONICS, model no. Eq-610) using a combined glass electrode. The concentration of chromium (VI) in the solutions before and after equilibrium was determined by using Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) technique.

**Experimental procedure**:

The static (batch) method was employed at temperature (300C) to examine the biosorption of Chromium(VI) by biosorbent. The method was used to determine the adsorption capacity, stability of biosorbent and optimum biosorption conditions. The parameters were studied by combining biosorbent with chromium (VI) solution in 250 ml separate reagent bottles. The reagent bottles were placed on a shaker with a constant speed and left to equilibrate. The samples were collected at predefined time intervals, centrifuged, the content was separated from the adsorbent by filtration, using Whatmann filter paper and amount of Chromium(VI) in the supernatant/filtrate solutions was determined by ICPAES. The following equation was used to compute the percent removal (% Adsorption) of chromium (VI) by the adsorbent,

$$\%\,\mathrm{Ad} = \frac{(\mathrm{C_i} - \mathrm{C_e})}{\mathrm{C_i}} \times 100$$

where $C_i$ and $C_e$ are the initial concentrations and equilibrium concentrations of the Lead(II) in mg/L. The equilibrium adsorptive quantity (qe) was determined by the following equation,

$$q_e = \frac{(C_i - C_e)}{w} \times V$$

where $q_e$ (mg metal per g dry biosorbent) is the amount of chromium (VI) biosorbed, V (in liter) is the solution volume and w (in gram) is the amount of dry biosorbent used.

**Results and discussion**

**Effect of pH:**

The biosorption capacity of the biosorbent and speciation of metals in the solution is pH dependent. The optimization of pH was done by varying the pH in the range of 1-8 for bisorption of chromium (VI) and pH trend observed in this case is shown in Figure 1. It was found that at pH 3 the biosorption process was maximum with 92.75 % and after increasing pH, biosorption was decreases. As the pH of the solution increases, charges on the surface of biosorbent becomes negative, this leads to generation of repulsive forces between chromium (VI) and biosorbent and inhibits biosorption and resultantly percent chromium (VI) uptake may decrease

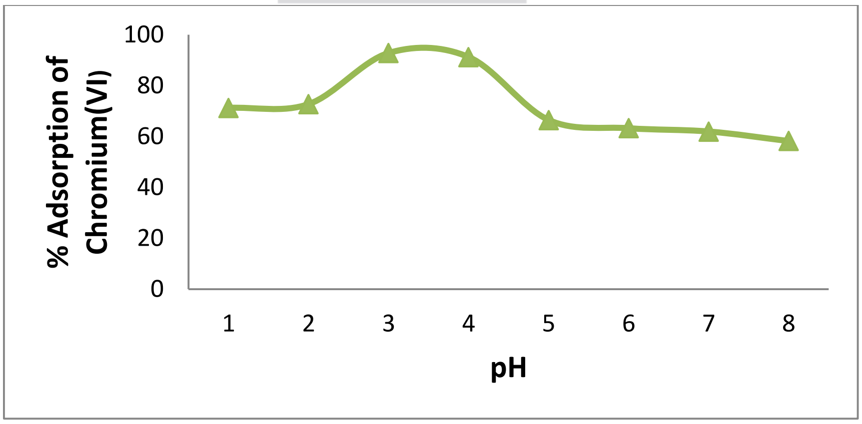


**Figure 1: Effect of pH on Chromium(VI) biosorption by Polyvinyl alcohol- alginate- Glutaraldehyde immobilized Sugarcane bagasse (Saccharum offcinarum L.) (biosorbent dose concentration: 5 g/L, Chromium(VI) concentration: 10 mg/L, contact time: 180 minutes, temperature: 30$^0$C**

**Effect of biosorbent dose concentration:**

Biosorbent dosage is an important parameter studied while conducting batch mode studies. The sorption capacity of on to Polyvinyl alcohol- alginate- Glutaraldehyde immobilized Sugarcane bagasse (Saccharum offcinarum L.) chromium (VI) ions by varying its dosage from 1 g/L to 15 g/L as shown in Figure 2. From the results it was found that biosorption of chromium (VI) ions increases with increase in biosorbent dosage and is highly dependent on biosorbent concentration. Increase in biosorption by increase in biosorbent dose is because of increase of ion exchange site ability, surface areas and the number of available biosorption sites (Naiya et al., 2009). The point of saturation for silica gel immobilized banana peels ( L.) was found at 5 g/L of biosorbent dose with 91.17 % of removal efficiency. The biosorbent dose 5 g/L was chosen for all further studies.

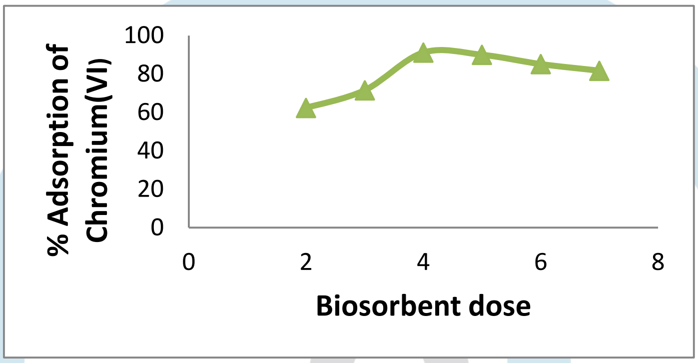



**Figure 2: Effect of biosorbent dose concentration on Chromium (VI) biosorption by Polyvinyl alcohol- alginate- Glutaraldehyde immobilized Sugarcane bagasse (Saccharum offcinarum L.) (biosorbent dose concentration: 5 g/L, Chromium(VI) concentration: 10 mg/L, contact time: 180 minutes, temperature: 30$^0$C**

**Effect of initial chromium (VI) concentration:**

The effect of initial chromium (VI) concentration from 10 mg/L-300 mg/L on the removal of chromium (VI) from aqueous solutions at biosorbent dose 5 g/L and at optimum pH 3 at 30 C was studied. On increasing the initial chromium (VI) ions concentration, the total chromium (VI) ions uptake decreased slightly at chromium (VI) ions concentration ranges from 10 mg/L-300 mg/L.

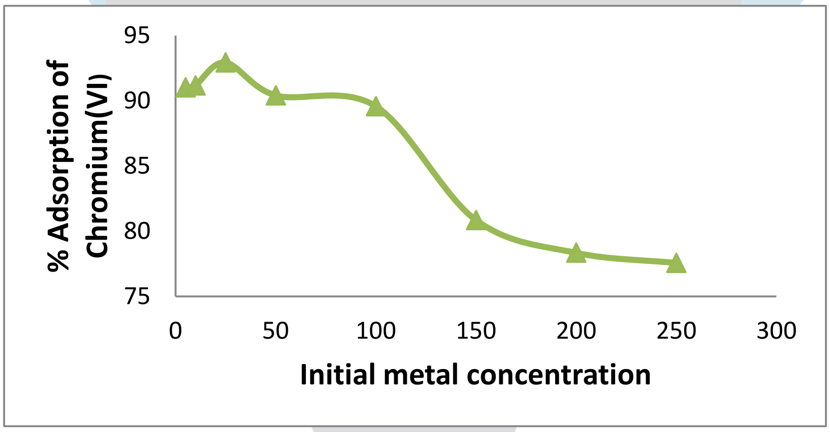



**Figure 6: Effect of chromium(VI) concentration on Chromium(VI) biosorption by Polyvinyl alcohol- alginate- Glutaraldehyde immobilized Sugarcane bagasse (Saccharum offcinarum L.) (biosorbent dose concentration: 5 g/L, Chromium(VI) concentration: 10 mg/L, contact time: 180 minutes, temperature: 30$^0$C)**

**Effect of contact time**:

In order to optimize the contact time for the maximum uptake of metals ions, contact time was varied between 10 minute-240 minute on the removal of chromium (VI) from aqueous solutions in the concentration of chromium (VI) 10 mg/L and adsorbent dose 5g/L at optimum pH 3.0 at 30 C. The results obtained from the biosorption capacity of chromium (VI) Polyvinyl alcohol- alginate- Glutaraldehyde onto immobilized Sugarcane bagasse (Saccharum offcinarum L.) showed that the biosorption increases with increase in contact time until it reached equilibrium. The optimum contact time for biosorption of chromium (VI) ions onto

Sugarcane bagasse (Saccharum offcinarum L.) was 180 minutes with 55.96% removal. The rapid uptake of chromium (VI) is due to the availability of ample active sites for sorption. So a contact time of 180 min was fixed for further experiments.

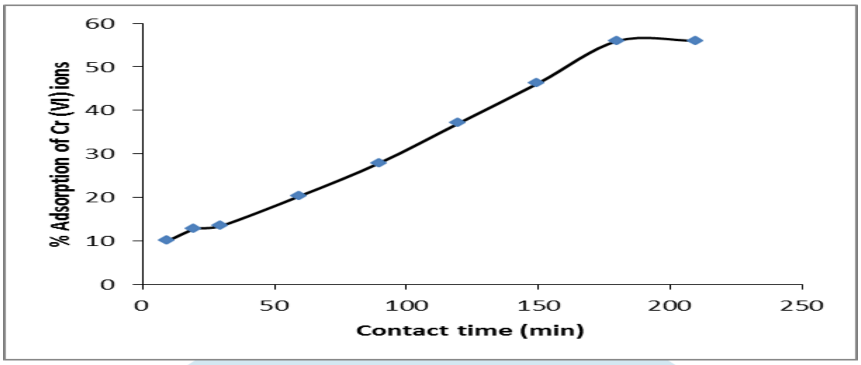



**Figure 4: Effect of contact time on Chromium(VI) biosorption by Polyvinyl alcohol- alginate- Glutaraldehyde immobilized Sugarcane bagasse (Saccharum offcinarum L.) (biosorbent dose concentration: 5 g/L, Chromium(VI) concentration: 10 mg/L, contact time: 180 minutes, temperature: 30$^0$C)**

**Effect of temperature:**

The effect of temperature on removal of chromium (VI) ions from aqueous solutions using banana peels ( L.) was studied at different temperatures from 20º C-40º C. The influence of temperature is depicted in Figure 5. Maximum sorption was seen at 30 C with percentage removal 92.63%.

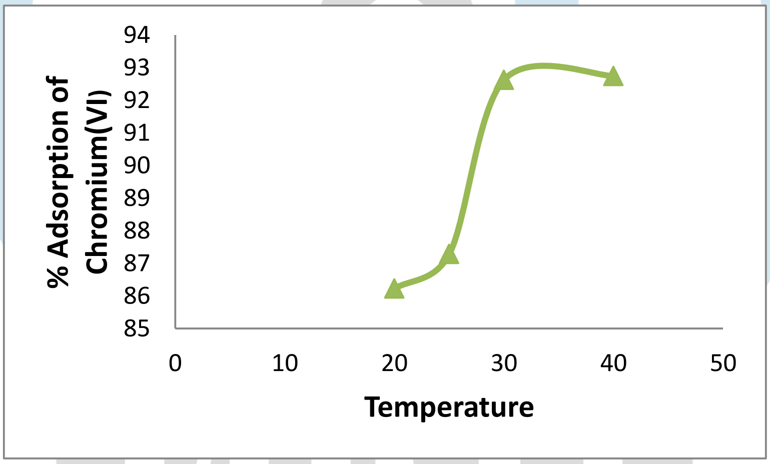



**Figure 5: Effect of temperature on Chromium(VI) biosorption by Polyvinyl alcohol- alginate- Glutaraldehyde immobilized Sugarcane bagasse (Saccharum offcinarum L.) (biosorbent dose concentration: 5 g/L, Chromium(VI) concentration: 10 mg/L, contact time: 180 minutes, temperature: 30$^0$C)**

**Effect of agitation rate:**

The effect of agitation rate on removal of chromium (VI) from aqueous solutions at biosorbent dose 5 g/L and at optimum pH 4 was studied at different rpm such as 40 rpm, 80 rpm, 120 rpm, 160 rpm and 200 rpm. The efficiency was highest at 120 rpm with percentage removal 92.75%. So, 120 rpm was chosen for all further biosorption studies

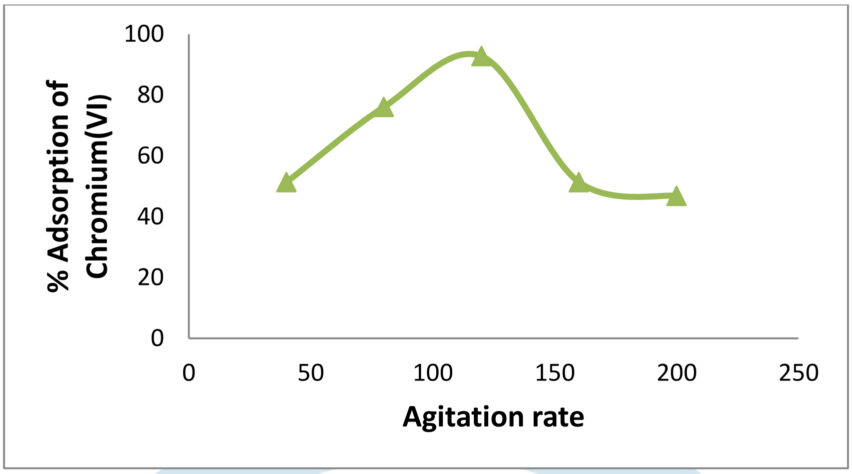



**Figure 6: Effect of agitation rate on on Lead (II) biosorption by Polyvinyl alcohol- alginate- Glutaraldehyde immobilized Sugarcane bagasse (Saccharum offcinarum L.) (biosorbent dose concentration: 5 g/L, Lead (II) concentration: 10 mg/L, contact time: 180 minutes, temperature: 30$^0$C)**

**Adsorption isotherm models**

The analysis of the adsorption isotherms data by fitting them into different isotherm models is an important step to find the suitable model that can be used for design process. The experimental data were applied to the two-parameter isotherm models: Langmuir, Freundlich, Dubinin-Kaganer-Redushkevich (DKR) and Temkin

**Langmuir adsorption isotherm (Langmuir, 1918):**

The Langmuir equation, which is valid for monolayer sorption onto a surface of finite number of identical sites, is given by:

$$q_e = \frac{q_m\, b\, C_e}{1+bC_e}$$

where qm is the maximum biosorption capacity of adsorbent (mg g-1). b is the Langmuir biosorption constant (L mg-1) related to the affinity between the biosorbent and biosorbate. Linearized Langmuir isotherm allows the calculation of adsorprtion capacities and Langmuir constants and is represented as:

$$\frac{1}{q_e} = \frac{1}{q_m\, b\, C_e} + \frac{1}{q_m}$$

The linear plots of 1/q vs 1/ce is shown in Figure 10 (a). The two constants b and qm are calculated from the slope (1/qm·b) and intercept (1/qm) of the line. The values of qm, b and regression coefficient (R 2 ) are listed in Table 1.

**Frenudlich adsorption isotherm (Frenudlich, 1939):**

Freundlich equation is represented by;

$$q = KC_e^{1/n}$$

where K and n are empirical constants incorporating all parameters affecting the biosorption process such as, biosorption capacity and biosorption intensity respectively. Linearized Freundlich adsorption isotherm was used to evaluate the sorption data and is represented as

$$\log q_e = \log K + \frac{1}{n}\log C_e$$

Equilibrium data for the adsorption is plotted as log q vs log Ce, as shown in Figure 10 (b). The two constants n and K are calculated from the slope (1/n) and intercept (log K) of the line, respectively. The values of K, 1/n and regression coefficient (R 2 ) are listed in Table 1

**Table 1: Adsorption isotherm constants for Chromium(VI) biosorption by Polyvinyl alcohol- alginate- Glutaraldehyde immobilized Sugarcane bagasse (Saccharum offcinarum L.)**

| Langmuir parameters | | | Freundlich parameters | | | DKR parameters | | | | Temkin parameters | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| $q_m$ | B | $R^2$ | K | 1/n | $R^2$ | B | $q_m$ | E | $R^2$ | $A_T$ | $b_T$ | $R^2$ |
| 151.5151 | 0.0137 | 0.9931 | 5.0792 | 2.2341 | 0.9486 | $-8\times10^{-7}$ | 24.3565 | 0.7601 | 0.7905 | 0.8504 | 246.791 | 0.9597 |

.**Dubinin-Kaganer-Radushkevich (DKR)** adsorption isotherm (Dubinin and Radushkevich, 1947): Linearized Dubinin-Kaganer-Radushkevich (DKR) adsorption isotherm equation is represented as;

$$\ln q_e = \ln q_m - \beta\,\varepsilon^2$$

where qm is the maximum biosorption capacity, β is the activity coefficient related to mean biosorption energy and ε is the polanyi potential, which is calculated from the following relation;

$$\varepsilon = RT\ln\left(1+\frac{1}{C_e}\right) \quad (10)$$

Equilibrium data for the adsorption is plotted as lnqe vs ε2 , as shown in Figure 10 (c). The two constants β and qm are calculated from the slope (β) and intercept (lnqm) of the line, respectively. The values of adsorption energy E was obtained by the following relationship,

$$E = \frac{1}{\sqrt{-2\beta}} \quad (11)$$

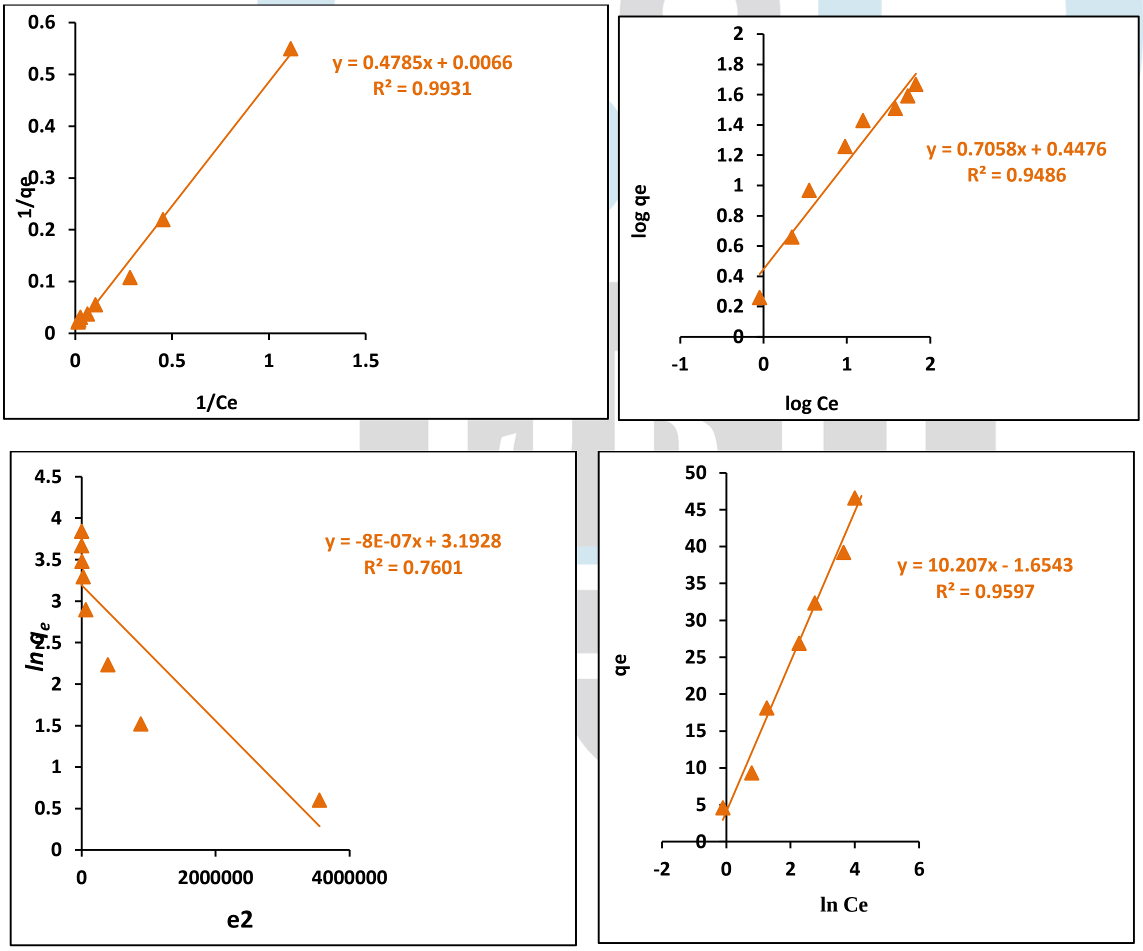



**Figure 7: Adsorption isotherms (a) Langmuir, (b) Freundlich (c) DKR and (d) Temkin for Chromium(VI) biosorption by Polyvinyl alcohol- alginate- Glutaraldehyde immobilized Sugarcane bagasse (Saccharum offcinarum L.) (biosorbent dose concentration: 5 g/L, Chromium(VI) concentration: 10 mg/L, contact time: 180 minutes, temperature: $30^0$C)**

**Temkin adsorption isotherm** (Temkin and Pyzhev, 1940):

Linearized Temkin adsorption isotherm is given by the equation;

$$q_e = \frac{RT}{b_T} \ln(A_T\, C_e) \quad (12)$$

where bT is the Temkin constant related to heat of biosorption (J/mol) and AT is the Temkin isotherm constant (L/g). Equilibrium data for the adsorption is plotted as qe vs lnCe, as shown in Figure 10(d). The two constants bT and AT are calculated from the slope (RT/bT) and intercept (RT/bT· lnAT) of the line.

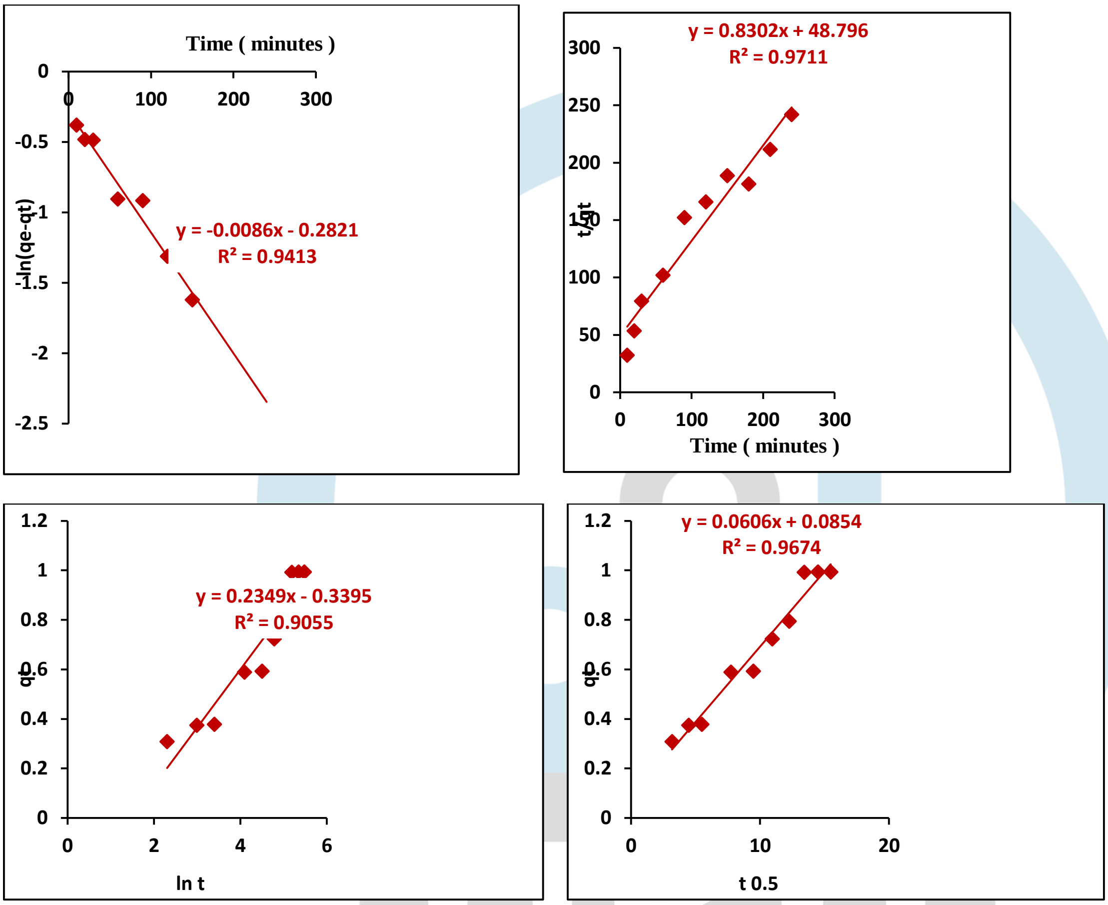
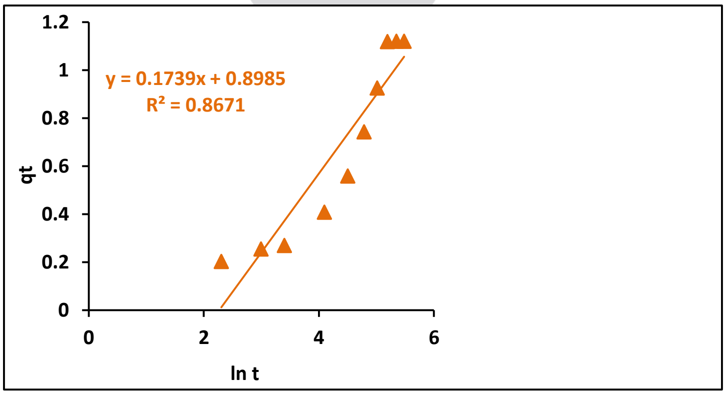



**Figure 8: Adsorption kinetic models (a) pseudo-first-order, (b) pseudo-second-order (c) Elovich and (d) Weber and Morris intra-particle diffussion equation, for biosorption of Chromium(VI) biosorption by Polyvinyl alcohol- alginate-Glutaraldehyde immobilized Sugarcane bagasse (Saccharum offcinarum L.) (biosorbent dose concentration: 5 g/L, Chromium(VI) concentration: 10 mg/L, contact time: 180 minutes, temperature: 30$^0$C)**

**Adsorption kinetics:** As aforementioned, a lumped analysis of adsorption rate is sufficient to practical operation from a system design point of view. The commonly employed lumped kinetic models, namely (a) the pseudo-firstorder equation (Lagergren 1898) (b) the pseudosecond-order equation (Mckay et al., 1999) (c) Elovich equation (Chien and Clayton 1980) (d) Weber & Morris intra-particle diffusion equation (Weber and Morris, 1963) are presented below;



$$\ln(q_e\text{-}q_t) = \ln q_e\text{-}k_1 t$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$$

$$q_t = \frac{1}{\beta}\ln(\alpha\beta) + \frac{1}{\beta}\ln t$$

$$q_t = k_i t^{0.5} + c$$

where qe (mg g-1) is the solid phase concentration at equilibrium, qt (mg g-1) is the average solid phase concentration at time t (min), k1 (min-1) and k2 (g mg-1 min-1) are the pseudo-first-order and pseudo-secondorder rate constants, respectively. The symbols of α (mg g-1 min-1) and β (g mg-1) are Elovich coefficients representing initial biosorption rate and desorption constants, respectively. $k_i$ ( mg g-1 min-1/2) is the intraparticle diffusion rate constant, c is intercept.

If the adsorption follows the pseudo-first-order model, a plot of ln (qe-qt) against time t should be a straight line. Similarly, t/qt should change lineally with time t if the adsorption process obeys the pseudo-second order model. If the adsorption process obeys Elovich model, a plot of qt against ln t should be a straight line. Also a plot of qt against t $^{0.5}$ changes lineally the adsorption process obeys the Weber and Morris intraparticle diffusion model. Kinetic plots depicted in Figure 8 (a) (b) (c) and (d) (Septhum et al., 2007).

**Determination of thermodynamic**

The effect of temperature on removal of Lead(II) from aqueous solutions in the concentration of Lead(II) 10 mg/L and biosorbent dose concentration 5 mg/ml with optimum pH 5.0 was studied. Experiments were carried out at different temperatures from 20$^0$C-40$^0$C. The samples were allowed to attain equilibrium. Sorption slightly increases from. The equilibrium constant (Catena and Bright, 1989) at various temperatures and thermodynamic parameters of adsorption can be evaluated from the following equations;

$$K_c = \frac{C_{Ae}}{C_e}$$

$$\Delta G^0 = \text{-}RT \ln K_c$$

$$\Delta G^0 = \Delta H^0\text{-}T\Delta S^0$$

$$\ln K_c = \frac{\Delta S^0}{R}\text{-}\frac{\Delta H^0}{RT}$$

where Kc is the equilibrium constant, $C_e$ is the equilibrium concentration in solution (mg/L) and CAe is the amount of Lead(II) biosorbed on the biosorbent per liter of solution at equilibrium (mg/L). $\Delta G^0$ , $\Delta H^0$ and $\Delta S^0$ are changes in standard Gibbs free energy (kJ/mol), standard enthalpy (kJ/mol) and standard entropy (J/mol K), respectively. R is the gas constant (8.314 J/mol K) and T is the temperature (K).

**Table 2: Adsorption kinetic data for for Chromium(VI) biosorption by Polyvinyl alcohol- alginate- Glutaraldehyde immobilized Sugarcane bagasse (Saccharum offcinarum L.)**

| Pseudo-first-order model | | | Pseudo-second-order model | | | Elovich model | | | Intraparticle diffusion model | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| $q_e$ | $k_1$ | $R^2$ | $q_e$ | $k_2$ | $R^2$ | *a* | *β* | $R^2$ | *Ki* | *C* | $R^2$ |
| 2.4041 | 0.0096 | 0.7083 | 0.2492 | 04013 | 0.998 1 | 30.4930 | 5.7504 | 0.867 1 | 0.0408 | 1.2526 | 0.7655 |

The values of $\Delta H^0$ and $\Delta S^0$ were determined from the slope and the intercept from the plot of lnKc versus 1/T (Figure 12). The values of equilibrium constant ($K_c$), standard Gibbs free energy change (ΔG0 ), standard enthalpy change ($\Delta H^0$ ) and the standard entropy change ($\Delta S^0$ ) calculated in this work were presented in Table 3.

**Table 3: Thermodynamic parameters of Chromium(VI) biosorption by Polyvinyl alcohol- alginate- Glutaraldehyde immobilized Sugarcane bagasse (Saccharum offcinarum L.)**

| Sr. No. | Time (min) | K | Kc | $-\Delta G^0$ | $\Delta H^0$ | $\Delta S^0$ |
|---|---|---|---|---|---|---|
| 1 | $20^0C$ | 293 | 6.5930 | 4.594 | 30.829 | 120.611 |
| 2 | $25^0C$ | 298 | 6.8667 | 4.773 | | |
| 3 | $30^0C$ | 303 | 12.5685 | 6.376 | | |
| 4 | $40^0C$ | 313 | 12.7931 | 6.632 | | |

**Conclusions:**

The present investigation revealed that Polyvinyl alcohol- alginate- Glutaraldehyde immobilized Sugarcane bagasse (Saccharum offcinarum L.) used as inexpensive, excellent biosorbent for the removal of chromium (VI) from aqueous solutions. The optimal parameters such as solution pH, biosorbent dose, agitation rate, initial chromium (VI) concentration, contact time and temperature determined in the experiment were effective in determining the efficiency of chromium (VI) onto that Polyvinyl alcohol- alginate- Glutaraldehyde immobilized Sugarcane bagasse (Saccharum offcinarum L.)Sorption equilibrium exhibited better fit to Langmuir isotherm than Freundlich isotherm, Temkin isotherm and Dubinin-KaganerRedushkevich (DKR) isotherm. The maximum chromium (VI) loading capacity of Polyvinyl alcohol- alginate- Glutaraldehyde immobilized Sugarcane bagasse (Saccharum offcinarum L.) determined from Langmuir adsorption isotherm was found to be 151.51 mg g . The second order model was found to be correlate the experimental data strongest than other three kinetic models. The thermodynamic study confirmed that reaction of biosorption of chromium (VI) was spontaneous, endothermic and increasing randomness of the solid solution interfaces. From these observations it can be concluded that Polyvinyl alcohol- alginate- Glutaraldehyde immobilized Sugarcane bagasse (Saccharum offcinarum L.) has considerable biosorption capacity, available in abundant, nonhazardous agro material can be used as an effective indigenous material for treatment of waste water stream containing chromium (VI).

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