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Uptake of Toxic Hexavelantchromium Element from Aqueous Solution by Annona Squamosa Carbon

Renukadevi C.[1] and Santhi T.[2]
[1]Department of Chemistry, Gandhi Polytechnic College, Tamil Nadu, INDIA
[2]Department of Chemistry Karpagam University, Coimbatore-641 014, INDIA

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Abstract

Annona Squamosa was used for the preparation of activated carbon for the removal of toxic elements like chromium (V1) from waste water. The effects of concentration of pH, temperature, adsorption capacity have been investigated. Adsorption capacity of Annona Squamosa activated carbon was showed highest value around pH=3. A direct proportionality between the percentages of Cr (V1) removal (86.11%) requested within 15 min from start of every experiment .The Adsorption kinetic data were tested using pseudo-first order, pseudo-second order, and Elovich and intra-particle diffusion models. Kinetics studies showed that the adsorption followed a pseudo second order reaction of Cr(V1) followed pseudo second order kinetics equations and fits the Langmuir, Freundlich , Tempkin and Elovich equation well Activated carbon developed from Annona Squmosa seed can be highly efficient option for Cr(V1) from wastewater showed better removal percentage of Cr (V1)[1].

Keywords: Annona squmosa, adsorption, kinetics, activated carbon, adsorption isotherm.

Introduction

The contamination of natural and Industrial water by heavy metals is recognized as a major environmental concern due to the impact and persistence of these pollutants. People have paid attention to it which will make us sick such as cancer or even make us to die. Among the heavy metals arsenic, copper, cadmium, lead, mercury, nickel and zinc are severely affected the environment. It will established that the presence of heavy metals in the environment even moderate concentration is responsible for producing a variety illness related with risk of dermal damage and respiratory diseases[1].

Chromium compounds are widely used in industries such as leather tanning, electroplating manufacture of dye, paint and paper. The industrial effluent[2-7] contained 1508 tones Cr (V1) in china in the year of 2004. Chromium exists in the aqueous mainly in Cr(III) and Cr(V1) oxidation states. The hexavalent form has been most hazardous due to its carcinogenic properties. Cr(V1) causes skin irradiation resulting in the ulcer formation. Adsorption remains the most economical and widely used process for removal of toxic pollutant from wastewater. Annona Squmosa as adsorbents using the batch experiments. Several isotherm and kinetic models were used to evaluate the adsorbents.

Hence removal of Cr (VI) ions from waste water assumes importance. The conventional method for heavy metal removal from waste water includes oxidation, reduction, precipitation, membrane filtration, ion exchange and adsorption. Among all, adsorption is highly effective and economical. Though the commercial activated carbon is a well-known adsorbent for the removal of heavy metals from waste water, the high cost of activated carbon limits its use as an adsorbent in developing countries[8]. Hence several research workers used different low-cost adsorbent such as coconut coir pith**,** sawdust, sludges ash banana pith, activated phosphate rock vermiculite and montmorillonite. In spite of several researchers adopted various low-cost adsorbents there is still a need to develop suitable adsorbents for the removal of chromium metal ion from aqueous solution.

The objective of the present study is to evaluate the capacity of Annona Squmosa as adsorbents using the adsorption process. Several isotherms and kinetic models were used to evaluate the adsorbents. Condition parameters such as contact time pH, initial concentrations and adsorbent concentration were considered.

Methodology

Biomass: Annona Squmosa plant was used for fencing purpose. The fruit was rich in protein and after using this fruit, the seed is used as good adsorbent by activating it in different methods. The seed was collected form house in Andhrapradesh. The seed is soaked with distilled water and dried.

Preparation of activated carbon Annona Squmosa (CAS): The dried Annona Squmosa 1.0 kg was added in a small portion to 1000 mL of 98% sulphuric acid for 12 hrs and washed thoroughly in a distilled water until it pH attained neutral pH and soaked in two percent $NaHCO_3$ overnight. Then the material was washed with distilled water and dried 110 ± 2^0C. Then preserved in desicators for use.

Preparation of Chromium solution and analysis: A stock solution of Cr (VI) was preparing by dissolving 1g of $K_2Cr_2O_7$ in 1L of deionized water, and the solution was used for further experimental studies. The pH values were adjusted with 0.1M HCL or 0.1M NaoH. Analytical grade reagents were used throughout this study. The Cr (VI) ion content in the adsorption solutions was determined by atomic absorption spectrophotometer.

Batch Adsorption Studies: Effect of pH: The initial pH values were adjusted to 1.0, 2.0, 3.0, 4.0, 5.0 and 6.0 with 0.1M HCL and 0.1M NaOH. The effect of initial pH on the chromium ions adsorption on activated Annona Squamosa adsorbent in 50mL of Cr (VI) solution in 50ppm and agitation time 1.30 minutes.

Kinetic Studies: Kinetic studies were carried out at solution pH 3.0 of Cr(VI) solution 50mL (50mg/L) and agitated time 1.30 minutes at room temperature 1. Samples were collected from duplicate flasks at different time intervals, viz, 5, 10, 15, 20, 25… 120 min, filtered by whatman filter paper no.40 the filterate was analyzed for residual chromium concentration 3.

Effect of Biomass Dose: To observe the effect of adsorbent dose on metal adsorption different amounts of adsorbent 0.2g, 0.4g, 0.6g, 0.8g, and 1g were added into initial concentration of 50 mg/L solution. The mixtures were shaken in 250 mL stoppered flask at 25^0C at pH=3 until equilibrium time was reached.

Absorption Isotherm: Batch adsorption experiments were carried out in a rotary shaker at 150 rpm using 250mL shaking flasks at room temperature for 100 min. The CAS (0.2g) was thoroughly mixed with 50mL of chromium solution. The isotherm studies were performed by varying the intial CAS concentrations from 50 mg to 250mg/L at pH 3.0. This was adjusted using 0.1M HCL or 0.1M NaoH before addition of CAS and maintained throughout the experiment. After shaking the reaction mixture was analyzed for chromium concentration. The concentration of chromium was measured by using colorimeter.

Results and Discussion

Effect of pH on Cr (VI) adsorption: The results of the pH effect in the adsorption process were analyzed. The effect of pH changes due to the adsorbent type, its behavior in the solution and the type of ions in adsorbed[9]. In this study, the optimum pH value for the Cr (VI) ions were determined as pH=3. So further experiments were carried on by using this selected value.

Effect of adsorbent dosage and contact time: The effect of dosage and contact time on the removal of Cr (VI) ions increase of adsorbent dosage from 0.2g to 1g the removal of Cr (VI) ions was observed at the beginning of the contact time. Following the rapid removal, the removal rate decreased, and an apparent equilibrium was reached after 1 hour 30 minutes depending upon on the adsorbent dosages. The high removal rate at the start of the contact time was due to the large amount of surface area available for adsorption of the Cr (VI) ions. At this stage, the adsorption mainly occurred on the surface of the adsorbent[10]. After the rapid uptake, the capacity of the adsorbent became exhausted and the adsorption would be replaced by the transportation of Cr (VI) ions from the external sites to the internal sites of the adsorbent particles. Therefore, the uptake late began to drop down, which was explained by intraparticle diffusion model[11].

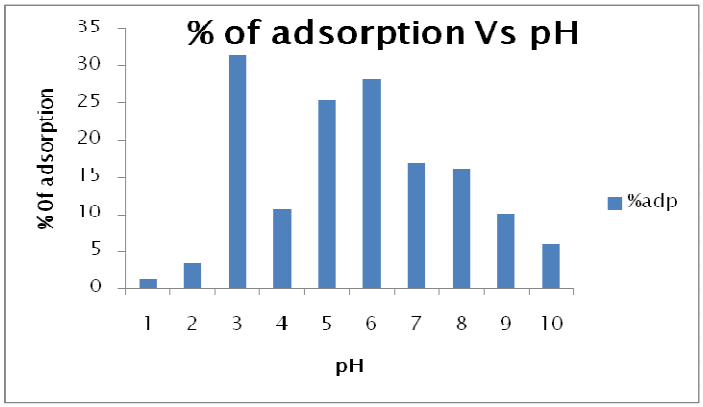


Figure-1
Effect of pH on the adsorption of Cr (VI) 50mg/L

Effect of Adsorbent dosage on Cr (V1) Adsorption:

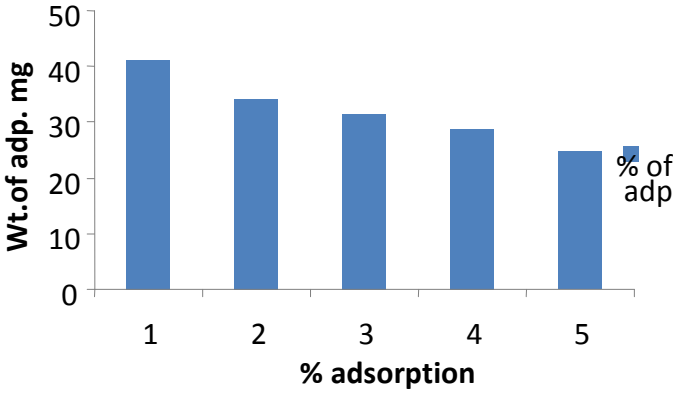


Figure-2
Effect of adsorbent concentration on Cr (VI) removals (pH 3.0, Agitation speed: 150 rpm, 27±2 ^{0}C)

Equilibrium Isotherms: To optimize the design of an adsorption system for the adsorption of adsorbates, it is important to establish the most appropriate correlation for the equilibrium curves[12]. The equation parameters of these equilibrium models often provide some insight into the sorption mechanism, the surface properties and the affinity of the adsorbent[13]. Langmuir, Freundlich, and Dubinin-Radushkevich (D-R) were used to describe the equilibrium characteristics of adsorption[14].

Langmuir isotherm: Langmuir theoretical isotherm is 10 valid for adsorption of a solute from a liquid solution as monolayer adsorption on a surface containing a finite number of identical sites. Langmuir isotherm model assumes uniform energies of adsorption onto the surface without transmigration of adsorbate in the plane of the surface[15]. Therefore, the Langmuir isotherm model was chosen for estimation of the maximum adsorption capacity corresponding to complete monolayer coverage on the adsorbent surface. The Langmuir non-liner equation is commonly expressed as followed:

$$qe = Q_m \ Ka\ C_e/1+K_a\ C_e \quad (1)$$

In equation (1) q_e and C_e are the *(q_e)* amount of Cr (VI) ion adsorbed at equilibrium onto carbon,(C_e) equilibrium liquid-phase concentration of (VI) ions, Q_m is a constant and reflect a complete monolayer (mgg^{-1}); K_a is the adsorption equilibrium constant (Lmg^{-1}) that is related to the apparent energy of sorption. The Langmuir isotherm equation (1) can be linearized into the following form[16,17].

$$\text{Langmuir-1}\ C_e/q_e=1/K_aQm+1/Q_m \times C_e \quad (2)$$

A Plot of C_e/Q_e versus C_e should indicate a straight line of slope $1/Q_m$ and an intercept of $(1/K_aQ_m)$.

The applicability of the linear form of Langmuir model to CAS was proved by the high correlation coefficients R^2=0.968.This suggests that the Langmuir isotherm provides a good model of the sorption system.

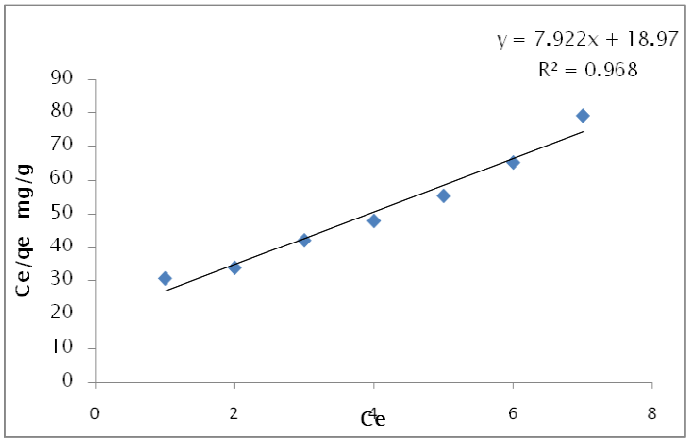


Figure-3
Langmuir isotherm for the adsorption (VI) onto CAS, (pH 3.0, contact time=1.30 min, CAS dose=0.2g/L)

The Freundlich isotherm: The Freundlich isotherm model[18] is earliest known equation describing the adsorption process. It is an empirical equation and can be used for non-ideal sorption that involves heterogeneous adsorption. The Freundlich isotherm can be derived assuming a logarithmic decrease in the enthalpy of adsorption with the increase in the fraction of occupied sites and is commonly given by the following non-linear equation:

$$qe=K_F\ C_e^{1/n} \quad (3)$$

Where K_F is a constant for the system, related to the bonding energy, K_F can be defined as the adsorption or distribution co-efficient and represents the quantity of metal absorbed onto adsorbent for unit equilibrium concentration 1/n is indicating the adsorption intensity of metal onto the adsorbent surface heterogeneity, becoming more heterogeneous as its value gets closer to zero. A value for 1/n below 1 indicates a normal Langmuir isotherm while 1/n above 1 is indicative of co-operative adsorption equation 4. Can be linearized in the logarithmic form equation 4. And the Freundlich constants can be determined.

$$\text{Log}\ q_e=\log K_F+1/n \log C_e \quad (4)$$

The applicability of the Freundlich adsorption isotherm was also analyzed using the same set of experimental data, by plotting log (qe) versus log (Ce*)*. The data obtained from linear Freundlich isotherm plot for the adsorption of the metal onto CAS presented in table 1. The correlation coefficients (1>0.994) showed that the Frendlich model is comparable to the Langmuir model. The 1/n is lower than the 1.0 indicating Cr (VI) is favorably adsorbed by CAS[19].

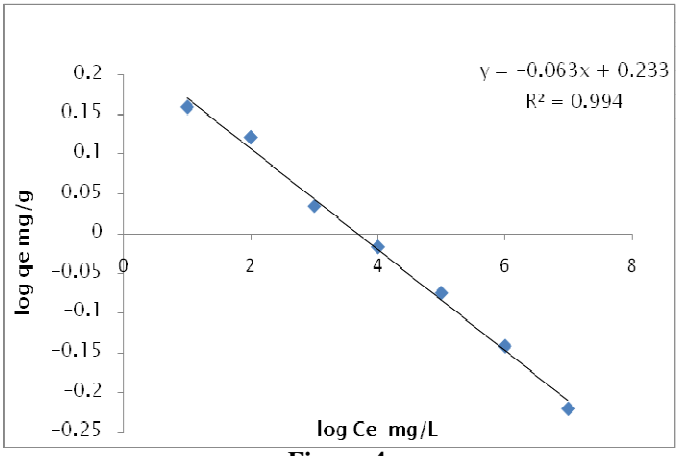


Figure-4
Freundlich isotherm for the adsorption (VI) onto CAS, (pH 3.0, contact time=1.30min, CAS dose=0.2g/L)

The Tempkin isotherm: Tempkin adsorption isotherm model was used to evaluate the adsorption potentials of the CAS for Cr (VI) metal. The derivation of the Tempkin isotherm assumes that the fall in the heat of adsorption is linear rather than logarithmic, as implied in the Frendlich equation. The Tempkin isotherm has commonly been applied in the following form

$$q_e=RT/b \ln (AC_e) \quad (5)$$

The Tempkin isotherm equation (5) can be simplified to the following equation.

$$q_e=ß \ln a+ß \ln C_e \quad (6)$$

Where ß= (RT)/b, T is the absolute temperature in Kelvin and R is the Universal gas constant, 8.134 J (mol K) $^{-1}$.The constant

b is related to the heat of adsorption[20,21]. The adsorption data were analyzed according to the linear form of Tempkin isotherm equation [6]. Examination of the data shows that the Cr (VI) adsorption the linear isotherm constants and coefficients of determination are presented in table 1. The correlation coefficient R^2 obtained from Tempkin model were comparable to that obtained for Langmuir and Freundlich equations which explain the applicability of Tempkin model to the adsorption of Cr (VI) onto CAS.

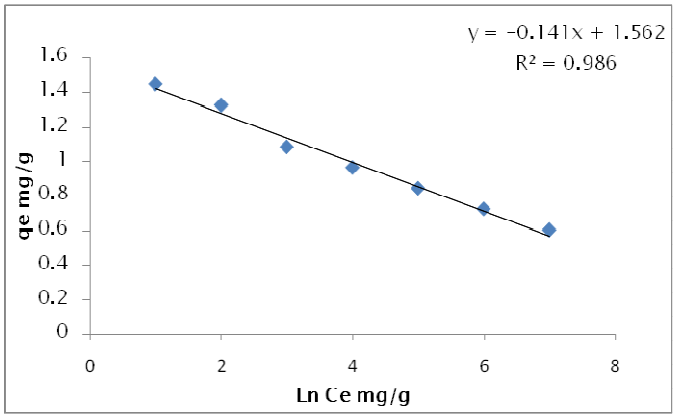


Figure-5
Tempkin isotherm for the adsorption (VI) onto CAS, (pH 3.0, contact time=1.30min, CAS dose=0.2g/L)

The Dubinin-Radushkevich (D-R) Isotherm: The D-R model was also to estimate the porosity apparent free energy and the characteristics of adsorption[22,23,24]. The D-R isotherm does not assume a homogeneous surface or constant adsorption potential. The D-R model CAS commonly been applied in the following equation 7. and its linear form can be shown in equation 8.

$$q_e = Q_m \exp(-K\varepsilon^2) \quad (7)$$

$$\ln q_e = \ln Q_m - K\varepsilon^2 \quad (8)$$

Where K is a constant related to the adsorption energy, Q_m (mg g^{-1}) is the theoretical saturation capacity is the Polanyi potential, calculated from equation 9.

$$\varepsilon = RT \ln (1+1/C_e) \quad (9)$$

The slope of the plot of ln q_e versus ε^2 gives K (mol^2 (KJ^2) $^{-1}$) and the intercept yields the adsorption capacity, Q_m (mg g^{-1}). The mean free energy of adsorption (E), defined as the free energy change when one mole of ion is transferred from infinity in solution to the surface of the solid, was calculated from the K value using the following relation [25].

$$E=1/\sqrt{2K} \quad (10)$$

The calculated value of D-R parameters is give in table 1. The saturation adsorption capacity Q_m obtained using D-R isotherm model for adsorption of Cr (VI) onto CAS is 3.45 mgg^{-1} at 0.2g/50mL adsorbent dose. Which is close to that obtained (0.126mgg^{-1}) from the Langmuir isotherm model (table 1). The values of E calculated using equation 11 is 0.1032 $kJmol^{-1}$ which indicates that the physical sorption process plays a significant role in the adsorption of Cr (VI) onto CAS

D-R isotherm:

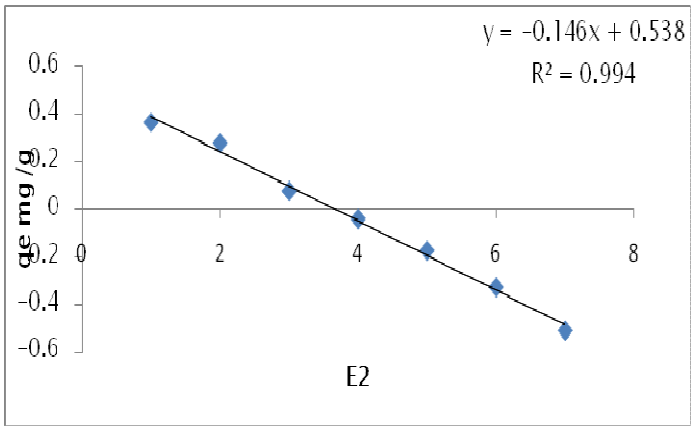


Figure-6
D-R isotherm for the adsorption (VI) onto CAS, (pH 3, contact time=1.30min, CAS dose=0.2g/L)

Table-1
Comparison of the coefficients isotherm parameters for Cr (VI) adsorption onto CAS

Isotherm Model	CAS Concentrations (g/50mL) (0.2g/50mL)
Langmuir	
$Q_m(mg.g^{-1})$	0.126
$K_a(Lmg^{-1})$	0.05271
R^2	0.9688
Frenundlich	
1/h	0.233
$K_F(mgg^{-1})$	1.710
R^2	0.994
Tempkin	
$\alpha(Lg^{-1})$	3.68
ß(mgL^{-1})	0.141
K^2	1.7866
R^2	0.986
DubininRadushkevich	
Q_m (mg.g^{-1})	3.45
K (x 10^{-5} mol^2 KJ^{-2})	0.538
E=(KJ mol^{-1})	0.1032
R^2	0.994

Kinetic Models Applied To the Adsorption of Cr (VI) Onto CAS: Several steps can be used to examine the controlling mechanism of adsorption process such as chemical reaction, diffusion control and mass transfer; Kinetic models are used to test experimental data from the adsorption of Cr (VI) adsorption onto CAS. The kinetics of Cr (VI) adsorption onto CAS is required for selecting optimum operating conditions for the full-scale batch process. The Kinetic parameters, which are helpful for the prediction of adsorption rate, give important information for designing and modeling the adsorption processes. Thus the Kinetics of Cr (VI) onto CAS was analyzed using pseudo-first-

order[22], pseudo-second-order[26]. Elovich[27,28] and intraparticle diffusion[29,30] kinetic models. The conformity between experimental data and the model predicted values was expressed by the correlation coefficients (R^2, values close or equal to 1). The relatively higher value is the more applicable model to the Kinetics of Cr (VI) adsorption onto CAS.

Pseudo- First –Order Equation: The adsorption Kinetic data were described by the Lagergren Pseudo-First-order model[31.] which is the earliest known equation describing the adsorption rate based on the adsorption rate based on the adsorption capacity. The differential equation is generally expresses a follows:

$$dq_t/d_t = k_1 (q_e-q_t) \quad (11)$$

Where qe and qt are the adsorption capacity at equilibrium and time t, respectively (mg g^{-1}). k_1 is the rate constant of pseudo-first- order adsorption ($Lmin^{-1}$) Integrating equation (11) for the boundary conditions t=0-t and q_t=0-q_t it gives.

$$\log (q_e/q_e-q_t)=k_1/2.303 \times t \quad (12)$$

Equation (12) linear form:

$$\log (q_e-q_t) = \log (q_e)-k_1/2.303 \times t \quad (13)$$

In order to obtain the rate constants the values of log (q_e-q_t) were linearly correlated with of log (q_e-q_t) versus t to give a linear relationship from which k_1 and predicted q_e can be determined from the slope and intercept of the plot. The variation in the rate should be proportional to the first power of concentration for strict surface adsorption. However, the relationship between initial solute concentration and rate of adsorption will not be linear when pore diffusion limits the adsorption process. The pseudo-first order equation fits well for the first 50 min and there after the data deviate from theory. Further more, the correlation coefficient R^2 was relatively low and adsorption constant (Q_m) was different from experimental value (table 2). This shows that the adsorption of Cr (VI) cannot be applied and the reaction mechanism is not a first-order reaction.

Pseudo-Second-order-equation: The adsorption Kinetics may be described by the Pseudo-Second-order model[32]. The differential equation is generally given as follows:

$$dq_t/d_t =k_2(q_e-q_t)^2 \quad (14)$$

Where k_2 (g(mg min)$^{-1}$) is the second-order rate constant of adsorption. Intergrating Eq.(14). for the boundary conditions qt=0-qt at t=0-t is simplified as can be rearranged and linear equation is:

$$(t/q_t) = (1/k_2 q_e^2) +1/q_e (t) \quad (15)$$

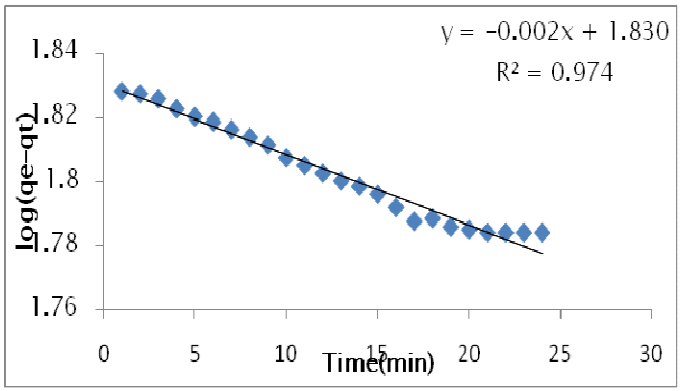


Figure-7
First order kinetics for the adsorption (VI) onto CAS, (pH3, contact time=1.30min, CAS dose=0.2g/L)

This second-order rate constants were used to calculate the initial sorption rate.

$$h=k_2qe^2 \quad (16)$$

Values of k_2 and equilibrium adsorption capacity q_e were calculated from the intercept and slope of the plots of t/q_t versus t. The linear plots of t/qt versus t show good agreement between experimental and calculated q_e values at 0.2mg/50mL adsorbent concentrations (table 2). The correlation coefficients for the Second-order Kinetic model are 0.972 and the adsorption constant (q_m) was close to experimental value, which led to believe that pseudo-second-order Kinetic model provided good correlation for the bio adsorption of Cr (VI) onto CAS.

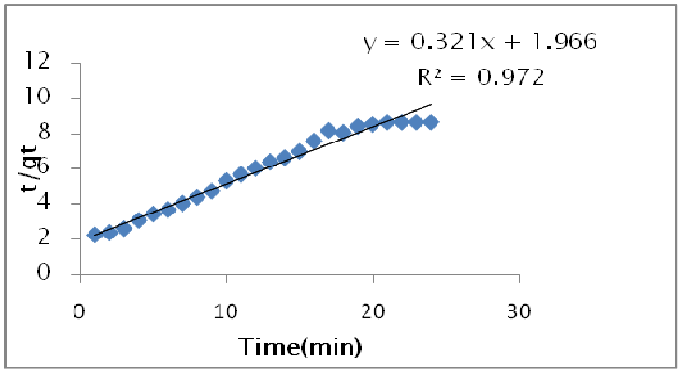


Figure-8
Pseudo-Second-Order kinetics for the adsorption (VI) onto CAS, (pH 3, contact time=1.30min, CAS dose=0.2g/L)

Table-2
Comparison of first order and second order adsorption data, [CAS 50mL/0.2mg]

Parameter	First-order kinetic model	Second - order kinetic model
R^2	0.974	0.972
q_e	67.60	3.1152
K_1	0.004606	----
K_2	----	0.63180
h	----	6.131
Experimental (q_e)	69.5381	69.5381

K_1 (min^{-1}) k2g (mg min^{-1}), q_e (mg g^{-1}), h (mg (g min) $^{-1}$)

Elovich Equation: The Elovich equation is another rate equation based on the adsorption capacity generally expressed as[33,34]

$$dq_t/dt=B_E \exp(-A_E qt) \quad (16)$$

Where B_E is the initial adsorption rate (mg (g min^{-1})) and A_E is the de-sorption constant (g mg^{-1}) during any experiment. It is simplified by assuming $A_EB_E >> t$ and by applying the boundary conditions qt=0 at t=0 and qt=qt at t=t equation (16) can be written as follows:

$$q_t=1/A_E \ln(B_EA_E) +1/A_E \ln(t) \quad (17)$$

If Cr (V1) adsorption by CAS fits the Elovich model, a plot of qt versus ln (t) should yield a linear relationship with a slope of $(1/A_E)$ and an intercept of $(1/A_E) \ln (A_EB_E)$ t Thus, the constants can be obtained from the slope and the intercept of the straight line (table 3). The intial adsorption rate was 3.1152mg (g min)$^{-1}$ for the initial Cr (VI) concentration of 50mgL^{-1} on CAS dose 0.2gmL^{-1}.Similar pattern is mentioned above for the Initial adsorption rate, h, obtained from pseudo-second-order model. The desorption constant, A_E was 3.1152g mg^{-1} for initial Cr(V1) concentration 50mg L^{-1} over CAS dose of 0.2g mL^{-1}.

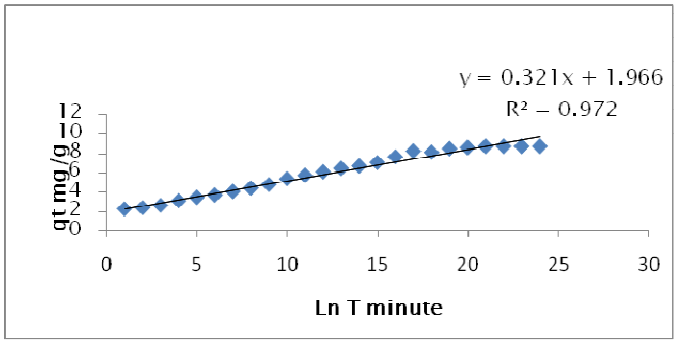


Figure-9
Elovich model plot for the adsorption (VI) onto CAS, (pH 3, contact time=1.30min, CAS dose=0.2g/L)

The Intraparticle Diffusion Model: The adsorption mechanism of adsorbent follows three steps viz, film diffusion, pore diffusion and intra-particle transport. The slowest of the three steps controls the overall rate of the process. The slowest of the three steps controls the overall rate of the process. Generally, pore diffusion and intra-particle diffusion are often rate-limiting in a batch reactor[35], which for continuous flow system film diffusion is more likely the rate limiting step. The liner form of intra particle diffusion model can be expressed as follows.

$$q_t=K_{diff} t^{1/2}+B_L \quad (18)$$

Where K_{diff} (mg g^{-1} min$^{-1/2}$) is the intra-particle diffusion rate constant. The correlation coefficients of the plot range between 0.972 which indicate low linearity for the adsorption of Cr (VI) by CAS. if intra-particle diffusion is the Sole rate-limiting step, it is for the qt versus $t_{1/2}$ plots to pass through the origin, which is not the case in this study, it may be concluded that surface adsorption and intra-particle diffusion were concurrently operating during the Chromium-adsorbent interactions. The evidence for such conclusion is the rate constants of Second-order and intra-particle diffusion, where their values are the highest among other Kinetic models.

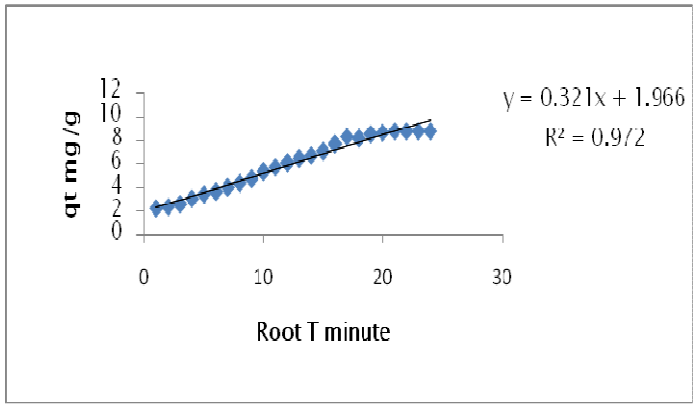


Figure-10
Intraparticle Diffusion plot for the adsorption Cr (VI) onto CAS, (pH 3, contact time=1.30min, CAS dose=0.2g/L)

Table-3
Comparison parameter obtained from the Elovich kinetic model and intraparticle diffusion model

Parameter	Elovich	Intraparticle Diffusion
R^2	0.972	0.972
A_E	3.1152	-------
B_E	456.352	--------
K_{dif}	----------	0.321
C	----------	1.966

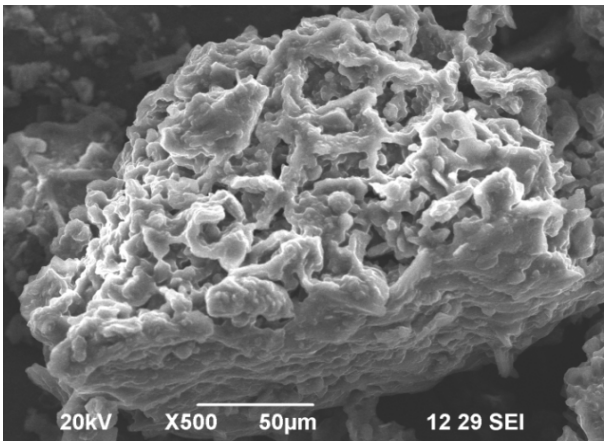
k_1 (min^{-1}) k_2(mg min^{-1}), q_e (mg g^{-1}), h (mg (g min^{-1})

SEM Test of CAS: Scanning electron microscopy test was used to know about the surface morphology and pore size of activated and non-activated carbon. The international union of pure and applied chemistry (IUPAC) classified pores and micropores (<2 nm diameter) mesopores (2-50nm diameter) and macropores (>50nm diameter). According to this classification, most pollutant molecules have different size ranging from 0.4nm to 0.9nm in diameter therefore micropores are preferred for the adsorption onto activated carbon.

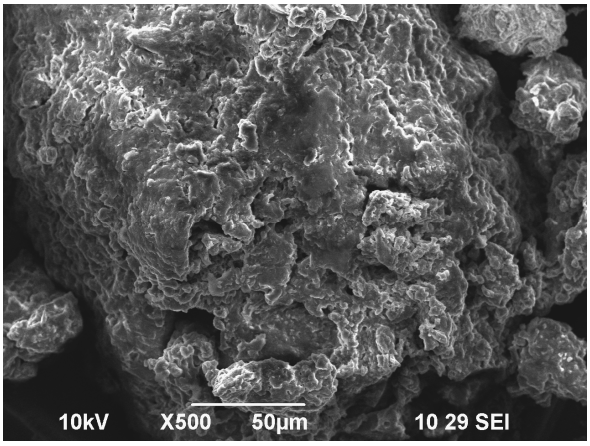
Conclusion

The results of this investigation show that activated carbon was prepared from Annona Squmosa seed CAS a suitable adsorption capacity for removal of Cr (VI) from aqueous solutions. The equilibrium adsorption is achieved in 120 min. The isotherms like Langmuir, Freundlich, and Tempkin and Dubinin Radushkevich isotherm equations are well fitted. Monolayer adsorption behaviour was explained using Langmuir-type isotherm. The kinetic study of Cr (VI) on CAS was explained using Pseudo-first-order, Pseudo-second-order, Elovich and intraparticle diffusion equations.

Through this study the adsorption kinetics follow the Pseudo-second-order rate with intraparticle diffusion as one of the rate determining steps. The present study concludes that the CAS was low-cost adsorbents as alternatives to commercial activated carbon for the removal of Cr (VI) from water and waste to water.



(a) SEM micrographs of the CAS before adsorption



(b) SEM micrographs of the CAS after adsorption

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