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**Kinetics and isotherm studies on the adsorption of
hexavalent chromium onto phosphoric acid activated
Mimusops elengi leaves carbon**

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Abstract

Hexavalent chromium [(Cr (VI))] ion is toxic and therefore, its removal from wastewater is extremely important. This study reports on the adsorption of Hexavalent Chromium from aqueous solutions using activated carbon prepared from leaves of *Mimusops elengi* by phosphoric acid activation. Adsorption experiments were carried out in a batch process with various experimental parameters such as effect of contact time, initial chromium ion concentration, carbon dosage and pH. Kinetics of the system was studied with linearised forms of Lagergren, Ho and Webber Morris models. Equilibrium data were fitted with Langmuir, Freundlich, Temkin and Dubinin-Raduskevich isotherms.

Keywords: Activated carbon, Phosphoric acid activation, Isotherms, Kinetics, and Hexavalent Chromium.

1. Introduction

Heavy metals are not biodegradable and tend to accumulate in living organisms. Those metals are considered to be potentially hazardous and can cause physiological and neurological disorders [1]. Out of the various toxic pollutants chromium and its compounds are considered as the most dangerous inorganic water pollutants. Chromium compounds present in the effluents as a result of electroplating, metal finishing, magnetic tapes, wood preservation, leather tanning, pigments and chemical manufacturing industries [2, 3]. Cr (VI) causes genetic mutations and cancer because of its elevated hyper toxicity [4], mutagenicity [5] and carcinogenicity [6]. Adsorption by activated carbon is one of the effective techniques for Cr (VI) ion removal from wastewater because of the high surface area, highly porous character and relatively low cost of the adsorbent [7]. Activated carbon is especially

known for the effective removal of organic chemicals, inorganic and heavy metal ion pollutants from wastewater in the laboratory as well as in various industries [8, 9]. In the past years, several investigations have been reported for the removal of metal ions using activated carbons developed from industrial or agricultural wastes [10].

Mimusops elengi is a medium-sized evergreen tree found in tropical forests in South Asia, Southeast Asia and northern Australia. Therefore activated carbon is prepared from the leaves of *Mimusops elengi* using phosphoric acid as activating agent and ability of the prepared carbon to remove Cr(VI) ions from aqueous solution through adsorption was investigated.

3. Data Processing Tools

2. Experimental

2.1 Preparation of activated carbon

20 g of the crushed and ground *Mimosops elengi* leaf powder were soaked in 100 ml H_3PO_4 solution (60%). The liquid/solid mixture was stirred continuously at ambient temperature for 2 h and left to soak for 12 h to allow penetration of the H_3PO_4 into the leaf powder of *Mimosops elengi* [11]. After the stipulated soaking time, the slurry was oven dried at $110^\circ C$ for 24 h. The activation temperature and time for this work was fixed at the lowest possible value [12,13]. The samples were cooled and washed with cold deionized water until phosphate ions were no longer detected by the lead nitrate test. The product was finally dried at $105^\circ C$ for 3 h, after which it was ground, sieved to get the particle size range of 75 to 105 μm and characterized. This carbon was designated as Phosphoric Acid Treated *Mimosops elengi* Activated Carbon (PTMAC).

2.2 Preparation of stock solution

Cr (VI) ion stock solution (1000 mg /L) was prepared by dissolving 2.835 g $K_2Cr_2O_7$ (AR grade) in one litre of double distilled water. The experimental solutions were prepared by proper dilution [14-15].

2.3 Adsorption experiments

The effect of adsorption parameters such as initial concentration of Cr (VI) ion, adsorbent dose and contact time was studied by batch mode technique because of its simplicity. Pre-determined dose of the adsorbent was taken in 250 mL iodine flask and 50 mL and pre-determined concentration of the Chromium solution was poured into the flask. Desired pH of the solution was brought by adding drops of either Con HCl or 6 N NaOH solution. Then the content of the flask was agitated using rotary shaker with 180 rpm for pre-determined duration. Then adsorbents were then separated by centrifugation and the concentration of the solution was determined by 'Diphenylcarbazide' method. The percentage removal of the Cr (VI) from the solution was calculated by the mass balance relationship.

2.4 Diphenyl carbazide method

1 mL of the sample solution was pipette out into 25 mL standard flasks. To this 1 mL of 6 N H_2SO_4 was added followed by 1 mL of 0.25% (W/V) diphenylcarbazide solution was prepared in 50% (V/V) acetone) and the total volume was made up to 25 mL using double distilled water. Concentration of Cr (VI) ion was estimated by measuring intensity of the reddish brown color developed due to complex formation using Systronics Double Beam UV-visible Spectrophotometer: 2202 at the wave length of 540 nm [16, 17].

3.1 Determination of percentage of removal and quantity adsorbed

The percentage removal of the Cr(VI) ions from the solution was calculated by the following mass balance relationships.

$$\begin{aligned} \% \text{ of Removal} &= (C_i - C_t) \times V / C_i \\ q_e &= (C_i - C_e) \times V / W \\ q_t &= (C_i - C_t) \times V / W \end{aligned}$$

Where, C_i , C_t and C_e are the liquid phase concentrations in mg/L of Cr(VI) ion at initial, at the time 't' and at equilibrium respectively. V is the volume of the adsorbate solution in liter (L) and W is the mass of the adsorbent in gram (g).

3.2 Isotherms

3.2.1 Langmuir isotherm

Langmuir equation is written in the following form [18]

$$Q_e = Q_0 b C_e / 1 + b C_e$$

This equation is often written in linear forms as

$$C_e / Q_e = 1 / Q_0 b + C_e / Q_0$$

where Q_e is the amount of solute adsorbed per unit weight of adsorbent (mg/g) at equilibrium, C_e the equilibrium concentration of solute in the bulk solution (mg/L), Q_0 is adsorption efficiency and also called as the maximum monolayer adsorption capacity or saturation capacity (mg/g) and b is the adsorption energy, 'b' is the reciprocal of the concentration at which half saturation of the adsorbent is reached.

The essential characteristics of Langmuir isotherm can be described by a separation factor, R_L , which is defined by the following equation.

$$R_L = 1 / (1 + bC_0)$$

Where, C_0 is the initial concentration of the adsorbate solution. The separation factor, R_L , indicates the shape of the isotherm and the nature of the adsorption process which is given below,

R_L value	Nature of the process
$R_L > 1$	Unfavourable
$R_L = 1$	Linear
$0 < R_L < 1$	Favourable
$R_L = 0$	Irreversible

3.2.2 Freundlich isotherm

This equation has the following form[18]

$$\log Q_e = \log K_f + 1/n \log C_e$$

Where Q_e is the amount of adsorbate adsorbed (mg/g), C_e is the equilibrium concentration of adsorbate solution (mg/L) and K_f and n are the constants incorporating all factors affecting the adsorption capacity and intensity of adsorption respectively.

3.2.3 Temkin Isotherm

The linear form of Temkin equation is [19].

$$q_e = RT/b_T \ln a_T + RT/b_T \ln C_e$$

Where b_T is the Temkin constant related to heat of sorption (J/mg) and a_T is the equilibrium binding constant corresponding to the maximum binding energy (L/g).

3.2.6 Dubinin – Raduskevich Isotherm

The Linear form of Dubinin-Radushkevich isotherm, [18].

$$\ln q_e = \ln q_D - B^2$$

Where, q_D is the theoretical saturation capacity (mg/g) B is a constant related to the mean free energy of adsorption per mole of the adsorbate (mol^2/J^2) and \ln is Polanyi potential which is related to the equilibrium concentration as follows;

$$= RT \ln (1+1/C_e)$$

The mean free energy of adsorption E calculated from B using the following equation

$$E = 1/ (2B)^{1/2}$$

3.3 Kinetics studies

3.3.1 Pseudo First order kinetics

Lagergren equation is[20].

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

Where q_e and q_t are the amounts of adsorbate adsorbed (mg/g) at equilibrium and at time t (min), respectively and k_1 is the rate constant of adsorption (l/min).

3.3.2 Pseudo Second order kinetics

Ho equation is [21].

$$t/q_t = 1/ k_2 \cdot q_e^2 + 1/q_e t$$

The initial adsorption rate, h (mg/(g min)), as $t \rightarrow 0$ can be defined as

$$h = k_2 q_e^2$$

3.3.4 Intra particle diffusion

Weber–Morris equation is[22].

$$q_t = k_p t^{1/2} + C$$

Where k_p is the intra-particle diffusion rate constant, a plot of q_t versus $t^{1/2}$ should be a straight line with a slope k_p which is the rate constant for intra particle diffusion and intercept C is the thickness of the boundary film.

3.3.5 Test for Kinetics Models

The sum of error squares is given as follows;

$$\text{SSE (\%)} = [(q_e)_{\text{exp}} - (q_e)_{\text{cal}}]^2 / N$$

Where N is the number of data points, $(q_e)_{\text{exp}}$ is the experimental q_e and $(q_e)_{\text{cal}}$ is the calculated q_e .

4. Results and Discussion

4.1 Effect of pH

The pH of the solution is an important variable which controls the adsorption. Hence, the influence of pH on the adsorption of Cr(VI) ion onto activated carbon was examined in the pH range of 2 to 10. Figure 1 shows the effect of initial pH of the solution on the removal of Cr(VI) ion. The percentage removal of Cr(VI) ions increased significantly, with the decrease of solution pH value and the maximum removal was attained at pH (2.0). This kind of result was reported earlier [23].

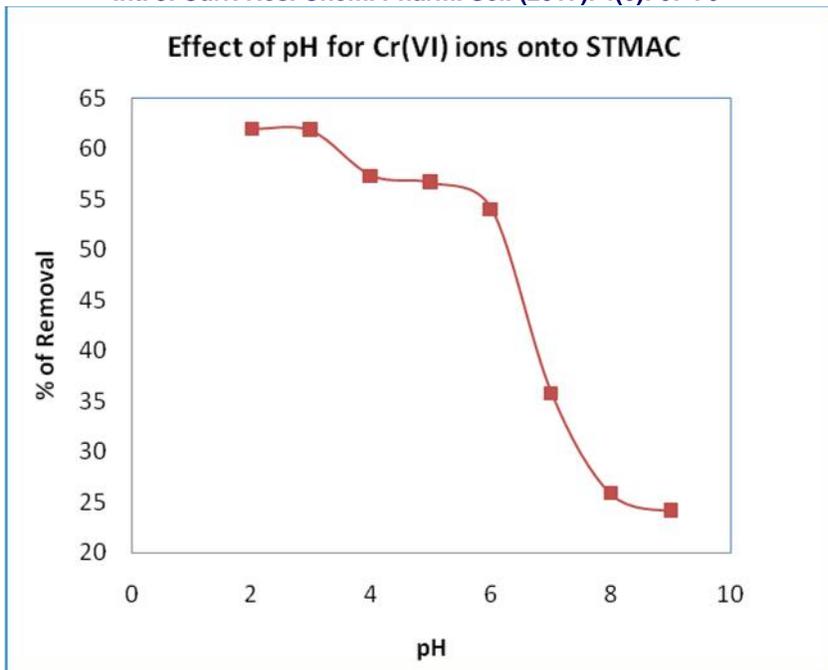


Figure 1 Effect of pH

4.2 Effect of Contact time and Initial concentration on adsorption

The effect of contact time on the percentage removal was studied by taking 10 mg/L, 20 mg/L, 30 mg/L, 40 mg/L and 50 mg/L solutions as initial concentrations.

The adsorption process was characterized by a rapid uptake of the adsorbate at the initial stages. Then the rate of percentage removal was found to decrease gradually as the contact time increases and become constant after attaining equilibrium stage in all the cases.

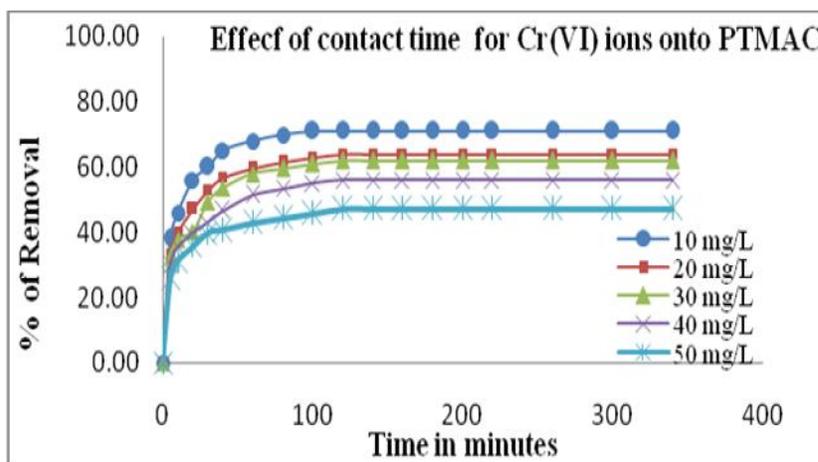


Figure 2 Effect of contact time

4.2 Isotherm studies

The equilibrium data obtained from the experiments were processed with different isotherm models such

as Langmuir, Freundlich, Temkin and Dubinin-Radushkevich. Inference obtained from each isotherm was discussed in detail one by one.

4.2.1 Langmuir Isotherm

The Langmuir isotherm represents the equilibrium distribution of adsorbate molecules between the solid and liquid phases [24]. The squares of correlation coefficient (R^2) values ranged from 0.993 to 1 for the three studied temperatures viz. 305, 315, 325 and 335 K. These results show the best fitting of the equilibrium

data with Langmuir isotherm. The mono layer adsorption capacity Q_0 values (mg/g) for adsorption of Cr(VI) ion onto PTMAC ranged from 57.143 to 63.694 mg/g. The adsorption capacity increased with the increase of temperature. The maximum removal of Cr(VI) ion was 200 mg/g. The values of R_L in all cases lie between 0 and 1, indicating that the adsorption process was favourable.

Table 1 Langmuir isotherm results

Temperature (K)	Q_0 (mg/g)	B (L/mg)	R^2	R_L
305	57.143	0.093	0.9809	0.32
315	59.524	0.114	0.9907	0.28
325	61.728	0.150	0.9948	0.23
335	63.694	0.224	0.9960	0.17

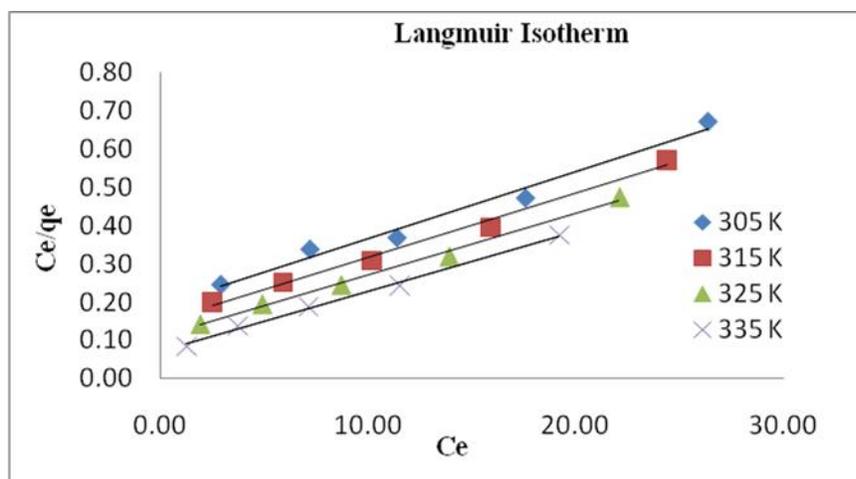


Figure 3 Langmuir Isotherm

4.2.2 Freundlich Isotherm

The Freundlich adsorption capacity constant K_f (mg/g) values ranged from 6.8234 to 14.0929 mg/g. The

magnitude of 'n' reveals the favorability of the adsorption. The values of $0 < 1/n < 1$ represent favourable adsorption conditions [25].

Table 2 Freundlich isotherm results for the adsorption of Cr(VI) ion onto PTMAC

Temperature (K)	1/n	K_f (mg/g)	R^2
305	0.5723	6.8234	0.9634
315	0.5559	8.1846	0.9601
325	0.5249	10.3514	0.9645
335	0.4720	14.0929	0.9770

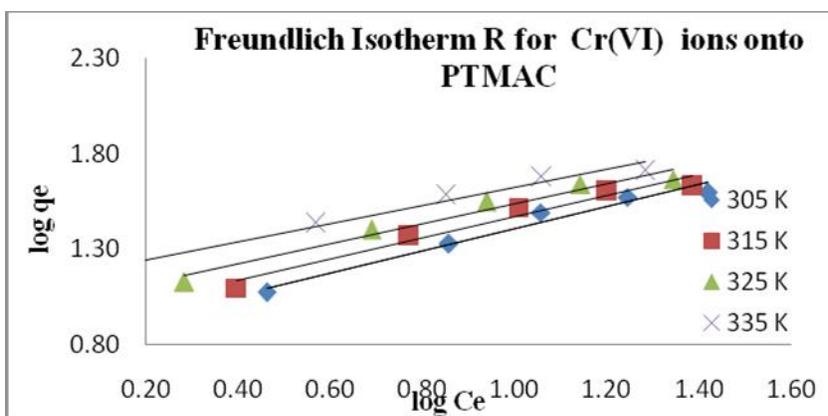


Figure 4 Freundlich Isotherm

4.2.3 Temkin Isotherm

Equilibrium binding constant a_T values (L/g) ranged from 0.3319 to 64.2190 L/g and the heat of sorption constant b_T values ranged from 190.4160 J/mg to

199.2838 J/mg for the three studied temperatures viz. 305, 315, 325 and 335 K. The lower values of a_T and b_T with respect to adsorption of Cr (VI) ion adsorption indicate physisorption rather than chemisorption.

Table 3 Temkin isotherm results for the adsorption of Cr (VI) ion onto PTMAC.

Temperature (K)	b_T (J/mg)	a_T (L/g)	R^2
305	190.4	0.33	0.9761
315	187.7	0.90	0.9872
325	189.6	4.35	0.9875
335	199.2	64.21	0.9869

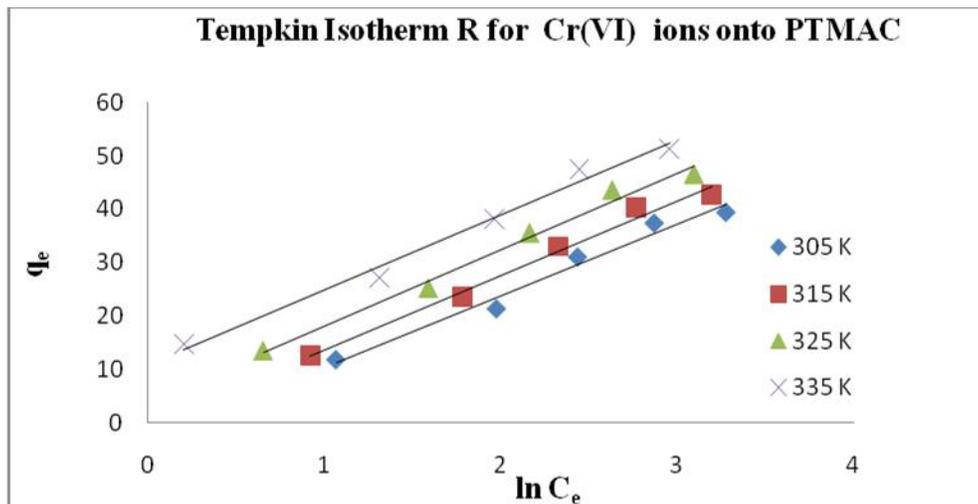


Figure 5 Temkin Isotherm

4.2.4 D-R Isotherm

Results of D-R isotherm are collected in Table 4. 'E' is a parameter used in predicting the type of adsorption. An E value less than 8 kJ/mol is an indication of physisorption. The mono layer adsorption

capacity q_D values (mg/g) are ranged from 82.2041 to 87.7238 mg/g for all the studied temperatures. Further it is noticed that adsorption capacity increased with the increase of temperature. The very low value of E infers the physisorption interaction.

Table 4 D-R isotherm results for the adsorption of Cr(VI) ion onto PTMAC

Temperature (K)	q_d (mg/g)	E (kJ/mol)	R^2
305	82.2	0.3536	0.9163
315	85.5	0.4082	0.9299
325	87.4	0.5000	0.9185
335	87.7	0.2673	0.8953

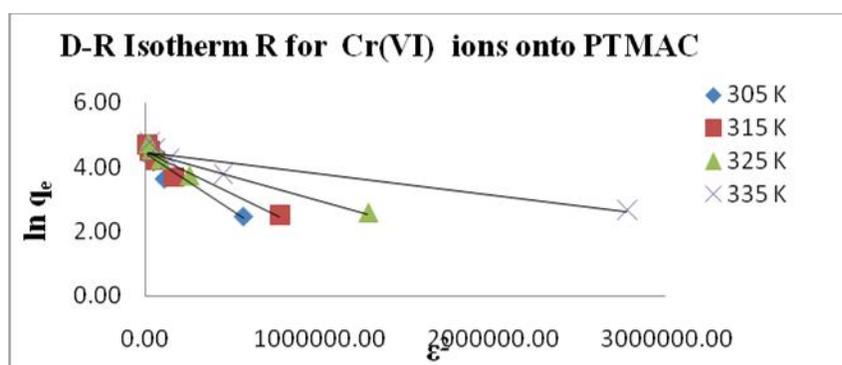


Figure 6 D-R Isotherm

4.3 Kinetic study

Several adsorption kinetic models have been established to understand the adsorption kinetics and rate-limiting step. These include pseudo-first and second-order and Weber Morris sorption kinetic models [26].

The pseudo first order rate constant, k_1 (min^{-1}) ranged from 0.0311 to 0.0486 min^{-1} . The pseudo first order theoretical adsorption capacity (q_e cal) values, obtained from the intercept of the linear plots, were compared with the experimental adsorption capacity (q_e exp) values. The initial sorption rate 'h' increased directly with the increase of initial Cr(VI) ion concentration (3.50– 5.75 $\text{mg/g}\cdot\text{min}^{-1}$), while an

inverse relationship exists between the overall sorption rates and initial Cr(VI) ion concentrations.

Results obtained in the two kinetic models are presented in Table 5. Pseudo second order kinetic model seems to best describe the above adsorption system as the R^2 value are very close to unity. Moreover, the difference between calculated adsorption capacity (q_e cal) and experimental adsorption capacity (q_e (exp)) values of second order is little when compared to the first order kinetic model. Statistically it is tested with a tool 'Sum of error squares' (SSE %) [27]. Low SSE value of pseudo second order kinetic model infers that this model is more appropriate.

Table 5 Kinetics results

Concentration mg/L	First Order				Second Order			
	$q_{e(\text{Cal})-I}$ (mg/g)	k_1 (min^{-1})	R^2	SSE %	$q_{e(\text{cal})-II}$ (mg/g)	k_2 (g/mg.min)	R^2	SSE %
10	7.3	0.0486	0.9794	4.57	12.0	0.0243	0.9998	0.28
20	15.6	0.0368	0.9719		21.7	0.0087	0.9998	
30	20.9	0.0373	0.9814		31.8	0.0051	0.9996	
40	24.6	0.0352	0.9761		38.3	0.0042	0.9996	
50	24.8	0.0311	0.9620		40.4	0.0035	0.9995	

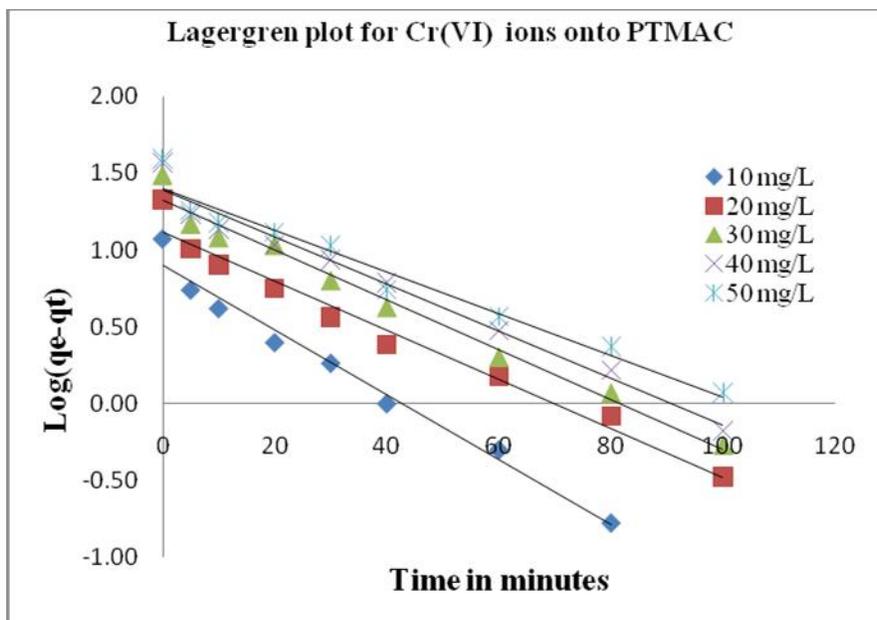


Figure 7 Lagergren plot

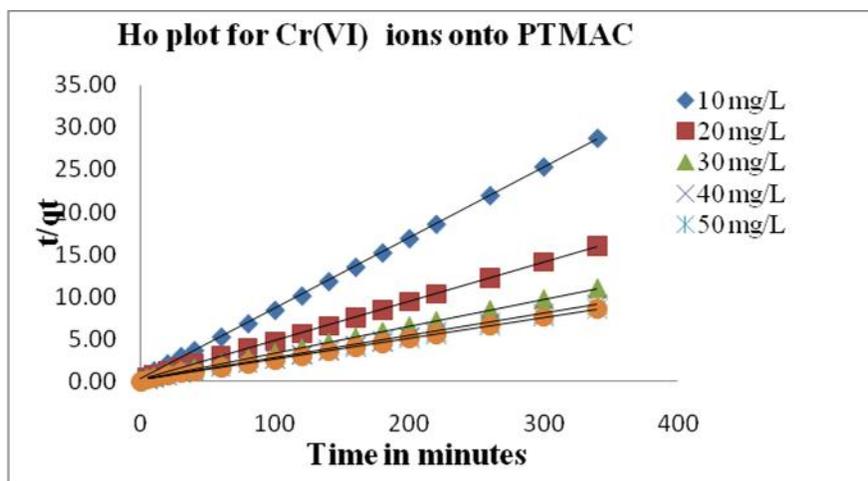


Figure 8 Ho plot

4.3.1 Intra particle diffusion

The k_p values were found to increase with an increase of Cr (VI) ion concentration which reveals that the rate

of adsorption governed by the diffusion of Cr (VI) ions within the pores of the adsorbent. [28].

Table 6 Intra Particle Diffusion results

Concentration mg/L	k_p (mg/g.min)	h	R^2
10	0.28	3.50	0.9767
20	0.47	4.15	0.9906
30	0.62	5.13	0.9974
40	0.94	6.10	0.9906
50	1.14	5.75	0.9995

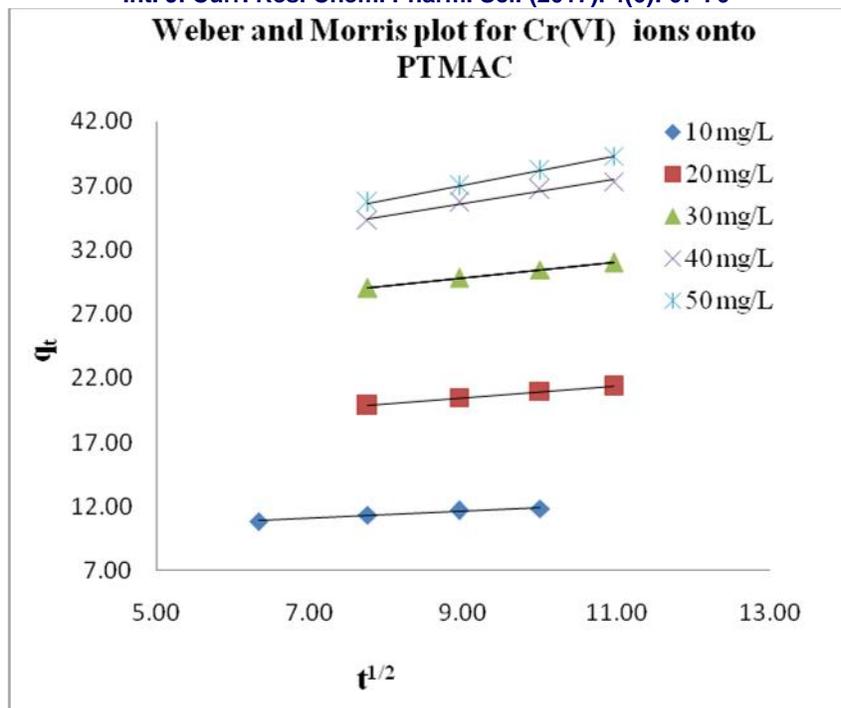


Figure 9 Weber and Morris plot

5. Conclusion

This study shows that Phosphoric Acid Treated *Mimosops elengi* Activated Carbon (PTMAC) was an effective adsorbent for the removal of Cr(VI) ion from aqueous solution. Adsorption of Cr(VI) ion was highly pH dependent and the results showed that the optimum pH for the removal was found to be 2.0, at which Cr(VI) exists as the most easily absorbable form. Equilibrium adsorption was achieved around 80 minutes for the dosage of 30 mg/50 mL of solution at room temperature of 305 K for the initial concentration of Chromium (VI) solutions ranging from 10 to 50 mg/L. The equilibrium parameter R_L values obtained from Langmuir isotherm study were in between 0 and 1 showing the favourable adsorption process. The values of 'n' the intensity of adsorption, obtained from Freundlich isotherm were found to be greater than one indicating a favourable adsorption. Values obtained for other parameters from Freundlich, Temkin and Dubinin-Radushkevich isotherms revealed the possibility of multi-layer, physisorption and heterogeneous pore distribution. The statistical tool 'Summation of error square revealed that present adsorbent – adsorbate system followed pseudo second order kinetics. The variation of intra particle diffusion constant k_p values with the initial adsorbate concentrations indicates that the intra-particle diffusion limits the rate of the process.

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