

Characterization and Application of Powder Activated Carbon from Water Hyacinth as Adsorbent for removal of Cr (VI) from Aqueous Solution

Teshibelay A., Dr. K.R. Aswin S.

Abstract: Hexavalent chromium, Cr (VI), is a toxic metal present in industrial effluents. The present study focuses on adsorption of Cr (VI) from aqueous solution on activated carbon prepared from water hyacinth shoot. The shoots taken from hyacinth was washed with distilled water and then it was dried. The dried sample were crushed into powder form in a circular saw mill. The powder thus obtained was activated with 10% of H₂SO₄ and carbonized at 500 ° C for 30 minutes in an electric furnace. The characteristics such as functional groups and surface morphology of activated carbon were analyzed using FTIR spectroscopy and SEM analysis respectively. From the characterization studies WHAC was assigned to the O–H stretching mode of hydroxyl groups and from the SEM studies it was observed that surface was highly porous and acidic. The Cr (VI) concentration was measured using UV-V Spectrophotometry.

Batch Batch adsorption experiments were performed to examine the effects of contact time, adsorbent dose, pH of the simulated solution, Cr (VI) concentration, agitation speed and temperature. Results show the maximum adsorption capacity of Cr (VI) was found to be 95% at pH 2, 1hr contact time, 40 mg/L Cr (VI) initial concentration, 1.4g/L adsorbent dose and temperature 30 ° C. The Langmuir and Freundlich adsorption isotherm models were used to fit the experimental data. The Langmuir adsorption isotherm best fit the equilibrium data and a pseudo-second order model represented the adsorption kinetics.

A column study was conducted for 30 minutes with 2 minutes interval using a Cr (VI) solution 20 mg/L, an influent pH of 5.53 and a depth of 5cm. From the result shown that Yoon Nelson model better than Thomas model at constant bed height and various flow rate. Therefore, WHAC can be

an effective adsorbent for Cr (VI) from the aqueous solution.

Keyword: Activated Carbon, Adsorption, Adsorption Isotherm, Adsorption Kinetic, Low Cost adsorbent and Hexavalent Chromium.

1. Introduction

Industries such as leather tanning, Ferrochromium production, Chromium plating and steel production are high consuming of chrome [1] and play role in discharging chromium into the environment. Cr (VI) is the main chromium species used in industrial processes [2]. It is one of the most dangerous form of chromium compounds and Carcinogens lists Cr (VI) compounds as known human carcinogens [3] like kidney and liver damage [4]. According to the WHO, the tolerance limit for Cr (VI) for discharge into surface water bodies is 0.05 mg/L and its concentration in industrial wastewaters especially developing countries varies from 0.5 to 270 mg/L [5-7]. So, it is obligatory to treat the Cr (VI) from wastewaters. Several chemical and physical methods have been developed to treat chromium bearing effluents to bring its level down to the permissible effluent standards [8-12].

Many numbers researchers have done to use conventional and unconventional adsorbents including carbonized powder biomass and un-carbonized powder biomass of activated carbon for removing Cr (VI) from wastewater. By various unconventional adsorbents, researchers used for removing Cr (VI) from real and artificial wastewaters (Table 1). Recently, activated carbon is effective to substitute convectional method by prepared from agro-wastes commercially obtainable for removal of Cr (VI) using modified activated carbon [13-19]. Similarly, activated carbon can be prepared water hyacinth by save woody plants [20]. This study indicates the application of activated carbon of water hyacinth for removal of Cr (VI) as low-cost adsorbent.

Water hyacinth (*Eichhornia crassipes*) is a floating macrophyte whose requirement for nutrients and explosive growth rate has been put to use in cleaning up municipal and agriculture wastewater. It is available almost through the world and high potential substrate for production cellulose [26]. From above case, an initiated to study the WHAC with the objective to use it as adsorbent for removing Cr (VI). The results of characterization of an adsorbent and batch adsorption experiments such as contact time,

initial concentration, adsorbent dosage, pH and temperature are obtained in this paper.

Table1: Summary of Activated Carbon Adsorbents used for Cr (VI) Removal Aqueous Solution

Adsorbent	pH	Optimal Removal	Reference
Palm date pits	4.5-6.5	65%	[20]
Yam Peel	4	80 %	[21]
Rice Husk	8	96%	[22]
Flamboyan t pod	6	99%	[23]
Peanut shell	2	90%	[24]
Rubber tires	4-6	90%	[25]
Water hyacinth	2	95%	Present study

2. Material and Method

2.1 Preparation of Adsorbent

Water Hyacinth were collected from a district Korattur, Chennai, India. The shoots were washed with distilled water and then it was dried in sunlight for 24hr. The dried sample was carbonized at 500 °C for 30 minutes in an electric furnace and crushed into powder form in a circular saw

mill. The crushed powder was sieved with particle size (150µm) and then powder sample was activated with 10% of H₂SO₄. The activated sample was washed with distilled water to a pH of 6-7, and then the sample was dried at 105° C for three hours with Hot air oven. The final activated carbon (WHAC) was kept in an air tight polyethylene bag to ready for use.

2.2 Characterization of Adsorbent

The WHAC was characterized in terms of functional groups and surface morphology. The presence of various functional groups presents on the surface of the adsorbent and their role in adsorption was analyzed using FTIR spectrum within the range of 400–4000 cm⁻¹. The surface morphology of WHAC was investigated using SEM to study the surface texture and the porosity of the WHAC.

2.3 Preparation of Adsorbate

Stock solution of hexavalent chromium (1000mg/L) was prepared by dissolving 2.875g potassium dichromate, K₂Cr₂O₇ (Lab chemicals wholesaler, Chennai) in 1L of deionized distilled water [27]. Desired concentration 50mg/L of Cr (VI) for experimental purpose was prepared from stock solution. Solution pH was

adjusted using 0.01-1M of HCl and /or NaOH.

2.4 Batch Adsorption Experiments

Batch adsorption experiments for adsorption of Cr (VI) on prepared adsorbent were conducted using aqueous solutions. For each run, a definite amount of adsorbent was added to 50 ml of Cr (VI) solution taken in 100 mL Erlenmeyer flasks. All the adsorption experiments were carried out at constant agitation speed of 120rpm in an incubator shaker. The adsorbate was decanted and separated from the adsorbent using filter paper (Whatman No-1). Final residual metal concentration after adsorption was measured by UV-Visible Spectrophotometer at 540 nm. To estimate the percentage adsorption of Cr (VI) from aqueous solution equation (1) was used. The metal uptake q_e (mg/g) at equilibrium time was calculated from the following equation (2).

$$\text{Adsorption (\%)} \text{ of Cr(VI)} = \frac{C_0 - C_e}{C_0} * 100 \dots\dots\dots (1)$$

$$q_e = \frac{(C_0 - C_e)V}{w} \dots\dots\dots (2)$$

Where, C_0 , C_e , v and w are the concentrations of Cr (VI) at the beginning and final concentration(mg/L), volume of

aqueous solution (mL) and adsorbent weight (g) respectively.

The effect of the contact time on the adsorption was studied by varying the time from 10 to 120min. The quantity of adsorbent needed for the study for the removal of chromium was tested between the range 0.2 to 0.12g/L by using 40mg/L initial Cr (VI), pH 5 and temperature at 30 ° C. The maximum adsorption time was taken after experiment and it was taken as equilibrium time in all experiments. The effect of pH was examined by performing adsorption experiments at various pH (2 to 10) using 40mg/L initial Cr (VI) with 1.4g/L adsorbent dose and temperature at 30 ° C. Effect of Cr (VI) concentration was examined at various with adsorbent dose of 1.4g/L at pH 2 and temperature of 30 ° C. Similarly, the effect of temperature was studied by performing adsorption experiments at temperature ranges 10 to 50 ° C using 40mg/L initial Cr (VI), adsorbent dosage 1.4g/L, pH 2 and at equilibrium time.

3. Result and Discussion

3.1 Characterization of activated carbon

Surface functional groups of activated carbon were determined by FTIR spectra at room temperature. The % of

transmission of samples was recorded over 400 – 4000 cm^{-1} (Figure 1). This spectra show a wide transmittance band at 3726.76 - 3000.69 cm^{-1} with a maximum at 3413.39 cm^{-1} was assigned hydroxyl groups [28]. The band around 3580.2 – 3558.99 cm^{-1} can be assigned to the C-H stretching and double bond regions at peak 1617.02 cm^{-1} . In addition N-H and CH₃ bend was observed at 1617 cm^{-1} for amines and at 1409.71 cm^{-1} for alkanes respectively[29]. The peak bands around 1487.82 cm^{-1} and 441.62 cm^{-1} show the hydrocarbons on the surface [30].

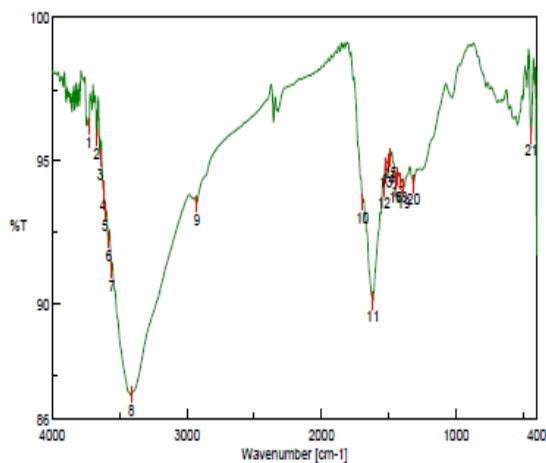


Figure 1: FTIR spectra of WHAC

The SEM micrograph of WHAC adsorption is shown in Figure 2. It is seen that WHAC has a highly porous structure with greater homogeneity and average pore diameter of 52.5 μm . Activated carbons produced at 77.25% have cavities. EDAX spectra of bounded adsorbent indicates of

metal adsorption has occurred on the adsorbent surface (Figure 3) which was calcium most abundant in WHAC. However, the carbon content has most dominated from other metals and non-metals.

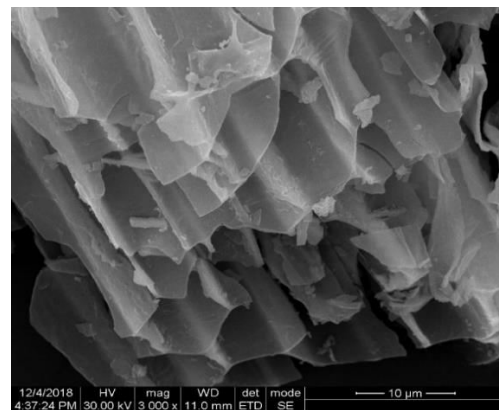


Figure 2. SEM micrograph of WHAC

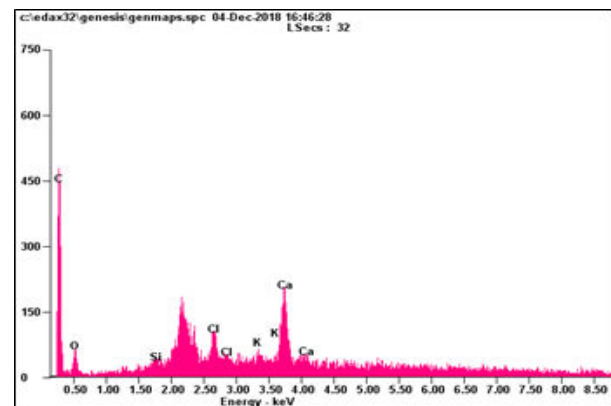


Figure 3. EDAX spectra of WHAC

3.2 Effect of contact time

Fast uptake of equilibrium time suggests the efficiency of particular adsorbent in terms of usage in wastewater treatment. Figure 4 shows the effects of contact time and it was observed that the adsorption rate was fast at the initial stages

and then gradually decreased. The maximum removal efficiency was found to be 89.5% at contact time of 60 min and the adsorption phase reached to equilibrium. Therefore, the contact time is decided 60 min for all experiments.

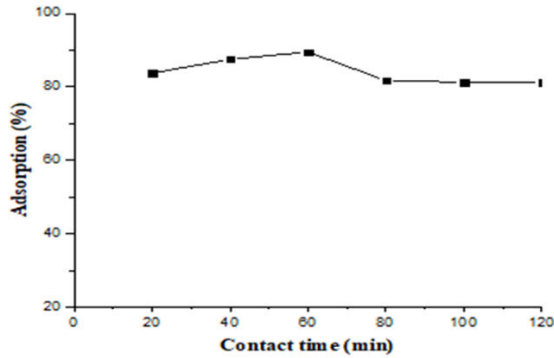


Figure 4: Effect of Contact Time

(dosage: 1.2g/L, initial concentration: 40mg/L, PH: 5, Temp.: 30 °C)

3.3 Effect of adsorbent dosage

The effect of adsorbent dose on adsorption of Cr (VI) using WHAC was illustrated in figure 5. The results show that the adsorption has increased from 45 to 81% that can be directly linked to the accessibility of increasing adsorption places with increasing amount of adsorbent. At increase of adsorbent dose, there is no enough occupy active sites and then adsorption to reached at constant. In all other experiments to the adsorbent dose was taken an average of 1.4g/L.

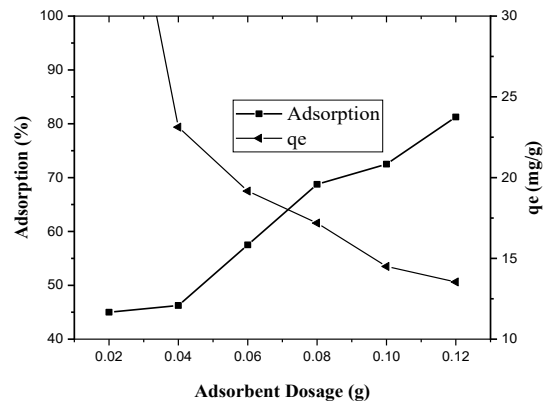


Figure 5: Effect of Adsorbent Dosage

(Contact time: 60min, initial concentration: 40mg/L, PH: 5, Temp.: 30 °C)

3.4 pH of the Simulated Solution

Figure 6 show the removal of Cr (VI) pH 2.0 to 10.0. The maximum percentage adsorption was found at acidic pH 2.0, reaching maximum of about 95 %. The adsorption capacity of WHAC for Cr (VI) at pH 2.0 was 95% mg/g, which was reduced to 17.50 mg/g with increase of pH to 10.0.

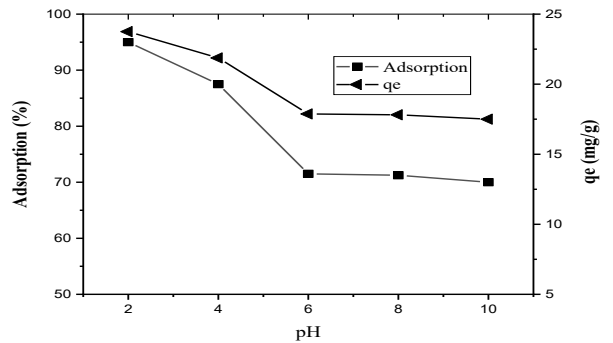


Figure 6: Effect of pH

(Contact time: 60min, initial concentration: 40mg/L, dosage: 1.4g/L, Temp.: 30 °C)

3.5 Effect of Cr (VI) Concentration

Figure 7 depicts the changes in adsorption trends of ACs with variation in initial concentration of adsorbate in the solution. AC prepared by H₂SO₄ activation (WHAC) showed rapid adsorption of Cr (VI) until first 40mg/L increased and it was decreased gradually. It indicated that the presence of adsorbent and adsorbate were highly reached adsorption sites on the surface of WHAC.

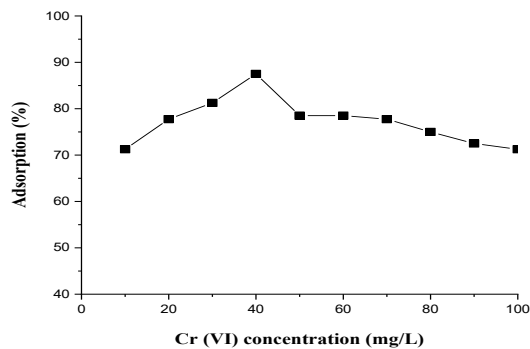


Figure 7: Effect of initial concentration
(Contact time: 60min, dosage: 1.4g/L, PH: 2, temp.: 30 °C)

3.6 Effect of temperature

Effect of temperature on Cr (VI) adsorption on WHAC was shown in Figure 8. Adsorption of Cr (VI) on all the adsorbents witnessed the increase in adsorption percentage and adsorption capacity (q_e) from 10 to 50 °C and no change with further decrease after 50 °C

temperature. The adsorption of Cr (VI) on WHAC increased from 62 to 76%, and with the rise in temperature from 10 to 50 °C.

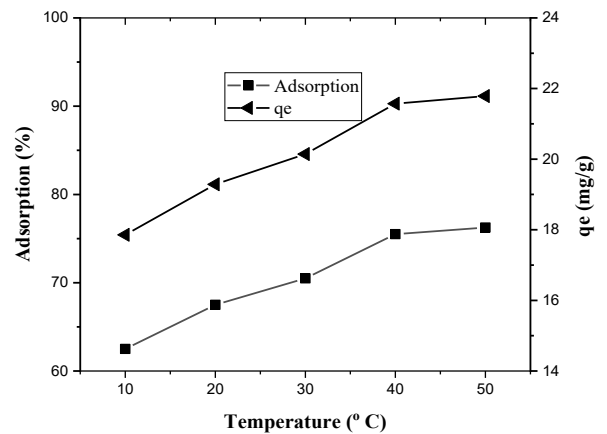


Figure 8: Effect of temperature
(Contact time: 60min, Initial concentration: 40mg/L, pH: 2, Dosage: 1.4g/L)

3.7 Adsorption of Isotherm Study

In this project Langmuir and Freundlich isotherms were applied to investigate the adsorption process of Cr (VI) on prepared WHAC at temperature ranges 10 to 50 °C (Figure 9 and Figure 10). From Langmuir isotherm the equilibrium parameter R_L which was found between ($0 < R_L < 1$) and others parameters represented in table 2. This value indicated that Langmuir isotherm give a best fit. But, Freundlich isotherm model was not fit by $1/n$ less than zero.

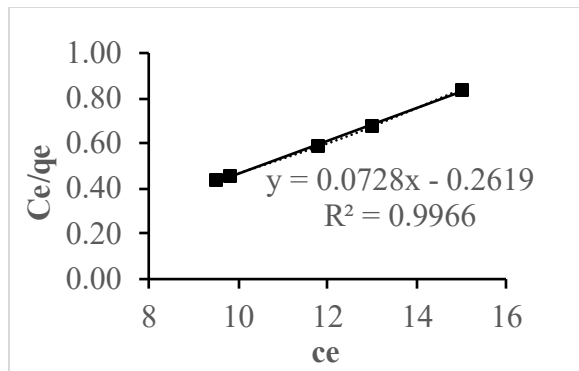


Figure 9: Langmuir Isotherm

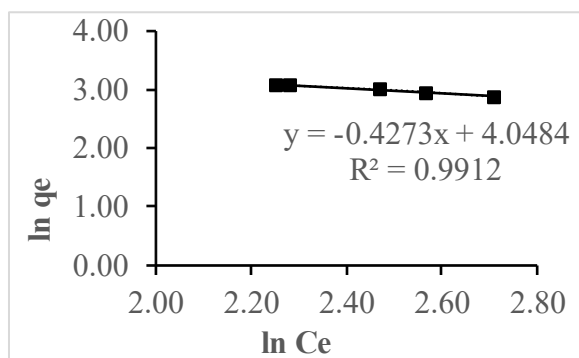


Figure 10: Freundlich Isotherm

Table 2: Langmuir and Freundlich Isotherm

Isotherms	Isotherm parameters		
	Q ₀ (mg/g)	Temp. (30 ° C)	Temp. (50 ° C)
Langmuir isotherm $\frac{C_e}{q_e} = \frac{1}{LQ_0} + \frac{q_e}{Q_0}$		17.513	13.736
	b	0.0855	0.006
	R _L	0.2262	0.288
	R ²	0.9947	0.9966
Freundlich isotherm $\ln q_{eq} = \ln K_F + (1/n)\ln C_{eq}$	K _f	28.216	38.813
	1/n	-0.2524	-0.4273
	R ²	0.9664	0.9912
	q _e (mg/g)	38.3	70.7

3.8 Kinetic Study

The linearized form of pseudo-first-order kinetic model equation and the

pseudo-second-order sorption kinetics equation can

be expressed (Table 3) respectively. The values of k₁ and q_e were calculated from the slopes and intercepts of log (q_e – q_t) versus ‘t’ plots (Figure 11). The value k₁ was obtained low and the graph was not linear. So that the adsorption of Cr (VI) on WHAC doesn't follow the pseudo first order kinetic model.

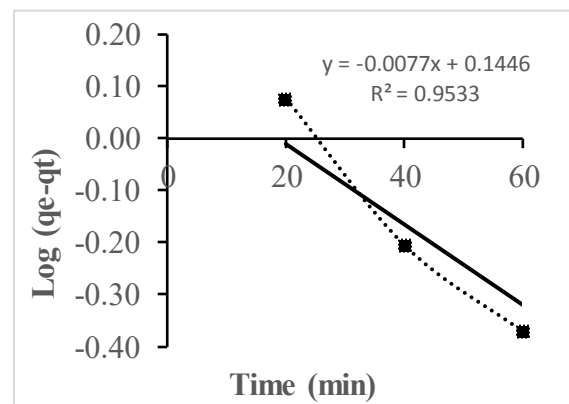


Figure 11: Pseudo First Order Model

By using the slope and intercept of the linear graph (Figure 12) the value of q_e, R² and K₂ were determined in table 3. From the result showed that WHAC in pseudo second order model has a very high regression coefficient.

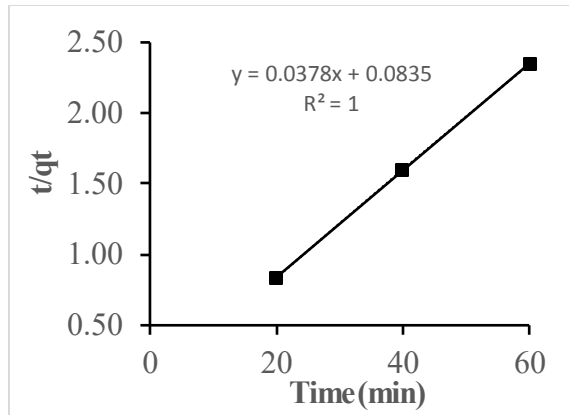


Figure 12: Pseudo Second Order Model

Table 3: Pseudo First Order and Pseudo Second Order parameters on WHAC

Kinetic model type	Kinetic parameters	
Pseudo First Order	K_1 (min^{-1})	0.000295
	R^2	0.9533
	q_e (mg/g)	25.57
Pseudo-Second Order	K_2 (min^{-1})	0.01916
	R^2	1.0
	q_e (mg/g)	26.45

3.9 Column Study

A column study was conducted for 30min using a Cr (VI) solution with an influent concentration of about 20mg/L, an influent pH of 5.53 and a depth of 5cm. The higher uptake of Cr (VI) ion was observed at the beginning of the fixed bed column and the the effluent rapidly increased after breakthrough volume (Figure 13).

The Thomas model was used in describing the adsorption kinetics and

evaluating the maximum solid phase concentration (q_e) and the Thomas rate constant (K). using values from the column experiments K_{Th} and q_e were calculated by plotting $\ln(C_o/C_{t-1})$ versus 't' (Figure 14). From the parameters and regression coefficient, it was determined that the experimental date fitted to the Thomas model (Table 4). The result that the Thomas model did not predict the initial part of the breakthrough well has also been testified.

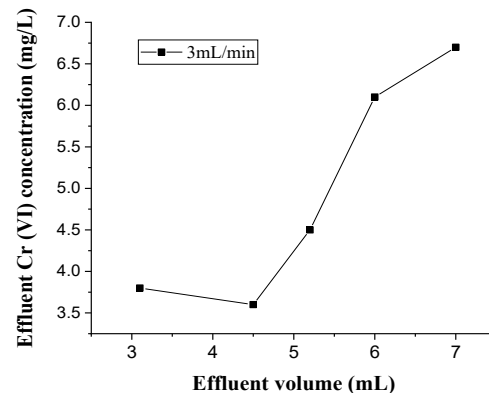


Figure 13: Breakthrough Curve

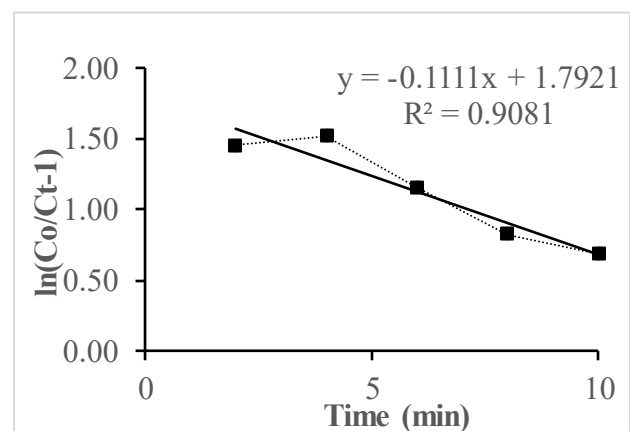


Figure 14: Experimental and Theoretical Breakthrough Curve form Thomas Model

The theoretical model developed by Yoon Nelson was used to investigate the breakthrough behavior of Chromium on the WHAC. The values of 'K_{Th}' and 'τ' were calculated from the plot graph ln (Ct/Co-Ct) versus 't' using values from column experiments at different time (Figure 15).

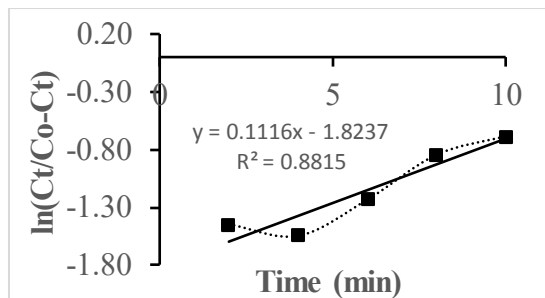


Figure 15: Experimental and Theoretical Breakthrough Curve form Yoon-Nelson Model

Table 4: Thomas Model and Yoon Nelson Model Constant Parameters at 3mL/min

Model type	Time (min)	constant	
		K _{Th} (mL/min.mg)	Q _e (mg/g)
Thomas Model	2	0.0448	1.859
	4	0.0224	3.719
	6	0.0164	5.080
	8	0.0112	7.439
	10	0.0089	9.299
Yoon Nelson model	Time (min)	K _{YN} (L/min)	τ (min)
	2	0.9044	0.123
	4	0.4522	0.245
	6	0.3015	0.368
	8	0.2261	0.491
	10	0.1809	0.614

The values of 'K_{Th}' and 'τ' were found to increase with increase in flow time and flow rate (Table 4). From the result shown that Yoon Nelson model fit the data and suitable.

4. Conclusion

This study paper indicated that the applicability WHAC as adsorbent for removal of Cr (VI) from aqueous solution. WHAC showed high porosity and had functional groups advantageous in adsorption. The maximum adsorption of Cr (VI) was found 95% at pH 2.0, contact time 60min and adsorbent dose 1.4g/l with room temperature. The Langmuir isotherm best fit the equilibrium data and a pseudo-second order model represented the adsorption data. From the column studies faster breakthrough curves were observed at 3mL/min. The flow rate was directly influenced by the accommodative capacity of the filtering media.

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AUTHORS PROFILE



Teshibelay Ashagre, PG Scholar, Department of Civil Engineering at Vel Tech Rangarajan Dr. Sagunthala R&D Institute of Science and Technology. He has three years Research experience at STIC, Ethiopia.



Dr. K.R. Aswin Sidhaarth, working as Associate Professor in Department of Civil Engineering, Vel Tech Rangarajan Dr. Sagunthala R&D Institute of Science and Technology, Chennai-600062, India. He completed his Ph.D. in Adsorption of Lead, Zinc and Congo Red Dye from Aqueous Solution Using Cobalt Ferrite and Manganese Ferrite Nanoparticles in Anna University. He has published above 8 research papers in reputed journals.