

# Removal of Chromium (VI) metal ions from waste water using alternative adsorbents - A comparative study

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**Abstract-** Alternative adsorbents like Activated charcoal, modified activated charcoal, wheat bran, activated neem leaf powder and groundnut shell powder were used as adsorbents for effective removal of Cr (VI) ions and thus a comparative study between the adsorbents are presented. Waste water containing Cr (VI) ions was treated with all adsorbents for a calculated amount of time. Further, filtration was carried out and was analysed for Cr (VI) metal ions using Spectrophotometric method. By varying the concentration of the solution and studying its effects we have selected the best result obtained for percentage removal of Chromium for the different adsorbents. Other parameters like time of contact, amount of adsorbent used and pH were also varied and the required result i.e. percentage removal of Chromium is shown.

**Index Terms-** Chromium VI (Cr VI), Waste water, Adsorbents, Spectrophotometric method, metal ions.

## 1. INTRODUCTION

With water pollution increasing as a result of enhanced industrial activities, the problem of removing pollutants from wastewater is growing daily. Heavy metal pollution is of great concern, due to growing awareness of potentially hazardous effects of elevated levels of these materials in the environment. Toxic metals (i.e., Cr, Cu, Ni, Zn, Cd, Pb) make their way into water bodies via wastewater from metal plating industries, leather industries, pulp and paper mills, refineries and steel work foundries, etc.

The current study focuses on the removal of Cr (VI), heavy metal ions from industrial waste water using unconventional adsorbents. Groundnut shells and wheat bran are available abundantly as agricultural wastes. Neem leaves are available as a natural adsorbent along with activated charcoal.

The advantages of non conventional adsorbents are as follows:

- (1) The efficiencies of various non-conventional adsorbents towards adsorbate removal vary generally between 50% and 90% depending on the characteristics and particle size of the adsorbent, and the characteristics and concentration of the adsorbate, etc. Hence, alternate adsorbents can be employed efficiently in removal of heavy metals.
- (2) Non-conventional adsorbents are much cheaper relative to conventional adsorbents, and when readily available locally which leads to much reduced transportation costs.
- (3) Non-conventional adsorbents require simple alkali/and or acid treatment for the removal of lignin before application in order to increase their efficiency.

(4) Non-conventional adsorbents require less maintenance and supervision. [13]

## 1.1 SOURCES OF CHROMIUM

Chromium emission sources can be divided into two broad classes, direct and indirect. The direct category primarily includes sources that either produce chromium or consume chromium or a chromium compound to manufacture a product.

The sources categories within the direct category are [3]:

Chromites ore refining	60%
Ferrochromium production	71%
Refractory production	3%
Chromium chemicals	17-18%
Chromium plating	70%
Steel production	12-28%
Leather tanning	90%

## 2. MATERIALS AND METHODS

### 2.1. REAGENTS AND APPARATUS

All the primary chemicals used were of analytical grade.

Potassium dichromate, caustic soda, hydrochloric acid solution and other necessary chemicals were obtained from the college laboratory.

A standard solution of 1000 ppm of  $K_2Cr_2O_7$  was prepared as the stock solution. This was further diluted to certain concentrations ranging from 50 mg/L to 300 mg/L. The adsorbents used were modified activated charcoal, wheat bran, groundnut shell powder, neem leaf powder, modified neem leaf powder. Conical flasks were used for the preparation and storage of different concentration solutions. Appropriate amount of adsorbents were weighed and added to each conical flask for shaking in a rotary shaker for a given contact time. Spectroscopic analysis was then conducted to get the final concentrations of the solutions. The percentage removal of Cr (VI) metal ions was thus obtained.

### 2.2. EXPERIMENTAL SETUP:

The equipments used were rotary shaker, pH meter, UV visible spectrophotometer. HACH-DR-4000 UV Visible spectrophotometer was used for determination of chromium content in standard and treated solutions. The pH of the solution was measured with a EUTECH make digital microprocessor based pH meter previously calibrated with standard buffer solutions. The particle size distribution analysis was carried out with the different adsorbents using a particle size distribution analyzer. The chemical analysis was carried out by standard methods of chemical analysis.

**UV spectroscopy:**

The solutions were analysed for absorbance at a wavelength of 540 nm.



Fig 1: UV Spectrophotometer

**Rotary Shaker:**

Rotary shaker was applied for shaking to all the solutions in conical flasks for a constant time. The contents of the flask were shaken at a constant value of 120 rotations per min.



Fig 2: Rotary Shake

**2.3 PROCEDURE:**

Preparation of standard solution (stock solution)

The stock solution containing 1000 ppm of Cr(VI) was prepared by dissolving a known quantity of  $K_2Cr_2O_7$  in 500 ml of deionised, double distilled water.

**Activated charcoal** was obtained from the laboratory and it was used directly in the powdered form.

The properties of this activated charcoal were then modified. It was oxidised with 0.1 N  $HNO_3$ . 10 ml of this acid was mixed with 0.2 g of activated charcoal, soaked for 2 hours and sun dried for 6 hrs. This sample was again mixed with a known quantity of NaOH and NaCl for 8 hrs. Thus, the activated charcoal was surface modified and used for the adsorption.

**Neem powder** was collected from local markets. After collection it was washed thoroughly with double distilled water to remove water soluble impurities and then sun dried. This was then ground to pass through 15-20 mesh screens. This was then treated with HCl in the ratio 1:1.8 by weight and dried in the sun for 24 hrs. This was then washed with distilled water to remove the free acid content and then dried in an oven at 400 °C for 4 hrs. To this 100 ml solution, 100 mmol/l copper solution (initial pH 8.5) was added 10 g of treated Neem leaves. The mixture was shaken for 6 hrs at 26±20 °C. [16]

**Groundnut shells** were obtained from local markets and were washed with tap water. They were then sun dried and ground to 200-300 micrometer size. 20g of the powder was soaked for 24 hours in 0.1M concentrated  $HNO_3$ . This mixture was filtered using three layers of filter paper. The residue was washed with distilled water to remove the acid content and dried at room temperature for 1 hour. It was then dried in an oven at 420 °C for 5 hours. [16]

**Wheat bran** was obtained locally and ground to 200-300 micrometer. It was washed with distilled water and dried for 6 hours under the sun.

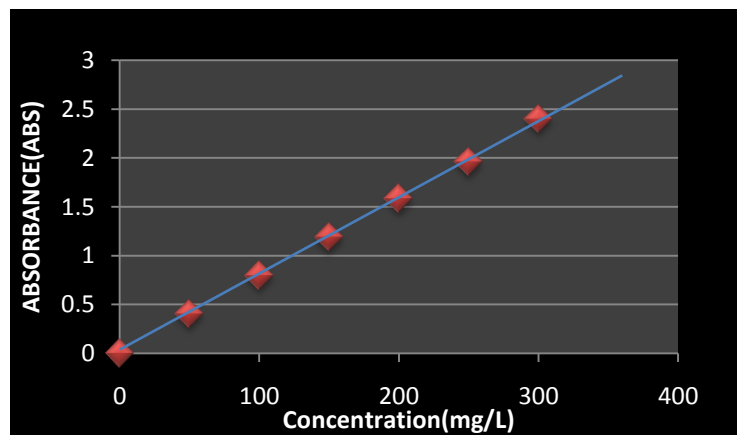
**2.4 CALCULATIONS:**

Using the calibration chart the corresponding values of concentration were obtained.

The absorbance readings were obtained at 540nm and they are noted as shown in the table below:

Table 2.1 Absorbance readings

SL. NO.	Initial Concentration of Cr(VI), mg/L	Absorbance (ABS)
1.	50	0.41
2.	100	0.8
3.	150	1.19
4.	200	1.58
5.	250	1.96
6.	300	2.40



Graph 2.1: Absorbance vs Concentration of Chromium (VI)

**2.4.1 BATCH ADSORPTION STUDIES:**

All experiments were carried out at room temperature, i.e., 30±20 C. Using the 250 ml stopper conical flask containing 50ml of test solution batch adsorption studies were carried out at the desired pH value, contact time and adsorbent dosage level. pH of the solution was monitored by adding 0.1M HCl and 0.1M NaOH solution as required. Necessary amount of adsorbent material was then added and contents in the flask were shaken for the desired contact time in a rotary shaker working at 120 rotations per minute. The time required for reaching the equilibrium condition estimated by drawing samples at regular intervals of time till equilibrium was reached. The contents of the flask were filtered using three layers of filter paper and the filtrate was analysed for remaining Cr(VI) concentration in the sample using the UV Visible Spectrophotometer with 1,5-

diphenylcarbazide in acid medium by following APHA, AWWA standard methods for examination of water and wastewater. The amount of Cr (VI) adsorbed per unit mass of the adsorbent was evaluated by using the following mass balance equation,

$$q = (C_o - C_f) v/w$$

The percent removal of Cr (VI) was calculated as follows:

$$\text{Percentage removal of Cr (VI)} = ((C_{\text{initial}} - C_{\text{final}})/C_{\text{initial}}) \times 100$$

Where,  $q \Rightarrow$  Adsorption capacity (mg/g)

$C_o \Rightarrow$  initial concentration (mg/L)

$C_f \Rightarrow$  final concentration (mg/L)

$v \Rightarrow$  volume of the solution (L)

$w \Rightarrow$  weight of the adsorbent (g)

Effect on variation of initial concentrations was studied for all the adsorbents, with the variation of Chromium (VI) ion from 50mg/L to 300mg/L with an adsorbent dosage of 4g/L and shaking time of 60 minutes. Further parameters were varied for the adsorbent showing the best removal percentage of Chromium (VI) under the given conditions.

The effect of adsorbent dosage level on percent removal of chromium was studied using Chromium (VI) concentration of 300 mg/L having adjusted the pH to 2. The selected adsorbent concentration was varied from 4g/L to 20g/L.

Adsorption experiments for the effect of pH were conducted by using a solution having concentration 200mg/L of Chromium (VI) with an adsorbent dosage of 4g/L and shaking time of 60 minutes.

### 2.5 PARAMETERS STUDY:

Various parameters affecting the removal of chromium (VI) from waste water is studied. The parameters include concentration of Cr (VI) solution, Adsorbent dosage, contact time and the pH. The effect of all these parameters on the adsorptive capacity of the different adsorbents used is studied.

#### 2.5.1 CONCENTRATION OF CR (VI) SOLUTION:

50 ml solutions were prepared of different concentrations by appropriate dilution of the standard solution. The concentrations taken were ranging from 50 mg/L to 300mg/L. The six different concentration solutions were prepared in 250 ml conical flasks. The selected adsorbents were used for different contact times to be shaken with the solution samples and the final concentration readings were taken to determine the percentage removal of Cr (VI).

#### 2.5.2 ADSORBENT DOSAGE:

50 ml of different concentration solutions were taken in 250ml conical flasks and different adsorbents were used for the adsorption process. The best adsorbent, modified activated charcoal was chosen and its amount was varied ranging from 2g/L to 20g/L. Experiments were carried at room temperature at

a constant pH determined experimentally. The results were obtained showing that with increase in the amount of adsorbents used, the adsorption increased initially. After a certain point the graph was constant meaning no further increase in adsorbent dosage had no effect on the removal of Cr (VI).

#### 2.5.3 CONTACT TIME:

Three concentrations were chosen for this experiment, 100mg/L, 200mg/L and 300mg/L. The contact time was varied from 5 min to 140 min keeping the pH value and temperature value constant. The results were obtained and it was seen that with the increase in contact time, i.e., the shaking time of the samples with the adsorbent, the adsorption also increases, till a certain time, after which the graph became constant.

#### 2.5.4 pH:

The concentration of Cr (VI) solution chosen for this experiment was 200mg/L. The pH was varied from 1 to 11 and the shaking time was taken to be 60 minutes. The results were thus obtained and it was found that maximum removal took place at a pH of 2.

## 3. RESULTS AND DISCUSSIONS

This chapter deals with the result analysis. A brief discussion on each of the results and the inferences drawn from them are presented. Graphical figures have been plotted to indicate the variations of the percentage removal. The best adsorbent is chosen on the basis of percentage removal by varying the concentrations of initial metal ions in the solutions. Different parameters such as time of contact, pH, and amount of adsorbent are varied for the best adsorbent. Tables show the observations recorded and the calculations performed.

### 3.1 EFFECT OF INITIAL METAL ION CONCENTRATION:

The efficiency of removal of Cr (VI) was affected by the initial metal ion concentration, with decreasing removal percentages as concentration increases from 50mg/L to 300mg/L at pH of 2-4, adsorbent dosage level of 4g/L and contact time of 60 minutes. At low metal ion is to adsorbent ratios, metal ion adsorption involves high energy sites. As this ratio increases the higher energy sites are saturated and adsorption begins on lower energy sites, resulting in decrease in the adsorption efficiency.

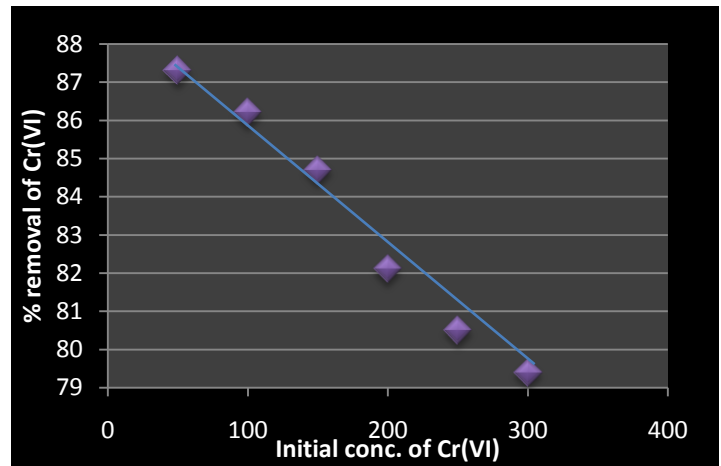
### OPERATING CONDITIONS

pH	2
Adsorbent dosage level	4g/L
Contact time	60 minutes
Temperature	32 °C(room temperature)

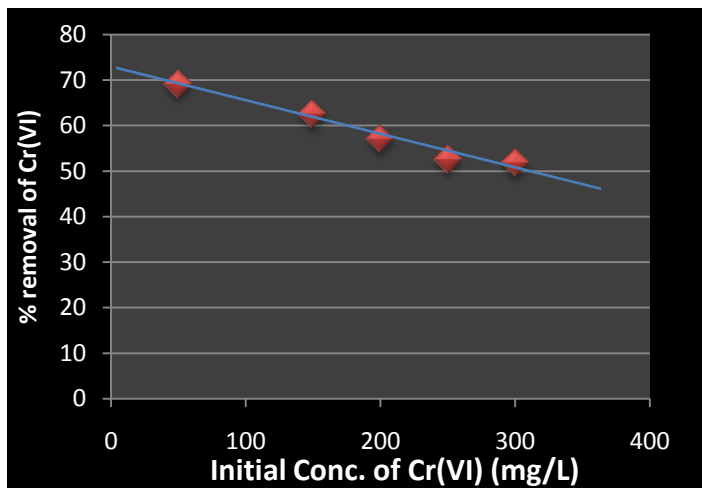
**GROUND NUT SHELL:**

Table 3.1: Effect of initial metal ion concentration, Ground nut shell

Sl. No.	Initial concentration (mg/L)	% Removal of Cr(VI)	Adsorptive capacity, q (mg/g)
1.	50	69	8.625
2.	150	62.5	5.462
3.	200	57	28.500
4.	250	52.6	32.875
5.	300	51.7	38.775



Graph 3.2: % removal of Cr (VI) vs Initial conc. of Cr (VI)



Graph 3.1: % removal of Cr (VI) vs Initial conc. of Cr (VI)

**MODIFIED POWDERED NEEM LEAVES**

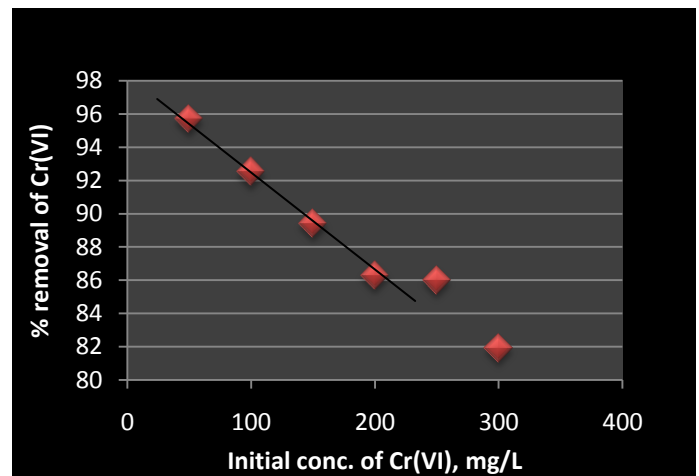
Table 3.3: Effect of initial metal ion concentration, modified powdered neem leaves

Sl. No.	Initial concentration (mg/L)	% Removal of Cr(VI)	Adsorptive capacity, q (mg/g)
1.	50	95.7	11.962
2.	100	92.5	23.125
3.	150	89.4	33.525
4.	200	86.3	43.150
5.	250	84.2	52.625
6.	300	81.9	61.425

**POWDERED NEEM LEAVES:**

Table3.2: Effect of initial metal ion concentration, neem leaves

Sl. No.	Initial Concentration (mg/L)	% Removal of Cr(VI)	Adsorptive capacity, q (mg/g)
1.	50	87.3	10.910
2.	100	86.2	21.550
3.	150	84.7	31.770
4.	200	82.1	41.050
5.	250	80.5	50.325
6.	300	79.4	59.550

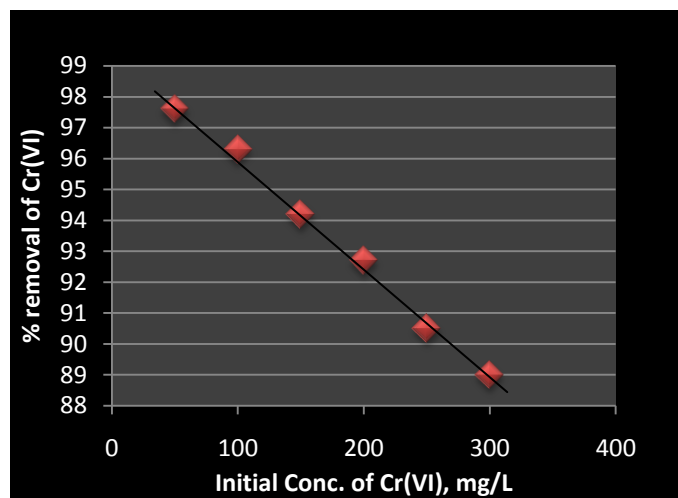


Graph 3.3: % removal of Cr (VI) vs Initial conc. of Cr (VI)

**WHEAT BRAN:**

Table 3.4: Effect of initial metal ion concentration, wheat bran

Sl. No.	Initial concentration (mg/L)	% Removal of Cr(VI)	Adsorptive capacity, q (mg/g)
1.	50	83.3	10.418
2.	100	81.2	20.312
3.	150	80.32	30.120
4.	200	79.5	39.750
5.	250	79.2	49.500
6.	300	78.6	59.110



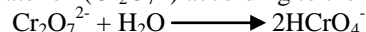
Graph 4.5: % removal of Cr (VI) vs Initial conc. of Cr (VI)

Based on the results, the best adsorbent under the given conditions was found to be modified activated charcoal. Thus further experimentation was done by varying various parameters and analysing their effects on the removal of Cr (VI).

**3.2 EFFECT OF INITIAL pH:**

The percentage of Cr (VI) adsorbed by modified activated charcoal decreased from 96.3% to 50.1% when the pH was increased from 1-7 and this value further decreased to 35.5% when the pH was further increased to 7-11. The maximum percent of removal of Cr (VI) was obtained between pH 1-3 at the initial concentration of metal ion at 100 mg/L, adsorbent was taken as 4g/L and shaking time of 60 minutes at room temperature of 28°C and thus pH of 2 was chosen for the rest of the experiments.

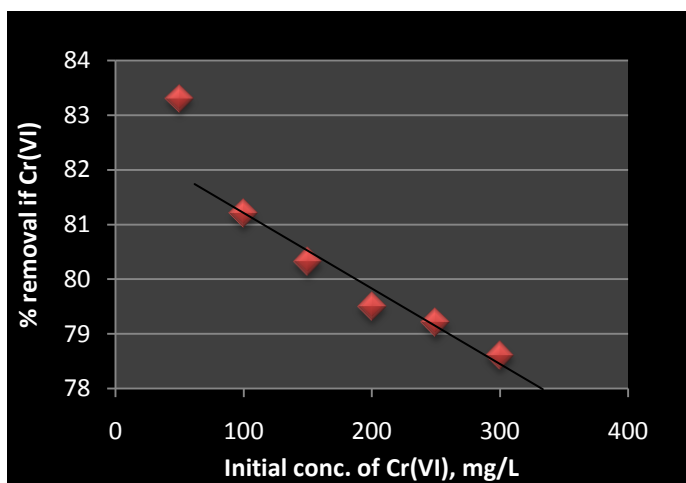
Chromium exists in two oxidation states which are Cr (VI) and Cr (III) and the stability of these forms is dependent on the pH of the system. It is well known that the dominant form of Cr (VI) at pH 2 is  $\text{HCrO}_4^-$  which arises from the hydrolysis reaction of the dichromate ion ( $\text{Cr}_2\text{O}_7^{2-}$ ) according to the equation-



Increasing the pH will shift the concentration of  $\text{HCrO}_4^-$  to  $\text{Cr}_2\text{O}_7^{2-}$  and other forms as  $\text{CrO}_4^{2-}$ . Maximum adsorption at pH of 2 indicates that it was the  $\text{HCrO}_4^-$  form of Cr (VI), which was the predominant species at this pH range and adsorbed preferentially on the adsorbents. Better adsorption capacity observed at low pH between 2 and 3 may be attributed to the large number of  $\text{H}^+$  ions present at these pH values, which in turn neutralize the negatively charged hydroxyl groups ( $-\text{OH}^-$ ) on adsorbed surface thus reducing the hindrance to the diffusion of dichromate ions. At higher pH values, the reduction in adsorption may be possible due to abundance of  $\text{OH}^-$  ions causing increased hindrance to diffusion of dichromate ions. [25]

**3.3 EFFECT OF ADSORBENT AMOUNT:**

When the amount of activated charcoal was varied from 2g/L to 20g/L at initial concentration of Cr (VI) at 300 mg/L, contact time of 60 minutes, pH of 2 was maintained and 50 ml. of sample was taken, the percentage of Cr (VI) removed increased from 70.1% to 97.44%.



Graph 3.4: % removal of Cr (VI) vs Initial conc. of Cr (VI)

**MODIFIED ACTIVATED CHARCOAL:**

Table 3.5: Effect of initial metal ion concentration, activated charcoal

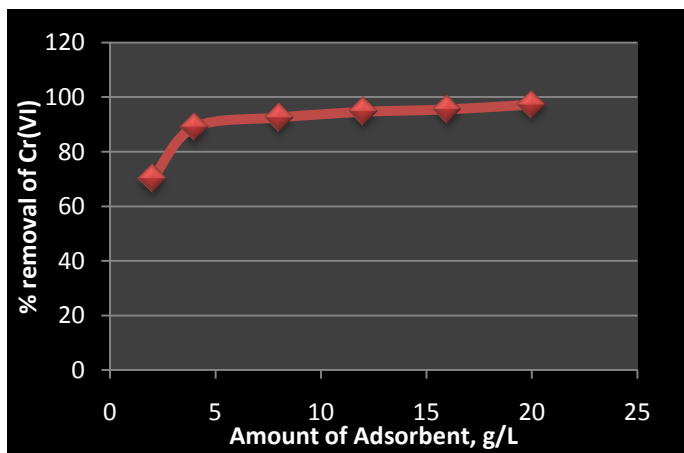
Sl. No.	Initial concentration (mg/L)	% Removal of Cr(VI)	Adsorptive capacity, q (mg/g)
1.	50	97.6	12.2
2.	100	96.3	24.075
3.	150	94.2	35.325
4.	200	92.7	46.350
5.	250	90.5	56.575
6.	300	89	66.750

**OPERATING CONDITIONS**

pH	2
Concentration	300mg/L
Time of contact	60 minutes
Temperature	32 °C(room temperature)

Table 3.7: Effect of amount of adsorbent used on percentage removal

Sl. No.	Amount of adsorbent (g/L)	% Removal of Cr(VI)	Adsorptive capacity, q (mg/g)
1.	2	70.1	89.98
2.	4	89	66.75
3.	8	92.6	34.72
4.	12	94.7	23.67
5.	16	95.6	18.05
6.	20	97.4	14.61



Graph 3.7 %removal of Cr (VI) vs Amount of adsorbent (g/L)

**3.4 EFFECT OF CONTACT TIME:**

The experimental runs measuring the effects of contact time on the batch adsorption of Cr (VI) was carried out at pH maintained at 2 and initial Cr (VI) concentrations of 100 mg/L, 200 mg/L and 300 mg/L.

Over the first 20 minutes the percentage removal of Cr (VI) from the solution increased rapidly and reached 82.4% for 300 mg/L and 90.3 % for 100 mg/L. After this the percentage removal tapered off until about 70- 80 minutes when it reached about 93% for the 300 mg/L and 96.8% for 100 mg/L solution. Further increase in contact time had a negligible effect on contact time. Thus taking an optimum time and economic considerations into

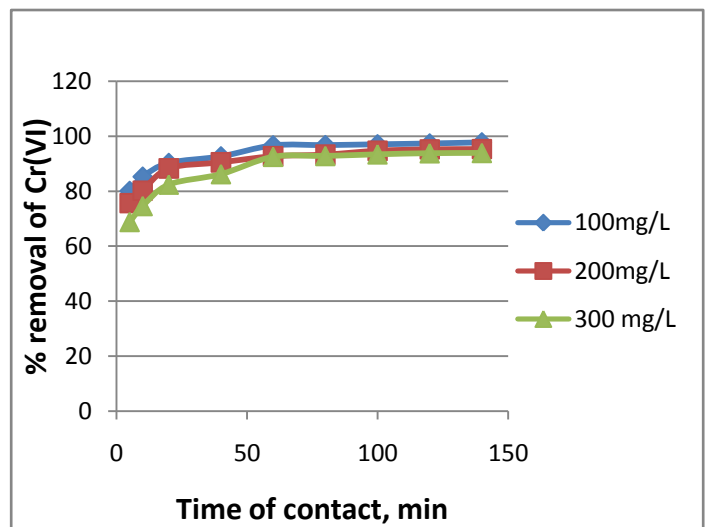
account, a contact time of 60 minutes was used for all batch experiments.

**OPERATING CONDITIONS**

pH	2
Adsorbent dosage	4g/L
Temperature	32 °C(room temperature)

Table 3.8: Effect of contact time on percentage removal

Sl. No.	Time (min)	%Removal 100mg/L	%Removal 200mg/L	%Removal 300mg/L
1.	5	80.1	75.7	68.8
2.	10	85.3	80.2	74.6
3.	20	90.3	88.3	82.4
4.	40	92.7	90.5	86.2
5.	60	96.7	92.7	92.5
6.	80	96.8	93.3	92.8
7.	100	97.1	94.7	93.4
8.	120	97.4	95.2	93.8
9.	140	97.8	95.3	93.9



Graph 3.8 % removal of Cr (VI) vs time of contact (min.)

**3.5 ADSORPTION ISOTHERM:**

The equilibrium of adsorption is an important physio-chemical parameter for evaluation of the adsorption process. To model the adsorption behaviour two adsorption studies were studied and their correlation with the experimental data was assessed. These included the Freundlich and Langmuir isotherms, which are the earliest and simplest known relationships describing the adsorption equation. [13]

### 3.5.1 LANGMUIR ISOTHERM:

The linear form of the Langmuir equation is shown in the following equation

$$C_e/q_e = 1/bq_m + C_e/q_m$$

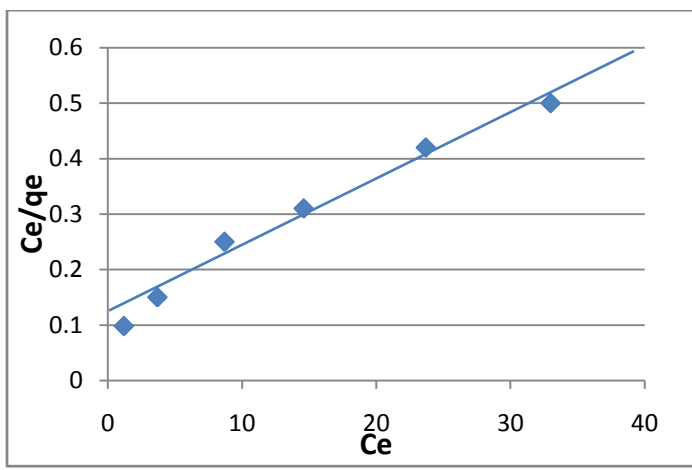
Where, b and  $q_m \rightarrow$  constants related to the apparent energy of adsorption and the adsorption capacity, respectively

$q_e \rightarrow$  the amount adsorbed per unit mass of the adsorbent ( $\text{mg g}^{-1}$ ) with an equilibrium concentration of  $C_e$  ( $\text{mg L}^{-1}$ ).

A plot of  $(C_e/q_e)$  vs.  $C_e$  was linear and the constants  $q_m$  and b were determined from the slope and intercept of the plot. The correlation coefficient obtained with the Langmuir equation was high, which indicated a good fit between the parameters. The dimensionless parameter ( $R_L = 1/(1+bC_e)$ ), which is a measure of adsorption favourability, was found to be in the range of  $0.03105 < R_L < 1$  and confirmed that Cr(VI) removal using activated charcoal at pH 2 and  $27 \pm 2^\circ\text{C}$  was a favourable adsorption process.

Table 3.9: Langmuir isotherm

Sl.No.	$C_e$	$C_e/q_e$
1.	1.2	0.098
2.	3.7	0.15
3.	8.7	0.25
4.	14.6	0.31
5.	23.7	0.42
6.	33	0.50



Graph 3.9:  $C_e/q_e$  vs  $C_e$

### 3.5.2. FREUNDLICH ISOTHERM

The freundlich isotherm is expressed by the equation

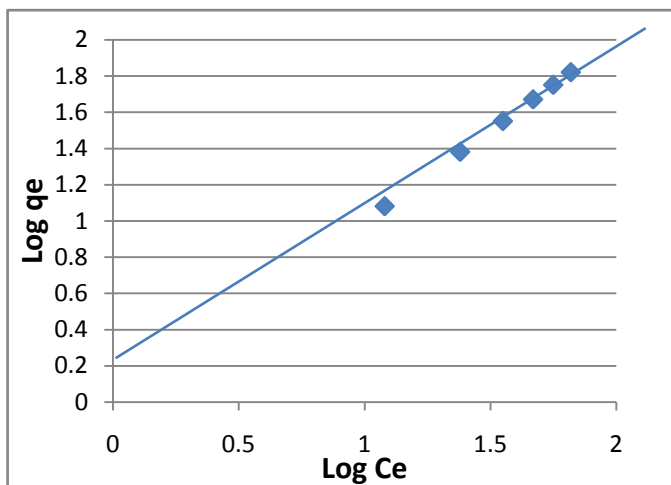
$$\text{Log } q_e = \text{log } K_f + (1/n_f) \text{log } C_e$$

Where,  $K_f (\text{g}^{1-1/n_f} \text{L}^{1/n_f} \text{kg}^{-1}) \rightarrow$  the freundlich constant which indicates the relative adsorption capacity of the adsorbent related to the bonding and regime

$n_f \rightarrow$  the heterogeneity factor representing how the adsorption deviates from linearity.

The value of 'n' as shown in the table lies between 1 and 10, which represents a favorable adsorption.

Sl. No.	$\text{Log } C_e$	$\text{Log } q_e$
1.	0.07	1.08
2.	0.56	1.38
3.	0.93	1.55
4.	1.16	1.67
5.	1.370	1.75
6.	1.510	1.82



Graph 3.10  $\text{Log } q_e$  vs  $\text{Log } C_e$

### RESULTS

Table 3.11: Linear regression equations, co efficient of determination and isotherm constants for Langmuir and Freundlich isotherms

<b>Langmuir isotherm</b>	$q_m$	<b>b</b>
	55.24	0.624
<b>Freundlich</b>	$K_f$	$n_f$
	1.08	1.92

### 4. CONCLUSION

Batch adsorption studies for the removal of Cr (VI) from aqueous solutions have been carried out using five different adsorbents. The study indicated the suitability of the adsorbents used for removal of Cr (VI) aqueous solution. The selected adsorbents may be viewed as a useful material while considering the economic aspects of wastewater treatment. The obtained results may be summarised as follows:

- 1) The concentration variation studies showed that percentage of Cr (VI) removal from waste water using groundnut was found to be ranging from 51.7% to 69%, for neem powder 79.4% to 87.3%, for modified

neemleaf powder 81.9% to 95.7%, for wheat bran 78.8% to 83.35%, and for modified activated charcoal the percentage removal was found to be ranging from 89% to 97.6%.

- 2) The best results were obtained by using modified activated charcoal in quantities that are economical for large scale purpose.
- 3) Other parameters such as pH, amount of adsorbent, contact time were varied for this particular adsorbent.
- 4) The pH variation studies showed that the adsorption process is highly pH dependent. The pH was varied from 1 to 11 and the optimum removal was found to be at pH 2.
- 5) Increase in adsorbent dosage leads to increase in Cr (VI) adsorption due to increased number of adsorption sites. Maximum uptake of Cr (VI) was obtained as the adsorbent dosage was increased from 2g/L to 20g/L.
- 6) The contact time, i.e., the time of shaking in the rotary shaker was varied from 5min to 140 min for three different concentrations. The percentage removal increases as the contact time is increased.
- 7) The results suggest that adsorption of Cr (VI) on the selected adsorbents involves a complex mechanism and in the adsorption process there are two distinct stages – the initial stages of boundary layer diffusion due to external mass transfer effects and the later stages it was due to intra particle diffusion which contributes to the rate determining step.
- 8) The adsorption isotherm studies showed that both Langmuir and Freundlich adsorption isotherm model fits well with the experimental data.
- 9) The adsorption capacities of the adsorbents for the removal of Cr (VI) have been compared with those of other adsorbents reported in the literature.
- 10) Modified neem leaf powder can be used as the next best adsorbent according to the comparative study made above.

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