REMOVAL OF CHROMIUM (VI) BY PREPARED ACTIVATED CARBON

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ABSTRACT

*Heavy metals are continuously released into the aquatic environment in various ways. They adversely affect the ecosystem. Heavy metal pollution has become one of the most serious environmental problems now-a*days. The removal of heavy metals from wastewater is one of special concern. So, removal of heavy metals is *primary important aspect. A variety of traditional and advance techniques are available. Adsorption is one of the techniques. Activated carbon is one of the well-known adsorbents. It is having high adsorption capacity, due to large surface area and pore volume. In recent years, huge research has been focused towards converting the agricultural wastes into activated carbon to make the adsorption process economical.*

In this study karanja seeds with shells (Pongamia pinnata shell) are used for the preparation of activated carbon. These are agricultural waste and so economical. The activated carbon prepared from karanja seeds with shells has been used for removal studies of Cr (VI) from wastewater. Different characteristics of prepared activated carbon such as moisture content, ash content, specific gravity, and decolourising power are to be found out. Experimental studies were conducted to find out the effect of contact time, pH, carbon dosage on Cr (VI) removal. *There by finding out optimum contact time, optimum dosage and optimum pH for decided initial concentration of Cr (VI).*

Keyword - *Karanja1, Heavy metals2, Adsorption3*

1. Introduction:

Water of high quality is essential for human existence and agricultural, industrial, domestic and commercial use and all these activities are also responsible for polluting the water. Majority of the industries are water based and a considerable volume of wastewater originated from these is generally discharged into water sources either untreated or inadequately treated resulting in water pollution. A study conducted by the Centre for Science and Environment, New Delhi, India, has suggested that over 70% of available water in India is polluted (C.S.E. Survey, 1982).**[5]** The contamination of water due to toxic heavy metals through the discharge of industrial wastewater is a global environmental problem. The heavy metals reach the water bodies through many industrial activities. From the heavy metals chromium is such metal which is to be found in aqueous system as both ionic forms i.e. Trivalent and Hexavalent chromium. Recently much importance has been given on removal techniques and developments of new process for heavy removal from waste water. There are large number of industries which discharge chromium containing waste, namely tanning, electroplating, textile cement and asbestos, refractories, cooling towers of thermal power stations and many other industries. Adsorption has been advocated as most promising among the currently known methods for waste water treatment, especially for removal of heavy metals. The adsorption process can be carried out using abundantly available low-cost adsorbent. In the present study *Pongamia Pinnata* seed is selected for preparation of activated carbon for removal of Cr (VI) from waste water.

1.1 Objectives of the research:

- 1. To arrive at the low cost adsorbent for removal of heavy metal i.e. hexavalent chromium.
- 2. To study the physical and chemical properties of the prepared activated carbon.
- 3. To determine the efficiency of the prepared activated carbon.
- 4. Economical way to remove heavy metals from industrial waste water.

1.2 Layout:

This study shall provide a better solution to hexavalent chromium pollution in aqueous phase by developing an efficient adsorbent. It will also provide an ideal technology to utilize and convert waste water into usable form i.e., activated carbon which scan be commercialized for the removal of contaminants from aqueous phase.

2. LITERATUR REVIEW

M. Makeswari, T. Santhi et al⁶ studied the removal of Cr (VI) from aqueous solution by using activated carbon prepared from Ricinus Communis leaves. Adsorption experiments were carried out by batch experiment to investigate the influence of different factor such as contact time, initial concentration of metal ion and pH of solution. The adsorption study revealed that contact time of 35 minutes for raw leaves of Ricinus Communis (RLRC) and 40 minutes for zinc chloride activated leaves of Ricinus Communis (ZLRC) is satisfactory to reach evenness at the solution pH of 7.05, ideal adsorbent does of 200mg/500ml and the metal ion concentration of 100mg/l. The efficiency of Cr (VI) adsorption increases with the increase in the contact time and adsorbent dosage, but it decreases with increase in the initial concentration of the adsorbate solution.

Muthukumaran K. and Gophie Beulah et al⁹ studied the removal of Cr (VI) from wastewater using chemically activated Syzygium jambolanum nut carbon by batch studies. In their study large amount of removal is occurred at pH of 2 as $HCrO₄$.he optimum carbon dose needed was only 0.1g and the optimum time needed was 4 hours for quantitative removal of Cr (VI) in batch modes. Cr (VI) could recovered up to 85% using 10% H_2O_2 and 1M NaOH. The method of preparation of chemically activated high temperature Syzygium jambolanum nut carbon is viable and could be used as a promising tool remediate Cr(VI) from wastewater.

Adhena Ayaliew, Werkneh, Nigus Gahbiye Habta, Hayelom Dargo Beyene et al¹ studied the removal of hexavalent chromium from tannery wastewater using activated carbon primed from sugarcane bagasse by adsorption process. The removal and recovery of chromium from tannery wastewaters is crucial for environmental protection and economic reasons. Thus, this work focuses on investigating low cost activated sugar cane bagasse for effective removal of hexavalent chrome from aqueous solutions. The maximum adsorption of Cr (VI) is observed at pH of 1.It was found that the Cr (VI) removal was increased with the increment of the adsorbent dosage and contact time. The result showed that the percentage of Cr (VI) recovered and adsorbent regenerated, increase with increasing pH and strength of regeneration solution, contact time and temperature.

 Renuga Devi N., Majasha K. and Lalitha P. et al¹⁰ studied the Removal of Hexavalent Chromium from aqueous solution using an eco-friendly activated carbon adsorbent (Delonix pods).The adsorption of Cr(VI) was found to be concentration dependent. Percentage of removal Cr (VI) is increased with decrease in pH and it was found to increase from 36.75% to 90.12% and pH varies from 4 to 2. The maximum Cr (VI) is removed at pH of 2. Thus the result shows that activated carbon produced from Delonix regia pods, is an inexpensive plant material and use of this material provides an effective solution for treatment of waste water containing Cr(VI).

 S. Vijay Kumar, R. Narayanaswamy, M. Sripathy, Prof. K.V. Pai et al¹² studied the Comparative Study of Removal of Chromium -IV Ion from Aqueous Solution Using Eucalyptus, Neem and Mango Leaves. The removal of Cr (VI) from waste water is initially depends on pH of solution, agitation rate, contact time and Cu concentration particle size. Maximum adsorption of Cr(VI) is obtained at pH 2 and 11, agitation rate of 150 rpm, particle size is 75µm, initial Cu concentration is 5mg/l and contact time 20 minutes. Maximum removal of Cr (VI) from waste water is obtained if all of the five parameters are optimized.

 Karnib Mona, Kabbani Ahemad, Holail Hanafy, Olama Zakia et al³ has conducted the batch experiment to test the ability of activated carbon for the removal of lead, cadmium, nickel, chromium and zinc from water. In his study nickel showed the highest removal percentage by activated carbon and the removal percentages decrease as the concentration of heavy metals increase. Silica / activated carbon (2:3) composite was more efficient in the

removal of nickel ions than activated carbon and silica nanoparticles. In their study, activated carbon showed the greatest affinity towards nickel with 90% removal percentage.

Muataz Ali Atieh et al⁸ has conducted the batch experiment to remove Cr (VI) from polluted water using activated carbon nanotubes supported with activated carbon. He found that the activated carbon coated with carbon nanotubes is considered as an excellent adsorbent to Cr in their study the application of both the activated carbon and the carbon nanotubes attached to the activated carbon have been investigated as potential adsorbents to Cr(VI) from waste water.

Ahmed El Nemr, AmanyEl-Sikaily, Azza Khaled, Ola Abdelwahab et al² has conducted the batch experiment by using which is a red marine macro algae, was tested for its ability to remove toxic hexavalent chromium from aqueous solution. A new activated carbon obtained from Pterocladia capllacea via acid dehydration was also investigated as an adsorbent for toxic chromium the experiments were conducted to study the effect of important parameters such as pH, chromium concentration and adsorbent weight. The maximum sorption capacities for dried red alga and its activated carbon about 12 and 66mg /g respectively, as calculated by Langmuir model. The ability in activated red alga P. capllacea and developed activated carbon to remove chromium from synthetic sea water, natural sea water and waste water was investigated as well. Different isotherm models were used to analyse the experimental data and models parameters were evaluated. This study shows that the activated carbon developed from red alga P. capllacea is a promising activated carbon for removal of toxic chromium.

Khalid R. Alhooshani et al⁴ had conducted batch experiment in which an efficient and fast adsorptive removal of dichloromethane, trichloromethane and carbon tetrachloride from aqueous media at ambient conditions was achieved by using AC loaded with cerium oxide nanoparticles (CeO₂-NP/AC). The adsorption removal of tested organic chlorinated compounds onto $CeO₂-NP/AC$ composite can be well described by Freundlich adsorption isotherm. In their study, kinetic characterization of the adsorption process onto the developed adsorbent was well described by the pseudo second order model, and the adsorption fit the intraparticle diffusion model. His study shows that at optimum conditions, 82.72%, 99.40% and 89.42% of dichloromethane, chloroform, and tetrachloride, respectively, were removed by $CeO₂-NP/AC$, at concentration between 0.25 and 5.00g/L

3. METHODOLOGY:

3.1. Selection of material:

To evaluate a feasible and economical low cost treatment. To remove the heavy metal Cr (VI) present in synthetic sample by using abundantly available seeds of karanja. By preparing activated carbon from this karanja seeds (*pongamia pinnata*) as an adsorbent.

3.2. Properties of karanja (*pongamia pinnata***):[11]**

3.2.1. Botanical classification:

3.2.2. Botanical name:

Pongamia pinnata (L.) Pierre

3.2.3. Synonyms:

Derris indica (Lam.) Bennett

Millettia novo- guineensis Kane.

Pongamia glabra Vent.

Pongamia pinnata Merr.

3.2.4. Common names:

Hindi, Beng, Mar, and Guj: karanja, karanj

3.2.5. Botanical Description:

According to Allen and Allen (1981) the *Pongamia Pinnata* is a fast – growing tree which reaches 40 feet in height and spread, forming a board, spreading canopy casting moderate shape. All the description characteristics of this plant are listed in table 3.2.5.

Plant type		Medium-sized, evergreen, perennial and deciduous trees
		(figno.3.2.7.1)
		Height $-35-40$ ft
		Chromosome no. -22
Growing requirements		Soil tolerance: - clay; loam; sandy; slightly alkaline; acidic; well – drained.
(a)	Leaf	Alternate, odd pinnately compound 2 to 4 inches, evergreen, hairless.
(b)	Flower	Lavender, pink; white, 2-4 together, short -stalked, pea shaped, 15-18 long.
(c)	Pods	$3 - 6$ cm long and 2-3cm wide, smooth, brown, thick-walled, hard, indehiscent, 1-2 seeded.

Table 3.2.5:- Botanical Description of *pongamia pinnata* **[11]**

3.2.6. Characteristics of karanja:

It is essential to know few characteristics of seeds of karanja (*pongamia pinnata*) carbon which is an adsorbent, like moisture content, ash content, decolourising power, PH, surface area, density & specific gravity of prepared activated carbon.

3.2.7. Figures of karanja:

Fig. 3.2.7.2: Seeds

3.2.8. Uses of karanja:

i) Wood: The wood of *pongamia pinnata* for cabinet making, cart wheels, agricultural implants, tool handles, &combs (GOI 1983).

ii) Oil: In India the oil which is extracted from the karanja seeds is used as fuel for cooking & lamps. Also it is used as lubricant, water – paint binder, pesticides, in soap making.

Iii) Medicinal properties: karanja seeds are antiseptic in nature. It is excellent for skin & hair & used in the manufacturing of soap, cream, lotions.

iv) Seed: The seeds of karanja are used to prepare activated carbon & this AC is then to remove the Cr (VI) from the waste water.

v) Other uses : Dried leaves are used as an insect lateral roots makes this trees ideal for controlling soil erosion and binding sand dunes

3.3. Methods:

3.3.1 Preparation of activated carbon:

For removal of Hexavalent chromium from aqueous solution adsorption technique was employed using activated carbon prepared from abundantly available seeds of Karanja (*Pongamia Pinnata*).

There are two methods to prepare activated namely:

- 1. Physical Activation
- 2. Chemical Activation

 In the present study both physical and chemical activation are employed to prepare active carbon powdered seeds of Karanja (*Pongamia Pinnata*).

3.4 Physical Activation:

The seeds of Karanja (*Pongamia Pinnata*), broken into pieces, and churned into powder form, washed in distilled water for 2 to 3 times. The powder was then oven dried at 105±5ºC for 24 hours. The oven dried powder was filled in a small container in three layers, by compacting each layer without any air space to avoid the loss in weight of the powder otherwise it would result in burning of material directly, leaving behind only the ash. The small container was then placed into a bigger container, such that, sand surrounded the small container completely. The lid of container was tightly fitted. Then the set-up was kept in muffle furnace at the temperature of 650ºC. After attaining the required temperature, the furnace was allowed to cool for about 10 hours. Before the container was taken out. The sketch furnished in fig. below was the set-up of the containers.

The activated carbon thus obtain was sieved to 300µ(sieve) size and packed in a polythene cover and stored in desiccator.

The activated carbon thus obtain was sieved to 300µ(sieve) size and packed in a polythene cover and stored in desiccator.

3.4.1 Moisture Content:

 About 1 gm of the material was weighed into a petri dish. The dish was placed in an electric oven maintained at 100± 5°C for about 4 hours. The dish was covered, cooled in a desiccator. Heating, cooling and weighing was repeated at 30 minutes intervals until the difference between the two consecutive weighing was less than 5 mg. Data's are shown in table No- 3.4.1

Moisture Content = $100 (M - X)/M$

(Percent by mass)

Where,

 $M =$ Mass in grams of the material taken for the test.

 $X =$ Mass in Grams of the material after drying.

3.4.2 Ash Content:

 One gram of the carbon variety under examination was weighed accurately into a tarred porcelain crucible. The crucible and its contents were placed in an electric oven at $110 \pm 5^{\circ}$ C for about 4 hours. The crucible was removed from the oven and the content were ignited in an electric muffle furnace at a temperature of 1000 °C for about 3 hours. The process of heating and cooling was repeated until the difference between two consecutive weighing's was less than 1 mg. The ash content of the different carbons are shown in the table – 3.4.2.

 $M1 =$ Mass of the ash in grams.

 $M =$ Mass of the material taken for the tests in grams.

 $X =$ Percentage of moisture content in the material taken.

3.4.3 Decolourising Power:

About 0.1 g of the carbon material was transferred to 50 ml glass stoppered flask. 1 ml of methylene blue solution (0.15%) was added from a burette and shaken for 5 min. Addition of methylene blue solution and shaking was continued till the blue colour persisted for at least 5 minutes.

 Decolourising power of carbon is expressed in terms of milligram of methylene blue adsorbed by 1 g of activated carbon.

Decolourising power $(mg/g) = 1.5$ ^{*} V/M

Where, $V = Volume in ml of methylene blue solution consumed.$

 $M =$ Mass of the material taken for the test in grams.

The decolourising power of different carbons are shown in table -3.3

3.4.4 pH Values of Prepared Activated Carbons:

10 g of the dried material was weighed and transferred into a one litre beaker. 300 ml of freshly boiled and cooled distilled water (adjusted to pH 7.0) was added and heated to boiling. After digesting for 10 min, the solution was filtered while hot, rejecting the first 20 ml of the filtrate, the remaining filtrate was cooled to room temperature and pH was determined using digital pH meter.

The P^H values of different carbons are shown in table 3.4.4

3.4.5 Determinations of Surface Area (Acetic Acid Adsorption Method)

1 Gram of carbon was accurately weighed to the nearest milligram and added to a series of 300 ml stoppered glass bottles. Acetic acid solution in the range of 0.015 M to 0.15 M were prepared separately and 100 ml of these solutions were added. The another bottle 100 ml of 0.03 M acid alone was added which is in absence of carbon served as a control. The flasks were tightly stoppered and then shaken in a rotary mechanical shaker.

 At the end of equilibrium period the samples were filtered through fine filter paper. The first 10 ml of the filtrate was rejected, from the remaining filtrate. 25 ml aliquots were withdrawn and filtrated against 0.1 N standard sodium hydroxide solution using phenolphthalein indicator.

 The final concentrations of acetic acid were calculated for each sample. By noting the difference in initial and final concentration of acetic acid, the number of moles of acetic acid adsorbed by the carbon was calculated. The number of moles of acid adsorbed per gram of carbon was then computed which is designated as N.

 The concentration of acetic acid remaining in each instant (C) was divided by number of moles of acid adsorbed per gram (N) of the carbon to get the ratio C/N. A plot of C/N versus the concentration of acetic acid remaining in the containers after adsorption process s(C) was made. A straight-line plot was obtained. The reciprocal of the slope of the straight line gives the number of moles of acetic acid required per gram to form a

monolayer which is designated as (Nm). By assuming that the molecular cross-sectional area of acetic acid as 21 areas available in square metre per gram of the carbon calculated from the following equation.

3.4.6 Determination of Specific Gravity:

The specific gravity of the prepared carbon was determined using pycnometer as follows by determining

- 2. Weight of pycnometer + distilled water W2
- 3. Weight of pycnometer + 1/3 carbon W3 4. Weight of pycnometer $+1/3$ carbon + distilled water W4
	-

W3 – W1

The specific gravity of the prepared carbon = -----------------------------

 $(W2 - W1) - (W4 - W3)$

The specific gravity of the prepared carbon are shown in Table 3.4.6

Table 3.4.6.1 Specific Gravity of Prepared Carbon

Table 3.4.6.2 Density of Prepared Carbon

3.5 Instruments:

3.5.1 Spectrophotometer:

 Chemito model UV 2100 UV- Visible Spectrophotometer with 10 mm cell path cuvettes was used for all measurements.

3.5.2 pH Meter:

Hanna Instruments Model-HI 98107 Pocket pH Tester (digital pH meter accuracy \pm 0.1 pH unit with combined electrode) was used for all pH measurements.

3.6 Solutions:

3.6.1 Preparations of Synthetic Hexavalent Chromium Solution:

 Synthetic hexavalent chromium solution was prepared by dissolving 282.8 mg potassium dichromate in one litre of deionised and distilled water, such that each solution contains 100µg of chromium.

3.7 Titration Curve For Acidification Of Chromium (VI) Solution:

5 ml of chromium solution and 45 ml distilled water were taken in a beaker. 0.1 N H_2SO_4 was taken in burette and slowly added, the resulting P^H was noted using P^H meter. The titration curve can be obtained by plotting pH versus 0.1 N $H₂SO₄$ added. The resulting plot is shown in figure 3.7.

Fig No. 3.7:- Calibration Curve for Cr(VI)

3.8 Batch adsorption Experiment:

In batch sorption, a pre-determined amount of adsorbent will be mixed with the sample, stirred using magnetic stirrer for a given contact time and subsequently separated by filtration.

Effect of several parameters such as pH, concentration of metal ion, concentration of adsorbent and contact time on adsorption of copper on powdered raw *pongamia pinnata* seed shell was studied by batch technique. All experiments were carried out at room temperature so as to avoid the heating of effluent in case this study would be . Batch experiments were carried out at an agitation of 100 rpm, samples at predetermined time intervals were collected, filtered by whatman filter paper and remaining chromium was analysed by UV Spectrophotometer as per standard methods. All experiments were carried out at pH values ranging from 2 to 10, the initial concentration of metal ion of 150 mg/L . Adsorbent dose of 2.5 to 15 gm and particle size of 300μm and the contact time of 30min to 180min based on equilibrium conditions. The percentage removal of toxic metal from the solution was calculated using the equation,

% Removal = Ci– Cf/Ci \times 100

Where, Ci- is initial concentration of toxic metal,

Cf- is final concentration of toxic metal

4.RESULT AND DISSUSSION

The chapter deals with the study of prepared carbon efficiency for removing hexavalent chromium for,

Effect of pH

Effect of adsorbent dosage

Effect of initial metal ion concentration

Effect of contact time

4.1 Effects of pH variation on Cr (VI) removal from aqueous solution:

In order to optimize the pH for maximum Cr (VI) removal, experiments were conducted with 150ml of Cr (VI) solution with 15 gm adsorbent by varying the pH, pH - 2 to 10 at constant contact time and the results are depicted in table - 2 .The results indicated a maximum adsorption of (99.44%) at pH 2, in 30 minutes of contact time. The decrease in adsorption at higher pH may be due to the negative charges on the surface of the adsorbent repelling the negatively charged chromate ions.

Table 4.1: Effect of pH on % removal of chromium ions by *pongamia pinnata* **seed shell adsorbent**

pH: 2to 10; temp: 32°c; conc. Of Cr(VI): 150ppm; contact time: 30min

Fig No.4.1:- Effect of pH on % removal of chromium

4.2 Effect of adsorbent dosage on Cr(VI) removal from aqueous solution:

The effect of variation of adsorbent dosage was determined by varying the adsorbent dosage from 2.5 to 15 gm with 150ml of solution. The results have been tabulated in Table 3. It is evident from the table that, The increase in percentage adsorption of Cr(VI) with increase in the adsorbent dosage may be due to the availability of more surface area of the adsorbent for adsorption of Cr(VI) species.

Table 4.2: Effect of amount of adsorbent on % removal of chromium ions by *Pongamia Pinnata* **seed shell Adsorbent**

Adsorbent Dosage: 15 gm; Temp: 32ºc; concentration of Cr (VI) solution: 150 gm/lit; Contact time: 30 min.

Fig No. 4.2:- Effect of amount of adsorbent on % removal of chromium

4.3 Effect of variation of initial concentration of Cr (VI) solution on adsorption of Cr (VI) from aqueous solution:

The adsorbate concentrations were varied from 10 to 50gm/lit. and batch studies were Performed to optimize the initial concentration of the adsorbate. The percentage removal of Cr (VI) with variation in initial concentration of Cr (VI) solution is given in table -4 .

The increase in percentage removal Cr (VI) when the adsorbate concentration was varied from 10 to 50gm/lit. As the concentration increases percentage removal of Cr(VI) decreases.

Table 4.3 Effect of initial metal ion concentration on % removal of chromium ions by *pongamia pinnata* **seed shell adsorbent**

Adsorbent dosage: 15gm, pH 2±0.02, Temp.: 32ºC, Contact time: 30 min.

Fig No. 4.3:- Effect of initial metal ion concentration on % removal of chromium

4.4 Effect of Contact Time:

Contact time has great influence in the adsorption process. The effect of contact time on the removal of chromium (VI) for synthetic sample at pH 2 ±0.02 using physically carbon of Seeds of Karanja (*Pongamia pinnata*) in figure- and model values are shown in the table from the table it is observed that contact time differs for different carbons (30 to 180 min) i.e. for physically activated carbons. It is further authenticated by studying pore diffusion of carbons. As the contact time increases Hence, percentage removal of (Cr VI) also increases.

Table 4.4: Effect of contact time on % removal of chromium ions by *Pongamia Pinnata* **seed shell Adsorbent**.

Adsorbent Dosage: 15 gm; Temp: 32ºc; Contact Time: 30 to 180 min.

Fig No. 4.4:- Effect of contact time on % removal of chromium

5.CONCLUSION:

Based on the present study the following conclusion can be drawn.

- 1. Raw material of Seeds and shells of Karanja (*Pongamia Pinnata*) has high efficiency in Cr (VI) removal.
- 2. Seeds and shells of Karanja (*Pongamia Pinnata*) can be effectively used for the preparation of activated carbon.
- 3. Activated carbon of Seeds and shells of Karanja (*Pongamia Pinnata*) has good Cr (VI) removal potential.
- 4. Statically analysis shown that chemical activation of carbon increases the surface area of adsorbent.

5. The adsorption of Cr (VI) is pH dependent. The removal efficiency of adsorbent increases with decreases in pH. Maximum adsorption takes place at pH 2.

6. The maximum recovery of Cr (VI) can be achieved by washing adsorbed carbon with 1N NaOH compare to 1N HCL and distilled water.

7. Reused carbon is also effective in removing Cr (VI). Small amount of efficiency lost compared to fresh carbon using the recovery and reuse technique.

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