

## Removal of Chromium (VI) From Aqueous Solutions Using Discarded *Solanum Tuberosum* as Adsorbent

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**Abstract:** Industrial polluting effluents containing heavy metals are of serious environmental concern in India. Chromium is frequently used in industries like electroplating, metal finishing, cooling towers, dyes, paints, anodizing and leather tanning and is found as traces in effluents finding their way to natural water bodies causing hazardous toxicity to the health of humans, animals and aquatic lives directly or indirectly. Many methods for the removal of Chromium such as chemical reduction, precipitation, ion exchange, electrochemical reduction, evaporation, reverse osmosis and adsorption using activated carbon etc. have been reported but all being expensive and complicated to operate. Experimental practices reveal that adsorption by agricultural and horticultural wastes are quite simple, inexpensive and efficient method. Agra is famous for Potato farming, a lot of discarded potato waste from cold storages is thrown along road side drains causing solid waste generated which either creates solid waste disposal problem or otherwise it finds way to Yamuna river resulting high BOD and posing a serious threat to the aquatic environment. For developing countries like India adsorption studies using discarded potato (*Solanum tuberosum*) waste from cold storages (DPWC) a solid waste as low cost adsorbent for Chromium removal was dual beneficial i.e., an ideal solution to these solid wastes disposal problem of Agra and removal of Chromium from tannery effluents and thereby saving aquatic life from Chromium contamination in Yamuna river. Keeping this in view batch experiments were designed to study the feasibility of discarded potato waste from cold storages to remove chromium (VI) from the aqueous solutions. During the study various affecting parameters, such as pH, adsorbent dose, initial concentration, temperature, contact time, adsorbent grain size and start up agitation speed were optimized as 5.0, 10-20 g/l, 50 mg/l, 25°C, 135 minutes, average size and 80 rpm respectively on chromium removal efficiency. Various Isotherms such as Langmuir, Freundlich, Tempkin also fitted suitably and various corresponding constants determined from these Isotherms favor and support the adsorption. Thermodynamic constants  $\Delta G$ ,  $\Delta H$  and  $\Delta S$  were found to be 0.267 KJ/mole, 0.288 KJ/mole and 0.0013 KJ/mole respectively.

**Keywords:** Adsorbent, Toxicity, Discarded, Potato, DPWC, Disposal, Cold Storage and BOD.

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### I. Introduction

Developing countries like India have a serious environmental concern of industrial effluents containing trace quantities of heavy metals such as nickel, manganese, lead, chromium, cadmium, zinc, copper, iron and mercury. Though some of these in micro content act as essential nutrients for biological growth (Metacalf and Eddy et al, 1994) but larger concentrations, , cause harmful effects on living beings like humans, animals and plants. Right under the district administration around 50 illegal tanneries are functioning in the city of Taj Agra, causing immense damage to the storm water drain system as well as posing a serious health hazard to the locals as well as for aquatic life. Tanneries are located in the heart of Agra city-Khatikpada near Raghunath Cinema. This is a quite congested area and entry of outsiders is closely scrutinized, while doing photography or collecting samples for investigation (TOI, Jan 02, 2016). Hence, it is desirable to adopt preventive measures so as to control and reduce the concentrations of these toxic substances. Many investigations regarding heavy metal concentrations and their toxicity in the aquatic environment have attracted the researchers attention and the general awareness. Chromium causes Nausea, vomiting, epiesgestric pain, severe diarrhoea, haemorrhage, dermatitis by skin contact, nasal mucous membrane, ulcer (Satyanarayan et al, 1995), lung cancer and tissue necrosis in humans, reduction in fish production at high concentrations, chromium accumulates in fish tissues and reaches to consumers. Gold fish and trout are killed at a concentration of 180 mg/l in Aquatic life and reduction in Soil fertility if chromium-bearing effluents are discharged on land (Sohail, 1997) and this way Chromium affects human beings, aquatic life and soil.

## II. Sample Collection

Agra, a historical place is worldwide famous for the world's seventh wonder Taj situated at the bank of holy river Yamuna, a big tributary of the great Ganga river and also a big industrial city located in northern India 195 Km Eastward from New Delhi on NH-2. The city is famous for the shoe industries, tanneries, and cold storages. The surrounding area of Agra is full of dense potato agriculture, the product potato is usually stored in cold storages for its use round the year where from tones of potato goes waste and discarded potato is directly thrown along the road side drains in which it rotten every year in huge amount creating foul gases and after rain this rotten discarded potato waste joins Yamuna river increasing its BOD, as a result the Yamuna gets polluted. Millions of money is being funded on Yamuna Action Plan a part of Ganga Action Plan by the central government and hence that ultimately goes waste despite of more many efforts. Many samples from various tanneries of the city were collected and analyzed to find the chromium content contaminating the Yamuna river water. The tannery effluent samples were also collected from the outlet points of the industries surroundings and it's nearby places and preserved as per standard methods before the physico-chemical analysis. All the samples were tested for pH, chloride, alkalinity, suspended solids, hardness, total dissolved solids and chromium content. The study resulted that most of the cases the surface water was within the limit except a few cases where the water was found not very well for direct disposal in river and contained chromium (VI). However, the chromium content in the surface water was found above the prescribed safe limit 0.1 mg/L (Federal Register 1981).

The surface water samples from various locations were collected and analyzed for the various parameters and results have been listed in Table-1.

**Table-1** Quality Parameters of Surface Water of Agra City

S. No.	Location	pH	Chloride mg/l	Alkalinity mg/l as CaCO <sub>3</sub>	Suspended Solids mg/l	Hardness mg/l as CaCO <sub>3</sub>	Total Solids mg/l	Chromium (VI) mg/l
1	Kuber Pur	7.5	60.2	55.6	250	380.6	965.2	9.2
2	Runkata	6.9	140.5	72.4	450	690.5	1610	30.5
3	Sikandra	7.2	115.6	62.2	380	450.3	1500	32.4
4	Dayal Bagh	7.0	120	55.6	360	432.4	1600	34.4
5	Poiya	6.7	132.5	55.2	365	445.6	1460	31.6
6	Bichpuri	7.2	112	55.8	260	358.4	900	1.2
7	Tajganj	6.8	175	84.6	360	410.4	1100	1.0

The local Agra region i.e. Agra-Hathras-Aligarh-Mathura, is a hub of potato farming aiming later use and exports purpose about 80% potato is kept in cold storages, existing on road side in a considerable number. Almost 10% of this cold stored potato is wastage which is usually left as it is in almost semi rotten stage along the road side drains where anaerobic activities take place in the later stage creating nuisance and objectionable foul smell. Every year during monsoon this all discarded potato waste finds its way to the natural streams Yamuna where BOD increases resulting considerable reduction in dissolved oxygen level causing survival threat to aquatic life. This very local and severe problem created an idea to investigate reuse, utility, feasibility and performance of waste potato to adsorb hexavalent chromium from local tannery effluent and thus it became a double fold pollution control as one pollutant is being used as a pollution controller for other pollutant.

### Sorption of Chromium (VI) on the Surface of Potato Waste

Many methods of removing chromium from wastewater such as chemical reduction and precipitation, electrochemical reduction, ion exchange, evaporation and reverse osmosis etc. have been reported in literature but all these methods include high costs along with regular expenses of chemicals and electricity. Researchers have still paid a very less attention in the direction of chromium removal using agro and horticultural wastes as adsorbent which have been proved to be potential (Sohail et al, 1998, 1999, 2014, 2014, Sharma, et al, 2010, 2013, 2015, 2016).

## III. Materials And Methods

To study hexavalent chromium adsorption capacity of discarded potato waste from cold storage batch experiments were conducted. Synthetic wastewater with known concentrations of Cr (VI) were prepared from the AR grade, Mono-Chromium Standard Solution for AAS prepared by Chromium Nitrate as 1000 mg/l Chromium (Cr) in 0.5 mol/l Nitric Acid (HNO<sub>3</sub>), prepared by IQBAL, Chemie (P) Ltd. and separately kept in glass stoppered conical flasks. Then known quantities of the adsorbent were mixed in wastewater. The process was equilibrated by shaking the contents of the flask at room temperature 25<sup>o</sup>C so as to have adequate contact time between adsorbent and the chromium ion. The suspension was filtered through Whatman No.1 filter paper and the filtrate was analyzed to evaluate the remaining concentration of Cr (VI) metal in the treated wastewater as per Standard Methods (Standard Methods, 1989) using Atomic Absorption Spectrometer (Perkin Elmer, Atomic Absorption Spectrometer, Pin AAcle 900F)

### Site Description, Collection and Preparation of Adsorbents

From local cold storage at Khandauli town (Agra) the discarded potato waste was collected, meshed and sun dried for 10 days in the month of March, 2014. After removing peel dust the dry meshed potato was grinded to the powder form and oven dried for two days at 90°C five hours daily. The oven dried powder was sieved through a 225 mesh (Indian Standard Sieve) to get an average and uniform sized particle. Then we washed this powder several times with distilled water to get rid of lighter materials, color releasing substances, dirt and other impurities. The adsorbent was dipped in 0.1N NaOH for a period of 9 hrs and washed several times with distilled water to remove the lignin and then dried again. The adsorbent was again rinsed separately with double distilled water two times and dipped into 0.1N H<sub>2</sub>SO<sub>4</sub> for the period of 9 hrs again to remove traces of alkalinity. The acid treated adsorbent was washed thoroughly with double distilled water. Then the dry powder so prepared was exposed to sun and stored in a desiccator. Before using the powder as adsorbent a size range was also achieved by sieving through a sieve set 1.7 mm, 1.18mm, 600µm, 425µm, 300µm, 150µm, 75µm and pan i.e. less than 75µm (Indian Standard Sieve) so as to optimize the size of the adsorbent.

### Chemicals

In the present study hexavalent chromium was used as an adsorbate and to get a waste of uniform characteristics to avoid interference with other impurities the laboratory wastewater was prepared by dissolving a known amount of AR grade, Mono-Chromium Standard Solution for AAS prepared by Chromium Nitrate as 1000 mg/l Chromium (Cr) in 0.5 mol/l Nitric Acid (HNO<sub>3</sub>), prepared by IQBAL, Chemie (P) Ltd., Laboratory Reagents & Fine Chemicals, Mumbai, India, the certificate of this standard solution is being attached in Appendices. All other chemicals such as Sodium hydroxide (NaOH), Sulphuric acid (H<sub>2</sub>SO<sub>4</sub>) were also from the same chemical company of LR grade. The Standard solution was stored in safe location and the required quantity was diluted with the deionized double distilled water to make solution of required concentration used in the studies. To avoid any kind of contamination the deionized and double distilled water was stored safely.

Though the collection of tannery wastewater samples for investigation purpose was not an easy task out of 24X7 close scrutinized watches yet on assurance of not disclosing the names of these illegal industries some effluent discharges being directly thrown in to the drains were obtained from Agra (India). The Chromium concentration were in the range of the hexavalent chromium concentration found in Agra city as mentioned in Table-2

**Table-2.** Physico-chemical characteristics of some tanneries of Agra City

S.No.	Parameter name	Tannery-1	Tannery-2	Tannery-3	Limits (Mg/L) discharge norms in India	
					Surface	sewer
1	Color	Dark Grey	Dark Blue	Dark Grey	Light blue	No color
2	BOD <sup>5</sup> (mg/l)	620	624	630	30	350
3	COD (mg/l)	1450	1560	1520	100	250
4	pH	8.8	9.1	9.6	5.5-9.0	5.5-9.0
5	SS (mg/l)	3880	4068	5050	100	100
6	Chloride (mg/l)	3950	4060	6062	-----	-----
7	Total solids (mg/l)	10,560	9860	10580	-----	-----
8	Total Chromium (mg/l)	18.6	22.00	17.40	1.0	0.5
9	Sulphate (mg/l)	180	240	196	1.0	1.0

### Experimental Procedure

A series of batch experiments were conducted to investigate the effects of various parameters such as pH, adsorbent dose, initial concentration of the chromium metal, contact time, temperature, adsorbent particle size and agitation speeds etc on adsorption. For all these batch set ups, synthetic wastewater of different concentrations of hexavalent chromium was prepared from the stock solution and distributed in glass stoppered conical flasks. Calculated doses of adsorbents had been added to these synthetic wastewater samples in conical flasks each of capacity 50 ml. The contents of the system were shaken for equilibration at room temperature on a mechanical shaker ( Indian Scientific Instruments Factory, Ambala Cantt, India) so as to achieve adequate contact time between adsorbent and the metal ions. After maintaining proper time of quiescent adsorption, the suspension was filtered through Whatman (No. 1) filter paper and then the filtrate was analyzed for the concentration of hexavalent chromium metal in the treated wastewater by using Atomic Absorption Spectrometer (Perkin Elmer, Atomic Absorption Spectrometer, PinAAcle 900F). Adsorption studies were made

for various times and as a result optimum time of adsorption was also determined. During the study the room temperature was 25°C. To study the effect of temperature variation the temperature was maintained using heaters in a closed chamber.

#### IV. Results And Discussions

During the study of effect of pH the initial chromium (VI) was kept 50 mg/l, as reported in tannery and chrome plating wastewater varying from 3-30 mg/l (ISI 7453-1977) but later it was optimized.

##### 4.1. Effect of pH on Adsorption

The adsorption of other ions is severely influenced due to strong adsorption of hydrogen from the solution and hydroxyl ions on the adsorbents. The pH of the solution also influences the degree of ionization affecting the adsorption process. Various pH values maintained by 1M sulphuric acid and 1M sodium hydroxide ranging from 1.0 to 6.0 have been tried with an initial chromium concentration of 50 mg/l. An adsorbent dose of discarded potato waste from cold storages (DPWC), 10 g/l was added to 50 ml of synthetic wastewater for the adsorption of hexavalent chromium. The optimum value of pH for the above set of parameters was found to be in the range 4.5 to 5. as shown in Fig.-1(Khan, et al 2001).

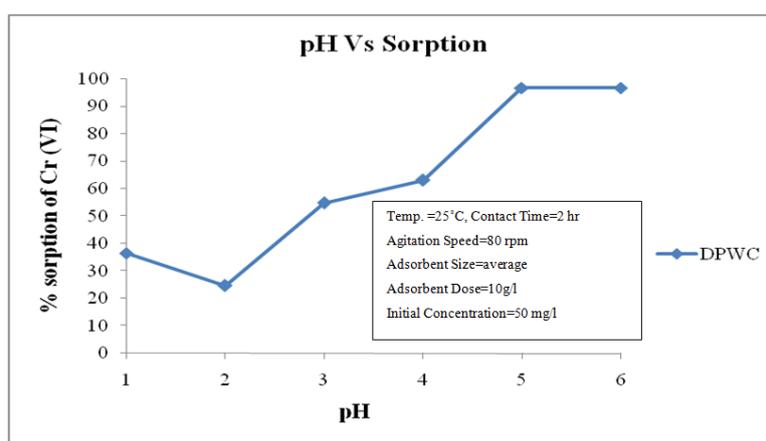


Fig-1 Effect of pH on Chromium Cr (VI) Sorption

##### 4.2. Effect of Adsorbent Dose on Adsorption

After optimizing the pH range and maintaining it at 4.5 to 5 at room temperature 25°C all the samples with amount 50 ml having initial chromium concentration 50 mg/l were tested for 2 hour time of contact for various doses of adsorbents right from 10 to 60 g/l with average sizes of adsorbent particles at rate of 80 rpm agitation speed and the observations for Discarded Potato Waste from Cold storage (DPWC) as adsorbent were analyzed. It has been observed that adsorption capacity of the adsorbent discarded potato waste from cold storages (DPWC) is the maximum at 20 g/l i.e. the optimum value as shown in Fig.-2. Here in the present study the optimum range of adsorbent dose was found to be 10 to 20 g/l and there after the adsorption capacity remains almost the same i.e. meaning to say that beyond a proper dose additional doses are ineffective, so there is no use of wasting extra adsorbents which could be used further. Perhaps this might be due to the saturation in adsorption capacities and a very few amount of adsorbate remaining in the solution further to be adsorbed. (Sharma, et al, 2016)

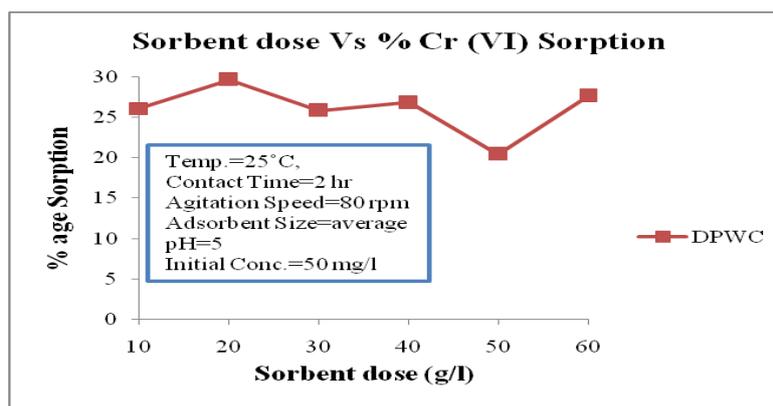


Fig-2 Effect of sorbent dose on Sorption

#### 4.3 Effect of initial chromium concentration on Fraction Removed.

Synthetic wastewater samples each 50 ml, having different concentrations of hexavalent chromium ranging 10 to 60 mg/l were taken separately in glass stoppered conical flasks, and then already optimized doses of adsorbents @ 20 g/l were added to each flask. At initial stages the contact time was 2 hour for average sized adsorbent at room temperature  $25^{\circ}\pm 1^{\circ}\text{C}$  maintaining pH 5. It was found that if we increase the initial chromium concentration the adsorption capacity of the adsorbents decreases; the optimized initial chromium concentration was found 10 mg/l. The reason of decrease in adsorption with increased amount of initial chromium concentration may be because of all sites of adsorbents get saturated and there remains no further scope of adsorption. The study graphs have been shown in Fig-3. (Sharma, et al, 2015)

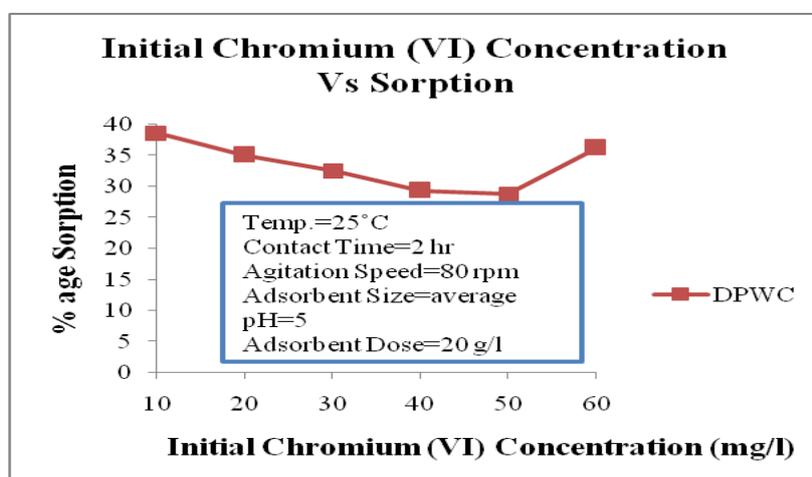


Fig-3 Effect of Initial Chromium (VI) Concentration on Sorption

#### 4.4 Effect of Contact Time on Fraction Removed.

Contact time plays a very crucial role in adsorption. Up taking of adsorbate species is found quite rapid in the initial stages of the contact period between adsorbent and adsorbate combinations but gradually become slower as soon as equilibrium reaches. Various synthetic samples in optimum pH 5, with average size, adsorbent dose 20 g/l, initial chromium concentration of 50 mg/l, at room temperature  $25^{\circ}\pm 1^{\circ}\text{C}$  having agitation speed 80 rpm at start, were tested for different periods of contacts right from 45 minutes to 270 minutes. The optimum value of contact time was observed 135 minutes and in first 45 minutes the adsorption recorded was more than 50%. The study analysis has been depicted in Fig.4.(Ayub, et al, 2014)

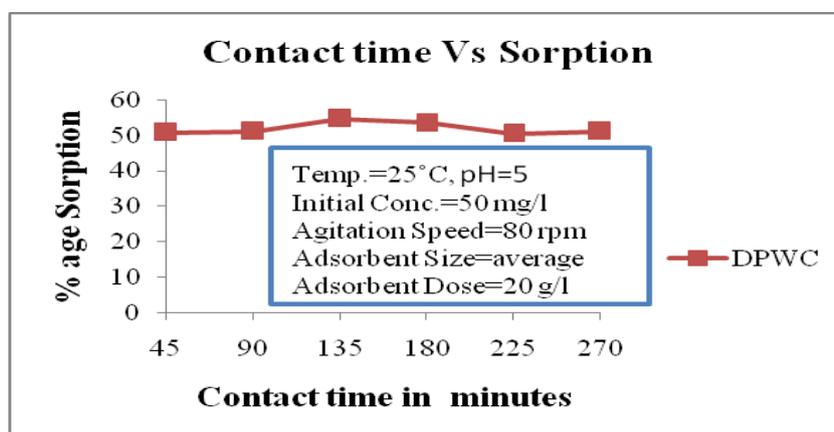


Fig- 4 Effect of Contact time on Sorption

#### 4.5 Effect of Adsorbent Particle size on Fraction Removed.

Indian Standard Sieved seven particle sizes < 75, 75, 150, 300, 425, 600, 1180  $\mu\text{m}$  of various adsorbents at pH 5, with 20 g/l doses, with initial chromium concentration 50 mg/l, at room temperature  $25^{\circ}\pm 1^{\circ}\text{C}$  with contact time 2 hour, at rate of 80 rpm starter agitation speed, were tested taking 50 ml samples in flasks. The study reveals that very very small size also not very effective for adsorption, also it takes longer time and extra labour to grind it to micron size, making the adsorbent costlier. The optimum size that is why has been taken in the range 300  $\mu\text{m}$ -600  $\mu\text{m}$  as an average size. The study graphs analysis is shown in Fig.-5.

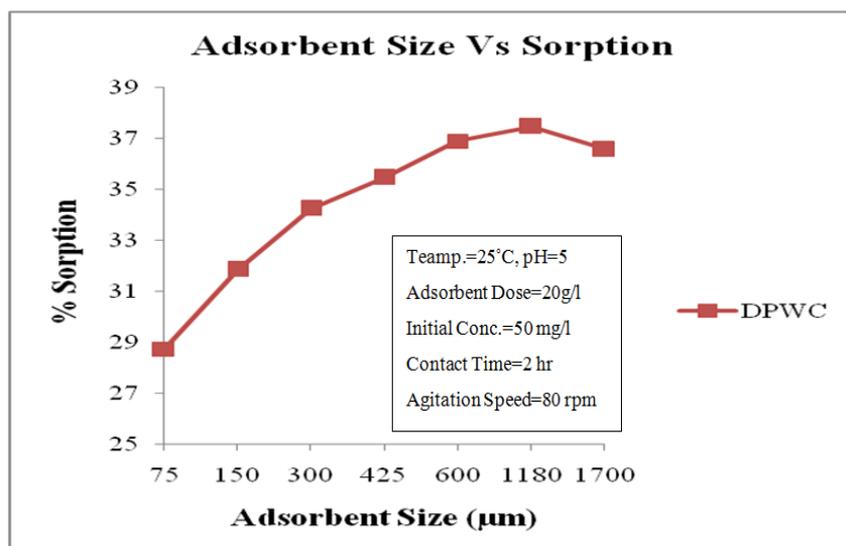


Fig-5 Effect of Adsorbent Size on Sorption

#### 4.6 Effect of Temperature on Fraction Removed.

Maintaining pH=5 using 1M Sulphuric acid and 1 M Sodium hydroxide, initial adsorbate concentration 50 mg/l optimized average sized adsorbent dose of 20 g/l, with contact time 2 hour, starter agitation speed 80 rpm in all experimental set ups, the effect of temperature variation, taking 50 ml of each sample was studied for varying temperatures from 10<sup>o</sup>C to 50<sup>o</sup>C by keeping them in temperature controlled oven ( Indian Scientific Instruments factory, Ambala Cant, India), were studied and after a series of observations literated temperature was verified as optimum temperature to obtain the optimum adsorption. The residual concentrations of chromium were subsequently determined. The maximum adsorption was found between 25<sup>o</sup>C to 30<sup>o</sup>C. At higher temperatures desorption starts taking place (Ayub, et al, 2014). The study analysis and graphs have been shown in Fig.-6.

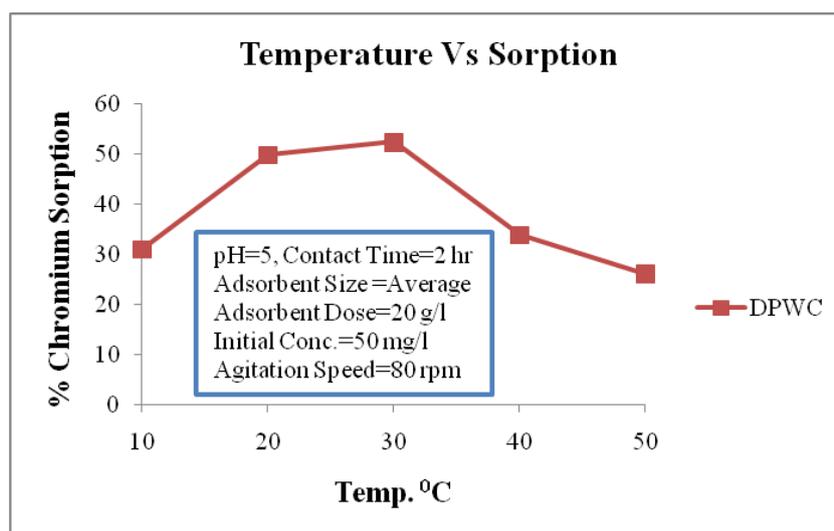


Fig-6 Effect of Temperature on Sorption

#### 4.7 Effect of startup Agitation Speed on Fraction Removed.

Maintaining optimized values of all affecting parameters such as pH=5, initial adsorbate concentration 50 mg/l, optimized average adsorbent grain sized from 300  $\mu\text{m}$  to 600  $\mu\text{m}$ , adsorbent dose of 20 g/l, with contact time 2 hour, various agitation speeds such as 80 rpm, 100 rpm, 120 rpm and 140 rpm, were tried in different samples each of 50 ml in flasks kept over different rpm set shaker. The observation revealed that more the agitation speed more greater was the adsorption but beyond a certain range of starter agitation speed i.e. 70 rpm to 90 rpm (optimum 80 rpm); the results were not very well, more over the higher speed was time, energy and maintenance consuming, resulting tear and wear and higher costs involved. The study graph so obtained is being depicted in Fig.-7.

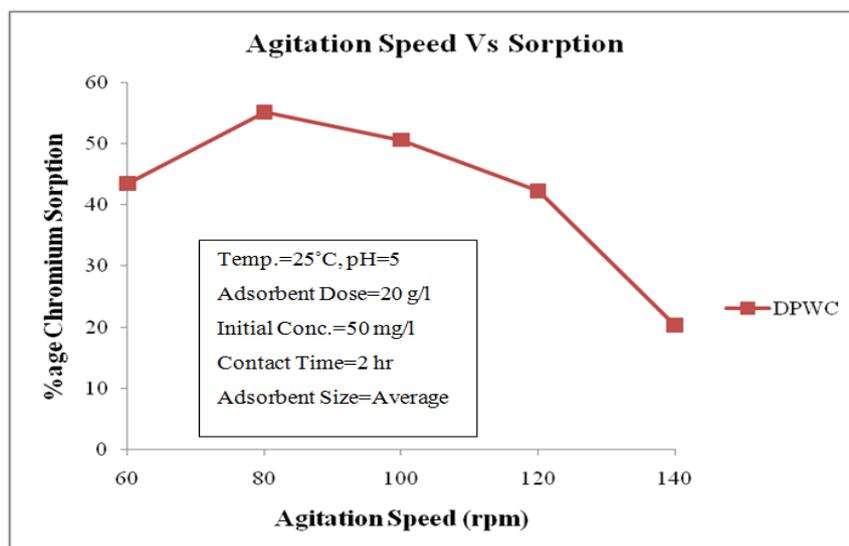


Fig-7: Effect of Agitation Speed on Chromium Cr (VI) Sorption

#### 4.8 Results of Optimized Affecting parameters

Taking all optimized values of all affecting parameters the batch study was made to obtain maximum adsorption of hexavalent chromium from synthetic wastewater using discarded potato from cold storages (DPWC) as adsorbent and the result so obtained has been depicted in Fig.-8.

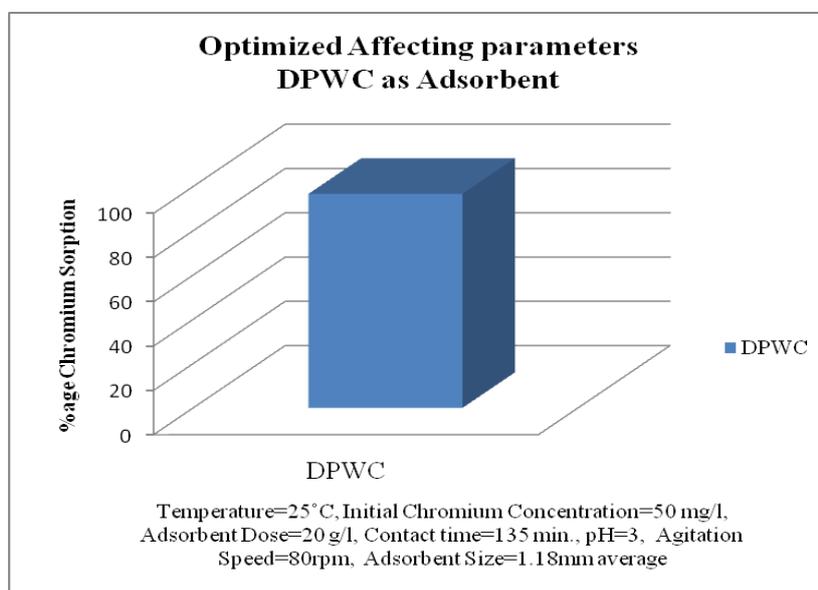


Fig-8, Result of Optimized Affecting parameters for various sorbants

#### Column Study

Though the information obtained from the results of batch experimental set ups for the application of adsorption to the removal of wastes constituents study were very useful, yet the column study provides the practical application in wastewater treatment technology for the design of continuous adsorption columns. When wastewater enters the top of adsorbent clean bed, most of adsorbate is initially removed in a rather thin layer of top of the column which is referred as the adsorption zone and this way the top layer gets saturated with adsorbate. As the adsorption continues, the adsorption zone increases downward through the bed resulting the whole packing adsorbent material to be the adsorption zone up to the bottom of the column and eventually, saturated and the adsorbate concentration effluent thereby increases.

A plot effluent adsorbate concentration versus time usually shows an S-shaped curve which is known as break through curve and the point where adsorbate concentration in effluent reaches its maximum allowable value is known as break through.

A burette of internal diameter 1.0 cm was used for the column study. The optimized dose of adsorbent as obtained from batch study was suspended in distilled water on magnetic stirrer for 15 minutes dispersing the particles by shaking it at agitation speed of 80 rpm so as to avoid conglomeration and then transferred in to the burette. The cotton was placed at top and bottom ends of the adsorbent in the column to avoid the loss with the liquid flow or floating. The flow rate was controlled by regulator used in outflow pipe maintaining 6.0 l/d. Concentrations of the influent stream to the burette and exit stream from the burette were determined using Atomic Absorption Spectrometer (Perkin Elmer, AAS, Pin AAcle 900K). The experimental set-up has been shown in figure-9. The column study was also conducted at the room temperature.



Fig-9: Experimental Set-up of Column Study and AAS

## V. Rate Kinetics

### 5.1 Thermodynamic Parameters

For adsorption data “Gibb’s free energy versus Temperature in Kelvin” graph was plotted and then from the linear equation  $\Delta G = (-\Delta S) x + \Delta H$  the values of various thermo dynamic constants are determined.

Fig-10 shows thermodynamic analysis for discarded potato waste from cold storage (DPWC). The values of  $\Delta G$ ,  $\Delta H$  and  $\Delta S$  were found 0.267, 0.288 and 0.0013. (Sharma, et al, 2012, a & b)

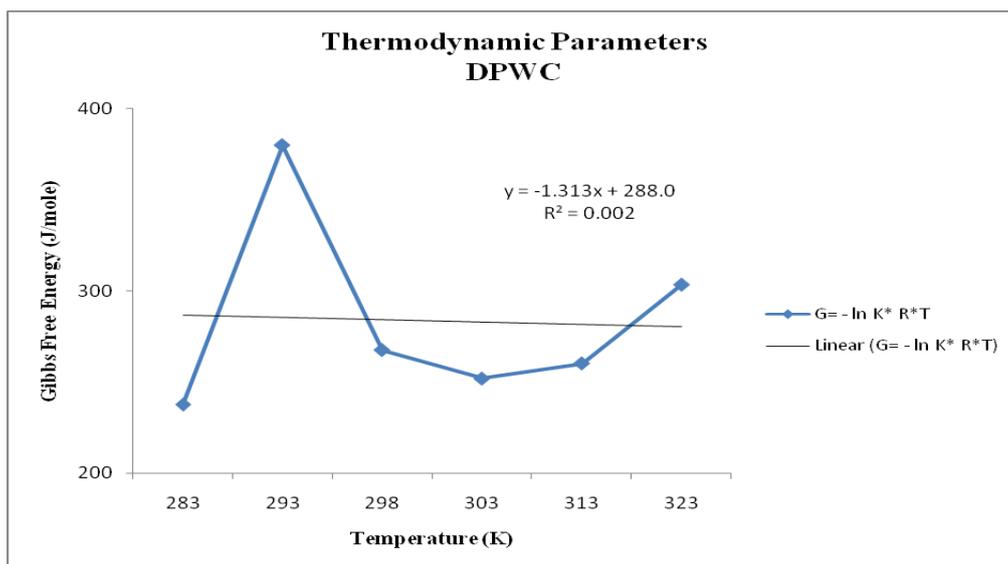


Fig-10 DPWC

### 5.2 Using Freundlich Isotherm

Keeping fixed initial chromium concentration 50 mg/l and varying doses of adsorbents in batch adsorption study, the data were fitted to the linearized Freundlich adsorption isotherm as

$$\text{Log}(q_e) = \text{log } K + 1/n \text{ log } C_e \quad (\text{Dada, A.O., et al, 2012})$$

Where,  $q_e = x/m$  = the amount of hexavalent chromium adsorbed per unit mass of adsorbent (mg/mg).

$C_e$  = the equilibrium concentration of aqueous solution.

$K$  = Constant of adsorption capacity

$1/n$  = Constant of adsorption intensity

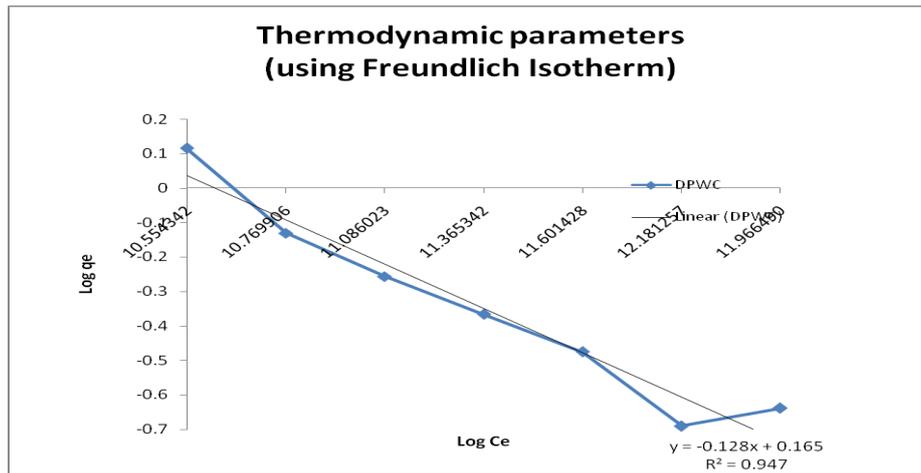


Fig.11 Plot of Freundlich Isotherm for (DPWC)

The values of the thermodynamic constants using Freundlich isotherm as drawn in Fig.-11 can be calculated using best linearized equations (Dada, A.O., et al, 2012)

$$y = -0.128x + 0.165$$

$K = 1.179 \text{ mg/g}$ ,  $1/n = 0.165$  for discarded potato waste from cold storages (DPWC) is  $<1$ , which indicates the favourable adsorption (Shrichand, 1994).

### 5.2 Using Langmuir Isotherms

Langmuir adsorption parameters were determined by transforming the Langmuir equation (Dada, A.O., et al, 2012)

$$q_e = (Q_o K_L C_e) / (1 + K_L C_e) \text{ in to linear form as } 1/q_e = 1/q_o + (1/q_o \cdot K_L) (1/C_e)$$

Where,  $C_e$  = the equilibrium concentration of adsorbate in mg/l

$Q_e$  = the amount of metal adsorbed per gram of the adsorbent at equilibrium (mg/g).

$Q_o$  = maximum monolayer coverage capacity (mg/g)

$K_L$  = Langmuir Isotherm Constant (L/mg)

The values of  $q_{max}$  and  $K_L$  were computed from the slope and intercept of the Langmuir plot  $1/q_e$  versus  $1/C_e$ .

The separation factor or equilibrium parameter  $R_L$ , which is a dimensionless quantity and its value indicates the nature of adsorption means if  $R_L > 1$  unfavorable, linear if  $R_L = 1$ ,  $0 < R_L < 1$  favourable and irreversible if  $R_L = 0$ .

$$R_L = 1 / [1 + [K_L \cdot C_o]]$$

Where,

$C_o$  = initial concentration of adsorbate = 50 mg

$K_L$  = Langmuir constant related to the energy of adsorption

All the above Langmuir constants were determined for all the adsorbents of which plots have been shown in Fig-12 below.

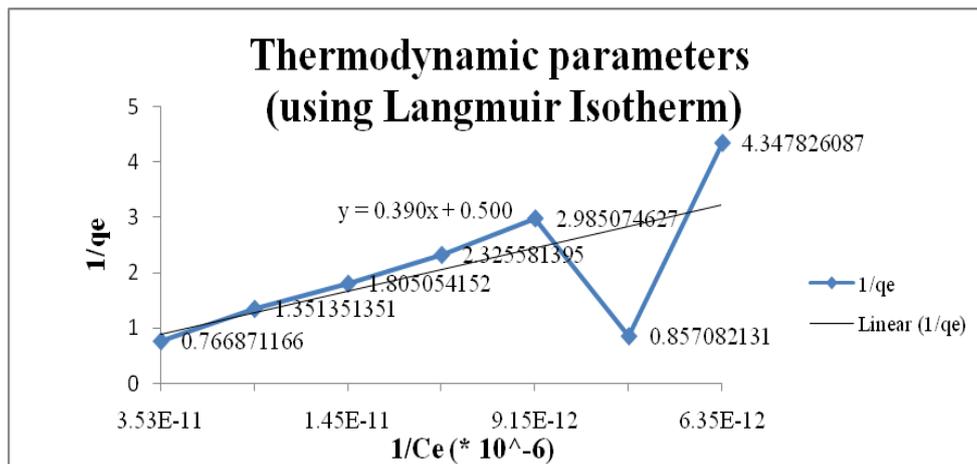


Fig-12 Plot of Langmuir Isotherm for (DPWC)



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