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Study of the Using Effect of Chromium Adsorption Remnants as Rodienticide for Sprague Dawley Rats *Rattus rattus* Accessing to Zero Residue Level (ZRL)

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Abstract:

This paper deals with the study of the potential of Pomegranate Peel Residue (PPR) (after extraction of antioxidant) to remove chromium (Cr^{+6}) ions from simulated synthesis aqueous solutions (SSAS) through various operating parameters using adsorption technique in sorption unit. Results show that the removal efficiency were 92.84% for chromium ions from (SSAS) and the removal efficiency decreased with increasing of initial concentration, pH and SSAS flow rate while the removal efficiency increased with increasing packing height of adsorbent material and feeding temperature. A mathematical model was proposed for an adsorption packed bed column with adsorbent media. Statistical model is also achieved to find an expression relates the overall operating parameters with the removal efficiency of chromium in a general equation. The waste of PPR (remaining after adsorbed chromium) were predestined to investigate the utilization of it in useful method. It can prepare a cheap rodenticide from these PPR waste without any further treatment. The results explain that the PPR adsorb chromium ions showed a good behaviour as rodenticide for sprague dawley rats Rattus rattus. By this way it can perform different benefits which are: remove the chromium ions polluted the water, get rid of agricultural waste PPR, in the same time, prepare a cheap and active rodenticide. It can discard more type of waste in a non-cost and eco-friendly method accessing to zero residue level (ZRL).

Key words: chromium, pomegranate peel, adsorption, residue, sprague dawley rats, Rattus rattus and ZRL

1. INTRODUCTION

Chromium is a chemical element with a steely-gray, lustrous, hard and brittle metal which takes a high polish, resists tarnishing, and has a high melting point. The name of the element is derived from the Greek word χρῶμα, chrōma, meaning colour because many of its compounds are intensely coloured. Chromium is the 22nd most abundant element in Earth's crust with an average concentration of 100 ppm. Chromium compounds are found in the environment, due to erosion of chromium-containing rocks and can be distributed by volcanic eruptions. Chromium is mined as chromite ($FeCr_2O_4$) ore. About two-fifths of the chromite ores and concentrates in the world are produced in South Africa, while Kazakhstan, India, Russia, and Turkey are also substantial producers. **(Wikipedia, 2015)**. Naturally occurring chromium is composed of three stable isotopes; ^{52}Cr, ^{53}Cr and ^{54}Cr with ^{52}Cr being the most abundant (83.789 % natural abundance). 19 radioisotopes have been characterized with the most stable being ^{50}Cr with a half-life of (more than) 1.8×10^{17} years, and ^{51}Cr with a half-life of 27.7 days. All of the remaining radioactive isotopes have half-lives that are less than 24 hours and the majority of these have half-lives that are less than 1 minute. This element also has 2 meta states **(Wikipedia, 2015)**. In aqueous systems the chromium can be mainly found as both Cr^{+3} and Cr^{+6}. The relation between Cr^{+3} and Cr^{+6} strongly depends on pH and oxidative properties of the location, but in most cases, the Cr^{+3} is the dominating species, although in some areas the ground water can contain up to 39 μg/liter of total chromium of which 30 μg/liter is present as Cr^{+6}. Trivalent chromium Cr^{+3} ion is considered a bio-element and it is possibly required at low concentrations since it plays an important role in the sugar and lipid metabolism of plants and animals. In larger amounts and in different forms, chromium can be toxic and carcinogenic. The most prominent example of toxic chromium is hexavalent chromium Cr^{+6} **(Radjenovic and Medunic, 2015; Wikipedia, 2015)**. Cr^{+6} is directly toxic to bacteria, plants and animals (Richard and Bourg, 1991). Due to the sever toxicity of Cr^{+6}, the USEPA has set the maximum contaminate level (MCL) for Cr^{+6} in domestic water supplies to be 0.05 mg/l. Cr^{+6} is typically present as anion and its direct precipitation is not usually practical **(El-Shafey, 2005)**. Chromium is used widespread in many industries such as electroplating, refractory of material, leather tanning, metal finishing, metallurgy, cement preservations, wood preservative, nuclear power plants, textile, dye and pigment, steel fabrication, catalysts, synthetic ruby and the first laser chromate preparation, etc. So, Chromium is an important toxic pollutant released to natural water from effluents of these industries **(El-Shafey, 2005; Radjenovic and Medunic, 2015)**. Various conventional methods have been employed for the treatment of heavy metal wastewater, such as chemical precipitation, electrolysis, ion exchange, solvent extraction, liquid membrane and reverse osmosis **(Chaudhuri and Azizan, 2012)**. These processes have significant disadvantages, technical and/or economic constraints for instance, incomplete removal, high-energy requirements, and produce a voluminous toxic sludge which may pose disposal problems **(El-Shafey, 2005; Radjenovic and Medunic, 2015)**. Although bioremediation of heavy metals by bacterial fungal or algal biomass can be considered as a suitable method to remove the heavy metals from aqueous solutions or industrial wastewater but the most significant limitation of this method is that microbial growth is inhibited when the concentrations of metal ions are too high or when significant amount of metal ions are adsorbed by microorganisms **(Hasan et. al., 2008)**. Adsorption technique with activated carbon has been recognized as a potential alternative, most commonly and efficacious methods in wastewater treatment and it represented the green chemistry direction to reduce the use of toxic and carcinogenic solvents in remediation processes of wastewater. Though activated carbon adsorption has high efficiency, but the problems related with regeneration and reuse in addition to the higher capital investment made the removal treatment less attractive and suffers from many limitations **(Abbas, 2014a)**. So, there is a continuing search for cheap, high capacity adsorbents instead of activated carbon for heavy metal ions removal **(Radjenovic and Medunic, 2015)**. Recently, great attention has been paid to the adsorbents based on natural materials, such as rice husk which is used as adsorbent media for adsorption of many pollutants such as heavy metals **(Abbas et. al., 2012; Abbas et. al., 2013a; Abbas and Abbas (2013a), (2013b), (2013c) and (2013d); Abbas and Abbas 2014a)**, dyestuffs **(Abbas and Abbas 2014b)**, pesticides **(Abbas and Al-Madhhachi, 2015)**, phenols **(Abbas et. al. (2013b), (2013c))** non-organic toxins **(Abbas, 2014b)** and cesium as a radioactive waste **(Abbas, 2014a)**, banana peel for adsorption of cyanide **(Abbas et. al., 2014)** and thorium **(Abbas, 2013)** and pomegranate peel **(Abbas, 2014c)** because it is available, very inexpensive and ability to remove different types of contaminants. Pomegranate peel also can be used as a natural adsorbent like rice husk and banana peel in the same way, thus the pomegranate peel usage will decrease the cost of wastewater treatment considerably and would represent practical benefit of agriculture waste residue in disposal toxic matter in save way **(Abbas, 2014d)**. The objective of this paper was to evaluate and determine the efficiency of pomegranate peel residue (after extraction of antioxidant) for recovering of chromium from simulated synthetic aqueous solutions and exploit from the remaining in eco-friendly method accessing to zero residue level (ZRL).

2. EXPRIMENTAL WORK

2.1: Materials

2.1.1: *Pomegranate peel residue* (*adsorbent media*)

Pomegranate Peel Residue (PPR) were collected after extraction of antioxidant from the Fresh Pomegranate Peel (FPP) as described in **(Abbas, 2014d)**.

2.1.2: *Stock solutions*

In order to avoid interference with other elements in wastewater, the experiments in this study were carried out using simulated synthetic aqueous solution (SSAS) of different chromium concentrations. 1000 mg/l stock solution of aluminium was prepared by dissolving known weight of Potassium dichromate $K_2Cr_2O_7$ in one litre of double distilled water, all solutions using in the experiments were prepared by diluting the stock solution with double distilled water to the desired concentrations for the experimental work of this investigation. The chromium concentrations were measured using spectrophotometer method using spectrophotometer thermo – genesys 10 UV, USA.

2.2: Sorption unit

Fixed bed column of continuous mode experiments were conducted in order to test chromium hexavalent Cr^{+6} removal by treated SSAS of above chromium at desired concentration with the various bed heights of the adsorbent media PPR using different flow rates of SSAS of chromium at various pH. The pH value was adjusted using 0.1 N NaOH and 0.1 N HCl solutions where the flow direction is downward by gravity. The sorption unit consists of two glass container of SSAS of chromium one for inlet and another for outlet each of (1 liter) capacity. Glass column has 2.54 cm ID and 150 cm height. The sorption column packed with adsorbent media to a height of (10, 20, 30, 40, 50, 60, 70, 80, 90 and 100 cm) supported from the top and the bottom by glass hollow cylinder layer, each cylinder have (0.5 cm ID, 0.1 cm thickness and 1 cm long). Before starting the runs, the packed bed sorption column was rinsed by double distilled water down flow through the column. The PPR is packed in the column to the desired depth, and fed to it as slurry by mixing the media PPR with distilled water in order to avoid the formation of air bubbles inside the media. After the packed bed sorption column was accommodation and putting the required amount of adsorbent media, the adsorption process started by allowing the chromium SSAS of required concentration and pH down flow through the sorption column from inlet container by gravity at a precise flow rate in experiment which is adjusted by the valve. To determination the best operational conditions, the experiments were carried out at a temperature between (20–45°C), various pH values which are (1–8) and initial feed concentrations of SSAS of different chromium which are between (1–100) mg/l and at different flow rates which are between (5–100) ml/min for chromium initial feed concentration. Outlet samples in each experiment were collected every 10 minutes from the bottom of packed column and the unadsorbed concentration of chromium in SSAS was analyzed by spectrophotometer.

3. MATHEMATICAL MODEL

Fixed bed dynamics are describing by a set of convection-diffusion equations, coupled with source terms due to adsorption and diffusion inside adsorbent particles. Inside the particle, molecules of adsorbate diffuse into the inner portions of particle via surface diffusion, pore diffusion, or both. The solution of these equations will give rise to the prediction of the needed concentration distribution. This investigation focuses on understanding the mechanism of both surface diffusion and pore diffusion. To formulate a generalized model corresponding to the dispersion flow, surface diffusion and pore diffusion mechanism, following assumptions are made:

1. The system operates under isothermal conditions.
2. The equilibrium of adsorption is described by Langmuir isotherm.
3. Intraparticle mass transport is due to Fickian diffusion, and it is characterized by the pore diffusion coefficient, D_p and the surface diffusion, D_s.
4. Mass transfer across the boundary layer surrounding the solid particles is characterized by the external-film mass transfer coefficient, k_f.
5. Film transfer resistance for mass transport from the mobile to the stationary phase.
6. Local adsorption equilibrium between the adsorbate adsorbed onto the adsorbent surface and the adsorbate in the intraparticle stagnation fluid.
7. Both surface and pore diffusion are included in the mass transport mechanism.
8. Axial dispersion.

3.1: Simulation Technique

Equations used in simulation technique represent a set of simultaneous, nonlinear, partial differential equations (PDEs) that can be solved numerically. The discretatization was applied to space coordinates (Z and r) to convert the PODs to a set of ordinary differential equations (ODEs). The resulting ODEs can be solved using an existing ODE solver provided by **(Abbas et. al. (2013b), (2013c))**.

4. RESULTS AND DISCUSSION

The ability of PPR to remove chromium ions from SSAS in fixed bed column of continuous mode at various parameters which are pH's of SSAS of heavy metal ion (*pH*), height bed of adsorbent media (PPR) (*l*), flow rates of SSAS (*F*), SSAS temperature (T_{feed}) and time of treatment (t) was investigated. The experiments were achieved by varying all above parameters for different initial concentrations (C_o) of SSAS of chromium. Thus, the results obtained are explained below.

4.1: Effect of Initial Concentration

The results showed that using PPR as an adsorbent material, the percent removal of chromium ions was decreased when the initial concentration (C_o) of SSAS of chromium ions was increased at constant other variables as shown in **Figures 1**. This can be explained by the fact that the initial concentration of chromium ions had a restricted effect on chromium ions removal capacity; simultaneously the adsorbent media had a limited number of active sites, which would have become saturated at a certain concentration. This was lead to increase in the number of chromium ion molecules competing for the available functions groups on the surface of adsorbent material. Since the solution of lower concentration has a small amount of chromium ions than the solution of higher concentration of it, so the percent removal was decreased with increasing initial concentration of chromium ions. For adsorbent media, higher percent removal was 92.42 % for chromium ions Cr^{+6}, at initial chromium ion Cr^{+6} concentration of 1 mg/l, so adsorbent material was found to be efficient to chromium ions removal from SSAS and wastewater.

4.2: Effect of pH

The results showed that using adsorbent material, the percent removal of Cr^{+6} ions was decreased when the pH of SSAS increased at constant other variables, **Figures 2**. It is well recognized that the pH of the aqueous solution is an important parameter in affecting adsorption of heavy metal ions. High adsorption of Cr^{+6} ions at low pH can be explained in both terms; the species of the metal and the adsorbent surface. For this case, at low pH, i.e. acidic conditions, the surface of the adsorbent PPR becomes highly protonated and favours adsorb of Cr^{+6} ions in the anionic form. With increasing the pH of aqueous solution, the degree of protonation of the surface reduces gradually and hence adsorption is decreased. Furthermore, as pH increases there is competition between hydroxide ion (OH^-) and Cr^{+6} ions, the former being the dominant species at higher pH values. The net positive surface potential of sorbent media decreases, resulting in a reduction the electrostatic attraction between the (*sorbent*) metal ion species and the (*sorbate*) adsorbent material surface PPR, with a consequent reduced sorption capacity which ultimately leads to decrease in percentage adsorption of metals ion **(Abbas, 2013; Abbas, 2014a)**.

4.3: Effect of Adsorbent Media Bed Height

The results elucidated that when the adsorbent media bed height was increased, the percent removal of all Cr^{+6} ions was increased too at constant other variables as shown in **Figures 3**. The increased of bed height (l) meaning increased in the amount of adsorbent media, thus increasing the surface area of adsorbent material, hence increased the number of active sites in the adsorbent material surface i.e. increased the availability of binding sites for adsorption and consequently increase the Cr^{+6} ions removal capacity on PPR. This lead to increase the ability of adsorbent media to adsorb greater amount of Cr^{+6} ions from SSAS at different initial concentrations and ultimately the percent removal of Cr^{+6} ions increased.

4.4: Effect of Flow Rate

The results illustrated that when the flow rate of SSAS of Cr^{+6} ions was increased, the percent removal was decreased at constant other variables as shown in **Figures 4**. This may be due to the fact that when the flow of SSAS of Cr^{+6} ions increasing, the velocity of solution in the column packed with the adsorbent media PPR was increasing too, so the solution spend shorter time than that spend in the column while at low flow rate, the SSAS of Cr^{+6} ions resides in the column for a longer time, and therefore undergoes more treatment with the adsorbent media, thus the adsorbent media uptake low amount of Cr^{+6} ions from SSAS of Cr^{+6} ions for high flow rate, therefore the percent removal of Cr^{+6} ions was decreased when the flow rate was increased.

4.5: Effect of Feed Temperature

The results demonstrated that when the temperature of feed which was SSAS of Cr^{+6}ions was increased, the percent removal of heavy metal ions was increased too at constant other variables as shown in **Figures 5**. The effect of temperature is fairly common and increasing the mobility of the metal cation. Furthermore, increasing temperature may produce a swelling effect within the internal structure of the adsorbent media enabling Cr^{+6} ions to penetrate further. It was indicated that Cr^{+6} ions adsorption capacity increased with increasing feed temperature from 20 to 45°C. This effect may be due to the fact that at higher temperature an increase in active sites occurs due to bond rupture.

4.6: Effect of Treatment Time

The results demonstrated that when the treatment time of SSAS of Cr^{+6} ions increased the percent removal of Cr^{+6} ions increased too at constant other variables as shown in **Figures 6**. This may be due to the fact that when the time of treatment of SSAS of Cr^{+6} ions increasing and the velocity of solution in the column packed with the adsorbent material was remaining constant, the solution spend longer time than that spend it when the time of treatment decreased, so the adsorbent material uptake more amount of Cr^{+6} ions from SSAS, therefore the percent removal of Cr^{+6} ions from SSAS of was increased.

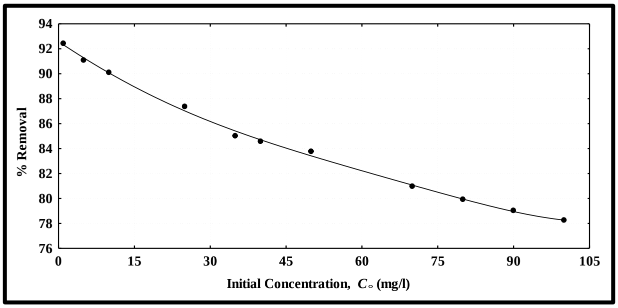

Figure 1: Effect of initial concentration ($C_\circ$) on the percent removal of Cr^{+6} ions @ T_f =45°C, l = 1 m, pH=1, t=60 min. and F=5 ml/min.

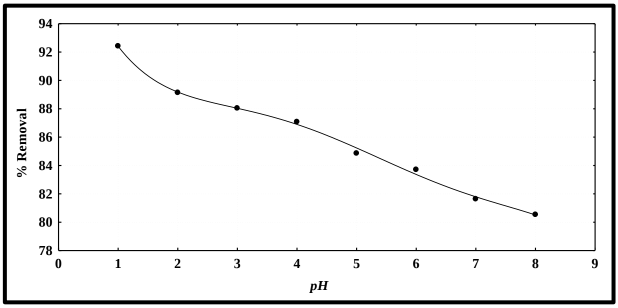

Figure 2: Effect of pH on the percent removal of Cr^{+6} ions @ $C_\circ$= 1 mg/l, T_f =45°C, l = 1 m, t=60 min. and F=5 ml/min.

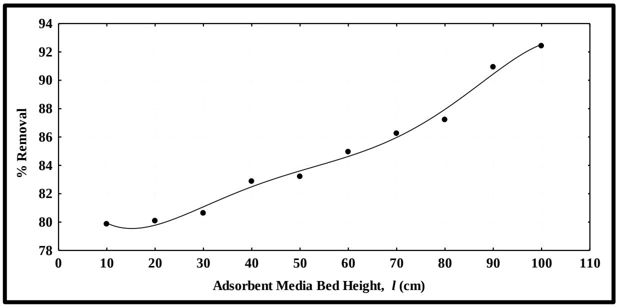

Figure 3: Effect of adsorbent media bed height (l) on the percent removal of Cr^{+6} ions @ $C_\circ$= 1 mg/l, T_f =45°C, pH=1, t=60 min. and F=5 ml/min.

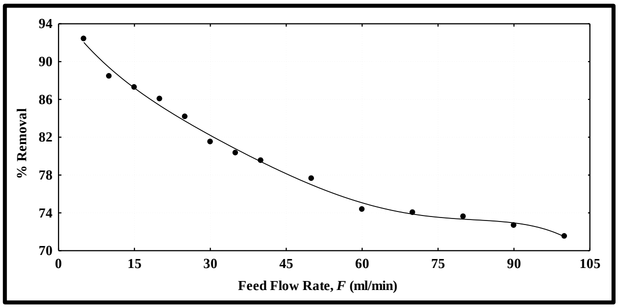

Figure 4: Effect of aqueous solution flow rate (*F*) on the percent removal of Cr^{+6} ions @ $C_{\circ}$= 1 mg/l, T_f =45°C, *pH* =1, *l* = 1 m and *t*=60 min.

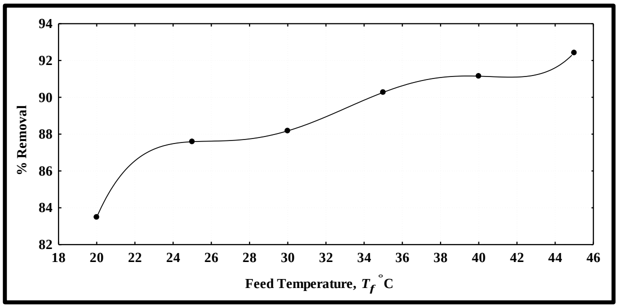

Figure 5: Effect of feed temperature (T_f) on the percent removal of Cr^{+6} ions @ $C_{\circ}$= 1 mg/l, *pH*=1, *l* = 1 m, *t*=60 min. and *F*=5 ml/min.

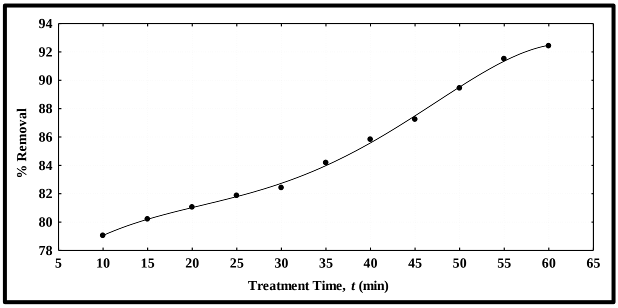

Figure 6: Effect of treatment time (*t*) on the percent removal of Cr^{+6} ions @ $C_{\circ}$= 1 mg/l, T_f =45°C, *pH*=1, *l* = 1 m, and *F*=5 ml/min.

5. STATISTICAL MODEL

A statistical model was carried out on the experimental results obtained from this study. Regression Analysis and **π** Theorem was adopted to maintain a relation between the percent removal of Cr^{+6} ions and the feed temperature, flow rate, pressure, pH of feed solution, initial concentration of Cr^{+6}ion, adsorbent media bed height, treatment time and column diameter. These relations are shown in **equation 1**, which has a correlation coefficient (R^2) 0.9954.

$$\%\boldsymbol{R} = \mathbf{1.741} \times \mathbf{10}^{-7} \left(\frac{\rho_{sol}}{C_{\circ}}\right)^{0.2574} . \left(\frac{l}{d}\right)^{0.2594} . \left(\frac{T_f . C_{P_{sol}} . t}{\vartheta}\right)^{0.2406} . \left(\frac{K_{sol} . T_f}{\gamma . u}\right)^{0.2363} . \left(\frac{1}{pH}\right)^{0.1039} \qquad (1)$$

where:	$\%\boldsymbol{R}$	Percent Removal of Chromium ions from SSAS
	ρ_{sol}	Density of SSAS, (kg/m^3)
	$C_{\circ}$	Initial Concentration of Chromium , (kg/m^3)
	l	Adsorbent Material Bed Height, (m)
	d	Internal Diameter of Sorption Column, (m)
	T_f	Feed Temperature, (K)
	$C_{P_{sol}}$	Heat Capacity of SSAS, (kJ/kg. K)
	t	Treatment Time, (s)
	ϑ	kinematic viscosity of SSAS, (m^2/s)
	K_{sol}	Thermal Conductivity of SSAS , (W/m. K)
	γ	Surface Tension, (N/m)
	u	Velocity of SSAS, (m/s)

6. UTILIZATION OF POMEGRANATE PEEL AFTER USES

Huge amount of PPR was lingered after using it in removal of Cr^{+6} ions from SSAS as illustrated above. The remaining waste of PPR after removal of chromium ions from SSAS by adsorption process were collected, segregated and classified according to its contain of chromium and utilization from these vestiges to prepare a simple and active rodenticide. Twenty groups (ten for male and ten for female) of an outbred multipurpose breed of albino rat which was Sprague dawley rats (*Rattus rattus*) were used in this test; each group has ten rats besides another animal control group to compare the results. Before the test is beginning, the rats were left for one week in clean and convenient cages and nurtured with normal feed to make sure that it's were not suffer from anything leading to death **(Ibrahim et. al., 2014; Abbas and Al-**

Madhhachi, 2015). The PPR waste were mixed with little amount of fruit to give sweet test and feed to the rats directly as rodenticide without any further treatment. The results were fate the rats in different periods and the lethal dose for chromium was also calculated as shown in **Table 1**.

The inorganic pesticide, is one of the most important type of pesticide used in rodent control, and perhaps the most important components used in this field are chromium, arsenic, barium, bismuth, boron, cadmium, chromium, cobalt, copper, iron, lead, lithium, manganese, mercury, nickel, tin, and zinc inorganic compounds. There are several ways to use these rodenticide in rodents places passing where the pesticide inter to the mouth of the rodent when its clean parts of their bodies, or through mixing with attractive materials to rodent like PPR or bread or vegetables pieces. Characterized compounds of this rodenticide being used successfully to combat rodent and where they are used as toxin infectious for their secured effectiveness, and severity of toxic, in addition to non-degradable and remain for a long time without changing their composition. There is more than one way to interpret the mechanical toxic effect of these elements compounds on rodents, which are:

1 – The compounds of these elements operate to prevent the phosphorylation process of Adenosine diphosphate (ADP) material in the process of oxidative phosphorylation and thus preventing the formation of Adenosine triphosphate ATP material necessary in the storage of energy required for the rodent.

2 – Theses compounds linked with many important enzymes required from the rodent body and discourage work, and these enzymes are dehydrogease, cytochrome oxidese, phosphatase where these inhibition process lead to an imbalance in the processes of chemical or biological eventually lead to the death of the rodent.

3 – The high concentrations of elements compounds lead to a deposition holistic and very fast for a protein in a living cell because it attack the sulphur bonds, which plays an important role in keeping the distinctive shape of the protein, it observed that the effect of these elements compounds are concentrated in the epithelium in the central gut of rodents.

4 – The low concentrations of these elements compounds leading to excessive drowsiness and lack of movement and the collapse of immune system and the failure of the ability to reproduce (infertility) of the rodent.

Table 1 LD_{50} (mg of Pesticide/kg of rat body) of Chromium Calculated in this Study

LD_{50} for male (mg/kg)	LD_{50} for female (mg/kg)	Standard Dose (mg/kg)
25.5	16.75	13 to 19 for female 21 to 28 for male **(Gad et. al., 1986)**

7. CONCLUSIONS

The following conclusions can be drawn:

1. PPR showed a good ability to remove chromium from SSAS using fixed bed adsorption unit. So, it could be recommended for removal of heavy metal ions from treatment plant wastewater instead of other material like activated carbon because it is valid, cheaper, economical, easy and simplicity for using, and has a high ability to adsorb heavy metal ions, and can be used finally in another benefit uses.
2. Maximum removal of Cr^{+6} ions were 92.42% at initial concentration of 1 mg/l.
3. The percentage removal of Cr^{+6} ions was increased with decreeing pH, flow rate of SSAS and initial concentration of Cr^{+6} ions while the percentage removal was increasing with increasing of treatment time and the height of adsorbent material PPR.

4. It can be prepared a good rodenticide for rodent control from the residual samples of PPR that adsorb the Cr^{+6} ions above from SSAS.

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