

# Adsorption of Heavy Metals from Industrial Wastewater using Palm Date Pits as Low Cost Adsorbent

Arshad I. Esmael, Minerva E. Matta, Hisham A. Halim, Farouk M. Abdel Azziz

**Abstract-**The removal of heavy metals from industrial wastewater is of great concern as heavy metals are non-biodegradable, toxic elements that cause serious health problems if disposed of in the surrounding environment. In this study, the removal of three heavy metals: copper, hexavalent chromium, and iron through adsorption using palm date pits was studied. Palm date pits are considered a low cost source of activated carbon. Heavy metals adsorption was studied for individual elements, and for industrial wastewater samples collected from a tannery and an electroplating factory. The kinetic studies showed that Cu, Cr<sup>+6</sup>, and Fe were adsorbed very rapidly within the first 30 minutes, while equilibrium was attained within 90 min, the optimum pH range for their adsorption was found to be (4.5-6.5), depth of adsorbent layer (70-90)cm, and particle size(0.5-0.75)mm. The adsorption capacity and removal efficiency for individual elements reached 89.17% for Cu, 71.30% for Cr<sup>+6</sup>, and 85.17% for Fe respectively. As for the removal of heavy metals from industrial wastewater collected from the tannery, removal efficiency reached 85.17% for Cu, 65.42% for Cr<sup>+6</sup>, and 87.03% for Fe and for the electroplating factory effluent: 82.857% for Cu, 61.65% for Cr<sup>+6</sup>, and 89% for Fe. The equilibrium sorption data for synthetic wastewater at temperature 27± 2°C was described by both the Langmuir and Freundlich isotherm models. Experimental data were better fitted to the Freundlich equation rather than to the Langmuir equation.

**Keywords:** Adsorption; heavy metals; industrial wastewater; low cost adsorbents; palm date pits.

## I. INTRODUCTION

The conventional methods for the removal of toxic metal ions from wastewater are not economically feasible especially at low concentrations. Adsorption as compared to other methods has appeared to be a simple attractive process in view of its high efficiency, easy handling and cost effectiveness as well as the availability of different adsorbents. In addition, the recovery of pure metal for recycle as well as reuse of the adsorbent are the added advantages [1]. In the recent years, the search for low-cost adsorbents that have metal-binding capacities has intensified. Materials locally available in large quantities such as: agriculture waste, natural materials or industrial by-products can be utilized as low-cost adsorbents. Some of these materials can be used as adsorbents with little processing.

Conversion of these materials into activated carbon, would improve economic value, help industries reduce the cost of waste disposal and provide a potential alternative to activated carbon [2]. Activated carbon can be prepared from a variety of carbon containing materials such as sawdust, rice hulls, palm shell, peat, coal, coconut shell, spent tea leaves ... etc.,[3,4]. The abundance of these materials in most continents of the world and their low cost make them suitable as adsorbents for the removal of various pollutants from wastewaters. The possibility of utilizing some low-cost adsorbents which would be an effective and economical option as peanut husk charcoal, fly ash, and natural zeolite was studied for the removal of copper (Cu+2) and zinc (Zn+2) from industrial wastewater. Batch isotherm experiments were carried out to evaluate the removal efficiency of these metals at various pH values (3-11), at heavy metal concentration of 10-100 mg/l using adsorbent dose of 5g/l. The kinetic studies showed that copper and zinc ions were adsorbed onto the different adsorbents very rapidly within the first 30 minutes, while equilibrium was attained within 2 hours, and the optimum pH range was found to be 6-8, the removal efficiency for copper and zinc ions could be from 30 to 98% of heavy metals using these different adsorbent [5]. The applicability of using locally available materials -Iraqi porcellanite rocks and activated carbon prepared from Iraqi palm date pits- and commercially activated carbon was evaluated for the adsorption of methylene blue dye using batch experiments and continuous system (fixed bed). Batch study showed that equilibrium isotherms for all the adsorbents used in the study are of favorable type and the maximum adsorption capacity was achieved by porcellanite followed by commercial AC and date-pits AC, with a very slight difference between porcellanite and commercial AC. The equilibrium data were well fitted to the Langmuir equation [6]. The adsorption of copper (II) and cadmium (II) ions from aqueous solutions on activated carbon prepared from rice hulls (ACRH) was investigated with varying pH values, activated carbon dosage, contact time, initial metal concentration and solution temperature. The optimum values of pH, ACRH dosage and contact time were determined to be 5-8, 0.5 g ACRH/25 mL solution and 60 minutes respectively for the adsorption of Cu (II) ions and 5-8, 1.5 g ACRH/25 ml solution and 60 minutes for the adsorption of Cd (II) ions[3]. Wastewater from dyeing industry was treated by activated carbon prepared from coconut tree sawdust at different agitation times, carbon dose and pH.

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The adsorption equilibrium for color removal was reached within 60 min., the study proved that pH did not have any significant effect on color removal. Removal of color, chemical oxygen demand (COD), biological oxygen demand (BOD), total solids and total hardness increased with increase in carbon dose. The use of sawdust as a source of activated carbon would be economical, since sawdust is a waste product and available in large quantities [4]. Barley straw was used as a raw material to produce activated carbon. The study investigated the removal of methylene blue dye from simulated water. The results showed that as the amount of the adsorbent was increased, the percentage of dye removal increased accordingly. Higher adsorption percentages were observed at lower concentrations of methylene blue. Optimum pH value for dye adsorption was determined as 7. Maximum dye was requested within 90 min after the beginning for every experiment [7]. Physicochemical processes were combined and granular activated carbon was used to remove phenol from pharmaceutical wastewater up to the allowable limit for reuse purpose. The objective of the laboratory investigation was to study the removal of color, Total Dissolved Solids (TDS), Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), turbidity and phenol and bring them up to the allowable limits for reuse purposes. Efficiency of coagulation, flocculation, sedimentation, sand filtration followed by activated carbon adsorption was determined. Activated carbon adsorption further reduces phenol, TDS, TSS, BOD, and COD up to 99.9%, 99.1%, 21.4%, 81.3% and 71.1% respectively. High removal of color was observed after activated carbon adsorption [8]. Wastes of the Iraqi date palm tree were used to study the removal of heavy metal cations (Cu+2, Cd+2& Zn+2) from simulated artificial wastewater using batch adsorption process. The dried parts of the date palm wastes were grinded to ≤ 1 mm in size and used directly in different adsorbent/metal ion ratios, starting with metal ion concentration of (1000ppm). Experiments were carried out at room temperature 25°C and pH value of (5-6). Date palm wastes succeeded to achieve 90% removal for Cu+2 ion, 57.5% for Cd+2 ions and 37.5% for Zn+2 ions within (60 min) contact time at adsorbent loading ratio of 30 g/l. Removal values for mixed ions were lower due to competition and interaction between ions, (80% Cu+2, 51% Cd+2 and 33% Zn+2) [9].

**II. EXPERIMENTAL WORK**

**A Materials**

**Adsorbent**

Palm date pits, also called pips, stones, kernels, or seeds are an integral part of the date fruit in the order of- depending on variety and quality grade- 6-12% of its total weight. Almost any organic matter with a large percentage of carbon could theoretically be activated to enhance its adsorptive characteristics [10]. Experiments conducted show that palm-date pits contain approximately 55-65% of carbohydrates. In view of this, high-grade activated carbon can be obtained from palm-date pits due to their high carbon content [11]. Palm date pits are shown in Fig.1.

**Production of Activated Carbon from Date Pits**

First, palm date pits were analyzed to study its composition, results are indicated in Table1.



Fig.1. Date Pits Used in the Present Study

**Table 1. Chemical Composition and Physical Structure for Palm Date Pits**

Chemical Composition	mg/Kg
Carbohydrates	550
Ash	1.17
Oil	700
Crude Fiber	9.18
Protein	5.27
Ether Extract (crude fat)	1.73
Ca	507
Mg	641
Fe	135
Cu	5.44
Zn	40.7
Al	6.88
Moisture	7%

Note: Tests were conducted in the laboratory materials for the National Research Center (NRC).

The preparation of activated carbon from the palm-date pits involves three steps: boiling, soaking and a combined step of carbonization and activation of pits. In the first step, the pits were boiled in a pressure cooker for about three hours at three stages with one hour interval for each stage. Boiling water was substituted with fresh water at the end of each interval. Then, the boiled pits were dried in an electric oven at 110°C for 24 hours and crushed with a hammer. The second step is soaking. The solution commonly used in the chemical treatment process of producing activated carbon is Phosphoric Acid (H3PO4). During this step, a concentrated phosphoric acid (85% H3PO4) was poured carefully into the containers containing the crushed pits with an impregnation ratio of 1 g of pits impregnated in 2 ml of 85% H3PO4 at 25 °C and for an impregnation time of 24 hours [12]. Then, the soaked pits were left in air for partial dryness and then dried in an oven at 120°C for one hour. The final step is the process of carbonization and activation. The date pits were placed on a metallic plate and subjected to an average temperature of 400°C for 2 hours [11].



The product was put in a flask and distilled hot water was added. Then, the pits were washed with distilled boiling water until the desired pH was achieved (6.5), and then the product was dried in an electric oven at 120°C overnight in order to remove any undesired moisture within the particles. The produced activated carbon is shown in Fig. 2.



Fig. 2. Palm Date Pits Produced

**Adsorbates**

**Column Adsorption System Set-up: Column Adsorption**

To determine the adsorption capacity of palm date pits under various conditions, a series of column adsorption experiments were performed in up-flow mode (expanded bed). A schematic diagram of the experimental model and process flow used in the study is shown in Fig.3. The model is composed of a column made of a PVC tube 1.00 m height and 0.05 m inner diameter. At the bottom of the column, a stainless steel sieve was fixed followed by a layer of glass wool beads. A known quantity of the prepared activated carbon was packed in the column to yield the desired bed heights of the adsorbent, and then an upper retaining sieve was inserted on top of the bed and firmly secured in place by layer of glass wool beads. The samples of the effluent were collected at the outlet of the column at regular time intervals and the concentrations were measured.

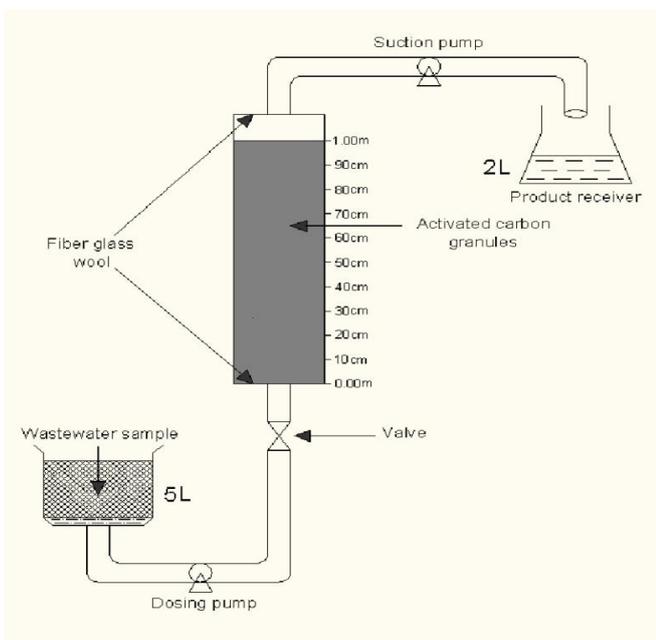


Fig.3. Experimental Setup

Once the activated carbon from palm date pits was packed with the desired height inside the column, the column was fully filled with deionized water for 2 h to ‘wet’ the column. This was important to ensure that all air was expelled between and within the activated carbon particles before the experiment began. If there was an air pocket inside the column, channeling and air entrapment would occur which would lower the bed performance [13].

**Heavy Metals**

The heavy metals chosen for this study were: Cu, Cr<sup>+6</sup>, and Fe. These elements were chosen due to their extensive use in several industries such as Tanneries, Fertilizers, Electroplating, and Chemical Industries... etc. and thus their presence in the final effluent of these industries in relatively high concentration causing negative effects on the surrounding environment. Each element was tested separately, and then samples of industrial wastewater containing those elements were tested as it is described in a following section. Synthetic stock solution of copper, chromium, and iron were prepared using their nitrated salts, in distilled water according to the Standard Methods for Analysis of water and wastewater, 20<sup>th</sup> Edition [14]. Metals’ initial and final concentrations were measured using Flam Atomic Absorption Spectrometry

$$\% \text{ Adsorption} = \left\{ \frac{C_o - C_e}{C_o} \right\} * 100 \quad (1)$$

Where, C<sub>o</sub> and C<sub>e</sub> are the initial and equilibrium concentrations of each metal (mg/l) in the solution.

**Adsorption Operating Conditions**

The effect of the following parameters on the removal of the previously mentioned elements was studied: pH, contact time, depth of layer, initial concentration of metals, and particle size according to Table 2.

**Table 2. Experimental Work Program for Individual Elements**

Run	Metal	pH	Contact Time (min)	Depth of Layer (cm)	Initial Conc. (mg/l)	Particle size (mm)
1	Cu	3.5-7.5	30	40	10	1.5
2		6.5	30-90	40	10	1.5
3		6.5	90	40-90	10	1.5
4		6.5	90	90	10-50	1.5
5		6.5	90	90	10	1.5-1-0.75
6	Cr	3.5-7.5	30	40	10	1.5
7		4.5	30-90	40	10	1.5
8		4.5	70	40-90	10	1.5
9		4.5	70	90	10-50	1.5



10		4.5	70	90	10	1.5-1-0.75
11	Fe	3.5-7.5	30	40	10	1.5
12		5.5	30-90	40	10	1.5

13		5.5	90	40-90	10	1.5
14		5.5	90	90	10-50	1.5
15		5.5	90	90	10	1.5-1-0.75

**B. Experimental Work on Industrial Wastewater**

**Industrial Wastewater from Tanneries (Case Study 1)**

The wastewater produced from ALFATEH Company is 8 m<sup>3</sup>/day and is discharged to the sewer system of the Maser el Kadima. Wastewater from the plating department of 1 m<sup>3</sup>/day represents the main source of pollution in this company’ effluent. The rinse water collecting from washing tanks and containers contains Cu, Cr<sup>+6</sup>, and Fe ions with concentrations 5.65 mg/l, 23.14 mg/l, and 42.56 mg/l respectively and actual pH was 3.

**Industrial Wastewater from Electroplating Industries (Case Study2)**

The wastewater produced from COMACX Company is 500 m<sup>3</sup>/day and is discharged to the sewer system of 6 October city. Wastewater from the electroplating department of 160m<sup>3</sup>/day represents the main source of pollution in this company. The rinse water contains high concentration of Cu, Cr<sup>+6</sup>, and Fe. Their concentration was as high as 7, 26, and 8 mg/l respectively. The COMACX has a chemical treatment of industrial and human wastewater by reduction of Cr<sup>+6</sup> in the electroplating wastewater was carried out prior to the end of pipe waste. Actual pH in raw wastewater was 4.3. For both factories, samples were tested first with their actual pH and then pH was adjusted according to the optimum value obtained from the results conducted on individual elements. Table 3 shows the experimental work conducted for the industrial wastewater.

**Table 3. Experimental Work for Industrial Wastewater**

Run	Case	pH	Contact Time (min.)	Depth of Layer (cm)	Initial Conc. (mg/l)	Particle size (mm)
1	Cu	actual	80-100	80	Actual Conc.	0.75
	Cr <sup>+6</sup>	actual	80-100	80	Actual Conc.	0.75
	Fe	actual	80-100	80	Actual Conc.	0.75
2	Cu	actual	100	70-90	Actual Conc.	0.75
	Cr <sup>+6</sup>	actual	100	70-90	Actual Conc.	0.75
	Fe	actual	100	70-90	Actual Conc.	0.75
3	Cu	actual	100	90	Actual Conc.	1.5-0.75
	Cr <sup>+6</sup>	actual	100	90	Actual Conc.	1.5-0.75
	Fe	actual	100	90	Actual Conc.	1.5-0.75
4*	Cu	6.5	100	90	Actual Conc.	0.75
	Cr <sup>+6</sup>	4.5	100	90	Actual Conc.	0.75
	Fe	5.5	100	90	Actual Conc.	0.75

These experiments were conducted twice for both case studies.

\* Adjusted pH according to the optimum for the synthetic wastewater

**III. RESULTS AND DISCUSSION**

The results of the individual elements are shown in Fig.4, the following was observed:

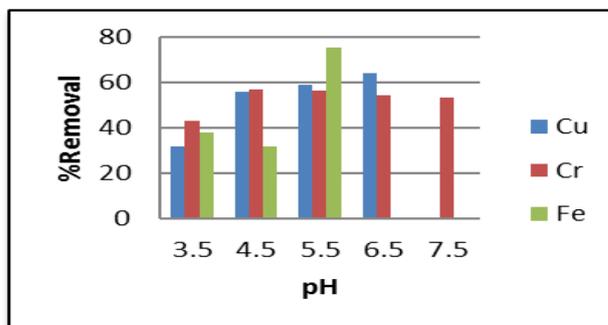


Fig. 4a Effect of pH on Cu, Cr<sup>+6</sup>, and Fe removal

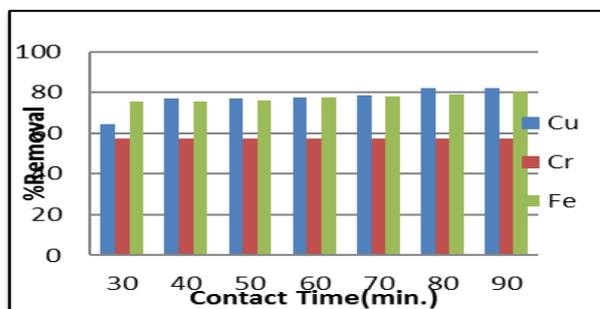


Fig. 4b Effect of the Contact Time on Cu, Cr<sup>+6</sup>, & Fe removal

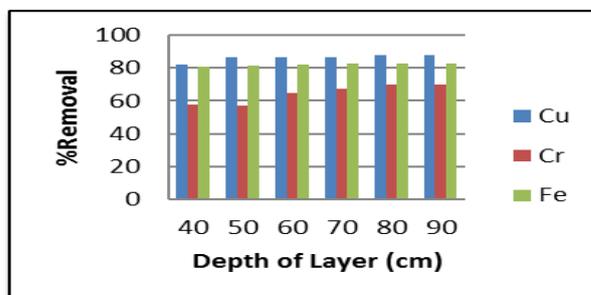


Fig. 4c Effect of the depth of layer on Cu, Cr<sup>+6</sup> & Fe removal

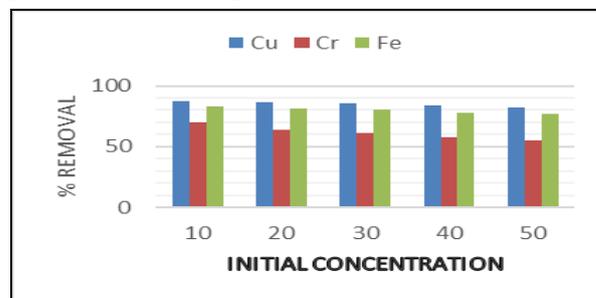


Fig.4d Effect of initial concentration on Cu, Cr<sup>+6</sup>, and Fe removal

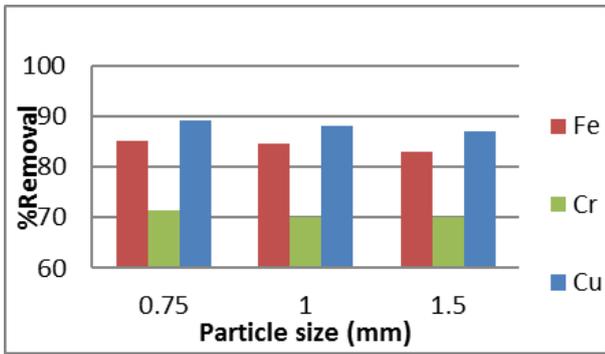


Fig. 4e Effect of the particle size on Cu, Cr<sup>+6</sup>, & Fe removal

First parameter tested was pH value, the percentage of adsorption of Cu increased almost linearly between pH 3.5 to 6.5, and the maximum removal was reached at pH 6.5, the Cr<sup>+6</sup> adsorption removal efficiency increased between pH 3.5 to 4.5 and removal decreased slightly from pH 5.5 to 7.5, and Fe removal efficiency increased between 3.5-5.5 and at higher pH values, Fe was found to precipitate. Second parameter was contact time, to study its effect, pH values were fixed at the optimum values obtained from the first set of experiments in order to reach the best operating conditions. The rate of uptake of metals was rapid; the metal removal in the first 30 minutes was 64.28%, 57.08% & 75.29% for Cu, Cr<sup>+6</sup>, & Fe respectively. Equilibrium was reached within 90 min for Cu with 82.05 % removal, equilibrium was reached within 70 min for Cr<sup>+6</sup> with 57.72 % removal, and equilibrium was reached within 90 min for Fe with 80.58 % removed from the solution. As for the results of varying the depth of layer, the metal removal in the first 40 cm of the layer was 82.05%, 57.47% & 80.58% for Cu, Cr<sup>+6</sup> & Fe respectively, and reached 87.82%, 70.02% and 83.05% for Cu, Cr<sup>+6</sup>, and Fe at 90 cm. For the results of initial concentration, the results showed that the percentage of removal increased with the decrease in the initial concentration from (50-10) mg/l, the maximum removal efficiency was obtained at initial concentration of 10 mg/l, and reached 87.77, 70.02, 83.03 % for Cu, Cr<sup>+6</sup> & Fe respectively. For the results of particle size, the results showed that the percentage of removal increased with the decrease in the particle size from (1.5 to 0.5) mm the maximum removal efficiency was obtained at size (0.75-0.5), and reached 89.17, 71.3, 85.17 % for Cu, Cr<sup>+6</sup> & Fe respectively. The obtained adsorption data were mathematically analyzed using both Langmuir and Freundlich models to determine the relevant adsorption parameters.

Langmuir equation 
$$\frac{C_e}{q_e} = \frac{1}{qm b} + \frac{C_e}{qm}$$

Freundlich isotherm has the form:  $q_e = k C_e^{1/n}$  and the linearized form can be represented as:  $\text{Log } q_e = \text{log } k + 1/n \text{ log } C_e$ , Where k & n are constants and  $n > 1$

It was found that the equilibrium data satisfied both the Langmuir and Freundlich isotherm models, as can be observed, experimental data were better fitted to the Freundlich equation, as the value of R<sup>2</sup> were found to be (0.995, 0.998, 0.996) than to the Langmuir equation, as the values of R<sup>2</sup> were found to be (0.989, 0.97, 0.983) for Cu, Cr<sup>+6</sup>, and Fe and therefore the palm date pits is more suitable

for the analysis of kinetics. The results and parameters obtained are shown in Fig. 5 and Tables 4 and 5.

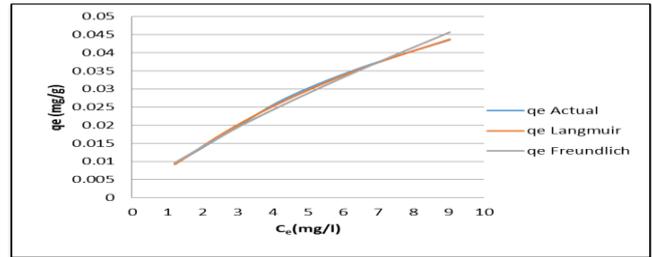


Fig. 5a Adsorption isotherm (q<sub>e</sub> Actual, q<sub>e</sub> Langmuir, q<sub>e</sub>Freundlich) for the sorption of Cu

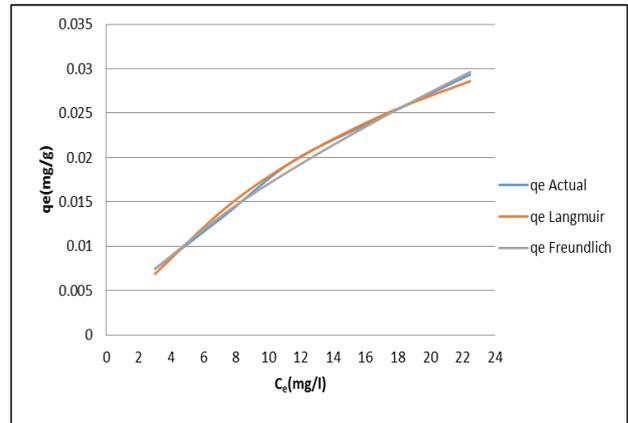


Fig. 5b Adsorption isotherm (q<sub>e</sub> Actual, q<sub>e</sub> Langmuir, q<sub>e</sub> Freundlich) for the sorption of Cr

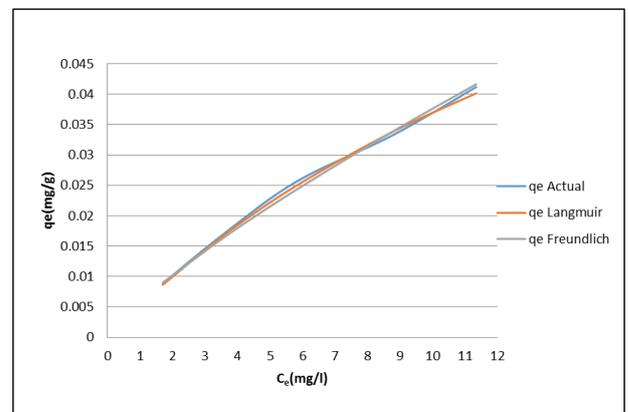


Fig.5c Adsorption isotherm (q<sub>e</sub> Actual, q<sub>e</sub> Langmuir, q<sub>e</sub> Freundlich) for the sorption of Fe

Table 4. Adsorption isotherm (q<sub>e</sub> Actual, q<sub>e</sub> Langmuir, q<sub>e</sub>Freundlich) for the sorption of Cu, Cr<sup>+6</sup> & Fe ion

Heavy Metal	C <sub>e</sub>	q <sub>e</sub> Actual	q <sub>e</sub> Freundlich	q <sub>e</sub> Freundlich
Cu	1.22	0.00934	0.009193	0.009578
	2.78	0.018319	0.018853	0.018223
	4.34	0.027298	0.026757	0.025804
	6.45	0.035691	0.035412	0.035163
	9.02	0.043596	0.043695	0.045691
Cr+6	3	0.007447	0.006883	0.007445
	7.32	0.013489	0.014258	0.01374
	11.56	0.019617	0.019609	0.018807
	16.97	0.0245	0.024714	0.024483
	22.45	0.029309	0.028597	0.029672
Fe	1.7	0.00883	0.00869585	0.009044
	3.67	0.017372	0.01721039	0.016804
	5.85	0.025691	0.02511992	0.024458
	8.78	0.033213	0.0338631	0.033913
	11.34	0.041128	0.04016401	0.041669

**Table 5 .Langmuir & Freundlich parameters for the sorption of Cu, Cr &Fe ions**

Langmuir parameters for the sorption of Cu, Cr &Fe ions			
Parameter	b	qm	R <sup>2</sup>
Cu	0.078	0.1058	0.989
Cr	0.047	0.0557	0.97
Fe	0.05	0.111	0.983
Freundlich parameters for the sorption of Cu, Cr &Fe ions			
Parameter	K	n	R <sup>2</sup>
Cu	0.0082	1.28	0.995
Cr	0.0035	1.4556	0.998
Fe	0.0059	1.242	0.996

Results for the industrial wastewater samples are summarized in Table 6. For the industrial wastewater collected from tanneries, the percentage of removal of the metals at actual pH (3) was 74.87, 35.18, and 77.5 % for Cu, Cr<sup>+6</sup>, & Fe and when the pH was adjusted to 6.5 for Cu, 4.5 for Cr<sup>+6</sup>, 5.5 for Fe –which is the optimum values obtained for individual elements-, the removal was found to be 85.8, 65.42, and 87.03 respectively. For the industrial wastewater from the electroplating factory, the percentage of removal of metals at actual pH (4.3) was 67.343, 33.277, and 83.15 % for Cu, Cr<sup>+6</sup>, & Fe and when the pH was adjusted to 6.5 for Cu, 4.5 for Cr<sup>+6</sup>, 5.5 for Fe, removal increased to reach 82.857, 61.65, and 89% . A cost estimate of using palm date pits as low cost adsorbent was conducted. It included its purchase cost, transportation and processing. It was found to be 5 times less than the cost of granular activated carbon.

**Table 6. Results for the industrial wastewater samples at the optimum operating conditions**

Case	Heavy Metal	pH	Initial Conc (mg/L)	Final Conc(mg/L)	% Removal
Case study 1	Cu	6.5	5.65	0.8	85.8
	Cr <sup>+6</sup>	4.5	23.14	8	65.42
	Fe	5.5	42.56	5.52	87.03
Case study 2	Cu	6.5	7	1.2	82.86
	Cr <sup>+6</sup>	4.5	26	9.97	61.65
	Fe	5.5	8	0.88	89

**IV. CONCLUSIONS**

- Kinetic studies showed that Cu, Cr<sup>+6</sup>, and Fe ions were adsorbed onto the palm date pits very rapidly within the first 30 minutes, while equilibrium was attained within 90 minutes for Cu, Cr<sup>+6</sup>, & Fe ions.
- The obtained adsorption data were mathematically analyzed using both Langmuir and Freundlich models to determine the relevant adsorption parameters and it was found that the equilibrium data satisfied both the Langmuir and Freundlich isotherm models, results showed that Freundlich model was more suitable for the analysis of kinetics for palm date pits.
- pH is the most important parameter affecting the adsorption capacity and thus the removal efficiency.
- The percentage removal efficiency was affected by:
  - The increase in contact time.
  - The increase in the depth of layer.
  - The decrease in particle size.
  - The decrease in initial concentration.
  - The cost of the palm date pits is 5 times less than the granular activated carbon.

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