Biosorption of Cr(VI) and Cu(II) by Palm Kernel Powder and Its Potential Application

Ragwan Mohamed, Alaa Mustafa, and Mohamed Erhayem

Abstract—**In this research, palm kernel powder (PKP) was utilized as a bioadsorbent for the removal of Cr(VI) and Cu(II) ions from aqueous solutions. Batch studies were preformed to evaluate the effect of various experimental parameters on Cr(VI) and Cu(II) adsorption onto PKP. The adsorption equilibrium was reached after 70 min. The adsorption capacity of Cr(VI) and Cu(II) was pH-dependent showing a maximum at equilibrium pH 2 and 5, respectively. The percentage removal of heavy metals by PKP varied in order unmodified adsorbents>HNO3>H3PO4. The adsorption isotherm and kinetics of Cr(VI) and Cu(II) were also investigated using Langmuir and Freundlich adsorption isotherm models and pseudo-first order (LSO) and pseudo-second-order (PSO) kinetic models. The data was better described by Freundlich adsorption isotherm model and PSO kinetics in the concentration range studied. For environmental study, it was found that the adsorption capacity for heavy metals decreased in the raw waste water sample in comparison with the synthetic waste water system.**

Index Terms—**Biosorption, chromium, copper, kinetics, palm kernel powder.**

I. INTRODUCTION

Heavy metals, such as Cr(VI) and Cu(II), release into the environment from plating plants, mining, metal finishing, welding and alloy manufacturing pose a significant threat to the environment and public health [1]. Unlike some organic pollutants, Cr(VI) and Cu(II) are not biodegradable and cannot be metabolized or decomposed [2]. The major concern with Cr(VI) and Cu(II) is their ability to accumulate in the environment and cause public health problems. For instance, the toxicity of Cr(VI), even in small concentrations, has been extremely studied causing damage to kidney, liver, etc [3]. The excessive intake of Cu(II) leads to severe mucosal irrational, widespread capillary damage, hepatic and renal damage, central nervous problems followed by depression, gastrointestinal irritation, and possible necrotic changes in the liver and kidney [4].

Several studies have successfully investigated the removal of heavy ions, such as Pb(II), Cd(II), Cr(II), Cu(II), Zn (II), from aqueous solutions under various operating variables like contact time, solution pH, initial metal concentration and temperature by agricultural by-products such as coconut shell charcoal, commercial activated carbon, Cankiri bentonite,

natural zeolite clinoptilolite, egg shells, palm shell activated carbon and green alga *Spirogyra* species using different models and techniques [4]-[6]. The PKP was found to be poor adsorbent using different activation process [4]. This study focuses on the PK collected from Libya as almost zero cost adsorbent for removal of Cr(VI) and Cu(II).

II. EXPERIMENTAL METHODS

A. Instruments

The FT-IR spectra of PKP before and after addition of heavy metals were done using Nicolet Impact 410 (Bruker-Tensor37) equipment and the spectra were recorded from 400 to 4000 cm^{-1}. The adsorption studies of metal ions were determined by using Atomic Absorption Spectrophotometer (AAS), Thermo AA spectrometer/M series. The pH value was measured by using pH meter (OAKTON 510 Series).

B. Materials

Raw material PK was collected from north of Libya and washed with distilled water and dried at 110℃ for 24 h. It was crushed and sieved to a particle size with 0.5 mm membrane and socked in HNO$_3$ (60%) for 3 hrs. The mixture was washed with distilled water. The PK was heated at 230℃ for 3 hrs and then stored in desiccator. Synthetic stock solutions of Cr(VI) and Cu(II) were prepared by dissolving of K$_2$Cr$_2$O$_7$ and CuSO$_4$.5H$_2$O, respectively, in deionized water. For the environmental study, raw waste water sample was collected from El-Souif waste crater plant, Alexandria, Egypt and 3 drops of HNO$_3$ were added to prevent metal precipitation. The sample was transferred to the laboratory and filtered. The filtered sample conditions were: original concentration of 0.181 mgL^{-1} Cr(VI) and 0.144 and mgL^{-1} Cu(II), respectively, the pH of 7, salinity 2.68 mgL^{-1}, conductivity 557 ms, TSS 200 mgL^{-1} and TDS 430 mgL^{-1}.

C. Adsorption Studies

In order to evaluate the time at which the equilibrium of metal ions and PKP was attained, 50 mg of PKP was added to 1.0 mgL^{-1} of each metal ion solutions. The suspensions were then shacked at 200 rpm. The samples were taken for different time intervals (1, 2, 3, 4, 5, 10, 15, 25, 40 and 70 min). In order to evaluate the effect of sorbent dosage on biosorption process, 0.1 to 1.0 g PKP doses were added to 150 mL of 1.0 mgL^{-1} of metal ion solutions. The Cr(VI) and Cu(II) solutions were adjusted to the desire pH 3 and 5 (based on pH study). The effect of metal ion concentration on biosorption process was investigated using 1, 5, 10, 20, 50, and 100 mgL^{-1} of 150 ml of metal ion solutions, Cr(VI) and Cu(II), containing 50

mg of PKP at constant pH 3 and 5. In order to determine the effect of pH on biosorption process, 50 mg of PKP (based on dosage study) was added to 150 ml of 1 mgL^{-1} of the metal ions at various values of pH from 3.0 to 9.0 using 0.1N HCl or 0.1N NaOH. In order to obtain the adsorption isotherms by using batch equilibrium method, 50mg of PKP were added to 150ml of various concentrations (1-1000 mgL^{-1}) of Cr(VI) or Cu(II). All suspensions were stirred at 200 rpm for 70 min to allow the suspension to equilibrate. The suspensions were allowed to settle down before filtering by Whatman 0.45μm filter paper. The remained metal concentrations were determined using AAS.

D. Environmental Study

In order to study the environmental impact of metal ions on biosorption process, 150 ml of the raw waste water sample was treated with 50 mg of PKP and continuous stirring at 200 rpm for 70 min. The adsorption procedures described above were followed. All experiments were carried out twice and the data was the means of duplicated experimental results.

III. RESULTS AND DISCUSSION

Characterization of PKP. FT-IR spectra were recorded for the raw PKP before metal ion addition and showed the presence of different functional groups (-C-C-, -OH, N-H, -C=C- and COOH), Figures not shown. The effect of solvent on these functional groups was also studied. These groups were affected by acid modification, namely solvent effect. The addition of Cr(VI) and Cu(II) caused to significantly decrease in the intensity and shift peaks of the functional groups due to Cr(VI) and Cu(II) adsorption onto PKP. This shift in wave number corresponds to a change in bonding energy of the functional groups. The -C-O-, -C=C-, -C=C-,-C=N-, -OH, -COOH and -NH groups are the most effective functional groups in Cr(VI) adsorption onto PKP surface functional groups. It can be noticed that Cu(II) adsorption was at –C-C- and –C-O- groups in case of raw PKP while in case of nitric and phosphoric acid modified PKP Cu(II) adsorption mainly occurred at -C-O-,-OH, -COOH and –NH groups.

Adsorption studies. Generally, the adsorption of Cr(VI) and Cu(II) ions showed a similar trend with time, which an initial rapid adsorption after the addition of PKP, followed by a final leveling off, Fig. 1. This is due to the availability of the PKP surface area and large numbers of vacant binding sites, which are available for adsorption during the initial stage of the addition and the surface gradually become covered with the metal ions after 40 min. The AA data indicated no significant changes in the adsorption of Cr(VI) and Cu(II) ions on the PKP that occurred after 60 min due to the repulsive forces between the metal ions on the solid and liquid phases. This result is in good agreement with previous research [7], [8]. Therefore, 70 min was chosen as the time at which the adsorption of Cr(VI) and Cu(II) ions onto the PKP was attained. Also, the adsorption reactions of Cr(VI) and Cu(II) were performed after modification of the used adsorbents by HNO$_3$ and H$_3$PO$_4$. The results indicated that the equilibrium time was shorter than that in case of unmodified adsorbents. Also, the percentage removal and adsorption

capacity increased in case of modified adsorbents with HNO$_3$, which was more effective in removing heavy metals than H$_3$PO$_4$. This could be due to heavy metals were not soluble in the presence of H$_3$PO$_4$ comparing to the presence of HNO$_3$.
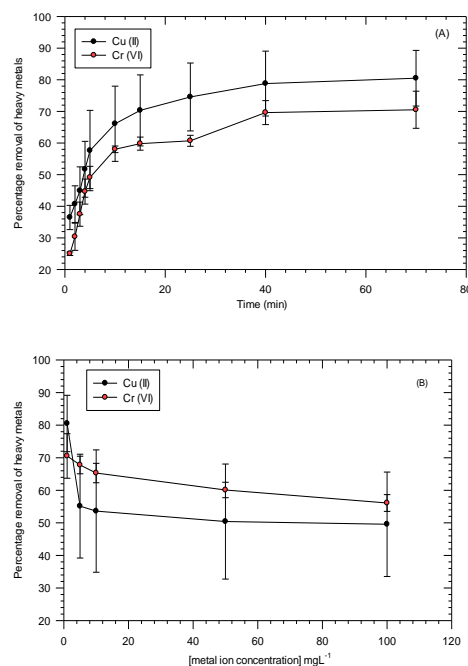

Fig. 1. Effect of (A) time and (B) ion metal concentrations on the percentage removal of Cr(VI) and Cu(II) onto PKP. (A) Fifty mg of PKP was added to 1 mgL^{-1} of each metal ion solutions. (B) Fifty mg of PKP was added to 150 ml of 1-100 mgL^{-1} of the metal ions. The pH value was adjusted to 3 for Cr(VI) and 5 for Cu(II). The suspensions were stirred at 200 rpm for 70 min at 25°C.

The effect of initial metal concentration on the adsorption process was investigated. Fig. 1 shows a decrease in the adsorption percentage of Cr(VI) and Cu(II) with a corresponding increase in initial concentration of metal ions. The trend which was an initial rapid adsorption of metal ions after addition of PKP followed by desorption and then a finial leveling off. Furthermore, the AA data indicated no significant changes in adsorption of metal ion onto PKP at high metal concentration likely due to the sites in the sorbents were occupied at higher concentration and fewer available sites were involved in the sorption process [9]. On the other hand, the adsorption capacity increased with increasing the initial concentration. This is probably due to the increasing initial metal concentration provides a key driving force to overcome all mass transfer resistance of metal ions between the aqueous and solid phases, thus increasing the adsorption capacity [10].
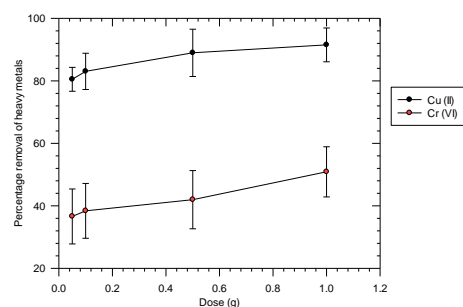

Fig. 2. Effect of PKP dose on the percentage removal of Cr(VI) and Cu(II) onto PKP. 0.1 to 1 g PKP doses were added to 150 mL of 1.0 mgL^{-1} of metal ion solutions. The pH value was adjusted to 5 and the suspensions were stirred at 200 rpm for 70 min at 25°C.

The effect of PKP dosage on the adsorption process was also studied, Fig. 2. An increase in adsorbent dosage resulted in a corresponding slightly increase in removal. This is likely due to the availability of more empty binding sites at higher dosages to adsorb the same amount of Cr(VI) and Cu(II) ions in the adsorbate solution [11]. The percentage removal of metal ions by PKP followed the order: Cu(II)>Cr(VI) likely due to overlapping of adsorption sites as a result of overcrowding of adsorbent particles and the lower adsorbate to binding site ratio where the Cr(VI) ions were distributed onto larger amount of adsorbent binding sites [12]. Also, neutralization of the charge on the PKP surface due to metal ion adsorption and the anionic radii of Cu(II)>Cr(VI), lead to an increase in the surface area onto PKP for adsorption.

Fig. 3 shows the effect of solution pH on biosorption process. The percentage removal of Cr(VI) adsorption onto PKP decreased significantly with increasing pH value and the maximum removal of Cr(VI) ions was found to be at pH 3. The improved removal of chromium (VI) at low pH value is probably due to reduction of Cr(VI) to Cr (III) which in turn adsorbed into the surface active sites [13]. Also, the increase of Cr(VI) adsorption at acidic pH should be due to the electrostatic attraction between positively charged groups of the sorbent surface and the $HCrO_4-$ anion which was the dominant species at low pH thereby increased the diffusion of chromate ions into the bulk of the adsorbent. Furthermore, Cr(VI) exists in aqueous medium as ($HCrO_4-$, $Cr_2O_7 2-$, $CrO_4 2-$) forms, among which the $HCrO_4-$ form is the dominant species. Increasing the pH shifts the concentration of $HCrO_4-$ to other forms, i.e., $CrO_4 2-$ and $Cr_2O_7 2-$. Consequently, Cr(VI), existing as oxo anions in water, which decreases in the adsorption due to the increased number of $OH-$ ions in the bulk which retarded the diffusion of chromate ions, and due to competitiveness of the oxy-anions of chromium. A high concentration of H+ ions facilitates the adsorption whereas a high concentration of OH- ions suppresses the adsorption reaction, thus accounting for the decrease in the adsorption percentage of Cr(VI) ion at high pH [3].
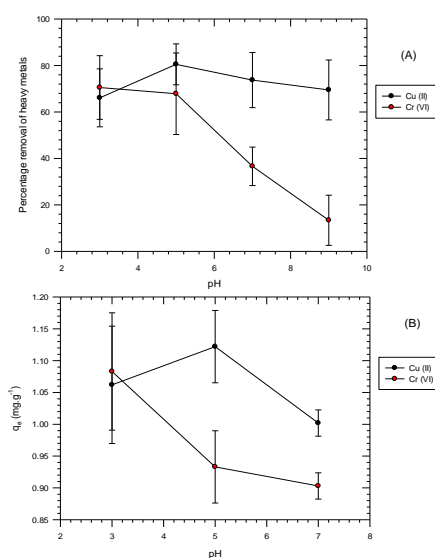
Also, the effect of pH on the percentage of Cu(II) removal by PKP was shown in Fig. 3. The results reveal that there is a decrease in removal of metal ions at lower pH 3 is apparently due to the higher concentrations of H+ in the solution, which compete with Cu(II) ions for the adsorption sites of the adsorbents. Generally, the positive charge of the adsorbent surface decreases with the increasing pH value to pH 5, leading to the decrease in the repulsion between the adsorbent surface and Cu(II), thus improving the adsorption capacity [14], [15]. At higher pH values than 5, the percentage removal and adsorption capacity due to metal precipitation (metal hydroxides precipitate) appeared and adsorbent was deteriorated with accumulation of metal ions. Therefore, pH 5 was selected to be the optimum pH for further studies [13]. The extent of Cr(VI) and Cu(II) adsorption onto PKP was greatly dependent on the solution pH.

Adsorption isotherms. Freundlich isotherm assumes that the uptakes of metal ions occur on a heterogeneous surface by multilayer adsorption and that the amount of adsorbate adsorbed increases infinitely with an increase in concentration. This isotherm does not predict any saturation of the sorbent by the sorbate thus infinite surface coverage is predicted mathematically, indicating a multilayer sorption of the surface using the following equation [7], [16], [17].

$$\log q_e = \log K_F + \frac{1}{n}\log C_e \tag{1}$$

C_e is the equilibrium activities of metal ions and q_e is the surface activity for metal ions on the solid surface [16]. Both K_F and n are Freundlich constants. The numerical value of $1/n$ <1 indicates that adsorption capacity is only slightly suppressed at lower equilibrium concentration. The values of $1/n$ and K_f can be calculated from the slope and intercept of the plot, respectively.

Langmuir isotherm is applied to equilibrium adsorption assuming mono-layer adsorption onto a surface with a finite number of identical sites using the following equation [18].

$$\frac{c_e}{q_e} = \frac{1}{q_m b} + \frac{c_e}{q_m} \tag{2}$$

In which q_e (mg.g^{-1}) is the equilibrium adsorption capacity of adsorbent; C_e is the equilibrium concentration of the adsorbate; q_m (mg.g^{-1}) is the maximum adsorption capacity corresponding to complete monolayer coverage, b is the Langmuir constant related to the adsorption energy and K_L is adsorption constant, $K_L=q_m.b$. A plot of C_e/q_e versus C_e resulted in a straight line of slope $1/q_m$ and an intercept of $1/b.q_m$.

The correlation coefficient (R^2) and q_m values that are regarded as a measure of the goodness-of-fit of experimental data on the isotherms model as shown in Table I. The R^2 values were elucidated that the Langmuir isotherm model represents a poor mathematical fit, while it is well fitted with the Freundlich isotherm model indicating the presence of heterogeneous sites. These results were in good agreement with previous research in a similar system [14].

Adsorption Kinetics. The linear form of LFO model was

Fig. 3. Effect of pH on Cr(VI) and Cu(II) removal by PKP in (A) synthetic water and (B) raw waste water. 50 mg of PKP was added to 150 ml of 1.0 mgL^{-1} of the metal ions. The pH value was adjusted at from 3 to 9 at 200 rpm for 70 min at 25°C.

applied in this study can be expressed by Eq. 3 The PSO kinetic model is given by Eq. 4 [19].

$$\ln(q_e - q_t) = \ln q_e - k_1 t \qquad (3)$$

$$\frac{t}{q_t} = \frac{t}{k_2 q_e^2} + \frac{t}{q_e} \qquad (4)$$

In which q_t (mg.g^{-1}) is the amount of adsorption time (min); q_e is the amount of adsorption equilibrium (mg.g^{-1}). K_1 and K_2 are the adsorption rate coefficients (rate constants) of the equations (min^{-1}). K_1 can be determined experimentally by plotting of $\ln (q_e - q_t)$ versus t. The applicability of PSO model can be examined by linear plot (t/q_t) versus t. Slopes and intercepts are respectively $1/q_e$ and $1/k_2 q_e^2$. The experimental data were evaluated by LFO and PSO models, which gave linear plots, Figures not shown, respectively. The rate constants and the calculated adsorption capacity qe of each model were determined and listed in Table II. For LFO study, it was found that the calculated q_e values did not agree with the $q_{e.exp}$ values which suggested that the LFO model gives a poor fitting. For PSO model, the R^2 values were 0.9980 and 1, Table II. The calculated and experimental capacities were fitted well using PSO. This result was in good agreement with previous research in a similar system [9], [20]. Thus, the PSO kinetics is the obeyed model for the biosorption of Cr(VI) and Cu(II) onto PKP since it has higher R^2 values than that obtained from the LFO model.

TABLE I: LANGMUIR AND FREUNDLICH BIOSORPTION CONSTANTS

	Freundlich constants			
	K_F			
Metal	(L.g^{-1})	1/n	R^2	±SD
Cr(VI)	6.47	0.872	0.9998	0.018
Cu(II)	6.45	0.747	0.986	0.136
	Langmuir constants			
	$q_{m.exp}$		K_L	
Metal	(mg.g^{-1})	b	(L.g^{-1})	R^2	±SD
Cr(VI)	394	0.0163	6.4	0.9614	0.016
Cu(II)	332	0.0143	4.75	0.631	0.094

* Fifty mg of PKP was added to 150 ml of various concentrations (1-1000 mgL^{-1}) of Cr(VI) or Cu(II) and stirred at 200 rpm for 70 min at 25ºC. SD=standard deviation.

Environmental study. In order to demonstrate a practical application value of this research, a real waste water sample from El-Souif waste crater, Alexandria, Egypt, was collected and added to the PKP. After 70 min stirring, the maximum removal of metal ion studied at different pH values was in order Cu(II)>Cr(VI), Fig. 3. It was found to be at lower pH values of the raw waste water sample as compared to higher pH values. Cr(VI) has the maximum recovery at pH 3 which was 87.8%, while Cu(VI) had the maximum recovery at pH 5 which found to be 88.2%. It was also noticed that the q_e for heavy metals decreased in the raw waste water sample in comparison with the synthetic waste water system which was reported above in Fig. 3A. This may be due to several reasons

which could be due to the lower initial concentrations (144-280 ppb) relative to our previously studied value (1 ppm) or the competition between metals are present in El-Souif wastewater crater. Moreover, the environmental study was performed on a raw waste water samples which contain many different metal ions which could be enhanced the adsorption capacity of metal ions onto the adsorbent surface [21]. The results above clearly suggest that the used adsorbents possess has a potential application value.

TABLE II: LFO AND PSO PARAMETERS FOR THE ADSORPTION OF CR(VI) AND CU(II) ONTO PKP AT VARIOUS INITIAL CONCENTRATIONS

	Pseudo-first-order model				
Metal	qe.exp. (mg.g^{-1})	K1 (min^{-1})	qe.cal. (mg.g^{-1})	R^2	±SD
Cr(VI)	90.39	0.075	55.2	0.9854	0.187
Cu(II)	76.2	0.071	48.1	0.971	0.244
	Pseudo-second-order model				
Metal	qe.exp. (mg.g^{-1})	K2	qe.cal. (mg.g^{-1})	R^2	±SD
Cr(VI)	90.39	0.003	94.6	1	0.005
Cu(II)	76.2	0.003	81.2	0.998	0.02

*Fifty mg of PKP was added to 150 ml of 1-1000 mg L^{-1} of the metal ions. The pH value was adjusted to 3 for Cr(VI) and 5 for Cu(II). The suspensions were stirred at 200 rpm for 70 min at 25 ºC. C$_o$=initial heavy metal concentration

IV. CONCLUSIONS

PKP has proven to be a promising material for removal of pollutant from environmental waters due to a fairly high capacity to remove Cr(VI) and Cu(II) ions from solution. The extent of Cr(VI) and Cu(II) adsorption onto PKP was found to be largely dependent on solution parameters such as metal ion type, initial metal ion concentration, biosorption dosage, solution pH and solvent used. The adsorption capacities of the used adsorbents toward Cu(II) was higher than those determined toward Cr(VI). The adsorption data of Cr(VI) and Cu(II) was best fitted with Freundlich isotherm more than Langmuir isotherm indicating the presence of heterogeneous sites on PKP for Cu(II) and Cr(VI). Adsorption Kinetics was followed the pseudo-second order rate equation. The adsorption capacity of heavy metals was increased in the presence of the real waste water sample when compared to the synthetic waste water system.

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