Treatment of Aqueous Solutions Contaminated by Heavy Metals with Low-Cost Adsorbent

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Abstract: This paper is related to study the potential of orange peel on the removal of copper ions from simulated wastewater using different design parameters by adsorption technique. The operating parameters studied to adsorb copper ions using orange peel as an adsorbent material were initial concentration of copper, absorbance material packing height which was orange peel, pH of copper feed inlet solution, and treatment time, these parameters were varied from (1-100) mg/l, (10-50) cm, (1-8), and (1-60) min respectively. All experiments were carried out at constant feed flow rate (1 l/min) and constant temperature 25°C. Results show that the higher removal efficiency was 89.25 % for copper from aquatic solution and this efficiency was decreased with increasing of initial concentration and pH while the removal efficiency increased with increasing adsorbent material bed height and contact time. The residues of orange peels that loaded with copper ions were prepared to use it as a rodenticide by applying it on the laboratory rats and calculate the half lethal dose (LD_{50}). The residues show a good ability to use it as a rodenticide and the LD_{50} calculated was in the same range mentioned in the literature. By this way, it can possess different benefits in the same time, which are remove the toxic copper ions from contaminated the water by highly efficiency, get rid of agricultural waste orange peel and finally prepare an active, cheap and simple rodenticide. So, it can discard more toxic waste in a coast and eco-friendly method accessing to Zero Residue Level (ZRL) concept.

Keywords: Copper ions, adsorption, toxic, rodenticide, Sprague dawley, and ZRL.

INTRODUCTION

Heavy metal released in the environment has been increasing continuously because of industrial activities and technological developments and poses a threat to human health [1]. Copper is a priority toxic pollutant in wastewater that is introduced into water from metal mining, melting, plating, batteries, pesticides, oil paint, pigments, and alloys [2, 3]. Large dosages of copper in humans can result in tubular and glomerular damage and cause proteinuria [4]. The presence of copper ions in the environment has become a potential threat to plant, animal, and human life due to their bioaccumulation tendency and toxicity and therefore they must be removed from municipal and industrial effluents before discharge into natural streams [5]. The traditional heavy metal treatment includes chemicals and processes in surface chemistry such as precipitation, adsorption, membrane processes, ionic exchange, floatation, and others [6, 7]. However, these techniques are rather expensive and are not selective enough to allow the recovery of very dilute heavy metals present in the effluent [8]. These disadvantages, together with the need for more economical and effective methods for the recovery of metals from wastewater, have resulted in the development of alternative separation technologies. One alternative is biosorption [9, 10], where certain types of biomass are able to bind and concentrate metals from even very dilute aqueous solutions. A biosorption process offers a number of advantages when compared to the conventional methods currently used. These include low operating costs, minimization of the volume of chemical and/or biological sludge to be handled, and high efficiency in detoxifying effluents [11, 12]. The mechanisms of biosorption are generally based on physicochemical interactions between metal ions and the functional groups present on the cell surface, such as electrostatic interactions, ion exchange, and metal ion chelation or complexation [13]. Functional groups most commonly implicated in such interactions include carboxylate, hydroxyl, amine and phosphoryl groups present within cell wall components such as polysaccharides, lipids, and proteins [14]. In this case, factors like pH, size of biosorbent, ionic strength and temperature influence the metal biosorption [15]. Therefore, the purpose of this study was to explore the feasibility of using orange peel waste for copper ion removal from aqueous solutions in very simple method. The effects of sorbent weight, initial copper ion concentration and pH on their adsorption capacities were studied.

EXPRIMENTAL WORK
Materials
Orange peel (sorption media)

Orange peel was collected from locally market in Baghdad. The orange peel was washed three times with doubled distilled water. Excess distilled water was used to remove the soluble materials present in the orange peel collected after bringing from the market, boiled to remove colour and other fine impurities may be found in the orange peel, and then dried at 50°C for 24 hours and the adsorbent thus processed was used in its original piece size.

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 copper concentrations. 1000 mg/l stock solution of copper was prepared by dissolving known weight of copper nitrate $Cu(NO_3)_2.4H_2O$ in one liter 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 copper concentrations were measured using spectrophotometer thermo – genesys 10 UV, USA.

Adsorption unit

Fixed bed column of continuous mode experiments were conducted in order to test copper removal by treated SSAS of above copper each one alone at desired concentration with the various bed heights of the adsorbent media orange peel using different flow rates of SSAS of copper at various pH. The pH value was adjusted using 0.1 N NaOH and 0.1 N HCl solutions. A schematic representation of the sorption unit is shown in Figure-1a and Figure-1b where the flow direction is downward by gravity. The sorption unit consists of glass container for SSAS of copper for inlet and another for outlet (5 liter) capacity. Glass column has 2.54 cm ID and 50 cm height. The sorption column packed with adsorbent media to a height of (10, 20, 30, 40, and 50 cm). Before starting the runs, the packed bed sorption column was rinsed by double distilled water down flow through the column. The orange peel is packed in the column to the desired depth, and fed to it as slurry by mixing the media orange peel 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 copper 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 (1 l/min) as shown in Figure-1a and Figure-1b. To determination the best operational conditions, the experiments were carried out at a constant temperature (25°C), various pH values, which are (1–8), and initial feed concentrations of SSAS of different copper which are between (1–100) mg/l and at constant flow rate (1 l/min) for copper initial feed concentration. Outlet samples after treatment in each experiment were collected every 10 minutes from the bottom of packed column and the unadsorbed concentration of copper ion in SSAS was analyzed by spectrophotometer.

RESULTS AND DISCUSSIONS

The ability of orange peel to remove copper from SSAS in fixed bed column of continuous mode at various parameters, which are pH's of SSAS of copper (*pH*), height bed of adsorbent media OP (*h*), and time of treatment (*t*) was investigated. The experiments were performed at constant flow rates of SSAS (*F*), SSAS temperature (T_{feed}) at 1 (l/min) and (25 °C) respectively and achieved by varying above parameters for different initial concentrations (C_o) of SSAS of copper. Thus, the results obtained are explaining below.

Effect of Initial Concentration

The results showed that using adsorbent material, the percent removal of copper was decreased when the initial concentration (C_o) of SSAS of copper was increased at constant other variables as shown in Figure-2. This can be explained by the fact that the initial concentration of copper had a restricted effect on copper 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 the increase in the number of copper molecules competing for the available functions groups on the surface of adsorbent material. Since the solution of lower concentration has a small amount of copper than the solution of higher concentration of it, so the percent removal was decreased with increasing initial concentration of copper. For adsorbent media, higher percent removal were 89.25 % for copper at initial copper concentration of 1 mg/l, so adsorbent material was found to be efficient to copper removal from SSAS and wastewater [16].
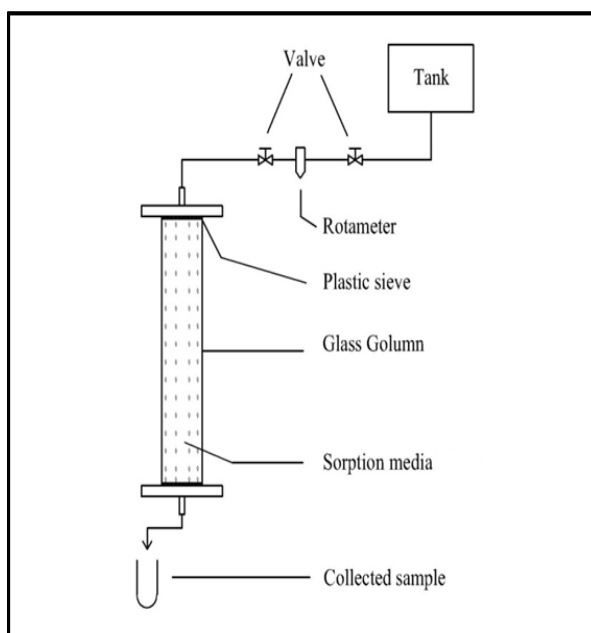
Fig-1a: The experimental setup for adsorption unit (sketch)

Fig-1b: The experimental setup for adsorption unit (photo)

Effect of pH

The results showed that using adsorbent material, the percent removal of copper was increased when the pH of SSAS of copper was decreased at constant other variables as shown in **Figure 3**. This decrease can be explained as follows: The adsorption of copper from aqueous solution is dependent on the pH of the solution, which affects the surface charge of the adsorbent, and the degree of ionization and speciation of the adsorbate species. This can be attributed to the depending of copper ionization on the pH value. Copper, will be adsorbed to a lesser extent at higher pH values due to the repulsive force prevailing at higher pH value. In addition, in the higher pH range copper forms salts, which readily ionize leaving negative charge on the copper ion. At the same time, the presence of OH^- ions on the adsorbent prevents the uptake of copper ions. pH also affects the surface properties of the sorbent, i.e., surface charge of the cells used as sorbent. At very low pH values, the surface of the sorbent would also be surrounded by the hydronium ions, which enhance the copper interaction with binding site of the sorbent by greater attractive forces, hence its uptake on polar adsorbent is reduced [17].

Effect of Adsorbent Media Bed Height

The results elucidated that when the adsorbent media bed height was increased, the percent removal of copper was increased too at constant other variables as shown in Figure-4. The increased of bed height (*h*) meaning increased in the amount of adsorbent media orange peel, 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 copper removal capacity on orange peel. This lead to increase the ability of adsorbent media to adsorb greater amount of copper from SSAS at different initial concentrations and ultimately the percent removal of copper increased [18].

Effect of Treatment Time

The results demonstrated that when the treatment time of SSAS of copper increased the percent removal of copper increased at constant other variables as shown in Figure-5. This result may be due to the fact that when the time of treatment of SSAS of copper increasing and the velocity of SSAS in the column packed with the adsorbent material was kept constant, SSAS spend longer time than spend it when the time of treatment decreased, so the adsorbent material uptake more amount of copper from SSAS, therefore the percent removal of copper from SSAS was increased [19].
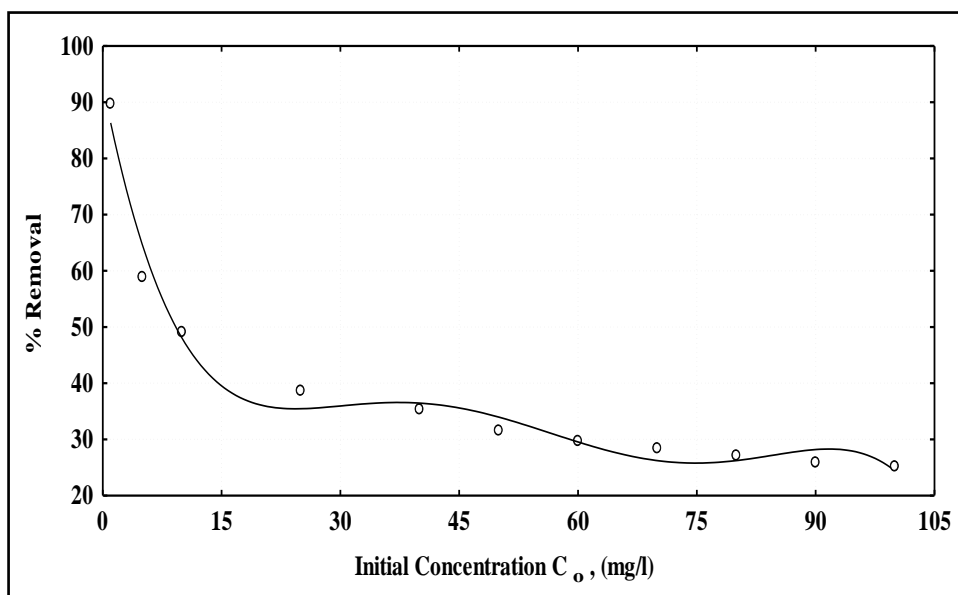
Fig-2: Effect of initial concentration ($C_\circ$) on the percent removal of copper @ T_f =25°C, h_b = 50 cm, *pH*=1, *t*=60 min. and *F*=1 l/min.
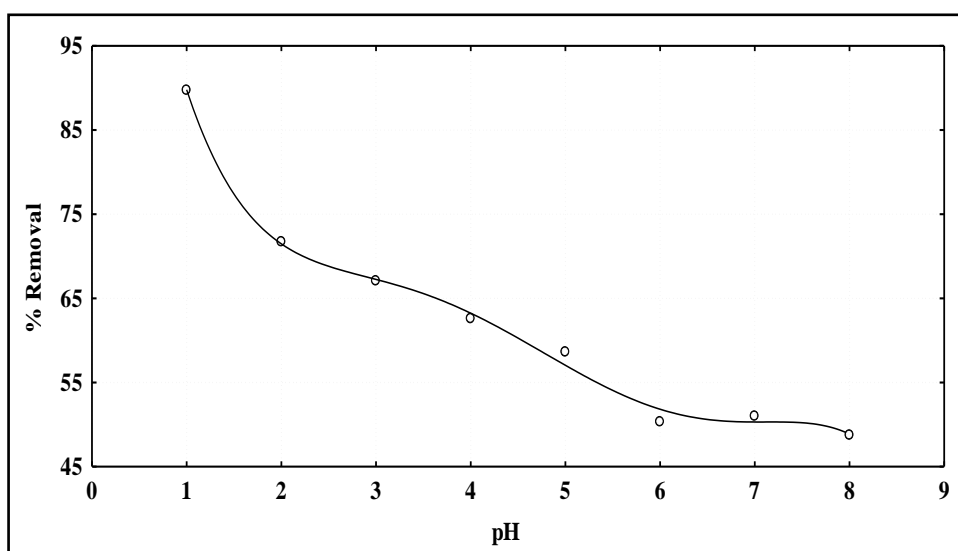
Fig-3: Effect of *pH* on the percent removal of copper @ $C_\circ$ = 1 mg/l, T_f =25°C, *h* = 50 cm, *t*=60 min. and *F*=1 l/min.
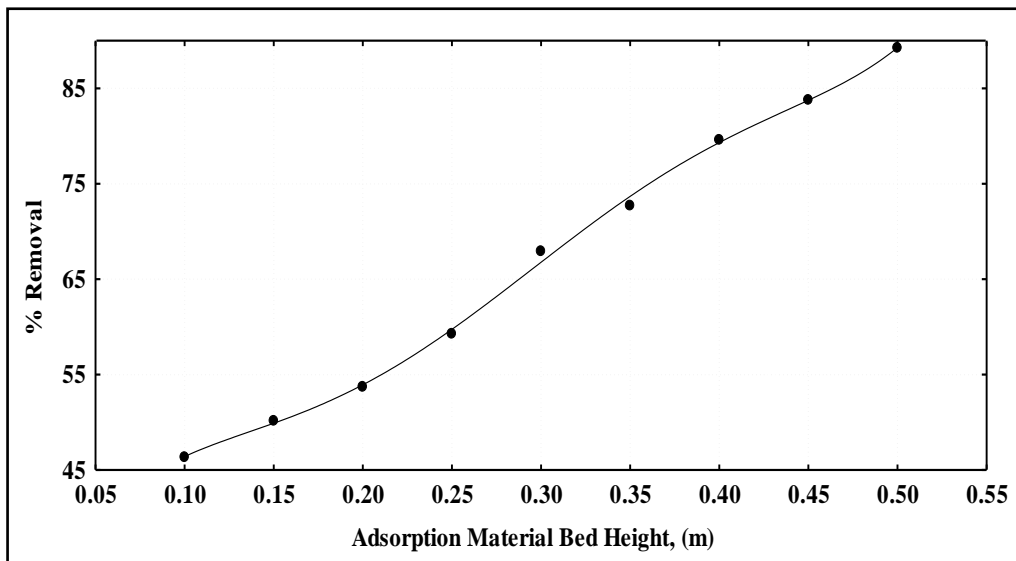
Fig-4: Effect of adsorbent media bed height (*h*) on the percent removal copper @ $C_{\circ}$ = 1 mg/l, *pH*=1, T_f =25°C, *t*=60 min. and *F*=1 l/min.
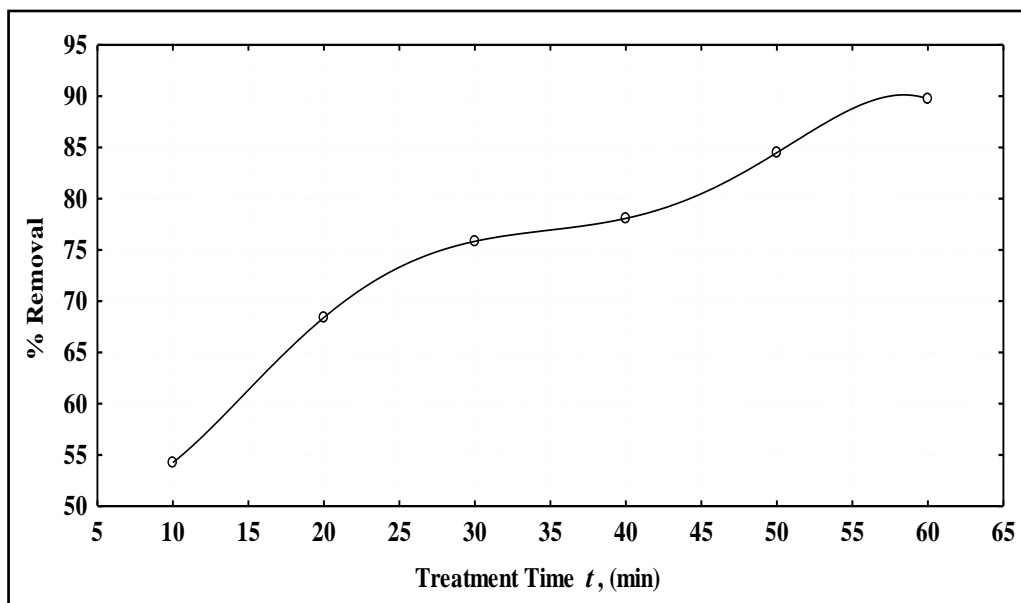
Fig-5: Effect of treatment time (*t*) on the percent removal of copper @ $C_{\circ}$= 1 mg/l, T_f =25°C, *pH*=1, *h* = 50 cm, and *F*=1 l/min.

Adsorption Isotherm

Langmuir and Freundlich models are used to describe the adsorption isotherm behavior in the present study [20].

Langmuir isotherm model

The Langmuir adsorption isotherm model supposes the adsorption surface is monolayer, homogeneous and the adsorption sites have similar adsorbate ability and each side in the adsorption surface independent on the adsorption in the neighboring sites. Langmuir isotherm model can be described by equation (1):

$$q_{eq} = \frac{q_m . K_L . C_{eq}}{1 + K_L . C_{eq}} \qquad \dots (1)$$

where: q_{eq}: is the copper ion quantity adsorbate per unit mass of adsorbent at equilibrium concentration C_{eq}, C_{eq}: is the adsorbate concentration in the solution at equilibrium, q_m: is the amount of solute adsorbed per unit mass of

adsorbent in forming a complete monolayer on the adsorbent surface and K_L: is a constant related to the energy or net enthalpy of adsorption (l/mg).

For very small amount of adsorption, when $K_L C_{eq} \ll 1$ it yields a linear adsorption isotherm: $q_{eq} = q_m K_L C_{eq}$. For large amount of adsorption, when $K_L C_{eq} \gg 1$ it will be equal: $q_{eq} = q_m$, Thus, the Langmuir isotherms model can be represented by linear form as:

$$\frac{C_{eq}}{q_{eq}} = \frac{1}{K_L q_m} + \frac{1}{q_m} C_{eq} \qquad \qquad ...(2)$$

Plotting of C_{eq}/q_{eq} against C_{eq} will give a straight line, has an intercept of $(1/K_L q_m)$ and the slop is $(1/q_m)$.

Freundlich isotherm model

The Freundlich adsorption isotherm model is an empirical equation and has been widely used for long time ago. This model depicts the equilibrium on heterogeneous surfaces and assumes multilayer capacity. Usually, for moderate concentrations, Freundlich model is completely concurred with Langmuir model and the experimental data, but in contrast, this isotherm model cannot represent a linear form in very low concentrations as in Langmuir adsorption isotherm model. However, Freundlich isotherm model can be described by equation (3) bellow:

$$q_{eq} = K_F C_{eq}^{\frac{1}{n}} \qquad \qquad ...(3)$$

Where n and K_F are the Freundlich isotherm constants.

Equation (3) can be rearranging to obtain the linear form of Freundlich adsorption model. The Freundlich isotherm model can be represented by linear form as:

$$\log q_{eq} = \log K_F + \frac{1}{n} \log C_{eq} \qquad \qquad ...(4)$$

The calculation of n and K_F values can be performed via plotting $\log q_{eq}$ vs. $\log C_{eq}$ where the intercept is $\log K_F$ and the slop is $1/n$

Adsorption isotherms calculations

The adsorption isotherm constants were calculated from the slopes and intercepts of (Langmuir isotherm) and (Freundlich isotherm) and presented in Table-3. The values of calculated correlation coefficients R^2 were higher for Langmuir isotherm than the Freundlich isotherm; that means Langmuir isotherm model represented the adsorption process well than Freundlich isotherm model. The maximum capacity of orange peels for copper ion removal copper ions was calculated in the range of 15.228 mg/g at constant temperature that indicated the best adsorbing capacity of orange peel for copper ion removal copper ions.

Table-3: Values of Langmuir and Freundlich isotherm constants for the adsorption of Copper ions on Orange peels at Optimum Conditions

Langmuir Isotherm Model				Freundlich Isotherm Model		
q_m	K_L	R_L	R^2	K_F	$1/n$	R^2
27.0182	0.04166	0.751	0.9239	2.3842	0.5408	0.9616

Adsorption Kinetics

In this paper, the adsorption kinetics is studied due to the important of it's to determine the physical and chemical behaviour of the adsorbent media and also to predict the mechanism of adsorption process occurred. Three kinetic models are proposed to investigate the experimental data obtained from the adsorption of copper ions on orange peels. These kinetic models are first order reversible model, pseudo first order model and pseudo second order model.

Pseudo-first order model

This model is depended on the capacity of solid adsorbate. It is assumed that adsorption between liquid and solid occurred on one layer on the adsorption surface (adsorbate). The mathematical expression of this model is described by Equation (7):

$$q_{eq} - q = e^{-k_1 t} \qquad \ldots (7)$$

Where: q_{eq}: is the copper ion quantity adsorbate per unit mass of adsorbent at equilibrium (mg/g), q: is the copper ion quantity adsorbate per unit mass of adsorbent at any time (mg/g), t: is the time and (k_1): is the first order rate constants (min⁻¹). The linear form of this model can be represented by equation (8):

$$\ln(q_{eq} - q) = \ln q_{eq} - k_1 t \qquad \ldots (8)$$

So from the plot of $\ln(q_{eq} - q)$ vs. t it can be calculating the rate constant k_1 which appears as the slop of the equation, while the intercept is equal to $\ln q_{eq}$.

Pseudo-second order model
Ordinarily it is the best model for expressing the kinetic model of adsorption process. It is assumed that adsorption between adsorbent (solid phase) and adsorbent (liquid phase) performed on two layer on the adsorption surface (adsorbate). This model is described by Equation (9):

$$q = \frac{q_{eq}^2 k_2 t}{1 + q_{eq}^2 k_2 t} \qquad \ldots (9)$$

Where: q_{eq}: is the copper ion quantity adsorbate per unit mass of adsorbent at equilibrium (mg/g), q: is the copper ion quantity adsorbate per unit mass of adsorbent at any time (mg/g), t: is the time (min) and (k_2): is the first second order rate constants (g.mg⁻¹.min⁻¹). The linear form of this model can be represented by equation (10):

$$\frac{t}{q} = \frac{1}{q_{eq}^2 k_2} + \frac{1}{q_{eq}} t \qquad \ldots (10)$$

From linearized form it is clear when plotting (t/q) against t the slop of equation 10 is $(1/q_{eq})$ while the intercept is $(1/q_{eq}^2 k_2)$

Adsorption kinetic calculations
The adsorption kinetic constants and correlation coefficients of three models used in this study were calculated and given in Table-4. Good correlation coefficients R^2 were observed for the copper ion uptake process and can be approximated with the pseudo second order kinetics model. Constants k_1 and k_2 for all kinetics models tested have been calculated and summarized in Table-4. Thus, the pseudo second order kinetics was pathway to reach the equilibrium.

Table-4: Values of pseudo first order and pseudo second order for the adsorption of copper on orange peels at Optimum Conditions

Pseudo first order			Pseudo second order			Experimental
q_{eq}	k_1	R^2	q_{eq}	k_2	R^2	q_{eq}
6.1034	5.439 ×10⁻²	0.931	8.527	1.725 ×10⁻²	0.9800	9.1158

Utilization of Wastes after Adsorption Process
Orange peels after used as an adsorption media to remove copper ions from SSAS (*as explained in experimental work mentioned above*) was collected and set up to the next step which is exploited it as a raw material for preparation of a simple and cheap rodenticide, the main goal of this paper. To perform this purpose, 20 groups (10 groups for males and 10 groups for females) of outbred multipurpose breed of albino rats, which are (Sprague dawley) and have scientific name (*Rattus rattus*). Each group (cage) contains 10 rats. Their weight ranged between (250-300) g and their age ranged between (6-10) months. In addition to that, there are another two control groups, (CG 1) and (CG 2) where the rats in which (males and females) were feeding with natural rat provender only and natural with non-contaminated orange peels (*non-adsorbed copper ions*) respectively to compare the results. Before testing, all rats were left for one week in clean cages and laboratory conditions suitable for living. The rats were feeding with normally rat provender to ensure that no rats suffer from any disease or ill-treatment that leads to change the results and for adapting to the place and food (provender) before starting experiments. The temperature was between (25±2°C) and the lighting was 14 hours per day using an ordinary light bulb [21]. After adoption to the place and provender, 120 male rats and 120 female rats were divided randomly to 12 groups for males and females as 10 rats/group, and the rats were treated daily for one week. After

the week, the rats were feeding by orange peels that adsorbed copper ions directly (without any further treatment) mixed with provender with very little of sweet food in order to entice the rats. These sweet foods were replaced continuously to avoid any aversion by the rats. The results showed that there were fatalities among males and female rats in all cages (except for rats in the control groups CG1 and CG2). The fatalities occur at unlike ratios and at different times depending on the amount of copper ions loaded on orange peels consumed by rats. The half lethal dose (LD_{50}) was also calculated, and the results determined were as explained in Table-1. From Table-1 it is noted that the value of LD_{50} calculated for prepared rodenticide in this study is identical with the LD_{50} cited in the literature and references. Thus, this way can be considered as one of the means that dispose of more than one type of harmful and contaminated wastes at the same time with non-cost, simple and eco-friendly method accessing to zero residue level (ZRL) [22].

Table-1: The Half Lethal Dose (LD_{50}) for Rodenticide Prepared from Orange Peels Residue Loaded with Copper ions

Type of food consumed by rats	The Half Lethal Dose (LD_{50}) Calculated for Male Rats (according to this study) , (mg/kg)	The Half Lethal Dose (LD_{50}) Calculated for Female Rats (according to this study), (mg/kg)	The Half Lethal Dose (LD_{50}) for Rats according to Literature
Control Group 1 (CG1): Feeding: Ordinary rat provender	There are no fatalities	There are no fatalities	-
Control Group 2 (CG2): Feeding: Ordinary rat provender + non-contaminated OPR only	There are no fatalities	There are no fatalities	-
Other Groups: Feeding: Ordinary rat provender + OPR loaded with Cu^{+2} ions	17.938	28.495	5.72 [23] – 30 [24]

Generally, the inorganic rodenticides are one of important pesticide types which are used to prevent or expel or kill or control rodents. The active components in these pesticides predominantly are compounds of chromium, arsenic, lead, tin, cyanide, phosphorus and copper as inorganic compounds [20, 25, 26]. There are many ways to use rodenticides which contain inorganic materials as active compounds, some of which are placed near their whereabouts as poisonous baits food that can be consumed by rats through oral nutrition. Another type of rodenticides is sprinkled like a powder in the path of rodents, attach to their bodies and enter the digestive system by cleaning the body. Or the poisonous pesticide is putting on an adhesive and when rat attach by the adhesive material, the toxic substance enters the body and is able to kill it. The rodenticide presents in this investigation follows the first type. Orange peels loaded with toxic copper ions were given to the rats by mixing it with very little sweet material approximately (1-5 wt %) of given food amount. Copper compounds are considered as one of distinctive substances that can be successfully used in the preparation of rodenticides due to their guaranteed effectiveness and toxic effects. Moreover, they are non-degradable and can remain for a long time without changing their composition.

CONCLUSIONS
The following conclusions can be drawn:
- Orange peel showed a good ability to remove copper from SSAS using fixed bed adsorption unit. So, it could be recommended for removal of copper from wastewater by this material because it is valid, cheaper, economical, easy and simplicity for using, and has a high ability to adsorb copper, can be used several times by costly regeneration method and can be used finally in another benefit uses.
- Maximum removal of copper was 89.25 % at initial copper concentration of 1 mg/l.
- The percentage removal of copper was increased with decreeing pH and initial concentration of copper while the percentage removal was increasing with increasing of treatment time and the height of adsorbent material orange peel.
- It can be prepared a good rodenticide for control rodent from the residual samples of orange peels residue that adsorb copper ions above from SSAS.

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