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# بررسی تعادل و سینتیک جذب زیستی مس (II) بر روی زائده ی پودر چای و قهوه

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# Equilibrium and Kinetic Studies of Cu (II) Biosorption Onto Waste Tea and Coffee Powder (WTCP)

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## چکیده

جذب زیستی یون‌های مس (II) از محلول‌های آبی توسط زائده‌ی پودر چای و قهوه به روش ناپیوسته مورد بررسی قرار گرفت. فرآیند جذب به pH محلول، غلظت اولیه یون فلزی، مقدار بیوجاذب و زمان تماس بستگی دارد. داده‌های جذب تعادلی که به طریق آزمایشی بدست آمده بودند توسط ایزوترم‌های لانگمویر، فروند لیچ، تمکین و دابینین-رادشکویچ مورد بررسی قرار گرفتند. مدل لانگمویر با ضریب همبستگی بالاتر تطابق بیشتری با داده‌ها داشت. بیشترین ظرفیت-های جذب با استفاده از ایزوترم لانگمویر به ترتیب ۵۲۶ و ۴۱۷ میلی‌گرم بر گرم برای قهوه و چای بدست آمد. مطالعات سینیتکی بیانگر آن بود که مراحل جذب توسط مدل شبه درجه دوم انجام می‌گیرد. براساس ظرفیت جذب، زائده‌ی پودر قهوه و چای می‌تواند به عنوان جاذب زیستی کم هزینه، موثر و دوست‌دار محیط زیست برای حذف یون‌های مس (II) از محلول‌های آبی مورد توجه قرار گیرد.

## واژه‌های کلیدی

جذب زیستی؛ مس؛ تعادل؛ مطالعه سینتیک.

## Abstract

Biosorption of Cu(II) ions from aqueous solutions onto waste Tea and Coffee powder (WTCP) has been investigated in a batch biosorption process. The biosorption process was found to be dependent on pH of solution, initial metal ion concentration, biosorbent dose, and contact time. The experimental equilibrium biosorption data were analyzed by Langmuir, Freundlich, Temkin and Dubinin−Radushkevic isotherms models. The Langmuir model gave a better fit than the other three models by higher correlation coefficient, $R^2$. The maximum biosorption capacities calculated from the Langmuir isotherm model were 526 and 417 mg/g, for coffee and tea, respectively at optimum conditions. The kinetic studies indicated that the biosorption process of the metal ions followed well pseudo-second-order model. According to the biosorption capacity, waste coffee and tea powder considered as an effective, low cost, and environmentally friendly biosorbent for the removal of Cu (II) ions from aqueous solutions.

## Keywords

Biosorption; Copper; Equilibrium; Kinetic Study.

## 1. INTRODUCTION

Expanded use of chemicals in our daily life and most industry processes have brought extensive amount of toxic heavy metals and their presence pose environmental-disposal problems. Our biological system has been sullied by high concentration of heavy metals released into the biosphere. Modern exercises, development, urban waste treatment, and vehicle deplete are a portion of the sources causing substantial amounts of heavy metal tainting in climate, water, and soil [1]. Copper, a widely used material in the world, is used as an excellent conductor of electricity and electronic devices. Copper metal contamination exists in aqueous waste streams from many industries constitutes one of the most serious pollution problems and can affect the quality of water supply and cause many problems on aquatic life [2].

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Several treatment technologies have been developed for the removal of copper from waters and waste waters such as solvent extraction, ultrafiltration, membrane filtration, ion exchange, and precipitation [3-12]. But these methods are not only expensive, they also create industrial problems. In recent years adsorption techniques have been shown to be the most promising option for the removal of copper from aqueous streams [13]. Activated carbon as an adsorbent has been widely investigated for the adsorption of copper due to their effectiveness and versatility, but it's high-cost limited its use in commercial application. Additionally, some difficulties have been reported for activated carbon in terms of engineering problems [14]. There are several different kinds of waste byproducts which have been used to remove heavy metals such as sunflower stalks, Eucalyptus bark, rice husk, wheat bran, mushroom biomass, fruit peel of orange, and Ficus religiosa leaves [15].

In our previous work, we have studied the efficiency of olive leaves and orange peels powders as a biosorbent for removal of copper from aqueous solutions [16,17]. In this paper, the adsorption of copper onto waste coffee and tea powders has been investigated. The adsorption capacity was evaluated under various conditions of pH, contact time, initial copper concentration, and adsorbent dosage. The adsorption isotherms were also investigated to explain the probable mechanism of adsorption and to provide several information such as maximum sorption capacity, energy of sorption, homogeneity/heterogeneity, and affinity between sorbent and adsorbent.

## 2. EXPERIMENTAL

### *1. 2. Reagents*

All chemicals used were of analytical reagent (AR) grade. 5000 ppm Cu (II) stock solution was prepared by dissolving an appropriate amount of $CuSO_4.5H_2O$ salt in deionized water. The stock solution was diluted to the required concentrations using deionized water. The solution pH was adjusted using 0.10 M HCl or 0.10 M NaOH.

### *2. 2. Determination of Cu (II)*

The concentration of Cu(II) ions in the solutions before and after equilibrium was determined by Molecular Absorption Spectrophotometer 6305 from JENWAY. The pH of the solution was measured with pH Meter 3505 from JENWAY. The determination of copper (II) as ammonia complex ($Cu(NH_3)_4^{2+}$) was carried out according to the published work [18]. The range of calibration curve concentrations of metal ions prepared from stock solution varies between 100-1000 ppm.

### *3. 2. Preparation of Adsorbents*

The coffee and tea waste powders were collected from a coffee shop, washed several times with double distilled water and then dried in an oven at $70^o$ C for 24 hrs. The dried materials were sieved through 500 µm size fraction using an American Society for Testing and Materials (ASTM) standard sieve.

### *4. 2. Adsorption Experiments*

The adsorption experiments were carried out in a series of 150 mL Erlenmeyer flasks containing 50 ml of metal ions solution, 0.100 g adsorbent powder and if necessary, an appropriate volume of HCl or NaOH solutions was used to adjust the pH of the solution. The solutions were shaken (175 rpm) at 25°C. Then solutions were filtered by Whatman filter paper. The removal percentage (% R) was calculated according to the following equation:

$$\% \, R = \frac{C_o - C_e}{C_o} \, X \, 100 \qquad (1)$$

Where: $C_o$ and $C_e$ are initial and final concentrations in ppm, respectively. The amount of adsorbed metal $Q_e$ mg/g (mg metal per gram adsorbent) was calculated based on the difference between the initial ($C_o$, ppm) and final concentration ($C_e$, ppm) in every flask, as follows:

$$Q_e = \frac{C_o - C_e}{M} \, X \, V \qquad (2)$$

where $Q_e$ is the metal uptake capacity (mg/g), V the volume of the metal solution in the flask (L) and M is the dry mass of biosorbent (g).

### *5. 2. Parameters Affecting Adsorption Experiments*

The following experiments were carried out to study the influence of pH, biosorbent dose, contact time, and metal ion concentration on adsorption of copper onto coffee and tea waste powders. I) The pH dependent study was carried out in series of 50 mL aqueous solutions of 400 ppm Cu (II) and containing 0.1 g powder. The investigated pH values for these solutions were varied between 1.5 to 6. The contact time was set at 30 min. II) The effect of contact time on Cu (II) adsorption was examined for series of 50 mL solutions with 400 ppm concentration and containing 0.1 g powder. The pH for all solutions was set to the optimum from (1). The contact time was varied for each solution between 10–60 min. III) The effect of biosorbent doses on metal uptake was investigated using series solutions with adsorbent masses of 0.1, 0.2, 0.4, 0.6, 0.8, and 1 g per 50 mL of 400 ppm Cu (II) solution.

The optimum pH and contact time were chosen. IV) Finally, various concentrations of Cu (II) were investigated to study the effect of metal ion concentration which were: 500, 600, 700, 800, 900, 1000 ppm per 50 mL solutions contain 0.1 g biosorbent. The optimum pH and contact time were chosen.

## 3. RESULT AND DISCUSSION

By UV VIS Spectrophotometer, the concentrations of copper (II) as ammonia complex in the solutions were determined [18]. The range of calibration curve of copper ions prepared from stock solution varies between 100-1000 ppm as shown in Fig. 1. The response of the Cu-complex was found to be linear in the investigation concentration range at $\lambda_{max}$ = 620 nm and the linear regression equation was y = 0.0011X with high correlation coefficient ($R^2$= 0.998). From the calibration curve, the concentrations of copper (II) in the solutions before and after equilibrium adsorption were determined.

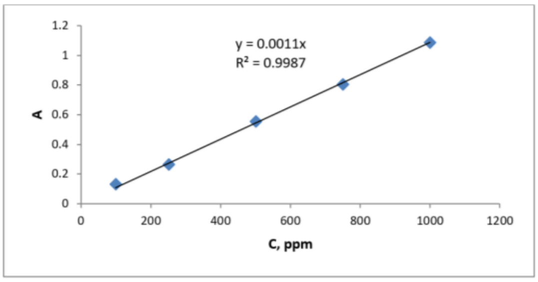

**Fig. 1.** Calibration curve for Cu - Complex at $\lambda_{max}$ = 620 nm.

### *1. 3. Effect of pH*

The biosorption and removal of metal ions from wastewater and aqueous solutions are strongly controlled by pH changes. The pH of the solution is considered to be as the most important parameters governing metal up taking and adsorption on adsorbent substrate. The adsorption of copper ions on (WTCP) was monitored over a range of pH from 1.00 to 5.50 of separate solutions Fig. 2 a & b. Low percent removal were observed at low pH values because of $H_3O^+$ ions will compete with Cu(II) for binding and also surrounded ($H_3O^+$) will prevent the copper ions from approaching the binding sites. However, at higher pH, (above pH 5), the Cu (II) started precipitating, so the removal was not achieved only by adsorption. The maximum percent removal % R and uptake capacity $Q_m$ of copper ions by both absorbents (WTCP) were observed at pH 4.0 - 4.5. Therefore, the remaining all biosorption experiments were carried out at this pH values.

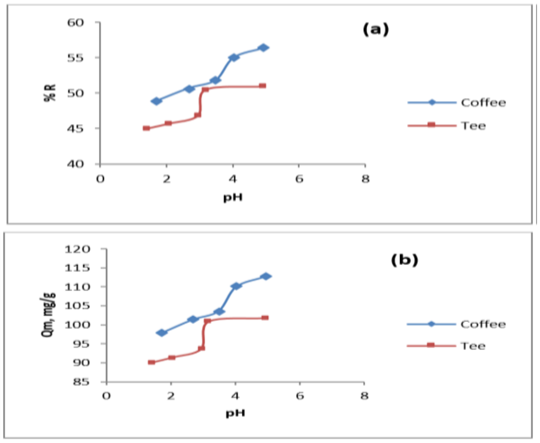

**Fig. 2.** Effect of PH on (a) percent removal of Cu (II) (% R) and (b) Copper uptake $Q_m$ onto biosorbent (WTCP).

### *2. 3. Effect of Initial Metal Concentration:*

The effect of different initial copper ion concentrations on equilibrium of biosorption experiments onto (WTCP) were investigated from 500 to 1000 ppm at pH 4.00 – 4.50. The percent removal % R of Cu(II) from solutions was a little bit decreasing with an increasing the initial concentration of metal as shown in Fig. 3 a. The relation between copper uptake $Q_m(mg.g^{-1})$ and initial metal ion concentration is shown in Fig. 3.b. The Copper ion adsorption is possible at lower concentrations, but as the concentration is increased, the driving force also increased, which favored the adsorption at higher concentrations. The equilibrium uptake of the adsorbent (WTCP) was observed increasing gradually with an increasing the initial concentration of Cu ions and then reach a maxima at a concentration of 1000 ppm. The increasing of adsorption capacity with the increasing in metal ions concentration is probably due to higher interaction between the metal ions and sequestering sites of biosorbent.

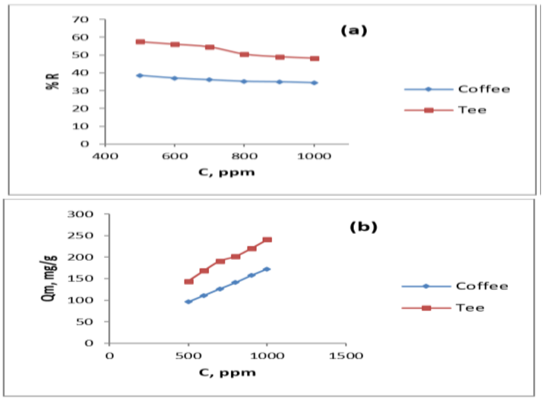

**Fig. 3.** Effect of initial concentration on (a) percent removal of Cu (II) (% R) and (b) Copper uptake $Q_m$ onto absorbent (WTCP).

### *3. 3. Effect of contact time*

The rate of biosorption is an important for designing batch biosorption experiments. Therefore, the effect of contact time of copper biosorption on (WTCP) was investigated Fig. 4. The biosorption of Cu(II) ions on (WTCP) increased considerably until the contact time reached 40 min at 25ºC**.** Further increase in contact time did not enhance the biosorption, so, the optimum contact time was selected as 40 min for further biosorption experiments.

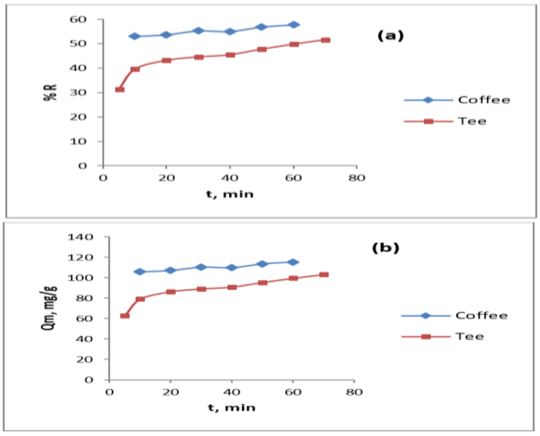

**Fig. 4.** Effect of contact time on (a) percent removal of Cu (II) (% R) and (b)Copper uptake $Q_m$ onto absorbent (WTCP).

### *4. 3. Effect of biosorbent dose on biosorption*

The biosorbent dosage of (WTCP) is an important parameter because this determines the suitable capacity of a biosorbent for a given metal concentration. The biosorption efficiency for Cu(II) ions as a function of biosorbent dosage (WTCP) was investigated Fig. 5. The percentage of the metal biosorption increases with the biosorbent loading up to 1 g/L.

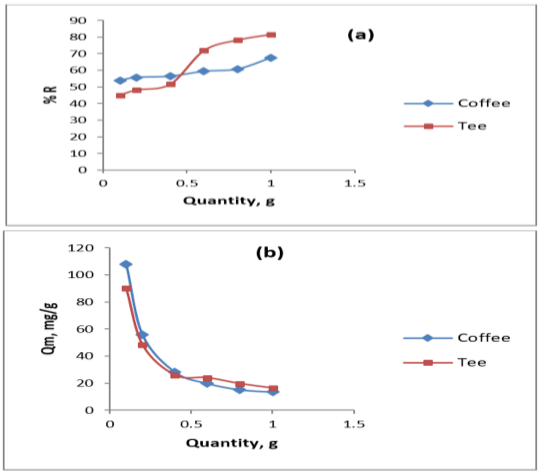

**Fig. 5.** Effect of adsorption dose on (a) percent removal of Cu (II) (% R) and (b) Copper uptake $Q_m$ onto absorbent (WTCP).

However, the metal uptake decreases with biosorbent dose. This result can be explained by the fact that the biosorption sites in (WTCP) remain unsaturated during the biosorption reaction whereas the number of sites available for biosorption site increases by increasing the dose of (WTCP). The further experiments were taken as 1 g/L because the maximum percentage of the metal biosorption was attained at 1.0 g/L of (WTCP) dosage Fig. 5 a & b.

### *5. 3. Biosorption Isotherms*

An adsorption isotherms describe the fraction of sorbate molecules that are partitioned between liquid and solid phases at equilibrium**.** Biosorption of Cu(II) ions onto waste tea and coffee powder (WTCP) was modeled using four adsorption isotherms.

The Langmuir isotherm assumes as monolayer adsorption on a uniform surface with a finite number of adsorption sites. Once a site is filled, no further sorption can take place at that site. As such the surface will eventually reach a saturation point where the maximum adsorption of the surface will be achieved. The linear form of the Langmuir isotherm model is described as [19]:

$$\frac{1}{Q_e} = \frac{1}{Q_m} + \frac{1}{b\,Q_m C_e} \qquad (3)$$

where b is the Langmuir constant related to the energy of adsorption(L.mg$^{-1}$) and $Q_m$is the maximum biosorption capacity (the monolayer adsorption saturation capacity mg/g), $Q_e$ (mg/g) the amount of metal ion bound per gram of the adsorbent at equilibrium; and $C_e$, the residual (equilibrium) metal ion concentration left in the solution after binding. Values of Langmuir parameters $Q_m$ and b were calculated from the slope and intercept of the linear plot of $1/Q_e$ versus $1/C_e$ as shown in Fig. 6**.** Values of $Q_m$, b and regression coefficient $R^2$ are listed in Table 1.

**Table1.** Langmuir, Freundlich, D-R and Temkin isotherm constants for biosorption of metal ions onto WTCP.

| Langmuir | $Q_m$ | b | $R_L$ | $R^2$ |
|---|---|---|---|---|
| Coffee | 526.3 | 7.16x10$^{-4}$ | 0.776 | 0.9956 |
| Tea | 416.7 | 25.0x10$^{-4}$ | 0.500 | 0.9866 |
| Freundlich | $K_f$ | n | | $R^2$ |
| Coffee | 1.158 | 1.300 | | 0.9982 |
| Tea | 7.967 | 1.838 | | 0.9808 |
| Temkin | B | A | | $R^2$ |
| Coffee | 10.75 | 122.18 | | 0.9844 |
| Tea | 10.51 | 51.64 | | 0.9828 |
| Dubinin-Radushkevich | $Q_d$ | β | | $R^2$ |
| Coffee | 188.02 | 0.0256 | | 0.9366 |
| Tea | 250.21 | 0.0098 | | 0.9601 |

These values indicated that Langmuir model describes the biosorption phenomena favorable.The level of conformity for coffee is high compared to Tea sorbent, according to the correlation coefficients ($R^2$) of 0.995 and 0.986, respectively. The essential characteristics of the Langmuir isotherm parameters can be used to predict the affinity between the sorbate and sorbent using separation factor or dimensionless equilibrium parameter, $R_L$ expressed as in the following equation:

$$R_L = \frac{1}{1 + b\,C_0} \tag{4}$$

The value of $R_L$ indicated the type of Langmuir isotherm to be irreversible ($R_L$=0), favorable (0 <$R_L$< 1), linear ($R_L$=1) or unfavorable ($R_L$>1). In concentration range of 500 to 1000 mg/L of Cu ions, the values $R_L$ of biosorbed (WTCP) were found to be 0.776 and 0.500 respectively.They are in the range of 0.0-1.0 which indicates the favorable biosorption.

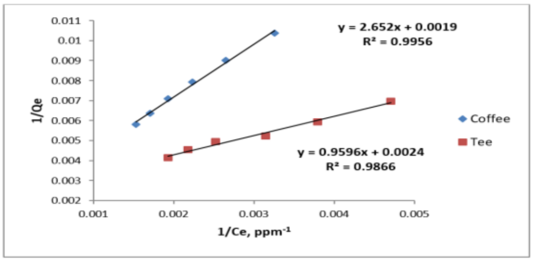

**Fig. 6.** Langmuir adsorption isotherm for the removal of Cu(II) by adsorption on (WTCP).

The Freundlich isotherm model is the well known earliest relationship describing the adsorption process. This model applies to adsorption on heterogeneous surfaces with the interaction between adsorbed molecules and the application of Freundlich equation. Also suggests that sorption energy exponentially decreases on completion of the sorption centers of an adsorbent. This isotherm is an empirical equation and can be employed to describe heterogeneous systems and is expressed as follows in linear form [20]:

$$\text{Log}\,Q_e = \text{Log}\,K_f + \frac{1}{n}\,\text{Log}C_e \tag{5}$$

where $K_f$ is the Freundlich constant related to the bonding energy. 1/n is the heterogeneity factor and n (g/L) is a measure of the deviation from linearity of adsorption. Freundlich equilibrium constants were determined from the plot of log$Q_e$ versus log $C_e$, Fig. 7 on the basis of the linear of Freundlich equation. The n value indicates the degree of non-linearity between solution concentration and adsorption as follows: if n = 1, then adsorption is linear; if n < 1, then adsorption is a chemical process; if n > 1, then adsorption is a physical process. The n value in Freundlich equation was found to be 1.3and 1.838 for tea and coffee respectively (Table 1). Since n lie between 1 and 10, this indicates the physical biosorption of Cu ions onto tea and coffee is suitable.

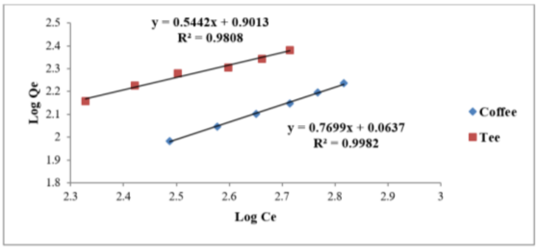

**Fig. 7.** Freundlich adsorption isotherm for the removal of Cu(II) by adsorption on (WTCP).

Temkin isotherm equation [21] assumes that the heat of biosorption of all the molecules in the layer decreases linearly with coverage due to adsorbent-adsorbate interactions and that the adsorption is characterized by a uniform distribution of the binding energies up to some maximum binding energy. The Temkin isotherm has been used in the linear form as follows:

$$Q_e = B\,\text{Log}\,A + B\,\text{Log}\,C_e \tag{6}$$

The plot of $Q_e$ versus log $C_e$ enables the determination of the isotherm constants B and A obtained from the slope and the intercept, Fig. 8. Where A (L/mg) is the equilibrium binding constant corresponding to the maximum binding energy and constant B is related to the heat of biosorption, Table 1.

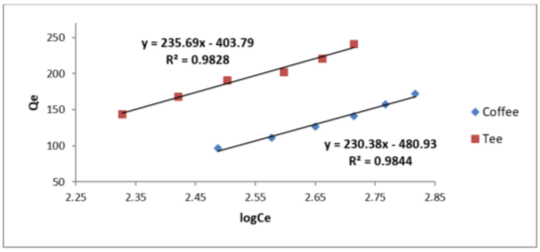

**Fig. 8.** Temkin adsorption isotherm for the removal of Cu(II) by adsorption on (WTCP).

Dubinin-Radushkevich (D-R) model , which does not assume a homogenous surface or a constant biosorption potential as the Langmuir model, was also used to test the experimental data [22].

$$\text{Log}\,Q_e = \text{Log}\,Q_d - \beta\,\varepsilon^2 \tag{7}$$

Where ε can be correlated to $C_e$ (mg/L) by the following equation:

$$\varepsilon = RT\,\text{Log}\left(1 + \frac{1}{C_e}\right) \tag{8}$$

Where R is the universal gas constant (8.314 J/mol K) and T is the absolute temperature (K) . The D-R isotherm parameters β and $Q_d$ were obtained from the slope and intercept of the plot of log $Q_e$ versus $\varepsilon^2$, Fig. 9. The calculated vales are listed in Table 1. The values of correlation coefficient were lower than that of other three isotherm values. In all cases, the D-R model represents the least fit to experimental data than the other isotherms models.

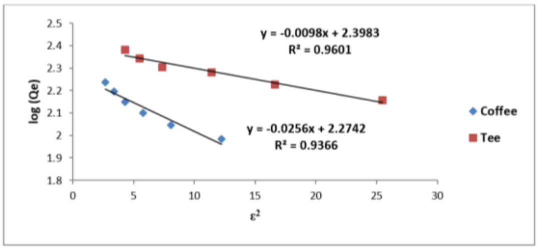

**Fig. 9.** Dubinin-Radushkevich (D-R) adsorption isotherm for the removal of Cu(II) by adsorption onto (WTCP).

The mean free energy of biosorption, E defined as the free energy change when 1 mole of ion is transferred to the surface of the solid from infinity in solution and calculated from the β value using the following equation:

$$E = \frac{1}{\sqrt{2\,\beta}} \qquad (9)$$

If the magnitude of E is between 8 to 16 KJ/mol then the sorption process is supposed to proceed via chemisorption reaction, while for values of E is less than 8 kJ/mol, the sorption process is of physical nature. The value of E calculated from equation (9) for the biosorption of metal ions by waste tea and coffee powder is 0.13 KJ/mol. This indicates that the biosorption process is of physical nature.

### *6. 3. Biosorption Kinetics*

To examine the biosorption kinetics of metal uptake onto (WTCP), the two kinetic models (pseudo first-order and pseudo second-order) were fit to experimental data. The pseudo-first order equation of Lagergren [23] is generally expressed as follows:

$$\frac{dQ_t}{dt} = k_1\,(Q_e - Q_t) \qquad (10)$$

Where $Q_e$ and $Q_t$ are the sorption capacities at equilibrium and at time t, respectively and $k_1$ is the rate constant of pseudo-first order sorption. The integrated form of equation (10) at boundary conditions, from $Q_t$ = 0 to $Q_t$ , and t = 0 to t; becomes:

$$\text{Log}\,(Q_e - Q_t) = \text{Log}\,Q_e - k_1\,t \qquad (11)$$

In order to fit the experimental data by integrated form of pseudo-first order equation, the equilibrium sorption capacity, $Q_e$ must be known. Furthermore, one has to find some means of extrapolating the experimental data to t = 0, on treating $Q_e$ as an adjustable parameter to be determined. For this reason, and for analyze the pseudo-first order model kinetics it is therefore necessary to use trial and error to obtain the equilibrium sorption capacity. The equation applicable to experimental results generally differs from a traditional first order equation in two ways: the terms $k_1$ and $(Q_e - Q_t)$ does not represent the number of available sites; and the log $Q_e$ is an adjustable parameter which is often not found equal to the intercept of a plot of log $(Q_e - Q_t)$ against t, where as in a true first order sorption reaction log $Q_e$ should be equal to the intercept and rate constant can be obtained from the slope Fig. 10. In most cases in the literature, the amount sorbed is still significantly smaller than the equilibrium amount and the Lagergren equation does not fit well for the whole range of contact time process. The calculated values and their corresponding linear regression correlation coefficient values are listed in Table 2. $R^2$ was found to be 0.8249 and 0.9547 which shows that this model cannot be applied to predict the adsorption kinetic model.

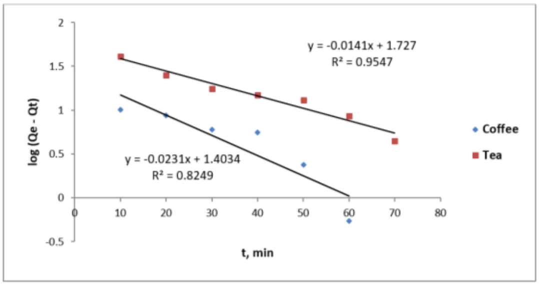

**Fig. 10.** Pseudo first order kinetic plots for the removal of Cu(II) by adsorption on (WTCP).

The pseudo second-order rate expression, which has been applied for analyzing sorption kinetics rate, is expressed as [24]:

$$\frac{d\,Q_t}{dt} = k_2\,(Q_e - Q_t) \qquad (12)$$

For the boundary conditions from $Q_t$ = 0 to $Q_t$ and t = 0 to t; the integrated form of equation becomes:

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{1}{Q_e}\,t \qquad (13)$$

Where t is the contact time (min), $Q_e$ and $Q_t$ are the amount of the solute adsorbed at equilibrium and at any time t (mg/g) respectively and $k_2$ is the rate constant of pseudo-second order sorption, (g/mg.min).

If pseudo-second order kinetics is applicable, the plot of $t/Q_t$ versus t of the equation should give a linear relationship, from which $Q_e$ and $k_2$ can be determined from the slope and intercept of the plot, Fig. 11. The pseudo-second order rate constant $k_2$, the calculated $Q_e$ value and the corresponding linear regression correlation coefficient value $R^2$ are given in Table 2. At all initial metal concentrations, the linear regression correlation coefficient $R^2$values were high (0.996 and 0.999). The higher values confirm that the adsorption data are better represented by pseudo-second order kinetics and the calculated $Q_e$ values agreed with the estimated experimental $Q_e$ values 115 – 103 (Table 1 and 2). This suggests that the adsorption of Cu ions follows pseudo-second order kinetics

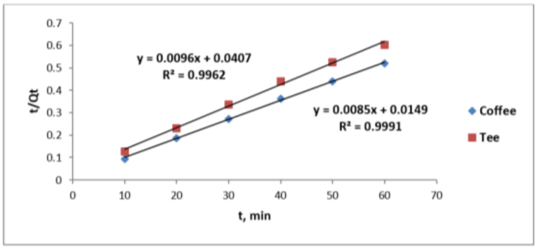

**Fig. 11.** Second order kinetic plots for the removal of Cu(II) by adsorption on (WTCP).

**Table 2.** Kinetic parameters of metal ions biosorption onto WTCP.

| First Order | $Q_e$ | $k_1$(1/min) | $R^2$ |
|---|---|---|---|
| Coffee | 25.32 | 0.0231 | 0.8249 |
| Tea | 53.33 | 0.0141 | 0.9547 |
| Second Order | $Q_e$ | $k_2$ (g/mg min) | $R^2$ |
| Coffee | Graphically (117.65)<br>Estimated (115) | $4.85x10^{-3}$ | 0.9991 |
| Tea | Graphically (104.17)<br>Estimated (103) | $2.26x10^{-3}$ | 0.9962 |

## 4. CONCLUSION

In our present work, we show that the waste coffee and tea powders could be used as a biosorbent for the removal of Cu (II) ions from aqueous solutions. The removal efficiency and metal capacity were found to be dependent on pH, contact time, initial metal ion concentration, and biosorbent dose. Biosorption isothermal data could be well simulated by Langmuir, Freundlich, Temkin and then Dubinin-Radushkevich (D-R) models. The maximum metal capacities determined by using the Langmuir isotherm were 526 and 417 mg/g for coffee and tea powders, respectively. The values of metal uptake capacities at equilibrium $Q_e$ calculated from equation (2) and pseudo-second-order plot were found comparable .The biosorption kinetics could be well predicted by pseudo-second-order kinetic. The results of our investigation indicate that the coffee and tea powder has a potential for use in removing Cu (II) from aqueous solutions.

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