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A Study on Using Molasses Production Waste Powder as a Green Adsorbent for Cu (II) Ions from its Aqueous Solution

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Abstract

This study utilized low-cost agricultural waste (molasses production waste powder) to extract copper ions from aqueous solutions. The present investigation explored a range of factors that influence the adsorption process, including temperature, pH, ionic strength, contact time, quantity of adsorbent, and particle size. Spectrophotometric analysis was used to determine the solution's absorbance both before and after the adsorption procedure. The Langmuir and Freundlich adsorption models were used to match the equilibrium data. The Freundlich model was determined to be the best isotherm model using the linear regression coefficient $R^2=0.9868$. Thermodynamic parameters, including enthalpy, entropy, and Gibbs free energy, were calculated for the continuous adsorption process, exothermic behavior of the system was observed by the heavy ion's adsorption process results on the green adsorbent. The process is good especially at low concentration in aqueous solutions.

Keywords: Adsorption, Copper(II), Langmuir model, Powder of molasses production waste.

دراسة استخدام مسحوق مخلفات إنتاج الدبس كمتنر لأيونات النحاس الثنائي من محاليلها المائية

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الخلاصة

أُستخدِمت هذه الدراسة المخلفات الزراعية منخفضة التكلفة (مسحوق مخلفات إنتاج الدبس) لازالة أيونات النحاس من المحاليل المائية. استكشف البحث الحالي مجموعة من العوامل التي تؤثر على عملية الامتزاز، بما في ذلك درجة الحرارة، ودرجة الحموضة، والقوة الأيونية، وزمن الاتصال، وكمية المادة المازة، وحجم . تم

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استخدام التحليل الطيفي لتحديد امتصاصية المحلول قبل وبعد عملية الامتزاز. تم استخدام نموذجي الامتزاز لانكماير و فروندليتش لمطابقة بيانات التوازن، وجد ان نموذج الأيسوثرم الأكثر ملاءمة هو نموذج فروندليتش باستخدام معامل الانحدار الخطي 0.9868، تم حساب الدوال الديناميكية الحرارية، بما في ذلك المحتوى الحراري والإنتروبي والطاقة الحرة لجيبس، لعملية الامتزاز، حيث أظهر نظام امتزاز الأيونات النحاس الثنائي على سطح المادة المازة السلوك الطارد للحرارة كما وجد ان الطريقة المستخدمة جيدة لإزالة التراكيز القليلة لايونات النحاس الثنائي من المحاليل المائية.

1. Introduction

The active ingredients used in healthcare products have become a growing concern as new environmental pollutants, due to their widespread use and presence in various ecosystems [1]. Industry has led to the development of many countries; however, it has also produced a wide range of pollutions. The hydraulic water sector suffers the most when industrial waste is discharged into lakes and rivers, tainting them with dyes, poisonous metals, and other materials [2, 3]. Wastewater contamination has an adverse effect on community water sources and may have detrimental health effects [4]. Currently, environmental activists have deleterious concerns due to the identification of unexpected pollutants found in many lakes and rivers worldwide, including chemical compounds from personal care and pharmaceutical products, polycyclic aromatic hydrocarbons and phenolic compounds [5,6]. A plethora of new types of contaminants found within water supply systems have recently been recognized being constituting creative environmental threats that need to be managed properly [7]. Although all heavy metals are extremely poisonous and the marginal inclination to ingest them averages less than 0.1 mg/L, it is best to clean up these effluents using sorbent technologies that allow for the extraction of trace levels of contamination [8,9].

Heavy metals pose significant risks above a particular concentration. They have the proclivity to accumulate throughout the food chain and amass in certain human organs. Among these heavy metals, which are indispensable to all living things, including humans, is copper. High copper levels in water can cause serious health issues such as verdigris vomiting, diarrhea, stomach pain, and damage to the liver and kidneys. Because copper is noxious, reducing its concentration in natural waterways is imperative to achieving drinking standards and avoiding health hazards. Furthermore, the significant decline in the concentration of copper in natural waterways can be attributed to the rigorous practices of heavy metal extraction following discharge into groundwater, as well as the proliferating agricultural industry.

Spectrophotometric methods can be more advantageous for identifying environmental samples due to their simplicity and cost in comparison to electrochemical methods [10, 12]. Heavy metal removal can be accomplished through a variety of techniques, including chemical precipitation [13], ion exchange [14], membrane separation [15], advanced oxidation processes [16, 17] and adsorption [18]. Nevertheless, adsorption has been found to be a more successful approach than the others since it is inexpensive, easy to use, and has a straightforward design [19,20]. Several studies on inexpensive adsorbents, such as wheat straw, waste from tea factories, olive leaves, date palm fibers, barley husks, pongamia pinnata leaf, sunflower stem, and activated charcoal, have been carried out as of late [21-23]. The adsorption process's outcome is determined by a wide range of operational variables. Contact time is an integral component, therefore the process needs sufficient time to reach an equilibrium point. Longer periods of contact between the adsorbent and the adsorbate are thought to have the potential to heighten adsorption capacity in some ways, depending on the active locale of the material [24].

In the present study, we looked at the molasses production waste powder's ability to remove the heavy metal ion Cu (II) from wastewater.

2. Materials and Methods

2.1 Apparatus

Copper ion concentration was measured using a Shimadzu ICPS-7510 Sequential Plasma Emission Spectrometer. The vibrational peaks of molasses production waste were identified using FTIR spectroscopy (Thermo Nicolet, AVATAR 330 Spectrometer). Korea is the manufacturer of the LabTech LSB-045S water bath shaker equipped with a thermostat. Germany supplied the centrifuge (6000 rpm). The weight of the adsorbent was measured using a Shimadzu electronic balance. The pH of the adsorbate was measured using a WTW pH 320 digital pH meter.

2.2 Materials

The copper nitrate trihydrate ($\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$) was supplied by Sigma Aldrich, USA. The molasses waste was provided from Diyala farms.

2.3 Standard solutions preparation

The standard stock solution of copper ion (100 mg/L) was made by dissolving 0.3801 g of $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ in 1 liter of distilled water and kept at 25°C. The stock solution was then serially diluted to form the experimental solution at different concentrations.

2.4 Preparation of Surface Powder

The powdered molasses production waste surface was cleaned using an amount of distilled water; this procedure was carried out multiple times to remove dust and other soluble materials. After an oven dry cycle lasting 90 min at 80°C, the surface was cleaned and then placed in airtight containers. After that, the surface was crushed and three filters were used for the powder nominal filters (75, 150, and 250) μm . For the entirety of this study, 75 μm particle sizes consistently used in all of our experiments. Nevertheless, only (150 and 250) μm particle sizes were used to investigate the effect of particle size.

2.5. Adsorption experiments

The purpose of every study was to assess the effects of temperature, length of contact, ionic strength, pH, and adsorbent weight. To obtain a copper ion calibration curve, the copper (II) concentration from (10 to 100) mg/L was plotted against the absorbance values as in Figure -1. The amounts adsorbed at equilibrium time (Q_e) and the percentage of Cu(II) removal (R%) were determined using Eq. (1 and 2).

$$R\% = \frac{C_o - C_e}{C_o} \times 100 \quad (1)$$

$$Q_e = \frac{V(C_o - C_e)}{m} \quad (2)$$

Where: C_o : The primary concentration (mg/L), C_e : Equilibrium concentration (mg/L), V: The solution volume (0.01L) and m: mass of adsorbent (0.3g).

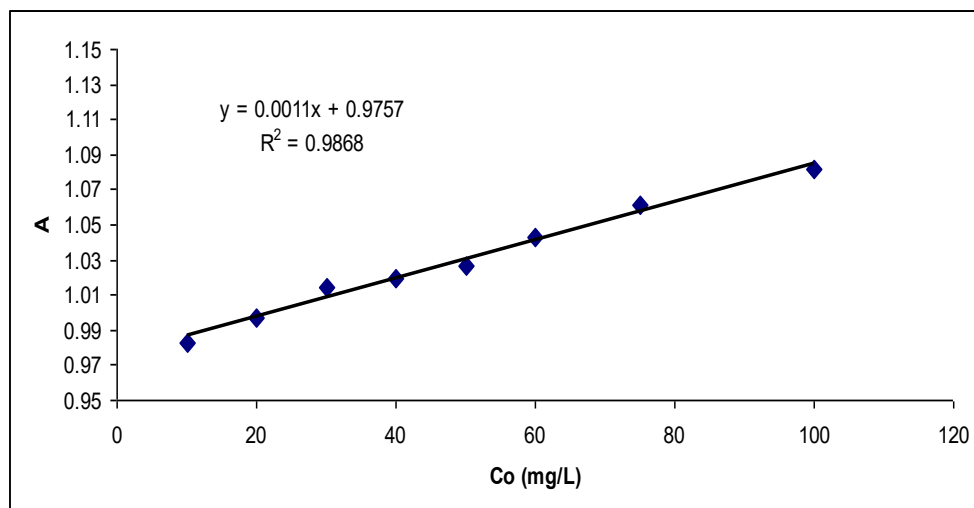


Figure 1: Calibration curve of copper ions (10-100) mg/L

3. Results and discussion

3.1 Identification of compounds

The chemical functional groups of the adsorbent and the sorbent were determined. Using the potassium bromide disc (KBr) method. To gain a comprehensive understanding of the adsorbent spectral characteristics, FTIR data was collected across a broad range of wavenumbers, spanning from 400 to 4000 cm^{-1} . A first qualitative analysis was conducted to identify the principal functional groups on the surface of molasses manufacturing waste using FTIR spectroscopy. The results of this investigation are presented in Table 1 and Figure 2. The vibrational peak of -OH is located at wave numbers 3500-3100 cm^{-1} . The stretching vibration of alkyl groups (-CH₂ and -CH₃) caused by the C-H bonds of aliphatic compounds is represented by the peaks at the range of 2950-2800 cm^{-1} . Peak values between 1760 and 1600 cm^{-1} correspond to the carboxyl groups (protein) and amide's C=O bonds. The vibration stretches involves the C-O was observed at 1550-800 cm^{-1} [25-27].

Table 1: The FTIR of assignment groups of molasses production waste.

Wave number (cm^{-1})	Assignment groups
3386	O-H Alcohols, carboxylic acid
2923	Stretching C-H (-CH ₃)
2854	Stretching C-H (-CH ₂)
1728	C=O carboxylic acid
1635	C=O amide stretching
1438	C-O (COO-), O-H bending
1377	C-H bending alkanes
1249	C-O stretching (COOH)
1033	Stretching alcohols C-O, carboxylic acids
894	Bending O-H carboxylic acids
620	O-H bending out of plan

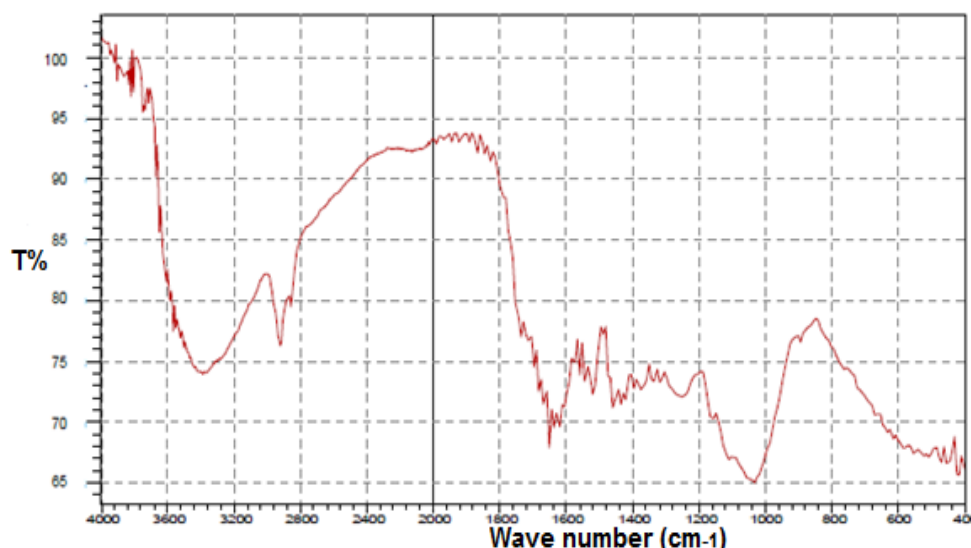


Figure 2: The FTIR of assignment groups of molasses production waste.

3.2 The effect of contact time

Contact time effects on copper ion adsorption on adsorbent surface were examined at 40 mg/L copper ions initial concentration and 0.3 g of adsorbent at 25 °C. As Figure 3 demonstrates, when the duration lengthened the adsorption increased, the Cu(II) adsorption attained saturation after 90 min. It was deduced that 70 min was sufficient for adsorption to attain equilibrium.

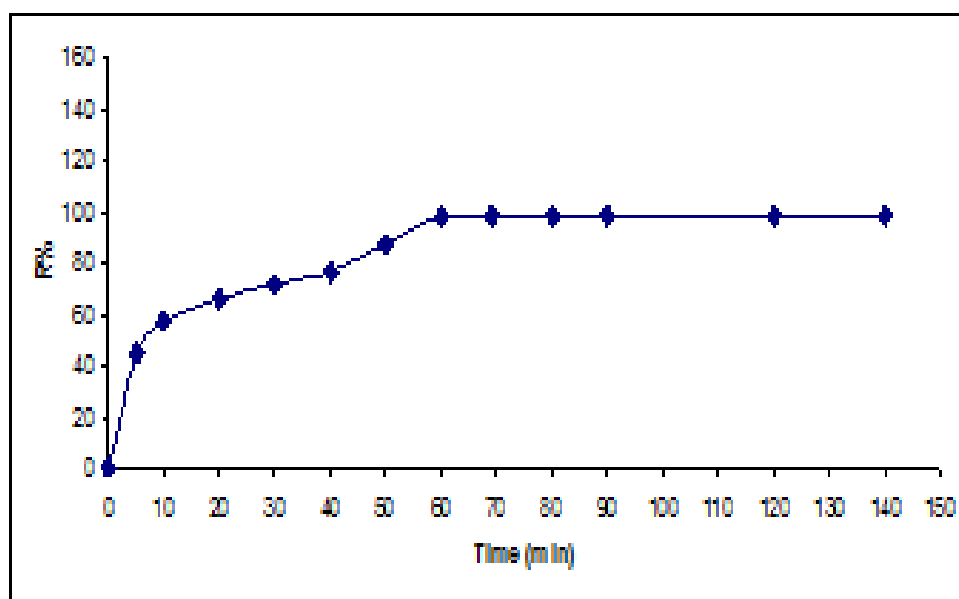


Figure 3: Effect of contact time

3.3 The effect of Adsorbent weight

The influence of molasses production waste weight on the removal percentage of adsorbed copper ions was investigated using 10ml of an initial 40 mg/L concentration of copper ions at 25 °C. The correlation between the adsorbent weight and the R% of copper ions is depicted in Figure-4. In contrast, a decline in effectiveness at higher adsorbent concentrations is attributed by a decrease in surface active sites as a result of partial adsorbent agglomeration. An increase in adsorbent dose causes an increase in adsorption because there

are more active sites available on the surface [28]. A weight of 0.3 g of adsorbent was selected for the remaining experiments to obtain the desired result.

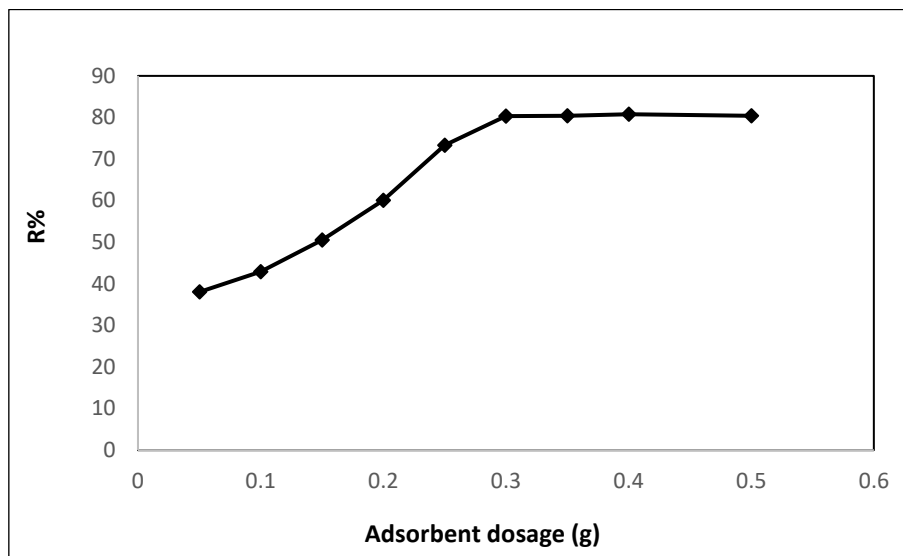


Figure 4: Adsorbent dosage effect.

3.4 pH effect

A variety of pH values, spanning from 1.5 to 10.0, were utilized in the adsorption test. To alter the pH of the solutions, the proper amounts of 0.1 M NaOH or 0.1 M HCl were added. Every other parameter in Figure 5 stayed the same throughout the experiment. Precipitation tended to occur at pH values over 6, while the pH range of 2- 4 demonstrated the maximum elimination capability for Cu(II) ions. Following the completion of each batch process, the solution's final pH of 2.5 somewhat dropped. The release and exchange of H^+ ions from functional groups like the hydroxyl and carboxyl groups found in molasses production waste explains this [29]. Numerous accounts in the literature state that Cu(II) ions are mostly present up to pH 5, after which a variety of hydroxides start to precipitate depending on the acidity. Therefore, additional batch tests were conducted at pH 2.5 using a 0.1 M HCl solution.

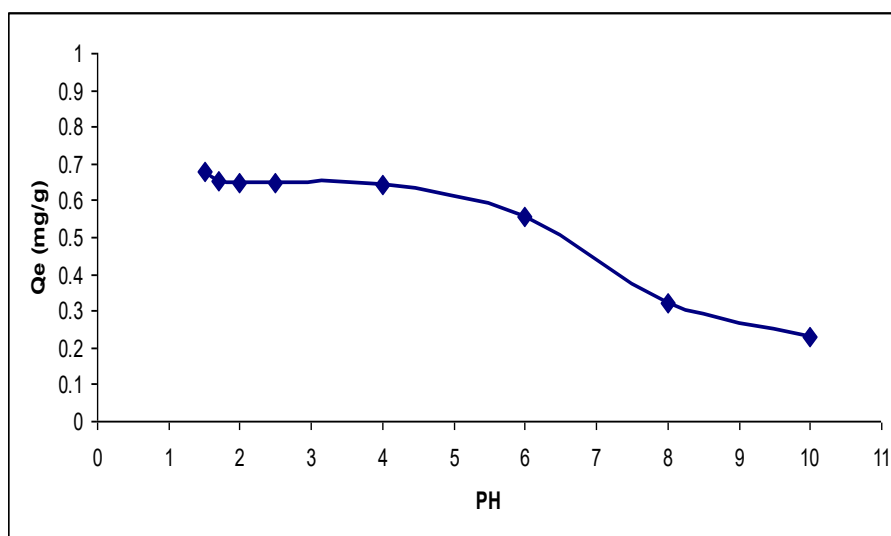


Figure 5: Effect of pH.

3.5. Ionic Strength's Effect

The effect of ionic strength on adsorption uptake of Cu(II) on molasses production waste was studied at variable concentration of sodium chloride (0.1, 0.2, 0.3, 0.4, and 0.5) F ionic strength ($I = 0.1, 0.2, 0.3, 0.4$ and 0.5 respectively) by using fixed concentrations of adsorbate (40 mg/L), 0.3 g of adsorbent at 25 °C. Figure 6, shows that when the concentration of NaCl increases, adsorption decreases. The adsorption of copper ions onto the surface of the adsorbent becomes less efficient as the NaCl solution concentration rises, due to the increasing ionic strength of the solution. Since their attraction to the surface is greater than that of copper ions, For the surface's active sites, the copper ions will face competition from the Na^+ and Cl^- ions [29].

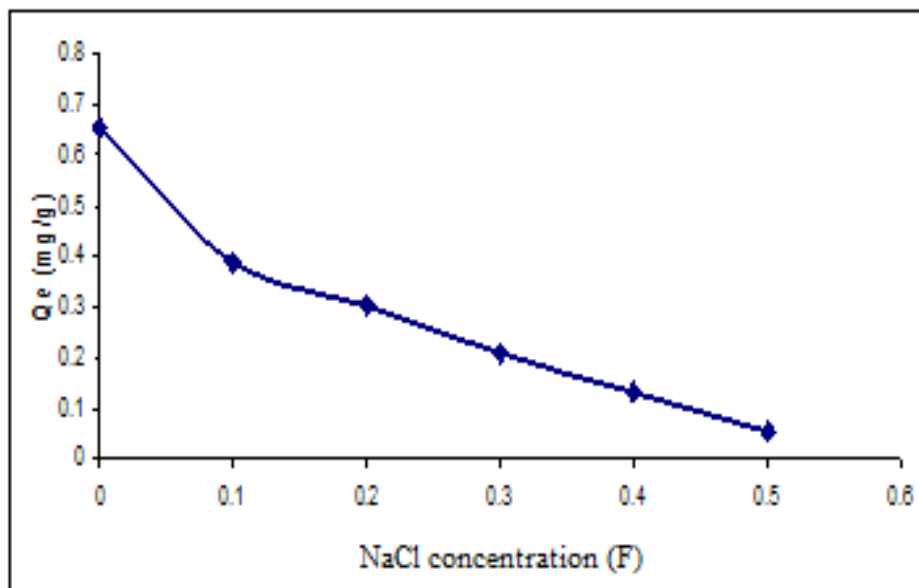


Figure 6: The impact of ionic strength.

3.6 Particle size effect

The particle size impact on adsorption was examined using various sieve sizes of (75, 150, and 250) μm . A constant adsorbate concentration of 40 mg/L and various particle sizes with an identical adsorbent weight of 0.3 g were used in these tests, Figure 7. The results showed that, in that order, the largest amounts of copper ions that were adsorbed on the adsorbent were 75 μm , 150 μm , and 250 μm . This phenomenon can be attributed to the increase in specific surface area, which enhances the extent of the adsorption process. As particle size decreases, copper adsorption effectiveness improves because smaller particles provide a greater specific surface area for adsorption. Larger particles may not be able to utilize the whole of their interior surface for adsorption and have a higher diffusion barrier to mass movement [28].

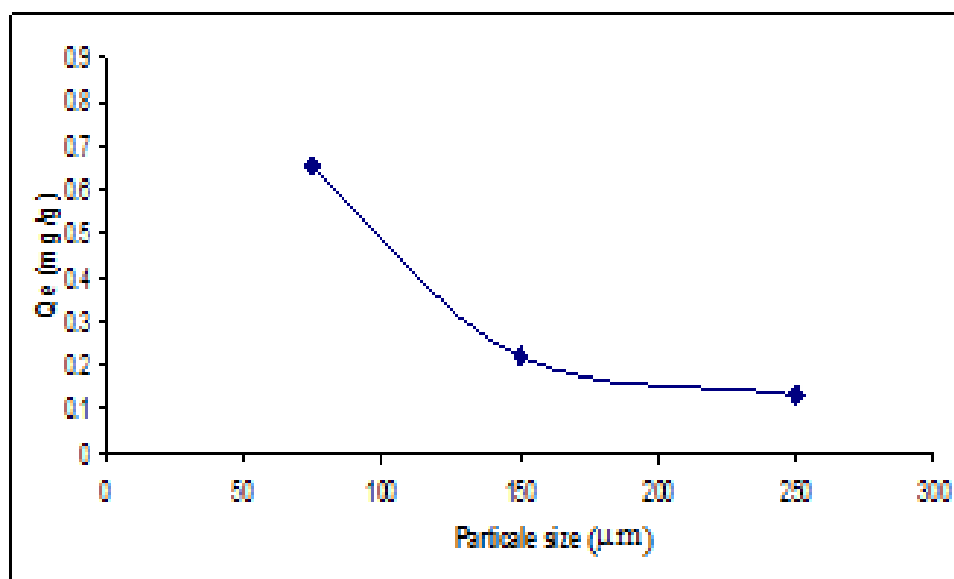


Figure 7: Particle size effect.

3.7 Effect of temperature

It is now evident that changes in temperature can significantly affect the analyte molecule's desorption [30]. Four distinct temperatures (283.15, 298.15, 310.65 and 323.15) K were used to investigate the influences of temperature on copper ion adsorption on adsorbent surfaces. The amount of Cu (II) ions extracted by adsorbents reduced as temperature increased, showing that the process of sorption was exothermic, as reported in Table 2 and Figure 8.

Table 2: Effect of temperature.

C _o (mg/L)	283.15K		298.15K		310.65K		323.15K	
	C _e (mg/L)	Q _e (mg/g)	C _e (mg/L)	Q _e (mg/g)	C _e (mg/L)	Q _e (mg/g)	C _e (mg/L)	Q _e (mg/g)
5	3.6282	0.0457	2.7756	0.0741	3.2435	0.0585	3.9252	0.0358
10	5.1452	0.1618	5.0384	0.1653	5.7473	0.1417	6.0427	0.1319
20	10.0598	0.3313	9.4145	0.3528	10.5940	0.3135	11.3632	0.2879
30	14.8247	0.5058	15.5320	0.4822	17.3675	0.4210	17.7735	0.4075
40	16.0470	0.7984	18.3311	0.7222	20.4658	0.6511	21.8119	0.6062
50	25.0000	0.8333	26.3568	0.7881	29.3526	0.6882	32.6666	0.5777
60	32.6303	0.9123	34.3269	0.8557	38.1794	0.7273	41.7682	0.6077

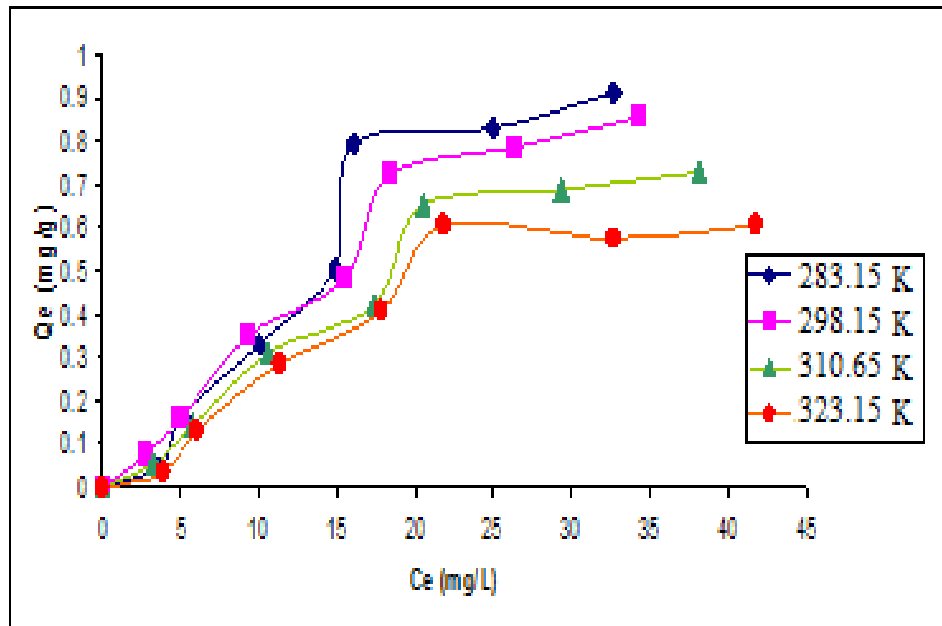


Figure 8: Effect of temperature.

3.8 Thermodynamic of adsorption process

Understanding the adsorption requires research on thermodynamics. Eqs. (3, 4 and 5) were used to compute the changes in enthalpy (ΔH), entropy (ΔS), and Gibbs free energy (ΔG).

$$\Delta G = -RT \ln K_{abs} \quad (3)$$

$$\ln K_{abs} = \left(\frac{-\Delta H}{RT} \right) + \left(\frac{\Delta S}{R} \right) \quad (4)$$

$$\Delta G = \Delta H - T\Delta S \quad (5)$$

The equation that follows was used to determine the equilibrium constant K_{ads} and R is the gas constant (8.314 J/mol.K).

$$K_{ads} = \frac{Q_e}{C_e} \quad (6)$$

As C_e is the adsorbate's concentration at equilibrium, expressed in mg/L, and Q_e is the amount adsorbed, expressed in mg/g. The slope and intercept of the linear plot of $\ln K_{ads}$ vs. $1000/T$ were used to calculate ΔH and ΔS respectively. Table -3 and Figure -9 display the obtained results.

Table 3: Impact of temperature on the thermodynamic equilibrium constant.

Temperature (K)	1000/T (1/K)	Qe (mg/g)	k	$\ln k_{abs}$
283.15	3.5336	0.84	1.14545	0.13579
298.15	3.3557	0.76	1.03636	0.03571
310.65	3.2206	0.67	0.91363	-0.09032
323.15	3.0960	0.60	0.81818	-0.20067

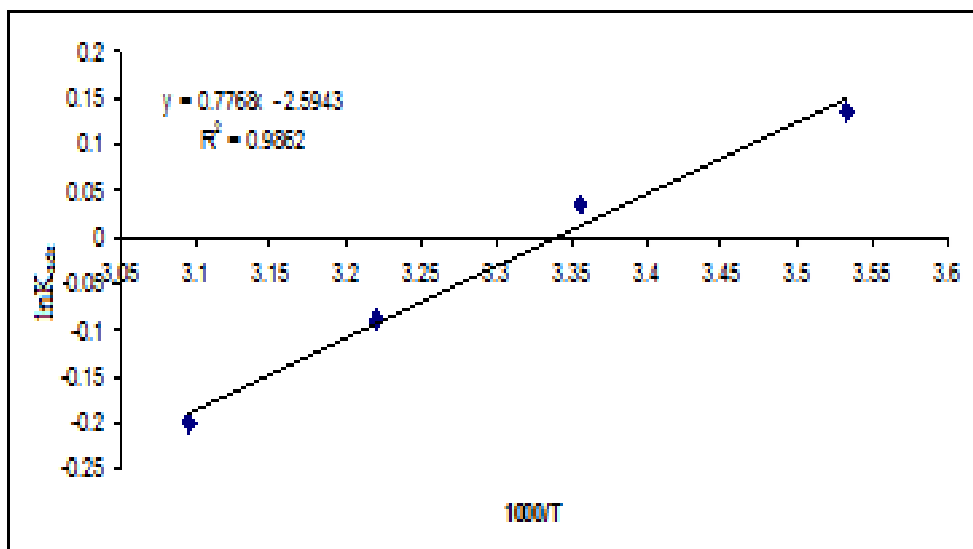


Figure 9: Relation between $\ln K_{ads}$ and $1000/T$ for adsorption process.

The adsorption process is exothermic because of ΔH has a negative value. Positive value for ΔG lends credence to the assertion that adsorption is a process that occurs naturally and that also bolsters the operation's viability. Since a more complex order structure is required for adsorption, a negative value of ΔS indicates a decrease of disorder process [31, 32] Table-4 displayed the values for ΔG , ΔH , and ΔS .

Table 4: Thermodynamic parameters at various temperatures of adsorption.

Temperature (K)	ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (J/mol.k)
283.15	-0.3192	-0.0065	-21.5690
298.15	-0.0884		
310.65	+0.2331		
323.15	+0.5388		

3.9 Adsorption model

Techniques such as adsorption isotherm studies can be used to compare the quantity of metal ions that are adsorbed throughout the operation. The Freundlich and Langmuir isotherm models are the most often used models by scientists to calculate the experimental equilibrium parameters of the adsorption process [31]. The empirical Langmuir isotherm Eq. (7) and Freundlich isotherm Eq. (8) and were matched with the experimental adsorption data.

$$\frac{C_e}{Q_e} = \frac{1}{a \cdot b} + \frac{C_e}{a} \quad (7)$$

$$\log Q_e = \log K_f + \frac{1}{n} \log C_e \quad (8)$$

The relationship between (C_e/Q_e) and (C_e) , and relationship between $(\log Q_e)$ and $(\log C_e)$ was linear and showed the appropriateness of Langmuir and Freundlich isotherms based on linearity (R^2) values, Figures- 10 and 11.

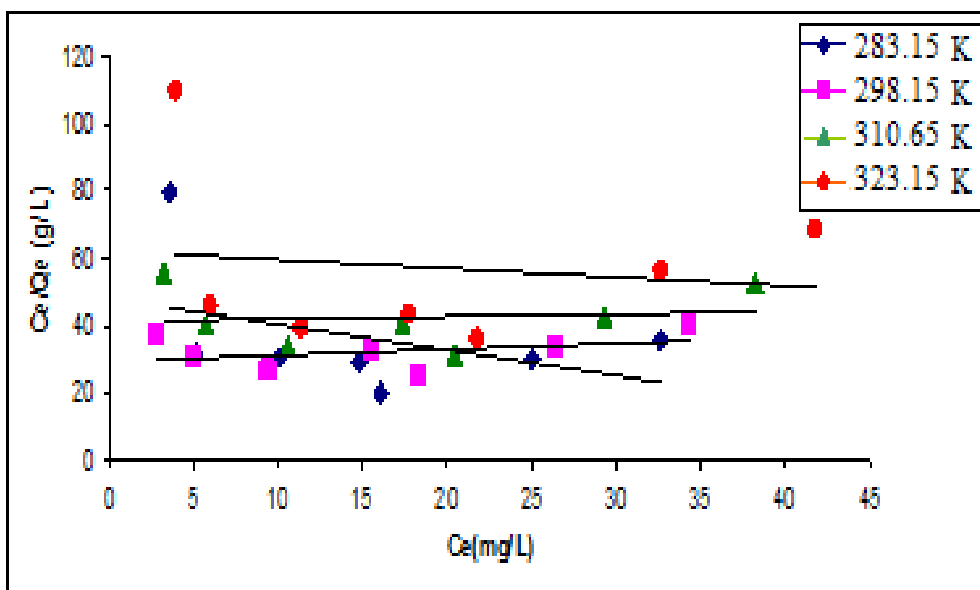


Figure 10: Langmuir linear adsorption isotherm of copper ions

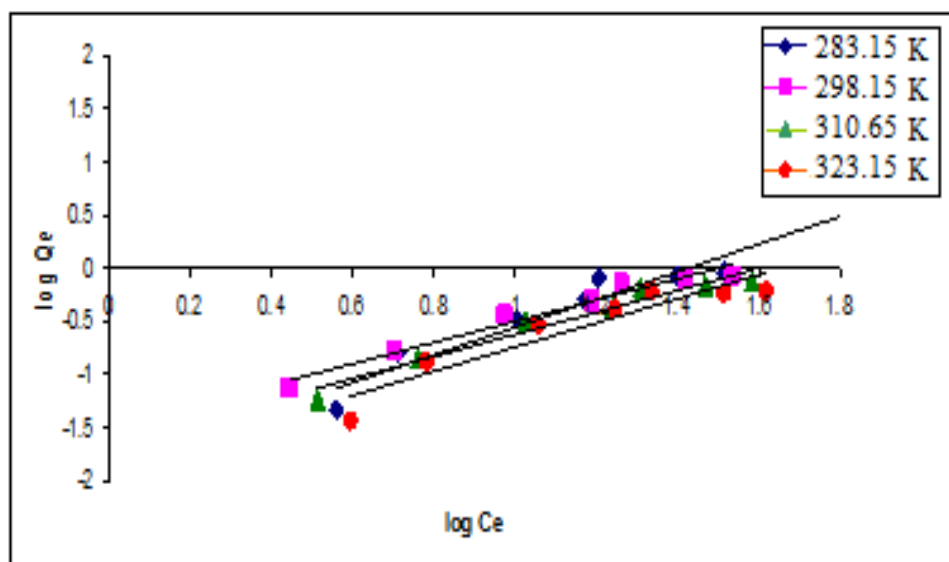


Figure 11: Freundlich linear adsorption isotherm of copper ions

The empirical values of the Freundlich and Langmuir constants at several temperatures were obtained using the linear regression analysis equation. The overview of the values may be provided by Tables 5 and 6. The results indicate that the best fit corresponds to the Freundlich model ($R^2 \geq 0.8782$), and the n value as showed by Freundlich model was (≥ 0.7) suggests that the Cu(II) favorable to by molasses production waste powder. In other words, the adsorption of Cu(II) on the adsorbent surface follows the Freundlich more than Langmuir model ($R^2 \leq 0.17$)

Table 5: The correlation coefficients and empirical values of the Langmuir constants for the adsorption of copper (II) at several temperatures on the adsorbent.

Temperature (K)	a (mg/g)	b (L/g)	R ²
283.15	-1.3099	-0.0157	0.1705
298.15	6.1652	0.0054	0.1207
310.65	13.0208	0.0018	0.0121
323.15	-3.9323	-0.0040	0.0190

Table 6: The Freundlich constants for the copper ions adsorption

Temperature (K)	n	K _f	R ²
283.15	0.7679	71.2534	0.9030
298.15	1.0064	31.3617	0.9669
310.65	0.9604	46.4408	0.9868
323.15	0.8791	76.9130	0.8782

Conclusion

Based on the previous studies, we have concluded that molasses production waste powder which is the disposed solid wastes from date palm are an efficacious adsorbent for removing copper ions from polluted water. The experimental findings obtained from the research showed that the ability to remove copper ions increases with increasing contact time. The copper absorption capacity was highest at pH 2.5, adsorption depends on the amounts, the particle sizes of the molasses production waste powder, and the ionic strength of the aqueous sodium chloride solution. It was found that the Langmuir and Freundlich isothermal models are applicable. For copper ion absorption, a negative value of ΔG indicates the spontaneous absorption process. A negative value of ΔS indicates an increase in the order of the process. The calculated thermodynamic parameters are very useful when using existing results in industrial processes.

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