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# INVERSE VULCANISED POLYSULPHIDE ADSORBENT FROM WASTE COOKING PALM OIL FOR $Cu^{2+}$ REMOVAL

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## ABSTRACT

*Heavy metals are considered to be toxic to humans. Commonly produced by petrochemical plants and pesticide productions, various techniques have been utilised to treat wastewater contaminated with heavy metal such as copper ($Cu^{2+}$). This article focuses on using adsorption by utilising polysulphide adsorbent to treat wastewater via inverse vulcanisation. The huge amount of waste cooking palm oil (WCO) that is generated daily in Malaysia has motivated the study to produce a low-cost adsorbent with high removal efficiency. The polysulphide adsorbent was prepared with sulphur powder and waste cooking oil with a ratio of 9:1. The effect of contact time, initial $Cu^{2+}$ concentration, and adsorbent dosage were studied using a batch adsorption process. The highest removal efficiency was 77.81% obtained while using 5 g of adsorbent dosage to treat 5 mg/L of $Cu^{2+}$ aqueous solution. The Freundlich and Langmuir isotherm was used for modelling $Cu^{2+}$ adsorption in an aqueous medium which was modelled best by the Langmuir isotherm. Overall, these results confirmed polysulphide as a low-cost and potential adsorbent to remove $Cu^{2+}$ from wastewater.*

**Keywords:** heavy metal removal, inverse vulcanisation, polysulphide adsorbent, waste cooking palm oil, waste to wealth.

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## INTRODUCTION

The adverse effects of heavy metal wastewater on human health and the aquatic system have concerned the scientific community. Heavy metal wastewater is toxic and carcinogenic in nature and sometimes is released without prior wastewater treatment. Globally, almost 78% of heavy metal wastewater remains untreated, which bioaccumulates along the food chain because of its non-degradable nature (Jones *et al.*, 2021; Kumar *et al.*, 2019; Worthington *et al.*, 2017). Copper ($Cu^{2+}$), an important micronutrient element, has a crucial role in bone formation with the help of enzymes and proteins, but extreme intake of this heavy metal wastewater can lead to brutal health damage including renal and hepatic damage, widespread capillary damage, mucosal irritation and corrosion, severe gastrointestinal and central nervous system irritation, and necrotic changes in the kidney and liver (Arbabi and Golshani, 2016). Highly concentrated $Cu^{2+}$ wastewater damages the osmoregulatory mechanism of the freshwater resources which also endangers the living organisms of the freshwater ecosystem. Different industries such as paint and pigments, metal plating and cleaning, petroleum refineries, smelting, mining, wood pulp, paper board, and fertiliser production are responsible for $Cu^{2+}$ wastewater (Braga *et al.*, 2021). Ion exchange, coagulation and precipitation, membrane filtration, distillation, reverse osmosis, and adsorption have been explored so far to remove $Cu^{2+}$ from aqueous solution (Ramos *et al.*, 2016; Rashed *et al.*, 2023). To deal with $Cu^{2+}$ wastewater, the adsorption process has been proven as an effective method (Ciobanu *et al.*, 2023). However, the high price and

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the nature of metal selectivity of the commercial adsorbents have caused some difficulties in the application of commercial adsorbents for $Cu^{2+}$ wastewater adsorption (Emenike *et al.*, 2022).

Recently, inverse vulcanised polysulphides have been used as adsorbents to remove heavy metals in wastewater (Nayeem *et al.*, 2023). Inverse vulcanisation utilises the surplus amount of elemental sulphur obtained from sour gas and refinery treatment plants all over the world exceeding almost 70 million t $yr^{-1}$ (Nayeem *et al.*, 2022a). Inverse vulcanisation enabled the use of a wide range of crosslinking monomers, which include cooking oils, agro-wastes, inorganic refinery by-products, *etc.* (Chalker *et al.*, 2019). The vinylic double bonds in the monomers break down during inverse vulcanisation and react with sulphur to form sustainable polysulphides (Chalker *et al.*, 2019). A recent investigation done by Nayeem *et al.* (2022) showed the use of waste cooking palm oil (WCO) in inverse vulcanisation, which created a new way to deal with the threat of environmental hazards caused by WCO (Nayeem *et al.*, 2022b). The use of different crosslinkers, simple synthesis process, and high selectivity towards heavy metals have made the inverse vulcanised polysulphide adsorbents suitable for heavy metal wastewater treatment (Chalker *et al.*, 2021; Nayeem *et al.*, 2022a).

Polysulphide was prepared in the current study from sulphur and WCO to remove $Cu^{2+}$ ions. The ratio of sulphur to WCO was 9:1. The characterisation of polysulphides was done using FTIR, TGA, and SEM-EDX to understand the functional bonds, thermal and morphological characteristics. The $Cu^{2+}$ removal was studied by varying the parameters including dosages, initial $Cu^{2+}$ concentration, and contact time. Later, isothermal studies were done by Langmuir and Freundlich isothermal models.

## MATERIALS AND METHOD

### Materials

Sulphur powder (Sigma Aldrich, USA, $S_8$ reagent grade sublimed powder, ±99.5%), sodium chloride (NaCl), (Sigma Aldrich, USA, ACS reagent, ≥99.5 %), copper nitrate [$Cu(NO_3)_2$], (Merck, Germany, >230 mesh ASTM) were used as received for polysulphide preparation and $Cu^{2+}$ solution preparation. WCO was sourced from the café of Universiti Malaysia Pahang (UMP), Gambang campus and used prior to the removal of existing impurities due to the frying process by 0.224 µm syringe filter. Deionised water (Millipore Elix 5, USA) was produced at the chemical lab (FTKKP), UMP.

### Polysulphide Synthesis Via Inverse Vulcanisation

The synthesis process was followed by the method done by Nayeem *et al.* (2022b) and Abraham *et al.* (2018) with some minor modifications (Abraham *et al.*, 2018; Nayeem *et al.*, 2022b). About 9 g of sulphur powder and 3 g of NaCl were weighed according to 3:1 and ground using mortar and pestle to make a fine and homogenous mixture. The mixture was heated in a 50 mL beaker at 195°C and 450 rpm on a hot plate. After 8-10 min the mixture became orange in colour to ensure the complete ring opening of the S-S bond (Chung *et al.*, 2013). Then, WCO of 10 wt.% of sulphur was slowly poured into the mixture using a syringe and heated up for the next 60 min to reach the gel point for successful polymerisation (Gomez *et al.*, 2016). As WCO is not a completely unsaturated vegetable oil, over 9:1 ratio may result in a large amount of unreacted sulphur. This phenomenon was physically observed in a study of the liquid sulphur-oil solution which was not polymerised at all (Worthington *et al.*, 2017). The mixture was then cooled at room temperature for 90 min and ground into a fine powder using a pestle and mortar. The powder was rinsed with deionised water and was stirred at 600 rpm for 30 min to remove the NaCl that was added earlier which acted as porogen. Then the polymer was placed in a vacuum drier for 12 hr at 50°C.

### Polysulphide Characterisation

The functional groups of the polysulphides were studied using FTIR with ATR (Thermo Fisher Nicolet iS5, USA) over the range of 400-4000 $cm^{-1}$. The thermal stability of the polysulphide was observed through TGA (Hitachi STA7200, Japan) at a constant heating rate of 10°C $min^{-1}$ from 30°C up to 700°C. Gaseous $N_2$ was used to create an inert environment. The surface morphology and elemental analyses were studied using scanning electron microscopy (SEM) equipped with an energy dispersive X-ray (EDX) system (Hitachi TM3030 Plus, Japan). During analysis, a platinum (Pt) sputter coat was applied to keep the sample surface uncharged. The main attributes of the adsorbents including the specific surface area, pore size, pore volume and pore distribution were studied through $N_2$ adsorption-desorption isotherm by a Micromeritics ASAP 2020 system based on the BET method.

### Batch Adsorption of $Cu^{2+}$

A 100 mg $L^{-1}$ of $Cu^{2+}$ stock solution was prepared in a 50 mL beaker using deionised water (Darweesh *et al.*, 2022). Later, different $Cu^{2+}$ solutions (1 mg $L^{-1}$, 3 mg $L^{-1}$ and 5 mg $L^{-1}$) were prepared through serial dilution to study the effect of initial $Cu^{2+}$

concentration on the adsorption process. $Cu^{2+}$ removal was studied using batch adsorption technique at room temperature. About 100 mL of each concentration was stirred at 450 rpm for 2 hr in a 200 mL beaker. To record the $Cu^{2+}$ concentration of the solution, 2 mL of sample was collected at a regular interval (every 20 min), centrifuged (Thermo Scientific MicroCL 21, USA) and run through atomic absorption spectroscopy (AAS). The effects of the adsorption parameters on $Cu^{2+}$ removal were studied like adsorbent dosages, initial $Cu^{2+}$ concentration and time. Numeric values of these parameters used in this study were varied according to the previous studies (Cheraghipour and Pakshir, 2021; Jorge Gonçalves *et al.*, 2021; Stanković *et al.*, 2019). Three different dosages (1, 3 and 5 g 100 $mL^{-1}$) were used to study the effect of dosages on $Cu^{2+}$ adsorption.

The equilibrium concentration of the adsorbed $Cu^{2+}$ was calculated with the help of Equation (1).

$$q_e = \frac{(C_o - C_e) \times V}{M} \quad (1)$$

Here, $q_e$ denotes adsorption capacity (mg $g^{-1}$). V stands for the total volume of solution (L) and, M for the mass of adsorbent amount (g). The initial and equilibrium concentration (mg $L^{-1}$) of $Cu^{2+}$ in solution are denoted respectively by $C_0$ and $C_e$. The percentage of $Cu^{2+}$ removal is denoted by R which is calculated using the following equation.

$$R(\%) = \frac{C_o - C_e}{C_o} * 100\% \quad (2)$$

Adsorption isotherm was analysed using both Langmuir [Equation (3)] and Freundlich [Equation (4)] isothermal model (Ciobanu *et al.*, 2023; Nayeem *et al.*, 2023).

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{q_m K_L}\frac{1}{C_e} \quad (3)$$

Here, the maximum possible amount of $Cu^{2+}$ that can be adsorbed per unit mass of adsorbent (mg $g^{-1}$) and constant for adsorption equilibrium is expressed by $q_m$ and $K_L$, respectively.

$$logq_e = logK_L + \frac{1}{n} logc_e \quad (4)$$

Adsorption intensity for Freundlich isothermal model is denoted by 1/n.

## RESULTS AND DISCUSSION

### Characterisation of Polysulphide Adsorbent

Generally, FTIR indicates the functional bonds through the characteristic spectral peaks. *Figure 1* shows the FTIR spectra for the synthesised polysulphide compound. The figure depicts the basic characteristic peaks in the spectrum of polysulphide. From the previous study, the characteristic peak at 3005 $cm^{-1}$ resembled the vinylic bond of C=C, which was not observed in *Figure 1* and transformed to C-C to form the C-S

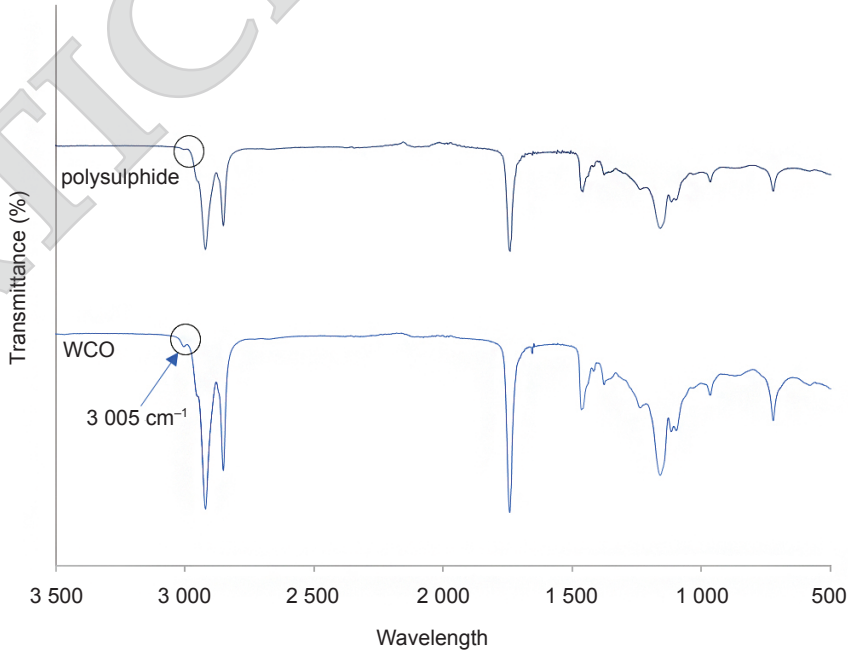


*Figure 1. FTIR spectra of polysulphides via inverse vulcanisation.*

polymeric chain during inverse vulcanisation (Nayeem *et al.*, 2022b). It demonstrates the successful polymerisation of WCO and sulphur. The observations obtained from FTIR follows the similar trends with the previous studies (Gomez *et al.*, 2016; Stanković *et al.*, 2019).

TGA curve showed the decomposition of polysulphide adsorbent in *Figure 2* where at first, the weight decreases slowly. Then, it becomes faster after 200°C. Maximum decomposition was a recorded at 289.6°C with 36.6% mass loss. Generally, WCO shows a single-step degradation. However, polysulphides produced from different vegetable oils through inverse vulcanisation tend to exhibit a two-step degradation (Nayeem *et al.*, 2022b; Tikoalu *et al.*, 2020).

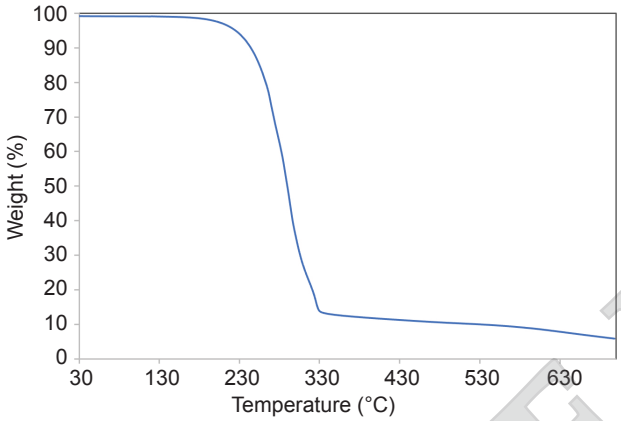


*Figure 2. TGA of inverse vulcanised polysulphide adsorbent.*

The SEM image of synthesised polysulphide adsorbent with 90 wt.% sulphur has been shown in *Figure 3*. The prepared polysulphide adsorbent has an irregular surface that is shaped like small bricks of different sizes and shapes. The polysulphides present a seemingly rough surface that indicates a high surface area for the adsorption. EDX image presented in *Figure 3*e shows that the percent amount of carbon, oxygen and sulphur composition in the adsorbent is 51.10%, 2.26% and 46.64% respectively.

**Effect of Different Parameters**

***Effect of dosages.*** The effect of adsorbent dosages on $Cu^{2+}$ removal from wastewater was investigated using three different loadings of polysulphide adsorbents, which were 1 g, 3 g, and 5 g respectively. The experiment was conducted at 35°C and a contact time of 120 min with a constant $Cu^{2+}$ initial concentration of 5 mg $L^{-1}$. The $Cu^{2+}$ removal rate in *Figure 4* indicated an increase in the percentage removal from 41.25% to 77.81% as the dosage was increased to 5 g from 1 g. At higher adsorbent dosages, there were higher unoccupied adsorption sites for removal of $Cu^{2+}$. As mentioned by Azman *et al.* (2020), the amount of adsorbed ions is increased as more surface area, active sites and functional groups can be obtained with higher dosages (Azman *et al.*, 2020). As depicted in *Figure 4*, $Cu^{2+}$ adsorption capacity decreased with higher adsorbent dosage. A similar trend was noticed in a previous study where adsorption sites

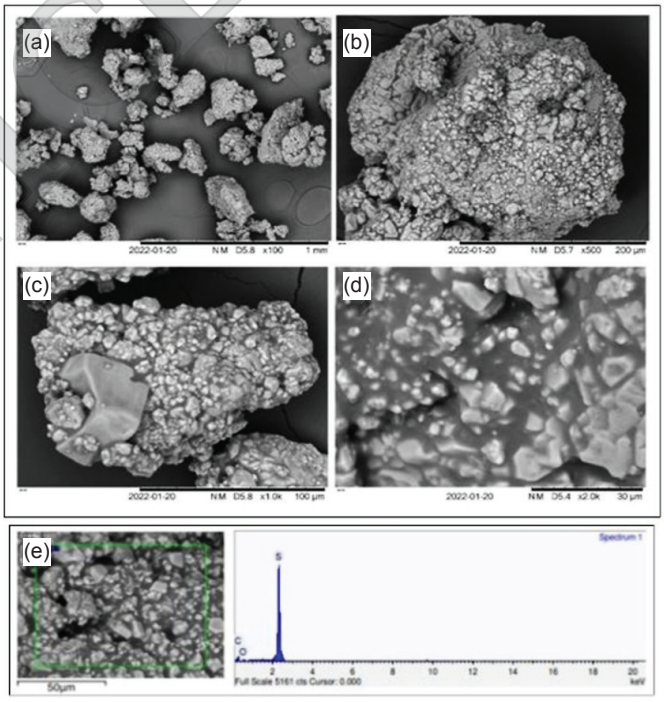


*Figure 3. SEM images of prepared polysulphide adsorbent from waste cooking oil and sulphur (a) 100X (b) 500X (c) 1000X (d) 2000X, and (e) EDX image.*

remained unsaturated during the removal because the higher amount of dosages produced more active adsorption sites and introduced steric hindrance for the polysulphide to adsorb the $Cu^{2+}$ ions (Ezeh *et al.*, 2017).

***Effect of initial $Cu^{2+}$ concentration.*** The effect of initial $Cu^{2+}$ concentration on the percentage removal is presented in *Figure 5*. The removal percentage dropped from 68.88% to 42.87% while the initial $Cu^{2+}$ concentration increased. On the contrary, the $Cu^{2+}$ adsorption capacity increased from 0.03 to 0.11 mg $g^{-1}$ when the initial concentration was raised from 1-5 mg $L^{-1}$. The maximum removal was 68.88% at 1 mg $L^{-1}$ of $Cu^{2+}$ solution. Because of steric hindrance, the active sites of the polysulphide adsorbent would be surrounded with more $Cu^{2+}$ in the solution when the initial $Cu^{2+}$ concentration was comparatively higher. Therefore, the equilibrium adsorption capacity of polysulphide adsorbent rises with the increasing $Cu^{2+}$ which enhances the adsorption process (Azman *et al.*, 2020). On the other hand, the removal rate tended to decrease at lower initial $Cu^{2+}$ concentration, as the $Cu^{2+}$ ions were removed by the sufficient available active sites of the polysulphide adsorbent (Radnia *et al.*, 2012).

***Effect of contact time.*** The removal efficiency of $Cu^{2+}$ was studied as a function of contact time at the initial concentration of 5 mg $L^{-1}$ at 35°C and adsorbent dosage of 2 g, which is presented in *Figure 6*. For predicting the equilibrium adsorption, it is crucial to consider the effect of contact time on the adsorption of metal ions on any adsorbent (Emenike *et al.*, 2022). As depicted in the figure, the adsorption of $Cu^{2+}$ into the polysulphide adsorbent increased with the increasing contact time. A similar trend was reported in a previous study (Gorzin and Bahri Rasht Abadi, 2017). At 0 to 80

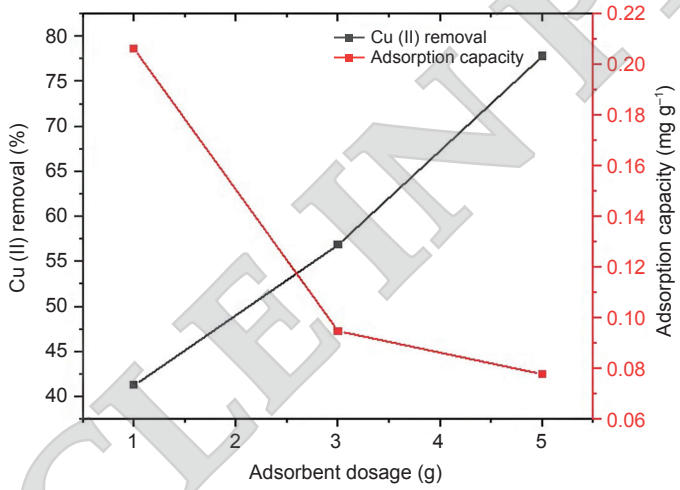


*Figure 4. Effect of adsorbent dosage on the percentage removal and $Cu^{2+}$ adsorption capacity (C0= 5 mg $L^{-1}$, T= 35°C).*

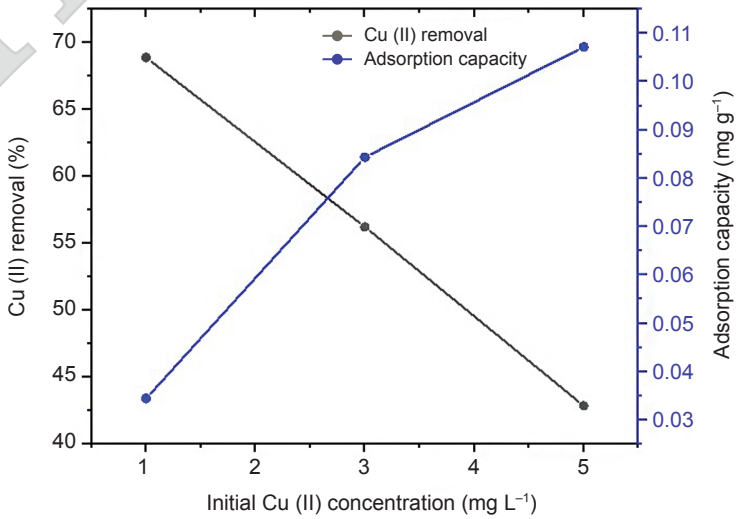


*Figure 5. Effect of initial $Cu^{2+}$ concentration on adsorption capacity and percentage removal (C0= 1 mg $L^{-1}$, T= 35°C and adsorbent dosage = 2 g).*

min, the adsorption of $Cu^{2+}$ removal was very fast. The contact time needed for $Cu^{2+}$ solution to achieve equilibrium was 80 min and saturated between 80-120 min with 77.81% of $Cu^{2+}$ uptake. Initially, the removal rate and $Cu^{2+}$ uptake capacity were rapid. Later, a gradual decline was observed at an equilibrium time beyond which no discernible rise in the removal rate and adsorption capacity was found. The initial rapid adsorption was caused by the abundance and availability of the active sites on polysulphide adsorbents. The active sites were rapidly occupied by $Cu^{2+}$ ions and then became saturated upon reaching equilibrium (Ezeh *et al.*, 2017; Gorzin and Bahri Rasht Abadi, 2017).

***Adsorption isotherm.*** Adsorption isotherm is important to understand the removal process. In this study, $Cu^{2+}$ removal was analysed by both Langmuir and Freundlich isotherm. But the process fitted better by Langmuir isotherm. The $R^2$ value of Langmuir value was higher than the Freundlich model. The $Cu^{2+}$-polysulphide interaction is the main reason behind this (Ko *et al.*, 2021). *Figure 7* represents the isothermal curves for both Langmuir and Freundlich isothermal models with a brief exhibition of their parameters in *Table 1*. The $R^2$ value is closer to 1 for Langmuir model, which means it is more favourable and efficient.

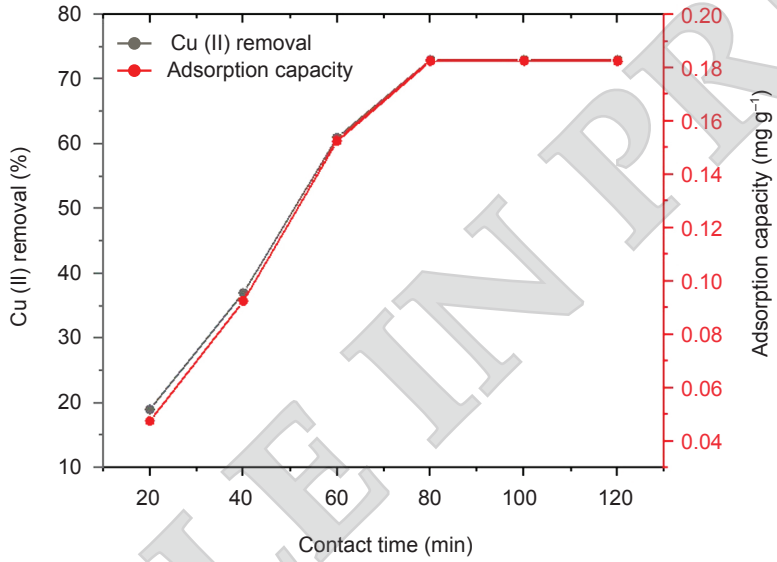


*Figure 6. Effect of contact time on adsorption capacity and percentage removal (C0= 5 mg $L^{-1}$, T= 35°C and adsorbent dosage = 2 g).*

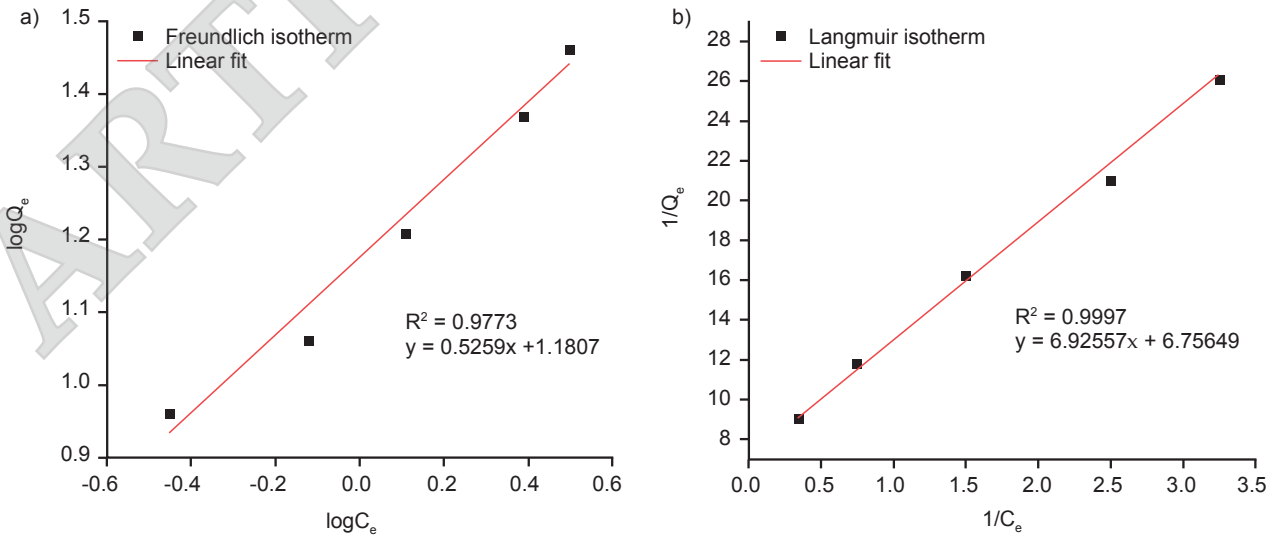


*Figure 7. Adsorption isotherm graph of $Cu^{2+}$ removal (a) Freundlich, and (b) Langmuir model.*

**TABLE 1. PARAMETERS OF ISOTHERMAL STUDY OF $Cu^{2+}$ REMOVAL**

| Langmuir | | | | Freundlich | | |
|---|---|---|---|---|---|---|
| $Q_{max}$ | $K_L$ | $R_L$ | $R^2$ | $K_f$ | 1/n | $R^2$ |
| 0.1480 | 0.9755 | 2.9755 | 0.99977 | 15.1600 | 1.90146 | 0.9773 |

The highest removal recorded in this study is 77.81%. It was achieved using a relatively higher polysulphide adsorbent dosage (5 g in 5 mg $L^{-1}$ $Cu^{2+}$ solution) which is higher than other heavy metals using the same adsorbent (Nayeem *et al.*, 2023). There are several factors behind this removal percentage for $Cu^{2+}$ in this study. The surface area, pore volume and pore diameter were 19.59 $m^3$ $g^{-1}$, 0.018 $cm^3$ $g^{-1}$ and 36.83 Å, respectively. Although the surface of the polysulphide was modified using NaCl, the attributes of the surface of the adsorbent were not high. The investigations showed that NaCl provides lesser surface area compared to other modifications processes such as electrospinning, carbonisation and supercritical carbon foaming (Nayeem *et al*., 2023). Moreover, the assumed hydrophobic nature and absence of hydrogen bonds of the vegetable oil based polysulphide adsorbent played a role for relatively low removal percentage (Hasell *et al*., 2016; Lin *et al*., 2019; Mann *et al*., 2021).

## CONCLUSION

In this study, polysulphides were prepared from sulphur and waste cooking oil in the ratio of 9:1 by inverse vulcanisation. The study was carried out to investigate the ability of polysulphide adsorbent in the removal of $Cu^{2+}$ ions. It was found that the adsorption is highly influenced by the following parameters such as initial $Cu^{2+}$ concentrations, adsorbent dosage, and contact time. The percentage removal of $Cu^{2+}$ was increased, with the increase of adsorbent dosage. On the other hand, the $Cu^{2+}$ removal efficiency was decreased when the initial $Cu^{2+}$ concentration was increased. The highest percentage removal of copper was recorded at 77.81% with 5 g of adsorbent in 5 mg $L^{-1}$ in aqueous solution. It was found that the equilibrium time for $Cu^{2+}$ adsorption onto polysulphide adsorbent was 80 min. The data obtained best fitted the Langmuir isotherm model with $R^2$= 0.9997. The results retrieved from this study have proven the ability of polysulphide adsorbent as a low-cost adsorbent. With proper research and development that can be carried out in the future, it shall be useful to be used in an up-scale treatment of contamination of other heavy metals, not only copper.

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