



## RESEARCH ARTICLE

## BIOSORPTIVE REMOVAL OF COPPER(II) FROM WASTEWATER BY HYBRID BIOSORBENT: ADSORPTION KINETICS AND MECHANISMS

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## ABSTRACT

Combining two well-known biosorbents, *Pleurotus sajor caju* and almond shell, with a newly created straightforward immobilization approach led to the creation of a unique hybrid biosorbent. The hybrid biosorbent's capacity to eliminate Cu(II) from aqueous solution was assessed. The immobilized hybrid biosorbent exhibited the greatest values for Cu(II) adsorption at a pH about 5.0. The ideal contact period and metal concentration were 240 minutes and 10 mg/L, respectively. The pseudo-second order kinetics and Langmuir adsorption isotherms models were used to explain the biosorption kinetics and equilibria. The presence of multiple functional groups and active sites may be seen in the hybrid biosorbent's FTIR spectra. Prior to biosorption, the hybrid biosorbent's SEM micrograph showed that its surface was uneven, rough, and porous. A successful and potentially alluring method for dealing with heavy metal-polluted aqueous solutions is the hybrid biosorbent technique, which is quite inexpensive and effectively removes a lot of Cu(II).

## KEYWORDS

Hybrid biosorbent, Cu (II), kinetic models, isotherm, SEM, FTIR.

## 1. INTRODUCTION

Natural metallic elements known as heavy metals have densities that are more than five times larger than the density of water (density > 5 g/cm<sup>3</sup>) (Enyoh et al., 2022; Ohiagu et al., 2021). People's health is seriously threatened by heavy metals. Specifically, among the metals that pose a serious risk to public health are Cr, As, Pb, Cu, Fe, Cd, Zn, and Ni. Even at low levels in the body system, these heavy metals are systemic toxins that cause a variety of organ problems in people (Sutton & Tchounwou, 2007; Yedjou & Tchounwou, 2007). Heavy metals are present in the environment naturally (Keshav Krishna & Rama Mohan, 2016). However, anthropogenic activities including mining, industrial and agricultural operations, careless dumping of solid waste, municipal wastewater discharges, incineration, etc. are the major ways that people are exposed to heavy metals (Rahimzadeh et al., 2017). Heavy metals are found as ores in the crust of the earth and are extracted as minerals through mining. In most ores, some heavy metals like Fe, Zn, Ar, Co, Ni, and Pb exist as sulfides, whereas other heavy metals like Mn, Al, etc. occur as oxides. Furthermore, heavy metals such as Co, Fe and Cu exist as both oxide and sulfide ores. Heavy metals are released into the environment from ores during mining activities and subsequently deposited in the soil, distributed through air and water to various regions of human habitation (Engwa et al., 2019).

In nature, copper (Cu) may be found as an element and in compounds. Cu ions can be either cupric ion (Cu<sup>2+</sup>) or cuprous ion (Cu<sup>+</sup>), depending on whether they are oxidized or reduced (Gaetke et al., 2014). Cu is released into the atmosphere by anthropogenic sources such iron and steel manufacturing, municipal incinerators, copper smelters, and volcanoes as well as natural sources like forest fires, wind-blown dust, and forest fires.

Contaminated drinking water, vitamin and mineral supplements, birth

control pills, copper cookware, copper intrauterine devices, copper water pipes, etc. are examples of environmental sources of copper (Pohl et al., 2011).

Reactive oxygen species, DNA, lipid, and protein deficiencies might result from elevated levels of Cu in the human blood, which could injure a number of important organs, including the liver, kidney, and pancreas (Gupta & Gogate, 2016). They harm aquatic animals' vital organs (such the liver, gills, and kidneys) and systems (like the nervous system) when they are in the aquatic habitat, in addition to affecting their sexual life. However, according to international regulatory organizations (such the USEPA, BIS, and WHO), the permitted international extent of Cu(II) ions in drinking or household water varies between 1.30, 1.50, and 2.00 mg L<sup>-1</sup>, respectively (Eldeeb et al., 2021; Rahman et al., 2021).

Many conventional methods, such as chemical precipitation, reverse osmosis, electrochemical methods, ion exchange, membrane filtering, coagulation, extraction irradiation, and adsorption, have been used to remove heavy metal ions (Sattar, 2021). Due to the need for safe, more effective, and less expensive methods of extracting copper ions from wastewater, research on low-cost alternatives to currently used techniques has become necessary. In recent years, a number of methods for removing heavy metals from aqueous solutions have been discovered. Solvent extraction, membrane filtration, chemical reduction and precipitation, coagulation, and ion exchange are a few of these techniques. The requirement for significant volumes of chemicals, residual metal solubility, expensive capital investments, high operational expenses, and the generation of a significant amount of sludge are the main downsides of these techniques (Fawzy et al., 2022). The removal of heavy metals from wastewater via biosorption has numerous benefits over conventional methods since it doesn't generate chemical slime, is more effective, is simple to apply, and is more economical (Ordóñez et al., 2023). Recent

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studies have demonstrated the effectiveness of many common agricultural waste products, microbes, algae, and natural polymers as biosorbents for heavy metal removal (Dil et al., 2019; Medhi et al., 2020).

An inventive approach for creating a hybrid biosorbent was examined, and an inventive hybrid biosorbent matrix was constructed by mixing *Pleurotus sajor caju* and almond shell waste biomass. Agricultural and microbial based materials have enormous biosorption ability as a biosorbent. To the best of our knowledge, however, no reports of hybrid biosorbent (*Pleurotus sajor-caju* and almond shell) for the biosorption of copper ions from aqueous solution have been made. Therefore, the goal of the current work was to reconsider and investigate the potential of employing hybrid biosorbent to remove copper from aqueous solution. To optimise copper removal by hybrid biosorbent, three independent variables—biosorbent pH, starting Cu(II) concentration, and time—were tuned. We also evaluated the effectiveness of an immobilized hybrid biosorbent as a Cu(II) ion biosorbent. In order to better understand the nature of the biosorbent and the biosorption process, kinetic and equilibrium experiments were conducted. SEM and FTIR were used to characterize the biosorbent.

## 2. MATERIALS AND METHODS

### 2.1 Chemicals

Sigma-Aldrich and Merck were used to acquire all of the compounds. To make stock Cu(II) solution, 29.5646 g of copper nitrate were dissolved in 1 L of DDW. By dilution of the metal stock solution, the necessary solutions were made. The pH of the solutions was changed using a total of 1M NaOH/HCl.

### 2.2 Hybrid Biosorbent Preparation and Characterization

Almond shell waste biomass was obtained from a nearby market, and *Pleurotus sajor-caju* was obtained from the Institute of Horticultural Sciences at the University of Agriculture in Faisalabad, Pakistan's Mushroom Laboratory.

The waste from almond shells was washed twice with distilled water, dried in an oven at 60°C to achieve a consistent mass, and then packed in a plastic box. *Pleurotus sajor-caju* slants were kept alive using potato dextrose agar (PDA) slants.

The liquid growth medium has the following composition (gram per liter of distilled water): Yeast (1.0), D-glucose (5.0), MgSO<sub>4</sub>·7H<sub>2</sub>O (0.2), KH<sub>2</sub>PO<sub>4</sub> (5.0), NH<sub>4</sub>NO<sub>3</sub> (2.0), Peptone (2.0), (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> (4.0), Trisodium citrate (2.5), and MgSO<sub>4</sub>·7H<sub>2</sub>O (2.0) are the other components.

The fungal strain of fungus (*Pleurotus sajor caju*) was developed by subculturing on potato dextrose agar slants for the manufacture of hyphal suspension. Hybrid matrix was created using the technique outlined by (Ashraf et al., 2015; Hanif et al., 2021). To create uniformly sized biomass beads (0.350 mm), waste from *Pleurotus sajor caju* and almond shells was combined with 2% sodium alginate and added dropwise into a calcium chloride (0.1M) solution (Sattar, 2021). With the samples manufactured as KBr discs, the surface morphologies of the samples were examined using a SEM (FEI Quanta 400F electron microscope), and the chemical properties of the hybrid biosorbent were deciphered using an FTIR Agilent N630.

### 2.3 Biosorption Studies

In a batch mode, it was estimated how different experimental variables, such as starting metal concentration, pH, and contact duration, would affect the sorption process. A Varian AA240, GTA 120 equipment was used to detect atomic absorption in order to discover the concentration of Cu in solutions.

The removal capacity was determined using the following equation:

$$q_e = \frac{(C_o - C_e)V}{W}$$

where V (L) is the volume of the solution, W(g) is the weight of the hybrid biosorbent utilized, and C<sub>o</sub> and C<sub>e</sub> (mg/L) stand for the initial and equilibrium metal ion concentrations, respectively (Pavithra et al., 2021).

### 2.4 Biosorption Isotherm Models

To ascertain the adsorption process for Cu(II) removal by hybrid biosorbent, the equilibrium adsorption data were fitted to the following isotherms in accordance with the equations provided in the literature,

including the Freundlich model (Udoka & Enenebeaku, 2019) and Langmuir model (Langmuir, 1918).

## 2.5 Statistical Analysis

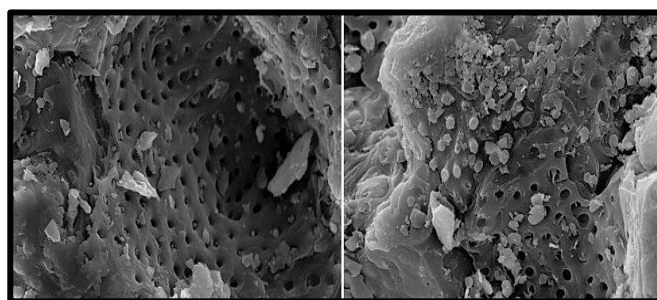
There were three copies of each experiment run. Regression analysis was used to statistically examine the data.

## 3. RESULTS AND DISCUSSIONS

### 3.1 Batch Study

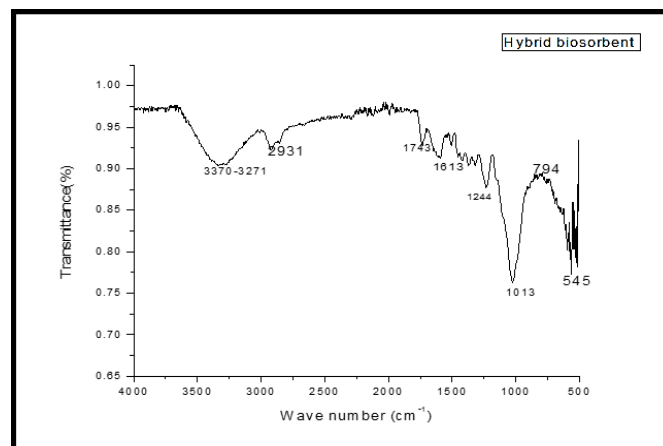
#### 3.1.1 Characterization

SEM may be used to assess the morphological distinctiveness of hybrid biosorbents. Figure 1a depicts a hybrid biosorbent SEM micrograph. Prior to biosorption, the hybrid biosorbent's SEM micrograph showed that its surface was uneven, rough, and porous. The accessible adsorptive surface is greatly increased by such a structure, increasing the ability of Cu(II) ions to bind (Figure 1). As a result, the adsorption process is more favorable with hybrid biosorbent. The holes on the surface of the biosorbents are filled with sorbate ions after metals have been biosorbed (Hanif et al., 2021).



**Figure 1:** SEM image of hybrid biosorbent before and after adsorption of Cu (II)

The presence of multiple functional groups and active sites may be seen in the hybrid biosorbent's FTIR spectra. An essential analytical method, FTIR spectroscopy identifies a molecule's chemical functional group based on the properties of its vibrations. In the 500-4500cm<sup>-1</sup> range, hybrid biosorbent was properly explored (Jerald et al., 2017). Figure 2 displays the hybrid biosorbent's FTIR spectrum.



**Figure 2:** FTIR spectra of Hybrid biosorbent

### 3.2 Effect of pH

Keeping all other experimental parameters constant (50 mg of metals L<sup>-1</sup>, 220 rpm of agitation, 0.1 g of adsorbent material, and 440 min of contact time), a batch adsorption experiment was conducted to evaluate the impact of initial pH on the removal of copper from aqueous solution. The maximal copper adsorption capacity q was (81.83) mg/g, while pH 5.0 was the optimal pH for hybrid biosorbent. Figure. 3 shows the impact of pH on the adsorption of Cu onto a hybrid biosorbent. In actuality, it is well known that most carboxylic groups do not dissociate at low pH and are hence unable to link metal ions in solution. Because of the dissociation of carboxylic groups at higher pH, there is an increase in biosorption (Iftikhar et al., 2009). In order to optimize other experimental conditions, pH=5 was chosen.

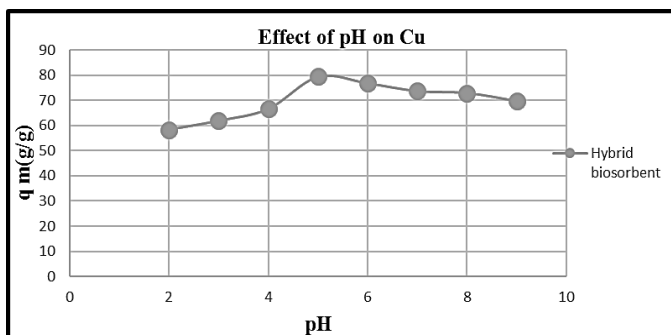


Figure 3: Effect of pH on Cu (II)

### 3.3 Effect of initial metal concentration

At pH 5.0 and using 0.1 g of adsorbent material, the effect of metal concentration (from 0 to 150 ppm) on the removal of Cu from the aqueous solution was investigated.

The hybrid biosorbent's adsorption capacity  $q$  (mg/g) for sequestering metals at varying starting metal concentrations of 10, 25, 50, 75, 100, and 150  $\text{mgL}^{-1}$  was assessed (Figure 4). The findings showed that while % removal falls with increasing metal concentration, absorption affinity  $q$  (mg/g) increases with starting metal concentration.

At a concentration of  $10\text{mgL}^{-1}$ , copper has a maximum adsorption capability of 91% (mg/g). As the metal concentration increased, the elimination of Cu reduced. This may be explained by taking into account the fact that all adsorbents have a finite number of active sites, and that the active sites become saturated at a particular concentration (Jagaba et al., 2020).

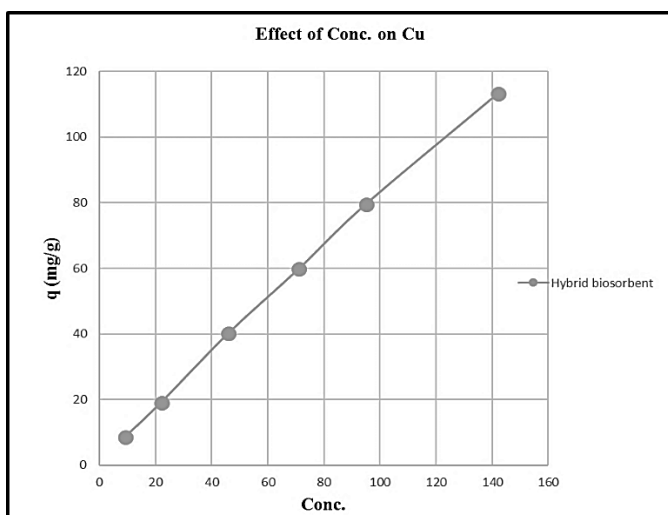


Figure 4: Effect of initial metal concentration on Cu(II)

### 3.4 Effect of contact time

At starting concentrations of 50 mg/l and a temperature of  $25^{\circ}\text{C}$ , the effects of contact time on the adsorption of metals copper were investigated. Figure 5 shows the experimental findings from this investigation. It seems that there is an initial quick absorption and that the adsorption capacity rises with concentration. The state of equilibrium reached after fewer than 240 minutes of shaking. The hybrid biosorbent achieved the highest absorption for copper (80%).

Metal uptakes first rise as contact duration increases. These differences in metal absorption may be the result of the early stage's increased driving force, which makes it easier for metal molecules to move to adsorbent surfaces, as well as the adsorbent's abundance of active sites. After that time, the reduction in the number of active adsorption sites and the long-range metal molecule diffusion effect may be responsible for the decline in adsorption rate (Zhang et al., 2019). Thus, equilibrium was always reached with a contact time of less than 240 minutes. Since there are solely sorbate and sorbent interactions, with little interference from solute-solute interactions, the rapid absorption of the metal molecules is caused by solute transfer. When a result, the initial rate of adsorption was high for high starting metal concentrations; however, when the mass transfer driving force is raised, the resistance to the metal absorption decreases (Majeed et al., 2012).

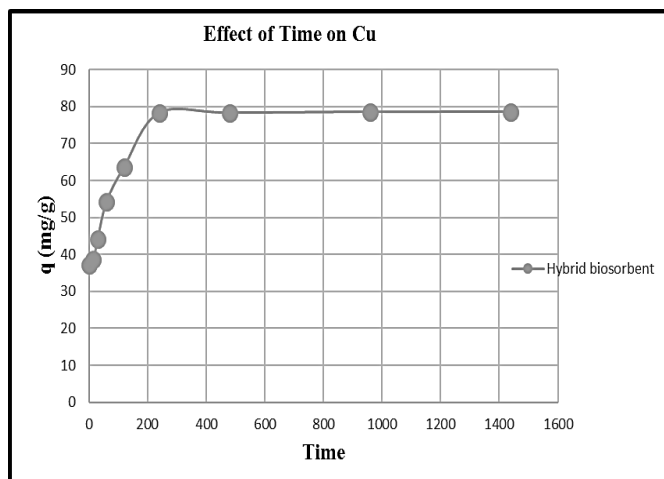


Figure 5: Effect of contact time on Cu (II)

### 3.5 Kinetic Modelling

Two kinetic models, pseudo-first-order and pseudo-second-order, were fitted to the experimental data in order to examine the kinetic process of Cu adsorption and possible rate regulating stages. Table 1 compares the pseudo-first-order kinetic model with the pseudosecond-order kinetic model. The calculated values of  $q_e$   $\text{mgg}^{-1}$  corresponded with the experimental ones given in Figures 6 (a, b) and the coefficient of correlation for the second order kinetic model was roughly equal to 1.

Both of these facts suggest that Cu is adsorbed according to the 2nd order kinetic model, which is based on the notion that adsorption might be a rate-limiting process. The beginning concentration, equilibrium concentration, and contact time were the experiment variables employed in the contact time inquiry (Wang et al., 2016).

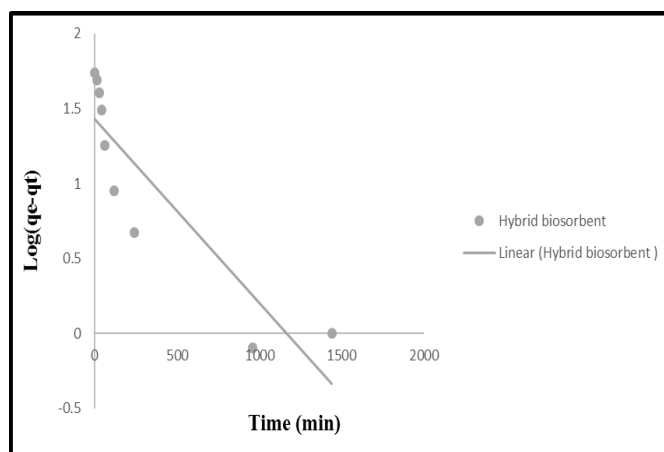


Figure 6(a): Pseudo 1st order plot for Cu(II)

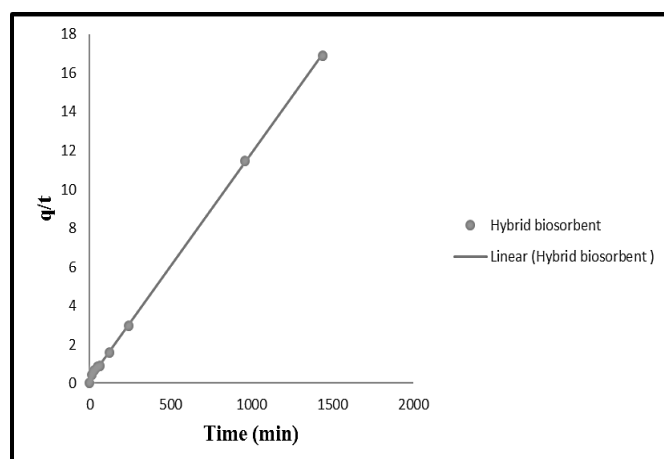


Figure 6(b): Pseudo 2<sup>nd</sup> order plot for Cu(II)

**Table 1:** Comparison between pseudo-first order and 2nd-order kinetic models for the adsorption of Cu (II)

Heavy metals	Biomass	Pseudo first order			Pseudo second order			
		q <sub>e</sub>	K <sub>1ad</sub>	R <sup>2</sup>	q <sub>exp</sub>	q <sub>e</sub>	K <sub>2ad</sub>	R <sup>2</sup>
	(mg/g)	(min <sup>-1</sup> )	(mg/g)		(mg/g)	(mg/gmin)		
	Hybrid biosorbent	28.464	0.000621059	0.7907	85.1769114	90.90909091	0.000527693	0.9996

### 3.6 Biosorption Isotherms

Many ideas have been employed in an effort to comprehend the adsorption process. Adsorption chemistry is a challenging field. Sorption isotherm models are frequently used for data fitting in order to analyze the connection between adsorbed and aqueous concentrations at equilibrium; the Langmuir and Freundlich adsorption isotherm are the most frequently utilized (Lalhruaitluanga et al., 2011).

The adsorption results (at 30 °C) were very near in value to the experimental and estimated maximum capacity. They also fit the Langmuir isotherm well (R<sup>2</sup> = 0.993). There was also evidence of a poor match to the Freundlich model.

Despite the need for extreme caution when extracting particular data for the degree of model fitting, it is possible to infer from the aforementioned results that both a monolayer and a more heterogeneous adsorption process occurred.

**Table 2:** Equilibrium modeling of data for the uptake of Cu (II) ions by hybrid biosorbent

Metal	Biomass	Langmuir model			Experimental Value q(mg/g)	Freundlich model			
		X <sub>m</sub> (mg/g)	K <sub>L</sub> (L/mg)	R <sup>2</sup>		q <sub>e</sub> (mg/g)	1/n	K <sub>F</sub> (mg/g)	R <sup>2</sup>
Cu	Hybrid biosorbent	277.77	0.003600101	0.99	273.14	342.1	0.25	64.18	0.85

## 4. CONCLUSION

In order to remove Cu(II) from aqueous solution, this study effort sought to combine two biosorbents, namely (Almond shell and *Pleurotus sajor caju*). Cu(II) is most readily absorbed at pH 5. At a concentration of 10 mg L<sup>-1</sup>, Cu(II) has a maximum adsorption capacity of q(mg/g) of 92%. The presence of multiple functional groups and active sites may be seen in the hybrid biosorbent's FTIR spectra.

A SEM micrograph of the hybrid biosorbent surface revealed that it is uneven and rough, which increases the metal ions' ability to attach to it. The pseudo-second order kinetic model describes the sorptions of Cu(II). The Cu(II) concentration data fit well with the Langmuir sorption isotherm model. According to the results of the entire investigation, hybrid biosorbent composed of microbial and plant waste biomass was very effective in excluding Cu(II) from aqueous solution.

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