



P-ISSN: 2349-8528

E-ISSN: 2321-4902

[www.chemijournal.com](http://www.chemijournal.com)

IJCS 2022; 10(3): 01-07

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Received: 01-03-2022

Accepted: 04-04-2022

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## Kinetic and equilibrium studies of copper (II) adsorption from synthetic wastewater by *Oedogonium hatei*, a locally available green alga

**Dr. Arshi Rastogi****Abstract**

The ability of non-viable *Oedogonium hatei*, green alga, to adsorb and remove Copper (II) metal ions from synthetic wastewater in a batch mode, is the subject of this research. The effects of key parameters such as sorption pH, temperature, initial metal ion concentration, contact time, and adsorbent dosage were investigated. The equilibrium data was applied to Langmuir and Freundlich isotherms, and it fitted well with Langmuir's model. The maximum adsorption capacity derived from the Langmuir isotherm was 56.18 mg/g at 90-minute contact time, 4.96 pH and 313K. The thermodynamic studies revealed that sorption onto algal biomass was feasible, spontaneous, endothermic, and physisorption process. Pseudo-first-order and pseudo-second-order kinetic models were applied to get insight into the adsorption process mechanism. Surface area calculation, elemental analysis, and FTIR spectral analysis were among the physical and chemical parameters of the algal biomass determined. According to the findings, *Oedogonium hatei* alga could be used as an adsorbent material to remove harmful copper (II) metal ions from wastewaters.

**Keywords:** Adsorption, copper (II), *Oedogonium hatei*, kinetic model

**1. Introduction**

Heavy metals found in high quantities in aquatic systems have been a major source of concern for scientists all around the world. Heavy metals are mostly introduced into water bodies by industrial and domestic effluents, which have a negative impact on people's health. Copper is one such harmful and poisonous heavy metal that, if present at high levels in drinking water, can produce immediate consequences in humans such as vomiting, diarrhea, stomach cramps, and nausea <sup>[1]</sup>, as well as being lethal in extreme cases. Solvent extraction, ion exchange, chemical precipitation, reverse osmosis, adsorption, oxidation, reduction, dialysis, electro-dialysis, and other physicochemical procedures have been documented for the treatment of heavy metal-containing wastewaters <sup>[2, 3]</sup>. However, due to its cost-effectiveness and environmental friendliness <sup>[4, 5]</sup>, the adsorption approach is considered to be the most promising of these.

Several low-cost and non-conventional natural materials, industrial products, agricultural wastes, and biomasses have been investigated, in the search for novel adsorbent materials for the removal of copper metal-containing wastewater <sup>[6-12]</sup>. However, algal biomass has lately proven to be more effective and advantageous due to its abundance and ease of biodegradability. The cell wall, which is made up of a fiber-like structure and an amorphous embedding matrix of different polysaccharides, has been credited with its adsorption ability. There have been reports of hazardous copper (II) metal ion adsorption by algae such as *Undaria pinnatifida* <sup>[13]</sup>, *Spirogyra neglecta* <sup>[14]</sup>, *Gelidium sp* <sup>[15]</sup>, *Cladophora fascicularis* <sup>[16]</sup>, *Caulerpa lentillifera* <sup>[17]</sup>, *lemna minor* L. <sup>[18]</sup>, but no reports of Cu(II) removal by *Oedogonium hatei*, alga.

*Oedogonium* sp. (a Chlorophyta) is a freshwater green filamentous alga that can be easily found in adjacent freshwater ponds and rivers, making it a cost-effective study material. Our lab previously demonstrated that this alga has a high adsorption capacity for heavy metal ions such as Cr (VI), Pb (II), Cd (II), and Ni (II) from aqueous solutions <sup>[19-22]</sup>. We present the adsorption capabilities of this alga in our ongoing search for a potential adsorbent material for hazardous Cu(II) removal from synthetic wastewater. The impact of major influencing variables on the sorption phenomena was investigated, followed by adsorption isotherm calculation, kinetic models, and thermodynamic analyses. The algal biomass was characterized

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physically and chemically, and the test alga's adsorption capacity for copper (II) metal was compared to that of previously reported algal biomasses.

## 2. Material and Methods

### 2.1. Chemicals and Equipment

All the chemicals and reagents used in this investigation were of analytical grade, either from Merck, Germany, or S.D Fine Chem. Ltd. India. A digital pH metre (PERFIT, India) was used to measure the pH of the solution, and an atomic absorption spectrophotometer-model Z 7000 (Hitachi, Japan) was used to record the absorbance of the samples. Using KBr pellets and a Thermo Nicolet FTIR (Germany), the spectra of biomass were obtained in the 4000-400  $\text{cm}^{-1}$  region. The chemical analysis of the adsorbent was performed using the elemental analyze system Vario MICRO CHNS V3.1.1 (GmbH, Germany).

### 2.2. Preparation of simulated synthetic wastewater

Synthetic wastewater refers to wastewater that has been artificially generated and contains Cu (II) ions as adsorbate. Cu (II) stock solution was made by dissolving the required amount of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  in double distilled water to reach a concentration of 1000 mg/L, which was then diluted as needed for experiments. To check for copper precipitation, 10 mL of  $\text{HNO}_3$  was added to the stock solution.

### 2.3. Algal biomass (adsorbent) preparation and Characterization

The test alga *Oedogonium hatei* was taken from a nearby freshwater pond and utilized as an adsorbent in this study. To eliminate extraneous material, the biomaterial was rinsed with tap water and then distilled water numerous times. After three days of sun drying, it was dried in the oven at 343K for 24 hours. After that, a domestic mixer and pestle mortar were used to crush the dried material. The powdered material was sieved to the required 100 mesh size and stored in vacuum desiccators before to use.

The surface area of the adsorbent was calculated using the BET method [23] and elemental analysis was utilized to estimate the percentage composition of Carbon, Nitrogen, and Sulphur components. Finally, an FT-IR transmission study was performed to determine the primary chemical functional groups present on the adsorbent's surface.

### 2.4. Experimental procedure

Adsorption experiments were carried out in batches using 100 mL Erlenmeyer flasks that had been cleaned with  $\text{HNO}_3$  to eliminate any traces of metal that had remained adsorbed on the flask's glass wall. Each flask was filled with the optimal biomass dose (adsorbent) and stirred at 150rpm on a rotary shaker until equilibrium was established. By adding 0.1 M HCl to the solution, the pH was kept at an optimal 5. The biomass was then separated using a membrane filter with a pore size of 0.45  $\mu\text{m}$ . Before analysis, the filtrate was collected in tiny tubes and diluted. An atomic absorption spectrophotometer set to 324.8 nm was used to determine the metal content (Cu (II)) in the filtrate.

The mass balance equation was used to calculate the amount of Cu (II) adsorbed by *Oedogonium hatei*, i.e., adsorption capacity, in each flask.

$$q_e = (C_o - C_e)V / M \quad (1)$$

where  $q_e$  is the algal adsorption capacity (mg/g),  $C_o$  and  $C_e$  are the initial and the equilibrium concentration of Cu(II) metal ion (mg/L),  $V$  is the volume of the reaction mixture (L) and  $M$  is the mass of adsorbent utilized (g). All of the experiments were repeated three times, with the average results provided. The standard deviations were determined to be around  $\pm 1.4\%$ .

### 2.5. Influence of operational parameters

The effects of key parameters such as the influence of contact time (range 1- 300 min), pH of the solution (range 3-7), adsorbent dose (0.005 - 1 g/L), and temperature (293, 303, and 313 K) on the adsorption capacity of algal biomass were investigated. The adsorbent was suspended in copper metal ion solution at three different temperatures, 293, 303, and 313 K, while the other parameters were kept constant to yield adsorption isotherms. At 313 K, kinetic experiments of algal biomass adsorption were conducted at two distinct concentrations (100 and 200 ppm), with the extent of adsorption measured at regular time intervals.

### 2.6. Adsorption isotherms and kinetic models

Two basic adsorption isotherm model equations explored to optimize the design of an adsorption system are summarized here (Table 1). The kinetic models listed in Table 1 were used to investigate the regulating mechanism and dynamics of the adsorption process.

**Table 1:** Adsorption Isotherms and Kinetic Models

Model	Equation	Parameters	References
<b>Adsorption Isotherm</b>			
Langmuir Isotherm	$\frac{1}{q_e} = \frac{1}{Q_0} + \frac{1}{bQ_0C_e}$	$q_e$ (mg/g): equilibrium adsorption capacity $Q_0$ (mg/g): maximum adsorption capacity $C_e$ (mg/L): equilibrium concentration of adsorbate (Cu(II)) $b$ : Langmuir constant	[24]
Freundlich Isotherm	$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e$	$K_F$ ( $\text{mg}^{1-1/n} \text{L}^{1/n} \text{g}^{-1}$ ): Freundlich constant $n$ : intensity of adsorption.	[25]
<b>Kinetic Study</b>			
Pseudo-first order	$\log (q_e - q_t) = \log q_e - \frac{k_{1,ads}}{2.303} t$	$q_t$ (mg/g): amount of Cu(II) adsorbed at equilibrium time $t$ $k_1$ ( $\text{min}^{-1}$ ): pseudo-first-order rate constant.	[26]
Pseudo-second order	$\frac{t}{q} = \frac{1}{k_{2,ads}q_e^2} + \frac{1}{q_e} t$	$q$ (mg/g): amount of the Cu(II) adsorbed at time $t$ $k_2$ ( $\text{g mg}^{-1} \text{min}^{-1}$ ): rate constant of second-order adsorption	[27]

## 2.7. Thermodynamic Studies

Thermodynamic parameters (Gibbs free energy change ( $\Delta G^\circ$ ), enthalpy change ( $\Delta H^\circ$ ), and entropy change ( $\Delta S^\circ$ )) were analyzed using conventional general equations to understand the system's overall performance given as:

$$\Delta G^\circ = -RT \ln(b) \quad (2)$$

$$\ln\left(\frac{b_2}{b_1}\right) = -\frac{\Delta H^\circ}{R} \left(\frac{1}{T_2} - \frac{1}{T_1}\right) \quad (3)$$

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \quad (4)$$

## 3. Results and Discussion

### 3.1. Characterization of the adsorbent

Standard procedures were used to determine the physical and chemical parameters of the algal mass (adsorbent). -0.065 V, 1.2 percent, and 1.1 g/cm<sup>3</sup> were calculated as the zeta potential, humidity, and apparent density, respectively. The

BET method determined the surface area of the adsorbent as 1.3 m<sup>2</sup>/g, and the elemental analysis revealed that the carbon, nitrogen, and sulfur content of the adsorbent as 25.4, 3.06, and 1.77%, respectively.

The FTIR transmission spectra of the test algae were recorded before and after copper heavy metal ion adsorption. Table 2 shows the IR absorption bands and their related potential groups that can interact with protons or copper metal ions. After Cu (II) adsorption on the algal surface, the wavenumber of the conspicuous IR peak changes, indicating that new bonds are formed between the adsorbent and the metal ion, and that the binding happens on the biomass surface<sup>[28]</sup>. As a result of this study, it was shown that the cell wall of non-viable algal biomass contains a complex chemical composition involving functional groups such as carboxyl, hydroxyl, amine, amide, and amino, which are responsible for Cu(II) heavy metal ion binding. It's worth noting that these findings don't provide a quantitative study of the level of affinity between the metal and the functional groups on the algal surface.

**Table 2:** Surface functional groups observed on the test alga before and after Copper (II) adsorption.

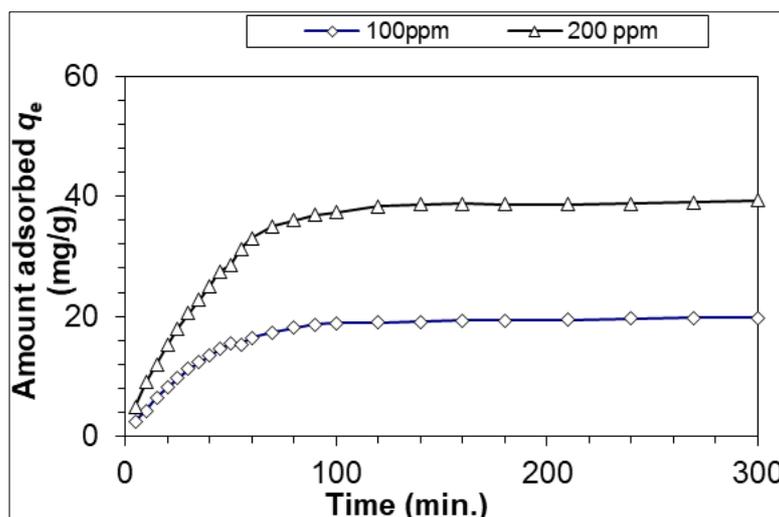
Test Alga before adsorption of Cu(II) (wavenumber in cm <sup>-1</sup> )	Test Alga after adsorption of Cu(II) (wavenumber in cm <sup>-1</sup> )	Bonds indicative of Functional groups
3407	3411	Carboxylic/OH stretch and N-H stretch
2917	2913	Phenolic/carboxylic
-	2356	-CH Stretch
1647	1650	C=O chelate stretching, amide I band
1540	1535	Amide II band, OH bonds,
1427	1425	Symmetric bending of CH <sub>3</sub> of the acetyl moiety
1241	1245	=C-C=, P=O
1169	1173	≡C-N<
1059	1062	-CN stretching, Plane deformation

### 3.2. Adsorption of heavy metal ion

#### Influence of contact time

The influence of contact time on adsorption efficiency of *Oedogonium hatei* biomass for Cu (II) was studied for an agitation time of 1-300 minutes, using 0.25 g of algal dose, 50 ml of 100 and 200 ppm Cu (II) solution at pH 4.96 and temperature 313 K. It can be observed from figure 1 that the

contact time of 90 min. was adequate to attain equilibrium, and the adsorption does not change considerably as the contact time is increased. The initial rapid phase may involve physical adsorption at the cell surface until it reached the equilibrium point in 90 min. Other mechanisms, such as complexation and binding site saturation, may be involved in the slower phase after equilibrium.



**Fig 1:** Influence of contact time on copper adsorption onto *Oedogonium hatei* biomass

#### Influence of adsorbent dose

To assess the effect of adsorbent dose, different amounts of algal biomass in the range of 0.005 to 1.0 g/L were examined.

These were suspended in 50 mL adsorbate solutions with a Cu (II) concentration of 200 ppm under optimal pH, temperature, and contact duration conditions. The effect of adsorbent dose

on the amount of copper adsorbed in mg/g and the extent of metal adsorbed (percent) / removal percent, of copper by the test alga is shown in Figure 2. The algae's maximal adsorption capacity achieved equilibrium at 0.25 g/L, as illustrated in

Figure. Such behavior is related to the increase in the number of active sites available for copper metal ion adsorption is in agreement with previous observations on Copper (II) and lead (II) sorption from aqueous solution by *Spirogyra neglecta* [14].

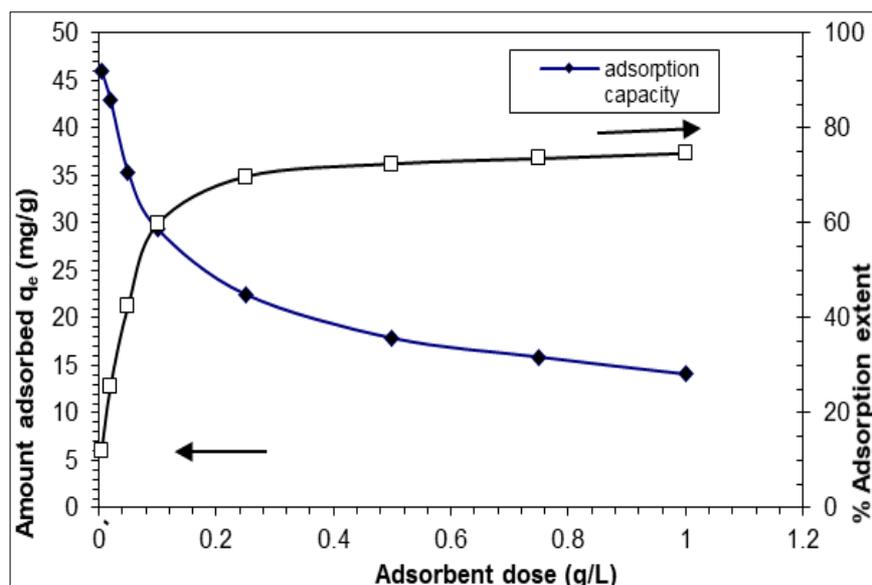


Fig 2: Influence of adsorbent dosage on copper adsorption onto *Oedogonium hatei* biomass

### Influence of pH

Figure 3 shows how the pH of the adsorption media altered the equilibrium adsorption of Cu (II) ions onto *Oedogonium hatei*, with uptake increasing from pH 3.0 to 4.96 and subsequently decreasing. Thus, the optimum initial pH corresponding to the maximum adsorption capacity of alga is an acidic pH of 4.96. At low pH, hydronium ions surround the adsorbent's surface, reducing copper interaction with the test

alga's binding sites via stronger repulsive forces. The overall surface on the alga becomes negative as the pH rises, and adsorption increases. After pH 6.01, insoluble copper hydroxide starts precipitating from the solution. Therefore, studies at higher pH values were not possible. Similar pH dependency behavior was reported by Ozer *et al.* for the adsorption of Cu (II) onto *Cladophora crispata* algal surface at 4.5 pH [29].

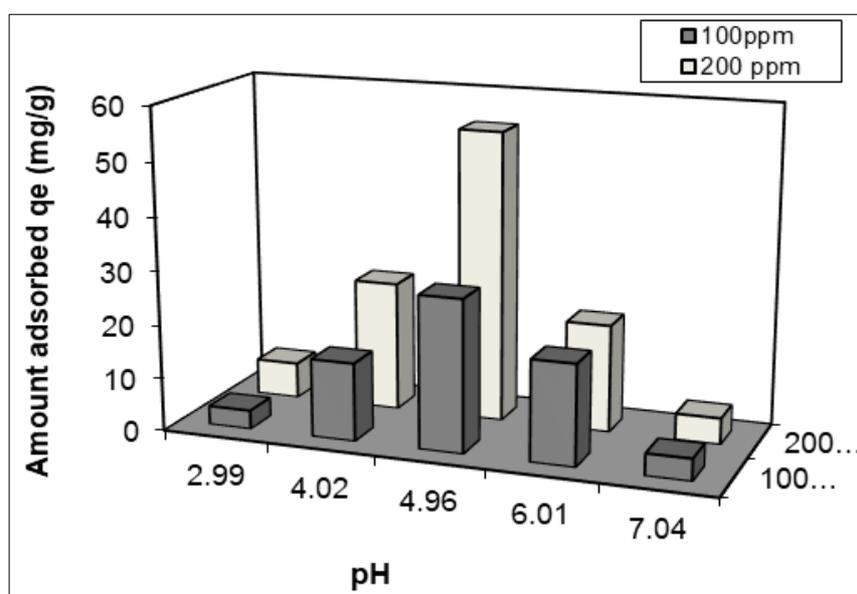


Fig 3: Influence of pH on copper adsorption onto *Oedogonium hatei* biomass.

### Influence of temperature and thermodynamic study

When the temperature was increased from 293 to 313K, the adsorption capacity of the adsorbent progressively increased (Figure 4), thereby suggesting that the copper metal

adsorption is an endothermic process. Deng *et al* had reported an increase in equilibrium with temperature indicating an endothermic process involved for Cu (II) ions uptake by *Cladophora crispata* alga<sup>29</sup>.

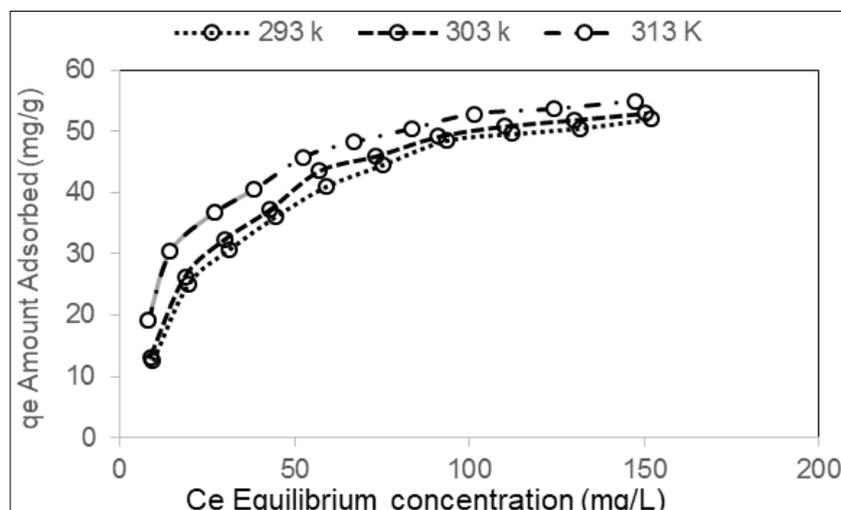


Fig 4: Influence of Temperature on copper adsorption onto *Oedogonium hatei* biomass.

The values of thermodynamic parameters are evaluated and represented in Table 3. The negative value of  $\Delta G^\circ$  at three different temperatures demonstrates that the adsorption process involved in the present study is feasible and spontaneous. Furthermore, the positive value of  $\Delta H^\circ$  implies the endothermic nature of metal ion sorption which is already evidenced by the increase in adsorption capacity with temperature. The experimental value of  $\Delta H^\circ$  is found to be less than 40 kJ/mol., which indicated that the adsorption of Cu (II) ions on *Oedogonium hatei* is physisorption in nature. The increased randomness at the solid-solution interface during the fixation of copper (II) metal ions on the active sites of the adsorbent is reflected by the positive value of  $\Delta S^\circ$ . These results are similar to those obtained by other researchers also [15, 17].

### 3.3. Adsorption Isotherms

The adsorption data were evaluated in terms of Langmuir and Freundlich isotherm models. The values of isotherm parameters and correlation coefficients ( $R^2$ ) are listed in Table 3. The value of  $q_e$  responsible for indicating adsorption capacity as calculated using the Langmuir model, has been observed to be 56.179 at 313 K with very high  $R^2$  values (>0.99). Dimensionless separation factor,  $R_L$ , can be

calculated from Langmuir constant,  $b$  at different temperatures, given as:

$$R_L = 1/(1 + bC_0) \quad (5)$$

The nature of the adsorption isotherm depends on the criterion that if the  $R_L$  value is greater than 1 then it is considered to be Linear, if the value of  $R_L$  falls between 0 and 1 it is favorable, and in case the value is equal to 0, it is considered to be irreversible. In the present study, the values for  $R_L$  are found to be less than unity and greater than zero at all temperatures, confirming the favorable adsorption process.

In order to indicate the surface heterogeneity nature of the adsorbent, Freundlich isotherm constants ( $K_f$ ,  $N$ , and  $R^2$ ) were also determined (Table 3). As the values of  $N$  (>1) indicate favorable and heterogeneous adsorption of metal ions onto algal biomass. The findings leads us to confirm that the Langmuir isotherm is the most appropriate isotherm in terms of high correlation coefficient values ( $R^2$ ) in comparison to Freundlich isotherms. The satisfactory fitting of the Langmuir model to the adsorption isotherms of copper (II) on marine alga *Bifurcaria bifurcate* has been reported earlier, confirming monolayer coverage of Cu(II) ions onto outer surface of the adsorbents.

Table 3: Isotherm constants and thermodynamic parameters for the adsorption of Copper (II) onto *Oedogonium hatei* at three different temperatures

Isotherm Parameters	<i>Oedogonium hatei</i>		
	293K	303K	313K
<b>Langmuir Isotherm</b>			
B (L mg <sup>-1</sup> )	0.02835	0.01750	0.01235
$q_e$ (mg g <sup>-1</sup> )	46.296	50.50	56.179
$R^2$	0.9906	0.9904	0.9903
<b>Dimensionless Separation Factor</b>			
$R_L$	0.002	0.004	0.006
<b>Thermodynamic parameters</b>			
$\Delta G^\circ$ (kJ mol <sup>-1</sup> )	-16.235	-17.669	-19.507
$\Delta S^\circ$ (kJ mol <sup>-1</sup> K <sup>-1</sup> )	0.0527	0.0462	0.0389
$\Delta H^\circ$ (kJ mol <sup>-1</sup> )	31.676		
<b>Freundlich Isotherm</b>			
N	1.5225	1.7259	1.9223
$K_f$ (mg g <sup>-1</sup> )	14.629	21.887	31.481
$R^2$	0.9371	0.9368	0.9037

\* Measured between 293 and 313 K.

### 3.4. Kinetic Models

Table 4 lists the results of the adsorption kinetics of copper metal onto the adsorbent surface. It was observed that the calculated value of  $q_e$  was close to the experimental value of  $q_e$  for pseudo-second-order kinetics, with a good correlation ( $R^2$ ) at two different concentrations of adsorbent (100 and 200

ppm), in comparison to that of the pseudo-first-order model. Therefore, it is concluded that the pseudo-second-order model best fits to describe the adsorption kinetics of Cu (II) ions onto *Oedogonium hatei* biomass. Similar results were reported by Saygideger *et al.* for Cu (II) adsorption onto macrophyte *Lemna minor* L<sup>18</sup>.

**Table 4:** Kinetic parameters for the adsorption of Copper (II) onto algal biomass *Oedogonium hatei*

Heavy metal ion	Initial dye Conc. (mg L <sup>-1</sup> )	$q_e$ (exp.) (mg g <sup>-1</sup> )	First-order model			Second-order model		
			$K_1$ (x10 <sup>-3</sup> min <sup>-1</sup> )	$q_e$ (cal.) (mg g <sup>-1</sup> )	$R^2$	$K_2 \times 10^{-3}$ (g mg <sup>-1</sup> min <sup>-1</sup> )	$q_e$ (cal.) (mg g <sup>-1</sup> )	$R^2$
Copper (II)	100	18.7	17.04	10.65	0.796	1.688	22.07	0.993
	200	42.9	19.34	24.84	0.898	0.76	44.44	0.991

### 3.5. Comparison with other adsorbents

The adsorbent utilized in this study (*Oedogonium hatei*) is compared to previously reported adsorbents for the removal

of copper (II) using the parameter ( $q_e$ ) adsorption capacity (Table 5). With the exception of a few, the test algae exhibited good outcomes in the comparison.

**Table 5:** Adsorption capacities of various biomass for Copper metal ion from aqueous solutions.

Algal biomass	Adsorption capacity ( $q_e$ ) (mg/g)	References
<i>Spirogyra neglecta</i>	115.3	[14]
<i>Gelidium sp.</i>	33.0	[15]
<i>Cladophora crispata</i>	57.5	[29]
<i>Spirogyra sp.</i>	133.4	[30]
<i>Oscillatoria limnetica</i>	23.96	[31]
<i>Anabaena spiroides</i>	0.24	[31]
<i>Eudorina elegans</i>	1.08	[31]
<i>Chlorella vulgaris</i>	9.47	[31]
<i>Gracillaria sp.</i>	37.5	[32]
<i>Ulva sp.</i>	47.7	[32]
<i>Bifurcaria bifurcata</i>	101.9	[33]
<i>Oedogonium hatei</i>	56.18	This study

### 4. Conclusions

The findings of this study showed that *Oedogonium hatei*, a locally available green alga, can be employed as an effective adsorbent for removing harmful copper metal ions from aqueous solutions. The adsorption was quick enough, with maximal removal occurring within 90 minutes of contact time. Batch adsorption studies showed that the total adsorption capacity (monolayer saturation at equilibrium) of the test alga for Cu(II) ions was 56.18 mg/g at an algal dose of 0.25g/L in 90 min of contact time with optimum pH 4.96 at 313 K. Thermodynamic studies revealed that the adsorption process was endothermic, spontaneous and physisorption. The results of FTIR analyses indicated the participation of -COOH, -OH, and -NH<sub>2</sub> groups in binding Cu(II) onto the adsorbent surface.

### 6. Acknowledgement

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

### 7. Conflict of interest

The author declares no conflict of interest.

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