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Kinetic and Isotherm Studies of Cu(II) Removal from Aqueous Solution Using *Gigartina Acicularis* Biomass

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Abstract

The biosorption characteristics of Cu(II) ions from aqueous solution using the red alga (*Gigartina acicularis*) biomass were investigated as a function of pH, biomass dosage, contact time and metal concentrations at room temperature (20-23°C). The Langmuir and Freundlich isotherm models were used to describe the equilibrium data and the isotherm constants were determined. The experimental adsorption data were fitted to the Langmuir isotherm model. The maximum adsorption capacity was 14.77 mg/g. Experimental data were also tested in terms of biosorption kinetics using pseudo-first-order and pseudo-second-order kinetic models. The results showed that the biosorption processes of both metal ions followed well pseudo-second-order kinetic. The sorption mechanisms were characterized using Fourier transform infrared spectroscopy (FTIR) and Energy dispersive X-ray emission (EDX). The *Gigartina acicularis* biomass investigated in this study exhibited a high potential for the removal of Cu(II) from aqueous solution.

Keywords: *Gigartina acicularis*; Cooper; Biosorption; Isotherm studies; Kinetics

1. Introduction

Protecting environment from contamination caused by modern industries and technologies is one of the concerns of today's world because they threaten humans public health (Inghui & Caoc, 2009). High tendency of heavy metals to maintain, circulate and accumulate in food chain is one of the most important factors that show their high importance. These metals enter the environment due to various industrial procedures including melting, extraction and purification. Presence of heavy metals and their compounds, even in small amounts, in industrial wastewater is very dangerous and must be removed (Hmetsarı, 2008).

Conventional methods which have been used for the removal of toxic metals such as copper from aqueous environment include ion exchange, reverse osmosis, evaporation, membrane filtration processes, chemical precipitation, adsorption and so on. However, these methods are expensive and more often, lead to secondary pollution problem. They also become inefficient when the concentration of the heavy metals in the effluent is low. Biosorption has been employed as an alternative method for removal of toxic metal ions from dilute aqueous solutions and industrial effluents (Sari et al., 2008; Sethunathan et al., 2005; Vieira et al., 2007; Anyurt et al., 2009 & Atolaiye et al., 2009).

Biosorption uses inexpensive dry biomass to extract industrial effluents of toxic heavy metals. The biomass can be composed of algae, mosses, fungi, bacteria, and various plant species. Seaweed biomass possesses a high metal uptake capacity due to its physicochemical characteristics (Padilha et al., 2005). Seaweed biomass contains various chemical groups attached to the cell wall polysaccharides and proteins, such as amino, carboxyl and hydroxyl groups (Yalçin et al., 2010), which are responsible for the interactions with metal cations (Yang et al., 2010).

The aim of the present work is to explore the possibility of utilizing *Gigartina acicularis* biomass for the adsorptive removal of copper (II) from aqueous solutions. The effect of such factors as initial pH, adsorbent dose, contact time and initial concentration was investigated. The kinetics of Copper (II) adsorption on this adsorbent was analyzed by pseudo-first-order and pseudo-second-order kinetic models. Cu(II) sorption equilibrium was evaluated using Langmuir and Freundlich isotherms. Cu(II) sorption mechanisms were elucidated using Fourier transform infrared spectroscopy (FTIR) and Energy dispersive X-ray emission (EDX).

2. Experimental

2.1. Marine alga collection and treatment

The biosorbent of red macroalga, *Gigartina acicularis* used in this study was collected on the coastal region of Rabat within North latitude of 34°03′ and a west longitude of 6°46′ with an altitude of 79 m. The beach of Rabat is located 35 km away from Kenitra city, on the left bank of the mouth of Bouregreg River. It is bordered by the Atlantic Ocean to the west. The alga was stripped of their epiphyte and debris adhering to their frond, rinsed on site with seawater and then placed in plastic bag. Upon arrival at the laboratory, alga was again rinsed with distilled water and dried in the open air, in a direct contact with the sun to ensure a complete dehydration. The dried seaweed was then crushed to reduce their size, and obtain a very fine powder which served for the remainder of our study.

2.2. Chemicals

Copper solution was prepared by dissolving a weighed quantity of Copper nitrate-2,5-hydrate in deionized distilled water. Before the adsorption study, the pH of the Cu(II) solution was adjusted to required value with 1M HCl and 1M NaOH solutions using a pH meter.

2.3. Sorption isotherm experiment

Cu(II) sorption experiments were conducted in shake flasks. The seaweed biomass (1 g) was added into a flask containing 50 ml metal solution and the solutions were agitated on a rotary shaker at 500 rpm for 3 hours. The samples were then filtered using filter paper (Whatman No 4). The residual concentration of copper in solution was analyzed using Inductively Coupled Plasma Spectrometry (ICP). The Cu(II) sorption capacities were calculated using the following Equation (1):

$$q = (C_i - C_e)\frac{V}{M} \qquad (1)$$

Where: q (mg/g) is the amount of metal adsorbed, C_i (mg/L) is the initial metal concentration, C_e (mg/L) is the residual metal concentration, V (L) is the volume of metal solution and M (g) is the mass of biosorbent used in the reaction mixture (Hashim et al., 2004).

Batch experiments were carried out at pH ranging from 2-9 to determine the effect of pH with a handheld pH meter provided with a combined glass electrode (Jacques et al., 2007).

Effect of initial metal concentration was investigated using initial Cu(II) concentration varied from 50 to 400 mg/L. In experiments concerning the effect of adsorbent dose, a range of algae biomass samples (5, 10, 20, 40, 60 and 80 g/L) were used. For contact time experiments, the procedure was similar with those presented above, with the difference that the phases were separated after a determined period of contact time. All adsorption experiments were performed at room temperature (20 - 23°C).

Table 1. Equations and linear form of kinetic studied here.

Equation	Linear form	kinetic type	References
$\frac{dq_t}{dt} = k_1(q_e - q_t) \quad (2)$	$log(q_e - q_t) = log(q_e) - \frac{k_1}{2.303}t \quad (3)$	Pseudo-first-order model	Lagergren, 1898
$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \quad (4)$	$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t \quad (5)$	Pseudo-second-order model	Ho & Ofomaja, 2006

2.4. Adsorption kinetic study

To understand dynamics of adsorption reactions, information resulted from adsorption kinetic can be examined. Two normal kinetic types studied in this research have been presented in table 1. Experimental kinetic data were conducted at room temperature, adsorbent dose 20 g/L, initial concentration of metals 100 mg/L, pH=5 and contact time (15-180min).

Where, q_e (mg/g) is the amount of copper (II) adsorbed at equilibrium, q_t (mg/g) is the amount adsorbed at time t, k_1 (1/min) is the rate constant of first-order adsorption and k_2 (g/mg.min) is the rate constant of second-order adsorption.

k_1 and k_2 values were determined from the slope and intercept of the plots of $\log(q_e - q_t)$ against t and t/q against t respectively (Fig. 5a and Fig. 5b).

2.5. Adsorption Equilibrium Study

It is important to evaluate the most appropriate correlations for equilibrium curves, to optimize the design of a sorption system. Langmuir and Freundlich isotherm models were used to describe the adsorption equilibrium (Table 2). Experimental isotherm data were conducted at room temperature, equilibrium pH=5, dose of biomass 20 g/L and equilibrium time of 90 minutes for different initial Cu(II) concentrations.

Table 2. Specifications of studied isotherms.

Equation	Linear form	Isotherm type	References
$q_e = \frac{q_{max} K_L C_e}{1 + K_L C_e}$ (6)	$\frac{C_e}{q_e} = \frac{1}{K_L q_{max}} + \frac{C_e}{q_{max}}$ (7)	Longmuir model	Langmuir, 1916
$q_e = K_F C_e^{\frac{1}{n}}$ (8)	$log\, q_e = log\, K_F + \frac{1}{n} log C_e$ (9)	Freundlich model	Freundlich, 1907

Where: q_{max} (mg/g) is the monolayer biosorption capacity of the biosorbent, C_e (mg/L) is the concentration of metal in solution at equilibrium, K_L (L/mg) is the Langmuir biosorption constant relating the free energy of biosorption, K_F $(mg/g.(L/mg)^{1/n})$ is related to the maximum adsorption capacity, and n is a constant that characterized the affinity between adsorbent and metals ions.

Separation Factor (R_L): The Langmuir separation factor (R_L) was applied to estimate either the sorption system favorable ($0 < R_L < 1$), unfavorable ($R_L > 1$), irreversible ($R_L = 0$) or linear ($R_L = 1$).

R_L is calculated from the Langmuir isotherm based equation as follow (Hall et al., 1966):

$$R_L = \frac{1}{1 + K_L C_i} \qquad (10)$$

Where, K_L is the Langmuir equilibrium constant and C_i is the initial metal concentration.

2.6. Analyses of biomass

The *Gigartina acicularis* biomass before and after Cu(II) adsorption were analyzed using Fourier transform infrared spectrometer (FTIR). The infrared spectroscopy (IR) spectra were collected using an AVATAR 360 Thermo Nicolet spectrophotometer within the range 400–4000 cm^{-1}.

The surface structure of The *Gigartina acicularis* biomass before and after Cu(II) adsorption biomass samples were analyzed by energy dispersive X-ray emission (EDX), (EDAX Genisis 2000).

3. Results and Discussion

3.1. Equilibrium Experiments

3.1.1. Effect of pH

The pH is probably the major factor influencing metal biosorbtion process (Esposito et al., 2003; Velkova et al., 2012). The pH of the solution influence both metal binding sites on the cell surface and the chemistry of metal in solution (Dursun, 2006). In order to demonstrate the effect of pH on biosorption capacity, uptake of Cu(II) ions onto *Gigartina acicularis* as a function of pH was studied in the pH ranges of 2 to 9 and showed in Fig. 1.

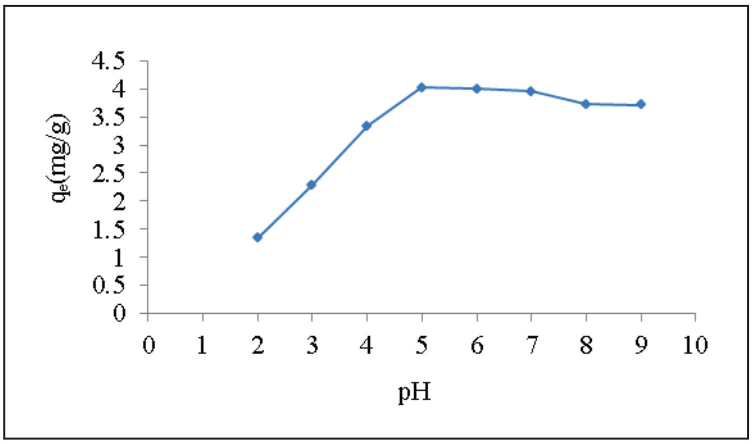

Figure 1. Effect of pH on copper biosorption using dead biomass of alga *Gigartina acicularis*, (initial Cu(II) concentration 100 mg/L, biomass dosage 20 g/L and Contact time of 180 min).

As the pH of the copper solution increased from 2 to 9, the adsorption capacity of Cu(II) increased up to pH 5 and then dramatically decreased. Increased positive charge (H^+) density on the sites of biomass surface at low pH values (2–4) restricted the approach of metal cations as a result of repulsive force. In contrast, when the pH value increased, biomass surface was more negatively charged and the biosorption of the metal ions with positive charge (Cu^{2+}) was reached maximum around pH 5. The decrease in biosorption at higher pH values (pH > 5) may be attributed to the formation of anionic hydroxide complexes of the metal ions and their competition with the active sites. Similar findings were reported for other types of biosorbents (Sari and Tuzen, 2008).

3.1.2. Effect of adsorbent dose

The effect of biomass dosage on Cu (II) biosorption was investigated in the range of 0.25g – 4g as presented in Fig. 2. Results showed a decreased uptake of Cu(II) with the increase of biosorbent dose. This may be due to the fact that some adsorption sites may remain unsaturated during the adsorption process where as the number of sites available for sorption increases by increasing the sorbent dose. This can be attributed to an insufficiency of metal ions in solution with respect to available binding sites (Fourest & Roux, 1992; Babalola et al., 2011).

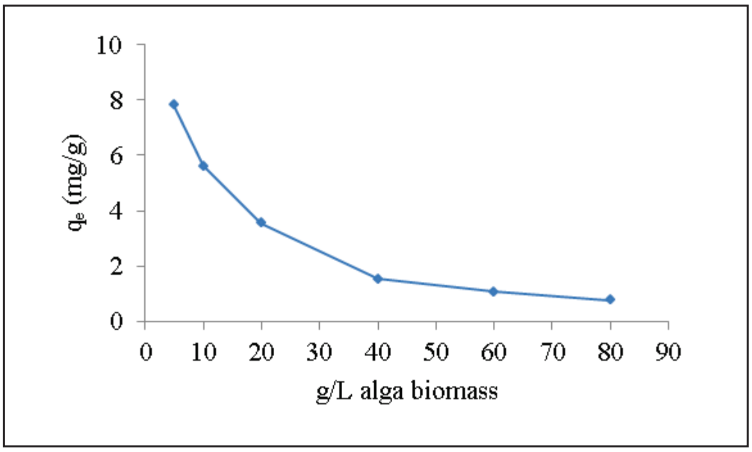

Figure 2. Effect of biosorbent dose on uptake, q_e of Cu(II) on *Gigartina acicularis* (initial Cu(II) concentration 100 mg/L, Contact time of 180 min and pH=5).

3.1.3. Effect of Initial metal Concentration

Effect of initial concentration of Cu(II) on adsorption capacity of *Gigartina acicularis* biomass was investigated by varying initial concentration of Cu(II) from 50 to 400 mg/L. For this study, pH, adsorbent dosage and contact time have been fixed as 5, 20 g/L and 180 min respectively. The results are presented in Fig. 3. The Cu(II) sorption capacity increased with increasing initial metal concentration, an increase of Cu(II) concentration accelerates the diffusion of Cu(II) ions from solution to the adsorbent surface due to the increase in driving force of concentration gradient. Hence, the amount of adsorbed Cu(II) at equilibrium increased from 1.8 to 10 mg/g as the Cu(II) concentration is increased from 50 to 400 mg/L.

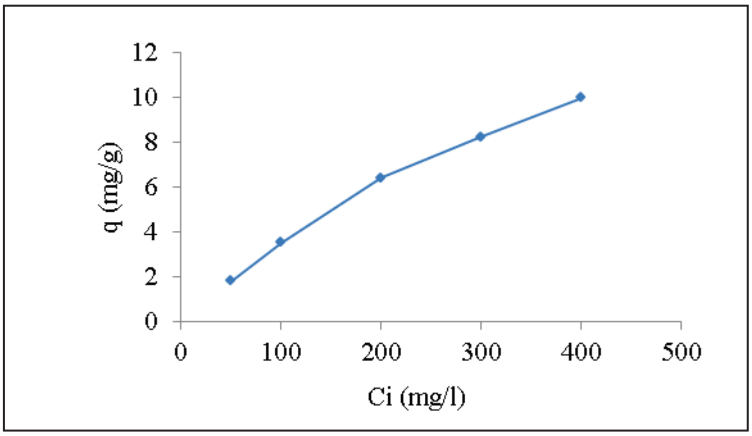

Figure 3. Effect of initial concentration of Cu(II) on adsorption capacity of *Gigartina acicularis* biomass

3.1.4. Effect of Contact Time

Effect of contact time between adsorbent (algae biomass) and a Cu(II) solution with an initial concentration of 100 mg/L, 1g of alga biomass and pH 5.0 at room temperature, on cooper removal is presented in fig. 4. The biosorption process can be divided into two phases. The first rapid phase passed during the first 45 min, followed by second slower phase until the equilibrium was reached at 90 min. The rapid phase is due to the availability of enough free active metal binding sites on the biosorbent surface. A second slow phase is due to decreasing of number of free binding sites.

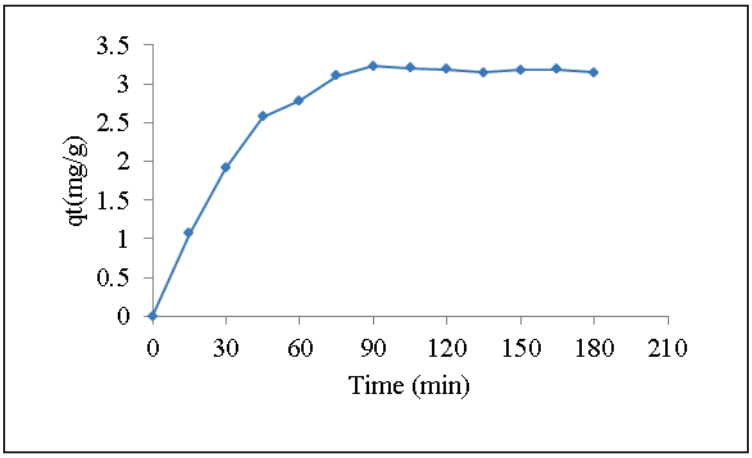

Figure 4. Effect of contact time on Cu (II) adsorption by alga biomass

3.2. Adsorption kinetics

In order to investigate the mechanism of sorption, the rate constants of biosorption process were determined by using Pseudo-First-Order and pseudo-second-order kinetic models (Fig. 5). The values of the first and second order rate constants are listed in Table 3.

Both the values of R^2 and $q_{e,cal}$ are crucial to evaluate how well the data fit the kinetic model.

Pseudo-First-Order Model: As shown in Table 3, the correlation coefficient was 0.74. Nevertheless the calculated adsorption capacity is much lower than experimental data ($q_{e,exp}$), indicating the insufficiency of the pseudo-first-order model.

Pseudo-Second-Order Model: Correlation coefficient presents good agreement at all condition. Moreover, $q_{e,cal}$ was quite similar to $q_{e,exp}$. Therefore, the adsorption behavior of red alga more accurately fit the pseudo-second-order model rather than the pseudo-first-order model. Kumar et al. (Kumar et al., 2006) and Vijayaraghavan et al. (Vijayaraghavan et al., 2004) had the same conclusion in copper removal by green alga.

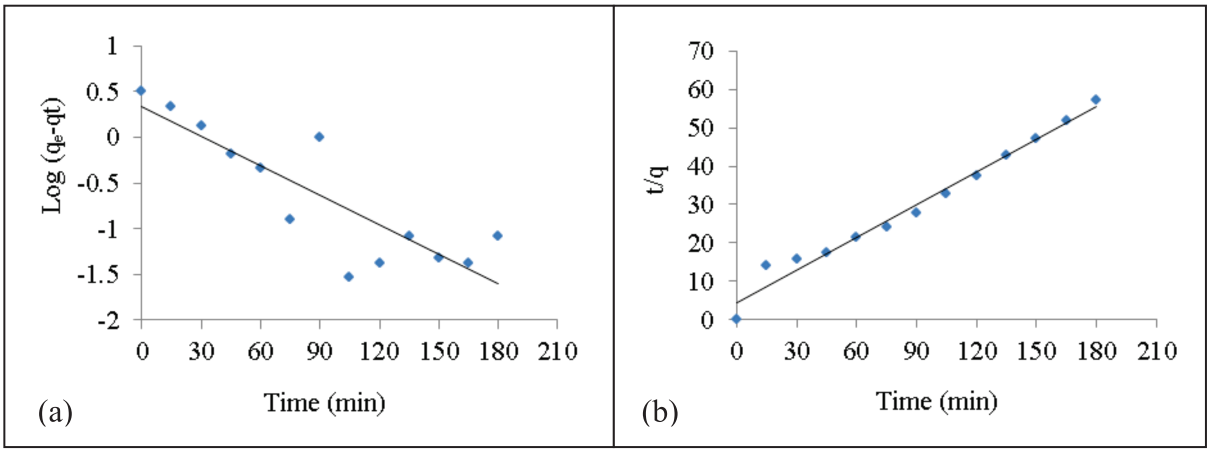

Figure 5. Pseudo-first-order (a) and Pseudo-Second-Order (b) sorption kinetics of Cu(II) onto *Gigartina acicularis*.

Table 3. Comparison of the pseudo-first-order, pseudo-second-order adsorption rate constants and calculated and experimental $q_{e,exp}$ values.

$q_{e,exp}$ (mg/g)	Pseudo-first-order kinetic model			Pseudo-second-order kinetic model		
	$q_{e,cal}$ (mg/g)	k_1 (1/min)	R^2	$q_{e,cal}$ (mg/g)	k_2 (g/(mg.min))	R^2
3.231	2.148	0.025	0.74	3.526	0.021	0.98

3.3. Adsorption isotherms

The graphical representations of Langmuir and Freundlich isotherm models are presented in fig. 6, and the values of isotherm models constants are summarized in table 4. The best fit equilibrium model was determined based on the linear regression correlation coefficient (R^2).

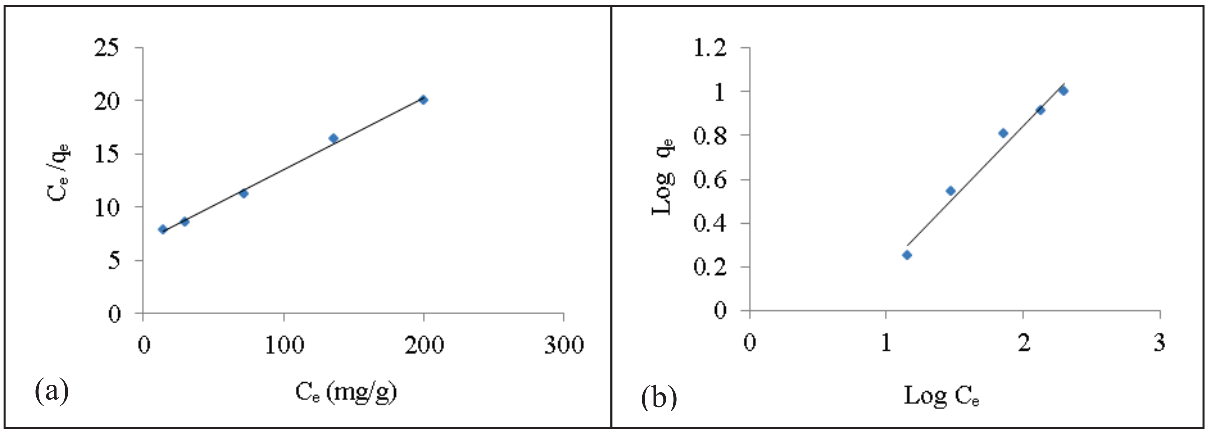

Figure 6. Isotherms Langmuir (a) and Freundlich (b) models

Table 4. Langmuir and Freundlich isotherm parameters

Isotherm type parameters	Longmuir model			Freundlich model		
	q_{max} (mg/g)	K_L (L/mg)	R^2	K_F (mg/g.(L/mg)$^{1/n}$)	n	R^2
	14.77	0.01	0.99	0.36	1.56	0.97

As seen in Table 1, the Langmuir isotherm fits quite well with the experimental data (correlation coefficient R^2 = 0.99), whereas, the low correlation coefficient (R^2 = 0.97) show poor agreement of Freundlich isotherm with the experimental data. The monolayer adsorption capacity according to this model was 14.77 mg/g. The fact that the Langmuir isotherm fits the experimental data very well may be due to homogeneous distribution of active sites onto *Gigartina acicularis* surface, since the Langmuir equation assumes that the surface is homogenous.

The good performance of the Langmuir model can also be found in other studies (Villaescusa et al., 2004; Villaescusa et al., 2000; Yu et al., 2001; Kumar et al., 2006).
The calculated R_L values indicated that Cu(II) sorption were favorable ($0 < R_L < 1$) at all initial metal concentrations (Fig. 7).

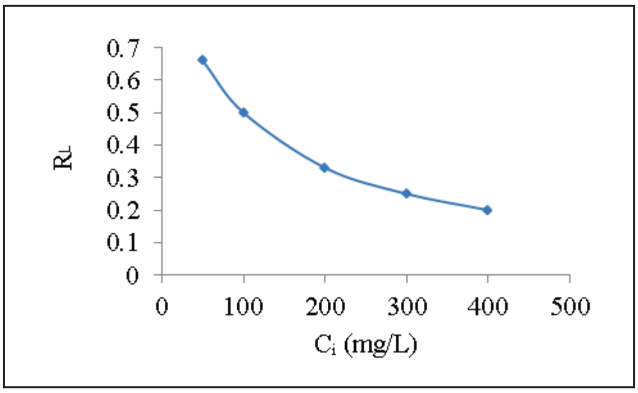

Figure 7. Separation factor profile

3.4. FTIR and EDX analyses

FTIR spectroscopy has been frequently used to detect vibrational frequency changes in seaweeds (Park et al., 2004; Sheng et al., 2004). It is well known that the cell wall composition of algal biomass contains a large number of complex organic components such as proteins, lipids, carbohydrate polymers (cellulose, xylane, mannan, alginic acid) and inorganic ions (Ca^{2+}, Mg^{2+}) (Davis et al., 2003). Numerous chemical groups have been proposed to be responsible for the biosorption of metals by macroalgae.

The most specific peaks of *Gigartina acicularis* before Cu(II) adsorption are the following: 3434 cm^{-1} (–OH and –NH stretching), 2921cm^{-1} and 2851 cm^{-1} (–CH2– stretching), 1637 cm^{-1} (C=O stretching), 1545 cm^{-1} (C=C stretching of aromatic rings), 1461 cm^{-1} (–CH3 bending), 1384 cm^{-1} (Asymmetric –SO3 bending), 1261 cm^{-1} (–C–O stretching of ester) and 1024 cm^{-1} (–C–O stretching of alcohol) and 802 cm^{-1} (S=O stretching).

Table 5 displays the main vibrational wavenumber correspond to functional groups that potentially interacted with Cu(II). The spectrum of the seaweed biomass after Cu(II) adsorption revealed a few changes in the frequencies, suggesting that these functional groups are involved in Cu(II) sorption.

The absorption peaks around 3434 cm^{-1} shifted to 3449 cm^{-1}, 2921 cm^{-1} shifted to 2923 cm^{-1}, 2851 cm^{-1} 1 shifted to 2852 cm^{-1}, 1637 cm^{-1} changed to 1636 cm^{-1}, 1461 cm^{-1} changed to 1462 cm^{-1} and the peak at 1024 cm^{-1} shifted to 1023 cm^{-1}, suggesting that hydroxyl, amino, alkanes groups and carboxyl groups are involved in Cu(II) uptake.

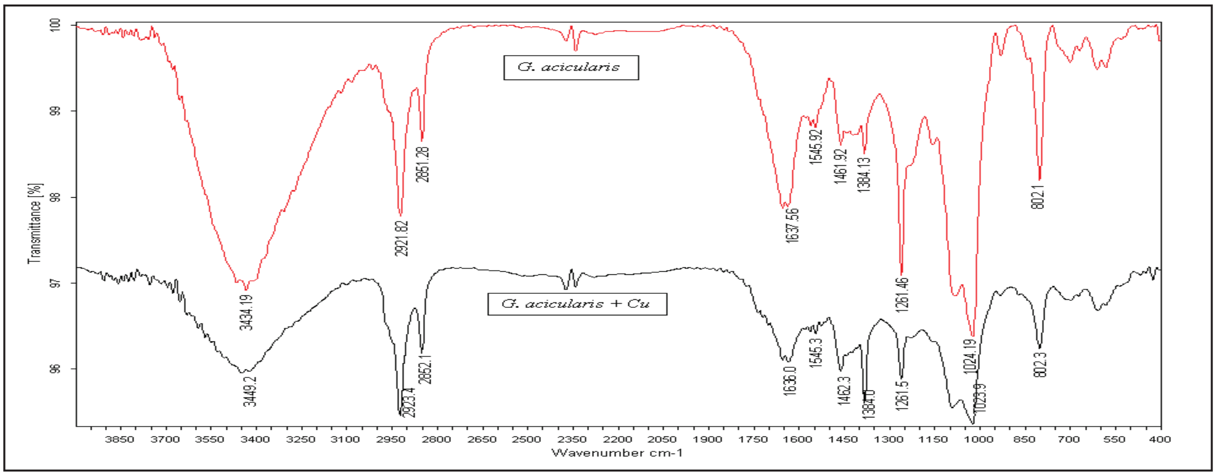

Figure 8. FTIR of *Gigartina acicularis* before and after Cu(II) adsorption.

Table 5. FTIR vibration wavenumber and corresponding functional groups observed on *Gigartina acicularis* biomass

Wavenumber (cm^{-1})		Functional groups
Gigartina acicularis before Cu(II) adsorption	*Gigartina acicularis* after Cu(II) adsorption	
3434	3449	–OH and –NH stretching
2921	2923	–CH2– stretching
2851	2852	–CH2– stretching
1637	1636	C=O stretching
1545	1545	C=C stretching of aromatic rings
1461	1462	–CH3 bending
1384	1384	Asymmetric –SO3 stretching
1261	1261	–C–O stretching of ester
1024	1023	–C–O stretching of alcohol
802	802	S=O stretching

The EDX spectrums of red alga before and after adsorption are shown in Fig. 9. The presence of Cu(II) peak in the spectrum 9b confirms the adsorption of Cu(II) onto alga biomass. It can be seen that copper appeared after adsorption with value 3.64%.

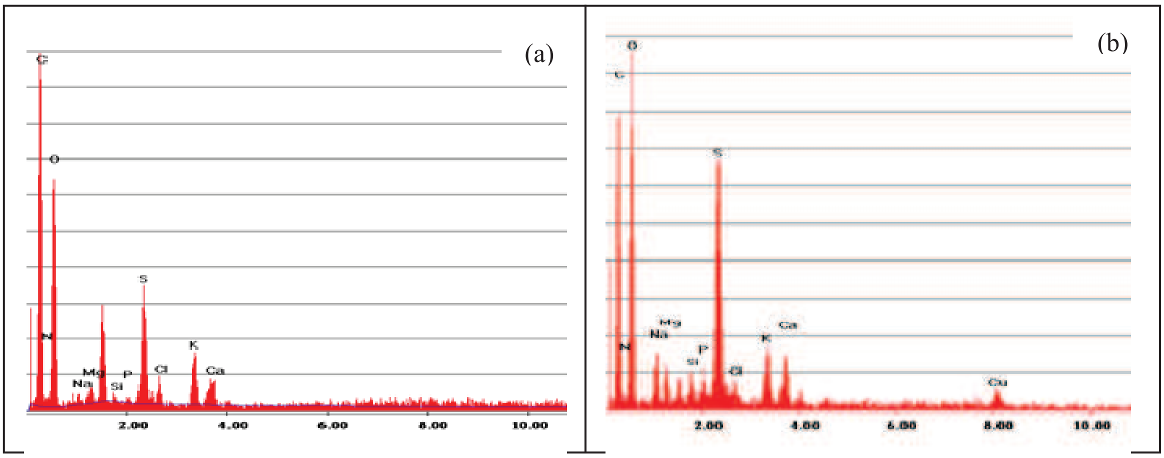

Figure 9. EDX spectrums of *Gigartina acicularis* biomass (a) before and (b) after Cu(II) adsorption.

4. Conclusion

The adsorption of cooper ions from aqueous solution using the red alga (*Gigartina acicularis*) biomass was investigated. Various impact factors such as contact time, initial cooper concentration ions, solution pH and adsorbent dosage were optimized.

Results obtained were modeled using Langmuir and Freundlich isotherm models. Equilibrium isotherms were well described by the Langmuir equation, giving maximum adsorption capacity of 14.77 mg/g at room temperature. The adsorption kinetics can be well described by the pseudo-second-order model equation. A large number of hydroxyl, amino and carboxyl groups were observed on the surface of the alga biomass by FTIR analysis. The application of adsorption of Cu(II) by *Gigartina acicularis* biomass had prove its efficiency in wastewater treatment applications.

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