

Utilization of *Cucumis Melo* Rind as Fe and Pb Ions Biosorbent

N. Othman* and S. Mohd-Asharuddin**

* Mircopollutant Research Centre, Faculty of Civil and Environmental Engineering, University Tun Hussein Onn Malaysia.

(E-mail: norzila@uthm.edu.my)

** Faculty of Civil and Environmental Engineering, University Tun Hussein Onn Malaysia.

(E-mail: hf110212@siswa.uthm.edu.my)

Abstract

The biosorption properties of the *Cucumis melo* (*C.melo*) rind to remove Fe and Pb ions from groundwater solutions were investigated. The effect of different metal optimized conditions (contact time, biosorbent dosage and pH) on metal uptake by *C.melo* were investigated and metal removal were up to 96.83% for Fe and 94.88% for Pb ion under Fe ion optimized conditions. While under Pb ions optimized conditions, the removal percentage obtained were up to 87.19% for Fe and 95.09 for Pb ion after 45 minutes agitation in 100ml of groundwater samples with biosorbent dosage of 0.05 g. Adsorption isotherm analysis data of both metal ions were fitted well to the Langmuir model with maximum adsorption capacity of 5.3505mg/g and 0.0830 mg/g for Fe and Pb ions respectively. The kinetic experimental data correlated well with the pseudo-second order kinetics model. From the characterization study of the biosorbent using SEM-EDX, FTIR and XRF, ion exchange and micro precipitation were estimated to be the main mechanism governing the biosorption process by *C.melo* rind. The results demonstrated that *C.melo* rind has potential to be developed as the adsorbent material for the removal of Fe and Pb ions from groundwater.

Keywords

Biosorption; *Cucumis melo*; Fe; Pb

INTRODUCTION

The rapid industrialization in Malaysia in the recent decade has affected the quality of natural resource including groundwater. The contamination of groundwater by heavy metals was triggered as threats to public health. Though, purification of contaminated groundwater is important since the utilization of groundwater as clean water supply for domestic use as well as agricultural and industrial use is still high especially in areas which are lacking of clean water supply [1].

Heavy metals such as iron and lead that leach through rocks and soils from various sources into groundwater are non-biodegradable and cause adverse effects not only to human, but also animals and plants [2][3]. The presence of these metals in groundwater above a certain level turns the water unusable mainly for esthetic considerations such as discoloration of food and beverages, metallic taste, odor, turbidity, staining of laundry and plumbing fixtures [4]. Apart from that, exposure to high concentration of metal contaminants has been associated with toxicity to affect human organ systems, causing liver damage, gastrointestinal pain as well as reducing immune function [5].

A number of technologies to remove metals in polluted groundwater have been developed including chemical precipitation, coagulation, flocculation, membrane separation, reverse osmosis and oxidation. Major drawbacks of these methods are toxic sludge generation, handling and disposal problems, high cost, technical constraints and incomplete metal removal [6] [7]. Hence, in considering techno-economic constraints, the biosorption method will be employed in this study to remove metal toxicants in groundwater.

Successful metal biosorption has been reported by variety of low-cost agricultural waste. Though, to the best of our knowledge there is no literature describe the potential of *Cucumis melo* (*C.melo*) rind to remove heavy metals in groundwater. The *C.melo* belongs to the family of Cucurbitaceae and is one of the most important fruit crops grown in many tropical countries. The pulp of the fruit is very refreshing and sweet in taste with a pleasant aroma, the intensity of which varies depending

upon the variety [8]. *C.melo* rind is a non-profitable part of the fruit, which is a by-product of fruit juice industries and fruit stalls. Therefore, it is totally inexpensive and available in abundant amount. The rind contains pectin and α -cellulose which provide the function of rigidity and resistance to tearing [9]. Pectin and cellulose are polymers that are rich in functional groups such as hydroxyl and carboxyl which known to strongly bind metal cations in aqueous solution [10]. The present work was conducted to evaluate the potential of *C.melo* rind as a new, low cost biosorbent for treating metal-laden groundwaters. Two sets of optimum working conditions (pH, biosorbent dosage and contact time) obtained from previous study were employed to determine the removal percentage of Fe and Pb from actual groundwater using *C.melo* rind [11]. Experimental data were fitted to isotherm and kinetics models.

MATERIALS AND METHODS

Biosorbent Material

C.melo rind was obtained from fruit stalls around Parit Raja, Batu Pahat, Johor, Malaysia. The rind was thoroughly washed using distilled water and soaked in 15% Nitric Acid for 24 hours. The rind was washed again in distilled water to remove the acid residue. The rind then left in distilled water for another 24 hours. The washed rind was oven-dried at 60°C until it reaches constant weight before ground into powder of <150 μ m particle size using laboratory ball mill.

Groundwater Sampling

A total of 16 groundwater samples were collected by grab sampling within 8 weeks period over dry season from January 2013 – February 2013. Samples of groundwater were collected from groundwater tube well located at Research Centre for Soft Soil, RECESS , UTHM in Parit Raja, Batu Pahat, Johor. All sampling bottles were acid washed prior to sampling. Groundwater was allowed to run for 30 minutes in order to remove stagnant water and allow representative groundwater to be sampled. All sampling methods were done according to APHA [12]. The concentration of the heavy metals in the groundwater was analyzed using AAS.

FTIR, SEM and XRF analysis

Fourier Transform Infra Red (Perkin Elmer Spectrum 100) analysis was conducted to identify the chemical functional groups present on native and metal-loaded *C.melo* rind. The IR adsorbance data were observed in the range of 650 – 4000 cm^{-1} . The surface structure of *C.melo* rind before and after metal adsorption was inspected by scanning electron microscopy (JEOL JSM-6380LA). The chemical composition of *C.melo* rind was studied using X-Ray Fluorescence microscopy (Bruker AXS S4 Pioneer) analysis.

Sorption Studies

The sorption capacity of *C.melo* rind was determined by contacting 0.05 g of the biosorbent with 100 ml of groundwater samples in 250 ml Erlenmeyer flasks. The pH of the solution was adjusted to desired pH value (see Table 1) using 0.1 M Glacial Acetic Acid and 0.1 M NaOH. The suspensions were shaken on an orbital shaker at 125 rpm for 45 minutes at room temperature of 25°C followed by filtration through 0.45 μ m nylon cellulose membrane filters to separate the biosorbent from the metal solution. Residual metal concentration in the filtrate was analyzed using ICP-MS analysis while the biosorbent residue was analyzed using SEM.

All experiments were performed in triplicate.

Table 1. Working conditions for adsorption studies [11]

Treatment	pH	Biosorbent dosage (g)	Contact time (min)
Fe optimized conditions	7.0	0.05	45
Pb optimized conditions	6.5	0.05	45

Data analysis of biosorption efficiency

The metal bound per gram of biomass was determined using the mass balance [13]. The percentage of metal removal and uptake capacity of Fe and Pb ions were determined using Eqs. (1) and (2) :

$$\text{Sorption (\%)} = [(C_0 - C_f) / C_0] \times 100 \quad (1)$$

where C_0 is the initial metal concentration (mg/L) and C_f is the final metal concentration (mg/L).

$$q = (C_0 - C_f) (V / M) \quad (2)$$

where q is the metal uptake (mg/g), C_0 is the initial metal concentrations in solution (mg/l), C_f is the final metal concentrations in solution (mg/l), V is the volume of solution (l), and m is the mass of biosorbent (g).

RESULTS AND DISCUSSION

SEM-EDX analysis

The micrographs of SEM shows the progressive changes observed on the surface of the biosorbent before and after the sorption of the metal ions. The surface of native *C.melo* rind was observed to be porous and rough which shows a sign of good candidate for potential biosorbent (see Fig. 1(a)) [14]. Larger and deepened pores were observed in acid-treated biosorbent (see Fig.1 (b)) which proven that the advantage of acid pretreatment in improving the quality of biosorbent by enlarging pores which increase the surface area for metal attachment [15]. Shiny particles were spotted on the smoothed surface of metal-loaded biosorbent (see Fig. 2).



Figure. 1. SEM micrographs of native *C.melo* rind (a) before acid treatment (b) after acid treatment



Figure 2. SEM micrographs of *C.melo* rind after sorption study

3.2. XRF analysis

The chemical composition of *C.melo* rind was presented in Table 2. The XRF analysis of the acid-treated biosorbent shows no element of heavy metal of interest present in the biosorbent. SiO₂ was found to be the most abundant component contains in the biosorbent. This finding was in parallel with the observation reported in a study of metal biosorption using rice husk. The major constituents of rice husk which may be responsible for sorption are carbon and silica [16]. While in other study done by Azizul-Rahman [17] on the adsorption of Zn by watermelon rind revealed that silica content was significantly reduced from 75.20% to 9.88% which reflects the involvement of silica in metal sorption. This finding could be due to the leaching of Si from the surface of the adsorbent material during the process of biosorption via redox reaction which forming metal oxides or due to substitution of metal ions at Si-OH bonds. Besides, Rao [18] suggested that when the substituted metal ions has a lower charge, the net negative charge at the site enables adsorption of cations thus the presence of Si-OH bonds would also act as cation exchange centers.

Table 2. Elemental composition of *C.melo* rind

Formula	Constitution (%)	
	Untreated biosorbent	Acid-treated biosorbent
CO ₂	0.10%	0.10%
SiO ₂	38.81%	41.23%
K ₂ O	18.90%	16.00%
SO ₃	5.39%	15.50%
CaO	14.00%	11.10%
P ₂ O ₅	10.40%	6.90%
Al ₂ O ₃	1.27%	4.28%
Cl	3.25%	1.59%
TiO ₂	1.25%	1.00%
MgO	2.39%	0.88%
CuO	1.01%	0.83%
ZnO	1.23%	0.59%
Fe ₂ O ₃	1.93%	-
Mn	0 <LLD	-

FTIR analysis

The pattern of metal sorption onto the sorbent material is attributable to the functional groups present of the surface of the sorbent material. Fig. 4(a-b) shows the FTIR spectra of *C.melo* rind before and after the biosorption study. There were shifting of wavenumber observed after the sorption study conducted. The broad peak between 3500-3200 cm⁻¹ can be attributed to the -OH groups of alcohols, phenols and carboxylic acids. The peak between 3000-2850 cm⁻¹ indicates symmetric or asymmetric CH or CH₂ stretching vibration of aliphatic acids [19]. While the peak between 1750-1680 cm⁻¹ may be assigned to the stretching vibration of C=O of carboxyl groups. Whereas the peak between 1640-1500 cm⁻¹ is may be due to the stretching vibration of ionic carboxylic groups (-COO⁻) [20].

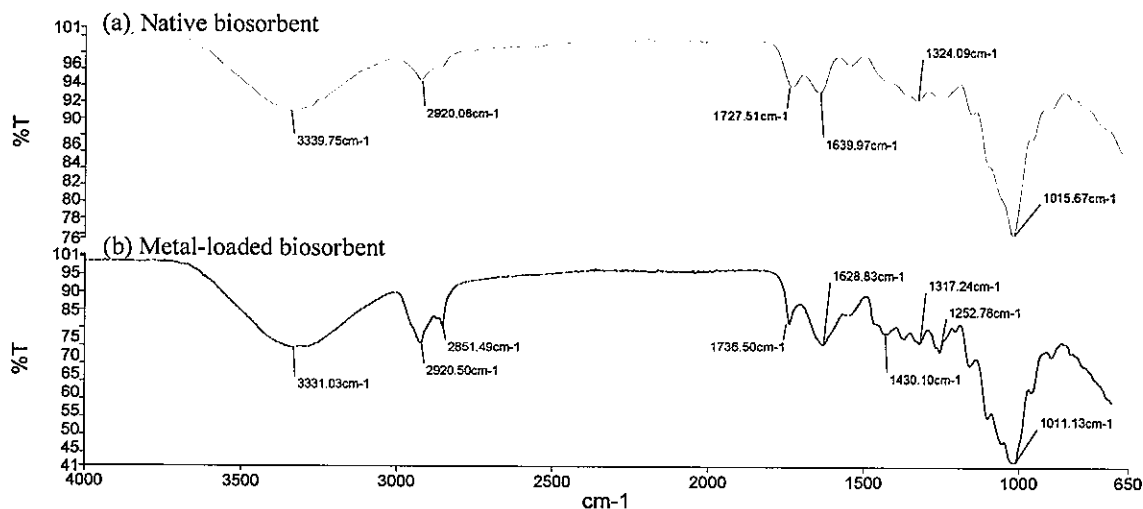


Figure 4. FTIR spectra of (a) native biosorbent, (b) metal- loaded biosorbent

Sorption studies

The analysis result of initial Fe, Mn and Pb concentrations in the groundwater samples were slightly fluctuated but no significant difference was found in Fe and Pb ions concentration between all samples collected ($p > 0.05$). The metals concentrations remains generally exceed the acceptable level by World Health Organization (WHO) [21] and Malaysia National Guidelines for Raw Drinking Water Quality (MNGRDWQ) (Benchmark for Groundwater) [22] (Table 3). Same observation was demonstrated by Azimah [23] on a study of metal contents in groundwater of RECESS monitoring well during 2012 where Fe and Pb were also found to be the highest metal contains in the groundwater. The observation is in agreement with the result demonstrated by Chin [24] on a study of metal contents in groundwater in Manukan Island, Sabah where the contents of Fe (2.538 mg/g) and Pb (0.590 mg/g) violated the WHO limit.

Table 3. Initial metal concentration of groundwater samples

Sample	Ferum Concentration (mg/l)	Lead Concentration (mg/l)
1 (1/1/2013)	2.335 ± 0.002	0.237 ± 0.002
2 (4/1/2013)	3.43 ± 0.001	0.155 ± 0.001
3 (8/1/2013)	3.148 ± 0.001	0.152 ± 0.001
4 (11/1/2013)	2.119 ± 0.001	0.216 ± 0.001
5 (15/1/2013)	3.67 ± 0.001	0.105 ± 0.001
6 (18/1/2013)	2.163 ± 0.001	0.213 ± 0.001
7 (22/1/2013)	3.263 ± 0.002	0.131 ± 0.001
8 (25/1/2013)	2.416 ± 0.001	0.137 ± 0.001
9 (29/1/2013)	3.293 ± 0.003	0.118 ± 0.001
10 (1/2/2013)	3.099 ± 0.001	0.122 ± 0.002
11 (5/2/2013)	2.968 ± 0.003	0.093 ± 0.002
12 (8/2/2013)	2.324 ± 0.003	0.073 ± 0.003

Table 3 (Continued)

13 (13/2/2013)	3.045 ± 0.003	0.228 ± 0.002
14 (15/2/2013)	3.341 ± 0.001	0.196 ± 0.004
15 (19/2/2013)	3.734 ± 0.001	0.178 ± 0.002
16 (22/2/2013)	3.128 ± 0.001	0.224 ± 0.001
Minimum Metal Concentration	2.119	0.073
Maximum Metal Concentration	3.734	0.237
Mean	2.967	0.161
WHO limits	2.000	0.010
NGRDWQ (benchmark for groundwater)	0.300	0.010

After sorption study was done under Fe-optimized conditions (pH: 7, biosorbent dosage: 0.05g, contact time: 45 minutes), the average removal percentage of Fe and Pb were 90.73% and 88.36% respectively (Figure 5). From the results, removal of Fe and Pb under Fe optimized conditions shows no significant difference ($p > 0.05$) in percentage removal compare to batch study reported by previous study [11]. This indicates that the application of Fe ion optimized-condition is also suitable for the removal of Pb from the groundwater.

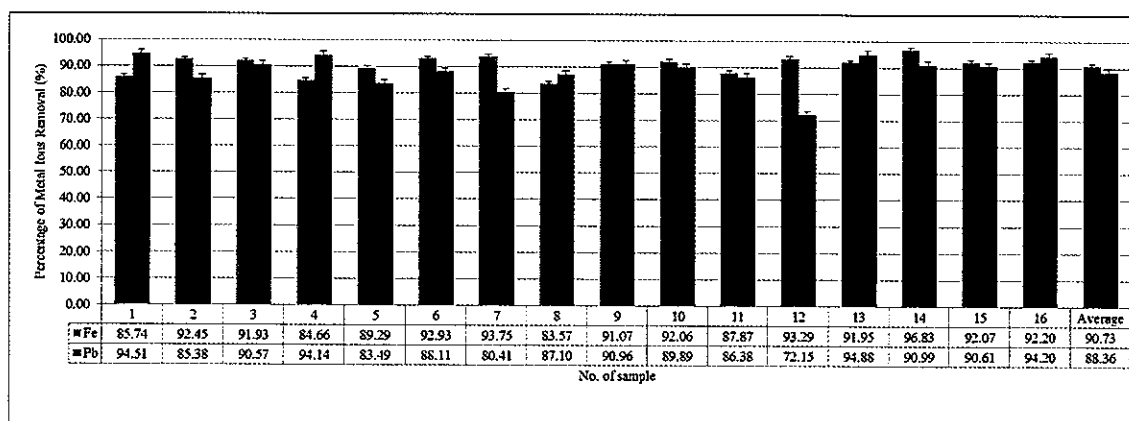


Figure 5. Removal of Fe and Pb ions under optimum condition of Fe ion (pH: 7.0, biosorbent amount: 0.05 g, and contact time: 45 minutes) (n=3)

In another sorption study conducted under Pb optimized conditions (pH: 6.5, biosorbent dosage: 0.05g, contact time: 45 minutes), the average removal percentage were 90.94% and 82.44% respectively for Pb and Fe (Figure 6). In comparison to the result obtained from batch study using synthetic groundwater, the removal percentage shows no significant difference ($p > 0.05$) [11]. So it can be concluded that the optimized working condition of Pb are also suitable for the removal of Fe ions in the groundwater. From the results, it was observed that *C.melo* rind have successfully reduced the concentration of Fe (0.50 mg/l) and Pb (0.01 mg/l) to achieve the standards recommended by WHO.

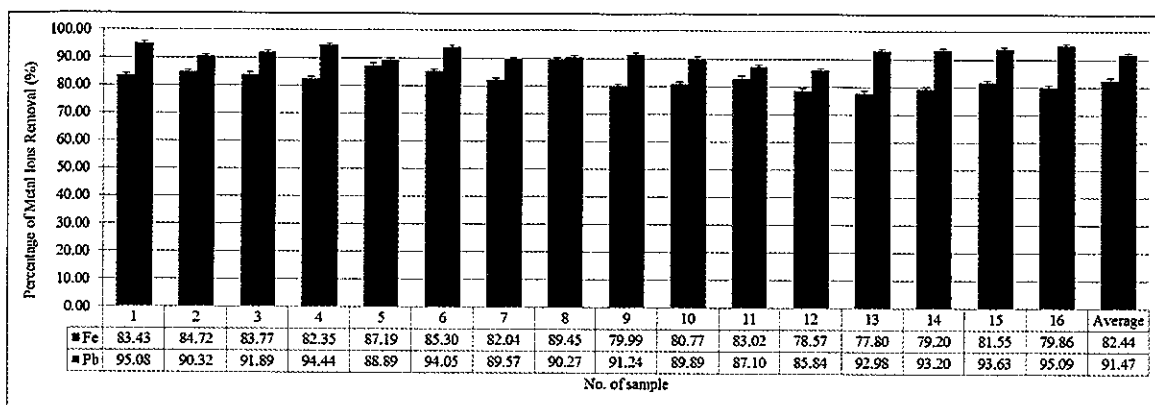


Figure 6. Removal of Fe and Lead ions under optimum condition of Pb ion (pH: 6.5, biosorbent amount: 0.05 g, and contact time: 45 minutes) (n=3)

The metal selectivity in the removal of metal by *C.melo* rind can be arranged in the order of Fe > Pb based on the average metal uptakes. Such trends could be explained in terms of the difference in the ionic sizes of the metal ions, the rate and distribution of active groups on the sorbent and the mode of interaction between metal ions and the sorbent [25]. The ionic radii (Pauling) for Fe²⁺ is 0.78 Å and Pb²⁺ is 1.33 Å. It is suggested that the smaller the ionic diameter, the higher adsorption rate [26].

Moreover, the sorption of metal ions in the presence of other common ions may be affected due to competition for the sorption sites. When the adsorption is particle diffusion controlled, it means that intraparticle mass transfer resistance is rate limiting. Therefore, in the presence of more than one metal ions, the metal ions compete for the adsorption sites on the adsorbent. This competition affects the diffusion properties of the metal ions, hence decreases the adsorption capacity of the metal ions. Thus, the metal ion that successfully reaches the adsorption site faster depends on the above factors and also on the ionic radii of the metal ions. A biosorption study using *Delonix regia* (Forest pods) on the removal of nickel and lead demonstrated that there is reduction of metal uptake of lead and nickel by the biomass in the presence of other competing cations such as magnesium and sodium [27]. Chatterjee and Schiewer study the effect of competing cations in biosorption using citrus peels. The study revealed that metal uptake was lower for bimetal systems than for mono-metal systems. The biosorbent showed higher affinity towards Pb which displaced previously bound Cd from the biosorbent [28].

Metal sorption kinetics

In order to analyze the biosorption kinetics of heavy metal ions, the pseudo-first and pseudo-second order kinetics models were applied to the kinetics data from previous batch experiment study [11]. Lagergren's first-order equation is among the most widely accepted for the sorption of a solute from liquid solution [29]

and represented as:

$$\log(q_e - q_t) = \log q_e - \frac{k_1 t}{2.303}$$

where q_e and q_t are the amounts of biosorbed (mg g^{-1}) at equilibrium and at time t , respectively, and k_1 is the rate constant of pseudo-first-order biosorption (min^{-1}). The q_e and rate constants k_1 were calculated from the slope and intercept of the plot of $\log(q_e - q_t)$ vs. t (see Fig. 7(a-b)). Pseudo-first order considers the rate of occupation of sorption sites to be proportional to the number of unoccupied sites. A straight line of $\ln(q_e - q_t)$ versus t indicates application of the pseudo-first order kinetics model.

While pseudo-second order equation [30] are based on the sorption equation which expressed as:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$$

where k_2 is the rate constant of pseudo-second-order biosorption ($\text{gmg}^{-1} \text{min}^{-1}$). The pseudo-second-order rate constant k_2 and q_e values were calculated from the slope and intercept of the plots t/q vs. t .

The results of the kinetics parameters for both Fe and Pb ions were calculated from the linear plots of pseudo-first order and pseudo-second order kinetics models are presented in Table 4. The theoretical q_{eq} values calculated from the pseudo-first order kinetics models for sorption of both metal ions gave significantly different values than that of q_{exp} values, and the R^2 values for both metal ions were also found to be lower. The R^2 regarded as a measure of the goodness-of-fit of experimental data on the kinetics model [30]. These results indicate that the biosorption of Fe and Pb ions by *C.melo* rind do not fit the first-order kinetic model.

The linear plots of t/qt against t for second-order equation result in straight lines for both metal ions (see Fig.8 (a-b)). The R^2 values for both metal ions were also near to 1(0.99). The q_{eq} values for both metal ions tested were also very close to the q_{exp} values. All these results implies that the biosorption of Fe and Pb ions by *C.melo* rind followed the pseudo-second order kinetic model which assumes that biosorption may be the rate-limiting step involving valence forces through sharing or exchange of electrons between biosorbent and sorbate [30].

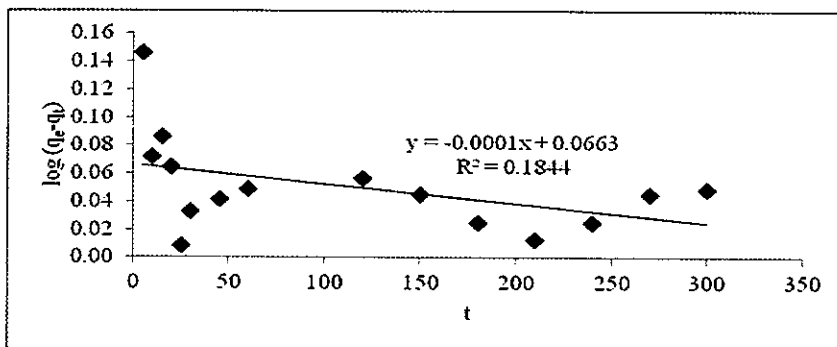


Figure 7 (a). Linearized pseudo-first order kinetic model of Fe ion uptake by *C.melo* rind

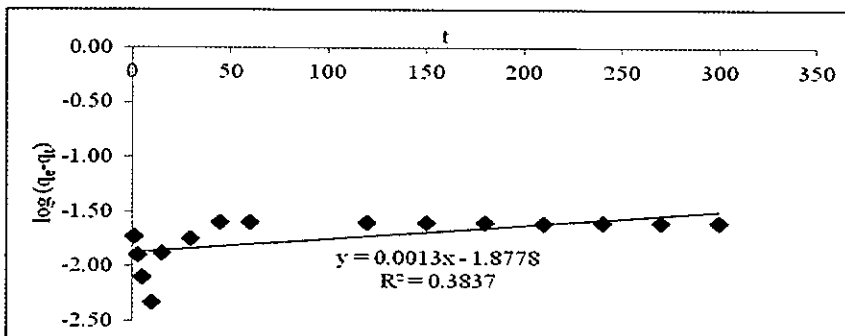


Figure 7 (b). Linearized pseudo-first order kinetic model of Pb ion uptake by *C.melo* rind

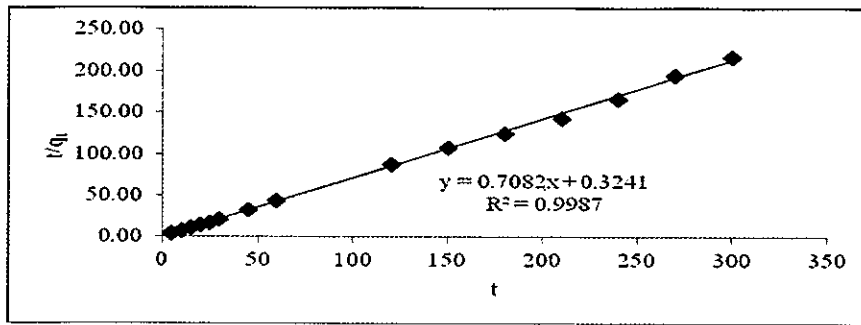


Figure 8 (a). Pseudo second-order kinetic model of Fe ion uptake by *C.melo* rind

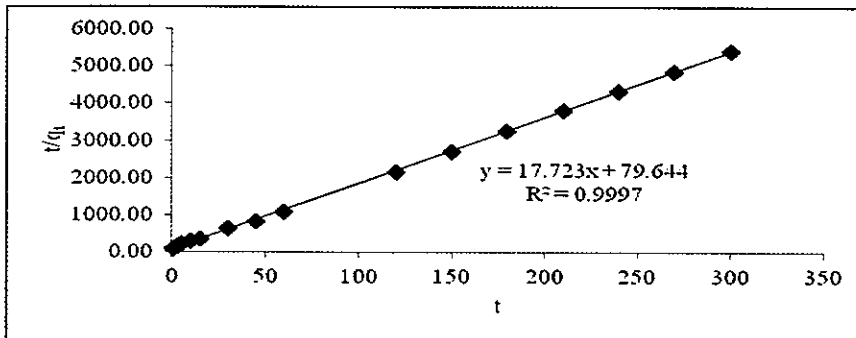


Figure 8 (b). Pseudo second-order kinetic model of Pb ion uptake by *C.melo* rind

Table 4. Kinetics parameters of biosorption of Fe and Pb ions by *C.melo* rind

Metal ions	q_e (exp.) (mg g ⁻¹)	Pseudo-first order			Pseudo-second order		
		q_e (cal.) (mg g ⁻¹)	k_1 (min ⁻¹)	R^2	q_e (cal.) (mg g ⁻¹)	k_2 (min ⁻¹)	R^2
Fe	1.4800	1.1649	0.0002	0.1844	1.4120	1.5476	0.9987
Pb	0.0550	0.8584	-0.0029	0.3837	0.0564	3.9472	0.9997

Metal sorption isotherms

To examine the relationship between the metal sorption capacity (q_{eq}) and the concentration of metal ions at equilibrium (C_{eq}), the experimental data from isotherm studies [11] of both metal ions were fitted to widely used adsorption isotherm models namely Langmuir, Freundlich and Brunauer, Emmett and Teller (BET). These isotherm models can be easily transformed into linear forms to obtain isotherm parameters by linear regression analysis [31]. The Langmuir isotherm model assume monolayer adsorption on the binding sites of the biosorbent, and is expressed by the following linearized equation [32]:

$$\frac{C_e}{q_e} = \frac{1}{q_{max}b} + \frac{C_e}{q_{max}}$$

Where where q_{max} is monolayer capacity of the biosorbent (mg g⁻¹), and b is the biosorption constant (L mg⁻¹). The plot of C_e/q_e versus C_e should be a straight line with slope $1/q_{max}$ and intercept $1/q_{max}b$ when the biosorption follows the Langmuir equation.

The Freundlich isotherm can be described by [33]:

$$\log q_e = \log K_f + \frac{1}{n} \log C_e$$

where K_F and $1/n$ are Freundlich isotherm constants related to biosorption capacity and intensity of biosorption, respectively.

The BET isotherm can be represented by the following equation [34]:

$$\frac{C_e}{(C_i - C_e)q_m} = \frac{1}{K_B \cdot q_m} + \left[\frac{K_B - 1}{K_B \cdot q_m} \right] \left[\frac{C_e}{C_i} \right]$$

Where C_i is equilibrium concentration of metal ions in solution (mg/L), C_e is final concentration of metal ions in solution at equilibrium (mg/L) while K_B is BET constant denoting energy interaction with the surface (mg/g).

The isothermal plots for all isotherm models for biosorption of Fe and Pb ions by *C.melo* rind are presented in Fig. 9-11. The data were perfectly fitted to Langmuir isotherm which yielded regression coefficient (R^2) value near to 1 for both metal (0.99 for Fe and 0.98 for Pb).

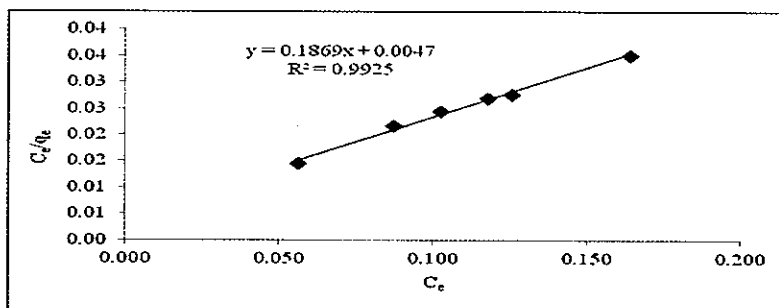


Figure 9(a). Langmuir isotherms of Fe ion uptake by *C.melo* rind

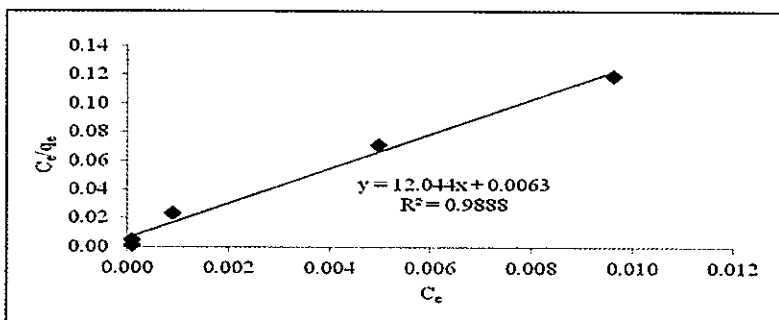


Figure 9(b). Langmuir isotherms of Pb ion uptake by *C.melo* rind

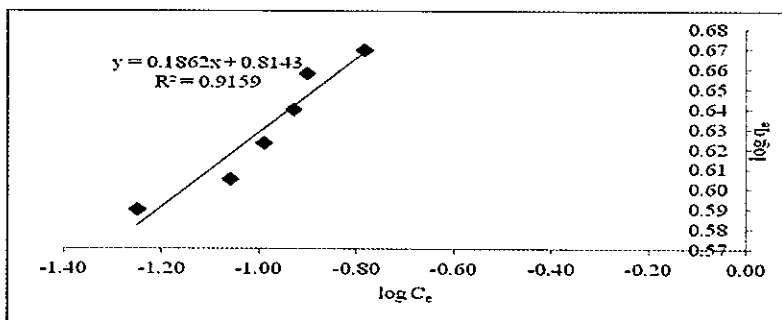


Figure 10(a). Freundlich isotherms of Fe ion uptake by *C.melo* rind

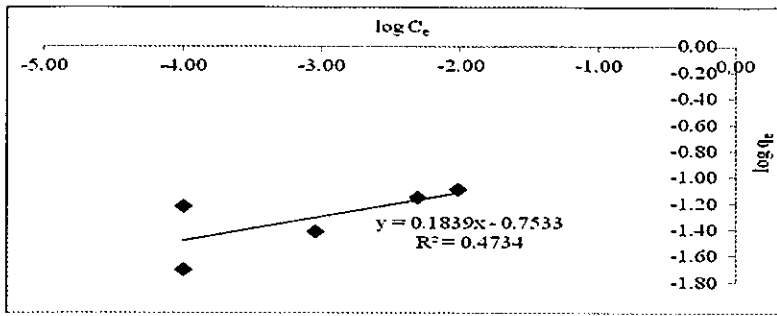


Figure 10(b). Freundlich isotherms of Pb ion uptake by *C.melo* rind

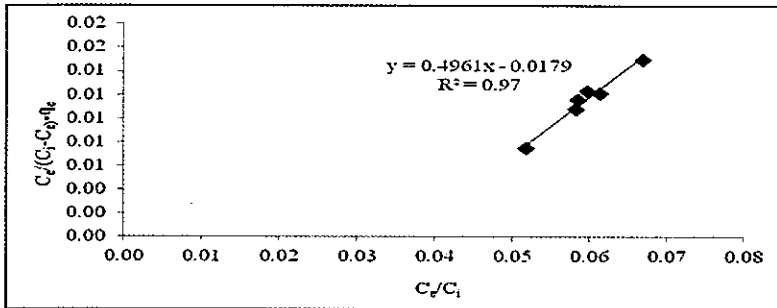


Figure 11(a). Brunauer-Emmett-Teller (BET) isotherms of Fe ion uptake by *C.melo* rind

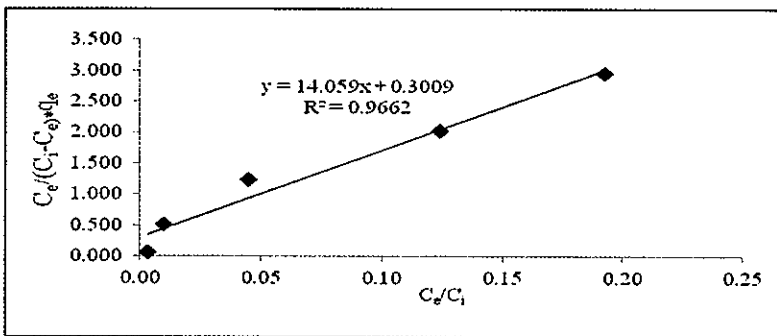


Figure 11(b). Brunauer-Emmett-Teller (BET) isotherms of Pb ion uptake by *C.melo* rind

The isotherms parameters are listed in Table 5. The sorption capacity, q_{\max} which is a measure of maximum sorption capacity corresponding to complete monolayer coverage showed that the maximum metal uptake for Fe and Pb ions were 5.3505 mg/g and 0.0830 mg/g respectively. The data conclude that the affinity of metal ions to bind to *C.melo* rind in the aqueous solutions was following the order of Fe > Pb.

Table 5. Isotherms parameters of biosorption of Fe and Pb ions onto *C.melo* rind

Metal ions	Langmuir			Freundlich			BET		
	q_{\max} (mgg ⁻¹)	b (lmg ⁻¹)	R ²	K _F	n	R ²	q_m (mgg ⁻¹)	K _B	R ²
Fe	5.3505	39.7656	0.9925	6.5208	5.3706	0.9159	2.0912	-26.7151	0.9700
Pb	0.0830	1912.4116	0.9888	0.1765	5.4377	0.4734	0.0711	46.7232	0.9662

Another essential characteristic of the Langmuir isotherm parameters can be used to predict the affinity between the sorbate and sorbent is using separation factor of dimensionless equilibrium parameter, R_L , which is expressed by the following equation:

$$R_L = \frac{1}{1 + bC_0}$$

Where b is the Langmuir constant and C_0 is the highest initial concentration of metal ions (mg/L). The value of R_L provides the important indication whether the isotherm is either unfavourable ($R_L > 1$), linear ($R_L = 1$), favourable ($0 < R_L < 1$) or irreversible ($R_L = 0$). Sorption of Fe and Pb ions by *C.melo* rind yields the R_L values of 0.009 mg/L and 0.011 mg/L respectively which indicating favourable biosorption process.

CONCLUSION

C.melo rind can be a promising low cost adsorbent material for the removal of Fe and Pb ions from groundwater. The data of experiments were identified to fit to the Langmuir isotherm with maximum adsorption capacity of 5.3505 mg/g and 0.0830 mg/g respectively for Fe and Pb ions. The R_L is less than 1 indicating of favourable sorption process. The kinetics study showed the biosorption of metal ions by *C.melo* rind follow the Pseudo-second order kinetics model. Based on the XRF analysis, it was identified that silica, SiO₂ is the most abundance constituent in the biosorbent. The surface of the native biosorbent was observed to be porous under the SEM. After the sorption process, new shiny particles were observed over the surface of the biosorbent. While the FTIR spectra showed broad spectrum of hydroxyl and carboxyl groups in the native biosorbent and detected shifting of wavenumbers in metal-loaded biosorbent which suggested ion exchange or chemisorption mechanism might involve in the biosorption process. The *C.melo* rind successfully removes up to 90.73%, 91.47% and 90.94% respectively to Fe and Pb in the groundwater samples which reduce the metals level below the WHO recommended limits of heavy metal for drinking water. Thus it is concluded that the *C.melo* rind has the potential to be employed as an inexpensive and eco-friendly biosorbent to remove metals in groundwater.

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