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RESEARCH ARTICLE

Sequestering of iron and lead from groundwater using chemically modified *Cucumis melo* rind

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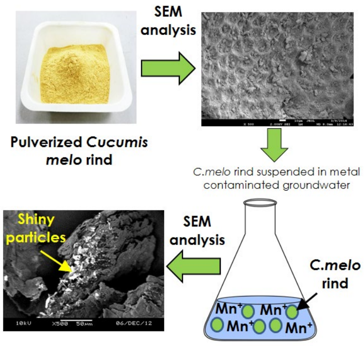
Graphical abstract



Abstract

Heavy metal contamination in groundwater is among the significant environmental issues which require high priority of remediation action due to its importance in our daily life. This study intended to upcycle *Cucumis melo* (*C. melo*) rind which a waste from agricultural industries as natural adsorbent to remove iron (Fe) and lead (Pb) ions from groundwater. The efficiency of *C. melo* rind to remove Fe and Pb under pre-determined optimum conditions were analyzed and the result showed that metal removal was up to 96.83 % for Fe and 94.88 % for Pb ions under Fe ion-optimized working conditions. While under Pb ion-optimized working conditions, the removal percentage obtained were up to 87.19 % for Fe and 95.09 % for Pb ion. Adsorption isotherm analysis data of both metal ions were fitted well to the Langmuir model with maximum adsorption capacity of 5.35 mg/g and 0.08 mg/g for Fe and Pb ions respectively. The kinetics experimental data correlated well with the pseudo-second order kinetics model. From the characterization study of the biosorbent using scanning electron microscopy coupled with energy dispersive X-ray spectrometry (SEM-EDX), X-ray fluorescent spectrophotometry (XRF) and Fourier transform infrared spectrometry (FTIR), 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 the potential to be developed as the adsorbent material for the removal of Fe and Pb ions from groundwater.

Keywords**:** Biosorption, biosorbent, cucumis melo, groundwater, Fe ion, Pb ion

INTRODUCTION

Metals such as iron and lead that are seeping into the environment naturally through rock and soils can cause adverse effect to human, flora, and fauna. The existence of the metals in groundwater beyond a certain level will turn the groundwater unfeasible for consumable. The metals polluted water may cause discoloration of food and beverages, metallic taste, odor, turbidity, staining of laundry, and plumbing fixtures (Mohamad Roslan *et al*., 2007; El-Araby *et al*., 2009; Abdul Aziz *et al*., 2017). Lengthen exposure to high concentration of metal contaminants may specifically cause organ system dysfunction that result in deficiency of growth and reproduction as well as neurological disorders (Hamilton *et al*., 2000).

Extensive number of researches has been done in exploring and improvising technologies for metals removal in polluted groundwater including chemical precipitation, coagulation, flocculation, membrane separation, reverse osmosis, and oxidation. Most of the methods demonstrate major shortcomings such as toxic sludge generation, chemical handling and disposal problems, high maintenance cost, technical constraints, and incomplete metal removal (Valdman *et al*., 2000; Hashim *et al*., 2011). Hence, in considering techno-economic constraints, the biosorption method was employed in this study to remove metal toxicants in groundwater.

Successful metal biosorption has been reported by numerous researches using variety of low-cost agricultural waste ranging from fruit seeds, husks, copra, leaves, and fibres (Othman *et al*., 2013; Othman *et al*., 2014). Though, to the best of our knowledge there is no literature that describes the feasibility of pre-treated *Cucumis melo* (*C. melo*) rind for heavy metals removal in groundwater. *C. melo* belongs to the family of Cucurbitaceae, is a tropical fruit that is best planted on soil temperature between 25 °C to 32 °C. In Malaysia, melons including *C. melo* are mostly planted in Johor, Kedah, Kelantan, Terengganu, and Pahang. The cultivation of these crops covers almost 7.1 % from the total agro food land in Malaysia which equivalent to 13, 814 hectares (Rasmuna *et al*., 2016). The 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 abundantly available.

The principal compounds present in the *C. melo* rind, pectin and α-cellulose provide the function of rigidity and resistance to tearing of this fruit (Mohd-Asharuddin *et al*., 2017). These polymers are rich in functional groups such as hydroxyl and carboxyl which are known to strongly bind to metal cations in aqueous solution (Mohd-Asharuddin *et al*., 2019).

The detailed batch optimization studies have been published (Othman *et al*., 2013). In the present work, the pre-determined working conditions as obtained in the previous work were employed to determine the removal percentage of Fe and Pb ions from groundwater samples using *C. melo* rind. Isotherm and kinetics studies were also conducted, and the experimental data was fitted to isotherm and kinetics model to obtain in-depth understanding on the adsorption process.

EXPERIMENTAL

Biosorbent materials

C. melo rind used in this study was obtained from various fruit stalls around Parit Raja, Batu Pahat, Johor. After the rind was thoroughly cleaned using distilled water to remove unwanted impurities, it was soaked in 15 % Nitric Acid. The rind was washed again in distilled water to remove the acid residue followed by oven-drying at 60 °C until it reached constant weight before ground into powder of <150 µm particle size using laboratory ball mill (Zayadi *et al*., 2013).

Preparation of metal solutions

Stock solution (1000 mg/L) of Fe and Pb ion solutions were prepared by dissolving the appropriate amount of $FeCl_3.6H_2O$ and $Pb(NO_3)_2$ in 100 mL pure water. Varied concentrations of metallic ion solutions (2.0 to 2.5 mg/L for Fe ion; 0.01 to 0.05 mg/L for Pb ion) were prepared by diluting the stock solution. The pH adjustment of the solutions was made using 0.1 M Glacial Acetic Acid and 0.1 M NaOH, utilizing a pH hand-held pH meter (Hach). Synthetic metal ion solutions were prepared for kinetics and isotherm studies.

Groundwater sampling

Samples of groundwater were collected from groundwater tube well located at Research Centre for Soft Soil, RECESS, UTHM in Parit Raja, Batu Pahat, Johor. The sampling process was done according to APHA (1998). The concentration of the heavy metals in the groundwater was analyzed using Perkin-Elmer Atomic Adsorption Spectrometer (AAS).

Sorption studies using actual groundwater samples

The working conditions applied to the present experiment as tabulated in Table 1 were obtained from the optimum working conditions based on the previous study on the removal of Fe and Pb ions using *C. melo* rind in synthetic aqueous solutions (Othman *et al*., 2013). The sorption experiments were carried out by using suspension of 0.05 g of *C. melo* rind in 100 mL of metallic solutions of various concentrations in 250 mL Erlenmeyer flasks. The suspensions were shaken on an orbital shaker at 125 rpm for 45 minutes at room temperature of 25 °C. The pH of the groundwater samples were adjusted to 6.5 and 7.0. The biosorbent was separated from the groundwater solution by glass filtration set. Residual metal concentration in the filtrate was determined using AAS analysis while the biosorbent residue was analyzed using SEM-EDX and FTIR. All experiments were performed in triplicates.

Kinetics studies

Kinetics study were carried out in batch at optimum conditions as listed in Table 1 and varied contact times (1, 3, 5, 10, 15, 20, 30, 45, 60, 120, 150, 180, 210, 240, 270, 300 min). The samples were immediately filtered after each time intervals and the residual metal concentration in the filtrate was analyzed using AAS. The data collected were modeled using Pseudo-first order and Pseudo-second order kinetics models.

Isotherm studies

The adsorption isotherm study was performed using predetermined optimum conditions with a series of Fe and Pb ion solutions containing different metal ion concentrations for each ion (in the range of 2.0–2.5 mg/L for Fe ion solutions; 0.01–0.05 mg/L for Pb ion solutions). All suspensions were filtered and metal residues in the filtrates were analyzed using AAS. The data collected were modeled using Langmuir, Freundlich, and BET isotherm models.

Biosorbent characterization using SEM-EDX, XRF, and FTIR analysis

Native and metal-loaded biosorbents were subjected to SEM-EDX microscopy (JEOL) for surface morphological and elemental analyses. Elemental compositions of the biosorbent were analyzed using XRF (Perkin Elmer) while chemical functional groups present on native and metal-loaded *C. melo* rind were characterized using FTIR (Perkin Elmer).

Data analysis of biosorption efficiency

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

$$Sorption\ (\%) = [(C0 - Cf) / C0] \times 100 \tag{1}$$

where C0 is the initial metal concentration (mg/L) and Cf is the final metal concentration (mg/L).

$$q = (C0 - Cf)\ (V / m) \tag{2}$$

where q is the metal uptake (mg/g), C0 is the initial metal concentrations in solution (mg/L), Cf 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

Sorption studies using actual groundwater

The adsorption study using groundwater samples were performed by employing the pre-determined optimum working conditions for both Fe and Pb ions as listed in Table 1 to evaluate the effect of different working conditions on metal selectivity of *C. melo* rind due to the fact of groundwater contains multi-ion compositions. The minimum and maximum Fe and Pb ions concentration in the groundwater samples are presented in Table 2. The metals concentrations generally exceed the acceptable level by World Health Organization (WHO, 2004) and Malaysia National Guidelines for Raw Drinking Water Quality (Benchmark for Groundwater) (MOH, 2000). Similar observation was demonstrated by Azimah *et al.* (2012) on a study of metal contents in groundwater of RECESS monitoring well in 2012 where Fe and Pb ionic concentrations were found to be the highest in the groundwater.

Table 1 Percentage of removal and uptake capacity of all parameters at optimum working conditions (Othman *et al*., 2013).

Metal ions	Optimum value				Percentage Removal (%)	Optimum Uptake Capacity (mg/g)
	pH	Contact time (minute)	Initial Metal Ion Concentration (mg/L)	Biosorbent Dosage (g)		
Fe	7.0	45	2.0	0.05	99.60	4.98
Pb	6.5	45	0.03	0.05	94.44	0.05

This observation is in agreement with the result reported by Nurazim *et al.* (2015) on a study of metal contents in pumping well near Sungai Kerian, Perak where the contents of Fe^{2+} (6.220 mg/L) violated the WHO limit.

Table 2 Fe and Pb concentration of RECESS groundwater samples.

Sample	Fe Concentration (mg/L)	Pb Concentration (mg/L)
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 ion-optimized conditions (pH: 7, biosorbent dosage: 0.05 g, contact time: 45 minutes), the average removal percentage of Fe and Pb ions were 90.73% and 88.36%, respectively. In another sorption study using Pb ion-optimized conditions (pH: 6.5, biosorbent dosage: 0.05 g, contact time: 45 minutes), the respective average removal percentage were 90.94 % and 82.44 % for Pb and Fe ions. Such removals have successfully reduced the concentration of Fe (0.50 mg/L) and Pb (0.01 mg/L) ions to achieve the standards recommended by WHO.

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 trend 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 (A-Jacques *et al*., 2007). The ionic radius (Pauling) for Fe ion is 0.78 Å while for Pb ion is 1.33 Å. It is suggested that the smaller the ionic diameter, the higher adsorption rate (Gerola *et al*., 2013).

Moreover, the presence of other competing ions may affect the metal uptake due to competition for the sorption sites on the surface of adsorbent. When the adsorption is particle diffusion controlled, it means that intraparticle mass transfer resistance is rate limiting (Yesim *et al*., 2000; Igwe *et al*., 2007). The competition between ionic species 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 including the ionic radii of the metal ions.

An interference study was performed using *Delonix regia* (Forest pods) on metal uptake with the presence of competing ions. The results indicated reduction in the biomass uptake of lead and nickel with the presence of competing ions of calcium, sodium and magnesium (Festus *et al*., 2013). Chatterjee and Schiewer (2014) study on the effect of competing cations of Pb and Cd in biosorption using citrus peels revealed that metal uptake was lower for bimetal systems than for mono-metal systems. The biosorbent showed higher affinity towards Pb than Cd that could be due to the surface adsorption on constituent group on substrates which plays a more vital contribution to adsorption capacity than the micro porous adsorption (Igwe *et al*., 2007).

Metal sorption kinetics

The results of the kinetics parameters for both Fe and Pb ions calculated from the linear plots of pseudo-first order and pseudo-second order kinetics models are presented in Table 3. 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 is regarded as a measure of the goodness-of-fit of experimental data on the kinetics model (Ho, 2004). These results indicate that the biosorption of Fe and Pb ions by *C. melo* rind do not follow 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 (Fig. 1 and 2). The R^2 values for both metal ions were also close 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 imply 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 (Khamidun *et al*., 2014).

Metal sorption isotherm

The data from the isotherm studies 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 ions) as presented in Fig. 3 and 4. The applicability of the Langmuir isotherm for the experimental data provides an initial indication that the governing mechanism is chemisorption with high possibility of monolayer adsorption of the metal ions which are occurring on the surface of the *C. melo* rind. Langmuir isotherm assumes that adsorption occurs at specific sites within the adsorbent (Langmuir, 1916). Therefore, once the metal ion occupying a site no further adsorption will take place at that site (Volesky *et al*., 1995).

The isotherms parameters are listed in Table 4. The sorption capacity, qmax 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 5.35 mg/g and 0.08 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.

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 Eq. 3:

$$R_L = \frac{1}{1+bC_0} \tag{3}$$

where b is the Langmuir constant and C_o 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) (Zeng *et al*., 2009). Sorption of both Fe and Pb ions by *C. melo* rind yields the RL values within the range of 0–1 which indicating favorable biosorption process.

Table 3 Kinetics parameters of biosorption of Fe and Pb by *C. melo* rind.

Metal ions	q_e (exp.) (mg/g)	Pseudo-first order			Pseudo-second order		
		q_e (cal.) (mg/g)	k_1 (min^{-1})	R^2	q_e(cal.) (mg/g)	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

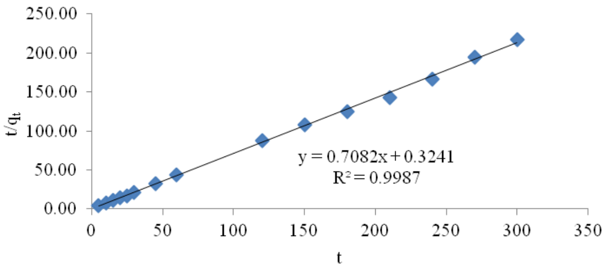


Fig. 1 Pseudo second-order kinetic model plot of Fe uptake by *C. melo* rind.

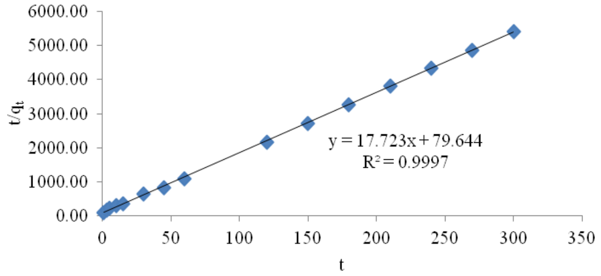


Fig. 2 Pseudo second-order kinetic model plot of Pb uptake by *C. melo* rind.

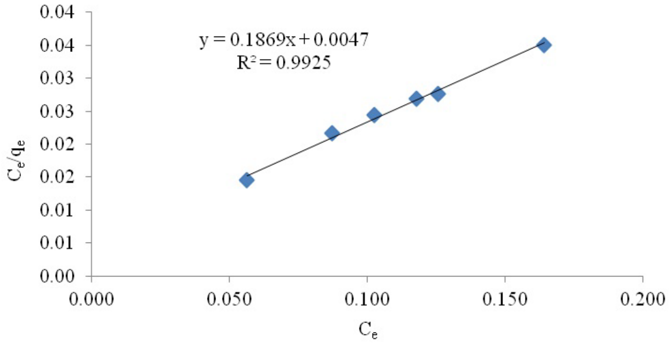


Fig. 3 Langmuir isotherm plot of Fe ion uptake by *C. melo* rind.

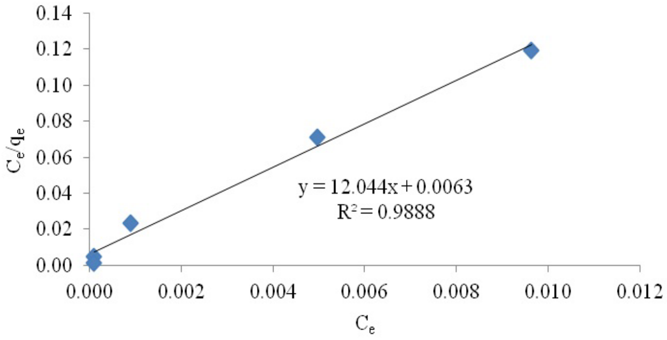


Fig. 4 Langmuir isotherm plot of Pb ion uptake by *C. melo* rind.

Table 4 Isotherms parameters of biosorption of Fe and Pb ions onto *C. melo* rind.

Metal ion	Langmuir			Freundlich			BET		
	q_{max} (mg/g)	b (L/mg)	R^2	K_F	n	R^2	q_m (mg/g)	K_B	R^2
Fe	5.35	39.76	0.99	6.52	5.37	0.92	2.09	-26.71	0.97
Pb	0.08	1912.41	0.99	0.18	5.44	0.47	0.07	46.72	0.97

SEM-EDX analysis

The surface of native *C. melo* rind was observed to be porous and rough as seen in Fig. 5. This shows a sign of good candidate for potential biosorbent. Larger and deepened pores were observed in acid-treated biosorbent (Fig. 6) which proven that the advantage of acid pretreatment in improving the quality of biosorbent by enlarging pores and consequently increasing the surface area for metal attachment (Noor-Syuhadah *et al*., 2012).

SEM micrographs of metal-loaded biosorbent were observed to be smoother than that of porous native biosorbent. There were shiny particles observed over the surface of biosorbent after the biosorption study was conducted. This observation evidenced the surface coverage of biosorbent by metal ions. The EDX analysis performed on the untreated biosorbent identified the presence of prominent C and O peaks and did not show the characteristics signal of Fe and Pb. The analysis conducted on the shiny spots revealed new peaks between 0.00 to 3.00 keV corresponding to Fe and Pb ions confirming the uptake of metals by the biosorbent.

XRF analysis

The chemical composition of *C. melo* rind is presented in Table 5. From the XRF analysis, it was also noted that the contents of several compound such as SiO_2, SO_3, and Al_2O_3 in acid-treated biosorbent were increased compared to untreated biosorbent. This could be due to pore enlargement which adding more surface area to the sorbent material which therefore contributing higher exposure of those elements content on the surface of the sorbent (Gerola *et al*., 2013). SiO_2 was found to be the most abundant component in the biosorbent. This finding is 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 (Noor-Syuhadah *et al*., 2012). While in other study done by Azizul-Rahman (2014) 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 forms metal oxides or due to substitution of metal ions at Si-OH bonds. Besides, Rao *et al*. (2010) 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.

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. 9 shows the FTIR spectra of *C. melo* rind before and after the biosorption study. The band shifts demonstrated in the FTIR spectra of metal-loaded biosorbent confirmed changes in functional group and surface properties of the biosorbent. These shifts may be attributed to the changes in counter ions associated with hydroxyl, carboxyl groups which are predominant contributors in the complexation of metal ions and ion exchange processes (Iqbal *et al*., 2010; Mohd-Asharuddin *et al*., 2017). *C. melo* rind is principally made up of lignin and cellulose which bear functional groups such as alcohol, ketone, and carboxyl. These groups can be involved in complexation reaction with Fe and Pb ions.

The changes observed in the band between 3500–3200 cm^{-1} may be assigned to complexation of metal ions with the ionized O-H groups of free hydroxyl groups and bonded O-H group in polymeric compounds such as alcohols, phenols and carboxylic acids presented in pectin, cellulose and lignin on *C. melo* rind. The changes in the band between 3000–2850 cm^{-1} may indicate the mechanism of ion exchange that took place between protons of symmetric or asymmetric CH and CH_2 of aliphatic acids (Iqbal *et al*., 2009). While the peak between 1750–1680 cm^{-1} may be assigned to the stretching vibration of C=O of carboxyl groups. The peak between 1640–1500 cm^{-1} is may be due to the stretching vibration of ionic carboxylic groups ($-COO^-$) of cellulose, which also may be indicative of electrostatic forces of attraction between negative charge of carboxylate anion and positive charge of Fe and Pb ions (Gerola *et al*., 2013). These observations are consistent with the study performed by Kamaruudzaman *et al*. (2013) where –OH and –C=O functional groups were reported to be involved in the biosorption of Pb ions.

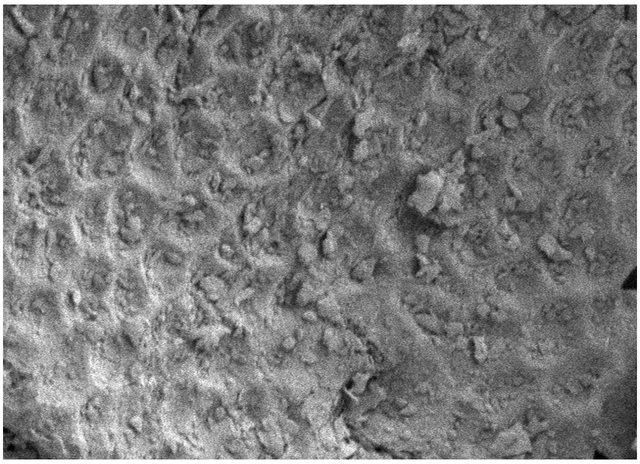


Fig. 5 SEM micrograph of native *C. melo* rind (×500).

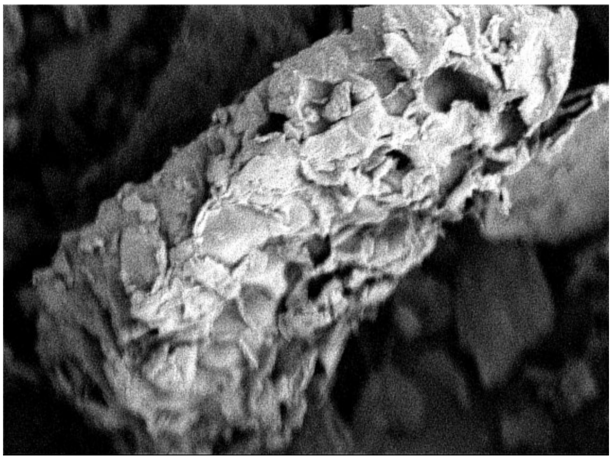


Fig. 6 SEM micrograph of acid-modified *C. melo* rind (×500).

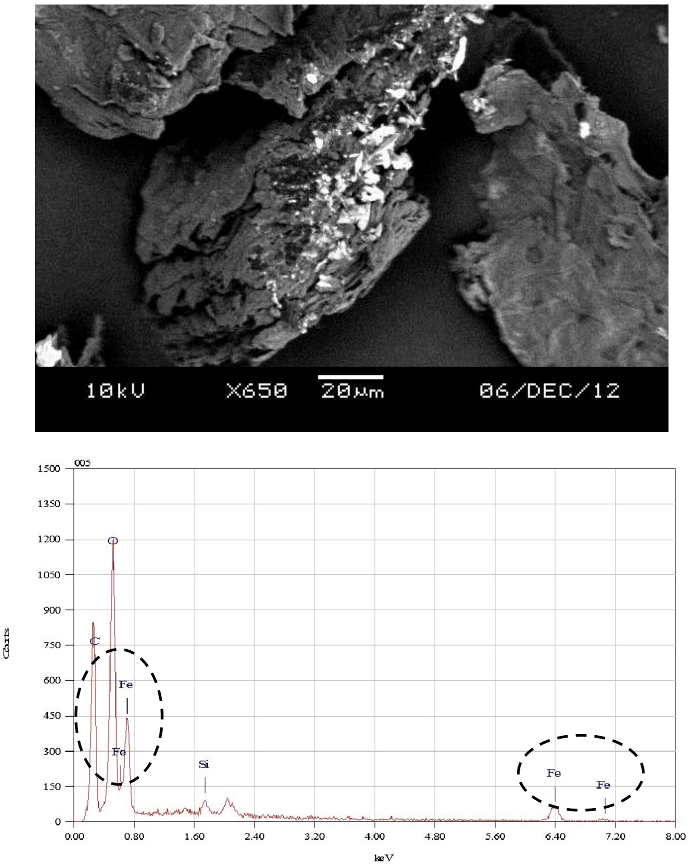


Fig. 7 SEM-EDX micrograph of *C. melo* rind after Fe sorption study (×500).

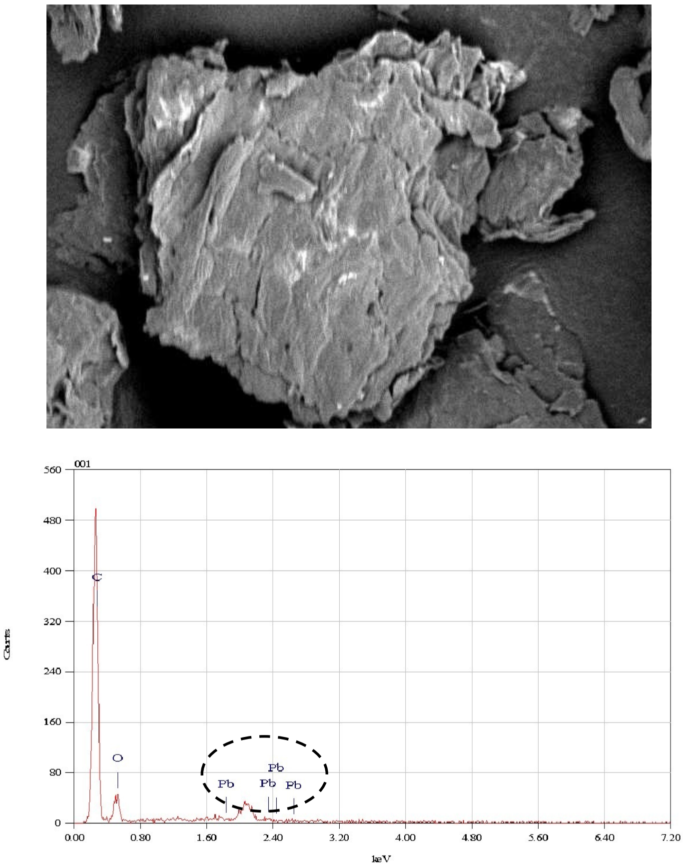


Fig. 8 SEM-EDX micrograph of *C. melo* rind after Pb sorption study (×500)

Table 5 The elemental composition of the *C. melo* rind.

Formula	Constitution (%)	
	Native biosorbent	Acid-treated biosorbent
CO_2	0.10%	0.10%
SiO_2	38.81%	41.23%
K_2O	18.90%	16.00%
SO_3	5.39%	15.50%
CaO	14.00%	11.10%
P_2O_5	10.40%	6.90%
Al_2O_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_2O_3	1.93%	-
Mn	0 <LLD	-

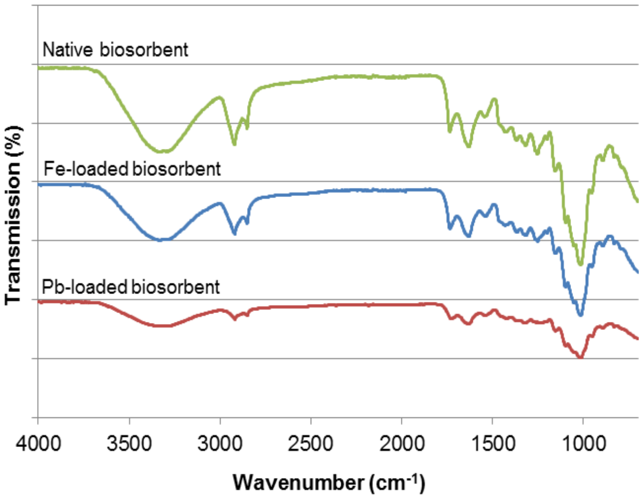


Fig. 9 FTIR spectra of native biosorbent, Fe- loaded biosorbent and Pb-loaded biosorbent.

CONCLUSION

C. melo rind can be a promising low-cost adsorbent material for the removal of Fe and Pb ions from groundwater. The sorption study showed that *C. melo* rind successfully removes Fe and Pb ions in the groundwater samples up to 90.73 % and 90.94 %, respectively, to a level below the WHO recommended limits of heavy metal for drinking water. The data were identified to fit to the Langmuir isotherm while kinetics study showed the biosorption of metal ions by *C. melo* rind follows the Pseudo-second order kinetics model. Based on the XRF analysis, it was identified that silica, SiO_2 is the most abundant constituent in the biosorbent. The surface of the acid-modified biosorbent was observed to be more porous under the SEM. After the sorption process, new shiny particles were observed over the surface of the biosorbent. 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 may be involve in the biosorption process. From the findings discovered in this research, it is concluded that the *C. melo* rind has the potential to be further explored and developed as an inexpensive and eco-friendly biosorbent to remove metals in groundwater.

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