Adsorption of Cd(II), Cu(II) and Ni(II) ions by *Lemna minor* L.: Effect of physicochemical environment

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Abstract

The free floating macrophyte *Lemna minor* L. was harvested locally. Untreated, acid pretreated (H_2SO_4), alkali pretreated (NaOH) biomass were used for adsorption of copper, cadmium and nickel ions from aqueous solutions. The effect of initial pH, initial metal concentration and multi metal interaction were carried out in a batch system. The equilibrium adsorption was reached within 40–60 min. The Langmuir and Freundlich models were used for describing of adsorption isotherm data. The maximum adsorption capacities of alkali pretreated biomass were determined as 83, 69 and 59 mg g^{-1} for the Cd(II), Cu(II) and Ni(II) ions, respectively. The pseudo first- and second-order intraparticle diffusion models were used to describe the adsorption kinetics. The experimental data fitted to pseudo second-order kinetic. Adsorption capacity decreased with acid pretreatment; however alkali pretreatment was not affected significantly adsorption capacity and adsorption capacity a little increased according to native biomass. The FT-IR results of *Lemna* biomass showed that biomass has different functional groups and these functional groups are able to react with metal ions in aqueous solution.
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1. Introduction

Toxic heavy metal contamination of industrial wastewater is an important environmental problem. Many industries, such as automotive, metal finishing, electroplating, battery manufacturing, mining, electric cable manufacturing, tannery, steel and textile industries, release various concentrations of heavy metals like cadmium, nickel and copper, etc. in wastewaters. These heavy metals are toxic to aquatic ecosystem and human health and these heavy metals also accumulate by organism in tolerance levels.

Many physicochemical methods have been developed for heavy metal removal from aqueous solution, including chemical coagulation, adsorption, extraction, ion-exchange and membrane separation process [1–3]. Most of these methods have been used in industrial wastewater treatment process. But these techniques have significant disadvantages including incomplete metal removal, requirements for expensive equipment and monitoring systems, high reagent or energy requirements or generation of toxic sludge or other waste products that require disposal. For dilute metal concentrations, ion exchange, reverse osmosis and adsorption can be applied. However, ion exchange and reverse osmosis have high operating cost [4].

Adsorption is an alternative technique for heavy metal removal. The activated carbon is the most widely used adsorbent material. In fact, use of activated carbon can be expensive for regeneration required and at lose of the application processes. Many researchers have been investigating new adsorbent materials for alternative to activated carbon, such as seaweeds [5], marine algae [6], clays [7], activated sludge biomass [8,9], perlite [10], maple sawdust [11], etc. for the removal of heavy metal from wastewater.

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In this research, adsorption ability of *Lemna minor* was investigated for removal of cadmium, copper and nickel ions from aqueous solution. The effect of initial metal concentrations, initial pH, multi metal interactions for native (untreated), acid and alkali pretreated biomass were examined. Biomass characterization was determined with FT-IR analysis. Langmuir and Freundlich adsorption isotherms were applied to the experimental data. The pseudo first-order, pseudo second-order and intraparticle diffusion model were used for determining of the adsorption kinetics.

1.1. Equilibrium modelling

The adsorption isotherms generally used for the design of adsorption system. The Langmuir [12] and Freundlich [13] adsorption isotherm models were used for describing the adsorption isotherm. The Langmuir and Freundlich equations are represented as follows Eqs. (1) and (2), respectively:

$$\frac{C_e}{q_e} = \frac{1}{k_L} + \left(\frac{a_L}{k_L}\right) C_e \tag{1}$$

$$\ln q_e = \frac{1}{n} \ln C_e + \ln k_F \tag{2}$$

where, a_L and k_L are the Langmuir isotherm constants, k_F the Freundlich constant and n is the Freundlich exponent. The ratio of k_L/a_L gives the theoretical monolayer saturation capacity of biomass (q_{max}).

1.2. Kinetic modelling

The pseudo first-order model [14], pseudo second-order model [15] and intraparticle diffusion model [16] were used to fit the experimental data.

The pseudo first-order model of Lagergren is given as:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \tag{3}$$

The pseudo second-order model is expressed as:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \tag{4}$$

The intraparticle diffusion equation can be described as:

$$q_t = k_i t^{0.5} \tag{5}$$

where k_1 is the rate constant of pseudo first-order model (min^{-1}), k_2 (g mg^{-1} min) the rate constant of pseudo second-order model and k_i is the intraparticle diffusion rate constant (mg g^{-1} min$^{0.5}$). q_e and q_t are amounts of metal adsorbed on adsorbent (mg g^{-1}) at equilibrium and at time t, respectively.

2. Materials and methods

2.1. Preparation of biomass

Lemna minor samples were supplied from Adana, Turkey. The biomass was washed with distilled water and than dried at 60 °C until constant weight and stored at room temperature. Acid and alkali pretreatment was carried out as follows: a weighed amount of dried biomass samples was treated with 0.01 M H$_2$SO$_4$ or 0.1 M NaOH solution (200 ml) for 30 min under slow stirring. The samples were washed with distilled water until removing excess H$^+$ and OH$^-$ ions. The biomass was dried at 60 °C until constant weight. Dried biomass was broken into pieces and was separated into certain particle sizes (0.12–0.5 mm) by sieve and it was used for adsorption experiments.

2.2. Adsorption studies

The adsorption of metal ions on *Lemna* biomass was studied by batch technique. Adsorption studies were conducted in 250 mL screw top flasks using 100 mL of biomass–metal solutions. The general method used for this study is described as follows: a 0.1 g dried biomass was contacted with 80 mL of distilled water before contacting of metal ions. Twenty millilitres of known concentration of metal solution was contacted with biomass–distilled water solution and the mixture was agitated on a shaker at 150 rpm. Samples were taken at time intervals for the analysis of residual metal concentration in solution.

Adsorption isotherms were studied at 20 °C and 60 min. Effect of the initial pH was studied at 2–6 pH ranges, 20 °C and 100 mg L^{-1} initial metal concentrations. The multi metal adsorption studies were conducted at pH 4, 20 °C and 1 mM initial metal concentrations of Cd(II), Cu(II) and Ni(II) ions. The solution pH was adjusted by dilute H$_2$SO$_4$ or NaOH at the beginning of the experiment and not controlled afterwards. The Cd(II) and Cu(II) stock solutions were prepared by nitrate salts and Ni(II) stock solution was prepared than chloride salt dissolving in distilled water.

The biomass–metal mixture was filtered for analysis of unadsorbed metal ions in the mixture. Analysis was made by using Hitachi 180-80 polarized Zeeman atomic absorption spectrophotometer with air–acetylene flame.

Metal uptake (q_e) was calculated using the following equation:

$$q_e = \frac{(C_0 - C_t) V}{1000 W} \tag{6}$$

where q_e (mg g^{-1}) is the amount of total adsorbed heavy metal ions, C_0 (mg L^{-1}) the initial metal concentration, C_t (mg g^{-1}) the equilibrium metal concentration in solution at time t, V (L) the solution volume and W (g) is the adsorbent weight.

Control samples were prepared from the biomass free solution for testing metal losses due to metal precipitation

and adsorption of bottle surface. All experiments were run at least in duplicate. Adsorbed metal concentrations were the means of the duplicate experimental results.

2.3. FT-IR analysis

The FT-IR analysis was made for detecting vibration frequency changes of native, acid and alkali pretreated biomass. KBr pellet were prepared and the proportion of biomass/KBr is 1/100. The background was obtained from the scan of pure KBr. Perkin-Elmer spectrum RX/FT-IR system was used for FT-IR analysis of biomass.

3. Results and discussion

3.1. FT-IR analysis

The cell wall compositions of algal and plant biomass have containing large number of complex organic components and there are proteins, lipids, carbohydrate polymers (cellulose, xylane, mannan, etc.) and inorganic ions Ca^{2+}, Mg^{2+}, etc. The carboxylic and phosphate groups in the cell wall are acidic functional groups of biomass and these functional groups direct affect the adsorption capacity of the biomass [17,18].

The comparisons of the FT-IR spectra of native, acid and alkali treated biomass were given in Figs. 1–3. The band at 3402–3344 cm^{-1} is O−H stretching of polymeric compounds, 2858 cm^{-1} is symmetric vibration of CH$_2$, 1740–1725 cm^{-1} is stretching vibration of COO, C=O, 1538 cm^{-1} band is streching vibration of C−N of peptidic bond of proteins, 1423–1417 cm^{-1} is of phenolic O−H and C=O stretching of carboxylates, 1233 cm^{-1} band is vibration of carboxylic acids, 1130–1000 cm^{-1} is vibration of C−O−C and O−H of polysaccharides, 873 cm^{-1} band is Si−H bend.

The bands < 800 cm^{-1} are finger print zone which is phosphate and sulphur functional groups.

Some functional groups of biomass are losing with acid or alkaline pretreatment. The acid and alkaline pretreatment can cause degradation of cellular compounds, such as cell wall, proteins and complex organic components of biomass. Changing of functional groups with pretreatment was given in Table 1. The FT-IR results showed that dried biomass have different functional groups for heavy metal ions binding, such as carboxyl, phosphate, amide, thiol and hydroxide groups.

3.2. Effect of initial pH

Solution pH is an important parameter for the adsorption experiments. The effect of pH on adsorption was given in Fig. 4 at 2–6 pH ranges, 20 °C, 100 mg L^{-1} initial metal concentrations for native biomass (pretreated biomass data was not given). The equilibrium adsorption capacity was observed at pH 4.0 for Cd(II), Cu(II) and pH 5.0 for Ni(II) ions. The initial pH of the solution was significantly affected the adsorption capacity of adsorbent, adsorption capacity was decreases with decreasing of pH however adsorption capacity was increase with increasing of pH. The lowest metal uptake values were determined at pH 2.0. At lower pH more protons will be available to protonate active groups of biomass surface, such as chitin, acidic polysaccharides, lipids, amino acids and other cellular components of the micro organism and metal ions are competing with the H$^+$ in the solution at low pH values for adsorption [19]. Similar results have been reported by other researchers for the uptake of heavy metal by activared sludge and aquatic moss *Fontinalis antipyretica* [9,20]. The adsorption capacity was determined as 72, 61 and 51 mg g^{-1} for native biomass, 79, 66 and 56 mg g^{-1} for
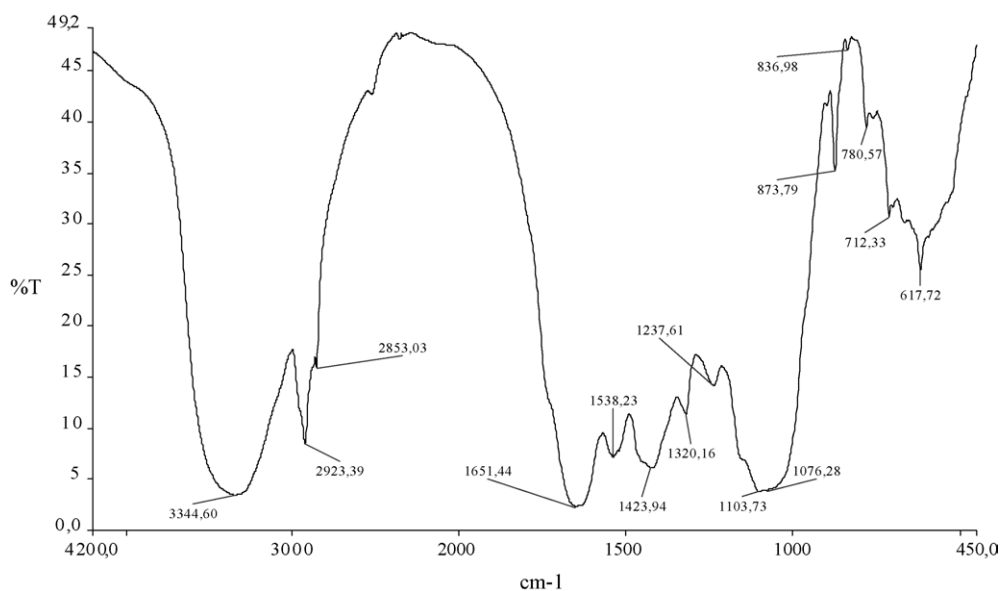

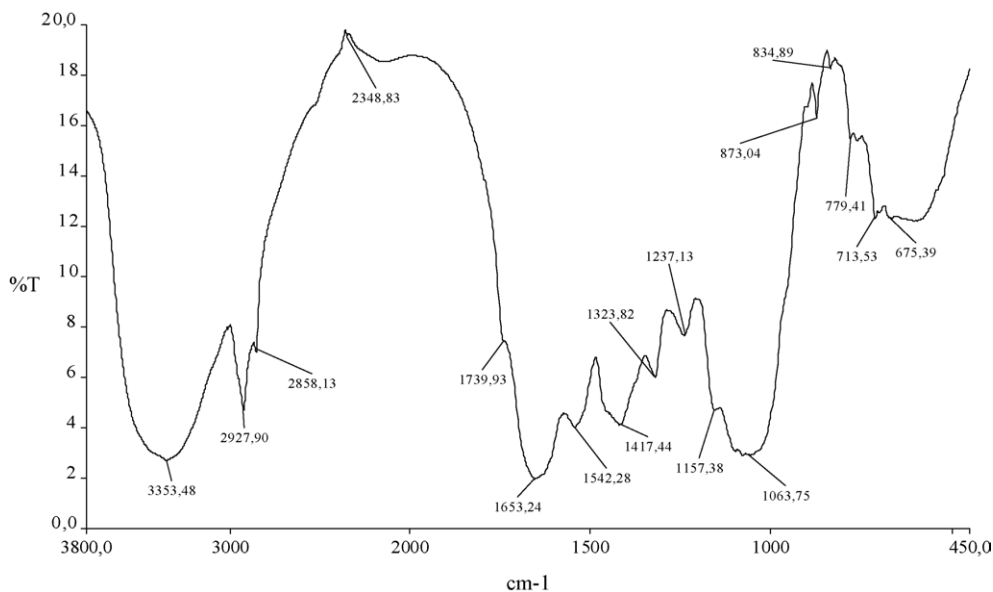Fig. 1. FT-IR spectra of native biomass.

Fig. 2. FT-IR spectra of alkali pretreated biomass.

alkali pretreated biomass and 26, 23 and 20 mg g^{-1} for acid pretreated biomass of Cd(II), Cu(II) and Ni(II) ions, respectively.

3.3. Effect of initial metal concentration and contact time

Effect of initial metal concentration on adsorption was studied at 50 and 100 mg L^{-1} initial metal concentrations and 20 °C. The results were given in Figs. 5–7. Initial metal concentration have important role for mass transfer between the aqueous and solid phase in. The effect of initial metal concentrations was investigated between 50 and 100 mg L^{-1} initial metal concentrations, at 20 °C. The equilibrium adsorption

capacity of biomass was increased with increasing initial metal concentration. The adsorption capacity was determined as 79, 66 and 56 mg g^{-1} of Cd(II), Cu(II) and Ni(II) for alkali pretreated biomass; 72, 61 and 51 mg g^{-1} of Cd(II), Cu(II) and Ni(II) for native biomass and 26, 23 and 20 mg g^{-1} of Cd(II), Cu(II) and Ni(II) for acid pretreated biomass at 100 mg L^{-1} initial metal concentrations.

The contact time between the adsorbate and adsorbent are important parameter for designed adsorption process. Adsorption studies were carried out for 100 or 180 min in order to determine the effect of time on adsorption (Figs. 5–7). Metal adsorption on to biomass increased with time and high amount of metal ions was adsorbed in the first 15–20 min and equilibrium was reached at 40 min for Cu(II), Ni(II) and
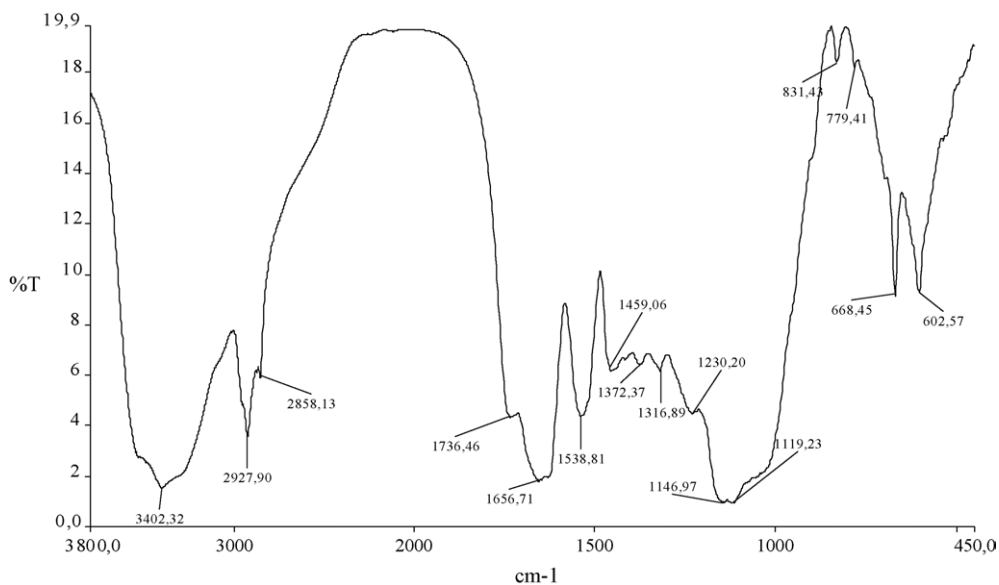

Fig. 3. FT-IR spectra of acid pretreated biomass.

Table 1
Comparison of infrared bands in the 3400–400 cm^{-1} spectral region

Functional groups of biomass	Treatment		
	Native biomass	Alkali pretreated biomass	Acid pretreated biomass
O—H stretching	3344	3353	3402
CH$_2$ vibration	2923	2927	2927
	2853	2858	2858
COO stretching	–	2348	–
C=O stretching	–	1739	1736
Amide I band	1651	1653	1656
Amide II band	1538	1542	1538
O—H and C=O stretching	–	–	1459
CH	1423	1417	–
CH bending	–	–	1372
C—O stretching	1320	1323	1316
COO vibration	1237	1237	–
C—O—C, C—O—P vibration	–	1157	1146
	1103	–	1119
	1076	1063	–
Si—H bend	873	873	–
	836	–	831
Finger print zone—SH, —PO$_4$ functional groups	780	779	779
	712	713	–
	–	675	668
	617	–	602
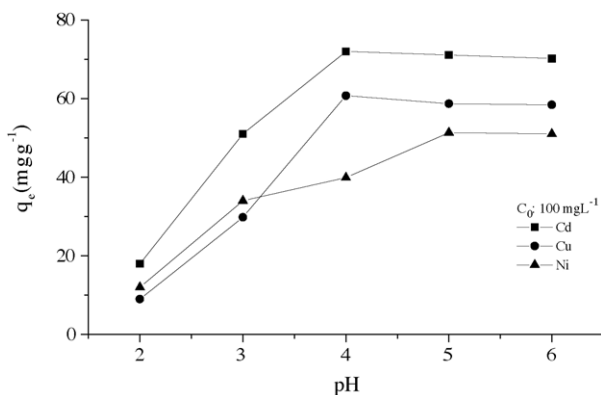
Fig. 4. Effect of initial pH on adsorption for native biomass.

60 min for Cd(II). After equilibrium time no more metal ions were adsorbed by *Lemna* biomass removal.

3.4. Adsorption isotherm

Adsorption isotherms are essential data source for practical design of adsorption systems and understanding of relation with adsorbent and adsorbate. The Langmuir and Freundlich isotherm models were used for determination of adsorption isotherm data at 20 °C. The adsorption isotherm constants, equilibrium monolayer capacities (q_{max}) were given in Table 2. The Langmuir adsorption isotherm results are shown in Fig. 8. The equilibrium monolayer capacities, q_{max}, were determined as 83, 69 and 59 mg g^{-1} for alkali

Fig. 5. Adsorption of Cd(II) ions on native, alkali and acid pretreated biomass.
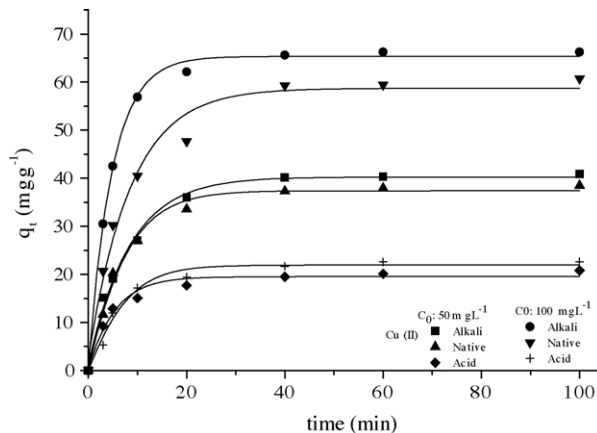

Fig. 6. Adsorption of Cu(II) ions on native, alkali and acid pretreated biomass.
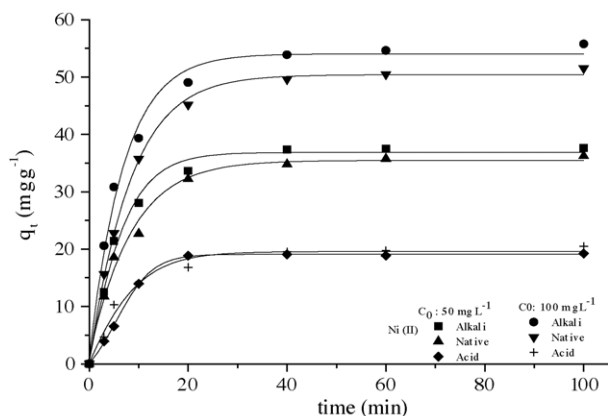
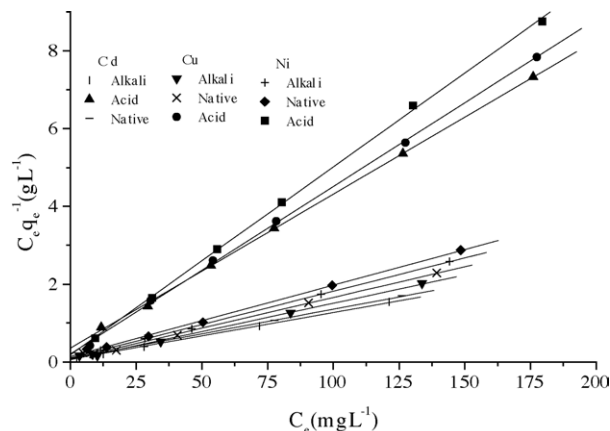Fig. 7. Adsorption of Ni(II) ions on native, alkali and acid pretreated biomass.

Fig. 8. Langmuir adsorption isotherms at 20 °C.

treated biomass, 79, 63 and 55 mg g^{-1} for native biomass and 25, 23 and 21 mg g^{-1} for acid treated biomass for Cd(II), Cu(II) and Ni(II) ions, respectively. The alkali pretreatment no more influenced the metal adsorption capacity of the biomass. The acid pretreatment was significantly decreased the adsorption capacity of the biomass.

Freundlich equation deals with physicochemical adsorption on heterogeneous surfaces. The Freundlich isotherm constants were given in Table 2, the magnitude of Freundlich exponent (n) showed that adsorption is good. The correla-

tion coefficient of linear Freundlich equation was greater than 0.830. The Langmuir model fits are quite well for adsorption data under the studied experimental conditions.

3.5. Multi metal ions adsorption

Multi metal ions adsorption of metal ions was conducted for solution containing 1 mmol of each metal ion at pH 4. The adsorption results were given in Fig. 9. The adsorption capacities of the biomass for multi metal ions were lower than single metal adsorption capacity. The ionic charge, ionic

Table 2
Langmuir and Freundlich adsorption isotherm constants

| Metal	Treatment	Langmuir isotherm				Freundlich isotherm		
		a_L	k_L	q_{max} (mg g^{-1})	R^2	n	k_F (L g^{-1})	R^2
Cd	Alkali	0.179	14.81	83	0.999	3.24	21.69	0.832
	Native	0.137	10.85	79	0.997	3.68	20.86	0.876
	Acid	0.112	2.82	25	0.999	11.09	15.13	0.991
Cu	Alkali	0.254	17.54	69	0.998	4.02	23.36	0.856
	Native	0.195	12.35	63	0.998	3.79	19.53	0.895
	Acid	0.211	4.89	23	0.999	11.74	14.74	0.982
Ni	Alkali	0.161	9.43	59	0.999	3.82	17.44	0.857
	Native	0.128	6.99	55	0.997	3.48	14.31	0.868
	Acid	0.260	5.38	21	0.999	23.42	16.23	0.922
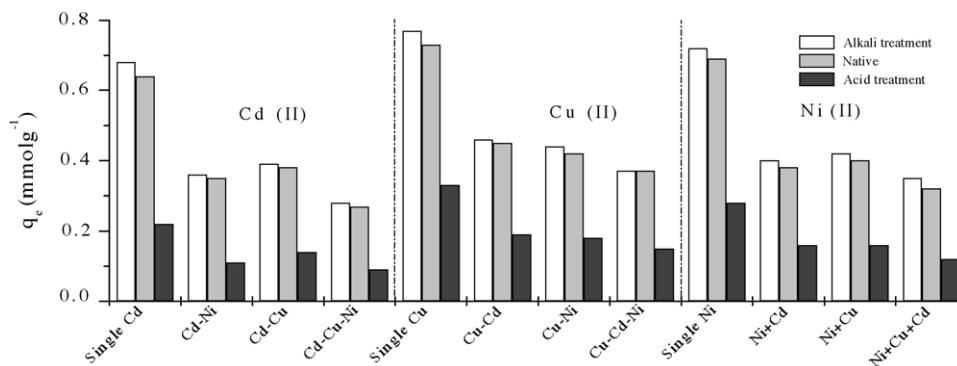
Fig. 9. Comparison of the single and multi metal adsorption capacity.

radii and electrode potential affect in the multi metal ions adsorption [18,21]. Many of the functional groups are present on the cell wall and different cations are able to react with these cellular components.

3.6. Adsorption kinetics

The pseudo first-, second-order and intraparticle kinetic models were used for analysis of adsorption kinetics. The effects of metal concentrations on adsorption kinetics were investigated for native and acid or alkaline treated biomass. The adsorption kinetic constants, correlation coefficients and the average percentage errors between the experimental and calculated data were given in Tables 3 and 4. Plots of linearized form of the pseudo first-order and pseudo second-order kinetic models were given Figs. 10–13. The first-order rate constant k_1 and $q_{e,cal}$ values determined from Figs. 10 and 11 are compared with the correlation coefficients in Table 3. The calculated q_e values of first-order did not give reasonable values, which were too low to compare with experimental q_e values. The adsorption of metal ions on *Lemna* biomass is not a first-order reaction.

The pseudo second-order kinetic rate constant k_2 and q_e values were determined from the slope and intercept of the Figs. 12 and 13. The correlation coefficients of the pseudo
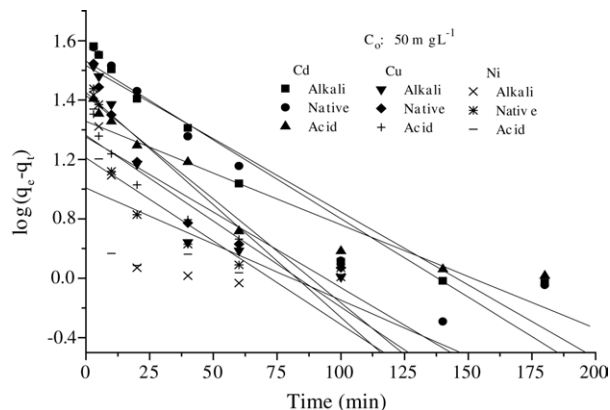

Fig. 10. Pseudo first-order adsorption kinetics at 50 mg L initial metal concentrations.

second-order kinetic model were higher than 0.992. Also, the calculated data ($q_{e,cal}$) were fitted the experimental data (Table 4). The pseudo second-order adsorption mechanism was predominant for adsorption of metal ions by native and pretreated *Lemna* biomass.

The plot of q_t versus $t^{0.5}$ may present multi linearity. The first shape portion is the external surface adsorption stage. The second shape is the gradual adsorption stage, where the intraparticle diffusion is rate-controlled. The third

Table 3
The pseudo first-order kinetic constants

Metal	Treatment	Initial concentration 50 mg L^{-1}				Initial concentration 100 mg L^{-1}			
		$q_{e,exp}$ (mg g^{-1})	$q_{e,cal}$ (mg g^{-1})	$k_1 \times 10^{-3}$ (min^{-1})	R^2	$q_{e,exp}$ (mg g^{-1})	$q_{e,cal}$ (mg g^{-1})	$k_1 \times 10^{-3}$ (min^{-1})	R^2
Cd	Alkali	43	27	22.57	0.914	79	35	25.56	0.847
	Native	41	29	24.41	0.912	72	36	22.57	0.899
	Acid	22	8	16.12	0.877	26	17	17.73	0.928
Cu	Alkali	41	22	37.77	0.827	66	24	38.00	0.786
	Native	38	21	37.77	0.838	61	36	43.76	0.903
	Acid	21	11	26.48	0.845	23	12	30.40	0.801
Ni	Alkali	38	14	34.78	0.845	56	24	38.92	0.909
	Native	36	17	31.51	0.839	51	24	33.85	0.862
	Acid	19	9	26.48	0.668	20	17	25.79	0.846

Table 4
The pseudo second-order kinetic constants

Metal	Treatment	Initial concentration 50 mg L^{-1}				Initial concentration 100 mg L^{-1}			
		$q_{e,exp}$ (mg g^{-1})	$q_{e,cal}$ (mg g^{-1})	$k_2 \times 10^{-3}$ (min g mg^{-1})	R^2	$q_{e,exp}$ (mg g^{-1})	$q_{e,cal}$ (mg g^{-1})	$k_2 \times 10^{-3}$ (min g mg^{-1})	R^2
Cd	Alkali	43	46	1.88	0.995	79	82	1.84	0.998
	Native	41	45	1.48	0.995	72	77	1.72	0.997
	Acid	22	22	5.98	0.997	26	27	2.8	0.996
Cu	Alkali	41	43	5.89	0.996	66	68	7.45	0.999
	Native	38	40	6.19	0.998	61	64	3.51	0.997
	Acid	21	21	15.14	0.999	23	24	9.12	0.996
Ni	Alkali	38	39	7.76	0.998	56	58	4.96	0.999
	Native	36	38	6.21	0.996	51	54	4.12	0.996
	Acid	19	21	8.31	0.999	20	22	8.63	0.992

Table 5
The intraparticle diffusion kinetic constants

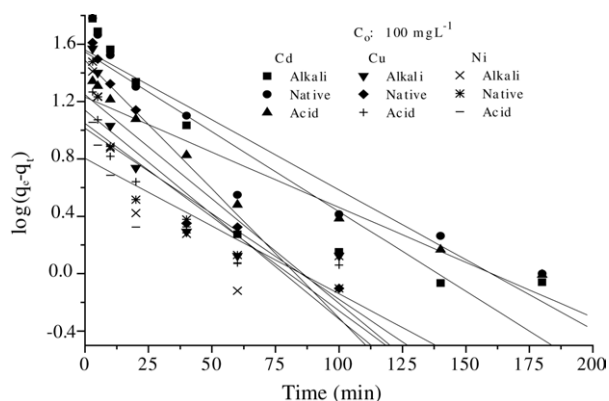| Metal	Treatment	k_1 (mg g min^{-1})	R^2	k_2 (mg g min^{-1})	R^2	k_3 (mg g min^{-1})	R^2
Cd	Alkali	13.21	0.993	6.06	1	0.10	0.965
	Native	12.83	0.978	2.70	0.996	0.41	0.999
	Acid	3.32	0.997	2.74	0.999	0.37	0.983
Cu	Alkali	18.19	0.998	2.71	0.955	0.24	0.911
	Native	11.01	0.978	3.72	0.856	0.42	0.906
	Acid	4.70	0.934	1.40	0.992	0.25	0.987
Ni	Alkali	12.71	0.992	4.47	0.928	0.52	0.999
	Native	10.49	0.987	4.24	0.901	0.52	0.992
	Acid	3.99	0.949	1.80	0.984	0.36	0.956

Fig. 11. Pseudo first-order adsorption kinetics at 100 mg L initial metal concentrations.

shape is the final equilibrium stage. When the adsorption of exterior surface reached saturation, the metal ions entered into the biomass particles by the pore within the particle and were adsorbed by the interior surface of the particle [22–24].

The plot of q_t versus $t^{0.5}$ is given in Fig. 14. The intraparticle diffusion rate constants (k_{i1}, k_{i2} and k_{i3}) and q_e were given in Table 5. If intraparticle diffusion rate constants are
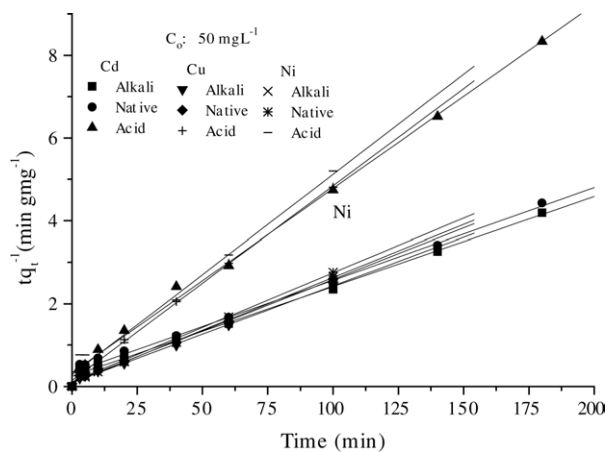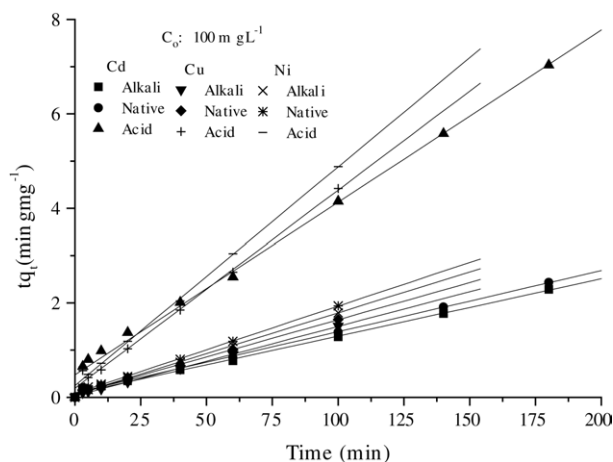

Fig. 13. Pseudo second-order sorption kinetics at 100 mg L initial metal concentrations.

compared, it is easy to see that k_{i1} (first stage) $>k_{i2}$ (second stage) $>k_{i3}$ (third stage).

The intraparticle diffusion model and pseudo-second kinetic models are applicable for adsorption system. The applicability of both models showed that sorption process is complex and involves more than one mechanism.
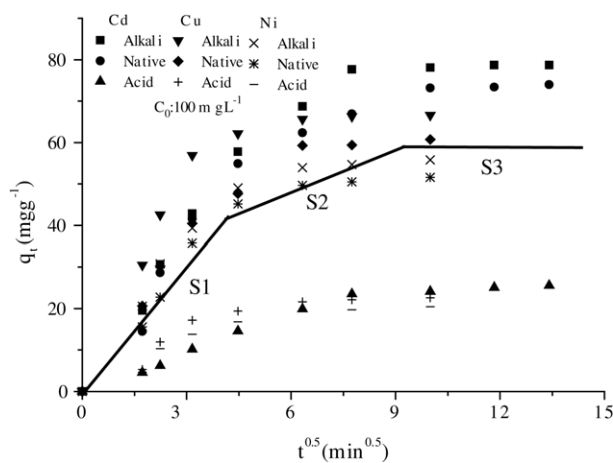

Fig. 12. Pseudo second-order sorption kinetics at 50 mg L initial metal concentrations.

Fig. 14. Intraparticle diffusion kinetics of native, alkali and acid pretreated biomass.

4. Conclusion

In batch adsorption studies, data show that dried *Lemna minor* has considerable potential for the removal of metal ions from aqueous solution. The Langmuir adsorption isotherm model fitted very well with studied metal concentration ranges at 20 °C. Adsorption capacity was little increase with alkali pretreatment but it was decreased with acid pretreatment according to native biomass.

The suitable kinetic models for the adsorption were also discussed. It was clear that the adsorption kinetics of metal ions obeyed pseudo second-order adsorption and intraparticle diffusion model. The single metal adsorption capacities of biomass were decreased with multi metal interactions in solution. According to FT-IR results biomass has different functional groups. These functional groups are able to react with metal ions in aqueous solution.

This study demonstrated that the dried *Lemna minor* biomass (native and alkali pretreated) could be used as an effective biosorbent for the treatment of wastewater containing Cd(II), Cu(II) and Ni(II) ions. However, *Lemna minor* is natural abundant environmental biomass and it may be alternative to more costly materials, such as activated carbon.

Acknowledgement

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