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ENVIRONMENTAL CHEMISTRY, POLLUTION & WASTE MANAGEMENT | RESEARCH ARTICLE

Adsorptive behavior, isothermal studies, and kinetic modeling involved in removal of divalent lead from aqueous solutions, using *Carissa carandas* and *Syzygium aromaticum*

Suresh Mahiya¹, Sanjay K. Sharma^{1*} and Giusy Lofrano²

Abstract: This study is focused on the biosorption of lead(II) ion onto surface of *Carissa carandas* and *Syzygium aromaticum* biomass from aqueous solution. The operating parameters, pH of solution, biomass dosage, contact time, initial metal ion concentration, and temperature considerably affect the biosorption efficiency of Pb(II). Biosorbent *C. carandas* leaf powder showed higher sorption efficiency than that of biosorbent *S. aromaticum* powder under identical experimental conditions. It was observed that the lead(II) removal percentage was found highest of 95.11% for *C. carandas* and 91.04% for *S. aromaticum* at contact period of 180 min. Also, it was observed that the regression coefficient ($R^2 = 0.99$) for the pseudo-second-order kinetic model is higher in comparison with the pseudo-first-order kinetic model and the calculated value of q_e for the pseudo-second-order kinetic model is very close to the experimental value, which indicates that it fits well with the equilibrium data for

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PUBLIC INTEREST STATEMENT

Potable water is a great concern worldwide and the researchers are trying hard for its purification and sustainability. Heavy metals are important pollutant present in water, which may cause serious adverse effects on the health of human beings, animals, and environment. Various modern and promising techniques are nowadays popular. Biosorption is one of them. The present research is a modest effort to remove lead from aqueous solutions by using *Carissa carandas* and *Syzygium aromaticum* as adsorbents. Where, we are reporting adsorptive behavior, isothermal studies, and kinetic modeling involved in removal of divalent lead from aqueous solutions. This research opens a new door to the researchers to try naturally available adsorbents or agriculture wastes to remove heavy metals from water and wastewater.

Pb(II) sorption from aqueous solutions on biosorbents. Also, the adsorption of Pb(II) onto *C. carandas* was best described by the Freundlich isotherm model.

Subjects: Applied & Industrial Chemistry; Environmental Chemistry; Environmental Sciences

Keywords: green chemistry; divalent lead; adsorption; biosorption; *Carissa carandas*; *Syzygium aromaticum*; Langmuir isotherm; Freundlich isotherm; kinetics

1. Introduction

Industrialization is the biggest source of heavy metals pollution in environment. Contrasting organic pollutants, the mainstream of which are vulnerable to biological degradation, heavy metal ions is not degradable into undamaging end products (Gautam, Sharma, Mahiya, & Chattopadhyaya, 2015). Heavy metals have been exceptionally released into the environment due to speedy industrialization and have become a major global concern. Heavy metals like cadmium (Cd), zinc (Zn), copper (Cu), nickel (Ni), lead (Pb), mercury (Hg), and chromium (Cr) are major habitually detected in industrial wastewaters, which instigate from metal plating, mining activities, smelting, battery manufacture, tanneries, petroleum refining, paint manufacture, pesticides, pigment manufacture, printing and photographic industries, etc. (Forgacs, Cserháti, & Oros, 2004; Mahiya, Lofrano, & Sharma, 2014a).

Lead exposure is toxic or poisonous. It causes many dangerous diseases like encephalopathy, nephropathy, anemia, mental retardation, seizures and it forms complexes with oxo-groups in enzymes and affect the hemoglobin synthesis (Ademorati, 1996; Schümann, 1990).

The WHO limit for lead in drinking water is 0.01 mg/L and JECFA established a provisional tolerable weekly intake (PTWI) for lead corresponding to 25 µg of lead per kilogram of body weight (WHO, 2011).

Already many methods are used to treat the lead(II) ion contamination, like chemical precipitation (Esalah, Weber, & Vera, 2000), ion exchange, membrane filtration (Canet, Ilpide, & Seta, 2002), electrolysis (Widner, Sousa, & Bertazzoli, 1998), reverse osmosis, solvent extraction, activated carbon (Khan, Alemayehu, Duraisamy, & Berekete, 2015), adsorption on minerals (Ahmad, Khalid, & Daud, 2002; Buerge-Weirich, Hari, Xue, Behra, & Sigg, 2002; Chamila, Murthi, Oksana, Songping, & Mietek, 2015), flotation (Zouboulis, Matis, Lanara, & Loos-Neskovic, 1997), magnetic nanoparticles and zeolites (Pankaj, Sharma, & Sambhi, 2015).

The Green Chemistry has more effective processes for removal of heavy metals than other conventional methods. There are many green techniques available using for removal of heavy metal ions from water and wastewater like biosorption (Mahiya, Lofrano, & Sharma, 2014b; Sharma, Mahiya, & Lofrano, 2015), phytoremediation (Mani & Kumar, 2014), photocatalytic processes (Sajad et al., 2014), and bioremediation (Mani & Kumar, 2014).

Adsorption is one of the most widely applied green techniques for removal of lead(II) ion from aqueous solution (Grassi, Kaykioglu, Belgiorno, & Lofrano, 2012). Various biosorbents were used for the removal of lead(II) ions from the aqueous solutions which includes orange peel, bamboo, morninga oleifera pods (Adelaja, Amoo, & Aderibigbe, 2011), cone biomass (Handan, Bayhan, Yusuf, Avni, & Algur, 2003), peanut shells (Şeyda, Fatih, & Ahmet, 2014), olive tree pruning waste (Blázquez, Martín-Lara, Tenorio, & Calero, 2011), *Punica granatum* L. peels (Cigdem, Safa, Yunus, & Adnan, 2012), and black cumin (Deniz, Merve, Sermin, & Erdal, 2012).

The purpose of the present study is to evaluate the efficiency of *C. carandas* and *S. aromaticum* as biosorbent for removal of divalent lead from aqueous solution. Maximum adsorption capacity of biosorbent, adsorption intensity of the adsorbate on biosorbent surface, and biosorption potentials of biosorbent were estimated by Langmuir and Freundlich isotherms, respectively. In the present study *C. carandas* and *S. aromaticum* leaves were used for biosorption of lead(II) from aqueous solutions.

Batch adsorption experiments were carried out at ambient temperature (300 K) as a function of solution pH (2–12), biosorbent dosage (20–100 g/L), contact time (60 min interval and up to 300 min), and initial metal ion concentration. Then, equilibrium isotherms and kinetic data parameters were evaluated. The prepared adsorbent was characterized by SEM analysis and FTIR analysis.

2. Experimental work

2.1. Materials and methods

2.1.1. Chemicals

Analytical-grade chemicals were used in this work without further purification. To avoid any interference of other ions, all solutions were prepared using double distilled water. To evaluate the significance of the adsorbent, stock solutions (1,000 mg/L) of Pb(II) were prepared by dissolving 1.831 g of $(\text{CH}_3\text{COO})_2\text{Pb}\cdot 3\text{H}_2\text{O}$ in 1,000 mL volumetric flask and make up to the mark with double distilled water. All the required working solutions were prepared by diluting the stock solution with double distilled water. Dilute solutions of 0.1 M HNO_3 and 0.1 M NaOH were used to adjust pH of metal ion solutions using a pH meter.

2.1.2. Adsorbents

The leaves of *C. carandas* and buds of *S. aromaticum* were collected from local field of Pushkar (India) and from local market of Jaipur (India), respectively. These were washed with distilled water, dried in sunlight, then 60°C for 24 h in hot air oven. Finally, the dried leaves of *C. carandas* and *S. aromaticum* were grinded in clean electric mixer and stored in a dry and clean plastic bag.

2.1.3. Experimental

The affinity of *C. carandas* and *S. aromaticum* to adsorb lead(II) were studied in batch experiments. All experiments, performed at room temperature and fixed volume of lead(II) ion solution in 100 mL was stirred with desired biosorbent dose (2–10 gm) for the period of three hours.

After a defined time interval, samples were withdrawn from the shaker, filtered by Whatman filter paper No. 1, and the supernatant solutions were analyzed for Pb(II) ion concentration using an atomic absorption spectrometer (thermo scientific solar S-series AA spectrometer). The removal of lead was calculated according to following expression:

$$\text{Metal removal \%} = \frac{(C_0 - C_i)}{C_0} \times 100 \quad (1)$$

where C_0 and C_i are the initial and equilibrium concentrations (mg/L).

3. Results and discussion

3.1. Effect of pH on biosorption

To study the effect of pH on adsorption of lead(II) ions, the batch equilibrium studies at pH values in the range of 2–10 were carried out. The results are furnished in Table 1 and the variation is presented in Figure 1. It was observed that after pH 10 solid precipitation of $\text{Pb}(\text{OH})_2$, PbOH^+ , $\text{Pb}(\text{OH})_2^-$, and $\text{Pb}(\text{OH})_3^-$ was occurred. Therefore, a pH range of 2–10 was used during the analysis (Issabayeva, Aroua, & Sulaiman, 2006; Rajkumar, Sefra, & Praveen, 2015).

It is observed that biosorption of heavy metal is critically linked with pH of the solution. The pH of solution is a very important contributing and controlling factor in the adsorption process, for this the role of hydrogen ion concentration was examined at different pH. The effect of pH was studied at the Pb(II) concentration of 100 mg/L, biosorbent dosage of 2 g/100 mL at the pH range 2–10. It was observed that with the increase in the pH of the solution the percentage removal of Pb(II) increased

Figure 1. Effect of solution pH on biosorption of Pb(II) by *C. carandas* and *S. aromaticum* ($C_0 = 100$ ppm, dosage = 2 g/100 mL, contact time = 180 min).

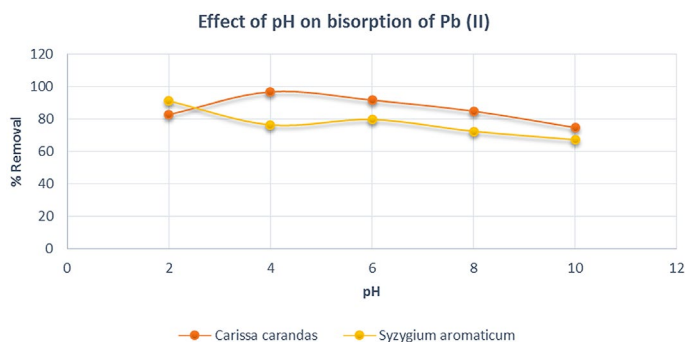


Table 1. Effect of solution pH on biosorption of Pb(II) by *C. carandas* and *Syzygium aromaticum* ($C_0 = 100$ ppm, dosage = 2 g/100 mL, contact time = 180 min)

S. No.	pH	Removal of Pb(II) (%)	
		<i>C. carandas</i>	<i>S. aromaticum</i>
1	2	82.43	91.34
2	4	96.57	76.32
3	6	91.46	79.65
4	8	84.56	72.34
5	10	74.38	67.02

Table 2. Effect of contact time on biosorption for Pb(II) by *C. carandas* and *Syzygium aromaticum* ($C_0 = 100$ ppm, dosage = 2 g/100 mL)

S. No.	Contact time (min)	Removal of Pb(II) (%)	
		<i>C. carandas</i>	<i>S. aromaticum</i>
1	60	87.985	82.22
2	120	89.57	86.39
3	180	95.11	91.04
4	240	95.11	91.04
5	300	95.11	91.04

up to the pH 4 for *C. carandas* and pH 2 for *S. aromaticum*. Further increase in pH value decreased the percentage removal of Pb(II) up to pH 10 (Table 1).

3.2. Effect of contact time on biosorption

As shown in Figure 2 the effect of contact time on biosorption of lead(II) was performed. It was observed that the lead(II) removal percentage was the lowest of 87.98% for *C. carandas* and 82.22% for *S. aromaticum* at 60 min of contact time and the highest of 95.11% for *C. carandas* and 91.04% for *S. aromaticum* at contact period of 180 min. After equilibrium reaction, increase in contact time did not affect the biosorption process of lead(II) ion. Hence the contact time of 180 min was selected for further experiments. The observations are given in Table 2. Similar results were observed for removal of lead(II) by Shaik, Yadamari, Yakkala, and Gurijala (2015).

3.3. Effect of biosorbent dose

Figure 3 illustrates the variation of adsorption efficiency with varying adsorbent dosage using *C. carandas* and *S. aromaticum*, which shows that the adsorption efficiency increases with an increase in adsorbent dosage. A graph was plotted between the different dosage of biosorbent *C. carandas* and *S. aromaticum* and the resultant percentage removal of Pb(II). The effect of different biosorbent

Figure 2. Effect of contact time on biosorption for Pb(II) by *C. carandas* and *S. aromaticum* ($C_0 = 100$ ppm, dosage = 2 g/100 mL).

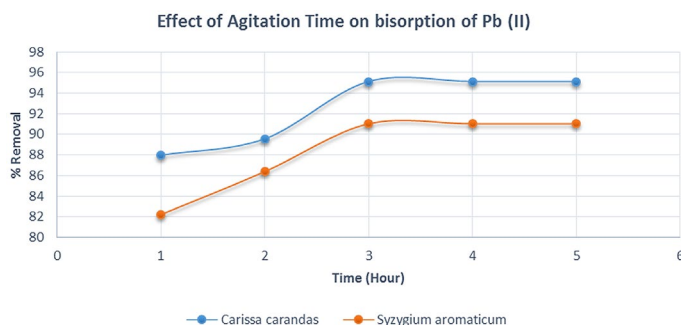
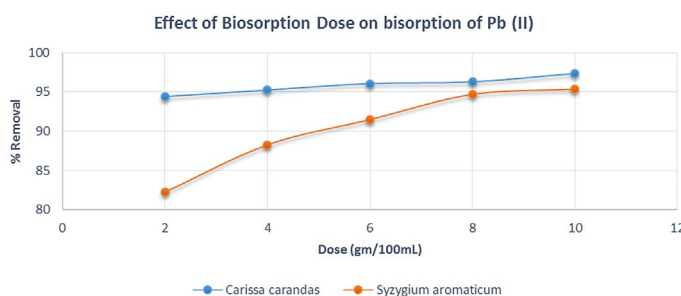


Figure 3. Effect of biosorbent dose on biosorption capacity of Pb(II) ($C_0 = 100$ mg/100 mL, contact time = 180 min).



dosage (2–10 g/100 mL) with percentage removal of lead is shown in Figure 3. The figure shows a marginal increase in lead removal with increasing biomass concentration (Table 3).

3.4. Effect of initial metal concentration

The biosorption capacity of *C. carandas* and *S. aromaticum* as a function of the initial concentrations of lead have been studied at different concentrations of Pb(II) in batch experiments. Increasing the initial concentration of Pb(II) in a batch study resulted in decreasing percentage of Pb(II) removal because evidently the biosorbent was approaching its saturation uptake capacity. In batch study using *C. carandas* and *S. aromaticum* biomass percentage removal of Pb(II) decreased from 95.11 to 71.12% and 90.04 to 68.98% when the initial concentration of Pb was increased from 100 to 1,000 mg/L (Table 4 and Figure 4).

3.5. Effect of temperature

Figure 5 shows the biosorption of Pb(II) for varied temperatures at 180 min of contact time. As shown in Figure 5 biosorption capacities at 20, 30, 40, and 60°C were found for Pb(II) as 76.21, 94.34, 90.91, and 61.17 mg/100 mL for *C. carandas* and 71.74, 88.98, 82.11, and 44.91 mg/100 mL for *S. aromaticum*, respectively. It was observed that the biosorption capacity of *C. carandas* and *S. aromaticum* decreased over 30°C for lead(II). It may be attributed to the deactivation of the biosorbent surface or the destruction of some active sites on the biosorbent surface. As a result, the optimum temperature for Pb(II) biosorption was chosen as 30°C for subsequent experiments (Table 5).

Table 3. Effect of biosorbent dosage on biosorption capacity of Pb(II) ($C_0 = 100$ ppm/100 mL, contact time = 180 min, $T = 300$ K)

S. No.	Biosorbent (g/100 mL)	Removal of Pb(II) (%)	
		<i>C. carandas</i>	<i>S. aromaticum</i>
1	2	89	74
2	4	91.43	82
3	6	93.23	85
4	8	96.12	89
5	10	97.57	94

Table 4. Effect of initial metal concentration on biosorption of Pb(II). (adsorbent dosage = 2 g/100 mL, contact time = 180 min, T = 300 K)

S. No.	Concentration of metal (mg/100 mL)	Removal of Pb(II) (%)	
		<i>C. carandas</i>	<i>S. aromaticum</i>
1	100	95.11	90.04
2	300	88.12	88.87
3	500	84.09	85.34
4	700	79.28	78.9
5	1,000	71.12	68.98

Figure 4. Effect of initial metal concentration on biosorption of Pb(II) (dosage = 2 g/100 mL, contact time = 180 min, pH = 6, T = 300 K).

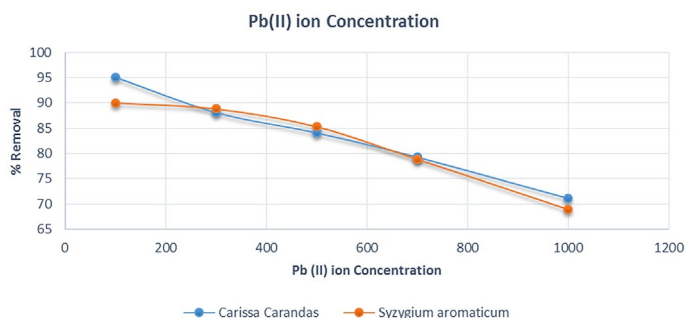


Figure 5. Effect of temperature on biosorption of Pb(II) ($C_0 = 100$ ppm, dosage = 2 g/100 mL, contact time = 180 min).

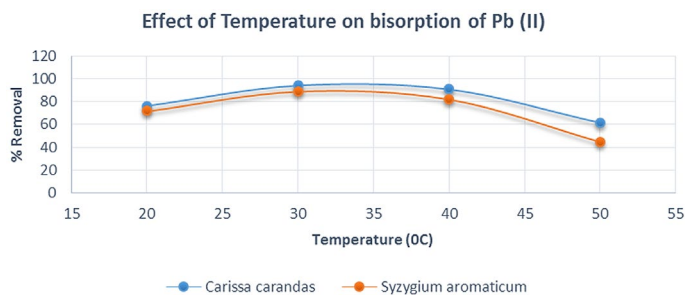
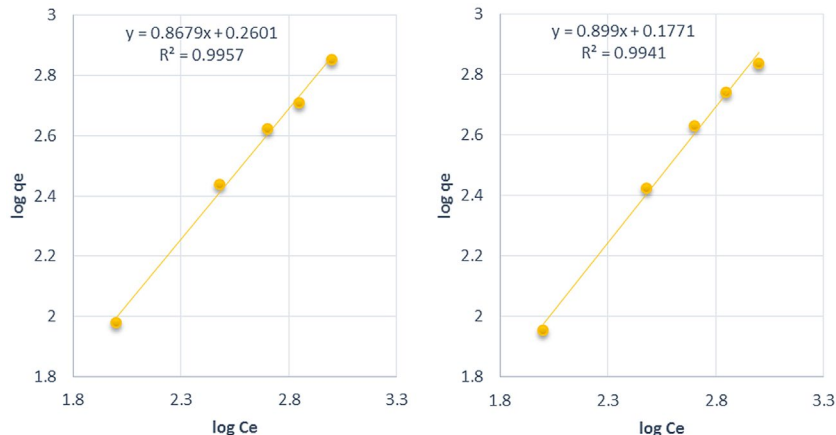


Table 5. Effect of temperature on biosorption of Pb(II) ($C_0 = 100$ ppm, adsorbent dosage = 2 g/100 mL, contact time = 180 min)

S. No.	Temperature (°C)	Removal of Pb(II) (%)	
		<i>C. carandas</i>	<i>S. aromaticum</i>
1	20	76.21	71.74
2	30	94.34	88.98
3	40	90.91	82.11
4	50	61.17	44.91

Figure 6. Freundlich isotherm for (a) *C. carandas* and (b) *S. aromaticum* for lead(II).



4. Isothermal studies

An adsorption is a quantitative relationship describing the equilibrium between the concentrations of adsorbate in solution and its concentration of adsorbent. To better understand the Pb(II) ion adsorption characteristics to the *C. carandas* and *S. aromaticum*, we applied two well-known two-parameter isotherms: Langmuir and Freundlich isotherm models. Langmuir adsorption (Langmuir, 1918) isotherm assumes monolayer adsorption of solutes onto a surface of adsorbent with a finite number of identical sites and can be expressed as

$$q_e = \frac{Q_0 b C_e}{(1 + b C_e)} \quad (2)$$

where C_e (mg/L) and q_e (mg/g) are the liquid phase concentration and solid phase concentration of adsorbate at equilibrium, respectively, and Q_0 (mg/g) and b (L/mg) are the Langmuir isotherm constants.

Figure 6(a) and (b) show the linear plots of C_e/q_e vs. C_e for both adsorbents, which are used to determine the value of q_{max} and b . The values obtained are given in Table 6. The R^2 values indicate that Langmuir isotherm fairly well predicts the adsorption process of Pb(II) ions by the *C. carandas* and *S. aromaticum*. The highest q_{max} was observed for the *C. carandas*. The Freundlich isotherm is known to well describe heterogeneous system for non-ideal adsorption. The experimental data on Pb(II) adsorption were fitted to the Freundlich adsorption isotherm (Freundlich, 1907), which can be expressed as:

$$Q_e = K_f C_e^n \quad (3)$$

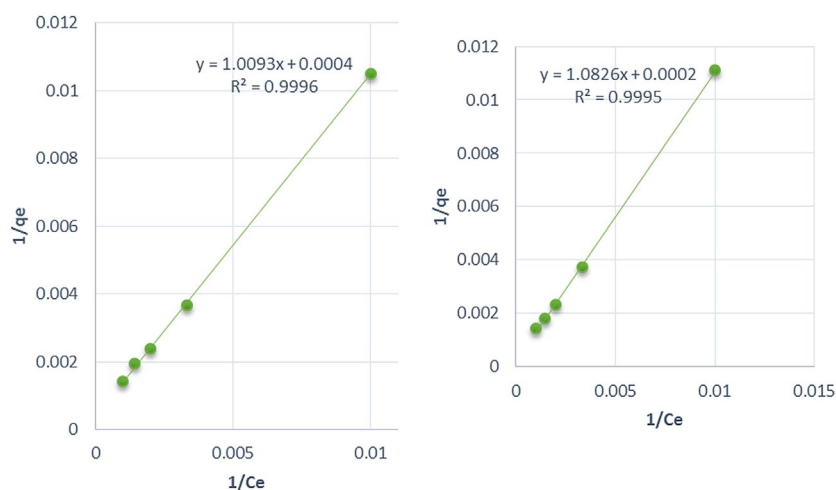
where K_f [mg/g (L/g)^{1/n}] is the Freundlich constant related to the bonding energy, n is the heterogeneity factor which depicts the extent of deviation from linearity of the adsorption. It indicates the degree of non-linearity between solution concentration and adsorption.

Table 6 lists the calculated Freundlich and Langmuir isotherm constants. Based on regression coefficient (R^2) values, the experimental data for adsorption of Pb(II) onto *C. carandas* and *S. aromaticum* powder fit better to Langmuir isotherm model than the Freundlich isotherm model. The adsorption of Pb(II) onto *C. carandas* was best described by the Langmuir isotherm model. The maximum adsorption capacities (K_L) estimated from the Langmuir isotherm model for Pb(II) were 2,018 mg/g and 5,414.18 mg/g for *C. carandas* leaves and *S. aromaticum*, respectively (Figure 7).

Table 6. Langmuir and Freundlich isotherm constants for Pb(II) ion biosorption

Adsorbent	Langmuir isotherm			Freundlich isotherm		
	q_{max}	K_L	R^2	K_F	N	R^2
<i>C. carandas</i>	1.009	2,018	0.9982	1.82	1.16	0.9957
<i>S. aromaticum</i>	0.923	5,414.18	0.9995	1.50	1.11	0.9941

Figure 7. Langmuir isotherm for (a) *C. carandas* and (b) *S. aromaticum* for lead(II).



5. Adsorption kinetics

The study of adsorption kinetics of biosorption of lead(II) ion by *C. carandas* and *S. aromaticum* is important as it provides efficacy of adsorption mechanism and valuable insight into the reaction pathways and it also describes the solute uptake rate. For analysis of sorption kinetics of Pb(II), kinetic models such as pseudo-first order and pseudo-second order have been used.

The linearized pseudo-first-order kinetic model takes the following form:

$$q_t = q_e - q_{e,exp} = (-k_1 t) \tag{4}$$

$$\log(q_e - q) = \log q_e - \frac{k_1}{2.303} t \tag{5}$$

where q is the amount of lead(II) (mg/g) at time t (min), q_e is the amount of lead adsorbed at equilibrium (mg/g), and K_1 is the equilibrium rate constant of pseudo-first-order adsorption (min^{-1}). The plot of $\log(q_e - q_t)$ vs. t gave a straight line for the first-order adsorption kinetics.

The pseudo-second-order kinetic model considered in this study is given as:

$$\frac{1}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \tag{6}$$

where K_2 ($\text{gmg}^{-1} \text{min}^{-1}$) is the second-order reaction rate constant.

The experimental data and the parameters of both models are tabulated in Table 7. It was observed that the regression coefficient (R^2 : 0.99) for the pseudo-second-order kinetic model is higher in comparison with the pseudo-first-order kinetic model and the calculated value of q_e for the pseudo-second-order kinetic model is very close to the experimental value. Similar experimental results indicate that the pseudo-second-order kinetic model fits the equilibrium data for heavy metal ion

Table 7. Kinetics studies of biosorption of Pb(II) on *C. carandas* and *S. aromaticum*

Biosorbent	Metal ion concentration (ppm)	Pseudo-first-order kinetic model			Pseudo-second-order kinetic model		
		$q_{e, cal}$ (mg/g)	K_1 (min^{-1})	R^2	$q_{e, cal}$ (mg/g)	K_2 ($\text{gmg}^{-1} \text{min}^{-1}$)	R^2
<i>C. carandas</i>	100	62.27	2.50×10^{-3}	0.95	106.38	3.7×10^{-4}	0.99
	300	149.90	3.4×10^{-3}	0.94	312.50	8.7×10^{-5}	0.99
	500	275.88	2.3×10^{-3}	0.95	476.19	8.2×10^{-5}	0.99
	700	368.70	2.9×10^{-3}	0.95	625	6.6×10^{-5}	0.99
	1,000	424.11	1.15×10^{-3}	0.95	833	3.7×10^{-5}	0.99
<i>S. aromaticum</i>	100	38.09	5.0×10^{-3}	0.92	121.95	1.2×10^{-4}	0.99
	300	113.29	5.0×10^{-3}	0.91	357.14	4.1×10^{-5}	0.99
	500	210.60	4.3×10^{-3}	0.91	526.31	3.9×10^{-5}	0.99
	700	284.29	3.9×10^{-3}	0.91	666.66	3.4×10^{-5}	0.99
	1,000	292.94	5.0×10^{-3}	0.91	909.14	1.6×10^{-5}	0.99

Figure 8. Pseudo-first- and pseudo-second-order kinetics for *C. carandas* and *S. aromaticum* ($C_0 = 100$ ppm, dose = 2 gm).

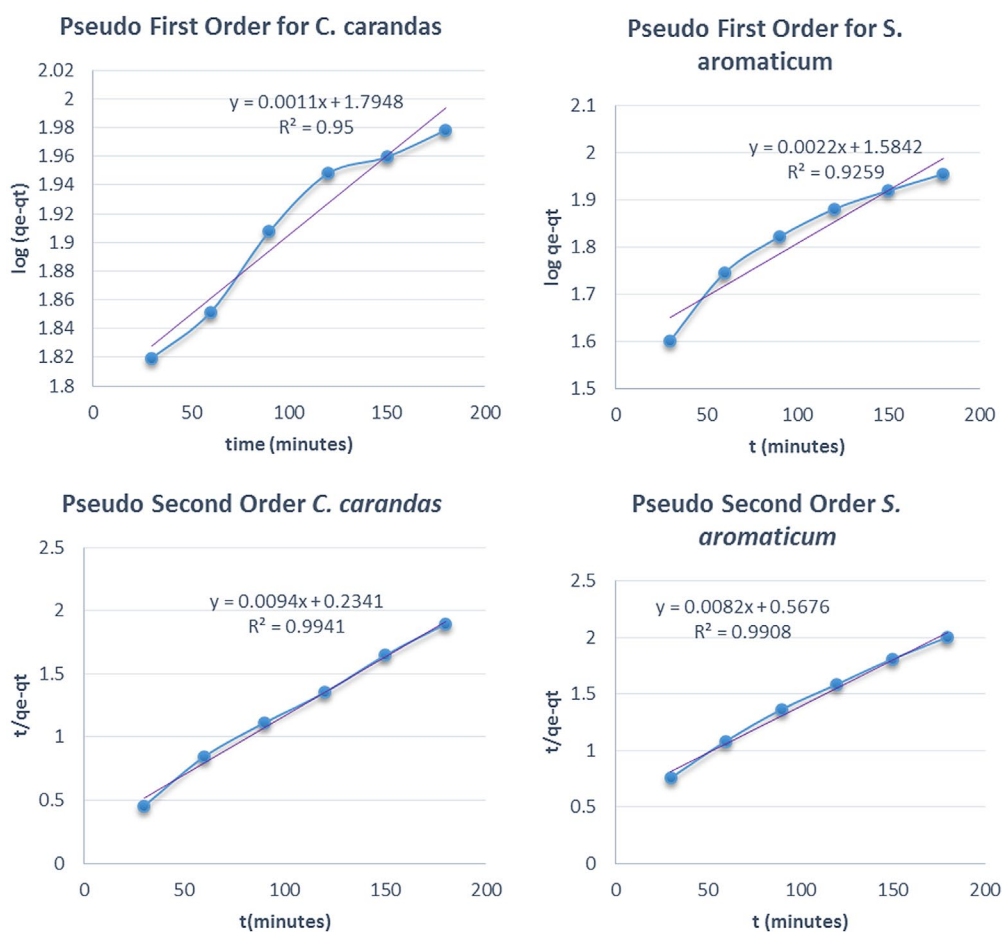


Figure 9. SEM image of *C. carandas* (a) before adsorption and (b) after adsorption of Pb(II).

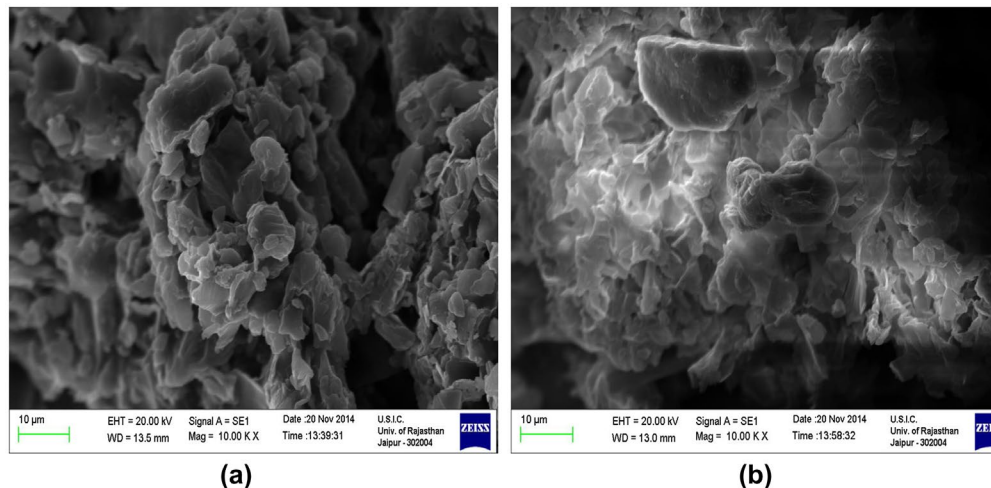
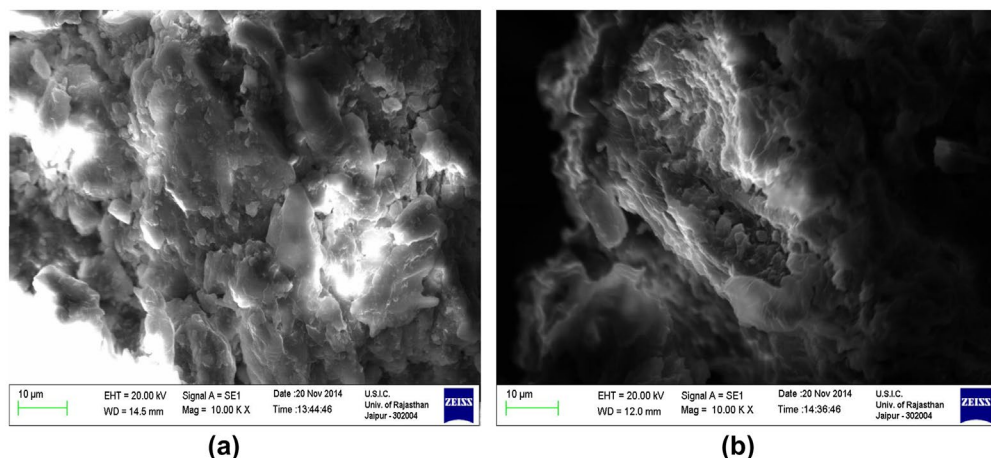


Figure 10. SEM image of *S. aromaticum* (a) before adsorption and (b) after adsorption of Pb(II).



sorption on biomasses from aqueous solutions quite well. Figure 8 shows the pseudo-first and pseudo-second order for both biosorbents and 100 ppm Pb(II) ion concentration.

6. SEM images

The SEM images of adsorbents before and after adsorption are given in Figures 9 and 10, respectively, for *C. carandas* and *S. aromaticum*. From these images, it is clear that there is significant difference in the appearance of the adsorbent surfaces. Images clearly highlight the action of adsorption on the surfaces of both the adsorbents and strengthen the view point of the researchers.

7. Conclusions

This study is focused on the biosorption of lead(II) ion onto of *C. carandas* and *S. aromaticum* biomass from aqueous solution. The operating parameters, pH of solution, biomass dosage, contact time, initial metal ion concentration, and temperature are effective on the biosorption efficiency of Pb(II). Biosorbent *C. carandas* leaf powder showed higher sorption efficiency than that of biosorbent *S. aromaticum* powder under identical experimental conditions. Also, the adsorption of Pb(II) onto *C. carandas* was best described by the Freundlich isotherm model.

However, we suggest that it also necessary to investigate the efficacy of *C. carandas* leaves powder to treat real industrial effluents. There is a ready supply of agricultural wastes worldwide. The

use of such materials will not only convert into low-cost effective adsorbents, but also provide a green solution to their disposal.

Since, several parameters including migration of metal ions from bulk solution to the surface of the adsorbent through bulk diffusion and the adsorption of metal ions at an active site on the surface of the adsorbent by chemical reactions play very important role in deciding the adsorption kinetics and adsorption mechanism; the actual adsorption mechanism is yet to be discussed and explained.

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