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Effective Removal of Lead Ions by Alternative Agro-waste Adsorbent from Aqueous Media

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Author's contribution

The sole author designed, analysed, interpreted and prepared the manuscript.

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ABSTRACT

Aims: The main goal of this work was to evaluate the removal of lead (II) from aqueous media by using low cost, natural, locally available, environmentally friendly and agricultural waste biosorbent of Molokhia husk (Mh) through batch experiments.
Methods: Different parameters were studied through batch experiments. The surface of Molokhia husk (Mh) before and after adsorption of lead ions was characterized by using FTIR analysis.
Results: It was observed that the maximum adsorption capacity 12.062 mg/g occurred at pH 5, adsorbent mass 0.8 g and contact time 90 min. The percentage removal of adsorption of Pb(II) onto Molokhia husk was 97.82%. Different adsorption isotherms models were used. Equilibrium data fitted well with the Freundlich isotherm (R^2 0.9429). Adsorption equilibrium was established after 90 min of contact time. The adsorption energy for Mh calculated from D-R equation 1.291 KJ/ mol indicating the physical adsorption process for removal of Pb (II). Different kinetic models were applied to study the kinetic data of adsorption. Kinetic studies demonstrated that the adsorption of Pb(II) by Mh followed a pseudo second-order model.
Conclusions: These results suggest that Mh could be used as a natural, cheap, low cost and environmentally friendly biosorbent and for removal of lead ions from aqueous media.

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Keywords: Molokhia husk waste; removal; lead (II); isotherms; kinetics; water treatment; environmentally friendly biosorbent.

1. INTRODUCTION

Pollution of water by heavy metals is considered the very important challenge in the world. Lead discharged into the environment through environmental conditions (soil, water, air, and food chain). Lead also may affect every organ in the human body even at low concentration in water. Also, effect on the damage of the brain and kidneys in adults or children when exposed to high concentrations of lead [1]. The primary source of lead present in many industrial processes such as paint, glass, and manufacturing of battery [2].

The permissible concentration of lead in drinking water as 0.1–0.05 mg/L which recommended by (WHO) [3]. There are many techniques used for removing of toxic heavy metals from water and wastewater includes flocculation, adsorption [4], oxidation [5], electrolysis, biodegradation [6], and adsorption [7]. But these techniques have some disadvantages such as high cost, incomplete removal of metal ion and a high amount of reagents and energy used [8]. On the other hand the process of adsorption is the most important, effective, economical and environmentally friendly techniques for removal of environmentally hazardous metals.

Among the variety of adsorbents, activated carbon may be logically the most preferred adsorbent for the removal of heavy metals because of its excellent adsorption ability. However, this process is proving to be uneconomical due to the high cost of activated carbon and also the additional cost involved in regeneration [9].

The advantages of these agricultural waste biomaterials include locally available, much cheaper, require fewer transportation costs, environmentally favorable and biodegradability [10].

There are many agricultural waste products and low-cost adsorbents such as peanut husk [11], wheat straw [12], pineapple leaves [13], garlic peel [14], sunflower, potato, [15-17].

Nowadays, the use of agricultural waste as adsorbents is currently receiving wide attention because of their coming from low cost, abundant, inexpensive, renewable and relatively high fixed carbons and presence of porous structures.

Molokhia (*Corchorus olitorius L.*) is the highly nutritious plant that originated in Egypt but has since spread throughout the different regions (the Mediterranean and the Middle East), and it is beginning to appear in western markets. Both leaves and young pods are edible and somewhat at mucilaginous. The composition of Molokhia includes vitamins (C, E, K, B6), phosphorous, selenium, calcium, iron, fiber and trace minerals [18]. Due to the high consumption of Molokhia plant, massive amounts of the husk are disposed, causing a severe problem in the community. Molokhia husk is available in large quantities in many countries of the world and free of cost.

To the best of our knowledge, there is no any study devoted to the potential applicability of Molokhia husk as a low-cost adsorbent for the removal of lead ions. The main objective of the present work was carried out to investigate the use of Molokhia husk (Mh) an agricultural waste material coming from Molokhia plant waste as a low cost, cheap and abundant adsorbent for removal of lead ions. pH, adsorbent dose, initial metal concentrations and contact time were investigated. Different models of kinetics and adsorption isotherm used through this study to describe the adsorption of Pb(II) onto Mh.

2. MATERIALS AND METHODS

2.1 Reagents

$Pb(NO_3)_2$ was used as the source of metal ions. All other chemicals used, such as hydrochloric acid and sodium hydroxide, were supplied by Merck.

2.2 Adsorbate

The stock solution of 1000 mg/L was prepared from $Pb(NO_3)_2$ (Merck) using double distilled water. The pH was adjusted with 0.1 M NaOH or 0.1 M HCl.

2.3 Preparation of Adsorbent

The Mh has been collected from a local market in Egypt and washed thoroughly with de-ionized water for removing impurities and dirt. The dried husk material has been ground and used as an adsorbent. The prepared Mh sample was stored in an airtight container for further use.

2.4 Adsorbent Characterization

Elemental analysis of the Molokhia husk biosorbent was measured for determining C, H, N and S content (Elemental C, H, N, S Analyzer, Vario EL III, Germany). The oxygen contents were calculated by the difference.

The functional groups presented in Molokhia husk was characterized by (FTIR) spectrometer (FT-IR-4100 JASCO, Japan).

2.5 Biosorption Experiments

To access the adsorption process, batch studies were performed by varying different physiochemical conditions and planned to determine the efficiency of Mh for the removal of Pb (II) ions. Initial lead ions concentration (5-100), pH (2–8) agitation speed 200 rpm and adsorbent dosage (0.1-1.0 g/100 ml) were studied on the removal of lead ions by Mh through batch adsorption experiments. The solutions were filtered through Whatman No. 40 filter paper, centrifuged (5000 rpm, 10 min) for solid-liquid separation and the concentrations of lead ions were determined using Microwave Plasma Atomic Emission Spectrometry (MP–AES).

2.6 Data Evaluation

The amount of biosorption was calculated based on the difference between the initial (C_0, mg/L) and final concentration (C_e, mg/L) in every flask, as follows:

$$q_e = \frac{(C_0 - C_e)V}{m} \quad (1)$$

Where q_e is the metal uptake capacity (mg/g), V is the volume of the lead solution in the flask (L) and m is the dry mass of biosorbent (g). The removal efficiency of Pb (II) from solution was calculated by the following equation:

$$\text{Removal efficiency (\%)} = \frac{(C0 - Ce)}{Co} x100 \quad (2)$$

2.7 Adsorption Isotherm Models

Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich (D-R) models were used in this study. The experimental results are fitted to these isotherm equations.

Langmuir isotherm model: The Langmuir adsorption model assumes that the maximum adsorption corresponding to a monolayer saturation of heavy metal molecules on the surface of adsorbent and that no lateral interaction between the molecules [19].

The Langmuir isotherm model is given by [20]:

$$\frac{C_e}{q_e} = \frac{1}{Q_m b} + \frac{C_e}{Q_m} \quad (3)$$

Where q_e (mg/g) is the amount of metal adsorbed per unit mass of adsorbent, b (L/mg) is the Langmuir equilibrium constant and Q_m (mg/g) is the maximum adsorption capacity.

Freundlich isotherm model: The Freundlich isotherm model [21], assumes heterogeneous sorption sites with different energies of sorption whereby the stronger sites are occupied first and the binding strength decreases as more sites are occupied The Freundlich isotherm equation is given as:

$$q_e = K_f C_e^{1/n} \quad (4)$$

Where q_e is adsorption capacity at equilibrium (mg/g), k_f is the Freundlich constant (mg $^{1-1/n}$ L $^{1/n}$ g^{-1}) related to the bonding energy.

Temkin isotherm model: The Temkin isotherm [22] is given as:

$$Qad = \frac{RT}{b} \ln A + \frac{RT}{b} \ln Ceq \quad (5)$$

Where RT /b = B is a constant related to the heat of adsorption and b shows the variation of the adsorption energy (J /mol).

Dubinin–Radushkevich isotherm model: Dubinin- Radushkevich (D-R) proposed another equation used in the analysis of isotherms [23]. It has commonly been applied in the following form (Eq. (6) and its linear form can be shown in Eq. (7):

$$q_e = q_m \exp(-K\varepsilon^2) \quad (6)$$

$$\ln q_e = \ln q_m - \beta \varepsilon^2 \quad (7)$$

Where K is a constant related to the adsorption energy, q_e (mg g^{-1}) is the amount of lead ions adsorbed per g of adsorbent and q_m represents the maximum adsorption capacity of adsorbent, β (mol^2J^{-2}) is a constant related to adsorption energy, while ε is the Polanyi potential that can be calculated from Eq. (8):

$$\varepsilon = RT \ln\left[1 + \frac{1}{C_e}\right] \quad (8)$$

The values of β and q_m can be obtained by plotting In q_e vs. ε^2. The energy of adsorption is calculated using the following relation (Eq. (9):

$$E = 1/\sqrt{2\beta} \quad (9)$$

2.8 Adsorption Kinetics

Adsorption kinetics is important in the design and modeling of the adsorption process. Pseudo-first order [24], pseudo-second order [25] and intraparticle diffusion [26] kinetic models were used to study the kinetic adsorption of pb(II) onto Mh.

The pseudo first-order rate can be calculated by the following equation:

$$\log(q_e - q_t) = \log(q_e) - \frac{K_1}{2.303}(t) \quad (10)$$

Values of k_1 were obtained from the plots of log (q_e – q_t) versus t.

The pseudo-second order equation is given as:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e{}^2} + \frac{1}{q_e}(t) \quad (11)$$

Where K_2 is the rate constant for the pseudo-second order kinetics (g mg^{-1} min^{-1}).

The intraparticle diffusion: The most commonly used technique for identifying the mechanism involved in the adsorption process is by fitting an intraparticle diffusion plot. The uptake is proportional to $t^{1/2}$ rather than with the contact time t. The intraparticle diffusion equation is given as:

$$q_t = k_i t^{1/2} \quad (12)$$

Where k_i is the intraparticle diffusion rate constant.

3. RESULTS AND DISCUSSION

3.1 Characterization of the Adsorbent

Elemental analysis: The elemental composition of Molokhia husk (Mh) contains carbon (37.71%) with high percentage compared with hydrogen (5.49%) and nitrogen (1.35%) indicates that carbon-hydrogen groups might be available for adsorption [27] and a small amount of sulfur (0.18%).

FT-IR analysis of the biosorbent: (FTIR) is often used to examine characteristic functional groups of the adsorbents and to determine those groups in the binding of metal ions [28,29].

The FTIR spectrum of the Mh was plotted to obtain information about the nature of functional groups at the surface. The spectra of FTIR of Mh before and after adsorption of Pb(II) were presented in Fig. 1. The spectrum Fig. 1(A) shows a dominant peak at 3423 cm^{-1} attributed to O–H stretching vibrations in hydroxyl groups of cellulose, pectin, hemicellulose and lignin [30,31], involved in hydrogen bonds. Whereas, bands observed at 2924.52 cm^{-1} are an indication of C-H stretch in alkanes groups [32]. Those groups probably act as proton donors that get deprotonated, the hydroxyl group or the carbonyl group adsorb the heavy metal ions [33]. The peak at 2361.41 cm^{-1} denotes the C=C stretching vibrations in alkyne groups [34]. The absorption peaks at 1732.73 cm^{-1} correspond to stretching of carboxyl groups. The peak at 1625.7 cm^{-1} corresponds to the C = O stretch in the carboxylic acids.

After adsorption of Pb (II) ions on Molokhia husk surface as illustrated in Fig. 1 (b), the peak representing the O-H bond was shifted from 3423.99 cm^{-1} to 3432.67 cm^{-1}. The peak representing the C-H shifted from 2924.52 cm^{-1} to 2922.59 cm^{-1}, which is an insignificant change. Whereas the peak of the C =O bond was displaced from 1625.7 cm^{-1} to 1629.55 cm^{-1}, and the peak of the C-O bond was significantly shifted from 1055.84 cm^{-1} to 1058.73 cm^{-1}. Those displacements are an indication that surface complication between Pb^{+2} ions and carboxylic acids functional groups is one of the mechanisms partially responsible for the biosorption of Pb^{+2} by rice straw. A similar trend was reported by other researchers for the removal of heavy metals by agricultural wastes [35-37].

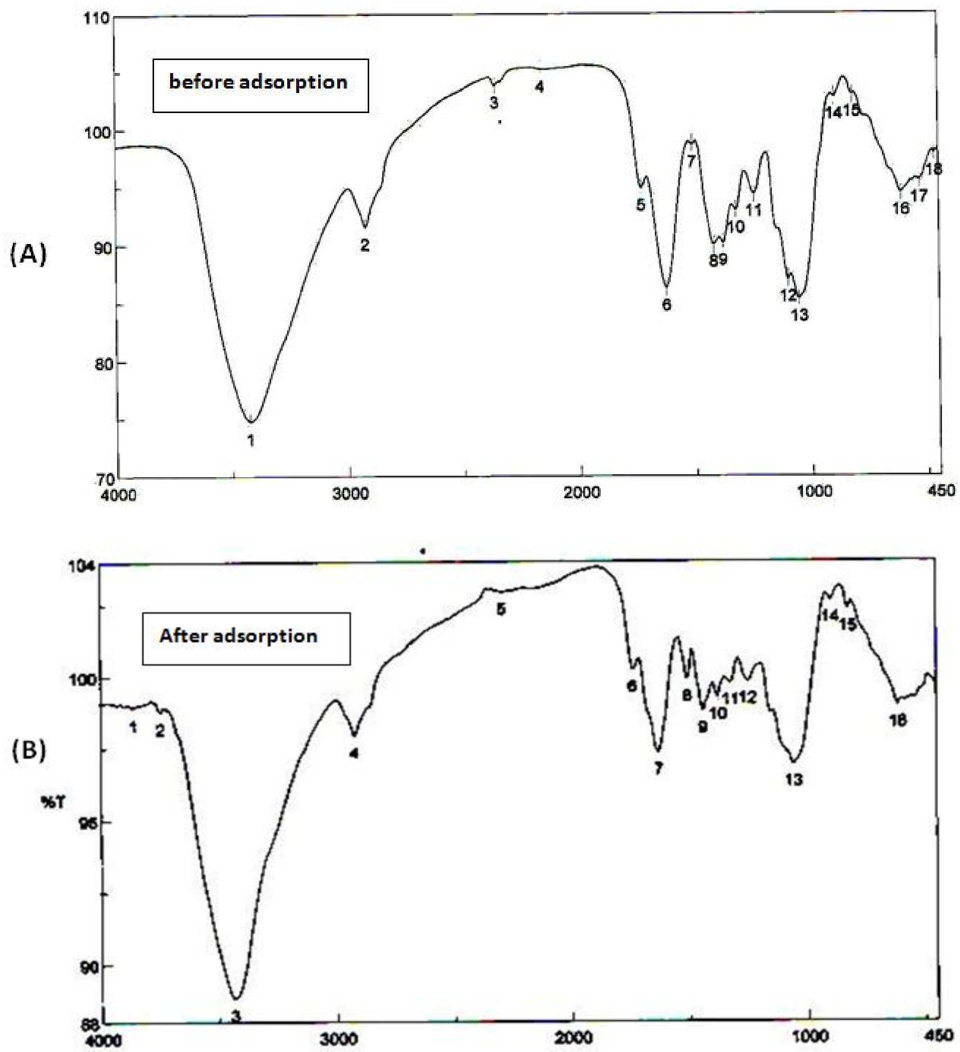

Fig. 1. FTIR Spectra of Molokhia husk (A) before adsorption; (B) after adsorption

FTIR results suggest that hydroxyl and carboxyl groups are presenting on the surface of Molokhia husk biosorbent that are believed to be responsible for metal biosorption [38].

3.2 Optimization of Adsorption Parameters

Optimization of pH, adsorbent dose and initial lead concentrations for the removal of lead ions by Molokhia husk (Mh) biosorbent from aqueous solution are shown in Fig. 2(A) -2(C).

Effect of pH: The pH is an influence on both of surface properties of adsorbent and adsorbate ionization [39]. The effect of pH on the removal of Pb (II) onto Mh (20 mg L^{-1}) was done at room temperature (25 ± 2°C), agitation speed 200 rpm for the minimum contact time required to reach the equilibrium (120 min). As evident from Fig. 2 (A), with an increase in pH of the solution the amount, adsorbed increases till pH 5.0. At low pH values, there are large quantities of H^+ may compete with exchange cations on the surface active sites of Mh [40]. As the pH increases, there are a small amount of H^+ in the solution may less competition with Pb (II) for binding sites.

Effect of adsorbent dose: The effect of adsorbent dose (0.1 – 1 g) on the removal of Pb(II) ions by Mh was studied at pH 5, initial metal ion concentration 20 mg/L and agitation speed 200 rpm is shown in Fig. 2 (B). It can be observed that removal of lead ions increased as the dose increased up to 0.8 g and becomes almost constant. This trend is due to the increase in surface area and active sites available for biosorption of Pb(II) ions onto Molokhia husk increased and consequently more biosorption occurred [41-42]. Hence, 0.8 g of Mh is the optimum dose for removing a considerable quantity of lead ions and was used for the rest of the experiment.

Effect of initial metal ion concentration: The effect of lead ion concentrations on the adsorption capacity of Mh was studied at (5 to 100 mg/L) is shown in Fig. 2 (C). As the concentration increases, the q_e value increased from 0.557 to 9.633 mg/g. At lower metal ion concentration the number of active sites was enough to accommodate the number of metal ion molecules, while the higher values of q_e at higher concentration. The number of active sites was limited and the exchange sites are occupied.

3.3 Adsorption Isotherm

The adsorption data were analyzed in terms of Langmuir, Freundlich, Temkin, and Dubinin-Radushkevich (D-R) isotherm models. The Langmuir adsorption isotherm of Pb (II) by Mh is presented in Fig. 3 (A).

Fig. 3(B) gives results of Freundlich isotherm fitting for adsorption of lead ions onto Molokhia husk with a correlation coefficient (R^2 0.9429) greater than the other models, which indicates that the Freundlich isotherm represents best the adsorption of Pb (II) onto Molokhia husk onto a heterogeneous surface under the experimental conditions. A similar trend has been observed by [43].

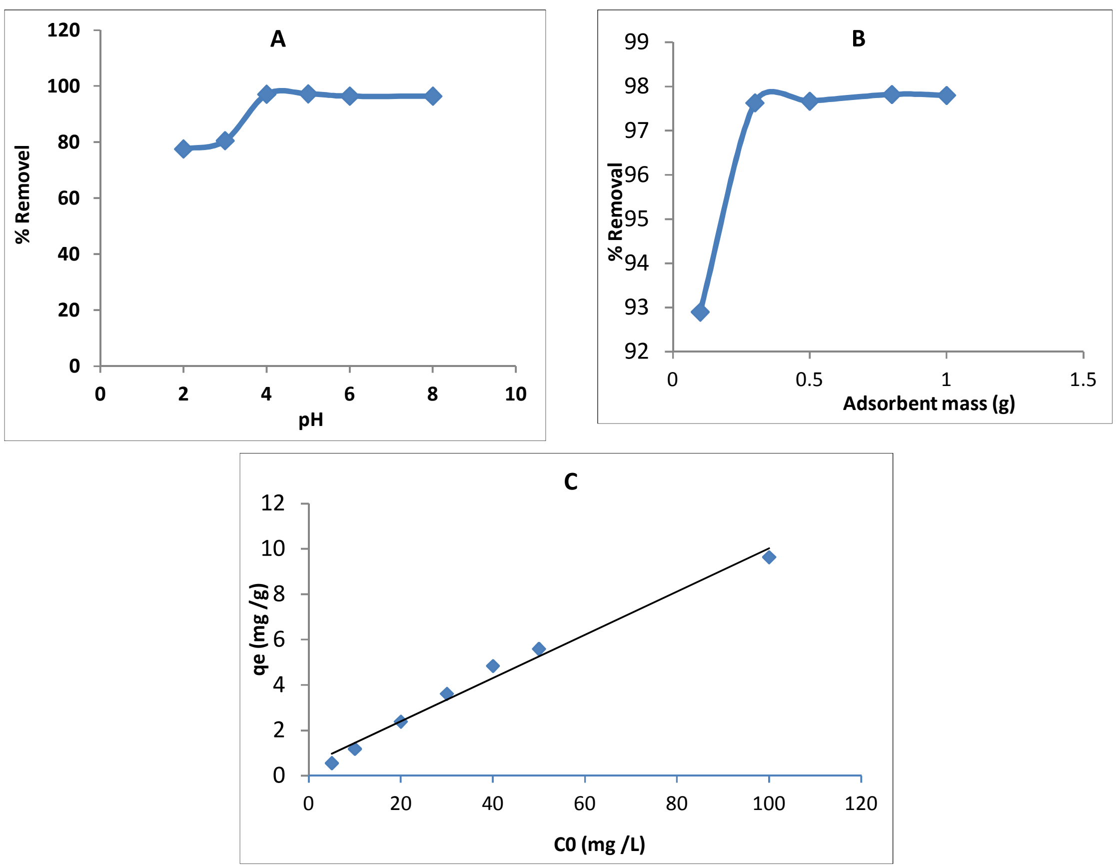

Fig. 2. (A) Effect of pH on the removal of Pb (II) by Mh, (B) Effect of adsorbent dose on the removal of Pb (II) by Mh, (C) Effect of different concentrations for removal of Pb (II) by Mh biosorbent (agitation speed 200 rpm, weight 0.8g, Volume 100 ml and pH 5)

The values of 1/n is related to the intensity of adsorption. If n=1, means linear adsorption; if n <1, means chemical adsorption; if n >1, means physical adsorption. The n value in Freundlich equation was found to be 2.596, this indicates the adsorption of Pb (II) onto Mh physically adsorbed understudied conditions [44].

Fig. 3(C) shows the fitting of the Temkin isotherm. Table 1 summarised the parameters of the four isotherm models. The results showed that the values of the coefficient of determination (R^2) are the highest for Freundlich isotherm when compared with Langmuir, Temkin and Dubinin-Radushkevich isotherm models. It is concluded that the Freundlich isotherm best fitted the experimental data than the other adsorption isotherm models for adsorption of Pb(II) onto Mh.

The fitting of D–R isotherm model is given in Fig. 3 (D). The saturation adsorption capacities q_m obtained using D–R isotherm model for adsorption of Pb (II) onto Mh 7.578 mg/ g. The adsorption energy is a useful indicator of the type of the adsorption process. The adsorption energy E (kJ mol^{-1}) for Mh calculated from D-R equation was found to be < 8 KJ mol^{-1} indicating that the physico-sorption process for removal of Pb (II) onto Mh.

3.4 Effect of Contact Time and Kinetics Modeling

It is essential to assess the effect of contact time required to reach equilibrium. The experiments were performed at a 0.8 g adsorbent dose, initial lead concentrations 20 mg/L as a function of contact time (5-120 min. The effect of contact time for the removal of Pb (II) ions by Mh is shown in Fig. 4 (A).

It was observed that the percentage removal of Pb (II) ions onto Molokhia husk was very high for the first 40 min and finally equilibrium was established after about 90 min. The rapid adsorption at the initial stage of contact time could be attributed to the availability of active sites on the surface of Molokhia husk. Afterward, with the gradual occupation of these sites, the adsorption become less efficient.

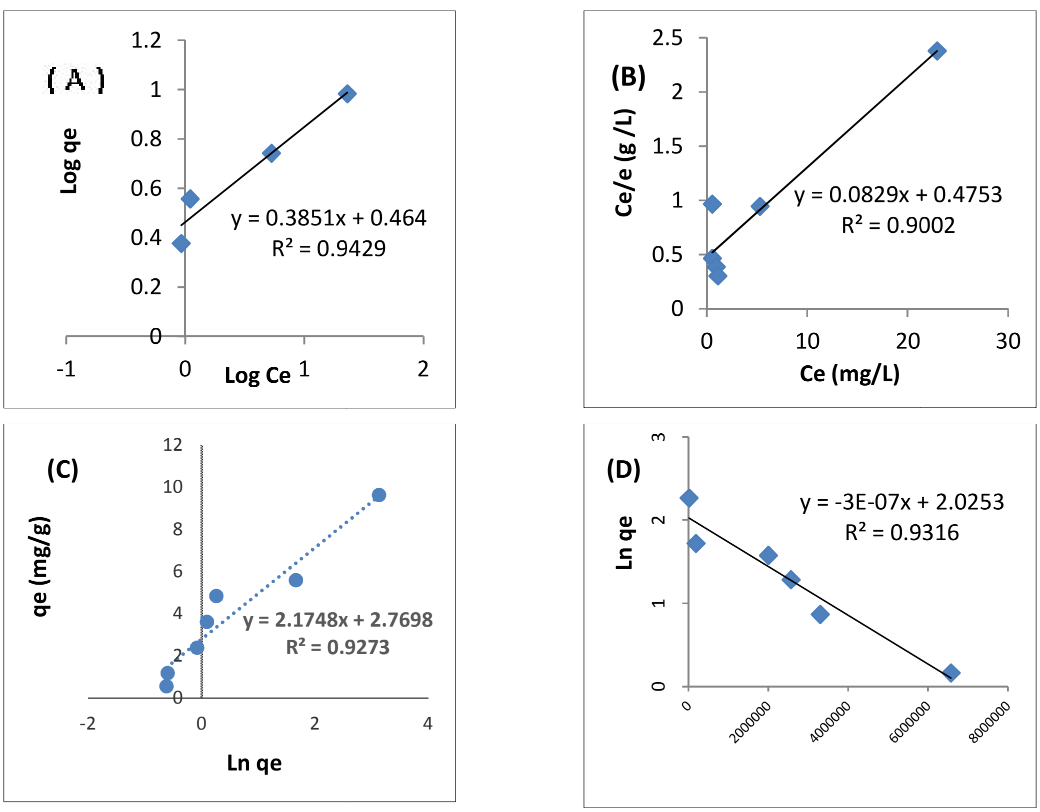

Fig. 3. (A) Freundlich adsorption isotherm for removal of Pb (II) by Mh; (B) Langmiur adsorption isotherm for removal of Pb (II) by Mh; (C) Temkin adsorption isotherm for removal of Pb (II) by Mh; (D) Dubinin- Radushkevich (D-R) isotherm for removal of Pb (II) by Mh

Table 1. Parameters of the Langmuir, Freundlich, Temkin, and Dubinin- Radushkevich (D-R) isotherms for the removal of Pb(II) by Mh

Isotherm	Parameters	Values
Langmuir	q_m (mg/g)	12.062
	b (L/mg)	0.183
	R^2	0.9002
Freundlich	K_f (mg $^{1-1/n}$ L $^{1/n}$ g^{-1})	2.910
	1/n	0.3851
	R^2	0.9429
Temkin	B	2.174
	A (L/g)	3.573
	R^2	0.9273
Dubinin- Radushkevich	q_m (mg g^{-1})	7.578
	E (KJmol^{-1})	1.291
	R^2	0.9316

A similar trend has been reported for different adsorbents that initially adsorption was very fast and then, decreased after certain contact time and finally equilibrium reached, i.e., banana peel [45], orange (*Citrus Cinensis*) waste [46], and walnut hull [47] as a function of time.

3.5 Adsorption Kinetics

The application of Pseudo-first order model to the data of adsorption of Pb (II) on Mh (data not shown) indicated the inapplicability of the model. The adsorption data were also analyzed by Pseudo-second order kinetic model. Table 2 showed the Parameters of the Kinetics models. A plot of t/q_t versus time (t) would give a straight line with a slope of $1/q_e$ and an intercept of $1/(k_2q_e^2)$, if the second-order model is suitable. The plot of t/qt versus t gave linear plot shown in Fig.4 (B). The results showed that adsorption kinetics of Pb (II) on Mh fitted well the pseudo-second-order kinetic model with high correlation coefficient R^2 = 0.9788. A similar trend has been reported by other researchers for removal of lead onto pine bark [48] and peanut shells [49].

The plot of q_t versus t $^{0.5}$ gave a straight line passing through the origin. If the linear plot passes through the origin, then intra-particle diffusion is the rate-limiting step. However, it is not the case in Fig.4 (C), and therefore, the intraparticle diffusion is not the only rate limiting step. This process is complex and may involve more than one mechanism. The similar trend has been obtained by Calvete et al. [50].

The correlation coefficients value for the intraparticle diffusion was lower than those of a pseudo-second order indicating that pseudo-second order is better fitted than the intraparticle diffusion model.

3.6 Comparison of Molokhia Husk Waste with Other Biosorbents

To evaluate the adsorption capacity of Mh and various agricultural waste materials for the removal of Pb (II) is shown in Table 3 as a comparative study. It is observed from the table that Molokhia husk (Mh) has got the highest monolayer adsorption capacity of 12.062 mg/g among all other adsorbents. Therefore, Mh can be used as an effective adsorbent for the removal of Pb (II) from aqueous solution.

Table 2. Parameters of the kinetics models for the removal of Pb (II) by Mh

Kinetic model	Parameter	Value
pseudo-second order	K_2 (g/mg.min)	0.050
	R^2	0.9788
	q_e (mg/g)	2.789
Intraparticle diffusion model	k_i (mg/g min$^{1/2}$)	0.047
	R^2	0.961

Table 3. Monolayer adsorption capacity of Pb(II) ion on various agro-industrial waste materials

Adsorbent	Q max (mg/g)	Reference
Areca Waste	3.57	[51]
Acecia Nilolica	2.51	[52]
Sawdust	6.57	[53]
Banana Peel	2.18	[54]
Rice husk	5.69	[55]
Okra waste	5.00	[56]
Almond shell (raw)	8.08	[57]
Molokhia husk waste	**12.062**	**(Present Study)**

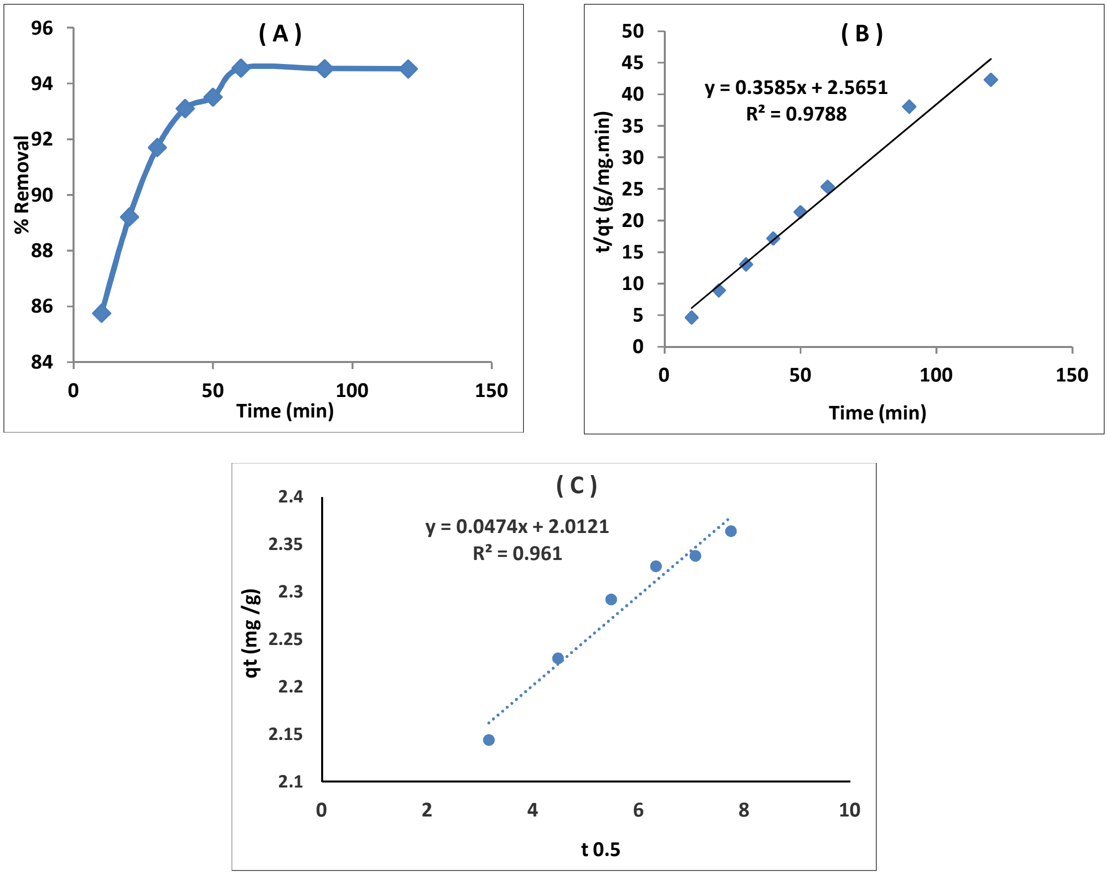

Fig. 4. (A) The effect of contact time for removal of Pb (II) by Mh, (B) Pseudo-second order kinetic model for removal of Pb(II) by Mh, (C) intraparticle diffusion model for removal of Pb (II) by Mh

4. CONCLUSION

The present work revealed that the Molokhia husk (Mh) is a promising and new low cost, effective, cheap adsorbent developed using agricultural waste for removal of Pb (II) ion from aqueous media. The use of this waste materials will not only convert into a low cost and effective adsorbent but also resolves the environmental problem issues as well. Different parameters such as Contact time, initial lead ions concentration, solution pH, and adsorbent dosage were optimized. It was observed that the maximum adsorption capacity 12.062 mg/g occurred at pH 5, adsorbent mass 0.8 g and contact time 90 min. The adsorption of Pb (II) onto Molokhia husk waste was best fitted by Freundlich isotherm with high correlation coefficient R^2 (0.9429). The kinetic studies indicated that the pseudo second-order model was the best one in describing the kinetics data of Pb (II) adsorbed onto Mh. The energy of adsorption was 1.291 KJ/mol indicating that the adsorption of Pb (II) onto Mh is physical in nature.

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COMPETING INTERESTS

Author has declared that no competing interests exist.

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