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Research Article

Biosorption of Pd(II) from Aqueous Solution using Leaves of *Moringa oleifera* as a Low-cost Biosorbent

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Abstract

Palladium ion (Pd(II)) is one of the hazardous metal pollutants commonly found in industrial effluents and poses severe environmental and human health impacts. The present study has chosen the leaves of *Moringa oleifera* (L-MO) as a potential biosorbent for removing Pd(II) from the aqueous solution. Various parameters such as pH, biosorbent dose, and initial Pd(II) concentration were optimized for maximum removal of Pd(II) using standard protocols. The Fourier-transform infrared spectroscopy study was performed to identify the functional groups involved in Pd(II) biosorption mechanism. The analysis of FTIR spectra confirmed the involvement of functional groups such as C–O, C=O, C–H, and O–H in the biosorption of Pd(II) on the surface of L-MO. The biosorption of Pd(II) was highest at pH 6. The sorption equilibrium data were well-fitted with the Langmuir isotherm model ($R^2 = 0.9896$). The highest adsorption capacity of L-MO was 41.15 mg/g. It is concluded that the L-MO could be used as a potentially low-cost novel biosorbent to remove Pd(II) from contaminated water.

Keywords: biosorption, *Moringa oleifera*, FTIR, isotherm models, palladium removal

1. INTRODUCTION

Metal polutan in the environment has raised significant concerns, as certain metals including arsenic, selenium, cobalt, chromium, mercury, nickel, cadmium, lead, and palladium exhibit highly toxic properties [1]. Palladium, an expensive and valuable metal, finds extensive use in various industries such as oil refineries, petrochemical complexes, jewelry, and the electrical equipment manufacturing sector. Given the scarcity of palladium, its increasing demand, and the associated environmental issues; the extraction of this metal from wastewater and solid waste becomes crucial [2]. Unlike organic pollutants, metal pollutants do not biologically degrade [3]. The global reserves of palladium are limited, with a proven quantity of only 67,000 tons, making it one of the most expensive resources. Its abundance in primary ores is extremely low, at approximately 0.01 g/kg. Extracting palladium from ores not only requires substantial amounts of energy and water but also generates significant waste volumes. On the other hand, end-of-life products like spent catalysts and waste-printed circuit boards contain relatively high concentrations of palladium, ranging from 0.1 to 10 g/kg. Therefore, recovering palladium from such end-of-life products holds great significance, offering advantages such as prolonging the lifespan of raw ores, conserving energy and water resources, and reducing waste generation [4]-[6].

In contrast to organic pollutants, metal pollutants lack the ability to biologically degrade. Consequently, various methods have been developed to eliminate metal ions from aqueous solutions, including oxidation [7], reduction [8], precipitation [9], membrane filtration [10], ion exchange [11], and adsorption [12]. Among these approaches, adsorption has emerged as a promising process for metal ion removal from water and wastewater due to its simplicity, cost-effectiveness, and suitability. Because of that, exploiting simple, novel, and highly efficient adsorbents is crucial in this context. However, commercial adsorbents are often expensive and not environmentally friendly. As a result, researchers today are directing their attention towards utilizing biological waste to create adsorbents. This approach not only transforms biological waste into valuable resources but also addresses the pollution issues associated with such waste [13].

Biosorption processes, utilizing plant-based sources, offer an environmentally friendly alternative due to their biodegradability and non-

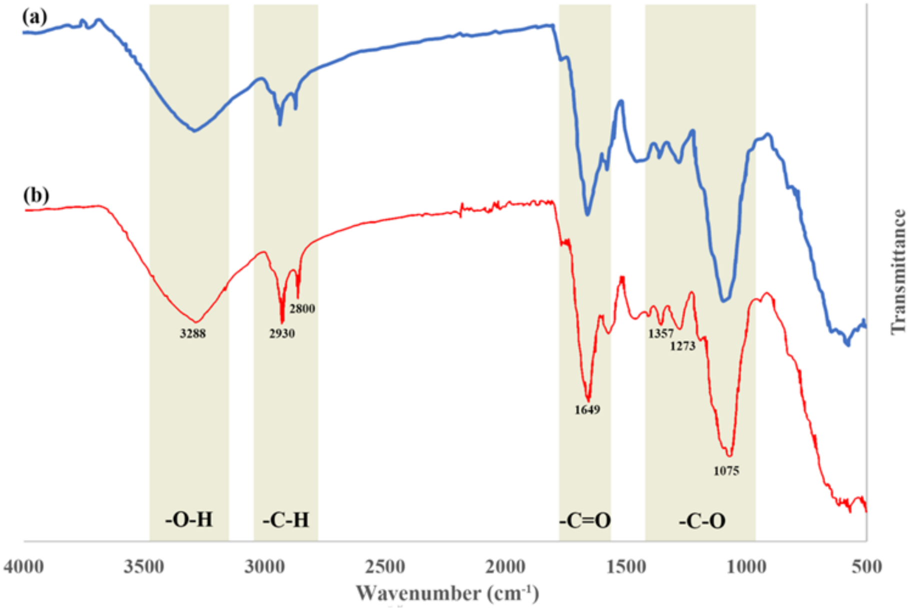

Figure 1. FTIR spectrum of (a) *Moringa oleifera* leaf; (b) *Moringa oleifera* leaf loaded Pd(II).

toxic nature. This technology possesses several advantageous characteristics such as affordability, availability, reusability, and biodegradability. Various materials, including algae, fungi, agricultural waste and residues, and bacterial strains, have been utilized as biosorbents for removing pollutants from aqueous solutions. While biosorption processes have been known for some time, they have gained recognition in recent decades as low-cost and eco-friendly technology [14]. Biosorption involves the uptake of chemical species from the aqueous solutions by biological materials through biological, physical, and chemical mechanisms. Biomaterials can accumulate pollutants from wastewater through passive binding to non-living cells, metabolically mediated processes within living cells, or physicochemical pathways. The mechanisms enabling metal uptake include extracellular accumulation, intracellular accumulation, and sorption/precipitation on the cell surface. Various types of plant-based substances, such as agricultural wastes [15], barks [16], leaves, seed husks [17], nutshells [18], fruit pods [19], and fruit shells [20], have been extensively reported as effective adsorbents in biosorption processes.

Moringa oleifera, belonging to the monogeneric family Moringaceae, is extensively cultivated and naturalized in various tropical regions. The leaves of *M. oleifera* contain a unique group of glycoside compounds called glucosinolates and isothiocyanates, which are the main chemical ingredients present in different parts of the plant [21]. Specifically, 4-(*R*-L-rhamnopyranosyloxy)-benzylglucosinolate and its three monoacetyl isomers are found in significant amounts in *M. oleifera* leaves. Additionally, quercetin-3-*O*-glucoside, quercetin-3-*O*-(6″-malonyl-glucoside), kaempferol-3-*O*-glucoside, kaempferol-3-*O*-(6″-malonyl-glucoside), 3-caffeoylquinic acid, and 5-caffeoylquinic acid are also identified, albeit in lower quantities [22]. The presence of polar groups on the surface of *M. oleifera* leaf powder is likely to enhance its cation exchange capacity.

The objective of this study is to investigate the biosorption of Pd(II) from aqueous solutions using leaves of *M. oleifera*. The optimal conditions for biosorption, including pH, biosorbent dosage, and initial metal ion concentration, have been experimentally examined. Furthermore, an attempt has been made to analyze the equilibrium adsorption isotherms based on the Langmuir and Freundlich models in order to gain insights into the adsorption process.

2. MATERIALS AND METHODS

2.1. Collecting and Processing Biosorbent

The leaves of *M. oleifera* (L-MO) were collected from Maringá city, Paraná State, Brazil. The L-MO samples were washed with deionized water for 5 min to remove any suspended materials on their surface and dried in an oven at 70 °C for 72 h. The dried samples were ground using a domestic blender and sieved through a 200 µm stainless steel sieve. The biosorbent samples were stored in glass bottles for further studies.

2.2. Preparation of Pd(II) Ion Solution

The stock solutions of Pd(II) of 1000 mg/L concentration were prepared by dissolving the appropriate mass of $PdCl_2$ (98%, Merck, Germany) in deionized water (resistivity, 18.2 MΩ cm). Deionized water was used for the preparation of these solutions as well as any other purpose.

2.3. FTIR Characterization

The as-received and dried samples of *M. oleifera* leaves were characterized by Fourier transform infrared spectroscopy (Nicolet Nexus 670 FTIR, Waltham, MA, USA) within the range of 4000–400 cm^{-1} using the KBr standard method. Spectra were collected for the pristine materials and the exposed ones.

2.4. Sorption Experiments

In this work, the sorption experiments, such as the effects of pH, contact time, biosorbent dosages, and the initial concentration of metallic ions, were performed by the batch system. The effect of different pH on Pd(II) sorption of biosorbent was estimated by adjusting the different pH (3, 4, 5, 6, 7, and 8) of the 50 mL of Pd(II) solutions (60 mg/L) by using 0.1 M HCl and 0.1 M NaOH with the biosorbent dose of 0.4 g and contact time 30 min. To optimize the dose of biosorbent in removing Pd (II), the different doses (0.1, 0.2, 0.3, 0.4, 0.5, 0.6, and 0.7 g) of biosorbent were used in 50 mL of Pd (II) solution (60 mg/L). The effect of initial Pd(II) concentrations on sorption of biosorbent was conducted in 50 mL solution of different Pd(II) concentrations (20–200 mg/L) using 0.4 g biosorbent. All experiments were conducted twice in a mechanical shaker (200 rpm) at room temperature, and mean value was considered for data analysis. The concentrations of Pd(II) ions *Cf* (mg/L) were determined by using the FAAS technique (Thermo Scientific).

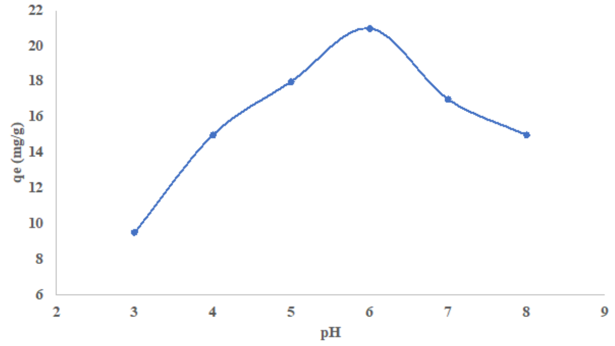

Figure 2. Effect of pH on the Pd(II) adsorption using the biosorbent.

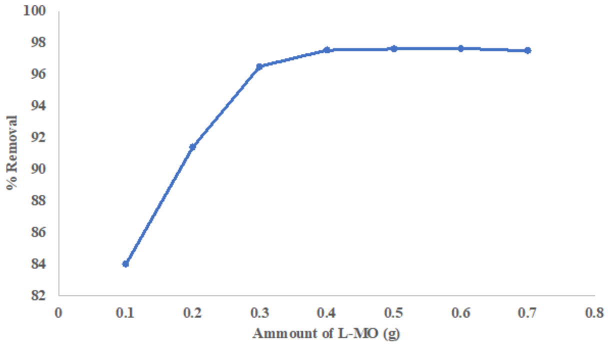

Figure 3. Effect of biosorbent dose for the adsorption of Pd(II).

The amount of metal ion sorbed onto the L-MO, q_e, was computed by the following equation 1 [17]:

$$q_e = \frac{(C_f - C_i)V}{m} \tag{1}$$

where C_f (mg/L) and C_i (mg/L) are the initial and equilibrium of Pd(II) concentration, V (L) is the volume of the adsorbate solution, and m (g) is the adsorbent's mass. In this case, C_i = 60 mg/L, V = 0.05 L, and m = 0.4 g.

The sorption isotherm for the L-MO was fitted with Langmuir (Eq. 2) and Freundlich (Eq. 3) models [23], respectively:

$$q_e = \frac{Q_m K_L C_e}{1 + K_L C_e} \tag{2}$$

$$q_e = K_F C_e^{\frac{1}{n}} \tag{3}$$

where Q_m is the maximum adsorption capacity (mg/g) and K_L is the Langmuir constant (L/mg), q_e is the amount of each heavy metal sorbed per unit mass of biosorbents, C_e is the amount of the heavy metals in solution at the equilibrium, K_F (mg/g·$(mg/L)^{-1/n}$) and n are Freundlich constants.

3. RESULTS AND DISCUSSIONS

3.1. FTIR Study and Pd(II) Removal Mechanism

The characterization of the biosorbent surface is crucial for understanding the biosorbent-metal interactions and biosorption mechanisms. FTIR spectroscopy is used to identify functional groups

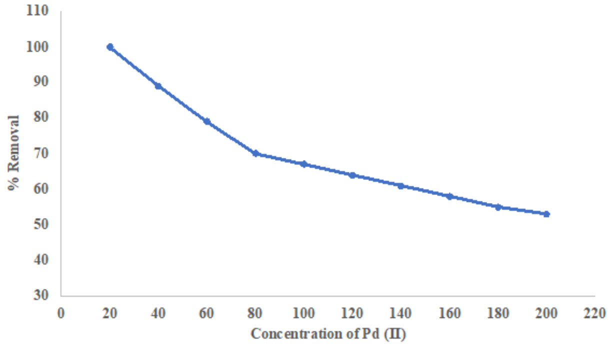

Figure 4. Effect of initial concentration of Pd(II) ions adsorption.

on the surface of the biosorbent material (L-MO) and the interactions between the biosorbent and Pd (II). The data obtained from the FTIR spectra of L-MO before and after Pd(II) adsorption show differences in the peak absorbance of functional groups (Figure 1). Plant biomass such as leaves, flowers, bark, and pods contain lignin, cellulose, and hemicellulose, which provide various functional groups that have a strong tendency to adsorb positively charged heavy metals. The variation in peak absorbance values is caused by the interaction between the functional groups present on L-MO and the target contaminant. The spectral shifts indicate that L-MO is a useful biosorbent for the removal of heavy metal ions. It is also observed that the absorbance peaks at 1075, 1273, and 1357 cm^{-1} indicate the presence of C–O functional groups. The absorbance of L-MO at 1649 cm^{-1} represents the C=O group. Similarly, the absorbance peaks at 2800–2930 cm^{-1} indicate the C–H bonds of methyl, methylene, and methane [24]. In Figure 1 (a), the stretching of O–H is observed at 3288 cm^{-1} on the surface of L-MO. The higher variation in the spectrum before and after Pd(II) attachment (Figure 1) indicates more interactions between the adsorbent surface and the metal ions. The main functional groups contributing to the adsorption of Pd(II) through L-MO are –C–O, –C=O, C–H, and –OH.

3.2. Process Parameters Optimization

3.2.1. pH

The pH of water, size of biosorbent particles, temperature, ionic strength, presence of competitive inorganic ions, stirring time, and adsorbent dosage are factors that control the removal of target contaminants. The pH of the solution is an important variable that controls the biosorption process for removing contaminants from aqueous solutions. The influence of solution pH on the removal of Pd(II) was determined at a Pd(II) concentration of 60 mg/L, a dosage of 0.4 g, and the selected pH values were: 3, 4, 5, 6, 7, and 8. The maximum biosorption of Pd(II) was observed at a pH range of 6 (Figure 2). A similar trend of Pb, Cd, Co, and Ni biosorption as a function of solution pH was observed by Abatal et al., where they used seeds and leaves of *M. oleifera* to remove Pb, Cd, Co, and Ni from aqueous solutions. The results showed that the removal of Pb, Cd, Co, and Ni using *M. oleifera* seeds and leaves was effective at pH 4 and 6, respectively [17]. Further increases in pH reduced the removal of metal ions from the solution. The variations in Pd(II) removal due to pH changes can be attributed to the different functional groups present on the biosorbent surface involved in the removal process. The pH effects at lower and higher pH values can be explained by the competition between H_3O^+ and metal ions in the solution. There is a decrease in H_3O^+ at higher pH, which supports the occupation of absorption sites by Pd(II) metal ions. On the other hand, at low pH, H_3O^+ increases and negatively affects the removal of other positively charged ions by the biosorbent surface [25].

3.2.2. Biosorbent Dosages

The effect of L-MO dose on the removal of Pd (II) is shown in Figure 3, which illustrates the biosorption of Pd(II) ion by changing the biosorbent dose from 0.1 to 0.7 g with pH 6, initial concentration 60 mg/L, and the contact time 30 min. As inferred from Figure 3, for a fixed metal

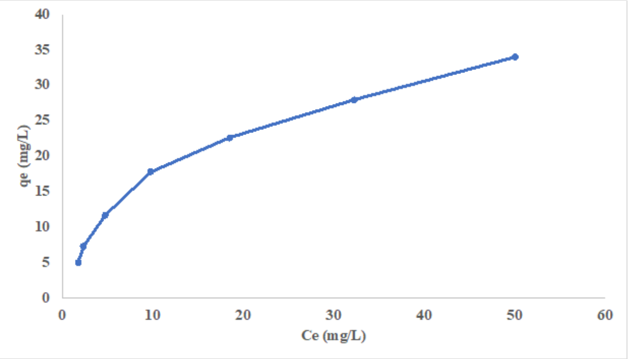

Figure 4. Adsorption isotherms of Pd(II) onto the biosorbent.

Table 1. Isotherm parameters for Pd (II) biosorption, using L-MO as biosorbent.

Model	Values
Langmuir	
Qm (mg/g)	41.15 ± 2.47
K_L (L/mg)	0.31 ± 0.05
R^2	0.9896
Freundlich	
K_F (mg/g)	4.44 ± 1.15
n (g/L)	1.83 ± 0.27
R^2	0.9718

initial concentration, increasing the sorbent dose provided greater surface area and availability of more active sites, thus leading to the enhancement of metal ion uptake. Adsorption increased from 84.00 to 97.54% with an increase in adsorbent dose. Maximum removal of Pd (II) was observed with an adsorbent dose of 0.4 g and thereafter a slow increase in the percentage removal was seen reaching a constant value with respect to the biosorbent dosage.

3.2.3. Initial Concentration of Pd(II) Ions

The effect of the concentration of Pd(II) was investigated by repeating experiments by using different initial concentrations (20–200 mg/L) of Pd (II). For the metal concentrations studied, it was observed that biosorption efficiency decreased as metal ion concentrations increased, indicating saturation. This was expected since, for lower metal ion concentrations, the ratio of surface-active sites to the total amount of metal ions is high, and, thus, all metal ions can more easily interact with the biosorbent and be removed from the solution. According to the results as shown in Figure 4, reveals that removal (%) of Pd(II) is decreased with an increase in Pd(II) concentration which is attributed to the higher number of metal ions at a higher concentration than the active sites at a given dosage. These results are in close agreement with the findings of Imran et al. [26]. It is reported that the saturation of the biosorbent surface depends on the initial concentration of metal [27].

3.3. Isotherm Study

In order to optimize the design of the sorption system to remove metal ions from effluents, it is important to establish the most appropriate correlation for the equilibrium isotherms. In these studies, the adsorption data were analyzed by the Langmuir and Freundlich isotherm models. The isotherms for adsorption of Pd(II) on L-MO at optimized experimental conditions, pH 6, biosorbent dose of 0.4 g/60 mL, contact time of 30 min, and temperature of 25 °C are presented in Figure 5. The corresponding Langmuir and Freundlich constants calculated are included in Table 1. The Langmuir model is valid for monolayer adsorption with a limited number of identical sorption sites on the surface of the biosorbent, which depends on the active sites with uniform energy for adsorption [28]. In the Langmuir model, all adsorption sites are equally capable of holding the metal (Pd(II)). In Eq. (2), parameters Qm and qe are unknown and their values were calculated from the linearized form of Eq. 2. The values of Qm (41.15 mg/g) and KL (Table 1) were measured from the slope and intercept of the curve Ce/qe vs Ce.

Table 2. Comparison of various biosorbents for removal Pd (II) from aqueous solution.

No	Biosorbent	Qm (mg/g)	Reference
1	Silica nanocapsule	51.28	[30]
2	Boron-nitrogen co-doping walnut shell biochar	47.18	[31]
3	*Lagerstroemia speciosa* leaf	46.30	[32]
4	Activated carbon coated with chitosan	43.48	[33]
5	*Terminalia catappa* L. leaf	41.86	[34]
6	*Moringa oleifera* leaf	41.15	Present study
7	*Racomitrium lanuginosum*	37.20	[35]

The Freundlich isotherm model, which assumes a heterogeneous sorption surface, is commonly used to describe the equilibrium adsorption isotherm (Eq. 3). In this model, the Freundlich distribution coefficient or adsorption capacity is represented by K_F, and the empirical parameter 1/n is known as the Freundlich constant, which indicates the medium's heterogeneity and adsorption intensity. The value of n is typically greater than one. A higher value of n in the Freundlich model indicates more favorable adsorption and greater heterogeneity of the particle surface [29]. The Freundlich parameters K_F (4.44 mg/g) and n (1.83 g/L) are obtained from the slope and intercept of the plot of log qe vs log Ce. While the Freundlich model is widely used in water and wastewater treatment, it does not provide information about the monolayer adsorption capacity. It assumes that the stronger adsorption sites are filled first and that there is a decrease in the binding energy of the adsorption sites as they become occupied. The high values of the Langmuir model correlation coefficient (R^2 = 0.9896) in Table 1 indicate that the Langmuir model provides a better representation of Pd(II) biosorption by L-MO, while the Freundlich model (R^2 = 0.9718) adequately fits the experimental equilibrium data.

Table 2 shows the reported removal capacities for other biosorbents which are collected to compare with the herein-studied material. The biosorption capacities of different biosorbents show the different sorption capacities and is mainly due to the type, surface structure, morphology and the type of functional groups present in the biosorbent, and the biosorption capacities are shown in Table 2 and the maximum biosorption capacity (qmax) of the biosorbent is obtained from Langmuir isotherm. The values shown in Table 2 indicate that the adsorption capacities of heavy metals on the leaves of *M. oleifera* are within the range of values reported for other biosorbents.

4. CONCLUSIONS

In this study, we studied the biosorption mechanism and isotherms for the removal of Pd(II) using *Moringa oleifera* leaves (L-MO) as the biosorbent. The results revealed that the efficiency (%) of Pd(II) removal decreased as the concentration of Pd(II) increased (20–200 mg/L). This can be attributed to a higher number of metal ions at higher concentrations, surpassing the active sites available at a given dosage. The maximum biosorption capacity of Pd(II) ions per unit mass of the adsorbent was determined to be 41.15 mg/g, while the maximum removal of Pd(II) reached 97.54% at an adsorbent dosage of 0.4 g. The Langmuir model provided a better fit for the experimental isotherm data. The analysis of FTIR spectra confirmed the involvement of functional groups such as C–O, C=O, C–H, and O–H in the biosorption of Pd(II) on the surface of L-MO, highlighting the suitability of L-MO as a cost-effective and environmentally friendly material. Based on these results, it can be concluded that the dried biomass of *Moringa oleifera* leaves exhibits a strong affinity for the removal of heavy metals, particularly Pd(II). Therefore, it can be effectively utilized for the removal of heavy metals from various industrial wastes containing such contaminants.

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Author Contributions

A.D. and B.C. conceived and planned the experiments; B.C. wrote the manuscript with input from all authors. A.D., B.C., and D.M. contributed to the characterization of the samples and the interpretation of the results.

Conflicts of Interest

The authors declare no conflict of interest.

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