



***Terminalia arjuna* Bark Biosorbent for Efficient Lead (II) Removal from Synthetic Wastewater**

Sachin Kumari^{a++*}, Sushila Singh^a, Vinod Sangwan^b and Indu Rani^a

^a Department of Chemistry, CCS Haryana Agricultural University, Hisar 125004, India.

^b Department of Biochemistry, CCS Haryana Agricultural University, Hisar 125004, India.

Authors' contributions

This work was carried out in collaboration among all authors. Author SK prepared the manuscript, designed and executed the study. Author SS helped in spectral analysis. Author IR collected plant bark and prepared the biosorbent. Author VS helped in lead ion analysis. All authors read and approved the final manuscript.

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ABSTRACT

Aim: Heavy metal such as lead ions is toxic and its entry in water environment is a threat to human as well as aquatic life. Plant based adsorbents have attracted a great attention as ecofriendly and low-cost adsorbent. The goal of this study was to prepare adsorbent from *Terminalia arjuna* bark using chemicals named hydrochloride followed by sodium hydroxide for lead ions removal.

Study Design: The research is experimental in nature.

Place and Duration of Study: This research was conducted in Department of Chemistry and Biochemistry at CCSHAU, Hisar in year 2022-23.

⁺⁺ Assistant Professor;

*Corresponding author: E-mail: sachindalal@hau.ac.in;

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Methodology: The functionality, surface morphology, and elemental analysis of that biosorbent was analysed by FTIR, FE-SEM with EDX respectively. The bark samples were treated with 400 mL of 0.1 M hydrochloride and 400 mL of 0.01 M sodium hydroxide, respectively, before being used as an adsorbent. Batch studies were done for biosorption of heavy metal ions. The maximum biosorption capacity of lead ions were optimized by varying the pH (2-8), lead ions concentration (10- 100 mg/L), biosorbent dose (0.01-0.04 g/50mL), and contact time (10–60 minutes).

Results: The maximum removal efficiency of about 71% was obtained at 6 pH having 0.03g of biosorbent at 27°C for 60 minutes. The Freundlich adsorption isotherms and pseudo first order kinetics models were the most suitable for the biosorption of lead ions and the maximum adsorption capacity was 125 mgg⁻¹ at optimized time 60 minutes.

Conclusion: The finding indicated that *Terminalia arjuna* bark based biosorbents would potentially be an inexpensive and environment friendly for lead ions removal from water environment.

Keywords: Biosorption; *Terminalia arjuna*; lead ions; adsorption.

1. INTRODUCTION

Water is a necessary component of life, civilization, and the earth's environment. Increase water pollution is one of serious concerns. Pollution from heavy metals has grown to be a significant issue in the current scenario [1]. However, due to unregulated release of all of these contaminants in the water, values of concentration of Cd (II), Ni(II), and Pb(II) up to 0.01 mg/L, 0.20 mg/L, and 0.006 mg/L were detected in different effluents [2]. The release of heavy metals into water bodies poses a major health risk since elevated levels of these metals in water can harm both the environment and human health [3-4]. The mining industry and industrial wastes, motor vehicle emissions, lead-acid batteries, paints, fertilizers, soil erosion, leaching of heavy metals, treated timber ageing water supply infrastructure, and micro plastics floating in the world's oceans are common sources of heavy metal contamination in this environment [5]. Lead ions, one of the heavy metals present in wastewater, is regarded as one of the most hazardous pollutants and results in hypertension, emphysema, testicular distortion, and kidney damage [6]. Conventional methods including membrane, separation, evaporation, ion exchange, chemical precipitation, and flocculation that are used to remove heavy metal ions are inefficient and expensive [7-8]. Compared to the previously described approaches, the use of biosorbent has increased in the past several decades due to its high efficiency, simplicity, affordability, and environmental friendliness [9-10]. The aim of this study was to support waste control by waste concept for develop *Terminalia arjuna* bark based biosorbent and to assess its adsorption capacity towards lead ion removal.

2. MATERIALS AND METHODS

2.1 Material Preparation and Characterization

Terminalia arjuna bark was collected from research farm area CCSHAU Hisar, Haryana, India, in during the fall season from May to July. The bark was shade-dried for a period of thirty day and was manually ground with a mortar and pestle and sieved. Before being utilised as an adsorbent, the powdered samples of arjuna bark were treated respectively, with 400 mL of 0.1 M hydrochloride (HCl, Hi-media) followed by 400 mL of 0.01 M sodium hydroxide (NaOH, Hi-media) for six hours of stirring at 60°C, the biomass was filtered out and given a thorough wash with distilled water. Washing proceeded until most of the colour disappeared. The biosorbent material was carefully cleaned with deionized water and then dried in an electric oven at 80 °C for ten days to make moisture free. The resultant biosorbents were stored in a desiccator for further use in present research work. For the experiment, the stock solution of concentration 1000 mg/L of Pb²⁺, was prepared by dissolving 1.598g of lead nitrate (Pb (NO₃)₂, Hi-media) in one litre of distilled water. The characteristics of the biosorbent before and after adsorption was examined by Fourier Transform Infrared spectrometer in the range of 4000–400 cm⁻¹ and scanning electron microscope with an energy dispersive spectrometer to determine changes in functional groups and morphology. Lead (II) ion concentration in the aqueous solution was measured using an air acetylene burner-equipped atomic absorption spectrometer (AAS) with a slit of 0.7 nm, analytical wavelength of 228.8 nm, the hollow cathode lamp was run at 1.8 L/min of gas flow. A digital pH meter was used for all pH measurements.

2.2 Adsorption Studies

The adsorption process was examined using batch procedures in order to determine the equilibrium data and rate of lead ion removal. The lead ion (%) removal and adsorption capacity were calculated using the following equations:

$$\% \text{ removal} = \frac{C_o - C_e}{C_o} \times 100 \text{ Eq.(1)}$$

$$q_e = \frac{(C_o - C_e)V}{m} \text{ Eq. (2)}$$

Whereas; C_o (mg/L) & C_e (mg/g) are initial and equilibrium concentrations of the adsorbate, q_e (mg/g) is amount of metal ion molecule adsorbed per unit of the adsorbent at equilibrium, V (mL) is the volume of metal ion, m (g) is mass of biosorbent.

2.3 Adsorption Kinetics Modelling

Pseudo-first-order model and pseudo-second-order model were used to interpret the experimental data for adsorption experiments and analyse adsorption mechanism of lead ions on *Terminalia arjuna* bark. The pseudo-first-order kinetic model described that the adsorption process follows a physisorption kinetics and the pseudo-second-order kinetic model described that the adsorption process follows a chemisorption kinetics.

Pseudo first order is in linear form is represented in equation 3.

$$\log(q_e - q_t) = \log q_e - \left(\frac{k_1}{2.303}\right)t \text{ Eq.(3)}$$

Here, q_e & q_t (mg/g) are adsorption capacity at equilibrium time and at time t (min.), respectively, k_1 (min^{-1}) pseudo first order rate constant, the value of k_1 , q_e , R^2 calculated from linear plot between $\log(q_e - q_t)$ vs t .

Pseudo second order is in linear form is represented in equation 4.

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \text{ Eq.(4)}$$

Here, k_2 (g/mg min^{-1}) is rate constant of pseudo second order. By plotting graph between t/q_t vs t we can calculate k_2 , q_e , R^2 values. The q_e (exp) values from the adsorption experiment are compared to the q_e (cal) value calculated using the kinetic models.

2.4 Adsorption isotherm Modelling

Langmuir and Freundlich models were comparatively explored to determine the equilibrium adsorption capacity data and analyse adsorption mechanisms (e.g. monolayer and multilayer adsorption).

2.4.1 Langmuir isotherm Model

Linear form of Langmuir equation is given in equation 5.

$$\frac{C_e}{q_e} = \frac{1}{q_{max} \cdot b} + \frac{C_e}{q_{max}} \text{ Eq. (5)}$$

Nonlinear form of Langmuir equation is given in equation 6.

$$q_{max} = \frac{q_{max} b C_e}{1 + b C_e} \text{ Eq. (6)}$$

Whereas, q_{max} (mg/g) is maximum monolayer adsorption capacity, b (L/g) is Langmuir constant.

2.4.2 Freundlich isotherm model

Equation 7&8 provides the generic formula for the Freundlich equation. The adsorbent's adsorption capacity is denoted by k_f , its adsorption intensity is shown by the Freundlich constant $1/n$, and its equilibrium concentration is represented by C_e .

$$q_e = k_f C_e^{1/n} \text{ Eq. (7)}$$

One taking logarithm of Eq. (7) shows linear form of Freundlich isotherm model can be written as Eq. (8).

$$\log q_e = \frac{1}{n} \log C_e + \log k_f \text{ Eq. (8)}$$

where, q_e is the total amount of lead ion adsorbed/total weight of adsorbents (mg/g). k_f (mg/g) is Freundlich constant, n is heterogeneity factor that relates to intensity of adsorption.

2.5 Thermodynamics Analysis

To understand the adsorption thermodynamics on arjuna bark biosorbent, the change in Gibbs free energy (ΔG^0), enthalpy (ΔH^0), and entropy (ΔS^0) were calculated from equation, (9-11).

$$\Delta G^0 = -RT \ln k_d \text{ Eq.(9)}$$

$$k_d = \frac{C_a}{C_e} \quad \text{Eq.(10)}$$

$$\log k_d = \frac{\Delta H^0}{RT} - \frac{\Delta S^0}{R} \quad \text{Eq.(11)}$$

Here, k_d is equilibrium constant, R gas constant (8.314 J/mol/K), T is temperature in K, C_a is amount of adsorbate on adsorbent surface at equilibrium. ΔH^0 & ΔS^0 was determined from linear plot between $\ln k_d$ vs $1/T$.

3 RESULTS AND DISCUSSION

3.1 FTIR and FESEM with EDS

The FTIR analysis of biosorbent provides insight into the interactions between functional groups during the adsorption process. FTIR plots for raw biosorbent and Pb(II) loaded biosorbents exhibit a shift in the wavenumbers of the main peaks as clearly shown in Fig. 1. The microstructure and surface morphology of the unloaded biosorbent and Pb (II) loaded biosorbents is done using the FESEM equipped with EDS. As shown in Fig. 2(a) arjuna bark has porous structure and after adsorption of cadmium ions porosity was degraded (Fig. 2(b)).

3.2 Adsorption Studies

To determine how arjuna bark biosorbent could effectively remove lead ions under different conditions, various batch experiments were investigated at 27°C. The following sections have

covered a number of aspects that affect biosorbent's efficiency in removing lead ions.

3.2.1 Influence of pH

The effect of pH was studied by varying the pH (2-8) in different sets of 250 mL polypropylene flasks that containing 50 mL of lead ion solution of known concentrations followed by the addition of 30 g of powdered arjuna bark biosorbent. As shown in Fig. 3(a) per cent removal of lead ion was increased with increase in the pH of the solution. At low pH values, adsorbent surface is highly protonated [11-12]. At higher pH values, the degree of protonation on the surface of adsorbent is reduced continuously. Thus, for adsorption of lead ion; studied pH range was observed at pH 6. Eventually at basic region, lead ion adsorption rate dropped due to formation of soluble hydroxylated groups [13].

3.2.2 Influence of contact time

The impact of contact times on the biosorbent's ability to remove lead ions was investigated by adjusting the contact time between 10 and 60 minutes at different biosorbent dose of 10 g/50 mL (10-100 mg/L lead), pH 6, and temperature of 27 °C. It was found that as contact time increased it also increased the percent removal of lead ions as shown in Fig. 3(b). Removal of lead ions was increased continuously up to initial 60 min. Further there is no significant effect on lead ion removal on increasing time up to 70min. At that time, equilibrium is attained and active sites get saturated [14-16].

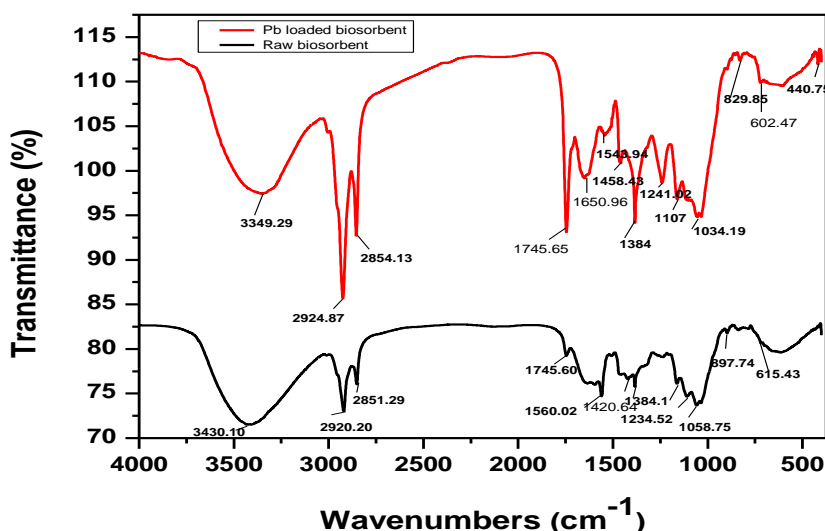


Fig. 1. FTIR spectra of biosorbent before and after metal adsorption

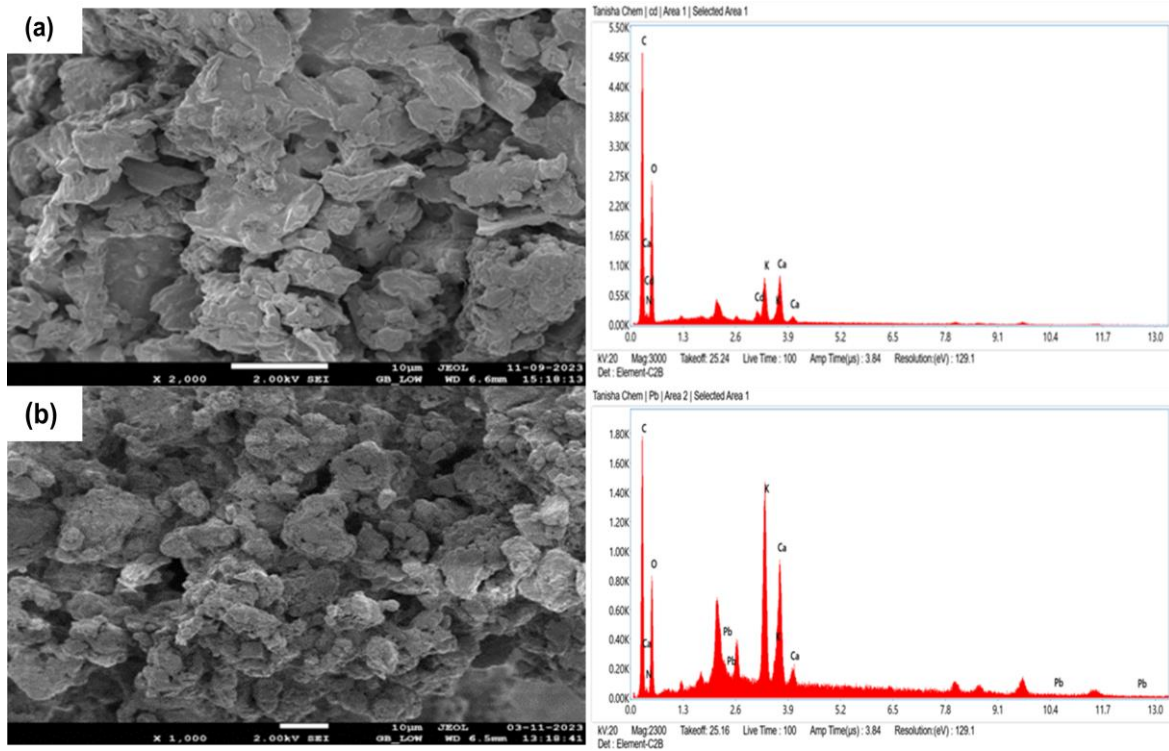


Fig. 2. FE-SEM micrograph of biosorbent- (a) Raw, (b) Pb loaded biosorbent

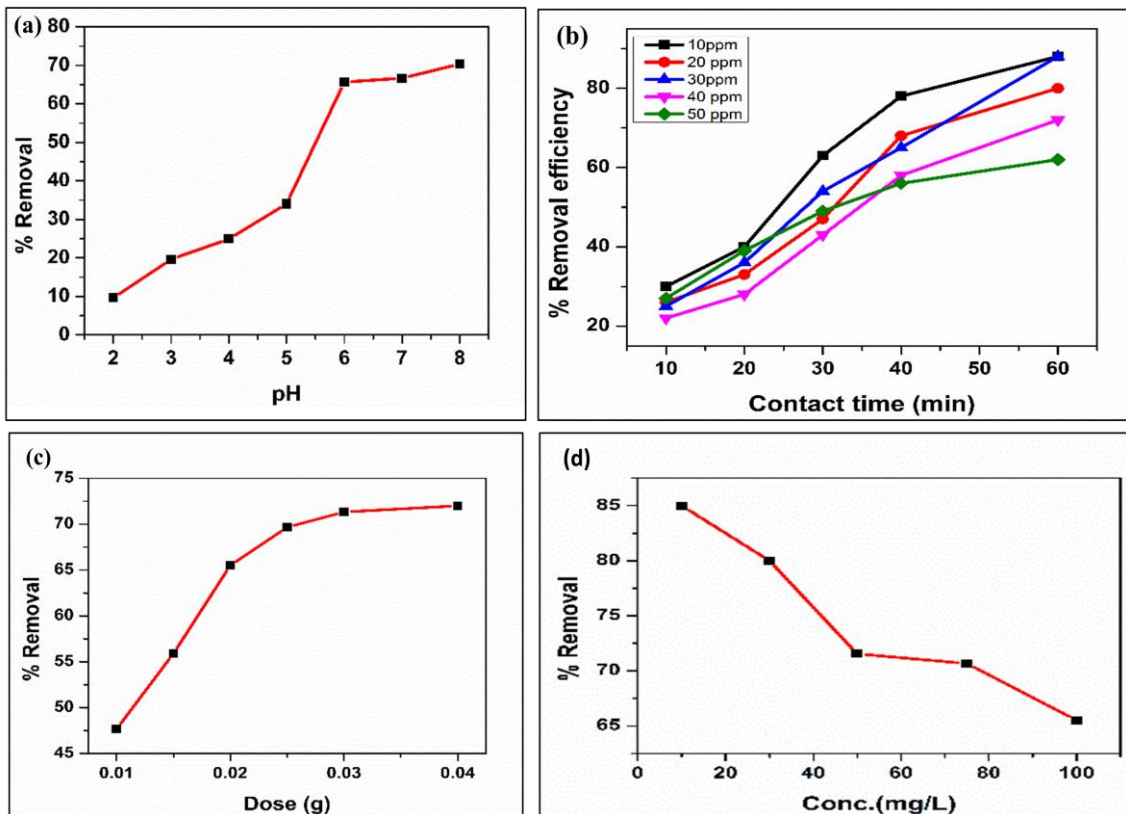


Fig. 3. Effect of (a) pH, (b) contact time, (c) adsorbent dose (d) various concentration

3.2.3 Effect of biosorbent dose

The amount of biosorbent dose was varied between 0.01 to 0.04 g/50mL to examine the impact of increasing adsorption dosages on the percentage removal of lead. The starting lead ion concentration was set at 10 mg/L, the contact time was kept at 60 minutes, and the pH of the adsorption medium was remained at 6. As the biosorbent dose increased concurrently with the removal effectiveness, as demonstrated in Fig. 3(c). The findings demonstrate that at an adsorbent dose of 0.03 g/50 mL, 72% of the lead ion was removed efficiently. This might be because more active sites were available for the adsorption of the biosorbent due to the surface area increasing with the adsorbent dose [17].

3.2.4 Effect of various initial lead concentration

The Pb (II) ions adsorption onto biosorbent was evaluated at different metal ions concentration (10, 30, 50, 75 and 100 mg/L) by keeping the remaining parameters unchanged (i.e. pH 6, adsorbent dose of 0.03g, contact time 60 min, temperature 27°C and stirring speed 200 rpm). The results showed that there was a considerable decrease in percentage removal of Pb (II) ions from 87 to 59.07% with increase in metal ions concentrations. The findings are displayed in Fig. 3(d). The capacity of the adsorbent materials is abruptly exhausted with a rise in initial lead ion concentration, at high concentrations, biosorbent site gets saturated and results in decrease in percent removal [18].

3.3 Adsorption isotherm Models

Table 1, summarizes the fitting parameters, Freundlich and Langmuir isotherms models fitted lead ion adsorption data well ($R^2 > 0.950$). However, Freundlich isotherm fitted slightly better than Langmuir isotherm due to the highest values of R^2 (0.995 and 0.965, respectively),

which implies that the arjuna bark biosorbent may have a heterogenous surface and a multilayer adsorption [19-22].

Table 1. Adsorption Isotherm parameters study for lead ions

Isotherm Models	Parameters	Values
Langmuir	qmax(mg/g)	125.0
	b(L/mg)	0.076
	R^2	0.965
Freundlich	N	1.823
	Kf(mg/g)	13.29
	R^2	0.995

3.4 Kinetics Study

In order to understand the kinetic behaviour of lead ion adsorption onto arjuna bark, the pseudo-1st-order and pseudo-2nd-order models were attempted in this work. Table 2 presents the validation findings and the kinetics model parameters. Experimental evidence indicates that the pseudo-first order regression coefficient is greater than that of pseudo-second order regression, indicating that adsorption of lead ions could be physisorption. The physical interaction may primarily involve the electrostatic interaction of lead ions (Pb^{2+}) on negatively charged surface of arjuna bark which agree with many previous biosorption studies [23-25].

3.5 Thermodynamic Studies

The thermodynamic parameter, including Gibbs free energy (ΔG^0) and entropy change (ΔS^0) and enthalpy (ΔH^0) for the adsorption of lead ions have been evaluated, verified and presented in Table 3. The increase in degree of randomness at the solid-solution interface is verified by the positive values of ΔS^0 for lead ions. Furthermore, the positive values of ΔH^0 for lead ions by biosorbent demonstrate that the adsorption process is endothermic [26]. Adsorption process feasibility and spontaneity are confirmed by

Table 2. Kinetics model parameters study for lead ions

Metal	Conc. (mg/L)	Pseudo-first order				Pseudo- second order			
		q _e (exp.)	k ₁ min-1	q _e (cal.)	R ²	K ₂ g/mgmin	q _e (exp.)	q _e (cal.)	R ²
Pb(II)	10	14.5	0.050	17.2	0.931	0.0007	15.8	28.2	0.846
	30	39.7	0.068	81.8	0.943	0.0001	45.3	91.7	0.842
	50	58	0.077	111.2	0.934	0.0003	74.4	97.1	0.898
	75	80.7	0.084	202.6	0.945	0.0001	103.3	166.7	0.802
	100	98.5	0.085	143.8	0.979	0.0004	126.1	133.3	0.979

Table 3. Thermodynamic parameters study for lead ions

Temperature (K)	ΔG° (kJ mol ⁻¹)	ΔH° (kJ mol ⁻¹)	ΔS° (J mol ⁻¹ k ⁻¹)
303	-1.92	63.74	211.19
313	-2.98	-	-
323	-3.27	-	-

negative ΔG° values of lead ions at increasing temperatures, indicating physical adsorption. The result is consistent with the finding reported for the removal of lead ions from water using different biomass adsorbents [27-29].

4. CONCLUSION

Chemically modified *Terminalia arjuna* bark have been demonstrated as biomass waste derived adsorbents for first time in Haryana. The removal of lead ions from simulated water using arjuna bark has been investigated under different experimental conditions in batch process. The optimal conditions of pH, initial lead concentration, adsorbent dosage, and contact time all affected lead adsorption. Adsorption kinetics analysis suggest that lead ions follow physisorption. The isotherm analysis further indicates that arjuna bark may have a heterogenous surface and a multilayered adsorption. The possibility and spontaneity of the adsorption process are validated by the negative ΔG° values of lead ions with increasing temperatures, which signify physical adsorption. The results of this study showed that lead ion may be successfully removed from an aqueous environment by using powdered arjuna bark as a low-cost adsorbent. Additional research is needed in the areas such as surface coating / functionalization of arjuna bark for different type of pollutants and adsorbent regeneration.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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