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Valorisation of dried biomass of water lentils for biosorption of Pb(II) from water in up-flow continuous column mode

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Abstract

The current research entailing a continuous adsorption approach in a fixed bed coulmn was accomplished to appraise the Pb(II) adsorption efficiency of dried biomass of a water lentile i.e. *Lemna gibba.* The study sought to understand the effect of operational parameters i.e.bed height and flow rate on the adsorption of Pb(II) by analysing the breakthrough curves. On the analysis of breakthrough curves it was observed that a reduced flow rate and higher bed depths resulted in higher adsorption efficiency. This also resulted in longer exhaustion and breakthrough times thus treating larger volumes of metal containing water before the bed gets fully saturated. The optimum operational parameters, aimed for the maximum removal 87.83 mg/g of Pb(II) from aquoeus solution containing 103.6 mg/L lead, are 19 cm bed depth and 2.0 mL/ min. The study also revealed the succesful application of Bed depth service time model with R^2 value greater than 0.996. Desorption experiments were conducted in order to regenerate, recycle and reuse the biosorbent for multiple cycles. Charaterisation of biosorbent before and after adsorption was done to study the surface morphology, elemental composition and crystalline phases of the biosorbent. It showed that Pb(II) was successfully adsorbed on the surface of adsorbent.

Keywords: *Lemna gibba* powder; Water lentil; Lead (II); Desorption; Regeneration.

Introduction

The release of heavy metal ions in water system from various sources is an issue recognized worldwide (Singh et al., 2024). Ca, Hg and Pb have been identified as 'Big three' heavy metals. Lead is one of the most dangerous metals which poses serious health concerns amongst all life forms as well as environment (Vélez et al., 2021; Carvalheiras et al., 2023; Saravanan et al., 2024; Thaçi et al., 20224). Major industries discharging lead into water include battery, oil refineries, paints and pigments, mining (Liu et al., 2012; Axtell et al., 2003). As per WHO, the maximum limit of Pb in potable water is set to be 0.01 mg/L (Edition, 2011). It thus becomes imperative that effluent containing metal ions are removed at the source before its discharge into natural water system.

Established methods and techniques like chemical coagulation and precipitation, chemical oxidation and reduction, ion exchange, membrane separation, and reverse osmosis have limitations related to operation and maintenance. They are also associated with low performance and generation of toxic secondary chemical residual (Aziz, 2023; Saravanan, 2024).

Employment of materials of biological origin for removal of water pollutants via adsorption has emerged as a non-conventional alternative of the above mentioned methods. The process of biosorption involves removal of water pollutants through active sites present on the surface of non living biomass (Singh et al., 1998; Davis et al., 2003). The process is advantageous in terms of being environmetally bening, easy to operate, cost effective, regeneration, reusability and thus contribution towards resource recovery and circular economy (Kuyucak, 1990; Bahadir et al., 2007; Kainth et al., 2024).

Adsoprtion process can be carried out via two system viz. the static or batch mode and the dynamic or continuous mode. In batch mode a fixed amount of adsorbent and adsorbate remain in contact and the changes in various parameters are observed over a specific time interval. It involves the optimization of process parameters as well as the study of isotherms and kinetics of adsorption. However, during studies conducted via packed bed continuous column mode the adsorbent and adsorbate always remain in contact till the need of saturation arise. The advantage of dynamic mode is that the process can be scaled up to industrial scale (Ashanendu et al., 2021; Farzaneh et al., 2021).

Various studies have been conducted for adsorption of lead (II) from water/wastewater (Sheba and Nandini, 2016; Tores, 2020; Bayuo et al., 2022; Tahir, 2026). The present work

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invetigates the biosoprtion effciency of dried powder biomass of a water lentil, also known as duckweed, *Lemna gibba*. Duckweeds are stem less, flowering free floating aquatic macrophytes growing in stagnent and slow moving water, which produce vegetatively by production of daughter fronds. They are reported to tolerate wide pH ranges (4.5-8.5) and high concentrations of total dissolved solids (up to 4000 mg/L) (Mkandawire et al., 2005; Younis and Nafea, 2015).

The studies are carried out in an up-flow dynamic mode using a packed-bed adsorbent column. As compared to down-flow mode, the up-flow mode has advantage of achieving high removal efficiencies. Because of no channeling, even fluid distribution, more residential period and maximum surface area exposure (Vijayaraghavan and Balasubramanian, 2013; Thirunavukkarasu et al., 2021). An attempt has also been made to desorb the metal ions from biosorbent surface using an appropriate eluent. It was then regenerated and further studied for its reuse as adsorbent for another cycle. This effort helps in making the process more economic, environment friendly and inclined with resource recovery and circular economy.

Material and methods

Preparation of adsorbent

Live duckweed plants were collected from a local pond, washed well with fresh and then distilled water. Biomass was then dried in an oven at 60ºC till it becomes completely dry. Dried biomass was then converted into powder form using blender and then finally seived (particle size <150 µm). Prepared biosorbent was stored in air tight containers to be used in experiements.

Preparation of adsorbate

Absorbate, lead(II) solution was prepared by dissolving $Pb(NO_3)_2$. First a standard stock solution (1000 mg/L) was prepared followed by further dilution as required for experiments. Very small quantity of conc. HNO_3 was added to the solution to prevent the precipitation of lead.

Packed bed Column design

A glass column (30 cm in height and 2 cm internal diameter) was used for packing of adsorbent in between layers of glass wool (1.0 cm), glass beads (3.0 cm) and a 0.5 mm stainless steel mesh at the bottom so that the adsorbent remains fixed in the column and also there is even distribution of adsorbate solution which passes throught the packed adsorbent bed. The column was packed with measured quantity of dried biomass powder so as to achieve desired height of the bed for experiements. A peristaltic pump of Miclins make was used to pump force the adsorbate solution (of known cncentration) into the packed bed column in an upward motion at desired flow rates (mL/min).

After saturation the desorption experiments were carried out using 0.1 N HCl as an eluent. After each cycle the bed was washed with double distilled water until pH reaches 7.0. Regenerated bed was used again for another cycle of sorption.

The treated solution was collected from top of the column for analyzing the remaining concentration of lead through UV-Visible Spectrophotometer (Spectrochem NV 201, AIMIL Ltd.). Other experimental conditions were pH 5, initial influent Pb(II) concentration at 103.6 mg/L, temperature 30 ± 1 °C.

Charaterization of adsorbent

The biosorbent before and after adsorption was charaterized through Scanning Electron Microscope (SEM), Transmission Electron Microscope (TEM), Energy Dispersive X Ray analysis (EDAX) and X Ray Diffraction (XRD) analysis. SEM and TEM were done to study the surface morphology and pore structure. EDAX confirmed the elemental composition of the adsorbent. With XRD crystalline structure is studied.

Study of Column Performance

In an up-flow fixed bed continuous column the adsorbate moves from bottom to the top of the column. While moving, depending on various factors (bed height, flow rate, initial metal concentration), the residential time of adsorbate in the column varies. At the time when bed becomes saturated (exhausted) the concentration of inlet and outlet adsorbate solution becomes virtually same. The breakthrough curves (time vs. C_o/ C_t) are analysed for invetigating the column performance (Shakeel et al., 2025). The volume of treated effluent is then calculated as follows with the help of flow rate (F) and time of exhaustion (t_e):

$$V_{eff} = F \times t_e \quad (1)$$

Total metal quantity (mg) transmitted to column:

$$m_{total} = C_o F t_e / 1000 \quad (2)$$

where C_o is the initial metal ion concentration (mg/L).

Percentage of metal removed:

$$\% \text{ removal} = (m_{ad}/ m_{total}) \times 100 \quad (3)$$

where, m_{ad} is the total amount of adsorbate retained with in the adsorbent (mg).

Equilibrium uptake capacity, q (mg/g), is metal adsorbed per unit mass of adsorbent (m) from the

total quantity of metal adsorbed (q_{total}) is determined as follows:

$q = q_{total}/ m$

(4)

Percentage of desorption is calculated using amount of metal desorbed (m_d, mg) as follows:

% desorption = (m_d/ m_{ad}) x 100

(5)

m_d is determined by multiplying flow rate with the area obtained by integration of desorption profile (time vs. effluent concentration).

Time of breakthrough (t_b) is the time when effluent concentration reaches 1 mg/L, while time of exhaution (t_e) is the time when effluent concentration crosses 95% of initial metal concentration.

Based on Bohart-Adams' equation, the Bed Depth Service Time (BDST) model is used in the evaluation of coefficients which are required for designing of a column. It provides systamatic approach for the designing of adsorption bed columns. This model is simple, versatile and hence used extensively across multiple process variables. The linear form of Bohart Adams' equation is given by Hutchins as follows:

$$t = \frac{N_o Z}{C_o u} - \frac{1}{k_a C_o} \ln\left(\frac{C_o}{C_t} - 1\right)$$

(6)

This relation predicts association between service time (t) and bed depth (Z). It estimates N_o (maximum adsorption capacity, mg/L). In this equation, u is the linear flow velocity (cm/ min), C_o and C_t are adsorbate concentrations (mg/L) at the begining and at time, t, k_a is the BDST rate constant ($Lmg^{-1}h^{-1}$).

Results and discussions

Study on effect of bed height

Fig. 1 presents the breakthrough curves obtained by studying the effect of changing the column fixed bed height on Pb(II) removal. The bed height was varied as 7.0, 10.0, 13.0, 16.0 and 19.0 cm by packing the column with different amount i.e. 5.1, 8.2, 10.5, 13.6, and 16.8 g of adsorbent. The flow rate was maintained at 2.0 mL/ min. It was observed (Table 1) from the parameters calculated by this plot, that the with the increase in bed height the column performance increased significanlty. The volume of treated effluent by the packed bed increased from 4342.8 to 7740 mL. Time of breakthrough (from 21 to 37.5 hours), time of exhaustion (36.19 to 65.50 hours), uptake capacity (64.23 to 87.83 mg/g), percentage removal (from 61.57 to 91.89 %) also increased with increase in bed height from 7.0 to 19.0 cm (Mohan, 2008). This performance improvement can be attributed to availabilty of more amount of adsorbent packed in the column, providing more surface with active adsorption sites. Also, there is prolonged residential period of adsorbate solution in longer beds facilitating improved mass transfer from liquid to solid phase. The adsorption sites were efficiently utilized, delaying the movement of mass transfer zone towards outlet of column, ultimately leading to longer breakthroughs and exhaustion of column. This also led to improved adsorption uptake capacity and percentage removal of Pb(II) (Radhika et al., 2018; Patel, 2019).

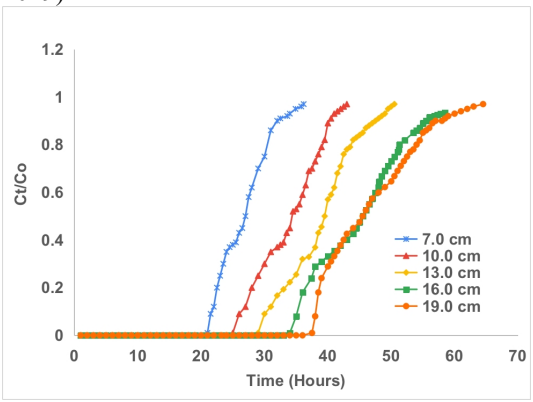


Figure 1: Breakthrough curve for Pb(II) sorption at different bed heights

Study on effect of flow rate

Different flow rates were maintained using a peristaltic pump to study the effect of flow rate on process parameters. Flow rates were maintained at 2.0, 4.0, 6.0, 8.0 and 10.0 mL/min. With the increase in flow rate a decrease in the values of volume of effluent (7740 to 21600 mL), breakthrough time (37.5 to 21.3 h) and exhaustion time (64.50 to 36.00 h) of the column, uptake capacity (87.83 to 61.38 mg/g) and percentage removal (91.89 to 71.94 %) was observed (Fig. 2, Table 1). This is because adsorbate gets lesser time of contact with the adsorbent in the column at higher flow rates, resulting in lesser intraparticle diffusion and mass transfer from liquid to the solid phase (Attia et al., 2008; Lim and Aris, 2014). With adsorbate getting short residential period in the bed, it exists from the bed early before saturation (Radhika and Palanivelu, 2006). Lower flow rates, however, leads to more residential period for Pb(II) solution in the bed, providing sufficient time for its adsorption. The column saturation is thus delayed resulting in higher removal efficiency (Fauzia et al., 2021).

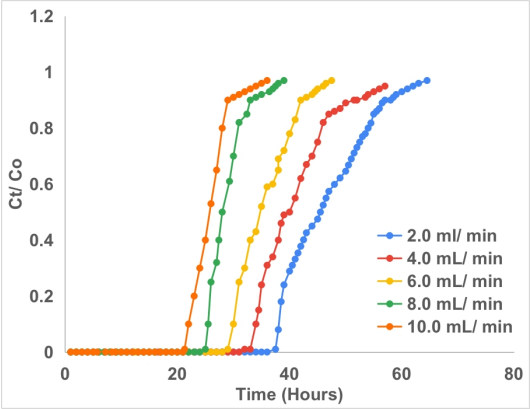


Figure 2: Breakthrough curve for Pb(II) biosorption at different flow rates

Table 1: Adsorption Data for Fixed-Bed dried powder *Lemna gibba* for Pb(II) Adsorption at Different Process Parameters

Process Pattern	**Veff (mL)**	**t_b (h)**	**t_e (h)**	**q (mg / g)**	**% removal**
Bed Height (cm)					
7	4342.8	21.00	36.19	64.23	61. 57
10	5160	25.00	43.00	69.53	70.12
13	6060	29.00	50.50	76.37	77.45
16	7020	34.00	58.50	81.71	82.84
19	7740	37.50	64.50	87.83	91.89
Flow rate (mL/min)					
2	7740	37.50	64.50	87.83	91.89
4	13680	32.00	57.00	83.36	85.88
6	17100	29.00	47.50	77.98	81.44
8	18720	25.00	39.00	68.54	76.43
10	21600	21.30	36.00	61.38	71.94

Bed Depth Service Time (BDST) Model

The values of bed sorption capacity per unit volume of the bed (N_o) and rate constant (K_a), determined by the slope and intercept of BDST plot (t vs Z, at various flow rates) (Figure 3). Higher value of correlation coefficient (>0.997) suggests the applicability of BDST model (Zang et al., 2017).

The rate constant, K_a, expresses the rate at which the substance diffuses from the liquid phase into the solid phase. The value of K_a was found to increase with an increase in flow rate (from 0.003996 to 0.009758 L/mgh). Higher K_a value means that regardless of the bed length, there will be no breakthrough, but as the K_a value reduces, increasingly longer bed lengths will be needed. The values of N_o were also found to increase with the increase in flow rate (Fadzil et al., 2015).

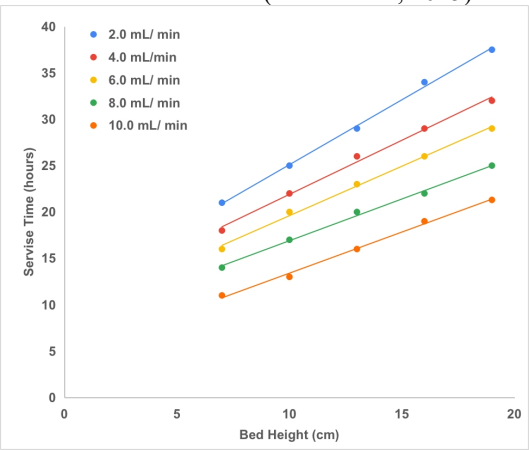


Figure: 3: Bed Depth Service Time plot

Desorption studies

Reusability of the biosorbent is another critical aspect of application and economic viability of any biosorption process. Multiple utilization of the same biosorbent not only results in a decrease in total costs of the treatment process but also significantly limits the necessity to prepare and add new biosorbents constantly. That is why the

ability of regeneration of the *Lemna gibba* powder as biosorbent was investigated through carrying out the three subsequent adsorption-desorption cycles. It is essential to choose a suitable desorbing agent since it must release the adsorbed metal ions without damaging the structure of the biosorbent or decreasing its adsorption ability. The effectiveness of the desorption is mainly defined by the mechanism of interaction of the metal and the chemical properties of the eluent and the biosorbent.

Desorption was carried out by packing the column with 16.8 g of biosorbent to maintain bed height of 19 cm. Based on the optimized adsorption conditions, initial lead concentration and flow rate were fixed at 103.6 g/L and 2.0 mL/min, respectively, throughout the desorption studies. 0.1 N HCl was used as an eluent for the used sorbent for all three sorption-desorption cycles.

Breakthrough curve for three sorption-desorption cycles are shown in Fig. 4, which shows that the curves become flat with each cycle, because with each cycle the breakthrough time decreases and exhaustion time increases. The results of desorption experiments are presented in Table 2. Good metal uptake (87.83 mg/g) and removal efficiency (91.89 %) was obtained showing the satisfactory performance of biosorbent in all three cycles.

Elution curves for three cycles are shown in Fig. 5. It can be seen that the curves are very sharp in the beginning with a gradual decrease. After each cycle of regeneration with dil HCl, the column was rinsed with distilled water till pH of 7.0 is obtained to make the column ready for next sorption cycle. The desorbent performed very well, exhibiting great desorption efficiency i.e. 95.54% (Table 2). Adsorption properties have decreased with each subsequent cycle due to shorter breakthrough time, alterations in the exhaustion time, wider mass transfer zone, and less steep breakthrough curves were obtained. All these parameters indicated that with each sorption-desorption cycle and regeneration, there is loss of adsorption sites available for adsorption. However, the biosorbent still had rather good regeneration capacity since the significant amount of adsorption properties remained during the experiment. Good results of regeneration in combination with the successful adsorption ability indicate that biosorbent under study can be considered a perspective, economic, and environmental-friendly biosorbent for removal of Pb(II) (Zang et al. 2017). It was also observed that in cycle 1, desorption was carried out for 6.50 h, as compared to 64.50 h for the sorption process, resulting in highly concentrated Pb(II) solutions in only a small volume of desorbent. For instance, in cycle 1 at t = 0.5 h the effluent Cr(VI) concentration was 924 mg/L.

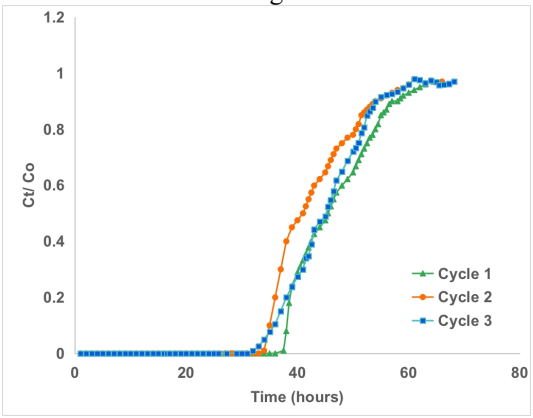


Figure 4: Breakthrough curves for Pb(II) sorption during
three sorption-desorption cycles

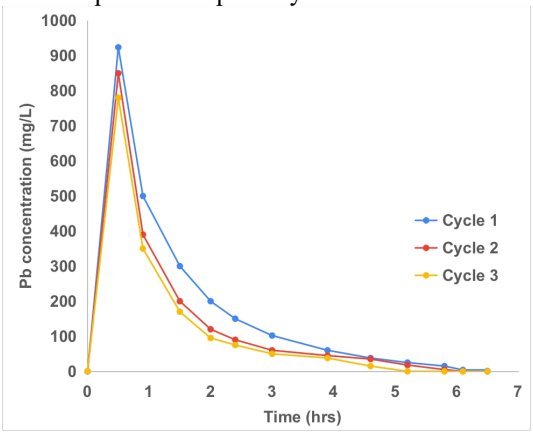


Figure 5: Elution curves for desorption of Pb(II) during three sorption-desorption
cycles

Table 2: Sorption-Desorption process studies for three cycles

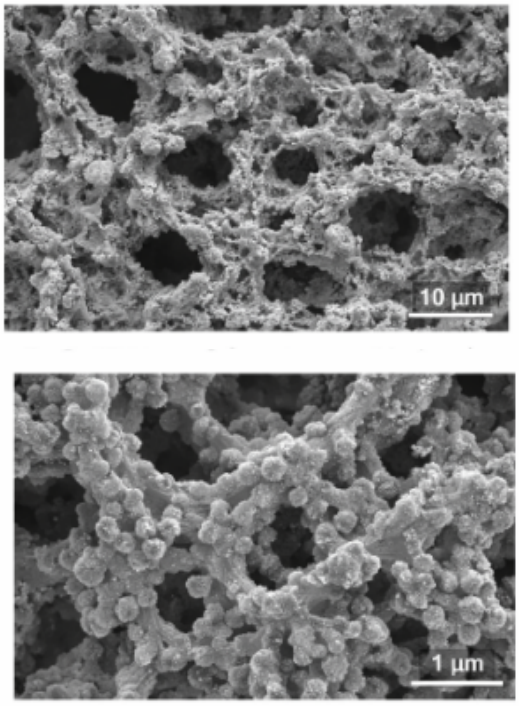
Cycle	Veff (mL)	t_b (h)	t_e (h)	q (mg/g)	% removal	Time for elution (h)	Elution efficiency (%)
I	7740	37.50	64.50	87.83	91.89	6.5	95.54

II	79 20	34 .0 0	66 .0 0	83. 39	87. 02	6.1	92.9 2
III	81 84	32 .0 0	68 .2 0	78. 91	85. 48	5.2	87.5 9

Characterization

SEM Analysis

Before and after adsorption, the surface morphology of the *Lemna gibba* biosorbent was examined using scanning electron microscopy (SEM). The rough and porous surface structure of the raw biosorbent provides active binding sites for heavy metal adsorption (Fig. 6(a), 6(b)). The pores appeared to be partially filled following the adsorption of lead ions (Fig. 6 (c)), indicating that the metal uptake was successful.



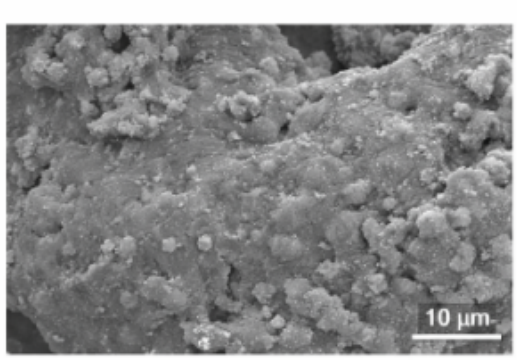


Figure 6: SEM micrographs of *Lemna gibba* biosorbent showing (a) raw biosorbent surface morphology, (b) high-magnification microstructure indicating porous adsorption sites, (c) biosorbent surface after Pb(II) adsorption

TEM Analysis

Transmission Electron Microscopy (TEM) provides high-resolution imaging of the internal pore morphology, connectivity and structural arrangement of *Lemna gibba* biosorbent (Figure 7(a) and 7(b)). The irregular shapes of the particles provides more active sites for the adsorption of metal ion and thus resulting in enhanced adsorption efficiency. TEM validates the successful adsotpion of Pb(II) through the appearance of dark areas (high contrast regions) (Fig. 7(c)) on the irregular particles.

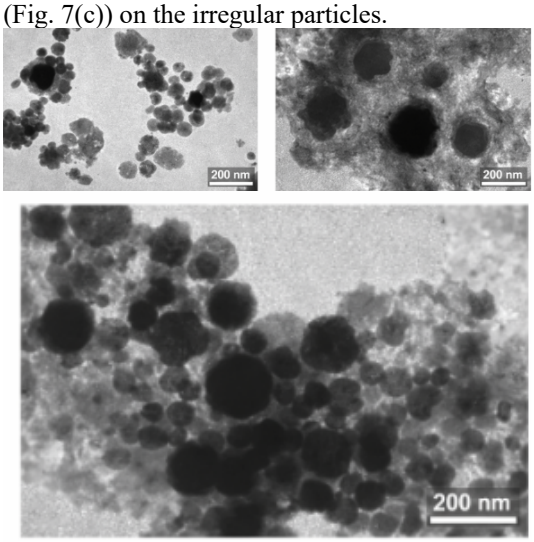


Figure 7: TEM micrographs of *Lemna gibba* biosorbent showing (a) raw biosorbent nanoparticles, (b) internal pore structure of biosorbent, (c) Pb(II) loaded biosorbent

EDAX Analysis

Lemna gibba powder's elemental composition was determined by Energy Dispersive X-ray Analysis (EDAX) (Table 3, Figure 8). The presence of carbon, oxygen, calcium, and silica was confirmed by the spectrum (Fig. 8(a)). Additional peaks

corresponding to lead demonstrated that the biosorption process was successful after adsorption (Fig. 8(b)).

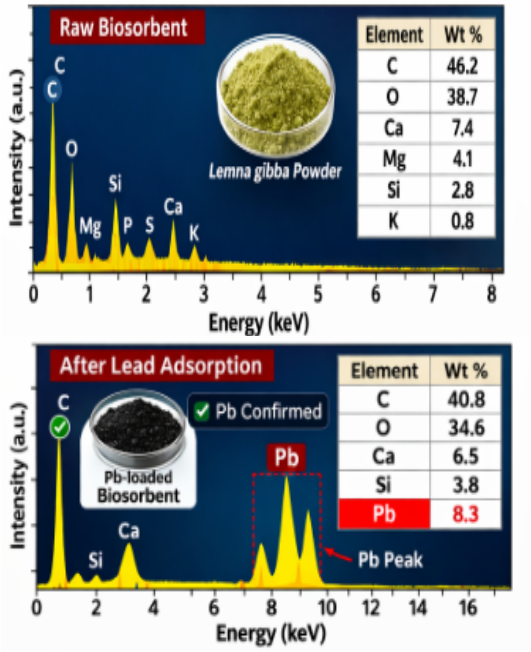


Figure 8: EDAX analysis of *Lemna gibba* biosorbent showing (a) elemental spectrum of raw biosorbent, (b) EDAX spectrum after Pb(II) adsorption,

Table 3: Elemental Composition

Element	Before Adsorption (%)	After Pb Adsorption (%)
C	**45.2**	39.8
O	**38.6**	34.9
Ca	**8.2**	7.2
Pb	–	7.4

XRD Analysis

The *Lemna gibba* biosorbent's crystalline phases were identified by X-ray diffraction analysis (Figure 9). The diffraction peaks proved that the structure was cellulose and lignocellulosic (Fig. 9(a)). The interaction between the biosorbent surface and heavy metal ions was demonstrated by the peak intensity reduction following lead adsorption (Fig. 9(b)).

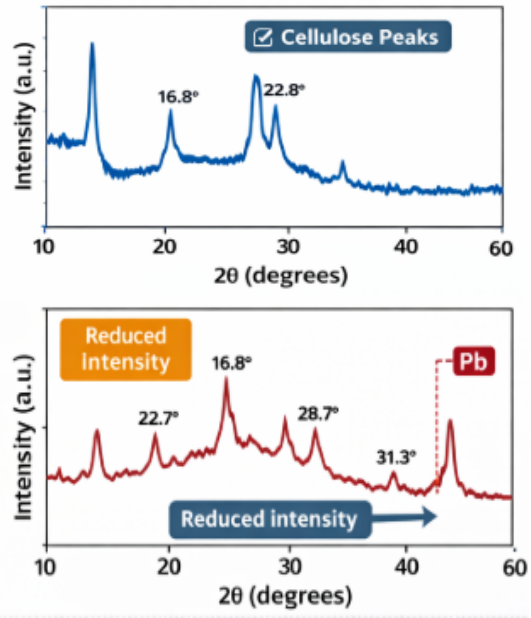


Figure 9: XRD patterns of *Lemna gibba* biosorbent showing (a) raw biosorbent powder, (b) biosorbent after Pb(II) adsorption

Conclusion

This study identifies the dried powder of water lentil, *Lemna gibba* (gibbous duckweed) as an effective biosorbent for continuous removal of Pb(II) from aqueous solution. Adsorption process is dependent on bed height and flow rate, showing that with increase in bed height the adsorption increased, however, it is adversely affected by the increase in flow rate. The maximum uptake percentage 91.89% (87.83 mg/g) was noticed to be at bed height 19 cm and flow rate 2 mL/ min. The BDST model fitted well to the sorption data and the values of bed sorption capacity (No) and Ka were found to increase with the increase in flow rates. Sorption performance of biosorbent was also successfully studied for three sorption-desorption cycle. All three cycles showed good sorption capacity. The rejuvenation and recycling show the possibility of using such biosorbent as an economical and sustainable biosorbent for the continuous removal of Pb(II). The recovery of the adsorbed metal prolongs the lifespan of the biosorbent, decreases the amount of material needed, and decreases the cost of the process. Additionally, the proper disposal of the spent biosorbent at the end of its working life helps to minimize the ecological footprint of the proposed biosorption technique. Adsorption of Pb(II) on the surface of dried powder of *Lemna gibba* was confirmed via SEM, TEM, EDAX and XRD analysis.

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