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Original Research Paper

Aquatic macrophytes for the effective remediation of hexavalent chromium from aqueous media in Continuous up-flow column

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Abstract

A continuous up-flow column mode was employed using dried powder of *Lemna gibba*, a gibbous duckweed for the biosorptive remediation of hexavalent chromium from aqueous solution. Breakthrough curves under different flow rates and bed heights were used to study sorption performance of the biosorbent. The maximum Cr(VI) removal efficiency of 65.32 mg/g corresponding to 84.18% obtained at a flow rate of 2 mL min^{-1} and bed height of 19 cm. The Bed Depth Service Time (BDST) model showed to be well agreeing with the experimental data ($R^2 > 0.995$). Desorption and regeneration studies showed that the biosorbent retained its adsorption efficiency well under three consequtive sorption-desorption cycles indicating good reusabilty and operational stability. Charaterization of the biosorbent was done before and after adsorbent using SEM, TEM, EDAX and XRD. While SEM and TEM showed noticiable modifications in surface after adsorption, EDAX confirmed the presence of Cr on the surface, and XRD further confirmed the surface structural changes, confirming its adsorption onto the biosorbent.

Keywords: ***Lemna gibba*** **powder; Chromium(VI); Adsorption; Bed Depth Service Time; Regneration.**

Introduction

Amidst several heavy metals, chromium (VI) has attracted much attention due to its extensive industrial application and its high toxicity (Rai et al., 2021; Suganya et al., 2019; Vunain et al., 2021; Adeleken and Abegunde, 2011; Devendra et al., 2017). The maximum permissible limit for Cr(VI) in potable water is 0.05 mg/L given by US EPA (Zhang et al., 2021). The traditional methods for the removal heavy metals from contaminated water are often associated with operational, cost and environmental issues (Obayomi et al., 2020). It is therefore imperative that treatment methods be developed that are cost-effective, environmentally friendly, and easy to manage. These options could also improve the efficiency of wastewater treatment and minimise adverse environmental impacts. In this regard biosorption has emerged as an unconventional method for not only metal detoxification but also recovery from water/ wastewater (Marghaki et al., 2020; Tayyab et al., 2026). During biosorption batch method determines the equilibrium time, optimal operating parameters, and adsorption efficiency. However, the continuous column mode evaluations provide the critical data to scale up the treatment system from laboratory to industrial operations (Borna et al., 2016; Ajmani et al. 2020). *Lemna gibba* is a small free-floating flowering plant (family Araceae) widely distributed in many parts of the world and also one of the most reproductive plants on earth. it typically grows floating on slow-flowing or still fresh and brackish water, particularly nutrient-rich water. Under conditions favourable to the plants, they form thick green mats on the water's surface. Duckweed generally propagates by vegetative propagation. During this, the daughter fronds emerge from the parent frond and remain attached for a while, until they establish small colonies, after which they separate when mature. At low temperatures, duckweed may produce special dormancy structures called turions. The cell wall is rich in cellulose and hemicellulose (Martinez et al., 2019; León et al., 2017; Topal et

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al., 2020). Current work investigates the utilization of dried powder of *Lemna gibba* biomass for the adsorptive remediation of hexavalent form of chromium employing the continuous column method in upward direction.

Material and methods

Chemicals and biosorbent:

Chemicals were procured from E. Merck India Ltd. For preparing 1000 mg/L standard solution of Cr(VI), 1.4143 g of $K_2Cr_2O_7$ was dissolved in 500 mL of double distilled water. Experimental solutions of the various concentrations were obtained by further dilution of the stock solution as desired.

A local natural pond was the source for collecting *Lemna gibba* which was then washed thoroughly with fresh and then with deionised distilled water for completely removing any foreign material. The clean biomass was oven-dried at 60 °C for approx 24 h. Dried biomass was crushed and seived to fine powder (particle size <150 µm) for being further use in adsorption.

Column Experiments:

For fixed bed up-flow continuous experiment a glass column of height of 30 cm and internal diameter of 2.0 cm was used. Different quantity of dried biosorbent powder was filled in column for achieving different bed heights. The biosorbent is packed in the glass column between a sieve (stainless steel - 0.5 mm width), a layer of glass wool (2.0 cm) and a layer of glass beads (2.0 cm) at the bottom of the column in order to ensure uniform distribution of influent through the fixed bed. A peristaltic pump (Miclins India Model 4651) was used for solution feed in upward flow in the columnas shown in Fig. 1. The effluent collected from column top was analyzed for remaining concentration of Cr(VI). Throughout the experiments the conditions were maintained as: atmospheric pressure (760 mm Hg), temperature 30 ± 1 °C, pH 1.5 and the initial Cr(VI) concentration at 103.99 mg/L. A UV-Visible spectrophotometer (Spectrochem NV 201, AIMIL Ltd.) was used to determine the Cr(VI) concentration.

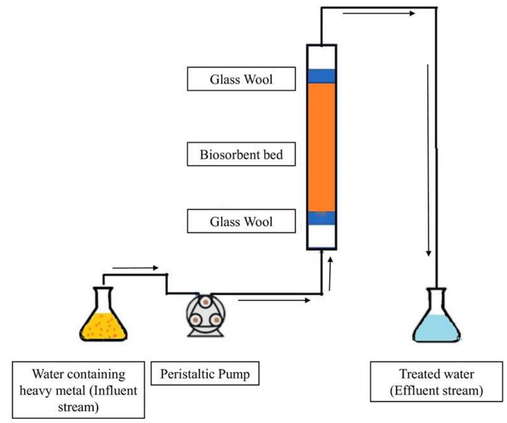

Figure 1: Experimental setup

Analysis of column results:

Breakthrough curves (C_t/C_o vs. time) were used to investigate the adsorption behaviour of hexavalent chromium under continuous column conditions. The experimental results were subsequently interpreted using equation as below (Shakeel et al., 2025):

Volume of treated Cr(VI) solution (Oliveira et al., 2020):

$$V_{eff} = F.t_e \tag{1}$$

Total quantity of Cr(VI) volume entering column (Oliveira et al., 2020):

$$Mtotal = \frac{C_o\ F\ t_e}{1000} \tag{2}$$

Total Cr(VI) removal percentage (Yahya and Odigure, 2015):

$$Total\ Cr(VI)\ \ removal\,(\%) = \frac{mad}{mtotal} x100 \tag{3}$$

Mass transfer zone length:

$$Zm = \ Z(1 - \frac{tb}{te}) \tag{4}$$

The % desorption was calculated using below equation:

$$Desorption efficiency(\%) = \frac{md}{mad} x100 \tag{5}$$

where, m_d is amount of desorbed Cr(VI), m_{ad} is quantity of metal adsorbed by the column bed, time of breakthrough (t_b) is time when effluent concentration reaches to 1 mg/L, while the time of exhaustion (t_e) is time when concentration of Cr(VI) in effluent (C_t) exceeds 95-99% the influent concentration (C_o), q (mg/g) is adsorbent uptake capacity.

Service time (t), represent time at which the bed achieves its limit and needs to be replaced or regenerated. Hutchins (1973) developed Bed Depth Service Time (BDST) model, as follows:

$$t = \frac{N_o Z}{C_o u} - \frac{1}{k_a C_o} \ln\left(\frac{C_o}{C_t} - 1\right) \qquad (6)$$

where, N_o is adsorption capacity of bed (mg/L), Z is bed height (cm), u is linear velocity (cm/min), C_o (mg/L) is initial concentration and C_t (mg/L) is effluent concentration and K_a (L/mgh) is BDST rate constant.

Results and discussions

Impact of bed height on chromium adsorption

To study change in bed height impact on chromium adsorption packing heights of 7.0, 10.0, 13.0, 16.0, and 19.0 cm were maintained by filling column with 6.2, 10.9, 13.5, 17.9 and 20.58 g of *Lemna gibba* powder respectively at metal ion concentration 103.99 mg/L and flow rate 2.0 mL/min.

The curves are presented in Figure 2 and results are presented in Table 1, which show that with bed height breakthrough volume increases as a consequence of this was that there was inadequate time for the Cr(VI) ions to diffuse into the internal pores and occupy all the available adsorption sites in the biosorbent. With the decrease in bed height from 19 cm to 7 cm, a remarkable reduction in treated solution volume prior to breakthrough from 2325.85 mL to 751.42 mL was observed. The bed height increase results in an improvement in uptake (mg/g), as well as both breakthrough and elution time. The reason is that high amount of adsorbent dose provides more active sites and contact period for adsorption (Han et al., 2008; Payel et al., 2021).

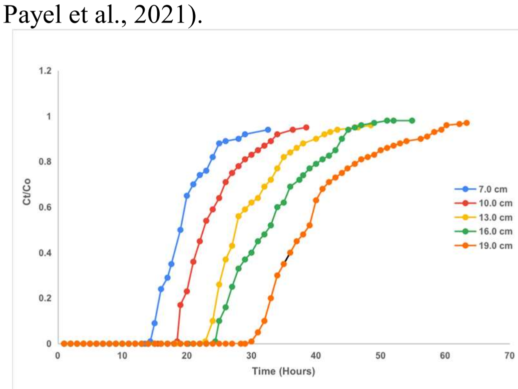

Figure 2: Effect of bed height during column adsorption of Cr (VI)

Impact of flow rate on Cr(VI) adsorption

The experiments were conducted at flow rates of 2.0, 4.0, 6.0, 8.0 and 10.0 mL/min at bed height of 19 cm and influent metal concentration of 103.99 mg/L. Alteration in flow rates influenced the hydraulic residence time between Cr(VI) ions and adsorbent fixed bed. The plot at various flow rates is represented in Figure 3 and various parameters calculated are given in Table 1. As flow rate increased, values of breakthrough time, elution time and uptake capacity of chromium (VI) decreased. It is because with flow rate increase, less mass transfer as well as intra-particle diffusion of metal ion occurs in the column bed (Abdolali et al., 2017). The volume of effluent, however, increased with flow rate. It is due to lesser interaction of adsorbate at higher flow rates, and thus lesser residential period (Syeda et al., 2025).

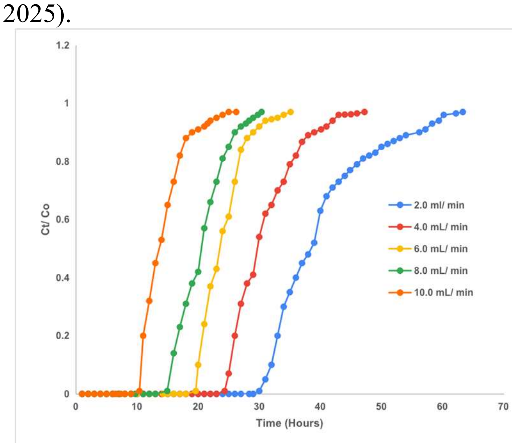

Figure 3: Effect of flow rate during column adsorption of Cr (VI)

Table 1: Continuous column experiment results during Cr(VI) Adsorption on to dried powder *Lemna gibba*

Process Pattern	Veff (mL)	t_b (h)	t_e (h)	q (mg/g)	% removal
Bed Height (cm)					
7	3907.2	14.34	32.56	48.72	58.36
10	4618.8	18.49	38.49	53.74	69.84
13	5817.6	22.86	48.48	58.12	74.48
16	6584.4	24.40	54.87	62.13	79.34
19	7598.4	28.35	63.32	65.32	84.18
Flow rate (mL/min)					
2	7598.4	28.35	63.32	65.32	84.18
4	9473.1	24.34	47.23	63.29	79.89
6	12479.6	19.61	35.13	58.32	73.47

8	14583.3	14.94	30.38	52.36	68.31
10	16931.1	10.39	26.23	46.39	62.26

Bed Depth Service Time (BDST) Model

Table 3 tabulates the results determined using intercept and slope of BDST plots (t vs Z). The plot showed good linearity, with a high correlation coefficient (>0.995). K_a obtained from plot intercept, represents the rate at which metal ion diffuses from the liquid to the solid phase. With increase in flow rates, values of K_a improved from 0.00630 to 0.3147 L/mgh indicating the need of longer adsorbent beds. (Goel et al., 2005)

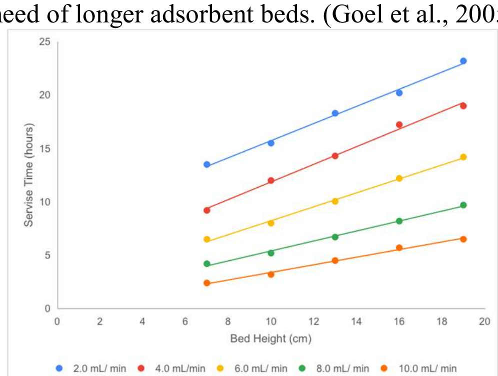

Figure: 4: BDST plot at different flow rates

Desorption studies

Desorption and regeneration of biosorbent becomes imperative keeping in mind the economic and environmental factors in mind. It also plays vital role in resource recovery and circular economy. For efficiently reusing the adsorbent, it should be easilty regenerated. The desorption process depends on how a biosorbent interacts with metal ion and the chemical properties of the eluent and biosorbent (Bayuo et al., 2024). The ideal eluent should be efficient, inexpensive, non-toxic, ecofriendly, and shoukd not damage the adsorbent surface (Taşdemir, 2021; Costa et al., 2020; Abdel-Aziz et al., 2023). Ability of regeneration of the *Lemna gibba* powder as biosorbent was investigated via three adsorption-desorption cycles at bed height 19 cm, metal ion concentration 103.99 mg/L and flow rate at 2.0 mL/min, respectively. 0.1 N NaOH was used an eluent for this study.

The results given in table 4 and represented in Fig. 5 display that the performance of the biosorbent across all three cycles was good. High uptake (65.32 mg/g), removal percentage (84.18%) with high elution efficiency of 93.35% were obtained. With progress of sorption-desorption cycles some active adsorption sites on adsorbent surface decreases. In spite of this the adsorbent under study showed good regeneration as well as adsorption efficiency, suggesting its prospective use as cost effective, and ecofriendly biosorbent.

Elution curves are shown in Fig. 6. The eluent (0.01 M NaOH) with good desorption efficiency (Table 4) performed very well. It was noticed that desorption was conducted for approximately 10.3 h. In cycle 1 the effluent concentration at time 0.61 h was 813 mg/L.

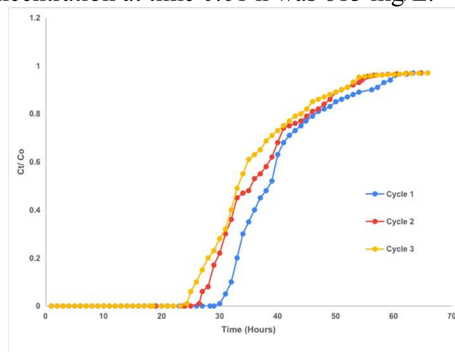

Figure 5: Sorption curves of Cr(VI) during three cycles of sorption-desorption

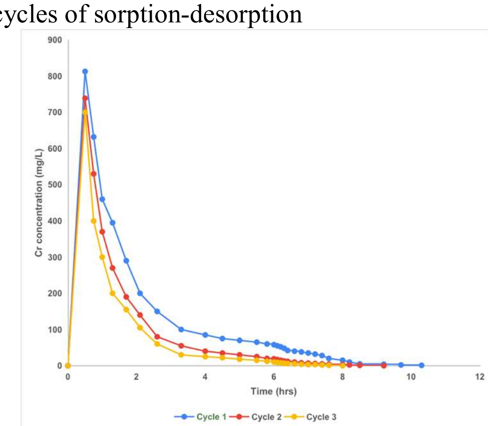

Figure 6: Elution curves for desorption of Cr(V) during three sorption-desorption cycles

Table 2: Sorption-Desorption process studies for three cycles

Cycle	V_{eff} (mL)	t_b (h)	t_e (h)	q (mg/g)	% removal	Time for elution (h)	Elution efficiency (%)
I	7598.4	28.35	63.32	65.32	84.18	10.3	93.35
II	7761.6	26.47	64.68	63.54	78.42	9.2	91.85
III	7900.8	24.35	65.84	60.71	77.04	8.1	88.57

Characterization

SEM Analysis

The morphology of the *Lemna gibba* biosorbent's surface before and after adsorption was analyzed through Scanning Electron Microscopy (SEM) (Fig. 7). Raw biosrobent surface (Fig. 7(a)) can be seen having porous structures, which act as active sites for heavy metal adsorption. In Fig. 7(b) biosorbent surface after adsorption is presented which clearly shows changes in surface morphology giving evidences of pores being partially filled after adsorption of chromium ions, which is further confirmed by other characterization techniques.

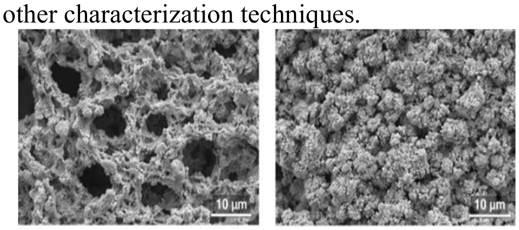

Figure 7: SEM micrographs of *Lemna gibba* biosorbent showing (a) raw biosorbent surface morphology, (b) surface after Cr(VI) adsorption

TEM Analysis

Fig. 8 shows Transmission Electron Microscopy (TEM) micrographs of (a) raw biosorbent (b) internal particle morphology and detailed porous structure of the biosorbent and (c) chromium loaded biosorbent. Presence of irregular particles on surface enhances the adsorption efficiency. In fig. 8 (c) the dark areas indicates the deposition of chromium representing metal adsorption.

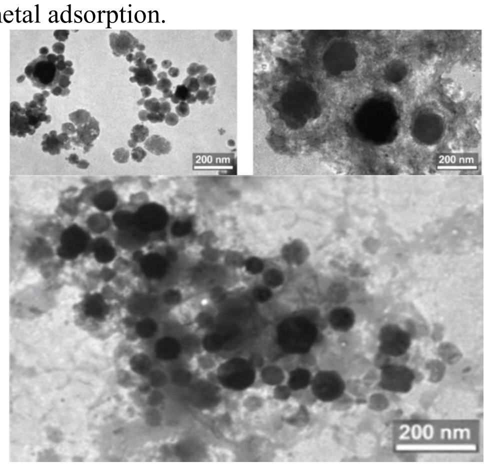

Figure 8: TEM micrographs of *Lemna gibba* **biosorbent showing (a) raw biosorbent, (b) internal pore structure of biosorbent, (c) chromium-loaded biosorbent**

EDAX Analysis

The elemental composition of *Lemna gibba* powder was determined by Energy-Dispersive X-ray Analysis (EDAX) (Fig. 9). Fig. 9 (a) shows elemental peaks before adsorption. The spectrum (Fig. 9(a)) confirms the presence of carbon, oxygen, calcium, and silica. Fig. 9(b) clearly shows peak corresponding to chromium indicating the success of biosorption process.

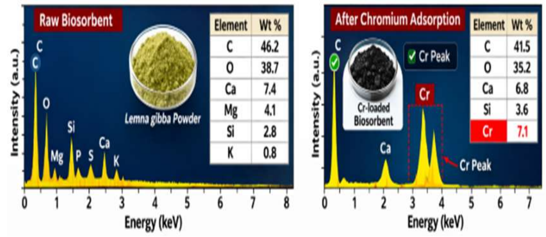

Figure 9: EDAX analysis of *Lemna gibba* biosorbent showing (a) elemental spectrum of raw biosorbent, (b) EDAX spectrum after chromium adsorption

Table 5: Elemental Composition

Element	Before Adsorption (%)	After Cr Adsorption (%)
C	45.2	40.1
O	38.6	35.4
Ca	8.2	7.5
Cr	–	6.8

XRD Analysis

Study of crystalline phase of biosorbent was done by XRD analysis. The diffraction peaks (Fig. 10) shows the biosorbent structure was cellulosic and lignocellulosic. With the adsorptive interaction of chromium metal ion with the biosorbent surface a reduction in the peak intensity was observed.

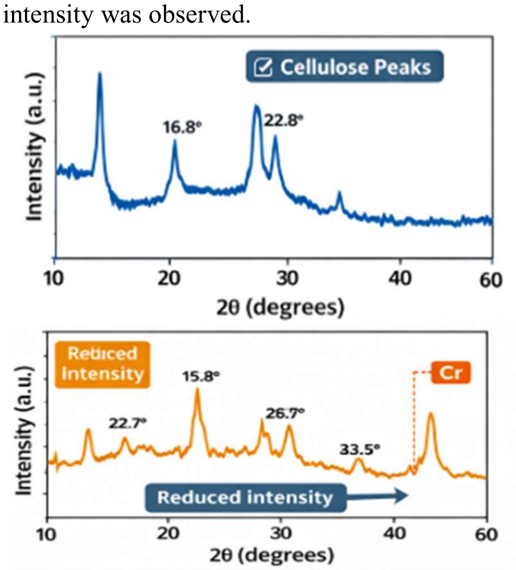

Figure 10: XRD patterns of Lemna gibba biosorbent showing (a) raw biosorbent powder, biosorbent after Cr(VI) adsorption

Conclusion

The performance of dried powder of gibbous duckweed, *Lemna gibba* for the adsorptive removal of chromium was analyzed under a fixed bed up-flow continuous column mode. Biosorbent performed well under varied conditions of bed depth and flow rates. It showed removal efficiency of 65.32 mg/g and percentage removal of 84.18 % at 19 cm bed height, 2.0 mL/min. flow rate, pH 2.0 and initial chromium concentration 103.99 mg/L. The uptake capacity increased with rise in bed height while decreased with the increase in flow rate. BDST model fitted well to the sorption data (R^2>0.995). Increase in rate constant (K_a) with flow rate showed an increased transport of metal ions from solution to adsorbent surface. Adsorbent was examined for three sorption-desorption cycles, and it maintained good update capacity for all cycles. 0.01 M NaOH was employed as a successful eluent giving high elution efficiency (93.35 %). This shows that the dried powder of *Lemna gibba* can be employed as a cheap, easily available and ecofriendly adsorbent. The surface morphology of biosorbent was also analyzed using SEM, TEM, EDAX and XRD analysis before and after adsorption. A clear modification in surface of biosorbent shows successful adsorption.

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