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Adsorptive Removal of Hazardous Cadmium(II) Ions from Synthetic Wastewater using Acid-Treated Algal Biomass *Vaucheria* sp.

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The purpose of this investigation was to examine the ability of acid-treated *Vaucheria* sp., to remove hazardous cadmium(II) ions from synthetic wastewater. The impact of variables such as solution pH, adsorbent dosage, initial metal ion concentration, alga-Cd(II) contact duration and temperature were examined *via* batch experiments. Using Langmuir, Freundlich and Temkin adsorption models, the equilibrium isotherm constants were calculated. The equilibrium data were found to be more compatible with the Langmuir model. At pH 5, 0.6 g of algal dosage, an equilibrium time of 70 min and a temperature of 318 K, the maximal adsorption capacity achieved by *Vaucheria* sp. biomass for Cd(II) was 92.59 mg/g. Adsorption kinetics were analyzed using both the pseudo-first-order and pseudo-second-order models, with the latter model demonstrating to be more suitable for the adsorption process by comparing R^2 values. The process was determined to be endothermic ($\Delta H^o > 1$) and spontaneous ($\Delta G^o < 1$) by analyzing the thermodynamic parameters. The biomass was pre-treated to a variety of chemicals in order to improve metal sorption capacity and it emerged that acid pretreatments significantly boosted the metal sorption capacity. Overall, the findings suggest that acid-treated *Vaucheria* sp. might be a cost-effective and beneficial adsorbent biomass for removing Cd(II) metal ions from synthetic wastewater.

Keywords: Adsorption, Cadmium ions Cd(II), *Vaucheria* sp., Synthetic wastewater, Equilibrium isotherm, Kinetic studies.

INTRODUCTION

The accumulation of toxic heavy metals in the food chain and the persistence of these metals in the environment make them a significant source of global pollution [1]. Removing harmful heavy metal ions from the wastewater and repurposing them is crucial from an ecological standpoint. Cadmium is an extremely toxic heavy metal that poses significant risks to human health and the environment. The largest contributions to Cd(II) pollution in wastewater streams come from a wide range of industries, such as battery manufacture, paint, pigment or fertilizer production, electroplating, smelting, alloy production and mining [2]. Long-term exposure to high levels of cadmium can cause renal failure, bone degradation, liver damage and even cancer [2]. The discharge limits for cadmium in wastewater and drinking water are 0.1 and 0.05 mg L^{-1}, respectively [3]. Therefore, it is essential to remove this toxic metal ions from the drinking water and wastewater systems.

To remove heavy metals from wastewater, various techniques such as evaporation, oxidation, reduction, chemical precipitation, coagulation, reverse osmosis, electrochemical process, ion exchange and surface adsorption are commonly employed [4,5]. However, when the concentration of dissolved metals ranges between 1 and 100 mg L^{-1}, these techniques may be ineffective or too expensive. Traditionally, the adsorption method for pollutant recovery uses activated carbon, which may be expensive and has a limited cycle duration. The last decade, however, has seen the synthesis and use of new materials such as nanocomposites in sorption and environmental applications [6]. Biosorption, an improved adsorption process that uses renewable natural resources as the adsorbent, is a greener alternative. The vast availability of many types of biomass makes this approach extremely promising [7].

The widespread occurrence of algae in nature has led to extensive research on their ability to sorb heavy metals [8-19]. These studies have shown that the presence of biopolymers on the surface of the cell wall is responsible for the exceptionally high efficiency of heavy metal adsorption. Metal ions can be bound to the biopolymers through their functional groups, such as amines, carboxyls, hydroxyls and sulfates [20]. However,

further study is needed to optimize the efficiency of this adsorption process for maximum removal, which could pave the way for its widespread application.

Previous works have also shown that the chemical and mechanical stability of biosorbents may be enhanced *via* pre-treatment of biomass by decreasing total organic carbon content, swelling volumes and attrition loss [21,22]. Therefore, the purpose of this study is to examine the potential for Cd(II) uptake by pre-treated *Vaucheria* sp. from synthetic wastewater. Moreover, this work also aimed to pre-treating the algal biomass with selected chemicals to activate the algal biomass and check its adsorption capacity improvements. Other goals of this work include characterizing the algal biomass by infrared spectrum analysis for Cd(II) sorption onto algal biomass and then applying several adsorption isotherms and kinetic models to better understand the adsorption mechanisms.

EXPERIMENTAL

Algae (adsorbent) preparation: This investigation used algal biomass, *Vaucheria* sp., as an adsorbent, obtained from the nearby pond. The natural substance was rinsed thoroughly with regular and distilled water to eliminate any remaining impurities. It was oven-dried for a day at 348 K followed by mechanically grinding. The dried biomass was sieved to collect 0.1 mm sized particles that were used further for experiments. The dried algal biomass was pre-treated with 0.1 M HCl and for this 50 g of dried biomass was soaked with 500 mL 0.1 M HCl and continuously shaken for 2 h at 318 K using a magnetic stirrer. It was filtered using a Whatman filter paper and washed with distilled water several times until neutral adsorbent biomass was obtained. This acid-treated biomass was then oven dried at 358 K for a day and stored in a vacuum desiccator until further used.

Preparation of synthetic wastewater (adsorbate): Initially, 1000 mg/L of Cd(II) stock solution was prepared by dissolving the necessary quantity of $[Cd(NO_3)_2{\cdot}4H_2O]$ in double distilled water. This investigation utilized analytical grade chemicals and reagents purchased from Merck, Germany. The pH of the solution was measured using a digital pH meter (PERFIT, India).

Characterization: The BET technique was used to calculate the adsorbent surface area and the percentage composition of the metals such as carbon, hydrogen, nitrogen and sulphur was calculated using the elemental analysis system Vario MICRO CHNS V3.1.1 (GmbH, Germany). An FT-IR transmission investigation was conducted with KBr pellets and a Thermo Nicolet FTIR (Germany) in the 4000-400 cm^{-1} range to identify the key chemical functional groups of the adsorbent.

Methodology: The experimental procedure entailed adding a 250 mg/L Cd(II) solution to each flask containing the appropriate adsorbent dosage and then agitating the mixture at 100 rpm until equilibrium was reached at 318 K. By adding 0.1 M HCl, the pH was maintained at the optimal level of 5. After that, the biomass was separated by passing it through a membrane filter with a pore size of 0.45 µ. Before doing the analysis, the filtrate was diluted. An atomic absorption spectrophotometer Z-7000 manufactured by Hitachi in Japan and calibrated at 228.8 nm was used to quantify the amount of Cd(II) metal that was present in the filtrate.

The adsorption capacity (q_e) of *Vaucheria* sp. for Cd(II) metal absorption in each flask was calculated using the following mass balance equation:

$$q_e = \frac{(C_o - C_e)V}{M} \times V \quad (1)$$

where q_e is the algal adsorption capacity (mg/g) for metal ions; C_o and C_e are the initial and the equilibrium (residual) concentration of adsorbate, *i.e*. Cd(II) in solution (mg/L); M is the mass of algal biomass (adsorbent) employed (g); and V is the volume of metal containing solution with the adsorbent (L). Each time, the average outcome was recorded and the average standard deviation was 1.2%.

Adsorption isotherms and kinetic modeling studies: For the purpose of determining the maximum adsorption capacity, q_e (mg/g), the Langmuir, Freundlich and Temkin isotherms were utilized [23-25]. The conventional equation for the Langmuir adsorption isotherm is presented with the assumption of monolayer coverage on the surface of sorbent (eqn. 2).

$$\frac{1}{q_e} = \frac{1}{Q_o} + \frac{1}{bQ_oC_e} \quad (2)$$

where q_e and Q_o represent the equilibrium adsorption capacity and the maximal adsorption capacity (mg/g), respectively. In this equation, C_e stands for the equilibrium concentration of adsorbate (mg/L) and b is the Langmuir constant.

The equation for the dimensionless separation factor (R_L) is represented as:

$$R_L = \frac{1}{(1 + bC_o)} \quad (3)$$

where R_L reflects the sort of isotherm, which may be either favourable (R_L value in between 0 and 1), irreversible (R_L value equal to 0), linear (R_L value equal to 1) or unfavourable (R_L value greater than 1), depending on where it falls on the scale.

When sorption occurs in layers onto a rough surface, the Freundlich isotherm occurs, which is represented as:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (4)$$

where n is the adsorption strength and K_F is the Freundlich constant.

Temkin isotherm can be represented as:

$$q_e = \frac{RT}{b_T} \ln(A_T C_e) \quad (5)$$

where b_T and A_T are the constants associated with the heat of adsorption, R is the gas constant (8.314 J mol^{-1} K^{-1}) and T is the absolute temperature in Kelvin.

Furthermore, pseudo-first-order and pseudo-second-order kinetic models were used to better understand Cd(II) adsorption onto the algal surface [26,27]. These models are linearized as follows:

Pseudo-first-order model:

$$\log(q_e - q_t) = \log q_e - \frac{k_{1,ads}}{2.303} t \quad (6)$$

Pseudo-second-order model:

$$\frac{t}{q} = \frac{1}{k_{2,ads} q_e^2} + \frac{1}{q_e} t \quad (7)$$

where k_1 and k_2 are the rate constants for pseudo-first-order and pseudo-second-order, respectively and q_e (mg/g) is the equilibrium adsorption of Cd(II), q_t and q (mg/g) are the adsorption amounts at time t.

Thermodynamic studies: In order to get insight into the overall efficiency of the system, the thermodynamic parameters such as Gibbs free energy change (ΔGº), enthalpy change (ΔHº) and entropy change (ΔSº) were analyzed using the standard equations. The mathematical equations are as follows:

$$\Delta G° = -RT \ln (b) \quad (8)$$

$$\ln\left(\frac{b_2}{b_1}\right) = -\frac{\Delta H°}{R}\left(\frac{1}{T_2} - \frac{1}{T_1}\right) \quad (9)$$

$$\Delta G° = \Delta H° - T\Delta S° \quad (10)$$

RESULTS AND DISCUSSION

Characterization studies: Acid-treated algae with a particle size of 0.1 mm were employed and their surface area was measured to be 3.248 m^2/g using a Quantasorb surface area analyzer. The elemental results for carbon, hydrogen, nitrogen and sulphur were 35.20%, 4.590%, 4.29% and 0.74%, respectively.

Table-1 lists the FT-IR absorption peaks that were found for the surface functional groups of the unloaded (raw) biomass and their changes following cadmium(II) ions sorption. When the dominant IR signal shifts in wavenumber, it means that new bonds are being formed on the biomass surface between the adsorbate and the adsorbent. Changes in the wavenumbers of individual peaks, indicating heterogeneity in the adsorbents, led researchers to speculate that hydroxyl, amide and carboxyl groups could play a role in the adsorption of Cd(II) ions onto *Vaucheria* sp. Related studies on the algal adsorption reported similar findings [11,14,19].

Optimizing basic parameters for cadmium removal: Acid-treated algal biomass was used and its capacity to adsorb Cd(II) was studied and optimized under varying conditions including pH, algal dosage, temperature and algal-Cd(II) contact duration.

Impact of pH: In an investigation conducted at 318 K, with initial Cd(II) ion concentrations of 150 and 250 mg L^{-1}, the impact of pH levels ranging from 2.11 to 7.11 on *Vaucheria* sp. Cd(II) ions adsorption capability was examined. Fig. 1 demonstrates that Cd(II) adsorption is highest within the pH range of 2.11 to 5.04, but decreases when the pH increased beyond that range. It is due to the water chemistry and metal binding sites on the cell surface that are influenced by the pH of solution. At low pH, due to the presence of hydronium ions (H_3O^-), the Cd(II) ions are repelled and not associated with the ligands present on the cell wall. But as the pH increases, ligands like carboxyl, phosphate, imidazole and amino groups become exposed and attract the positively charged metal ions and facili-tate their adsorption onto the cell surface. However, for pH levels greater than 5.04, the complexation of Cd(II) by OH^- groups hinders metal adsorption, leading to a decrease in fixation. Similar findings on the impact of pH on Cd(II) adsorption have been reported earlier [19].

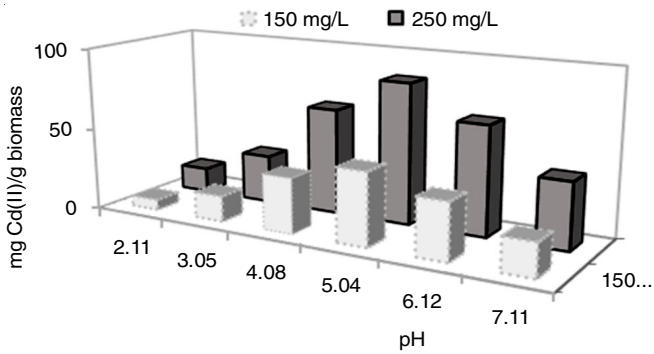

Fig. 1. Impact of solution pH on Cd(II) adsorption onto acid-treated algal biomass (*Vaucheria* sp.)

Impact of algal dosage: Fig. 2 depicts the impact of adsorbent dosage on Cd(II) ion adsorption by employing a range of biomass doses (0.1-1.0 g L^{-1}). The results revealed that increasing the amount of biomass in solution, significantly enhanced the efficiency of adsorption. To examine the impact of adsorbent dosage, different quantities of adsorbent were added to 50 mL Cd(II) solution having a concentration of 250 mg L^{-1} and a pH of 5.04. The maximum adsorption of metal ions was achieved at a biomass dosage of 0.6 g L^{-1} and at higher doses, the adsorption was essentially unchanged. This trend can be attributed to the fact that higher biomass concentrations result in the partial agglomeration of biomass, which decreases the effective surface area for adsorption. Furthermore, the adsorption capacity (q_e = 32 mg g^{-1} at 1.0 g L^{-1} dosage) decreased as the adsorbent dose increased for a given Cd(II) concentration (250 mg L^{-1}). These findings established 0.6 g L^{-1} of biomass as the optimal dose for future research, where the adsorption extent is approximately 75% (Fig. 2b).

TABLE-1
FTIR BAND POSITIONS OF *Vaucheria* sp. ALGA UNLOADED AND LOADED BIOMASS WITH Cd(II)

Vaucheria sp. band positions of unloaded (raw) biomass (wavenumber in cm^{-1})	*Vaucheria* sp. band positions when loaded with Cd(II) (wavenumber in cm^{-1})	Bonds that are a component of functional groups
3423	3428	Bonded –OH and N–H groups
2925	2927	C-H stretching
1644	1649	Amide I band, chelate stretching due to C=O
1535	1538	Amide N–H bending
1432	1429	–C–N stretching
1068	1072	PO_4^{3-} stretching

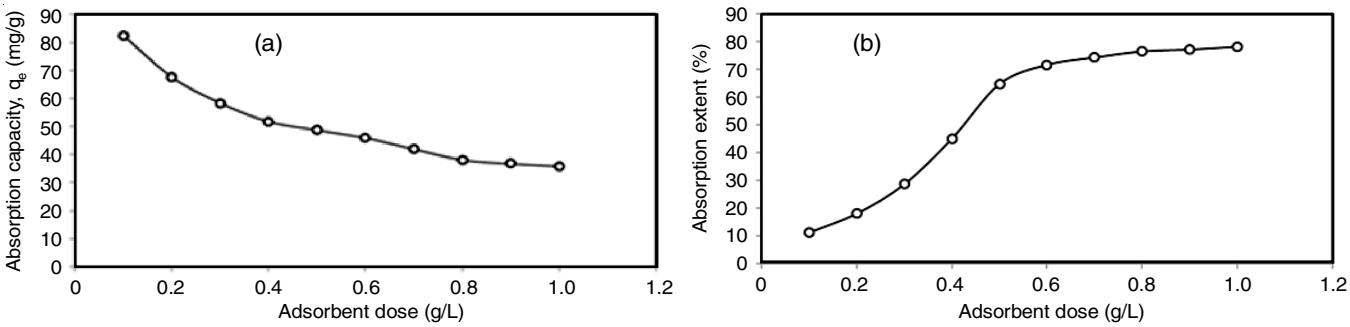

Fig. 2. Impact of adsorbent dosage on Cd(II) adsorption in terms of (a) adsorption capacity (b) Adsorption extent %, onto acid-treated algal biomass (*Vaucheria* sp.)

Impact of temperature: The cadmium adsorption isotherms of adsorbent at 298, 308 and 318 K are illustrated in Fig. 3, which demonstrates an endothermic process as the adsorption capacity increases with temperature from 298 to 318 K and the optimal solution temperature was found to be 318 K. The boundary layer thins out and the diffusion rate increases as the temperature rises. The metal(II) ions may be more easily removed if the solubility is improved by heating to a high temperature. For a variety of other adsorbent systems, similar results have been achieved in the past too [11]. On the other hand, Tüzün *et al*. [12] reported the temperature-independent effect of Cd(II) uptake on adsorption capacity. Alternatively, some findings also reported that the adsorption capacity is reduced at higher temperatures [14]. Benguella & Benaissa [28] observed a more complicated scenario for chitin towards the adsorption of Cd(II) ions, where sorption capacity first increased and then decreased. Thus, the literature survey shows that temperature affects adsorption by biomaterials differently.

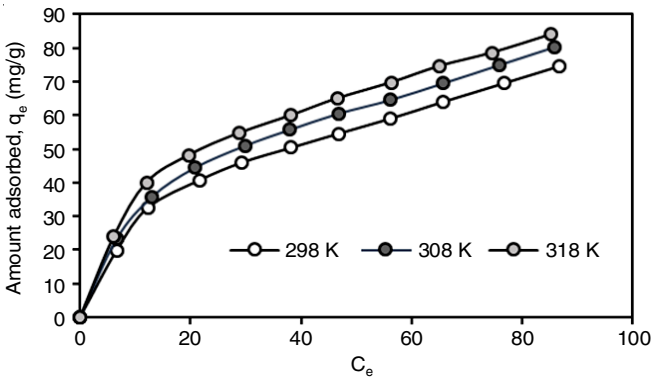

Fig. 3. Impact of temperature on Cd(II) adsorption onto acid-treated algal biomass (*Vaucheria* sp.)

Impact of contact time: Optimizing the adsorption applications can be achieved by modifying the contact duration and Fig. 4 illustrates the impact of contact time between adsorbent (algal biomass) and adsorbate (synthetic wastewater) on the adsorption capacity of *Vaucheria* sp. at two initial cadmium(II) ions concentrations. After 70 min, there is no change in the concentration of the residual metal ions and the curve rapidly approaches the time. This represents the point at which the metal ions concentration is assumed to reached equilibrium.

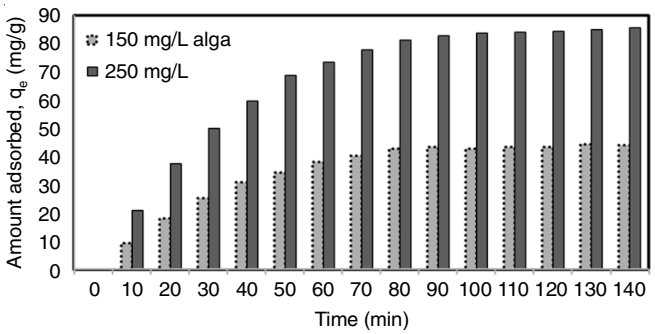

Fig. 4. Impact of contact duration on Cd(II) adsorption onto acid-treated algal biomass (*Vaucheria* sp.).

Adsorption isotherms: The Langmuir, Freundlich and Temkin isotherms were employed to find a good fit with the experimental results. Plots (Fig. 5a-c) were used to calculate the adsorption and correlation coefficients, which are shown in Table-2. The Langmuir isotherm relies on the idea that the binding sites on a monolayer surface are uniformly distributed. Adsorption capacity (q_e) was found to be 92.59 mg/g at 318 K with good R^2 values (> 0.99) using the Langmuir model. This investigation showed that the adsorption process is favourable at all temperatures.

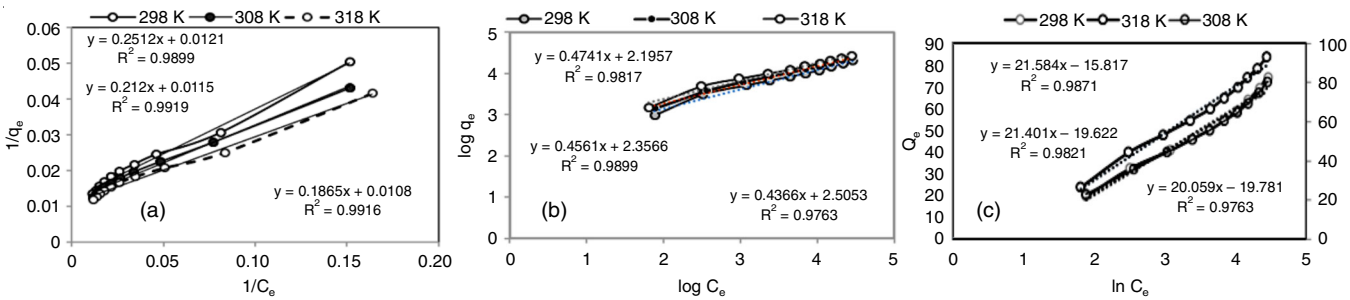

Fig. 5. Adsorption isotherms (a) Langmuir model (b) Freundlich model and (c) Tempkin model, of Cd(II) adsorption onto acid-treated algal biomass (*Vaucheria* sp.)

TABLE-2
THERMODYNAMIC PARAMETERS AND ISOTHERM CONSTANTS FOR Cd(II) ADSORPTION ONTO ACID-TREATED ALGAL BIOMASS (*Vaucheria* sp.) AT THREE TEMPERATURES

Parameters evaluated	Cd(II) sorption at three temperatures		
	298 K	308 K	318 K
Langmuir isotherm			
b (L mg^{-1})	0.04816	0.05424	0.0579
b (m mol^{-1})	5.4136	6.097	6.508
q_e (mg g^{-1})	82.644	86.956	92.592
R^2	0.9899	0.9919	0.9916
Dimensionless separation factor			
R_L	0.0007	0.0006	0.0006
Thermodynamic parameters			
ΔG° (kJ mol^{-1})	-21.298	-22.318	-23.215
ΔS° (J $mol^{-1}K^{-1}$)	0.0958	0.0960	0.0958
ΔH°* (kJ mol^{-1})	7.252		
Freundlich isotherm			
n	2.1092	2.1925	2.290
K_F (mg g^{-1})	8.9863	10.555	12.247
R^2	0.9817	0.9899	0.9763
Temkin isotherm			
A_T (L mg^{-1})	0.3730	0.3997	0.4805
b_T (g kg mg^{-1} mol^{-1})	20.059	21.401	21.584
R^2	0.9763	0.9821	0.9871

*Difference between 298 and 318 K.

Using the Freundlich isotherm, an empirical equation, the sorption sites and energy are assumed to be distributed in an exponential pattern. As can be seen from Table-2, Cd(II) adsorbs onto algal biomass in a selective and heterogeneous manner, with parameter 'n' values greater than 1 indicating a favourable adsorption process. At various temperatures, the Langmuir model had the highest correlation coefficient, followed by the Freundlich and Temkin models. Thus, according to the results, the Langmuir isotherm is superior to the other two isotherms studied, in terms of the correlation coefficient (R^2) being closer to 1.

Thermodynamic studies: The negative result for ΔG° suggests that Cd(II) ions is being adsorbed spontaneously by *Vaucheria* sp. The value increases with increasing temperature, showing that adsorption is more favourable at higher temperatures. Cadmium(II) ions is being taken up by the tested algae endothermically, as evidenced by the positive ΔH° value (Table-2). Increasing temperature also enhances the adsorption capacity, providing further support for the aforementioned conclusion. The growing disorderliness at the solid-solution interface is reflected as indicated by the positive value of ΔS°. These results are consistent with those reported by other researchers [13] investigating the removal of Cd(II) ions using algal biomass.

Kinetic studies: Table-3 displays the results of the kinetics of Cd(II) adsorption onto the adsorbent surface. The pseudo-first-order and pseudo-second-order adsorption rate constants k_1 and k_2, as shown in Table-3, were calculated from the slope of the linear plot of ln (q_e–q_t) *vs.* time (Fig. 6a) and t/q_t *vs.* t (Fig. 6b). Despite both kinetic models having strong correlation coefficients (> 0.98), Table-3 data demonstrate that pseudo-first-order R^2 values are better than pseudo-second-order values. Fig. 6a showed that the Lagergren pseudo-first-order kinetic plot fit better for the adsorption of Cd(II) onto algal biomass as they better follow a straight line (Fig. 6b). Similar reports have been reported by Durbaz & Rossta [11] for the adsorption of Cd(II) onto *Parachlorella* sp. alga [11].

Comparative studies: The adsorption capacity of algal biomass *Vaucheria* sp. for Cd(II) ions is much higher than the other studied algal biomass (Table-4). This demonstrates its efficiency, making it a promising biomaterial for the removal of toxic metal ions.

TABLE-3
SUMMARY OF Cd(II) SORPTION DATA FROM MULTIPLE KINETIC MODELS FOR ACID-TREATED ALGAL BIOMASS (*Vaucheria* sp.)

Initial Cd(II) conc. (mg/L)	Pseudo-first-order model			Pseudo-second-order model		
	k_1 ($\times 10^{-3}$ min^{-1})	q_e (mg g^{-1})	R^2	$k_2 \times 10^{-3}$ (g mg^{-1} min^{-1})	q_e (mg g^{-1})	R^2
150	32.472	68.093	0.993	0.382	56.818	0.982
250	32.933	127.849	0.996	0.227	106.383	0.989

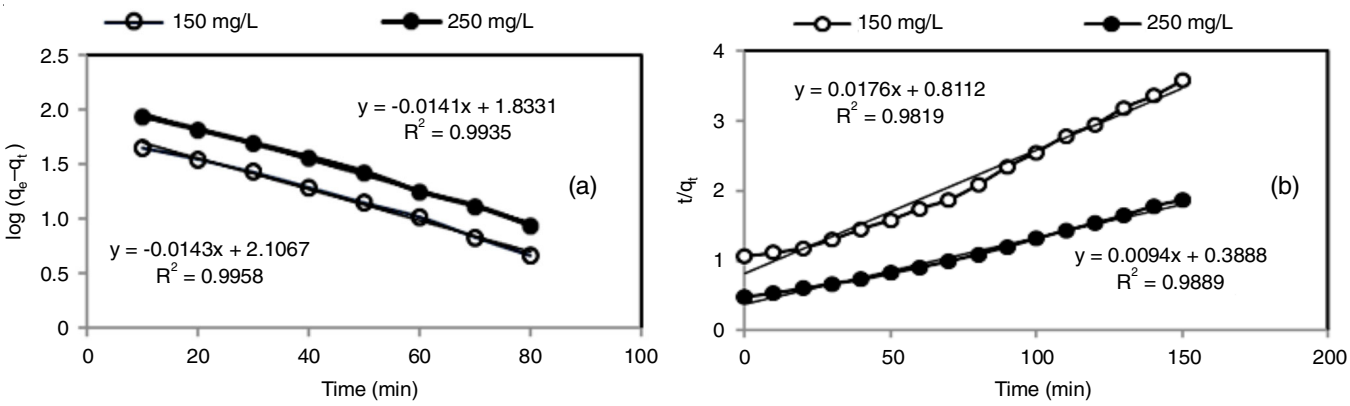

Fig. 6. Kinetic modeling (a) pseudo-first order model and (b) pseudo-second order model of Cd(II) adsorption onto acid-treated algal biomass (*Vaucheria* sp.)

TABLE-4
Cd(II) METAL ION ADSORPTION CAPABILITIES OF DIFFERENT ALGAL BIOMASSES IN AQUEOUS SOLUTIONS

Algal biomass	Adsorption capacity (q_e) (mg/g)	Ref.
Ulva pertusa	2.92	[9]
Enteromorpha linza	8.43	[9]
Ulva lactuca	29.1	[10]
Parachlorella sp.	96.72	[11]
Chlamydomonas reinharditili	42.6 ± 0.54	[12]
Caulerpa scalpelliformis	111.11	[13]
Caulerpa fastigiata	16.13	[14]
Chlorella vulgaris	86.6	[15]
Codium vermilara	21.8	[16]
Spirogyra insignis	22.9	[16]
Spirulina platensis	98.04	[17]
Gelidium algae	18.0	[18]
Oedogonium hatei	88.2	[19]
Vaucheria sp.	92.59	This study

Effect of pre-treatment of algal biomass: By pre-treating the dried algal biomass with different chemicals, such as hot water (323 K for 20 min), mineral acids (HCl, HNO_3), bases (NaOH), ammonium acetate and organic reagents like acetone, formaldehyde and methanol, respectively, efforts were made to increase the removal of Cd(II) from synthetic wastewaters. Table-5 shows that hydrochloric acid pretreatment significantly improved the Cd(II) sorption capacity of *Vaucheria* sp. (upto 28.1%). This conclusion is consistent with the findings of Mehta *et al.* [29], who observed a 39% increase in metal binding capability after acid pre-treatment.

TABLE-5
EFFECT OF SEVERAL PRETREATMENTS (60 min, 0.1 mM OF ALL CHEMICALS) ON THE CAPACITY OF *Vaucheria* sp. TO ADSORB Cd(II)

Pre-treating agent	Percentage change in metal sorption ability
Acetone	-32.3 ± 1.1
Methanol	-28.6 ± 0.8
Ammonium acetate	-20.5 ± 1.3
Hot water (323 K for 20 min)	-16.4 ± 0.4
Formaldehyde	-1.8 ± 0.9
HNO_3	5.1 ± 0.7
NaOH	12.9 ± 1.0
HCl	28.1 ± 0.8

The symbols – and + denote decrease and increase in percentage change relative to control

Disposal of the spent biomass: The used biomass was sterilized by microwave irradiation for 10 min before being encapsulated in deep concrete cavities. This was done to reduce the environmental impact of cadmium-laden biomass.

Conclusion

In this study, the adsorption of cadmium metal ions onto acid-treated alga *Vaucheria* sp. from synthetic wastewater was investigated using a series of batch tests. The most effective adsorption was achieved with a pH of 5.04, a dose of 0.6 g/L of algal biomass, a contact time of 70 min and a temperature of 318 K. The Langmuir adsorption isotherm involving monolayer coverage of Cd(II) ions on the adsorbents' outer surface was found to be the best-fit model in terms of coefficient correlation values (R^2 values highest for the Langmuir model) and the adsorption capacity (q_e) calculated from the Langmuir isotherm was 92.6 mg/g for 250 mg/L initial Cd(II) ions concentration. Adsorption of Cd(II) onto algal biomass was shown to occur with pseudo-first-order kinetics, according to the kinetic investigations. It was also shown that activating algal biomass by acid pre-treatment boosted its adsorption capacity significantly. Therefore, acid-treated algal biomass was employed throughout all trials. Also, while evaluating the ability of several algal biomasses to adsorb Cd(II) metal ions, the studied algae, *Vaucheria* sp., performed well in terms of adsorption capacity. Therefore, it is reasonable to conclude that acid-treated *Vaucheria* sp. biomass can be used as a low-cost and environmental friendly material to remove Cd(II) from synthetic wastewater.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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