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Biosorption characteristics of *Pennisetum glaucum* for the removal of Pb(II), Ni(II) and Cd(II) ions from aqueous medium

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ABSTRACT

Kinetics and thermodynamic studies were carried out for the removal of heavy metals (Pb^{+2} , Ni^{+2} and Cd^{+2}) from aqueous solution by using *Pennisetum glaucum*. Surface morphology and elemental analysis were investigated with scanning electron microscope and energy dispersion X-ray spectroscopy analysis. Functional group characteristics were found by a Fourier transform infrared spectrophotometer. The identified groups as binding sites for metal ions were found to be carbonyl (C=O), hydroxyl (OH) and amide (NH). Process parameters such as biosorbent dose, contact time, solution pH, initial metal ion concentration and so on were also studied in batch mode. The optimum pH for the studied process was found to be 5–6. Equilibrium data obtained from the batch process fitted well with the Langmuir isotherm and the process followed pseudo second-order kinetics. *P. glaucum* was found to be economical, ecofriendly and an effective biosorbent in sequestration of heavy metals from water.

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Introduction

Evolvement of technology is the basis of industrialization; hence, the increased production of utilities is required to sustain the societal needs. Industrialization, although pivotal to the structure of a developing country, comes with the great risk of environmental deterioration (1, 2). Industrial effluents in huge quantities are dumped untreated into water bodies. These pollutants show high solubility in the aquatic environment leading to bioaccumulation (3). Elevation of toxic elements in the water bodies poses a threat to the aquatic lives as well as the agricultural areas that are dependent on the water bodies for irrigation purposes, as even in extremely small quantities they are hazardous. The indigestion of these elements by humans through the food chain can lead to allergic reactions, kidney damages, dysfunctional organs, renal disorders, bone deformation and so on if they exceed the limit recommended by the World Health Organization (4, 5). In females, cadmium affects the reproductive system and has a detrimental effect on kidney and the nervous system (6). Nickel becomes the source of many incurable diseases such as renal edema, cystic fibrosis, skin

infections and allergies, it also leads to fatal disease, i.e. lung cancer (7). Chronic lead toxicity symptoms include memory deterioration, reduced nerve conductivity, peripheral neuropathy, muscle weakness, reduced dermal sensitivity and it affects the fetus developments and can cause miscarriages, still births and anemia (8, 9)

Industrial wastewater treatment is necessary to protect man and the environment. Many conventional methods are being used for the treatment such as chemical precipitation (10), ion exchange (11), membrane filtration (12), photo catalysis (13), coagulation (14), flotation (15) and so on. These methods, although significant have some disadvantages such as sludge formation, are expensive and difficult to practice (16). But their economic issue is valid enough for the industrialist to say no to them. Cheap and economically feasible solution for this serious issue is required.

The aim of this research work is to find the solution of heavy metal contamination for developing countries like Pakistan where multiple aspects must be kept in mind while finding an ecofriendly solution of heavy metal pollution. Selection of *Pennisetum glaucum* as biosorbent has multiple reasons, it has tendency to grow in arid

and semi-arid land where other plants such as rice, wheat, sorghum and barley cannot grow. The specie is adapted to grow in variety of soil even in high saline infertile soil where other plants of this family loss their nutritive value. So the growth of this plant will not affect the production of vital crops. It can bear drought stress so its production is favorable in Pakistan where water crises are crucial. It is a novel plant as a biosorbent so a complete biosorption study is required for its acceptance as a biosorbent.

Material and methods

Collection of biomass

P. glaucum was collected from the local farms of Lahore, Pakistan. It was chopped for easy handling then rinsed with distilled water to remove dust and particles other than *P. glaucum* and then air dried to remove all the moisture. Dried biomass was grinded and sieved through to mesh size 70–80 ASTM. It was then washed with distilled water to remove the soluble lignin and then it was oven (Memert) dried at 60–70°C.

Chemicals and instrumentation

All chemicals and reagents used in the study were of analytical grade. De-ionized or distilled water was used throughout the experiment. All glassware was cleaned by using nitric acid followed by distilled water washing. Nickel sulfate heptahydrate, lead nitrite, cadmium nitrate, sodium hydroxide and hydrochloric acid were bought from Scharlaw. Stock solutions of metal ions (1000 mg/L) were prepared for each metal ion using their corresponding salts. Standard solutions of metal ions of varying concentrations were prepared by diluting the suitable aliquots of the prepared stock solutions. A Fourier transform infrared spectrophotometer (FTIR) was used to characterize the biomass in terms of surface functionality. A scanning electron microscope (SEM, FEI Nova Nano SEM 450) was used for surface morphology and an energy dispersion X-ray spectroscopy (EDX, Nova SEM 450) for elemental analysis. An atomic absorption spectrophotometer (AAS; Perkin Elmer, Analyst 100) was used to determine the concentration metallic species at various stages.

Batch biosorption studies

The effect of experimental variables such as dose of biosorbent, concentration of metal ions, contact time, solution pH and temperature was investigated independently in batch

mode for Pb^{+2} , Ni^{+2} and Cd^{+2} biosorption on *P. glaucum*. Each parameter was studied step by step while keeping all other parameters constant. For the biosorption process, 50 mL of 50 mg/L solution of respective metal ion was taken into a conical flask and placed on an orbital shaker. It was allowed to contact with a pre-fixed quantity of biomass while constant shaking for a pre-defined time period. After shaking, the flask was removed and the solution was filtered. After filtration, the concentration of Ni^{+2} , Pb^{+2} and Cd^{+2} in the filtrate was determined by AAS (Perkin Elmer Analyst 100). Regulation of pH during the experiments was done by using HCl (0.1 M) and NaOH (0.1 M) solution and shaking speed was fixed at 100 rpm. The uptake capacity q_e (mg/g) was determined by the following formula (Equation (1)):

$$q_e(\text{mg/g}) = v \times \frac{(C_i - C_f)}{m} \quad (1)$$

Here v (mL) is the volume of solution under study, m (g) is the mass of biomass taken, C_i and C_f represent the initial and final concentrations of metal ions in mg/L.

All the experiments were performed in triplicate and results reported here are the mean values. The graphs were plotted using Microsoft Excel 2013 program. The suitability of the studied mathematical models was investigated by performing regression analyses (R^2 values). Root mean square error (RMSE) was also calculated to evaluate the error of model predictions (Equation (2)):

$$\text{RMSE} = \sqrt{\frac{\sum (q_{e(\text{cal})} - q_{e(\text{exp})})^2}{N}} \quad (2)$$

Here, $q_{e(\text{cal})}$ and $q_{e(\text{exp})}$ represent the uptake capacities of biosorbent predicted by mathematical model and experimental findings, respectively, whereas N represents the number of data points in each set.

Result and discussion

Biosorbent characterization

The FTIR is an important tool to determine the characteristic functional group on the surface of biosorbent which may be the possible binding sites for the metal ions. FTIR data indicated the presence of hydroxyl group (–OH,

Table 1. Main functional groups in *P. glaucum* identified by FTIR.

Functional groups	Peaks (cm^{-1})
Hydroxyl group	3300
Alkyl group	2918
Carbonyl group	1637
Carboxylate stretch (acid/ester)	1178

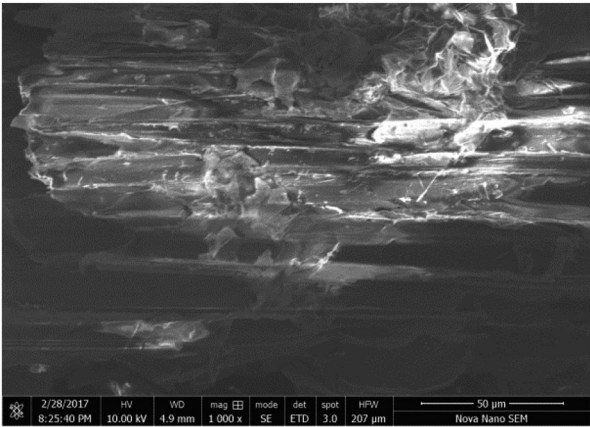


Figure 1. SEM image of *P. glaucum*.

3300 cm^{-1}), amino group ($-\text{NH}$ (str), 2918 cm^{-1}) and carbonyl group ($\text{C}=\text{O}$, 1637 cm^{-1}) (17). Peaks of functional groups are described in Table 1.

Surface morphology for biomass was determined by SEM. SEM analysis indicated the visualization of minor structural changes of the biosorbent (18). Rough morphology was visible in Figure 1 which is supportive for metal ion attachment.

Metal loaded plant biomass was also analyzed to observe the morphological changes. Shininess in the Figure 2 indicated the occupation of respective metal ions on the surface. Thus, affirming the attachment of metal ions to the binding sites of biosorption of Pb^{+2} , Ni^{+2} and Cd^{+2} on *P. glaucum*.

Table 2. Elemental analysis of *P. glaucum* by EDX.

Element	Wt%	Wt% Sigma
C	59.03	0.58
O	40.41	0.57
Si	0.16	0.02
Cl	0.05	0.01
K	0.27	0.02
Ca	0.07	0.01
Total	100	-

EDX analysis was carried out for simple as well as for metal loaded (Pb^{+2} , Ni^{+2} and Cd^{+2}) on *P. glaucum* (Table 2, Figure 3). EDX data also show the incorporation of respective metal ions when the data for simple and metal loaded biomass were compared. Data also give an idea about the possible biosorption mechanism involved. The data indicated that the studied heavy metal ions replaced calcium and potassium ions. Hence, the possible mechanism of metal attachment can be ion exchange.

Effect of adsorption dose

Experiments were conducted to check the effect of *P. glaucum* dose on biosorption of the studied metal ions. Behavior is well illustrated in Figure 4. With the increase in amount of the biosorbent, the removal percentage increases. This increase is due to the increased active and unsaturated sites for the attachment of the metal ions (19). At higher biosorbent concentration, a decreasing trend is observed, it might be due to the saturation point and aggregation (20). This step is helpful in the

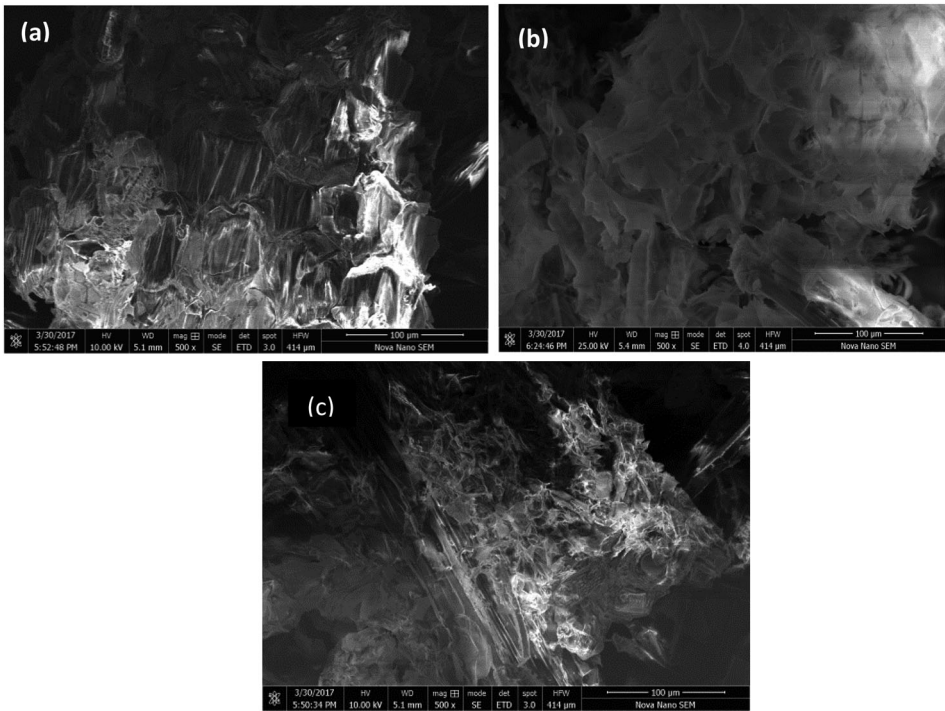


Figure 2. SEM images of Pb^{+2} (a) and Ni^{+2} (b) and Cd^{2+} (c) loaded modified biosorbent.

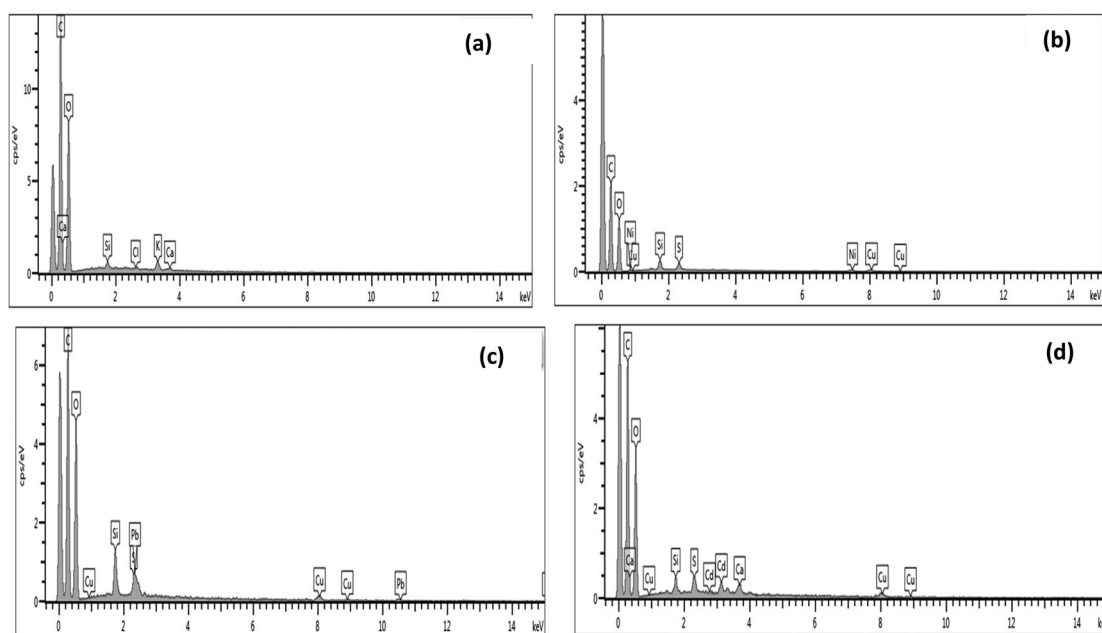


Figure 3. (a) EDX analysis of *P. glaucum*, (b) Ni^{2+} loaded *P. glaucum*, (c) Pb^{2+} loaded *P. glaucum*, and (d) Cd^{2+} loaded *P. glaucum*.

determination of quantitative uptake of metal ions by the biosorbent which is different for different metals ions.

At a lower concentration, all the Ni^{2+} , Pb^{2+} and Cd^{2+} interacted with the biosorption sites of the biomass because the ratio of the active sites to initial concentration of metal ions is large (21). While, on the other hand with the increase in the initial concentration of metal ion, this ratio became low because ions occupied maximum active sites of the biomass.

Effect of pH

The pH of the aqueous solution has a notable effect on the phenomena of the biosorption of heavy metals onto the biosorbent. The pH decides the degree of ionization, charge on the species and nature of metal ion (22). With the change in pH, the number of unsaturated sites

changes. This change can be related to the ionic state and types of the functional groups present. The optimum pH is slightly acidic (pH 5–6) as shown in Figure 5. The increase in removal percentage with the increase in pH is due to the effect that at low pH most of the active or unsaturated sites are protonated that inhibits the binding of the metal ions with these sites (23). As the pH increases, deprotonation occurs due to the availability of hydroxyl ions and the active sites become free for the attachment of the metal ions due to the ion exchange mechanism hence uptake capacity increases. Figure 5 also shows that uptake capacity of *P. glaucum* decreases at higher pH values. This is due to the precipitation of metal ions via hydroxide formation. Similar trends are observed by different adsorbents (24, 25).

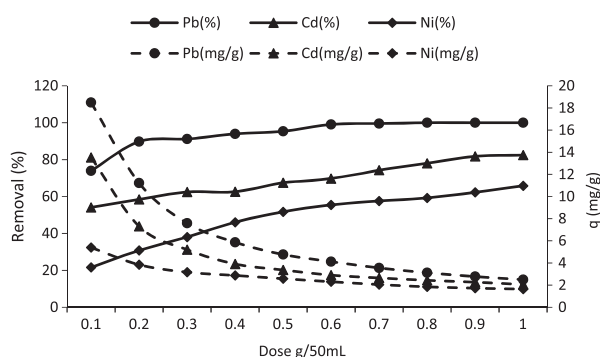


Figure 4. Effect of *P. glaucum* dose on removal of Pb^{2+} , Ni^{2+} and Cd^{2+} ions (initial conc. = 50 mg/L, time = 45 min, pH = 6, agitation speed = 125 rpm).

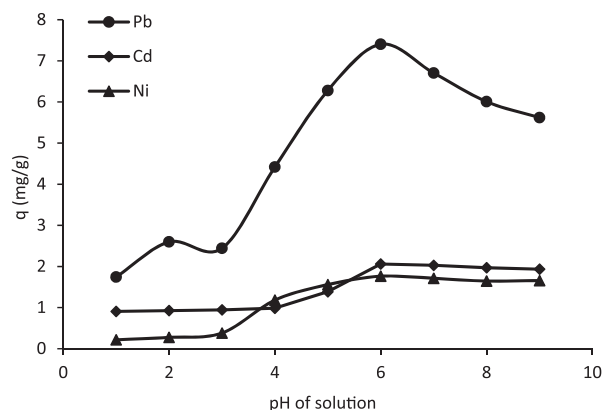


Figure 5. Effect of solution pH on the removal of Pb^{2+} , Ni^{2+} and Cd^{2+} ions (initial conc. = 50 mg/L, time = 45 min, dose = 100 mg/50 mL, agitation speed = 125 rpm).

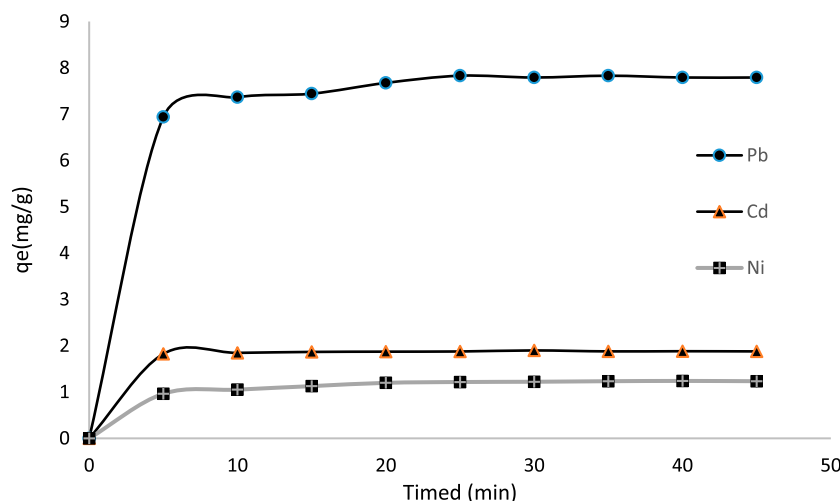


Figure 6. Effect of time on the biosorption of Pb^{2+} , Cd^{2+} and Ni^{2+} on *P. glaucum* (initial conc. = 50 mg/L, pH = 6, dose = 100 mg/50 mL, agitation speed = 125 rpm).

Effect of contact time – kinetic study

The time required for attaining equilibrium and maximum metal uptake for fixed concentration has been investigated and illustrated in Figure 6. The figure shows that there was a rapid uptake of metal ions by *P. glaucum* in the start, then gradually the rate of uptake decreases and finally became constant. The optimum time to attain equilibrium for Ni removal was found to be 40 min. While for lead 35 min and for cadmium 30 min were observed. The reason was attributed to the fact that after the equilibrium time maximum binding sites of the biosorbent became occupied and repulsive forces were developed between the solute particle in solid and bulk phases (26, 27).

Active sites are not the sole factor that is responsible for biosorption kinetics. The ease in accessibility of metal ions to the surface of biosorbent is also important. The most popular kinetic models, pseudo first-order (PFO, Equation (3)) and pseudo second-order (PSO, Equation (4)) models were employed to investigate the biosorption kinetics:

$$\ln(q_e - q_t) = \ln q_e - k_1 t, \quad (3)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + t/q_e, \quad (4)$$

where q_e (mg/g) and q_t (mg/g) are the uptake capacities of *P. glaucum* at equilibrium and at instantaneous time t , respectively. k_1 (min^{-1}) and k_2 (mg/g/min) are the PFO and PSO constants.

PFO assumes that rate of biosorption is directly proportional to the number of free active sites. The kinetic data for a fixed concentration of metal ions (50 mg/L) were plotted using Equation (3). The regression analysis shows that PFO is insufficient to explain the biosorption kinetic ($R^2 < 0.98$). In addition, the experimental q_e values and calculated q_e values for the studied metal ions are considerably different which reinforce the above statement (Table 3).

PSO determines that the rate of biosorption is directly proportional to the square of biosorption sites and metal ion concentration. The linear equation was employed to analyze the data for this specific model. The correlation coefficient (R^2) values close to 1 show the goodness of fit of the PSO model to kinetic data. The experimental and calculated q_e values closely resemble each other, indicating that the kinetic mechanism is governed by the PSO model.

Effect of temperature – thermodynamic study

The thermodynamic study is the prime factor to describe the process of biosorption. The uptake capacities of

Table 3. Kinetic parameters for the biosorption of Pb^{+2} , Ni^{+2} and Cd^{+2} on *P. glaucum*.

Adsorbates	PFO model			q_e (exp) (mg/g)	PSO model		
	k_1 (min^{-1})	q_e (cal) mg/g	R^2		k_2 (mg/g/min)	q_e (cal) (mg/g)	R^2
Pb^{+2}	0.109	1.544	0.942	7.83	10.638	7.955	0.999
Ni^{+2}	0.136	0.626	0.979	1.23	4.943	1.120	0.990
Cd^{+2}	0.073	0.106	0.976	1.90	3.094	1.889	0.999

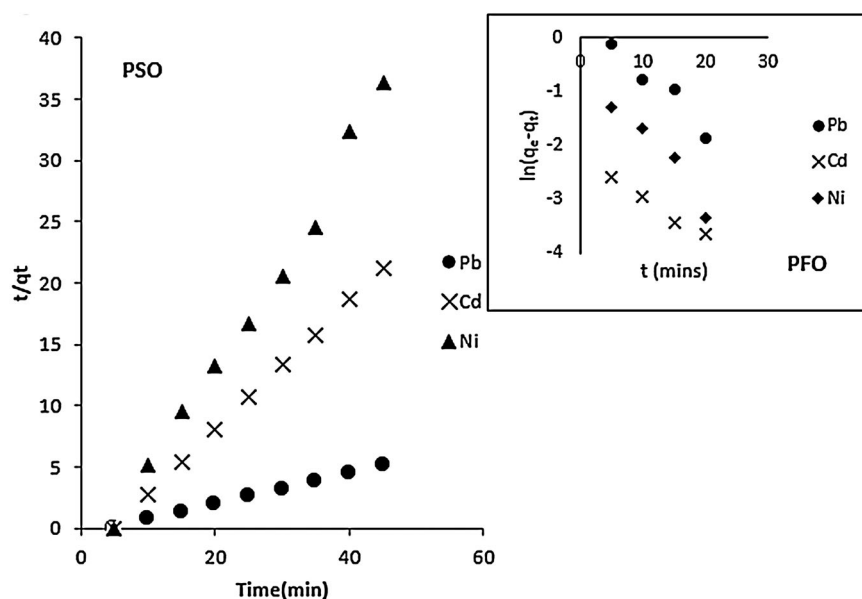


Figure 7. Plot for PFO and PSO.

P. glaucum for the studied metal ions removal as a function of temperature is shown in Figure 7. It was observed that biosorption increased with the increase in temperature that showed that the phenomenon was endothermic, biosorption increased either due to the increase in active sites or due to the increase in diffusion of the metal ion onto biosorbent. Closely a similar finding has also been reported in previous studies (28).

Enthalpy change (ΔH°), free energy change (ΔG°) and entropy changes (ΔS°) are promising factors for the determination of biosorption thermodynamics and calculated by the following equations:

$$\Delta G^\circ = -RT \ln K_D, \quad (5)$$

$$\ln K_D = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{RT}. \quad (6)$$

ΔG° (kJ/mol) is Gibbs free energy, T is temperature (K), R is universal gas constant (8.3143 J/mol/K) and K_D is the adsorption equilibrium constant derived from Equation (7). ΔS° and ΔH° were calculated from the intercept and slope by using Equation (6):

$$K_D = \frac{C_o - C_e}{C_e}. \quad (7)$$

Thermodynamic functions change their values in the biosorption process (29). Negative value of free energy changes supported the feasibility and spontaneity of the process (18). A positive value of enthalpy changes indicated that the process was endothermic in nature. This corresponds to the correct experimental findings. Entropy (ΔS°) determined the degree of randomness at the solid solution interface (30). Its positive value

enhanced the stability of adsorbed metal ions. Thermodynamic parameters are described in Table 4.

Equilibrium modeling – study of initial metal ion concentration

Interaction of sorbate with sorbent has a major contribution in optimizing and scale up the biosorption process. To investigate the famous Langmuir, Freundlich and Dubinin-Radushkevich (DR) models were employed to the equilibrium data that were obtained using different initial metal ions concentration and a fixed amount of the biosorbent. The said models were employed in a non-linear fashion and the RMSE values calculated from the respective plot are enlisted in Table 5. Langmuir (Equation (8)), Freundlich (Equation (9)) and DR models (Equation (10)) are frequently used models. A non-linear approach was employed to explain the equilibrium modeling and RMSE was set as a standard for comparison:

$$q_e = \frac{b \cdot q_m \cdot C_e}{1 + b \cdot C_e}, \quad (8)$$

$$q_e = K_f \cdot C_e^{1/n}, \quad (9)$$

$$q_e = q_m \cdot \exp(-\beta \varepsilon^2). \quad (10)$$

Table 4. Thermodynamic parameters for biosorption of Pb^{+2} , Ni^{+2} and Cd^{+2} on *P. glaucum*.

Adsorbate	ΔG° (KJ/mol)			ΔH° (KJ/mol)	ΔS° (KJ/mol)
	30°	40°	50°		
Pb^{+2}	−4.891	−5.40	−5.400	14.85	0.065
Ni^{+2}	−0.197	−0.723	−0.722	7.82	0.031
Cd^{+2}	−4.891	−5.400	−6.270	17.89	0.027

Table 5. Isotherm parameters for Pb^{+2} , Ni^{+2} and Cd^{+2} biosorption.

Adsorbates	Langmuir parameters			Freundlich parameters			DR			
	q_{\max} (mg/g)	b (dm^3/g)	RMSE	K_f	n	RMSE	q_m (mg/g)	β (mol^2/J^2)	E KJ/mol	RMSE
Pb^{+2}	15.243	0.135	0.873	3.066	2.67	3.25	11.8	1×10^{-6}	0.707	4.09
Ni^{+2}	5.534	0.067	0.413	0.171	1.36	0.312	2.99	1×10^{-5}	0.223	1.58
Cd^{+2}	5.555	0.102	0.645	0.755	2.01	0.360	3.96	1×10^{-6}	0.707	1.388

The Langmuir isotherm determines the solid liquid phase equilibrium (31). It determines the biosorption parameters and indicates the some important points like specific homogenous sites are more active for biosorption on the surface of biosorbent (9). Parameters of these models (Table 5) provided the information regarding monolayer capacity (q_{\max}) (32). When one metal occupies the surface site, no further attachment is possible and the intermolecular forces existing between site and metal ions decrease with an increase in distance. Each active site is independent in its behavior and neighboring molecules do not interfere in the process (33). The non-linear approach was used to calculate the RMSE value which is >1 which indicated goodness of the Langmuir model. It means enough active sites were available for lead attachment on the surface.

Behavior of the heterogeneous system is described well by the Freundlich isotherm. A higher value of K_f for lead indicated strong interaction strength. Lead and cadmium showed a higher value of n which determined the intensity of interaction between sorbent and sorbate (34). n represented the linearity between metal-contaminated solution and adsorption. More this value was near to 1, the more linear is the relation (35). A higher value of RMSE for lead showed that heterogeneous biosorption is not as much favorable as monolayer biosorption capacity. Data for Ni^{+2} and Cd^{+2} inferred that the RMSE value (<1) favors the multilayer biosorption and fitness to equilibrium data as shown in Figure 8(a–c).

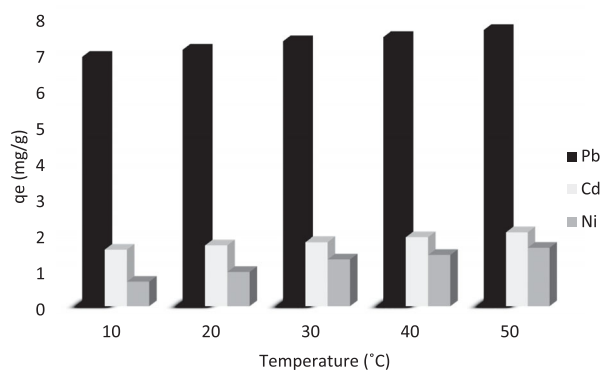


Figure 8. Effect of temperature of biosorption of Pb^{+2} , Ni^{+2} and Cd^{+2} on *P. glaucum* (initial conc. = 50 mg/L, pH = 6, dose = 100 mg/50 mL, agitation speed = 125 rpm, time = 45 min).

The DR model is more general than other isotherms. The values of q_m , β and RMSE for all selected metals are given in Table 5. It determines the information regarding the porosity of the surface and nature of biosorption. The informative value obtained by applying this model determined the nature of biosorption, for all selected metals, it was physio-sorption and not chemisorption. The value of E (kg/mol) is less than 16 for all three metals, which indicated that the interaction between metal ions and biosorbent was not chemical in nature. Values of lead cadmium and nickel are less than one which is supportive to the surface attraction behavior of the biosorption system. Observed behavior of all selected metals under the DR model is shown in Figure 8. It is concluded from the given RMSE values and metal ion behavior (Table 5 and Figure 8) that the Langmuir model fitted well to all metals as compared to other isothermal models. A comparison of the present study and some previously reported studies is tabulated in Table 6 (Figure 9).

Conclusion

P. glaucum is a drought- and saline-resistant crop and may grow in infertile land. It might be a good solution for heavy metal contamination for a country like Pakistan, where water crisis and surface water contamination are serious issues. It showed good biosorption capacity and percentage removal of heavy metals (in order $\text{Pb}^{+2} > \text{Cd}^{+2} > \text{Ni}^{+2}$). A previous study supported this interpretation (36). The results of operational parameters

Table 6. Comparison of biosorption capacity of *P. glaucum* with other materials.

Biosorbent	Metal ions	q_{\max} (mg/g)	References
Wheat straw	Pb^{+2}	0.38	Anis et al. (37)
Corncoobs	Pb^{+2}	8.29	Reddad et al. (38)
Banana Peels	Pb^{+2}	2.18	Anwar et al. (39)
<i>Triticum aestivum</i>	Pb^{+2}	0.85	Farooq et al. (40)
<i>P. glaucum</i>	Pb^{+2}	15.24	Present study
Corncoobs	Ni^{+2}	13.5	Reddad et al. (38)
Rice husk	Ni^{+2}	8.86	Bansal et al. (41)
<i>P. glaucum</i>	Ni^{+2}	5.53	Present study
Rice Straw	Cd^{+2}	13.9	Ding et al. (42)
Rice husk	Cd^{+2}	20.24	Kumar and Bandyopadhyay (43)
Corncoobs	Cd^{+2}	8.89	Reddad et al. (38)
Banana Peels	Cd^{+2}	5.71	Anwar et al. (39)
<i>P. glaucum</i>	Cd^{+2}	5.55	Present study

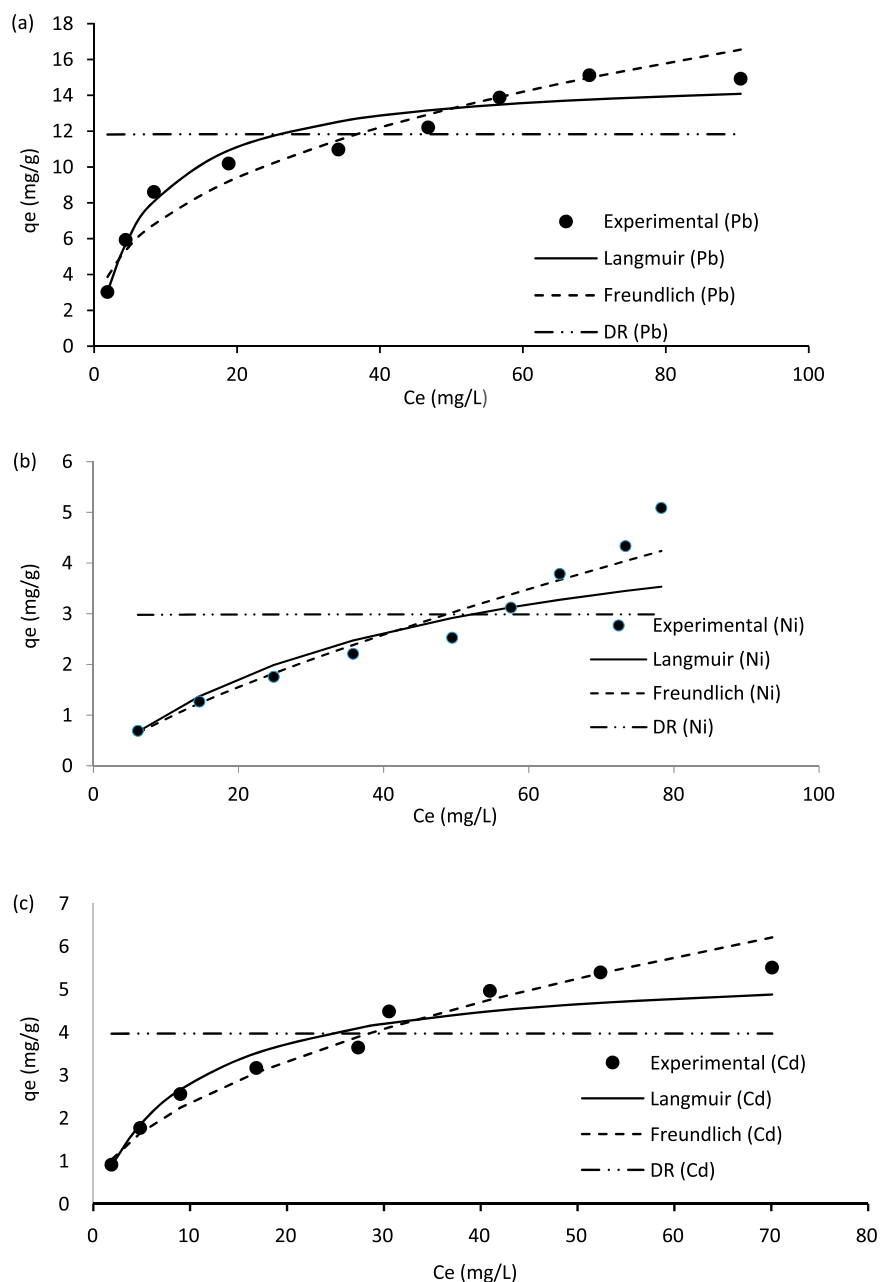


Figure 9. Non-linear isotherms plot for Pb^{+2} (a), Ni^{+2} (b) and Cd^{+2} (c) showing Langmuir, Freundlich and DR model.

indicated that the optimum pH range was 5–6 and the optimum time to attain equilibrium was 30–40 min. The non-linear approach was under consideration to study the equilibrium process and Langmuir fitted well for all selected metals with considerable q_{max} values for lead (15.243 mg/g), cadmium (5.555 mg/g) and nickel (5.34 mg/g). Freundlich was more suitable for Ni^{+2} and Cd^{+2} as compared with Pb^{+2} . Moreover, the thermodynamic parameter supported the feasibility and spontaneity of the reaction. Hence, it can be inferred that the investigated plant is efficient in its heavy metal removal characteristic behavior.

Disclosure statement

No potential conflict of interest was reported by the authors.

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Amna Yousaf is a PhD scholar at the Institute of Chemistry, University of the Punjab. Her research interest is in waste water treatment. Currently, she is working as a Lecturer in a well-renowned Institute, Kinnaird College, Lahore.

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Faraz Shahzad Chawla has completed his M.Sc from Govt. College University, Lahore, Pakistan. Previously, he worked with SGS labs Pakistan as In-charge Quality Control, currently working with Tti testing labs as Manager marketing.

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