

FULL LENGTH ARTICLE

Studies on the Potential of *Adiantum philippense* L. as a Biosorbent for Nickel Removal

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ABSTRACT

Increase in population and industrial development has resulted in threat to various water sources. Heavy metal pollution of water is presently of utmost concern. Release of untreated/ partially treated industrial effluents, pesticide run off from agricultural lands, mining activities are few of the many sources of heavy metal pollution of water bodies. Conventionally used techniques like chemical precipitation, membrane filtration etc. have few limitations that have prevented its application in effluent treatment. Currently, several studies are being conducted to find an effective replacement for heavy metal removal from polluted sources. Biosorption is one such method that employs inactivated biomass for the passive removal of heavy metals. Bacteria, yeast, fungi, algae, agricultural by-products and plant wastes have been used as biosorbents. Abundant and easy availability, low cost and minimal processing make a biomass a potential biosorbent. Pteridophytes, in general, exhibit rapid reproduction through spores, resilience to urbanization and heavy metal tolerance. Thus, the present study employed a locally and abundantly available fern, *Adiantum philippense* L. to learn its heavy metal sorption efficiency.

Keywords: Biosorption. *Adiantum philippense* L. Nickel ion. Isotherm. Adsorption kinetics. ATR-FTIR. Scanning electron microscopy.

INTRODUCTION

Water is one of the prime elements responsible for life on earth. Unfortunately, most of the water bodies are under threat due to the growing population and industrial development. Heavy metal pollution of water is becoming a common global environmental problem. Heavy metals are non-degradable and chronic even at low concentration leading to ecological and health problems in both plants and animals. Industrial effluents are the major source of heavy metal pollution. Industries that manage electroplating, mining, fertilizer or pesticide production, metal surface finishing are few of the many sources. Thus, the removal of heavy metal ions from the very source of pollution is a prerequisite. Nickel usually occurs in the form of sulphides, oxides and silicate minerals. The main route of exposure to nickel possibly takes place via inhalation, ingestion or dermal contact. Nickel as an environmental pollutant is of utmost concern as it increases the risk of lung and nasal cancer, chronic bronchitis and skin allergy. Release of nickel into the environment arises due to nickel mining and by industries such as paint formulation, electroplating, porcelain enameling and copper sulphate manufacturing [1]. The concentration of released nickel ion depends on the type of industrial effluent. The maximum permissible limit of nickel in industrial effluent discharge in accordance with Indian standards is 2- 3 mg/L. To meet the regulatory standards, removal of toxic nickel from the effluents is very essential, before they are released into water sources. Physicochemical treatment processes commonly used for removing metal ions from effluents containing heavy metals include chemical precipitation, lime coagulation, ion exchange, reverse osmosis, membrane processes etc. Although they are efficient, application of such process is restricted because of technical or economic constraints. Recently, biosorption has been proposed to be an attractive alternative to conventional processes. It is advantageous compared to conventional techniques [2] for the following reasons: low cost, high efficiency, minimization of chemical and or biological sludge, no additional nutrient requirement, regeneration of biosorbent and possibility of metal recovery. In recent years, the potential of numerous biomasses in heavy metal removal are being studied upon. Among them, pteridophytes or ferns have received good attention in heavy metal phytoremediation in polluted land. Ferns have very few applications apart from phytoremediation. They are by and large treated as invasive weeds and do not play a role in food chain. Their wide habitats, rapid reproduction, resistance to

accumulation of high concentrations of heavy metals, resiliency towards urbanization are few of the many characteristics that should be exploited. Thus, the use of ferns in heavy metal remediation can be justified either in its live or inactive form over other biomass.

The research work presented herein is a part of the investigation conducted to evaluate the potential of heavy metals removal of the fern *Adiantum philippense* L. pinnae. To the best of our knowledge, no study has been carried out on removal of nickel using dried pinnae of *Adiantum philippense* L., a very common terrestrial fern that grows abundantly in moist shady places. Various parameters like contact time, initial pH of the solution, biosorbent dose and initial metal concentration were investigated using batch studies.

MATERIALS AND METHODS

A. Reagents

All the chemicals used in the present study were of analytical grade. NiSO₄·6H₂O (Nice Chemicals Pvt. Ltd., India) was used to prepare the Nickel metal solution, 0.1 N Sulphuric acid (Merck, India) and 0.1 N NaOH (Merck, India) were used to adjust the pH.

B. Preparation of biosorbent

The pteridophyte, *Adiantum philippense* L. was collected from a specific area in the campus of National Institute of Technology Karnataka, Surathkal, India for the entire study. The pinnae were separated from the fronds of the fern, washed thoroughly and dried. Dried pinnae were ground and sieved to a range of 212 to 180 µm (average of 200 µm). The powder was then stored in an air tight bottle to protect it from moisture until further use.

C. Preparation and analysis of Ni (II) solution

100 mg/L solution of Ni²⁺ ions was prepared in a 500 mL standard flask by dissolving 0.2239 g of nickel salt (NiSO₄·6H₂O) in distilled water. The Ni (II) concentration, before and after the experiments were determined by using GBC 932 plus Atomic Absorption spectrometer.

D. Batch biosorption experiments

The factors that affect the biosorption of metals on the fern biomass in its powdered form in removal of Ni (II) were examined in the batch system. The batch study was performed with desired biosorbent dose (2g/L) in 250 mL Erlenmeyer flasks with a working volume of 100 mL in an incubator orbital shaker at 150 rpm, 32±2°C for 120 min. Different parameters of pH (2, 3, 4, 5, 6, 7 and 8), contact time (2, 5, 10, 15, 20, 25, 30, 40, 50, 60, 90, 120, 150 and 180 min), biosorbent dosage (0.5, 1- 10 g/L) and initial metal concentration (25, 50, 150 and 200 mg/L). The solutions were separated from the biomass by filtering it through Whatman filter paper. The filtrate was refrigerated until the analysis was done. All the experiments were conducted in triplicates and average values were used in the analysis of the results. The amount of Ni (II) uptake, q_e (mg metal adsorbed /g adsorbent) was calculated by using the Eq. (1):

$$q_e = \frac{(C_0 - C_e) \times V}{W} \quad (1)$$

Where, the initial concentration C_0 (mg/L), metal concentrations at various time intervals, C_e (mg/L), V is the volume of solution in 'L' and W is the mass of sorbent in 'g'.

Percentage removal of metal ions (% Biosorption) or removal efficiency at any time was calculated using Eq. (2):

$$\% \text{ Biosorption} = \frac{(C_0 - C_e) \times 100}{C_0} \quad (2)$$

E. Adsorption isotherms and kinetic studies

Adsorption isotherm describes a functional relationship between q_e , the amount of solute adsorbed per unit weight of adsorbent and C_e , the residual equilibrium concentration. Sorption isotherms were experimentally determined by varying the initial metal ion concentration. In the present study, Langmuir and Freundlich models were used to describe the equilibrium data.

Adsorption kinetics is expressed as the solute removal rate that controls the residence time of the sorbate in the solid-solution interface. In this study, pseudo first order and pseudo second order rate equations were tested with the experimental data (concentration-time profile) for kinetic modelling of Ni (II) biosorption. The applicability of both kinetic models was tested using linearized equations.

F. Characterisation of *Adiantum philippense* L.

1) Scanning Electron Microscopy: The surface characterisation of *Adiantum philippense* L. before and after metal biosorption was carried out using JEOL JSM 6460LV Scanning electron microscope to determine if any morphological changes occur during biosorption.

2) Attenuated total Reflectance Fourier Transform Infrared Spectroscopy (ATR- FTIR): ATR- FTIR studies were carried out using BRUCKER ECO- ATR to identify the functional groups present in the

biomass that might be responsible for the adsorption process. The ATR- FTIR spectrum of dry untreated, treated biosorbent (control) and metal loaded biosorbent were determined.

RESULTS AND DISCUSSION

A. Effect of pH on Ni (II) sorption

Solution pH is a critical parameter that governs the biosorption process. Preliminary pH study was conducted to find out the pH at which the metal removal by *Adiantum philippense* L. biomass is optimum. The percentage biosorption of Ni (II) ions increased from 37.71% to 58.44%, when the pH was increased from 2.0 to 8.0. Therefore, Fig. 1 clearly shows that sorption of Ni (II) was influenced by the initial pH of the solution. Ionic interaction is the main mechanism contributing to biosorption of metals on the biosorbent. At lower pH, hydrogen ions compete with metal ions resulting in protonation of active sites. Whereas, at higher pH, more negatively charged surfaces is available resulting in high metal removal [3]. Thus, at a lower pH, the sorption of cationic nickel should be less because of higher dissociation of Ni (II) and positive surface charge of *Adiantum philippense* L. However, at pH 9.0 Ni (II) ion precipitations occurred due to low solubility and by the formation of nickel hydroxide [2]. Although the percentage biosorption was slightly higher at pH 8.0 compared to pH 7.0, it is always preferred to operate any system at neutral condition. Therefore, the optimum pH was chosen as 7.0 for this study.

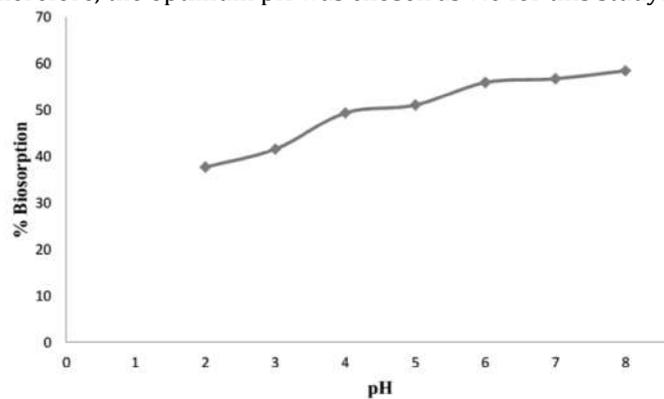


Fig. 1 Effect of initial solution pH on uptake of Ni (II) (Time= 24 hrs, biosorbent dose= 2 g/L, initial concentration= 100 mg/L, volume of solution= 100 mL, temperature= 32±2°C)

B. Determination of equilibrium time

Equilibrium time is the time taken to achieve equilibrium after which no further uptake of metal is observed [1]. Contact time influences the metal ion removal efficiency of the biosorbent as shown in Fig. 2. The Ni (II) sorption rate is high in the first 10 min at pH 7.0. However, the rate slows down. It is clear that biosorption of Ni (II) on *Adiantum philippense* L. consisted of two phases: a rapid primary phase and a slower second phase of biosorption. Initial higher rate of metal uptake may be due to (a) the high concentration of Ni (II) which exists initially (b) large number of active sites available on the biosorbent. Secondary slow phase may be due to (i) reduction in number of active sites being available and/or (ii) due to reduction in concentration gradient [4]. As the adsorption proceeds, the adsorbed solute tends to desorb back into the solution up to a certain extent. In due course, the rate of adsorption and desorption will attain an equilibrium state. Adsorption equilibrium was attained at 120 min, as shown in Fig. 2. Hence, all further experiments were conducted for a period of 120 min.

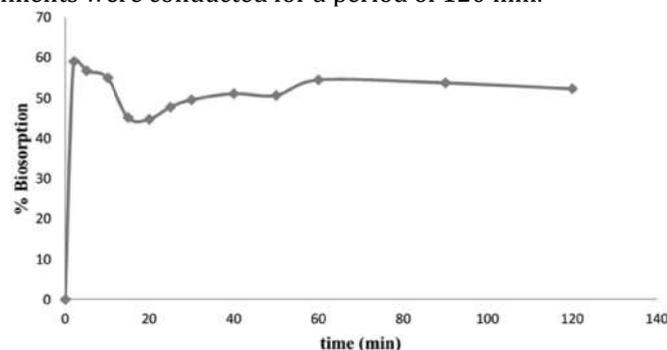


Fig. 2 Equilibrium time for the uptake of Ni (II) (pH= 7.0, biosorbent dose= 2 g/L, initial concentration= 100 mg/L, volume of solution= 100 mL, temperature= 32±2°C)

C. Effect of biosorbent dosage

Effect of biosorbent dosage was evaluated with experiments conducted at pH 7.0, initial metal concentration of 100 mg/L, temperature $32\pm 2^\circ\text{C}$ and contact time of 120 min. It is evident from Fig. 3, the percentage biosorption of Ni (II) increases with increase in adsorbent dosage. As the dosage of biomass was increased from 0.5 g/L to 10 g/L, the percentage biosorption increased from 33.56% to 93.46% for Ni (II). As the dosage was increased from 6 g/L to 10 g/L the removal efficiency has increased marginally by 6.25 %. Very small increase in percentage biosorption with increase in biosorbent dosage at higher dosage levels may be due to partial aggregation of biomass at higher concentration, which results in a decrease in effective surface area for biosorption [5]. Percentage biosorption exhibited was 93.14 % by 9 g/L and 93.46% by 10 g/L of biosorbent were very close. Hence, 9 g/L biomass is sufficient for optimum metal removal. Overall, increase in biosorption percentage with increase in biosorbent dosage could be due to an increase in the biosorbent surface area and availability of more biosorption sites ([6], [7]).

D. Effect of initial metal ion concentration

The initial Ni (II) ion concentration was increased from 25 to 200 mg/L, keeping all parameters constant as mentioned earlier. After the contact period of 120 min the percentage sorption of Ni (II) and the initial metal ion concentration were plotted, respectively, as y and x- axis (Fig. 4.). The removal efficiency decreased from 78.724% to 50.69%. At lower concentrations, metals adsorb on specific binding sites on the biosorbent and thus percentage removal of metal ion was high. The decrease in percentage biosorption at higher metal concentration was due to the saturation of the sorption sites available on the biomass [8]. This is due to the increase in the number of ions, competing for available binding sites in the biomass [9].

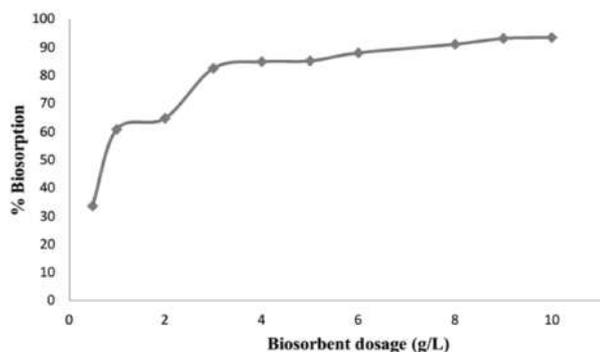


Fig. 3 Effect of biosorbent dosage for the uptake of Ni (II) (pH= 7.0, initial concentration= 100 mg/L, volume of solution= 100 mL, temperature= $32\pm 2^\circ\text{C}$)

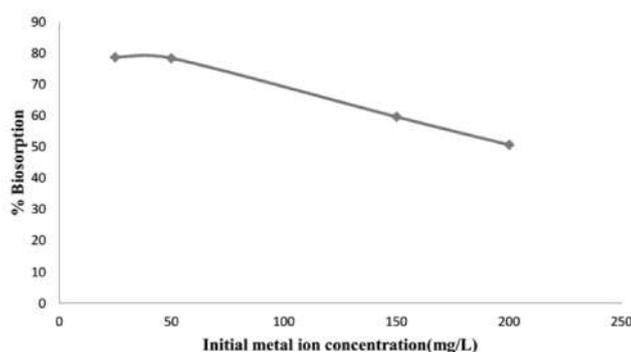


Fig. 4 Effect of initial metal ion concentration for the uptake of Ni (II) (pH= 7.0, biosorbent dosage= 2 g/L, volume of solution= 100 mL, temperature= $32\pm 2^\circ\text{C}$)

E. Adsorption isotherm

Sorption equilibrium is usually described by an isotherm equation whose parameters express the surface properties and affinity of the sorbent, at a fixed temperature and pH [10]. Sorption isotherms were analysed experimentally by varying the initial metal ion concentration. In the present study, Langmuir and Freundlich models were used to describe the equilibrium data.

1) Langmuir isotherm model: The model describes quantitatively the formation of an adsorbate monolayer on the outer surface of the adsorbent, and after that no further adsorption takes place [11]. Linear form of Langmuir equation (Eq. 3) was used to determine the Langmuir adsorption parameters

$$\frac{1}{q_e} = \frac{1}{Q_0} + \frac{1}{Q_0 K_L C_e} \quad (3)$$

Where, C_e = the equilibrium concentration of adsorbate (mg/L), q_e = the amount of metal adsorbed per gram of the adsorbent at equilibrium (mg/g), Q_o = maximum monolayer coverage capacity (mg/g) and K_L = Langmuir isotherm constant (L/mg). The essential characteristics of the Langmuir isotherm can be expressed using Hall separation factor R_L (dimensionless) which is given by the Eq. (4).

$$R_L = \frac{1}{1+C_oK_L} \quad (4)$$

Where, C_o is the highest initial metal ion concentration (mg/L) and K_L is the Langmuir constant (L/mg). R_L values are used for the interpretation of the sorption type as given below: $R_L > 1$: unfavourable; $R_L < 0$: unfavourable; $R_L = 1$: favourable (linear); $0 < R_L < 1$: favourable; $R_L = 0$: irreversible.

The Langmuir isotherm parameters, Q_o and K_L were determined from the intercept and slope of the isotherm plot, $1/q_e$ against $1/C_e$ (Fig. 5). The Langmuir constants, Q_o and b were calculated and their values are shown in Table 1. The R_L values obtained from this study was indicated that the adsorption of Ni (II) is favourable.

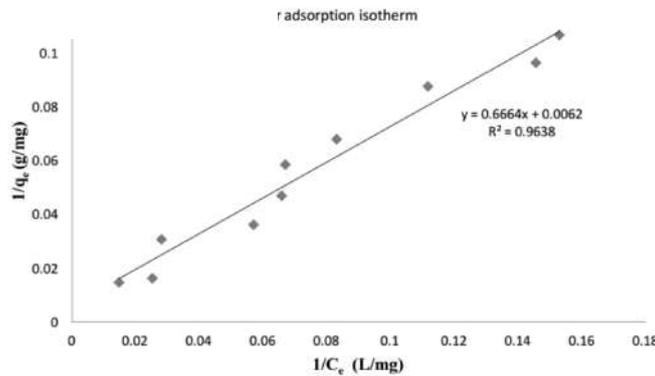


Fig. 5 Langmuir isotherm plot for biosorption of Ni (II) by *Adiantum philippense* L. biomass (pH= 7.0, biosorbent dosage= 2 g/L, volume of solution= 100 mL, temperature= 32±2°C)

2) Freundlich isotherm model: The model describes the adsorption characteristics for the heterogeneous surface. The adsorption is found to be infinite with the increase in the adsorbate concentration. The adsorption data was also analysed using Freundlich model using the linearized Eq. (5).

$$\log Q_e = \log k_f + \frac{1}{n} \log C_e \quad (5)$$

Where, q_e is the amount of metal adsorbed (mg/g), C_e is the equilibrium concentration of the metal (mg/L). The Freundlich constants, k_f and n were determined from the intercept and slope of the isotherm plot, $\log C_e$ against $\log q_e$ (Fig. 6). The Freundlich constants, k_f and n were calculated and their values are shown in Table 1. 'n' value between one and ten indicates a favourable sorption process and smaller $1/n$ value indicates greater heterogeneity (Dada et al., 2012). From the data in Table 1, value of $1/n = 0.883$ shows lower heterogeneity, while $n=1.1325$ indicating that the sorption of Ni (II) onto *Adiantum philippense* L. is favourable with the R^2 value, 0.9471 and the capacity of its surface as a possible Ni (II) biosorbent.

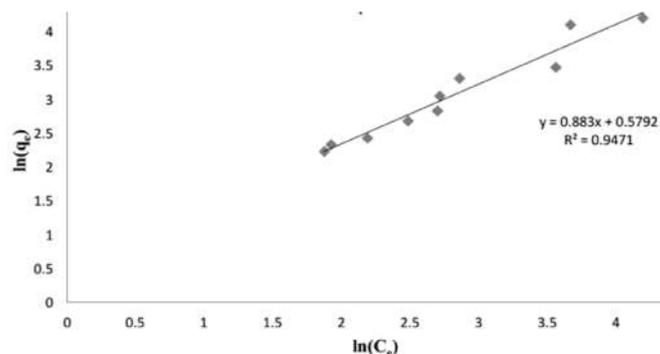


Fig. 6 Freundlich isotherm plot for biosorption of Ni (II) by *Adiantum philippense* L. biomass (pH= 7.0, biosorbent dosage= 2 g/L, volume of solution= 100 mL, temperature= 32±2°C)

Table 1. Freundlich and Langmuir Adsorption Constants Associated to Adsorption Isotherms of Ni (II) on Dried *Adiantum philippense* L. Biomass.

Langmuir isotherm			Freundlich isotherm		
Q _o (mg/g)	K _L (L/mg)	R ²	k _f (mg/g)	n	R ²
161.29	0.0093	0.9638	1.7846	1.1325	0.9471

Table 2 compares the maximum metal uptake efficiency of different plant and fern biomasses used for Ni²⁺ sorption. Dried *Adiantum philippense* L. biomass shows relatively higher metal uptake (mg/g) capacity in comparison to other sorbents. Thus, the fern can be used as a potent biosorbents for the removal of Ni (II).

Table 2: Comparison of Different Plant Biosorbents in Ni (II) Removal.

Biomass type	Metal uptake (mg/g)	Reference
Peepal leaves (<i>Ficus religiosa</i>)	6.35±0.54	[1]
Watermelon rind	35.3	[12]
<i>Azolla filiculoides</i> (dry)	45.193	[13]
Pomegranate peel (<i>Punica granatum</i>)	52	[14]
<i>Azolla filiculoides</i> (Activated)	71	[15]
<i>Adiantum philippense</i>	161	Present Study

F. Adsorption kinetics

Kinetics aspect helps in understanding the performance and mechanism of adsorption. From the kinetic analysis, the solute uptake rate, determining the residence time required for the completion of adsorption reaction may be found [16]. In the current study, pseudo first- order and pseudo second- order rate equations were tested with the experimental data for kinetic modelling of Ni (II) biosorption. The linearized expression of pseudo- first order is given by Eq. (6):

$$\ln[q_e - q_t] = \ln q_e - tK_{pl} \quad (6)$$

Where, q_e and q_t (mg/g) are the adsorption capacities at equilibrium and time t (min), respectively. K_{pl} (min^{-1}) is the pseudo-first-order rate constant for the kinetic model. A straight line of $\ln[q_e - q_t]$ versus t suggests the applicability of this kinetic model. q_e and K_{pl} can be determined from the intercept and slope of the plot respectively [17].

The linearized expression of pseudo- second order [18] is given by Eq. (7):

$$\frac{t}{q_t} = \frac{1}{kq_e^2} + \frac{t}{q_e} \quad (7)$$

Where, t (min) shows time, q_t (mg/g) shows uptake capacity at t and k ($\text{g mg}^{-1} \text{min}^{-1}$) shows the equilibrium rate constant of pseudo-second order adsorption, q_e the amount adsorbed (mg/g) at equilibrium. kq_e^2 ($\text{mg}/(\text{g}\cdot\text{min})$), initial adsorption rate. The values of rate parameters k and q_e can be obtained from the intercept and slope of the plot t/q_t against t (Fig. 7).

Table 3, provides pseudo first-order rate constants K_{pl} , pseudo second-order rate constants k , q_e , the (theoretical) and experimental equilibrium sorption capacity $q_{e, \text{exp}}$ (experimental) at various time intervals and the regression coefficient, R^2 to determine the validity of the kinetic models. Pseudo second-order model has a better regression coefficient of 0.9985. From Table 3 and Fig. 7, it is evident that the q_e , the value estimated from pseudo second- order kinetic model is very close to the $q_{e, \text{exp}}$ value. This indicates that pseudo second-order model is better obeyed than pseudo first-order model. The analysis of kinetic data by other studies also showed that the pseudo second-order rate equation was a reasonably good fit for Ni (II) adsorption ([19], [20]).

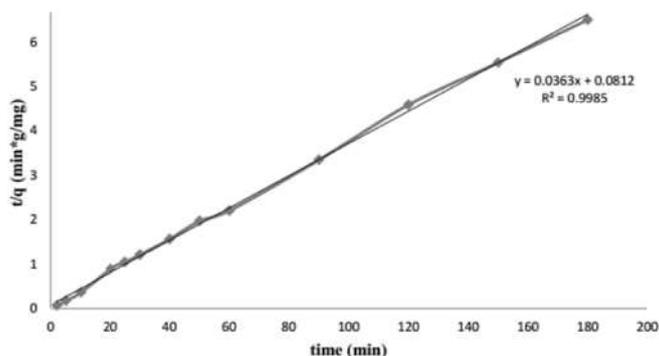


Fig. 7 Linearized pseudo-second order biosorption kinetics of Nickel (II)

Table 3: Comparison of Pseudo First- Order and Pseudo Second- Order Kinetic Models for Ni (II) Biosorption on Dried *Adiantum philippense* L. Biomass.

Experimental q_e , $q_{e, \text{exp}}$ (mg/g)	Pseudo first- order kinetics			Pseudo second- order kinetics		
	K_{pl} (min^{-1})	$q_{e, \text{the}}$	R^2	k ($\text{g mg}^{-1} \text{min}^{-1}$)	$q_{e, \text{the}}$	R^2
26.155	0.002	6.586	0.053	0.0162	27.55	0.9985

G. Characterization of biomass

1) Scanning electron microscopy: Scanning electron microscopy was used to acquire the surface morphology before and after the biomass was enriched with Ni (II) ions. Electron micrographs of *Adiantum philippense* L. biomass, both before and after exposure to nickel in aqueous solution, are presented in Fig. 8. Scanning electron micrograph indicated slight deformation of the surface of the metal-loaded *Adiantum philippense* L. biomass (Fig. 8B). Similar observations were reported by [21]. Layers observed in biomass (Fig. 8A) were found to be sticking to one another post metal binding (Fig. 8B). After the exposure to metal ions, the biomass was no longer found to be smooth [22].

2) Attenuated total Reflectance Fourier Transform Infrared Spectroscopy (ATR- FTIR) analysis: Numerous chemical groups have been proposed to be responsible for the biosorption of heavy metals. Their relative importance in metal sorption depends on biosorbent sites, accessibility, chemical state and affinity between active sites and metal. FTIR is an important tool to identify the functional group.

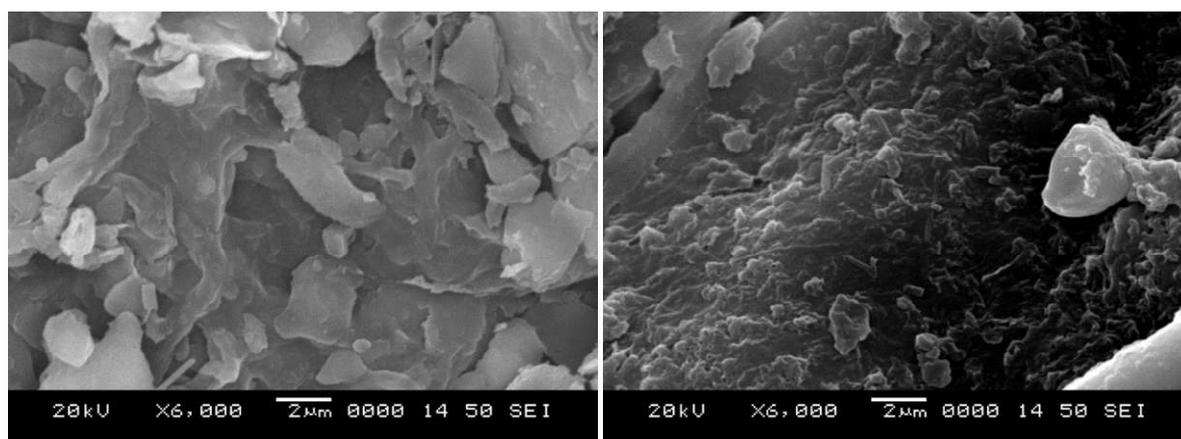


Fig. 8 SEM images of biomass before (A) and after the biomass are enriched with Ni (II) (B) of magnification 6000X.

In order to identify the functional groups responsible for biosorption, ATR- FTIR analysis was carried out. ATR- FTIR spectrum of dry native biosorbent (Fig. 9A) and metal loaded biosorbent was compared (Fig. 9B). The vibrancy signals before and after the biosorption of metal was different. In the native biosorbent the two intense peaks at 2922.64 - 2853.32 cm^{-1} were due to the asymmetric and symmetric stretching (C-H) of aliphatic compounds. A characteristic peak at 1739.98 cm^{-1} was assigned to (C=O) bond vibration of carbonyl group. The peak of medium intensity obtained at 1627.01 is due to the presence of (C=C) alkene

bonds. The peak present at 1229.43 indicates (C=C) aromatic bending and strong intense peak at 1020.25 cm^{-1} is characteristic to (C-C) stretching.

Upon binding of Ni ions both, change in peak intensity and shifting of peaks was observed. There was a decrease in intensity of peaks at 2922.64- 2853.32 cm^{-1} and increase in peak intensity at 2355.63 cm^{-1} for (N-H). Shift in peaks from 2362.48 to 2355.63 and 1627.01 to 1618.60 cm^{-1} corresponding to amine and alkene groups. Thus, indicating that carbonyl, amines and alkene Groups are involved in the sorption of Ni (II) ions. These results are in agreement with [23].

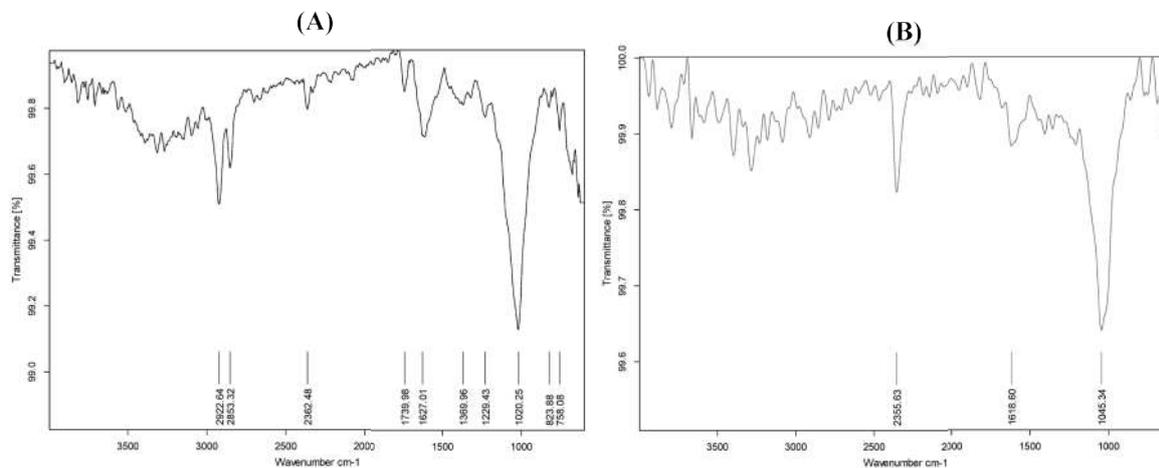


Fig. 9 ATR- FTIR spectra of the biomass before (A) and after (B) the biomass are enriched with Ni (II)

CONCLUSION

Adiantum philippense L. pinnae powder in its native form was found to be a relatively good adsorbent for Ni (II) from aqueous solution, compared to few other reported biosorbents like native and treated *Azolla filiculoides*, leaves of *Ficus religiosa*, watermelon rind and pomegranate peel. Neutral pH and higher fern dosage resulted in a higher metal loading onto the sorbent at an equilibrium time of 120 minutes. The maximum uptake of Ni (II) on *Adiantum philippense* L. was found to be 161.29 mg/g, obtained from Langmuir isotherm model. Furthermore, *Adiantum philippense* L. with no commercial value promotes its application in heavy metal adsorption. However, further research is needed to establish if chemical treatments can further improve its characteristics as an efficient biosorbent.

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