Biosorption of Ni(II) Ions from Electroplating Industrial Effluent using *Tinospora cardifolia* Biomass

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Abstract

A biomass derived from the plant *Tinospora cardifolia* has been found to be very efficient in Nickel(II) adsorption. An attempt was made to use this biomass for Nickel(II) removal under different conditions. The parameters optimized were contact time (5–1440 min), pH (1–10), concentration of adsorbent (1–7 gm/L), concentration of adsorbate (10–500 mg/L), etc. It was observed that the pH had a strong effect on biosorption capacity. The optimum pH obtained for nickel adsorption was 4.0. The influence of common ions such as Cu(II), Zn(II), Cd(II), Fe(III), Mn(II), Pb(II) and Cr(VI) and NO_3^-, SO_4^- PO_4^- and Cl^- at concentrations varying from 10 to 500 mg/l was investigated. To establish the most appropriate correlation for the equilibrium curves, isotherm studies were performed for Ni(II) ion using Freundlich and Langmuir adsorption isotherms. The pattern of adsorption fitted well with both models. The biomass of *T. cardifolia* was found to be effective for the removal of Ni(II) with 92.3% sorption efficiency at a concentration of 50 mg/l of Ni(II) solution, and thus uptake capacity(q_m) according to Langmuir isotherm is 4.48 mg/g of biomass and in column mode 2.49 mg/g of biomass.

Keywords: Nickel; Adsorption; Biomass; *Tinospora cardifolia*

1. Introduction

Many toxic heavy metals are being discharged into the environment as industrial wastes, causing serious soil and water pollution. Various physicochemical and biological methods for removal of toxic metal ions have been studied (Kubilay et al., 2007). The conventional methods for the removal of these heavy metals from wastewaters include chemical precipitation, membrane separation, ion exchange, adsorption and biosorption (Babel and Krniavan, 2003; Rusten et al., 1997). But, these processes are not economically feasible even for small-scale industries, due to huge capital investment. Adsorption of pollutants on solid adsorbents is an effective method used for heavy metals removal from aqueous solutions, especially when the adsorbent used is not very expensive (Babel and Krniavan, 2003; Bailey et al., 1999). Natural materials that are available in large quantities or certain waste products from industrial or agricultural operations may have potential to be removing the heavy metal as inexpensive sorbents. The moss peat is one example of such low-cost sorbents which can be utilized for the heavy metals removal from aqueous solutions (Brown et al., 2000; Coupal and Spiff, 1999).

Nickel is one of the toxic heavy metals that are common pollutants of the environment. In humans, presence of nickel can cause serious problems, such as dermatitis, allergic sensitization, and damage to lungs and the nervous system; it is also a known carcinogen (Axtell et al., 2003). The maximum permissible concentration of nickel in drinking water is 0.02 mg/L according to world health organization (WHO 2003) (Khan et al. 2011).

This work was carried out with the objective of evaluating the nickel biosorption process by means of the biomass *Tinospora cardifolia.* This indigenous plant; extensively used in Ayurvedic system of medicine, is commonly known as (Gulancha) which belongs to the family *Menispermaceae* (Bhawya et al. 2010). It is an herbaceous vine which is indigenous to the tropical areas of India, Myanmar and Sri Lanka. *T. cordifolia* has various common names such as Guduchi, Amrita, Giloe, Gado, Galo, Amrutha, Balli etc. This plant is commonly known as Giloya in hindi which is a mythological term that refers to the heavenly elixir that saved celestial beings from old age and kept them eternally young (V.Kumar et al. 2013).

The stem of the plant was studied for its antioxidant and phytochemical constituents. A number of compounds viz. alkaloids, glycosides, diterpenoid lactones, sesquiterpenoids, steroids, phenolics, aliphatic compounds, polysaccharides and flavonoids have been confirmed in aqueous extract of *T. cordifolia* by phytochemical analysis considered to be responsible for the biological activities like anti-inflammatory, anti-arthritic, anti-osteoporotic activity, anti-allergic, anti-hyperglycemic, anti-pyretic, anti-oxidant, diuretic and cardioprotective activity (Upadhyay et al. 2010).

Pandey et al. (2012) first reported the use of this biomaterial for removal of fluoride from waste water. In furtherance of the work now we report the removal of Ni(II) from industrial water and waste water. This biomaterial was subjected to various determinations for nickel sorption, optimum time for metal-biomass equilibrium, pH, biomass size, concentration, and the biosorption capacity in batch and column mode.

2. Materials and method

2.1. Collection and Preparation of the biosorbent

Biomass was collected from the different areas of Chhattisgarh in India. The collected biomass was dried under sun and cleaned manually. It was washed with distilled water to make it free from dust and impurities. The residual material so obtained was dried at 60^0C in hot air oven for few days, and then the material was grinded and sieved through 1.18µM mesh, to obtain uniform sized material for experiments. After that the biomass was cleaned with HCl (0.1M) for 30 min. Biomass was then filtered (Whatman Filter No.42) and washed with distilled water till the washing was neutral. The biomass was then dried in an air-oven at 60^0C till drying. The acid treatment also helped to remove any previously adsorbed metals. It was presumed that such a treatment will enhance the uptake capacity of the biomass. The biomass was stored in airtight plastic container till further use.

2.2 Chemicals

All chemicals used in this work were of analytical grade and obtained from Merck (India/Germany). All solutions were prepared with double distilled water. All utensils and bottles utilized in the experiments were washed with 5% HCI solution and rinsed with distilled water. Nickel ions were prepared by dissolving its corresponding nitrate salt in double distilled water. Nitrate salt was selected, because of the low chelating capacity of NO_3^{2-} with respect to metal ions. The pH of the solution was adjusted with 0.1M HCl and NaOH. All other solutions were prepared from the stock solution.

2.3 Instrumentation

An Atomic Absorption Spectrophotometer AA240FS (Fast Sequential Atomic Adsorption Spectrophotometer) was used for Ni(II) measurement and the Fourier Transform Infrared Spectroscopy (FT-IR) was used for FTIR analysis, A high precision electrical balance TB-214 was used for weighing. A vacuum filtration pump was used for filtration; a digital pH meter was used for pH measurement and a peristaltic pump IPC (High Precision Multichannel Dispenser was also used for providing constant flow in fixed bed column.

3. Batch mode adsorption study

Aqueous solution of Nickel (1000 mg/l) was prepared by dissolving Nickel nitrate (AR grade) in double distilled water. The aqueous solution was diluted with double distilled water to obtain the working solutions of desired concentration. For each adsorption experiment the desired amount of Nickel solution was taken and the pH was adjusted to 4.0 by adding drops of 0.01M HCl or NaOH solution and the mixture were stirred occasionally for 30 minutes at room temperature. After that the mixture was filtered to separate the exhausted adsorbent from solution. The residual Ni concentration in solution was determined by AAS. All experiments were replicated thrice and results were averaged. The removal percentage and adsorbent capacity of adsorbent were calculated as reported by this formula –

The definition of removal efficiency is as follows: Removal

$$Removal\ Efficiency(\%) = [Ci - Ce/Ci]100 \quad (1)$$

Where C_i and C_e are the metal concentration in the sample solution before and after treatment.

The adsorption capacity of an adsorbent which is obtained from the mass balance on the sorbate in a system with solution volume V is often used to acquire the experimental adsorption isotherms. Under the experimental conditions, the adsorption capacities of the adsorbent for each concentration of Ni(II) calculated using by this formula –

$$qe(mg/g) = \frac{Ci - Ce}{M} \times V \quad (2)$$

where Ci and Ce are the initial and final concentration of both ions in the solution respectively. V is the volume of solution (L) and M is the mass of adsorbent (in gm) used.

4. Column study

It is known that the packed column enhances the metal binding capacity to a greater extent than the batch mode. Hence to ascertain the same in this experiment packed bed experiment was conducted at room temperature (27 ± 2^0C). In a glass column packed with 70 gm. of *T. cardifolia* dry biomass, 50 mg/L of Ni solution, where the pH of the solution are 4.0 adjusted by 0.1M NaOH and HCl solution. The solution was pumped through the column at a flow rate of 13 ml/min adjusted by using a Peristaltic Pump IPC (High Precision Multichannel Dispenser) collector. 200 mL sample was collected from the exit of the column at regular time intervals until the biosrption saturation capacity of the packed column was reached at the stage when no adsorption is occurred. The effluent sample was analyzed by atomic adsorption spectroscopy (AA240FS).

5. Result and Discussion

Ni(II) uptake studies were conducted at (27 ± 2^0C) temperature to evaluate the effect of pH, contact time, adsorbent dosage and the initial metal ion concentration on the biosorption.

5.1 Effect of pH

The pH of the aqueous solution is an important controlling parameter in the heavy metals adsorption processes (Barrera *et al*., 2006). This parameter is directly related with competition of hydrogen ions with metal ions to active sites on the biosorbent surface (Lodeiro *et al*., 2006). The effect of pH on Ni(II) removal was conducted with biosorbent dosage 5 gm, equilibration time 30 min, and initial nickel concentration are 50 mg/L. They were given in Figure.1. The metal ions are in competition with the protons in the solution at low pH values for the biosorption on active sites biomass surface. At very low pH metal uptake was less but metal uptake increased with increasing pH from 3-4 this is due to more legends with negative charge being exposed with the subsequent increase in attraction sites to positively charged metal ions. Beyond this point there is not much further increase in efficiency of the biosorption, the process increase drastically due to the formation of metal hydroxide with their respective metal ions. This is mostly due to the metal precipitation as hydroxide ion which depend on the pH and ion concentration but not due to the biosorption. From which it was evident that Ni(II) removal was maximum in the pH 4.0.

5.2 Effect of Metal Ion Concentration

Figure.2 shows the effect of metal ion concentration on the adsorption experiment. It was observed that percentage adsorption of Ni(II) decreased from 92.3% to 72.2% with increased in metal concentration from 50 to 500 mg/L. At higher concentration, most of the Ni(II) is left unabsorbed due to saturation of adsorption sites. As the ratio of sportive surface and ion concentration percentage removal was decreases with increasing metal ion concentration. At low concentration of metal ions, more binding sites are available, but as the concentration increases, the number of ions competing for available binding sites in the biomass increases.

5.3 Effect of Adsorbents Dose

Effect of biosorbents dosage on percentage removal of Ni(II) was investigated by varying adsorbents dosage in the range of 0.5 gm/L to 20.0 gm/L. It was observed that the percentage removal of Ni(II) increases with the increase in the adsorbent dosage. The maximum percentage removal of Ni(II) was at 5 gm/L of biosorbent dose at constant initial metal ion concentration of 50 mg/L. The phenomenon of increase in percentage removal of Ni(II) with increase in adsorbent dose may be explained as with increase in adsorbent dose, more and more surface becomes available for metal ion to adsorb and this increase the rate of adsorption.

5.4 Effect of time

The data obtained from the biosorption of Ni(II) on the *T. cadifolia*. showed that a contact time of 30 min was sufficient to achieve equilibrium and the adsorption did not change significantly with further increase in contact time. 70% of Ni(II) was removed before 30 min, the removal of Ni(II) increased with and reached a maximum at 30 min. after that, which further increase in time did not bring about any improvement.

6. Adsorption Dynamics

Kinetics of adsorption, describing the solute uptake rate, which in turn governs the contact time of adsorption process, is one of the important characteristics defining the efficiency of adsorption. Kinetics of Ni(II) removal was there for carried out to understand the behavior of biomass. In this paper studied both Pseudo-First Order and Pseudo-Second Order rate reaction but this paper obey only Pseudo-Second Order rate reaction.

Pseudo-First Order Model

The rate constant of adsorption K_{ad}, was determine using the, following first order rate expression given by (Langergren 1898).

$$\mathrm{Log}(q_e - q_t) = \log q_e - \left(\frac{k_{ad}}{2.303}\right) \times t \qquad (3)$$

Here q_t & q_e (both in mg/g) are the amount of nickel adsorbed at time t (min) and at equilibrium respectively. A straight line plot of $\log(q_e-q_t)$ against time(min)

Pseudo- Second Order Model

(Ho and McKay, 1999) presented the pseudo-second order kinetic as:

$$\frac{dqt}{dt} = k_2\,(q_e - q_t)^2 \qquad (4)$$

For the boundary conditions t = 0 to t = t and qt = 0 to qt = qt, the integrated form of Eq. (4) becomes:

$$\frac{t}{qt} = \frac{1}{k_2 q_e^{\,2}} + \left(\frac{1}{q_e}\right) t \qquad (5)$$

Where k_2 is the pseudo-second order rate constant (g $mg^{-1}min^{-1}$). The initial adsorption rate, h (mg $gm^{-1}min^{-1}$) at t→0 is defined as:

$$h = k_{2q_e^2} \qquad (6)$$

The h, q_e and k_2 can be obtained by linear plot of t/q_t versus t in Figure. 3 and Table.1 Shows the parameters.

7. Interfering ion study:

The study of co-ion, adjusting the pH value is 4.0 by using 0.1M NaOH and HCl solution in the 50 mg/L of Ni(II) solution duration of time is 30 min, and biosorbent dose are 5 gm/L. Experimental result of the co-ion effect on the adsorption of ions from an aqueous solution cations containing Cu(II), Zn(II), Cd(II), Fe(III), Mn(II), Pb(II) and Cr(VI) in equal concentration reveal that to presence of foreign ions diminishes the adsorption of Ni(II) ions. The removal efficiency of nickel are decreases at higher concentration in case of Fe(II), Cu(II) Mn(II) and Zn(II), while in the presence of Pb(II), Cd(II) and Cr(II) percentage removal of Ni(II) was higher at higher concentration. In the study of anion like NO_3^-, SO_4^-, PO_4^- and Cl_2^- percentage removal of Ni(II) are high at lower concentration but at higher concentration it was decreases.

8. Isotherm Study

Adsorption is usually described through isotherms, that is, functions which connect the amount of adsorbate on the adsorbent. Distribution of metal ions between the liquid phase and the solid phase can be described by several isotherm models such as Langmuir and Freundlich.

Langmuir and Freundlich model

Studies of adsorption equilibrium isotherm were conducted varying the Ni(II) concentration from 10 to 500 mg/l at pH 4.0, for 30 min, of shaking at room temperature. These data were evaluated by Langmuir and Freundlich adsorption isotherm equations to interpret the efficiency of metal sorption. Langmuir isotherm model assumes monolayer adsorption, and is presented by the Eq. (7) and (8).

Langmuir model: $$q_{eq} = Q_{max} \times K_L \times C_{eq}/(1 + K_L \times C_{eq}) \qquad (7)$$

Langmuir model in linear form: $$\frac{C_{eq}}{q_{eq}} = \frac{1}{(Q_{max} \times K_L)} + \frac{1}{Q_{max}} \times C_{eq} \qquad (8)$$

Where q_{eq} is the metal amount adsorbed per unit mass of adsorbent (mg/g), C_{eq} is the equilibrium concentration of metal in the solution (mg/L), Q_{max} is the maximum adsorption capacity (mg/g), and K_L is the constant related to the free energy of adsorption. A straight line is obtained by plotting C_{eq}/q_{eq} against C_{eq}. The slope and intercept are used to calculate the Q_{max} and K_L, respectively. The Freundlich model is presented by the Eq. (9) and (10).

Freundlich model: $$q_{eq} = k_f \times C_{eq}\, 1/n \qquad (9)$$

Freundlich model in linear form: $$\log q_{eq} = \log k_f + \frac{1}{n} \times \log C_{eq} \qquad (10)$$

Where k_f is a parameter of relative adsorption capacity of the adsorbent related to the temperature and n is a characteristic constant for the adsorption system. A plot of log q_{eq} against log C_{eq} gives a straight line and the slope and intercept correspond to 1/n and log k_f, (Patil et al. 2012) respectively.

The equilibrium adsorption isotherm was obtained by plotting the amount of Ni(II) sorbed (q_{eq}) against equilibrium concentration (C_{eq}) in the solution, and was presented in Figure. 4. The Figure shows that the amount of Ni(II) sorbet increases with an increase in equilibrium concentration and attains a value related to the maximum adsorption capacity of biomass. The linearized Langmuir and Freundlich isotherms were applicated for the experimental data and the parameter values were calculated and presented in Table 2. The Langmuir and Freundlich plots were put in Figure. 4 and Figure. 5. The value of the correlation coefficient of the Freundlich model is higher than of the Langmuir model. This indicates the adsorption phenomenon was best described by this Model over the concentration range studied. According to the Langmuir model, a monolayer of adsorbed Ni(II) was formed with a maximum adsorption capacity of Ni(II) is 4.48 mg/g.

9. Behavior of adsorption column

The fixed bed column experiment was conducted with 50 mg/L Ni(II) solution maintaining a constant flow rate of 13 ml/min. The breakthrough curve of S-shaped was obtained as shown in figure. 6 and Table. 3 show the values of all the parameters of column study. The breakthrough time and exhaust time corresponding to (C/C_o) were found to be 153.6 min and 345.7 min respectively. The corresponding volume of treated Ni(II) solution was 4.4 liter respectively. About 70 gm of biomass were used in the column.

10. Comparative study of different biosorbent

Adsorptive removals of heavy metals from aqueous effluent have received much attention in recent years. It's true that the different bioasorbent have the ability to remove the heavy metal toxicity from aqueous effluent but, it is observed that *Tinospara cardifolia* has the cost effective and having a various medicinal properties, when compared with other bioasorbents. The values of adsorption capacities of Ni(II) for different biomass in batch study are presented in Table. 5.

11. FTIR (Fourier Transform Infrared Spectroscopy) Study

The FTIR technique is an important tool to identify the characteristic functional groups on the adsorbent surface. The FTIR spectra of unloaded and Ni(II) loaded *T. Cardifolia* biomass in the range of 4000-500 cm^{-1} were taken to obtain information on the nature of possible biosorbent Ni(II) ion interaction are presented in Figure 9 & 10. The spectra display a number of adsorption peaks indicating the complex nature of the examined biomass. Inspection of the spectra reveals the presence of a broad peak at wave number 3349.7 cm^{-1} representing surface bonded O-H group which was shifted to 3749.9 cm^{-1} O-H group and 3429.4 cm^{-1} N-H group after adsorption. The adsorption peak at 3429.4 cm^{-1} represent the Amine –NH group which was not present in unloaded biomass. Whereas, the adsorption peak around 2363.4 cm^{-1} indicates the existence of Nitrile –CN group which was disappear after adsorption. The strong adsorption peak at 2925.7 cm^{-1} can be assigned to –CH stretching and those at 1734.4 cm^{-1} assigned to C=O stretching groups respectively. These peaks also were shifted to alkane –CH 2922.9 cm^{-1}, Alkyne –CH 2364.0 cm^{-1} and C=O 1734.9 cm^{-1}. The adsorption peak at 1514.6 cm^{-1} and 1426.5 cm^{-1} can be attributed to the presences of Nitro –NO and Aromatic group –C=C stretching respectively. After adsorption these peaks were shifted to 1515.6 cm^{-1} and 1428.2 cm^{-1}. Another peaks of Alkyl halide group observed at the biomass surface before adsorption appeared at 1373.9 cm^{-1} was shifted to 1374.9 cm^{-1}. The peaks observed at 772.5 cm^{-1} and 668.3 cm^{-1} was shifted to 771.2 cm^{-1} and 666.7 cm^{-1}. Figure. 7 FT-IR spectra of Native adsorbent and Figure. 8 FT-IR spectra of Nickel loaded adsorbent and Table. 4 showing the important peaks of native and nickel loaded adsorbent.

12. Application of Biomass for Removal of Nickel from Industrial Effluents

The removal of Ni(II) ions from electroplating wastewater was carried out by the batch method under the optimum conditions used, in this study 50 ml of electroplating waste water containing 13.58 mg/L Ni(II) was taken in a conical flask. The pH of the solution was adjusted at 4.0 and treated with 5 gm/L of biosorbent. The removal of Ni(II) ions was carried out as described in adsorption experiments Electroplating wastewater was collected from one of the electroplating industries in Vishakhapatnam (India). Removal of Ni(II) by *T. Cardifolia* biomass from electroplating wastewater was found to be 92%.

13. Conclusion

From this study it is concluded that *T.cardifolia* is effective in removing nickel ion from aqueous solutions. The results obtained show that pretreatment, initial pH, biosorbent amount, initial metal ion concentration and time affect the uptake capacity of the biosorbent. The maximum nickel ion adsorption achieved in the batch experiment is 4.48 mg/g and in the column mode study it is 2.49 mg/g. It was found that at the optimum pH of 4 and at the 50 mg/L concentration of metal ion this method could successfully remove more than 90% of Ni(II). The biosorption equilibrium data obeyed Langmuir and Freundlich model.

Based on FTIR analysis it can be concluded that (O-H), (C-H), (C=O), (-NO) and (-C=C-) groups are involved in the process of Ni(II) sequestration. As this indigenous biomass has shown a very good adsorption capacity and is easily and abundantly available; its large scale utilization is envisaged.

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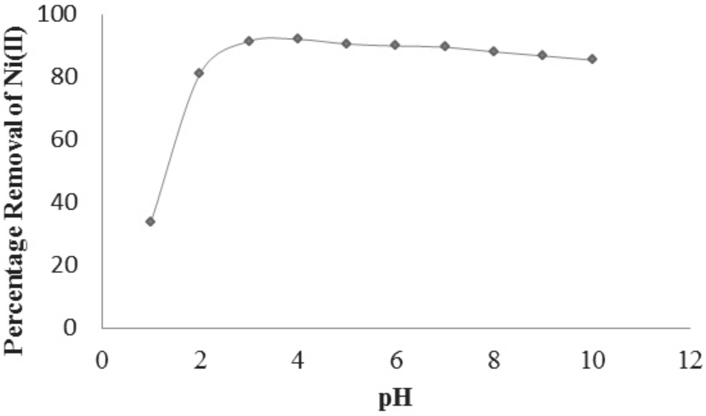

Figure. 1 Effect of pH on the percentage removal of Ni(II)

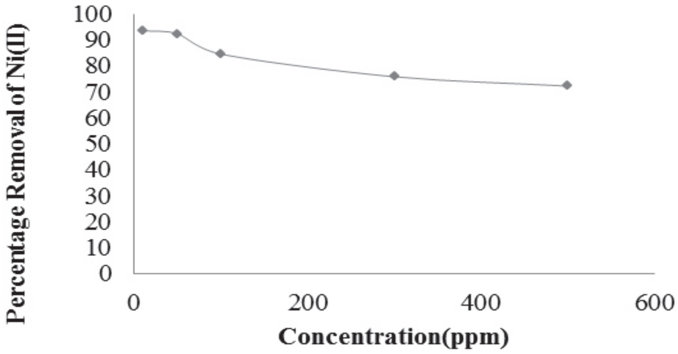

Figure. 2 Effect of concentration on the percentage Removal of Ni(II)

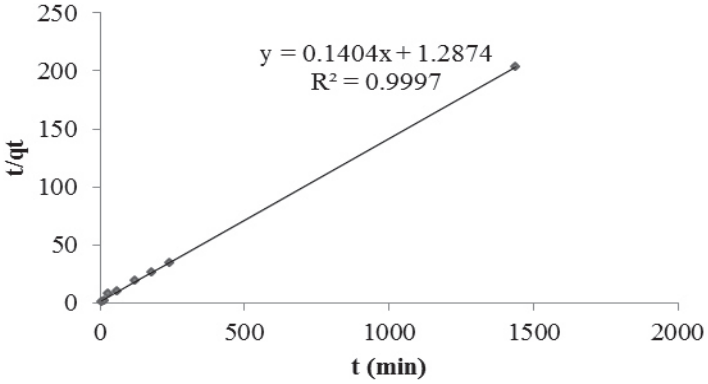

Figure. 3 Pseudo-Second-Order Kinetic Model for the Removal of Ni(II)
Where – t = Time (min), q_t = The amount of nickel adsorbed at time t (min)

Table 1. Parameters of kinetic study pseudo second order reaction.

Pseudo Second Order Equation Parameters:	
K_2(g/mg min)	0.355
R^2	0.999
$q_e$cal(mg/g)	4.48

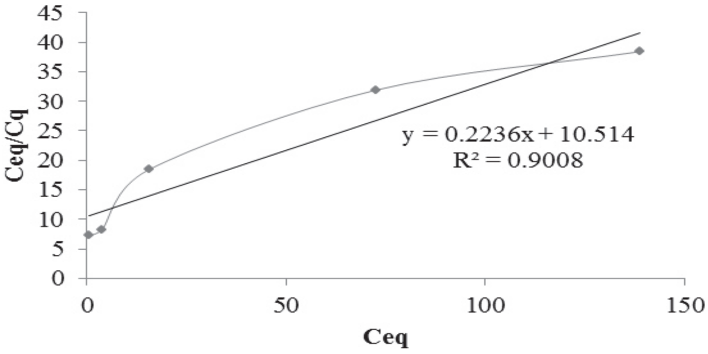

Figure. 4 Langmuir adsorption Isotherm for Batch study using chemically treated biomass
Where – C_{eq} = Equilibrium constant of metal in the solution (mg/l),
q_{eq} = The Metal amount adsorbed per unit mass of adsorbent (mg/g)

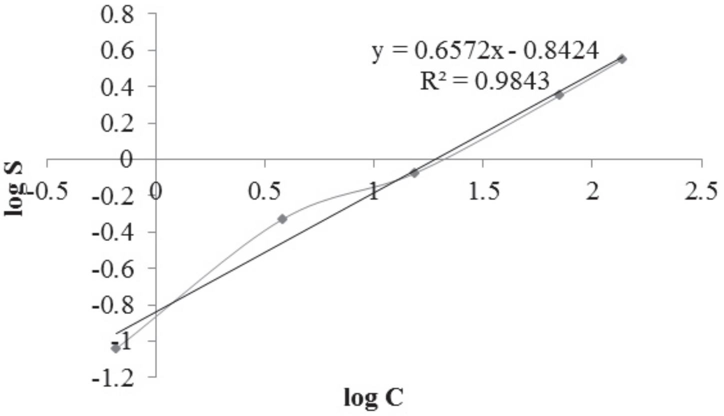

Figure.5 Freundlich adsorption Isotherm for Batch study using chemically treated biomass\
Where – log C = Equilibrium concentration of metal ion solution (mg/l), log S = Metal ion sorbed (mg/g)

Table 2. Langmuir and Freundlich constants and correlation coefficients of isotherm models.

	Langmuir Isotherm			**Freundlich Isotherm**		
Temperature (^{0}C)	q_m(mg/g)	K_L(Lmg^{-1})	R^2	1/n	K_F	R^2
27	4.48	0.0212	0.900	1.396	0.143	0.984

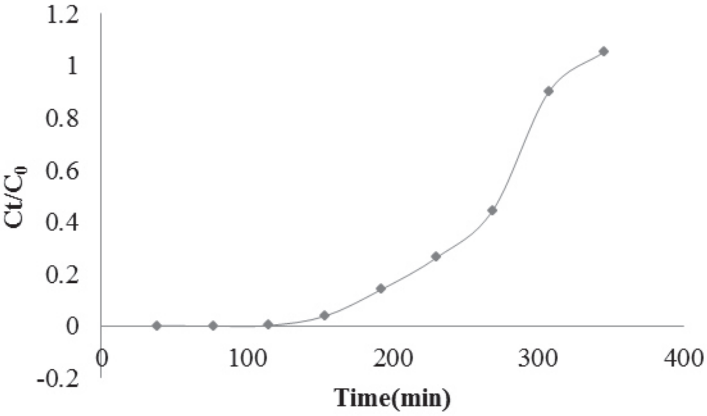

Figure. 6 Column Study of Ni(II) for using chemically treated biomass
Where – C_t = Effluent ion concentration (mg/l), C_0 = Influent ion concentration (mg/l)

Table 3. Parameters of Column Study

Metal	Concentration (ppm)	Flow Rate (ml/min)	Bed Volume (min)	EBCT	Removal Capacity (mg/g)
Ni(II)	50	13	55.31	4.25	2.49

Table 4. FT-IR Spectra bands of Native and Nickel Loaded Adsorbent

Adsorbent	O-H	-NH	-CN	Stretching Vibration
Native	3349.7 cm^{-1}	Disappear	2363.4 cm^{-1}	2925.7 cm^{-1} 1734.4 cm^{-1}
Nickel Loaded	3749.9 cm^{-1}	3429.4 cm^{-1}	Disappear	2922.9cm^{-1} 2364.0cm^{-1}

Table 5. Comparison of different biosorbents based on their maximum capacity for Nickel Biosorbent Biosorption Capacity

Biosorbent	**Biosorption Capacity (q_{m} = mg/g)**	**Reference**
Bagasse	0.001	Rao et al(2002)
Seyitomer fly ash	1.174	Bayat (2002)
Free cells	8.0	Al-Saraj et al. (1999)
Maple saw dust	0.294	Shukla et al. (2005)
Sawdust (*Dalbergia sissoo*)	10.47	Rehman et al. (2006)
Tectona grandis	4.975	Patil et al.(2012)
Coal dust	11.11	Kapur et al.(2014)
Tinospora cardifolia	**4.48**	**This Paper**

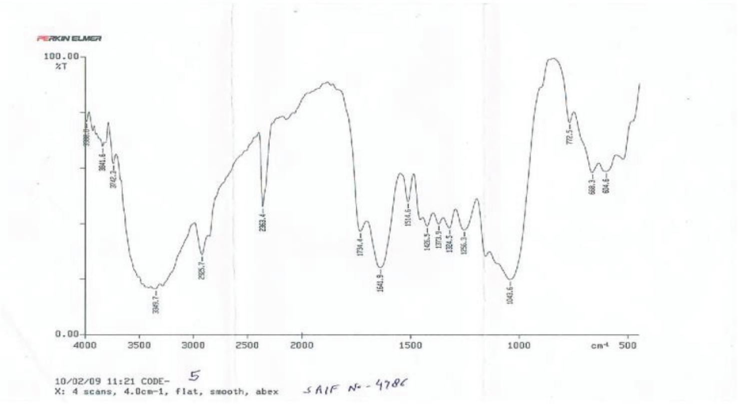

Figure. 7 FT-IR Spectra of fresh biomass of *Tinospara*

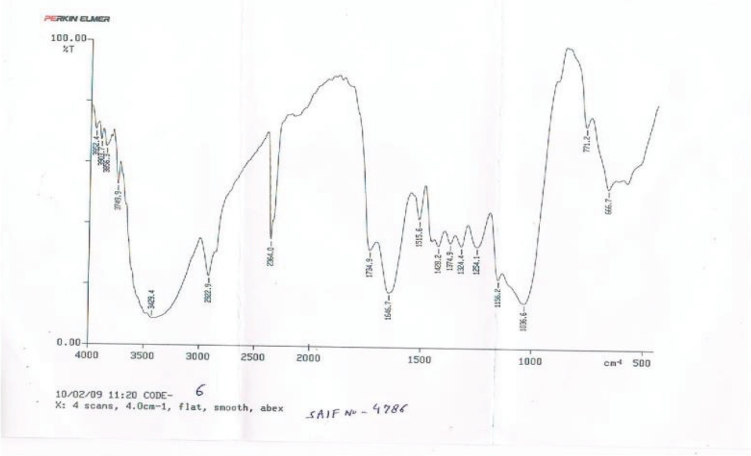

Figure. 8 FT-IR Spectra of Lead loaded biomass of *Tinospara cardifolia*