

# A Novel Eco-friendly Biomaterial *Ficus religiosa* Leaf Powder (FRLP) for the Removal of Ni (II) Ion from Water Bodies

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Received December 24, 2008; revised and accepted July 23, 2010

**Abstract:** The study explores the efficacy of *Ficus religiosa* leaf powder (FRLP) as an effective adsorbent for the abatement of Nickel (73.89%) from water bodies. Morphological changes observed in the scanning electron micrograph of treated biomass confirmed the phenomenon of biosorption and data of metal ion fitted well in Langmuir isotherm model indicating monolayer sorption. Fourier Transform Infra Red (FTIR) spectrometry analysis indicated that amino acid-metal interaction appears to be responsible for the metal binding. Desorption studies showed that the ease of metal ion recovery from metal-loaded biomass by the different eluents is of the order Nitric acid (0.05M) > Citric acid (0.5 M) > Sodium hydroxide (0.05M) > distilled water. The sorption capacity of regenerated biomass remained almost constant up to three cycles of sorption process. The successful application of this easily abundant agricultural waste FRLP, as a biosorbent has potential for a low technological pretreatment step, prior to economically non-viable high-tech chemical treatments for the removal of Ni (II) from water bodies.

**Key words:** Biosorption, nickel, *Ficus religiosa*, agricultural waste, regeneration.

## Introduction

Heavy metals even if present in dilute, undetectable quantities, their recalcitrance and consequent persistence in water bodies imply that through natural processes such as biomagnification, concentration, may be elevated to such an extent that they begin exhibiting toxic characteristics. Despite strict environment regulations, significant quantities of nickel is found in wastewater protruding from several industries such as plating plants, silver refineries, batteries, welding mining operations, tanneries, electronics, electroplating and petrochemical industries (Basso et al., 2002, Vasudaran et al., 2003). According to US Environmental Protection Agency, maximum permissible limit for Nickel in waste water is 0.1 mg/dm<sup>3</sup> (USEPA, 1997). Conventionally, methods

for removing metal ions from solution include ion exchange, reverse osmosis, chemical precipitation which are inherently problematic, expensive and ineffective at low metal concentration (Yan and Viraraghavan, 2004). Presently, there is an increasing trend to evaluate cheaper materials like agricultural wastes to remove toxic metals from water bodies. Biosorption technology removes heavy metals from wastewater without creating hazardous sludge at costs much lower than conventional methods. Several biosorbents such as grape stalk (Isabella et al., 2004), cocoa shells (Meunier et al., 2003) and *Lemna minor* (Nicholas et al., 2003) for nickel have been studied for removal from water bodies.

In continuation of our work on decontamination of metals using agricultural wastes (Srivastava et al., 2005; Kumari et al., 2005, 2006; Sharma et al., 2007; Goyal et

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al., 2007a,b, 2008a,b, 2009, Kardam et al., 2009), the present communication reports the unexploited properties of the plant *Ficus religiosa* Linn. one of the most appealing biomaterial for the removal of nickel from aqueous system. Desorption and regeneration of the biosorbent using different stripping agents was also studied thereby making the process more lucrative, beneficial and attractive than currently employed sophisticated technologies.

## Materials and Methods

### Collection and Biosorbent Preparation

Leaves of *Ficus religiosa* Linn. were collected and washed repeatedly with water to remove dust and soluble impurities, dried at 65°C for 24 hours, crushed and finally sieved through (105, 210 and 420  $\mu\text{m}$ ) mesh copper sieves.

### Metal-binding Experiments

Batch experiments were conducted in triplicates ( $N=3$ ) using standard practices as a function of particle size (105, 210 and 420  $\mu\text{m}$ ), biomass dosage (2.0, 4.0 and 6.0 g), contact time (10, 20, 30 and 40 min.), metal concentration (1, 5, 10, 25, 50 and 100 mg/l) and pH (2.5, 3.5, 4.5, 5.5, 6.5, 7.5 and 8.5). Nickel solution was prepared by dissolving the desired quantity of Nickel Sulphate (AR) in deionized water. Dilutions were made in order to have metal concentration in the range 1–100 mg/l. After pH adjustment, required quantity of biosorbent was added and finally metal bearing suspension was allowed to settle. The residual biomass sorbed with nickel ion was filtered using Whatman 42 filter paper (Whatman International Ltd., Maidstone, England). Filtrate was collected and subjected for metal ion estimation using Atomic Absorption Spectroscopy (Perkin Elmer-3100). Percent metal sorption by the sorbent was computed using the equation: % Sorption =  $(C_0 - C_e) / C_0 \times 100$ , where,  $C_0$  and  $C_e$  are the initial and final concentration of metal ions in the solution.

### Scanning Electron Microscopic Analysis

The morphological characteristics of *Ficus religiosa* leaf powder were evaluated using Steroscan 360, Scanning Electron Microscope (Cambridge, UK). The scanning electron micrographs of untreated and Ni (II) treated biomass at bar length equivalent to 20  $\mu\text{m}$ , working voltage 20 KV with 200X magnification were recorded.

### Fourier Transform Infrared Spectroscopy

In order to gain better insight into the surface functional groups that might be involved in metal sorption, FTIR

analysis in solid phase was performed using a Shimadzu 8400 Fourier Transform Infrared spectroscopy. Spectra of the sorbent before and after metal sorption were recorded.

### BET Surface Area Analysis

The surface area of the biosorbent was measured using a Micromeritics ASAP-2010 BET Surface Area analyser.

### Sorption Isotherm

The experimental data for the sorption of nickel ions by the FRLP over the studied concentration range processed in accordance with the two of the most widely used adsorption isotherms – Freundlich and Langmuir isotherm describing the adsorption phenomenon.

The classical freundlich equation is given below

$$q = K_f C_e^{1/n}$$

where,  $q$  is the heavy metal adsorbed on the biosorbent (mg/g dry weight);  $C_e$  is the final concentration of metal (mg/l) in the solution;  $K_f$  and  $n$  are the characteristic constants.

The classical Langmuir equation is given below;

$$C_e/q_e = (1/Q_0 b) + (C_e/Q_0)$$

where,  $C_e$  is the equilibrium concentration,  $q_e$  is the amount adsorbed of Ni (II) at equilibrium,  $Q_0$  and  $b$  are the Langmuir constants related to adsorption capacity and energy of adsorption respectively.

The biosorption capacity ( $K_f$  and  $Q_0$ ) and biosorption intensity/energy ( $1/n$  and  $b$ ) were estimated from the slope and intercept of the Freundlich and Langmuir isotherms.

### Desorption Experiment

Desorption studies (batch process) were conducted to restore the biomass as a function of concentration of different desorption reagents: hard acid (0.05 M  $\text{HNO}_3$ ), soft acid (0.5 M Citric acid), 0.05 M NaOH and distilled water. Metal loaded biosorbent obtained from our sorption experiments, were transferred to Erlenmeyer flasks and shaken with 50 ml of each desorption reagents as a function of time (20, 40, 60, and 80 min) at room temperature. At the end of each time interval the suspension was stirred for 5 min. The suspension was filtered using Whatman 42 filter paper and in the filtrate estimation of metal ion concentration was carried out.

The amount of metal ion remaining on the biomass as a function of time was calculated using a mass balance equation:

$$q_t = q_e - c_t (v/m)$$

where,  $q_t$  and  $q_e$  are the biomass phase metal ion concentration (mg/l) and  $c_t$  solution phase metal ion concentration (mg/l) at time  $t$  (min) respectively.

### Kinetics of Desorption

Kinetics of desorption was expressed in the linear form as follows:

$$\ln q_t/q_0 = \ln \theta - K_{\text{des}} t + (1 - \theta)$$

where,  $q_t$  is the metal concentration in the FRLP phase (mg/g biomass) at time  $t$  (min).  $q_0$  is the total desorbable metal concentration in the biomass at the beginning (mg/g biomass) and  $K_{\text{des}}$  is the pseudo first-order desorption rate constant ( $\text{min}^{-1}$ ) and values of desorbable fraction  $\theta$  and  $q$  fraction resistant desorption ( $1 - \theta$ ) can be obtained from a plot of  $\ln q_t/q_0$  against time (min).

### Statistical Analysis

Batch experiments were conducted in triplicates ( $N = 3$ ) and data represent the mean value. Correlation coefficient

and standard deviations were calculated using SPSS PC<sup>+</sup> statistical package. For the determination of inter-group mean value differences, each parameter was subjected to the student-t test for significance level ( $p < 0.05$ ).

## Results and Discussion

Sorption studies result into the standardization of the optimum conditions as biomass dosage (4.0 g), particle size (105  $\mu\text{m}$ ) metal concentration (25 mg/l), contact time (40 min) at pH 6.5 (Table 1). Maximum sorption for Ni (II) was found to be 73.89%. Results indicate that FRLP has considerable potential to be used as biosorbent for metal removal from the water bodies.

Sorption of metal was studied from pH range 2.5 to 8.5. Biosorption efficiency of the target leaf biomass for

**Table 1: Soluble Ni (II) ion concentration ( $\mu\text{M}$ ) after adsorption on *Ficus religiosa* leaf Powder (FRLP) as functions of contact time, particle size and biomass dosage at volume (200 ml), and pH (6.5)**

Initial Conc. ( $\mu\text{M}$ )	Time Interval (minutes)				
	10	20	30	40	60
<b>Biomass dosage (2.0 g)</b>					
03.41 <sup>a</sup>	02.73±0.15 <sup>bx+φ</sup>	02.45±0.13 <sup>x+</sup>	02.28±0.12 <sup>x+</sup>	02.18±0.11 <sup>x+</sup>	02.18±0.10 <sup>xx+</sup>
17.06	11.94±0.54 <sup>x+</sup>	10.85±0.52 <sup>x+</sup>	10.20±0.51 <sup>x+</sup>	09.72±0.48 <sup>x+</sup>	09.69±0.50 <sup>xx+</sup>
34.12	22.01±1.01 <sup>x+</sup>	19.72±0.92 <sup>x+</sup>	18.08±0.90 <sup>x+</sup>	17.06±0.85 <sup>x+</sup>	17.03±0.85 <sup>xx+</sup>
85.32	48.46±2.16 <sup>x+</sup>	42.45±2.16 <sup>x+</sup>	39.21±1.96 <sup>x+</sup>	36.58±1.82 <sup>x+</sup>	36.48±1.75 <sup>xx+</sup>
170.64	96.72±4.64 <sup>xx+</sup>	84.67±4.14 <sup>xx+</sup>	78.25±3.91 <sup>xx+</sup>	72.90±3.64 <sup>xx+</sup>	72.76±3.63 <sup>xx++</sup>
341.29	193.17±9.65 <sup>xx+</sup>	169.07±7.60 <sup>xx+</sup>	156.00±7.02 <sup>xx+</sup>	145.56±6.98 <sup>xx+</sup>	145.25±6.97 <sup>xx++</sup>
Correlation coefficient (R)	0.96	0.97	0.97	0.98	0.99
<b>Biomass dosage (4.0 g)</b>					
03.41 <sup>a</sup>	01.97±0.10 <sup>x+φ</sup>	01.60±0.09 <sup>x+</sup>	01.39±0.07 <sup>x+</sup>	01.33±0.07 <sup>x+</sup>	01.33±0.07 <sup>xx+</sup>
17.06	08.49±0.45 <sup>x+</sup>	06.99±0.37 <sup>x+</sup>	06.14±0.34 <sup>x+</sup>	05.76±0.29 <sup>x+</sup>	05.76±0.29 <sup>xx+</sup>
34.12	14.94±0.79 <sup>x+</sup>	12.49±0.66 <sup>x+</sup>	11.26±0.58 <sup>x+</sup>	10.22±0.52 <sup>x+</sup>	10.20±0.51 <sup>xx+</sup>
85.32	32.80±1.73 <sup>x+</sup>	26.05±1.38 <sup>x+</sup>	22.78±1.13 <sup>x+</sup>	20.78±1.07 <sup>x+</sup>	20.71±1.03 <sup>xx+</sup>
170.64	65.49±3.53 <sup>xx+</sup>	52.01±2.80 <sup>xx+</sup>	45.42±2.36 <sup>xx+</sup>	41.39±2.06 <sup>xx+</sup>	41.29±2.06 <sup>xx++</sup>
341.29	130.71±6.79 <sup>xx+</sup>	103.72±5.39 <sup>xx+</sup>	90.61±4.80 <sup>xx+</sup>	82.55±4.29 <sup>xx+</sup>	82.32±4.11 <sup>xx++</sup>
Correlation coefficient (R)	0.96	0.97	0.95	0.98	0.99
<b>Biomass dosage (6.0 g)</b>					
03.41	01.97±0.11 <sup>x+φφ</sup>	01.60±0.09 <sup>x+</sup>	01.39±0.08 <sup>x+</sup>	01.33±0.07 <sup>x+</sup>	01.33±0.08 <sup>xx+</sup>
17.06	08.49±0.45 <sup>x+</sup>	06.99±0.37 <sup>x+</sup>	06.14±0.34 <sup>x+</sup>	05.76±0.29 <sup>x+</sup>	05.76±0.29 <sup>xx+</sup>
34.12	14.91±0.76 <sup>x+</sup>	12.45±0.67 <sup>x+</sup>	11.22±0.60 <sup>x+</sup>	10.20±0.53 <sup>x+</sup>	10.17±0.52 <sup>xx+</sup>
85.32	32.74±1.57 <sup>x+</sup>	25.98±1.35 <sup>x+</sup>	22.68±1.22 <sup>x+</sup>	20.71±1.11 <sup>x+</sup>	20.64±1.07 <sup>xx+</sup>
170.64	65.35±3.59 <sup>xx+</sup>	51.80±2.84 <sup>xx+</sup>	45.25±2.39 <sup>xx+</sup>	41.26±2.18 <sup>xx+</sup>	41.12±2.17 <sup>xx++</sup>
341.29	130.30±6.25 <sup>xx+</sup>	103.31±5.16 <sup>xx+</sup>	90.23±4.51 <sup>xx+</sup>	82.21±4.27 <sup>xx+</sup>	82.01±4.26 <sup>xx++</sup>
Correlation coefficient (R)	0.95	0.95	0.93	0.95	0.96

<sup>a</sup> Number in parenthesis represent soluble metal concentrations in  $\mu\text{M}$ .

<sup>b</sup> Standard deviation values of replicate (N=3) determinations.

Mean difference [initial Ni (II) loaded ( $\mu\text{M}$ ) versus soluble Ni (II) ( $\mu\text{M}$ )] as functions of Time \*significance ( $p < 0.10$ ), <sup>xx</sup>insignificance ( $p > 0.10$ ).

Metal concentration <sup>+</sup>significance ( $p < 0.01$ ), <sup>++</sup>insignificance ( $p > 0.01$ ).

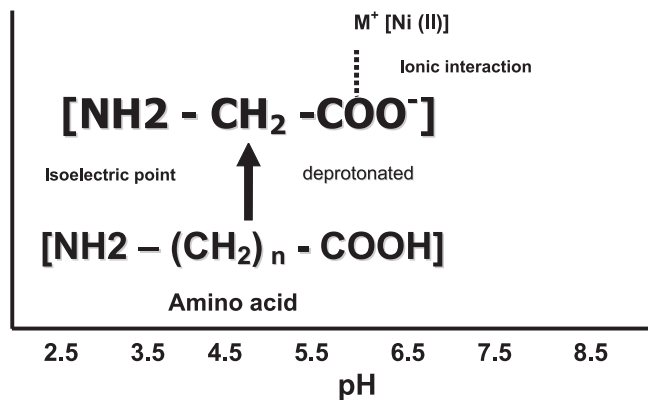
Biomass dosage <sup>φ</sup>significance ( $p < 0.01$ ), <sup>φφ</sup>insignificance ( $p > 0.01$ ).

Ni (II) ions in the pH range 2.5 to 4.5 did not vary significantly. However, there was a sharp increase in the sorption efficiency at further rise in pH from 4.5 to 6.5, thereby attaining a maximum value at pH 6.5 and onwards. Investigation on pH variation beyond 7.5 yielded an apparent increase in sorption up to pH 8.5, which might be due to precipitation carry over of metal starting at pH 7.5 onwards (Iqbal et al., 2002). Ni (II) precipitation is undistinguishable from sorption phenomenon at pH 7.5 (Ajmal et al., 2003). Studies would therefore have to be carried out to provide an insight into the mechanism of interactions between metal and FRLP.

Metal biosorption is a rather complex process affected by several factors, among which pH of the solution is the most prominent. pH not only influences functional group dissociation but also the chemistry of metals in a solution. In order to understand how metal binds to the biomass, it is essential to identify the functional groups responsible for metal binding. On the basis of our experimental findings and relevant literature, we synthesize a possible mechanism for metal binding to the FRLP biomass. The aqueous solution of leaf is a heterogeneous complex mixture with various functional groups, particularly low molecular weight amino acids (Tegbe et al., 2006). These proteinaceous amino acids have a variety of structurally related pH dependent properties of generating either positively or negatively charged sites for attracting the anionic or cationic species of metal ions, respectively (Costa et al., 1997). The majority of amino acids present in *Ficus religiosa* leaf powder have isoelectric point in the pH range 4.0–8.0 (Delvin, 2002). In this range of pH over 90% of the amino acid molecules are in the ionized state i.e. they have both positively charged amino groups and negatively charged carboxylic group. As observed sorption tendency of metal is very less at lower pH values. This is because at lower pH, binding sites (amino acid moieties) in the biomass are generally protonated thus electrostatic repulsion between cationic metallic species and positively charged amino groups seems to predominate. Our experimental findings showed that optimum sorption for nickel ion were recorded at pH 6.5. At this pH, more negatively charged carboxylic groups become available thus facilitating greater metal uptake. The sorption behaviour of the target plant biomass for Ni (II) species has been depicted in Figure 1.

### Biomass Characterization

Nickel interaction with target leaf biomass (FRLP) can further be highlighted on the basis of IR spectrum. The IR spectrum of the solid phase of untreated (native) and nickel treated samples reflects the complex nature of the



**Figure 1: pH profile and amino acid-metal (Ni (II)) interaction.**

biomass. The characteristic peak shifting of carboxylate ion from  $(1463.86 \text{ and } 1682.14 \text{ cm}^{-1})$  to  $1534.89 \text{ and } 1775.98 \text{ cm}^{-1}$  corroborates the electrostatic attraction between carboxylic ion and positively charged metal species. The powered FRLP sieved at  $105 \mu\text{m}$  had a surface area of  $4.17 \text{ m}^2/\text{g}$ . Figure 2 represents the scanning electron micrograph of untreated *Ficus religiosa* leaf powder. Morphology seems to be of large spherical clusters having pore size:  $0.17 \mu\text{m}$ ; pore area:  $6.47 \mu\text{m}^2$ . Figure 3 represents dense agglomerated, etched dendrite type morphology having pore size:  $0.04 \mu\text{m}$ , pore area:  $0.73 \mu\text{m}^2$  which is in sharp contrast to spherical type clusters appeared in untreated *Ficus religiosa* leaf powder. Observed aggregation and reduction of pore size and pore area in treated biomass of *Ficus religiosa* leaf powder indicates the liquid phase concentration of removed metal ions at experimental pH thus confirming the phenomenon of biosorption.

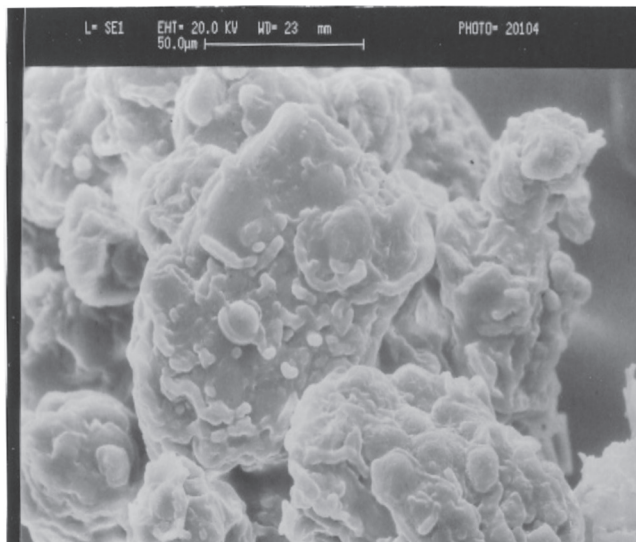
### Sorption Capacity using Isotherms

The extent of Ni (II) sorption on FRLP has been described by Langmuir isotherm. According to Langmuir model, maximum biosorption capacity ( $Q_0$ ) and energy of adsorption (b) for Ni (II) was found to be 2.51 and 0.36 which indicate its favourable sorption. Moreover, high value of the correlation coefficient of the linear regression ( $R^2 = 0.98$ ) indicate that adsorption data fitted well to Langmuir isotherms. The linearity of Langmuir plots suggested the formation of homogenous monolayer of Ni (II) on the outer surface of the biosorbent. Maximum metal uptake on LLSP was  $2.38 \text{ mg/g}$  in single metal solution.

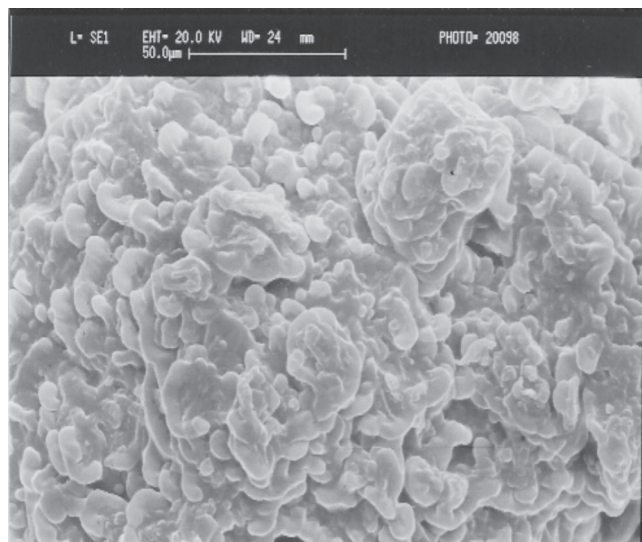
### Regeneration and Biomass Reusability

In order to design the proposed process of sorption more economical, attempts were made to regenerate the metal-treated biomass for its effective reuse. The recyclability





**Figure 2: Scanning electron micrograph of untreated FRLP showing large spherical cluster type morphology.**



**Figure 3: Scanning electron micrograph of Ni (II) treated FRLP showing dense agglomerated, etched cluster type morphology.**

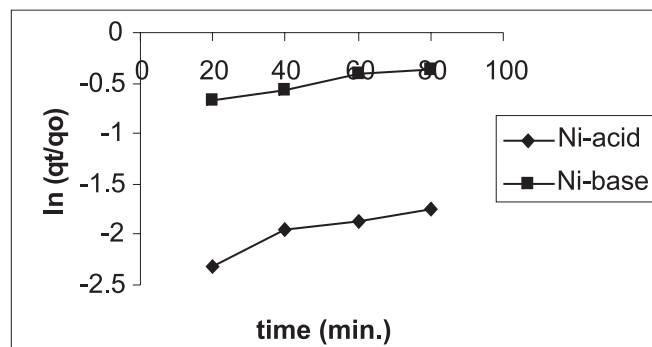
of the FRLP with desorption reagents in the acid, basic and neutral media were tried at varying contact times. It is inferred that desorption in the acidic media for metal ions were rapid and higher than in basic and neutral media. In the basic media, less than 20% of the metal ions were recovered from the metal-laden biomass. Insignificant level ( $< 4\%$ ) of desorption was recorded for distilled water. In acidic medium the carboxyl group present in the FRLP is protonated and might lose affinity for positively charged metal ions and protons replaced the bound metal ions. The poor desorption ( $< 20\%$ ) observed in basic medium may be ascribed to deprotonated co-ordinating ligands-bound metal ions which may find it difficult to be detached from the biomass. The data also indicates that desorption is a time dependent variable.

The kinetics of desorption assess the overall performance of desorbing reagent. The pseudo-first order kinetics of desorption  $K_{des}$  was used to evaluate the release constant. The larger the value of  $K_{des}$ , greater the desorption. The release constant,  $K_{des}$  and value of desorbable fraction ( $q$ ) for Ni (II) metal ion are  $8.02 \times 10^{-2}$  and 0.86 obtained from the regression lines (Figure 4). Further, the release constant of the acid reagent was double compared to the basic reagent. Among the acid eluents, hard acid (0.05 M Nitric acid) showed higher desorption, whereas comparatively low desorption rate was achieved with soft acid (0.5 M Citric acid). The regeneration of the biomaterial with nitric acid (0.05 M) sorption of metals on regenerated FRLP remained constant up to 3 cycles and then started decreasing. Citric

acid (0.5 M) elution of the metals resulted into the reusability of the biomaterial for four cycles.

## Conclusion

The laboratory based findings open up new avenues in the abatement of nickel by *Ficus religiosa* leaf powder which is infact an agricultural waste. Thus, it introduces a less expensive, domestic and environment friendly green method for the removal of nickel from water bodies. To add, *Ficus religiosa* leaf biomass is non-toxic, biodegradable and environmentally acceptable than chemical coagulants. It could be a potential challenge as primary coagulant and can be used as a pre-treatment step for the large scale removal of toxic metals from aqueous media, leading a step towards sustainable development of environment.



**Figure 4: Pseudo-first order desorption kinetics on the recovery of Ni (II) from metal loaded FRLP.**

## Acknowledgements

Authors gratefully acknowledge Prof. V.G. Das, Director, Dayalbagh Educational Institute and Prof. L.D Khemani, Head, Department of Chemistry, Agra for providing the necessary research facilities. Dr. T.K. Roy, Central Pulp and Paper Research Institute and Prof. K.K. Pant, Department of Biochemical Engineering and Biotechnology, Indian Institute of Technology, Delhi, are gratefully acknowledged for recording scanning electron micrographs and BET surface area. The authors are also grateful to Prof. Satya Prakash, Emeritus Professor of the Department for fruitful scientific discussions.

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