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Rapid Removal of Nickel(II) by Coconut Leaves Powder as Bioadsorbent

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ABSTRACT

In the present paper adsorption technique was employed for the removal of heavy metal Ni(II). The heavy metals are important for proper functioning of biological systems but their deficiency or excess lead to a large number of disorders. Industrial effluents discharged contain higher amount of heavy metals such as Ni, Cd, Hg, Cr and Cu. In this research article low cost and eco-friendly coconut leaves powder as a bioadsorbent were employed for the removal of Ni(II) from industrial wastewater. The batch adsorption study were conducted for the removal of Ni(II) along with different parametric studies of Initial concentration of nickel metal, adsorbent dose, pH, contact time and temperature. The coconut leaves powder bioadsorbent as an experimental research was applied for the systematic and effective investigation to determine the optimal conditions of the operation variables. The employed coconut leaves powder (CLP) bioadsorbent gives positive results, the adsorption of Ni(II) is very rapid and most of fixation occurs at first 15 minutes. The adsorption kinetics obeys second order kinetic. The CLP adsorbent were characterized by SEM analysis before and after treatment, which shows sufficient adsorption of Ni(II) on CLP. The result suggests that coconut leaves powder bioadsorbent can be beneficial in Ni(II) removal from the contaminated water.

1. Introduction

As the human population is increasing by leaps and bound, there is need for more production in all spheres. Many industries are coming up polluting more and more while leaving the biosphere foul and unpleasant. The incidence of air and water pollution from metals has reached such an alarming level that environment are finding it difficult to enforce control measures. All these pollutants seriously interfere with bioenvironmental process and thereby posing a menace to the life on this planet. Metals have classified into essential and non-essential group. The former consists of Ca, Mg, K, Fe, Mn, Zn, Cu and the latter of Hg, Pb, Cd, Ni, As and Se. The metals of the second group directly or indirectly, have metal pollutants cause direct toxicity, both to human and other living beings due to their presence beyond specified limits [1]. The environment and all the life forms on the earth face a very serious threats from the heavy metal pollution due to rapid industrialization and growth in world population [1]. At least 20 metals are classified as toxic and half of these are emitted into the environment in quantities that pose risk to human health [2].

Nickel as well as cadmium are the heavy metals, which are highly toxic to human, plants and animals. The metal is of special concern because it is non-degradable and therefore persistent the presence of toxic metal ions in wastewater remains a serious environmental concern. Therefore, it is necessary to develop various efficient technologies for their removal. A number of techniques have been used to remove the metal ions from wastewater, effluents including chemical precipitations [3], ion exchange processes [4,5], electrolytic methods [6], adsorption onto activated carbon [7], organic based ligand precipitation [8], membrane and reverse osmosis processes [9]. These methods have been found to be limited, because of the high capital and operating costs or the ineffectiveness in meeting stringent effluent standards. Therefore several approaches have been studied for the development of inexpensive and abundant adsorbents such as sawdust [10], live biomass [11], clay [12, 13] and agricultural byproducts, Zn(II) removal by magnetic nanoadsorbents [14-17]. Each methods has been found to be limited for the cost, complexity and efficiency, as well as secondary wastes, for example the electrolysis process often take higher operational costs and chemical precipitation may generates secondary wastes [18]. The main anthropogenic pathway

through which cadmium enters environment is via wastes from industrial processes such as electroplating, smelting, alloy manufacturing, pigments, plastic, cadmium, nickel batteries, fertilizers, pesticides, mining, pigments and dyes, textile operations and refining [19-23].

The main goal today is to adopt appropriate methods and to develop suitable techniques either to prevent the metal pollution or to reduce it to very low levels. Various physical and chemical methods like precipitation, electro dialysis, reverse osmosis, ion exchange and adsorption are involved in the removal of heavy metals [25]. Conventional adsorbents like activated carbon are effective in removal of heavy metals. But they are costly when compared to the non-conventional adsorbents like agricultural waste. The use of low-cost adsorbents in the wastewater treatment is recommended since they are relatively cheaper and available locally. In the present investigation an attempt is made to adsorb Ni(II) on coconut leaves powder (CLP). Nickel present in the wastewater of textile dyeing and printing industry. In India there are over 10,000 garment manufacturers ND 2100 bleaching and dyeing industries, mostly scattered in the urban areas. Most of the industries are not able to meet standard for the discharge of their effluent. WHO has prescribed a maximum concentration of Ni(II) in drinking water as 0.1 mg/L, where as in textile dyeing and purifying effluent water it may be up to 6.6 mg/L². Thus the effluent water has to be treated for the removal of Ni(II) [26].

The present investigation was aimed to study the effects of pH, contact time, initial concentration, equilibrium and kinetic study for the removal of Ni(II) ions from aqueous solution using the coconut leaves powder.

2. Experimental Methods

2.1 Reagents

All the chemicals used in the present study were of analytical grade. These chemicals include NiSO₄·6H₂O (Merck), 0.1 mol/L NaOH, 0.1 mol/L H₂SO₄ and DMG.

2.2 Preparation of Adsorbent

The coconut leaves were collected and washed with tap water and then dried in shadow. Dried leaves were ground sieved to 25 mesh sizes. The 50 g leaves powder was soaked in 1 liter 0.1 mg/L HNO₃ and solution kept for 12 hr to precipitate out already adsorbed metals on the biomass surface from the environment. It was then filtered and washed with

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distilled water to remove acid contents. Washing was continued till the pH of filtrate became neutral and filter cake was first dried at room temperature and then in an oven at 110 °C to constant weight. No other chemical and/or physical treatments were prior biosorption experiments. The biomass was then stored in air tight glass bottles to protect it from moisture.

2.3 Preparation and Analysis of Ni(II) Solution

In this biosorption experiments, stock nickel solution of 1000 mgL⁻¹ was prepared by dissolving an appropriate amount of nickel salt (NiSO₄·6H₂O) in deionized distilled water (DDW). Ni(II) Solution of different concentrations were prepared by adequate dilution of stock solution with DDW. All the glassware and polypropylene flasks to be used in experiments were overnight immersed in 10% (v/v) HNO₃ and rinsed several times with DDW before and after the experiments, Ni(II) content in the solutions were determined by Double Beam spectrophotometer (Systronics-2203) at λ_{max} = 232 nm. The p^H was adjusted by 0.1N HCl and 0.1N NaOH using digital pH meter.

2.4 Experimental

The biosorption and method followed to investigate the adsorption of nickel using nonconventional adsorbent, coconut leaves powder (CLP) have been described in detail. The entire chemical used was of analytical grade and double distilled water was used for all experimental work. Nickel was analyzed spectrophotometrically using standard of Ni(II) from aqueous solution using coconut leaves powder (CPL) as adsorbent. The effect of following parameter on the adsorption of Ni(II) was studied. The contact time was varied from 50 to 420 min. The adsorbent dosage was varied from 100 to 900 mg and pH was varied from 1 to 8. The initial metal concentration was varied from 10 to 50 mg L⁻¹. The biosorption experiment is carried out at room temperature.

2.5 Calibration Curve

2.5.1 Preparation of Calibration Curve

A set of known concentration of Ni(II) solution was prepared and their absorbance was determined at different wavelengths. A graph was plotted between absorbance vs wavelength, from a plot, the wavelength corresponding to maximum absorbance (λ_{max}) was determined for Ni(II) solution. The wavelength (λ_{max}) was used to measure the absorbance of further Ni(II) concentration to obtain the calibration curve of absorbance Vs concentration of Ni(II) solution. The unknown concentration of residual Ni(II) was determined using systronics-118, UV-visible spectrophotometer by using quartz's cell of 1 cm path length. In the adsorption no additional peaks formed for the dye solution after shaking with adsorbent. This indicate that there was no break down products of the dye, this indicate that the dye removal from the solution in this study were through adsorption [27-31]. The Ni⁺² samples calibrated in order to find out various absorbances at various concentrations. The calibrated results are very effective to identify the respective Ni⁺² removal capacities of coconut leaves powder as an adsorbent.

3. Results and Discussion

3.1 SEM Analysis

The scanning electron microscope (SEM) is widely used to study the morphological features and surface characteristics of adsorbent material. It is useful to determine particle shape, porosity and appropriate size distribution of the adsorbent material. The SEM micrograph of the adsorbent and Ni(II) adsorbed adsorbent were carried out at different magnifications to know the surface structure and porosity of the adsorbent. The morphological structure and surface features of CLP before and after adsorption was examine as shown in Fig. 1. The SEM micrographs observation of the CLP showed rough areas of the surface and micropores were seen before treatment of with metals solution but after treatment the micropore get adsorbed with Ni(II).

The adsorption kinetics for removal of nickel from aqueous solution of Ni(II) was studied using a non-conventional adsorbent coconut leaves powder (CLP). The results of these studies are presented below. The removal (%) at any instant of time and the amount of nickel absorbed were determined by the following equation.

$$\text{Ni(II) removal (\%)} = [(C_0 - C_e)/C_0] \times 100$$

$$\text{Amount absorbed}(Q_e) = [(C_0 - C_e)/M] \times 100$$

where C₀ and C_e are the initial and remaining concentration (mgL⁻¹) of nickel and M is the mass of adsorbent in g.

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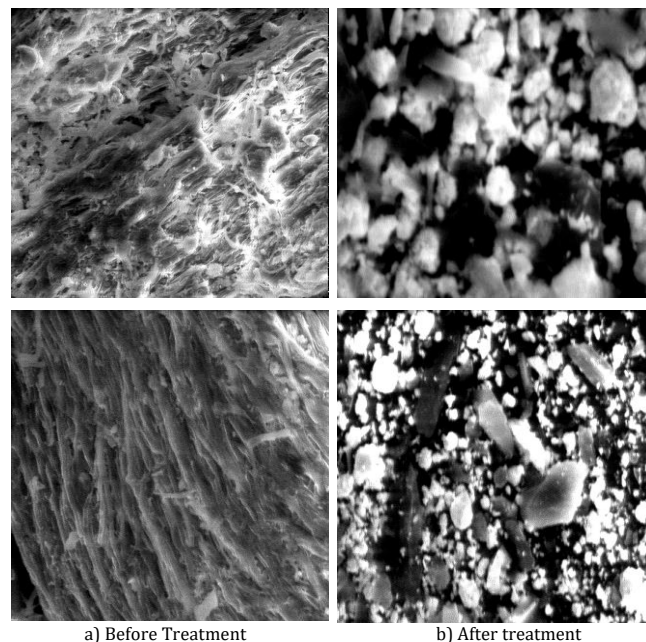


Fig. 1 SEM images of adsorbent before and after the metal removal

3.2 Effect of Contact Time

Fig. 2 shows that the contact time has a great effect on nickel removal with increasing the contact time, the adsorption percentage also increase up to 300 min. afterwards it decreases 45% reduction was observed in first 30 min of contact time due to the availability of vacant sites. As the contact time is increased further, the % reduction increasing at slower rate up to 300 min. thereafter, the % reduction decreases with increasing the contact time, probably due to desorption of the metal ions back to that solution. The maximum removal was noted at 5 hr (300 min) and the percent reduction in the metal ion observed was 88.6%.

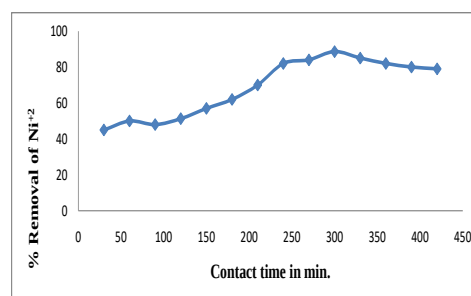


Fig. 2 Effect of contact time on percentage removal of Ni⁺² adsorbent dose 250 mg/L, at pH 5.5.

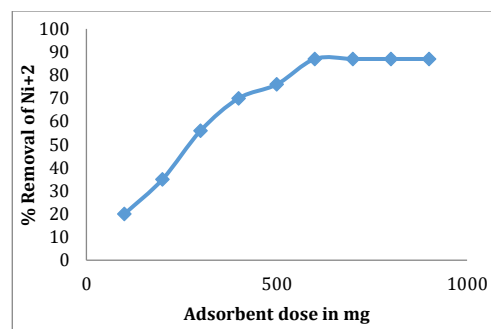


Fig. 3 Effect adsorbent dose of on percentage removal of Ni(II) at contact time 30 min, at pH 5.5

3.3 Effect of Adsorbent Dose

It is necessary to determine the dosage of adsorbent required to achieve a desired level of treatment with a view to economize on the amount of adsorbent to be used. It has been shown by several workers that the extent of adsorption at solid solution interface is a strong function of adsorbent dosage. In the present study, the effect of various adsorbent is strong function of adsorbent dosage. In the present study, the effect of various adsorbent dosages of coconut leaves powder. CLP on nickel removal was studied at optimum time 5 Hrs. In graph representation of

Fig. 3 shows that as the adsorbent dosage is increased from 100 to 600 mg, the % reduction of the metal ion also increased continuously After 600 mg while applying more dosages, viz, 700, 800, 900 mg. It was observed that the percent reduction becomes constant, might be due to less contact of the adsorbent with the adsorbate when present in bulk. The maximum removal was noted for 600 mg dosage.

3.4 Effect of pH

The pH of solution from which adsorbent occurs may affect the extent of adsorption for several reasons. Due to strong adsorption of hydrogen from the solution and hydroxyl ions, the adsorption of the ions is strongly influenced. The pH of the solution also affects the ionization and in turn affects the extent of adsorption. Increase in hydrogen ion concentration also results in neutralization of negative charge at the surface of the adsorbent thereby reducing hindrance to diffusion and making available more of the active surface of the adsorbent. The % removal of nickel was studied at optimum time 5 hr and optimum adsorbent dose 600 mg.

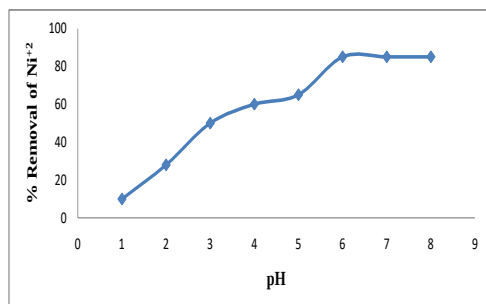


Fig. 4 Effect of pH on percentage removal of Ni(II) at adsorbent dose 250 mg/L, contact time 30 min, at pH 5.5.

The % reduction was found to be increased with increasing pH up to 6 due to less competition offered by H^+ ion which is of smaller size as compared with Ni(II). Since adsorption is affected by the presences of free metal ion concentration as P^H increased above 7 toward alkaline side, the solution was observed to be turbid probably due to formation of metal hydroxide. The Fig. 4 shows the higher percentage of reduction may be due to precipitation along with adsorption or adsorption of monodentate metal complex formed due to hydrolysis. Though, higher percentage of reduction was observed at pH 6, the optimum value chosen for further batch studies was pH 6 due to the lack of crystal structure of the adsorbent to show the hydroxide complex adsorption.

3.5 Effect of Concentration

The effect on concentration on adsorption of solute is dependent factors such as pH, temperature, ionic strength of solution, particle size, dosage etc. of the adsorbent. Several investigations have reported similar observation is reported from the data obtained from removal of Ni(II) using coconut leaves powder (CLP) as adsorbent. The Ni(II) concentration taken as 10 to 130 mg/L with optimum adsorbent dosage of 600 mg, optimum pH 6 and for an optimum contact time of 5 hr (300 min). It is observed that by increasing concentrations, the percentage removal of Ni(II) is decreasing simultaneously Fig. 5. The following equation was employed for adsorption data.

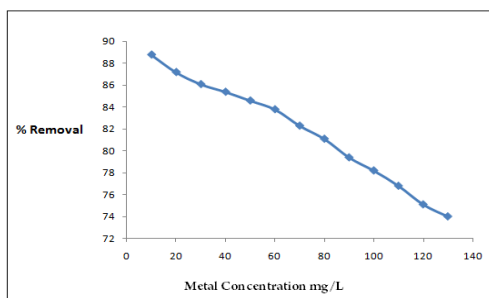


Fig. 5 Effect of concentration on percentage removal of Ni(II) at adsorbent dose 250 mg/L, contact time 30 min, at pH 5.5.

3.6 Adsorption Isotherms

The adsorption isotherm is highly significant in the removal of Ni(II) by the adsorption technique, as it provides an approximate estimation of the sorption capacity of the adsorbents. The equilibrium data for the removal of nickel by adsorption on adsorbents at 30 °C is used in Freundlich and Langmuir isotherm [25].

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$$K = (2.303/t) \log(C_0/C_e)$$

The rate constant $\ln C_0/C_e$ vs K (min^{-1}) was plotted Fig. 6 for varying metal concentration which was found to be linear indicating that the applicability of kinetic equation and is to be first order kinetics.

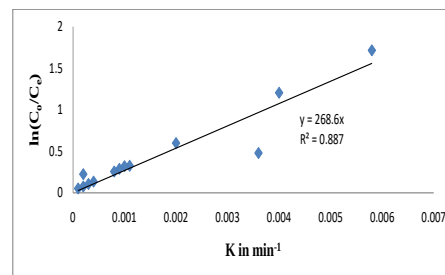


Fig. 6 First order kinetics of removal of Ni(II) at adsorbent dose 250 mg/L, contact time 30 min, at pH 5.5

3.6.1 Freundlich Adsorption Isotherm

The equilibrium data for the removal of nickel by sorption on adsorbents at 30 °C is used in Freundlich isotherm,

$$\log Q_e = (1/n) \log C_e + \log K_F$$

where C_e is equilibrium concentration of nickel in mg/L, Q_e is the amount of nickel absorbed per unit weight of adsorbent in mg/g.

K_F and $1/n$ are the measures of adsorption capacity and intensity of adsorption respectively. The equilibrium adsorption data at different Nickel(II) concentrations are fitted with Freundlich isotherm model. The values of K_F and n are calculated from the intercept and slope respectively of this linearized plot and are listed in Table 1 along with R^2 value.

Table 1 Freundlich isotherm constant for Ni(II) removal

Adsorbent used	K_F	$1/n$	R^2
Coconut leaves powder	12.02	0.144	0.674

The calculated value of $1/n$ is in between 0 and 1, which indicated the favorable adsorption of Ni(II) on coconut leaves powder. The R^2 value is very close to unity, which indicates the Freundlich isotherm is applicable.

3.6.2 Langmuir Adsorption Isotherm

The equilibrium data for the removal of nickel by sorption on adsorbent at 30 °C is used in Langmuir isotherm.

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

where C_e is equilibrium concentration of nickel in mg/L, Q_e is the amount of nickel absorbed per unit weight of adsorbent in mg/g, Q_0 is Langmuir constant related to the capacity, and b is Langmuir constant related to the energy adsorbent. The Langmuir isotherm plotted between C_e and C_e/Q_e was found to be linear over a wide range (Fig. 7).

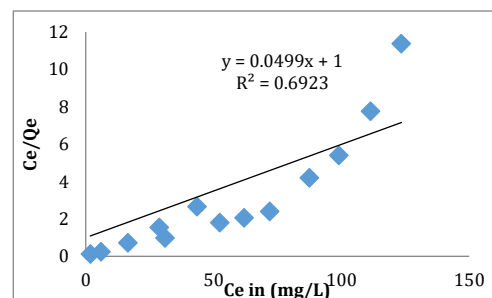


Fig. 7 Langmuir isotherm for adsorption of removal of Ni(II) at adsorbent dose 250 mg/L, contact time 30 min, at pH 5.5.

The value of the Langmuir constant Q_0 and b are given in the Table 2, which were calculated from slopes and intercepts of the Langmuir plot. The essential characteristics of the Langmuir isotherm may be expressed in term of a dimensional equilibrium parameter R_L , which is defined by,

$$R_L = 1/(1 + bC_0)$$

where, C_0 is initial concentration of nickel in mg/L and b is Langmuir constant in g/L. The value of $R_L > 1$ shows unfavorable adsorption. The value of $R_L = 1$ shows that linear adsorption and the value of $R_L < 1$ shows favorable adsorption. The value R_L is equal to 0.495 which is $0 < R_L < 1$

indicates that adsorption is more favorable with coconut leaves powder. Similar results were obtained to Aslam et al. [27].

Table 2 Langmuir isotherm constant for Ni(II) removal

Adsorbent used	Q_o	b	R_L	R^2
Coconut leaves powder	0.049	20.40	0.495	0.692

3.7 Adsorption Kinetics

The rate at which nickel will be removed from dilute aqueous solution by the adsorbents is a significant factor for application of this process in the treatment of nickel wastewaters. The rate limiting step for adsorption may either be film diffusion, pore diffusion or adsorption at the external surface sites of adsorbents. In a rapidly stirred batch reactor using adsorbents with little intra porous surface. Film diffusion and pore diffusion resistances are negligible. Thus, only step controlling the overall adsorption rate during nickel uptake will be adsorption at the active surface centers of the adsorption. The rate of adsorption which predict the contact time of the adsorption can be determined from Lagergren equation,

$$\log(C_e - Q_e) = \log C_e - (K_t/2.303)$$

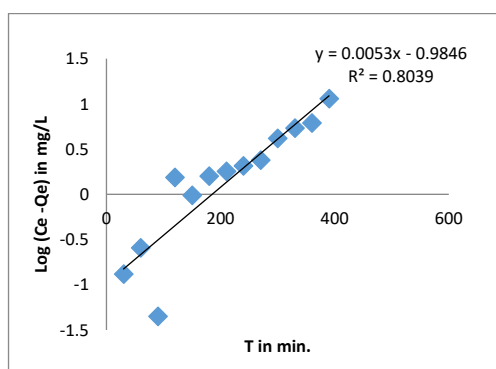


Fig. 8 Second order kinetics of Ni(II) at adsorbent dose 250 mg/L, contact time 30 min, at pH 5.5.

An attempt has made to analyze the result in the light of the Lagergren model [24] with a view to evaluate the mechanistic parameter associated with the adsorption process. This equation suggests linearity for the plot of $\log(C_e - Q_e)$ against time. A linear relationship was observed among the plotted parameters indicating the applicability of the above equation and the first order value of the uptake of the adsorbate (Fig. 8). It is of interest to study the probability of the adsorbate species to diffuse into the interior sites of the particles of the adsorbent. For his purpose, Weber Morris equation⁴ was tested. In diffusion rate studies, processes are usually expressed in terms of square root of time. The plot of mass of nickel adsorbed per unit mass of adsorbent vs $T^{1/2}$ for coconut leaves powder. The linear portion of the plot for wide range of contact period between adsorbent and adsorbate does not pass through the origin. The deviation of straight line from the origin or near saturation is due to difference in the rate of mass transfer in the initial and final stages of adsorption.

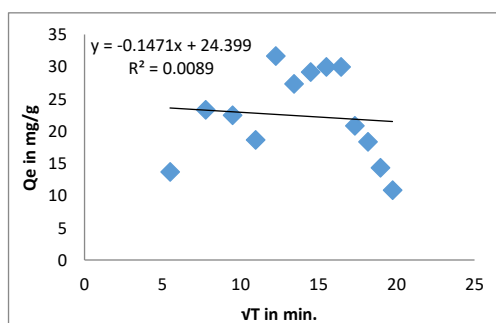


Fig. 9 Freundlich adsorption isotherm for adsorption of Ni(II) at adsorbent dose 250 mg/L, contact time 30 min, at pH 5.5.

Straight line from the origin indicates that the pore diffusion is not the only rate controlling step. It is also seen that there are two separate region – the initial pore diffusion due to external mass transfer effect followed by the intra particle diffusion. The linear plot indicates the diffusion of the adsorbate species from the surface film into the micro pores. The Fig. 9 shows the line is not passing through the origin thereby indicating that intra particle diffusion is not the only rate determining step.

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4. Conclusion

The experimental results reveal that the optimum contact time for removal of Ni(II) using coconut leaves powder was 5 hr (300min). The optimum adsorbent dosage for removal of Ni(II) was found to be 600 mg. The optimum pH for adsorption of Ni(II) from aqueous solution using coconut leaves powder was found to be 6. The percentage removal for an initial concentration of 10 mg/L for a dosage of 600 mg of coconut leaves powder at pH 6 and for a contact time of 5 hr (300 min) was found to be 82%, whereas for an initial concentration of 130 mg/L under same optimum conditions. It was found to be 5% indicating that a high concentration of metal ions, the efficiency of the adsorbent has been decreased. The adsorbent of Ni(II) using coconut leaves powder follows Freundlich isotherm. It was found to be linear over a wide range. Langmuir isotherm was fitted well into the adsorption of Ni(II) using coconut leaves powder. The limitless separation factor (R_L) for Langmuir isotherm was found to be < 1 , hence it predicts a favorable adsorption process. the plot of $\ln(C_o/C_e)$ vs $K(\text{min}^{-1})$ was found to be linear indicating the first order value of the adsorption process. Lagergren equation and Weber Morris equation were fitted well for the adsorption data and was found to be linear, further supporting the first order kinetics of the adsorption.

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