
ORIGINAL ARTICLE

Adsorption Isotherm, Kinetic and Thermodynamic Studies for the removal of Ni(II) from Aqueous Solution using Bamboo leaves activated carbon

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ABSTRACT

Activated carbon was prepared from bamboo leaves by chemical activation method and was used as an adsorbent for the removal of nickel from aqueous solution. Surface morphology and functional groups present on the surface of prepared activated carbon were studied by SEM and FTIR. The influence of different parameters such as contact time, metal ion concentration, adsorbent dose and pH of the solution on adsorption process was studied. The experimental data were investigated by the Freundlich isotherm and the Langmuir isotherm models. Langmuir model fitted well with the experimental data, with the maximum adsorption capacity 31.05 mg/g. This implies occurrence of monolayer adsorption of metal ions on the surface of bamboo leave activated carbon. The kinetic data followed the pseudo-second-order kinetic model which considers chemisorptions as the rate limiting step. Thermodynamic parameters showed that the adsorption process was spontaneous and endothermic in nature. The present study showed that the activated carbon has great ability for the removal of nickel from aqueous solutions and can be used for wastewater treatment because of their high adsorption capacity, easily available and economical.

Keywords: activated carbon, aqueous solution, adsorption isotherm, kinetic study.

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INTRODUCTION

Contamination by heavy metals is one of the critical environmental issues in the present world [1]. In the developing world, pollution of water bodies is highly contentious issue as it has become a menace due to the discharge of effluents from different industries. These pollutants find their way into the environment through anthropogenic and natural sources [2]. Heavy metal pollution needs special concern due to their carcinogenic effect and detrimental impacts on human health and the environment [3, 4]. Heavy metals are non biodegradable as compared to other organic pollutants [5] subsequently accumulating in different environmental reserves (i.e., water and soil) there are more chances of its bioaccumulation and transfer of them into the food chain [6, 7]. Removal of heavy metals is an exigent concern to trim down their noxious effects on human health [8].

Nickel is being released into the environment through nickel mining and industries that deal with the nickel compounds. An uptake of nickel has several health effects which include development of lung cancer, prostate, cardiovascular diseases, respiratory failure and birth defects. As per the BIS standard the permissible limit of nickel in drinking water is 0.02 mg/L. Various physico-chemical methods coagulation, precipitation, complexation, ion exchange, solvent extraction, foam flotation, electron deposition, membrane operation have been employed so far for the treatment of waste water which proved to be inefficient and costly [9,10]. Continuous efforts are being made to develop an alternative method which is more flexible and ecofriendly [11, 12]. Adsorption has evolved as an effective treatment technique for the removal of heavy metals from waste water as the method offers flexibility in operation. Adsorption involves the transfer of adsorbate from liquid solution onto the surface of adsorbent. The

main advantage of the adsorption method is that it can be carried out on spot at the contamination site without producing secondary pollutants. The adsorbate is bound to adsorbent either by physical or chemical interaction [13]. The adsorption mechanism of heavy metals on activated carbon involves electrostatic attraction, physical adsorption, ion exchange and surface complexation [14]. The activated carbon is mostly modified with the aim to increase the number of functional groups, surface area and pore volume which help in adsorption of heavy metals. Numerous studies revealed that activated carbon has more adsorption capacity as compared to virgin carbon because of increased surface functional groups [15, 16].

MATERIAL AND METHODS

Preparation of stock solution

All the chemicals were used of analytical grade and double distilled water used for the preparation stock solution. Nickel stock solution was prepared from nickel sulphate and the working solutions were prepared from serial dilution of stock solution.

Preparation of activated carbon

Bamboo leaves were used for the preparation of activated carbon. The carbon prepared from bamboo leaves were subjected to chemical activation. The prepared carbon were mixed with 45% w/w of H_3PO_4 at mole ratio of carbon to $H_3PO_4 = 1:2$. [17] for activation and were kept overnight. The carbons mixed with H_3PO_4 were again heated in muffle furnace at the temperature of 500 °C for 4 hours. The resultant activated carbon was washed with double distilled water till pH of the filtrate became neutral and was again dried in hot air oven at 105°C. The prepared sample was cooled down and was grinded to fine powder and was used as adsorbent. The activated carbon prepared from bamboo leaves were designed as BLAC.

Characterization

Various characterization techniques were used to characterize the surface of the prepared material. The FTIR spectra (400-4000 cm^{-1}) of the activated carbon before and after adsorption of metals were obtained using a Perkin Elmer Spectrum-2 spectrometer. The morphology and microstructure of the BLAC were examined on (SEM) scanning electron microscopy. The heavy metal ion concentration in solutions was detected by Atomic absorption Spectrometry instrument (Perkin-Elmer, Analyst, origin USA).

RESULTS AND DISCUSSION

FTIR

The FTIR spectra (400-4000 cm^{-1}) of the BLAC, and BLAC-Ni were obtained by using a Perkin Elmer Spectrum-2 spectrometer. FTIR analysis was performed to verify the different functional groups present on activated carbon which help in establishing contact with the heavy metals (Fig. 1a). The spectra of activated revealed the peaks at 2924 cm^{-1} which corresponds C - H stretching and the peak at 1589 cm^{-1} correspond to C=O carboxylic group and the band at 1489 cm^{-1} is due to C-H stretching. The peak at 1127 cm^{-1} may be due to C-O stretching vibration and the peaks between 614 - 827 cm^{-1} are due to aromatic C-H stretching. After the adsorption of metal the peaks shows evident shift which reflect electronic attraction between heavy metals and functional groups.

SEM

The surface morphology was visualized by using scanning electron microscopy. Samples were mounted on aluminium support using a double adhesive tape coated with gold in HUSSGB vacuum coating unit and observed in Hitachi S-520 SEM unit. The SEM micrographs of activated carbon (BLAC) are presented in (Fig. 2). SEM micrograph of BLAC provides clear picture of porosity and irregular size as it is evident from SEM micrograph that the external surface of the activated carbon (BLAC) is full of cavities. The development of pores on the surface of the activated carbon is attributed to H_3PO_4 activation, thus leading to large surface area and mesoporous structure of the activated carbon, which favours the adsorption process [18] and after the adsorption of metals, the adsorbent surface became abnormal and a great deal of substance adhered to the surface showing agglomeration.

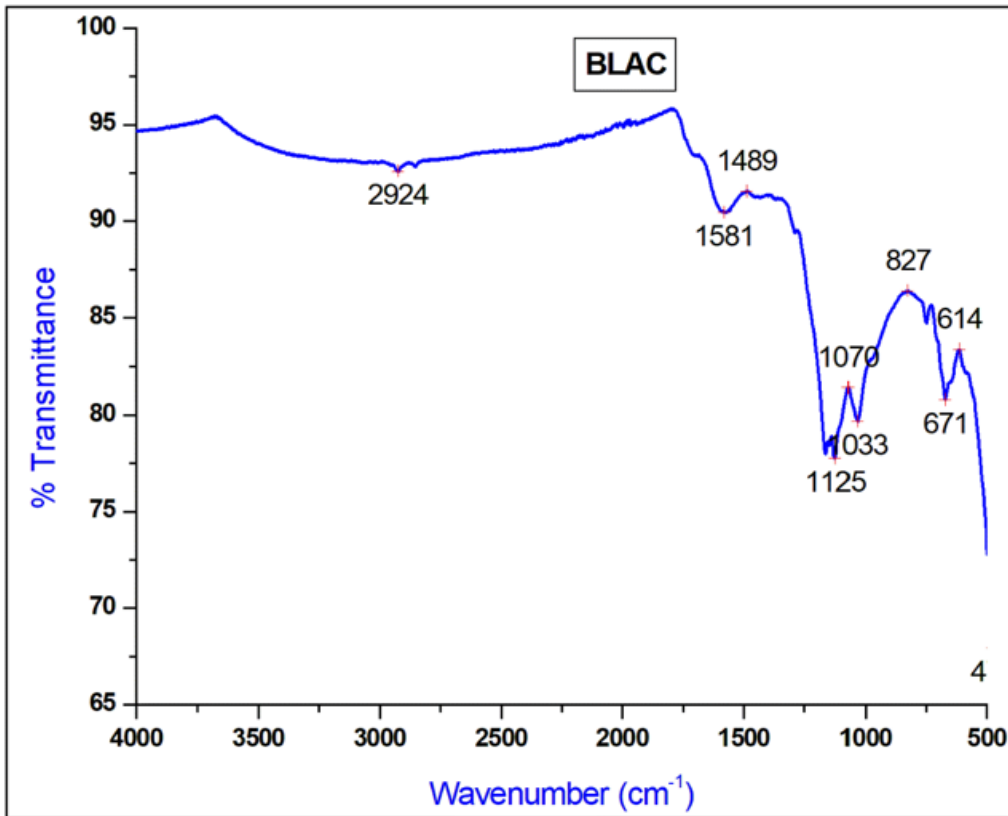


Fig. 1(a): FTIR spectra of activated carbon.

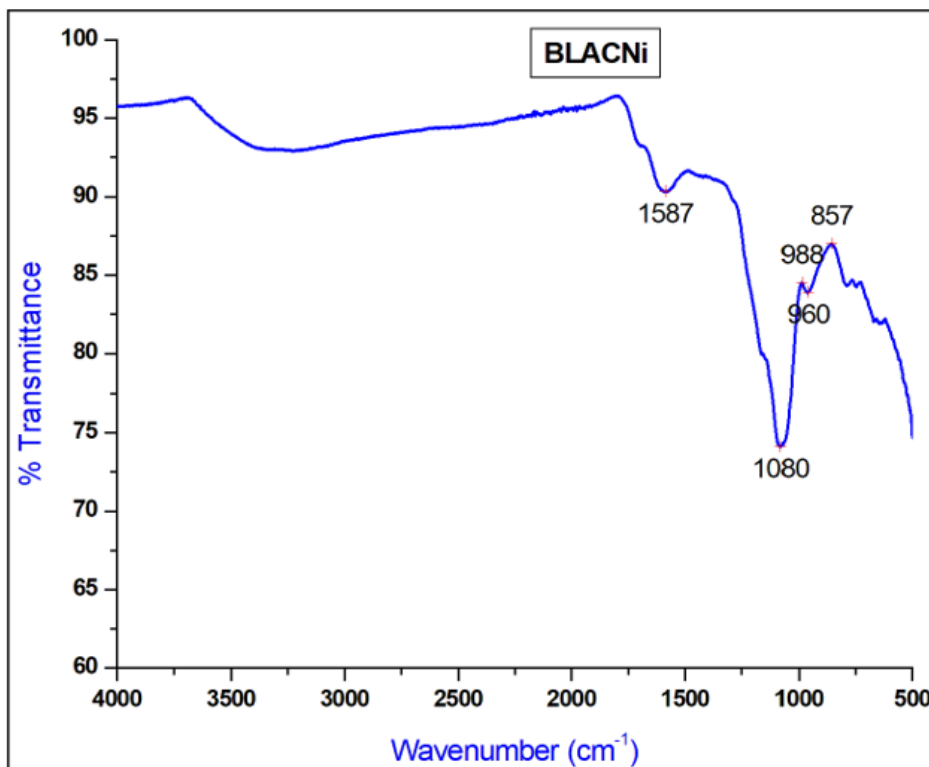


Fig. 1(b): FTIR spectra of activated carbon after the adsorption of Ni(II).

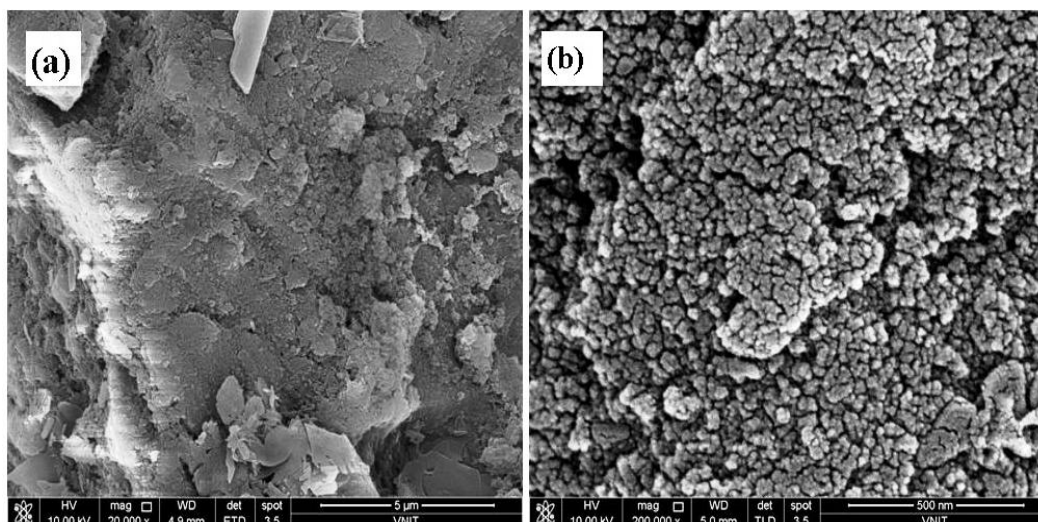


Fig. 2: SEM micrograph of activated carbon prepared from bamboo leaves.

Batch adsorption experiments

Batch adsorption experiments were carried out in order to evaluate the use of BLAC as adsorbent for the removal of Ni(II) ions from aqueous solutions. Batch adsorption was performed in a series of 250 mL Erlenmeyer conical flasks containing 100 mL metal ion solution of different concentration between 10 and 100 mg/L. All the samples were stirred on rotator shaker at different contact time between 15 and 150 minutes to ensure equilibrium, after the addition of adsorbent dose from 0.2 - 4.0g/L. The pH of the solution was adjusted between 2 and 12 by using HCl and NaOH. The amount of metal adsorbed per unit mass of modified adsorbent (mg/g) was calculated by using following equation

$$q_e = \frac{C_i - C_e}{m} \times 100 \quad (1)$$

and the removal percentage was calculated by the equation

$$\% = \frac{C_i - C_e}{C_i} \times 100 \quad (2)$$

Where C_i is initial concentration and C_e is the equilibrium concentration of the metal (mg/L) respectively, V is the volume of solution (L) and m is the mass of adsorbent (g/L).

Effect of contact time

The effect of contact time on removal of Ni(II) were investigated at a room temperature of 25°C, with increasing contact time from 15 minutes to 150 minutes for a set concentration of metal ions (10mg/l) and a fixed adsorbent mass (2 g/L) [19] as shown in Fig. 3. The adsorption of Ni(II) on the surface of adsorbent was found to be increased with increasing contact time. The metal ions adsorption increased steeply with time up to 90 min and thereafter showed static adsorption rate which defines that equilibrium is attained at 90 min. The maximum adsorption of Ni(II) ions was 76% at 90 min. However, after 90 min of contact time, percentage removal shows a constant trend. At initial stage maximum numbers of vacant sites are available for adsorption of heavy metals [20], however, after sometime these free sites are hard to occupy due to repulsive force between metal ion on the surface of adsorbent and liquid phase [21].

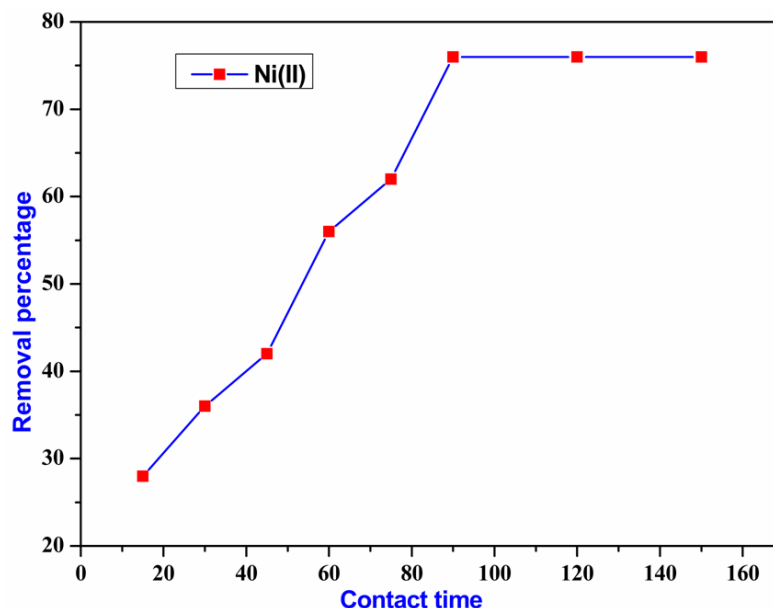


Fig. 3: Effect of contact time on adsorption of Ni(II) from aqueous solution.

Effect of concentration

The effect of metal ion concentration on removal of Ni(II) was evaluated at optimized experimental conditions. It is apparent from the results that removal of metal ions decreases by increasing the concentration from 10-100 mg/L. The removal percentage decreases from 79 to 45% for Ni(II), by increasing metal ion concentration from 10-100 mg/L using BLAC as an adsorbent (Fig. 4). The metal ion uptake particularly depends on the metal ion concentration present in the solution. At lower concentration, adsorption by available free binding sites of adsorbent is high because more binding sites are available while at higher concentration adsorption decreases due to the saturation of available binding sites and also due to lack of enough surface area to accumulate more available metal ions from the solution [22].

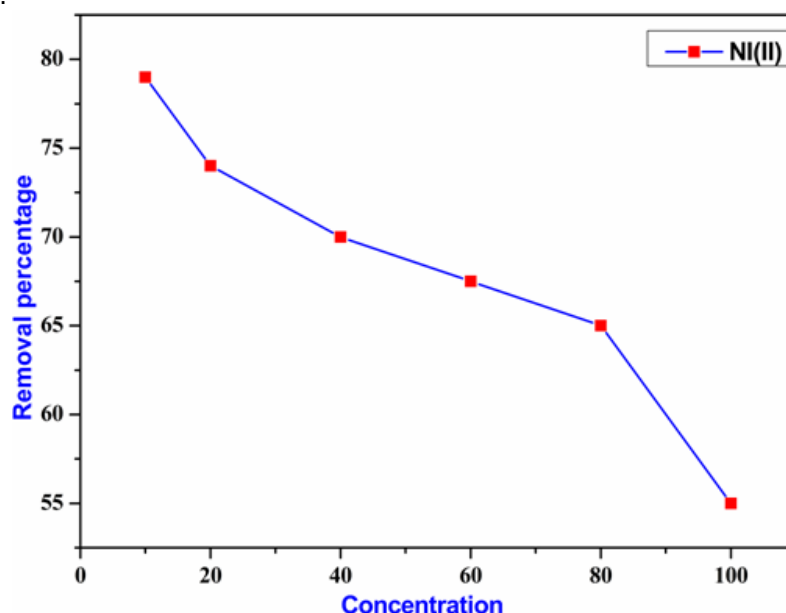


Fig. 4: Effect of metal ion concentration on adsorption of Ni(II) from aqueous solution.

Effect of adsorbent dose

The effect of adsorbent dose on adsorption of Ni(II) was evaluated at different amount of adsorbent dose (0.2 - 4.0 g/L) and keeping the other parameters constant like metal ion concentration (10 mg/l), contact time 90 minutes and pH (6.0). The results indicated that percentage removal increases by increasing the adsorbent dosage. The removal percentage of Ni(II) increases from 46 to 76% shows maximum removal

of 76% at adsorbent dosage of 2.0 g/L (Fig. 5). The increase in removal percentage by increasing adsorbent dosage may be attributed to increase of surface area of adsorbent which results in increase in number of adsorption sites on surface of adsorbent (BLAC) and by increasing the amount of dose further the removal percentage decreases because of overlapping of adsorbent material and only a part of active binding sites are exposed which are occupied by metal ions. The dosage, at which maximum removal was attained was taken as optimum dosage and was used in further studies [23].

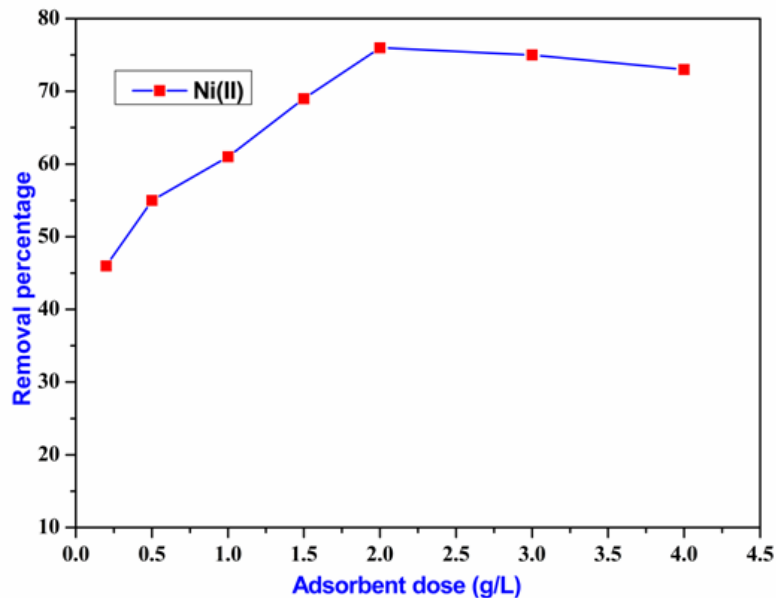


Fig. 5: Effect of adsorbent dose on adsorption of Ni(II) from aqueous solution.

Effect of pH

The pH plays a significant role in the removal of metal ions on the surface of adsorbents [24]. In this experiment, the effect of pH on the removal Ni(II) was studied at optimized contact time, concentration and adsorbent dose. The pH of solution was varied between 2.0-12 by using HCl and NaOH. The result revealed that percentage removal shows a drift of increasing metal ion removal with increasing pH from 2.0 to 6.0 [25] and the maximum removal were observed at pH 6 with removal percentage of 75%, whereas, beyond pH 6.0 the metal ion percentage removal start slight decreasing (Fig. 6). A decline in removal percentage observed was probably due to saturation of all binding sites present on the adsorbent surface on or beyond pH 6. At low pH the removal percentage declines because of the fact that adsorption sites are occupied by the protons which lead to electrostatic repulsion from protons present on the surface of adsorbent [26].

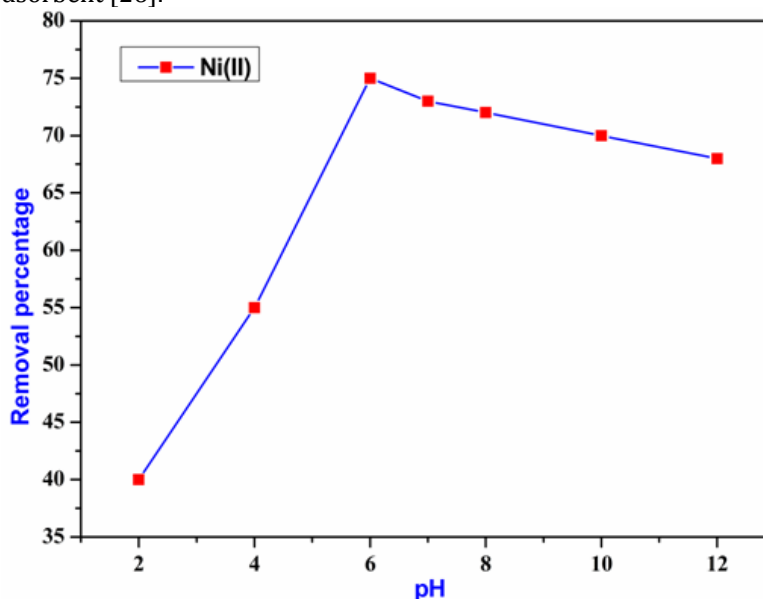


Fig. 6: Effect of pH on adsorption of Ni(II) from aqueous solution.

ADSORPTION ISOTHERM STUDY

Freundlich and Langmuir isotherm study

Adsorption isotherm models are used to determine the adsorbent adsorbate relationship. The adsorption process was investigated using following three equilibrium isotherms; Freundlich, Langmuir isotherm, models. The isothermal studies were performed at varying initial metal ion concentration from 10-100 mg/L at optimized contact time and constant adsorbent dosage of BLAC. When the metal ion comes in contact with the adsorbent in the solution, the metal ions are adsorbed onto the surface of adsorbent till the equilibrium is attained and this point clearly indicates that the distribution of metal ions on the surface of adsorbent from liquid phase [27]. The metal adsorption capacities were calculated by means of least squares regression using linear expression of Freundlich Isotherm Eq.(3) and Langmuir Isotherm [28] Eq.(4). Freundlich Isotherm was used to fit the equilibrium adsorption data and can be expressed by using following equation

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (3)$$

Where C_e the equilibrium metal ion concentration (mg/l), K_F provides an indication of the adsorption capacity and n is related to the intensity of adsorption [29]. The value of the parameters K_F and n was obtained using linear fit of the plot $\log q_e$ vs $\log C_e$ (Fig. 7a). Langmuir isotherm was used to fit the equilibrium adsorption data and can be expressed by using following equation

$$\frac{C_e}{q_e} = \frac{1}{K_L q_m} + \frac{C_e}{q_m} \quad (4)$$

Where q_e , q_m are equilibrium adsorption capacity under given condition and maximum adsorption capacity, respectively. C_e is the equilibrium concentration. K_L is the Langmuir constant which is related to the energy of adsorption [30]. Maximum adsorption capacity, q_m and K_L (l/g) were obtained from slope of the graph plotted between C_e/q_e versus C_e . (Fig.7b). The value of regression coefficient in case of Langmuir isotherm fits better as compared to that of Freundlich isotherm indicating that the equilibrium data are best fitted to Langmuir isotherm which indicates that there is occurrence of monolayer adsorption of metal ions on the surface of BLAC. The maximum adsorption capacity (q_m) obtained were 31.05 mg/g for Ni(II) as depicted in Table 1.

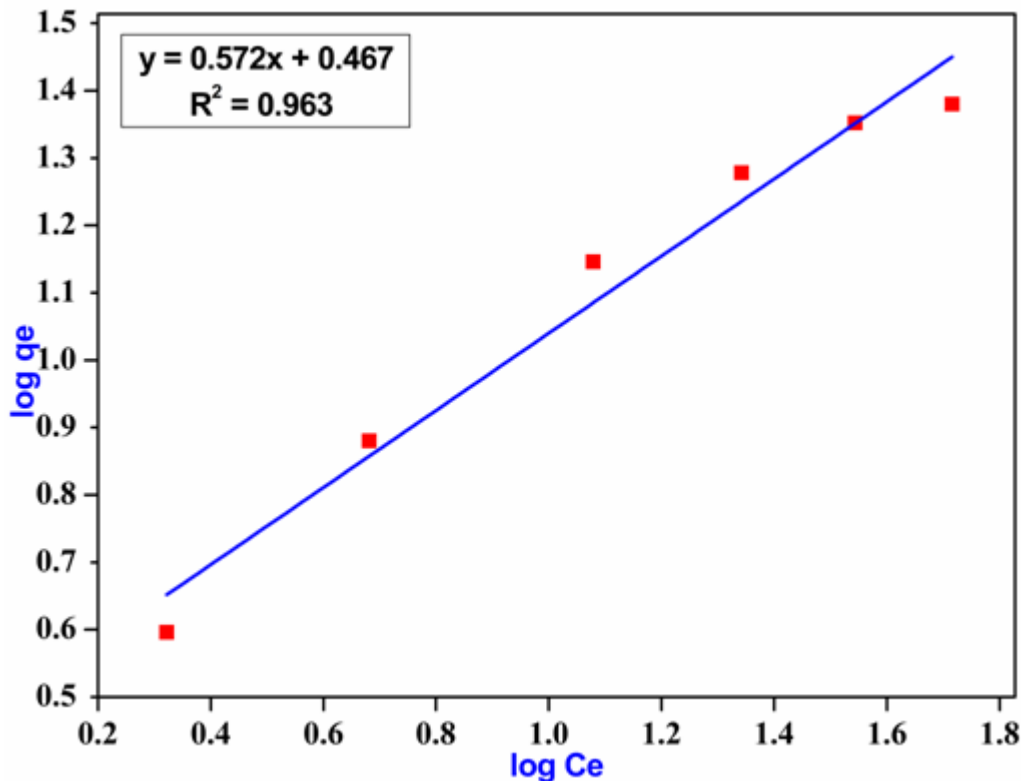


Fig. 7(a): Freundlich isotherm of Ni(II) on BLAC .

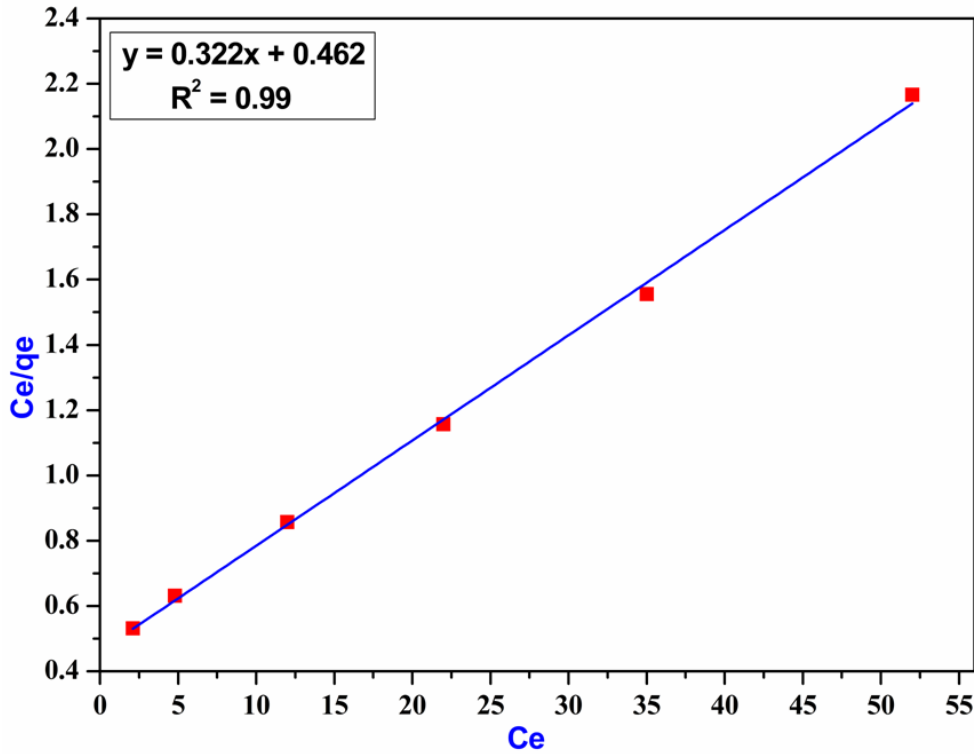


Fig. 7(b): Langmuir isotherm of Ni(II) on BLAC .

Table 1: Adsorption isotherm data of Nickel on BLAC.

	Freundlich Isotherm			Langmuir Isotherm		
	K_f	N	R^2	K_L (l/mg)	q_m (mg/g)	R^2
Ni(II)	2.93	1.748	0.963	0.069	31.05	0.99

Kinetics study

Kinetic study has been carried out using pseudo-first order and pseudo second- order models to investigate the sorption of heavy metals ion from 10 mg/L of metal ion concentration onto BLAC at room temperature (298 K) at different range of contact time. The integral form of the pseudo-first-order kinetic model [31] is generally expressed as follows:

$$\log(q_e - qt) = \log q_e - \frac{K_{1ad}}{2.303} t \tag{5}$$

The pseudo-first-order rate constant K_{1ad} and q_e were obtained from the plot of $\log (q_e - qt)$ vs t [32] (Fig.4.5a) and the assumption of the pseudo-second-order kinetic model is based on the fact that the adsorption follows chemisorption and the linear form of equation is written as:

$$\frac{t}{qt} = \frac{1}{K_{2ad}q_e^2} + \frac{1}{q_e} t \tag{6}$$

Where, K_{2ad} (g/mg min) is the second order rate constant. pseudo-second-order parameters i.e. q_e (mg/g) and K_{2ad} (g/mg min) were obtained from the slope and intercept of linear plots between t/qt vs t (Fig. 8b) [33, 34]. The validity of each model was estimated by comparing the R^2 values. Among the two kinetic models a good agreement of experimental data was observed with Pseudo second-order kinetic model with higher correlation coefficient as compared to that of pseudo first order kinetic model. Hence, the adsorption process follows the pseudo-second order kinetic model which suggests that chemisorption is the rate limiting step [35]. The values of pseudo first and second order kinetic parameters are summarized in the Table 2.

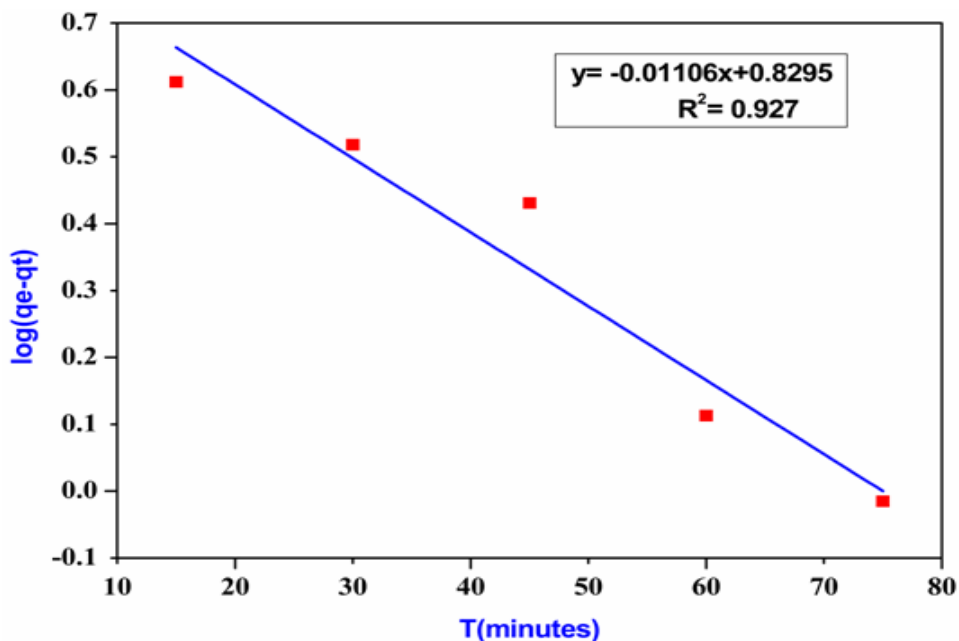


Fig. 8(a): Pseudo first order kinetics of Ni(II) on BLAC.

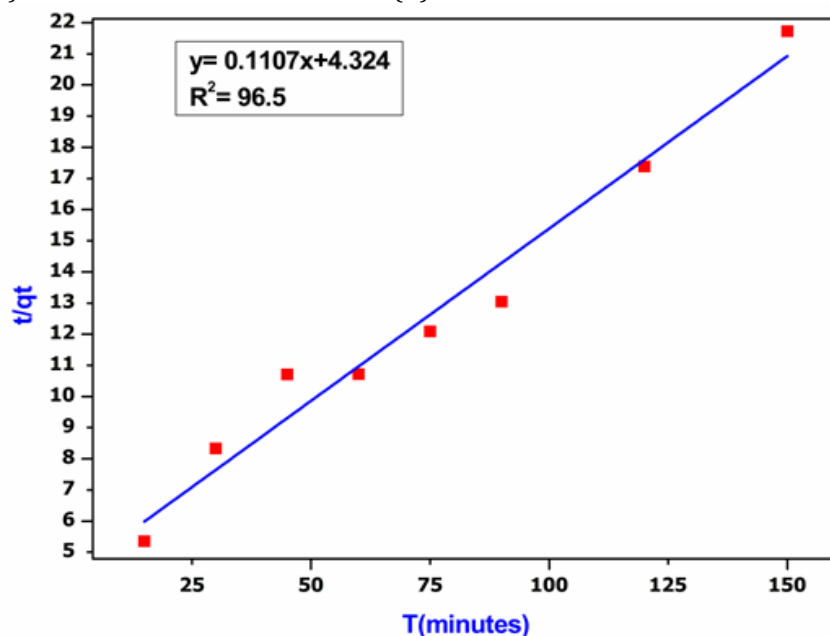


Fig. 8(b): Pseudo second order kinetics of Ni(II) on BLAC.

Table 2. Kinetic study parameters of Nickel adsorption onto BLAC.

	Pseudo first order reaction				Pseudo second order reaction		
	$q_{e\ exp.}$ (mg/g)	K_1 (g/mg min)	$q_{e\ cal.}$ (mg/g)	R^2	K_2 (g/mg min)	$q_{e\ cal.}$ (mg/g)	R^2
Ni(II)	6.9	2.5×10^{-2}	6.75	0.92	2.83×10^{-3}	9.03	0.965

Thermodynamic Study

Thermodynamic study and the analysis of its parameters are performed to get the critical information of adsorption of adsorbate onto the surface of adsorbent. Thermodynamic parameters were calculated from following equations [30]

$$\Delta G^{\circ} = -RT \ln K_d \quad (7)$$

$$\Delta G^{\circ} = \Delta H^{\circ} - T\Delta S^{\circ} \quad (8)$$

$$\ln K_d = \frac{\Delta S^{\circ}}{R} + \frac{\Delta H^{\circ}}{RT} \quad (9)$$

Where K_d is the equilibrium/distribution constant, R is the universal gas constant (8.314 kJ/mol K) and T is the absolute temperature (K) [36]. The values of ΔH° and ΔS° were calculated from the slope and the intercept from the plot of $\ln K_d$ vs $1/T$, respectively, (Fig. 9) and the calculated values are mentioned in the table 3. The negative values of Gibbs free energy (ΔG°) indicates feasibility and the spontaneous nature of metal adsorption on BLAC [35] and positive value of ΔH° indicates the endothermic nature of the adsorption of metal ions onto BLAC [37]. The positive value of ΔS° indicates increased randomness and the affinity of BLAC for metal ions at the adsorbent/solution interface during the adsorption of metal ions onto BLAC [38]. Further, positive value of ΔS° also contributes the adsorption process to become spontaneous and suggests that the adsorption is irreversible which is the characteristic of chemisorption [39].

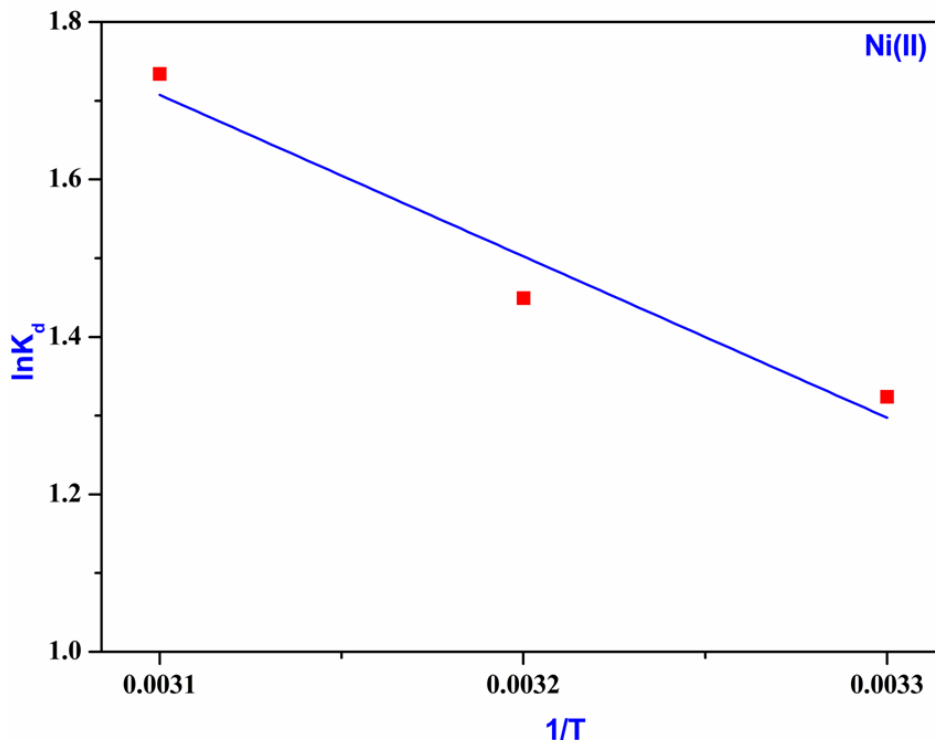


Fig. 9: Thermodynamic study of Nickel on BLAC.

Table 3: Thermodynamic parameters of Nickel on BLAC.

	ΔG° KJmol ⁻¹	ΔH° KJmol ⁻¹	ΔS° Jmol ⁻¹ K ⁻¹
Ni(II)	- 3.001	17.04	0.0670
	- 3.289		
	- 3.936		

CONCLUSION

In this study, it was observed that *bamboo leaves* activated carbon (BLAC) prepared by chemical activation method is carbon rich mesoporous material shows good adsorption capacity. The large surface area and the high pore volume and multiple functional groups enhance the adsorption performance of activated carbon. The maximum adsorption of 76% were observed at contact time of 90 minutes, from fixed metal ion concentration of 10mg/L at pH 6 and at the adsorbent dose of 2 g/L. The results showed that the experimental data were well fitted with Langmuir isotherm as compared to that of Freundlich and Temkin isotherm models. The kinetic studies showed that the rate of adsorption is better described by pseudo second-order model which is an indication for chemisorptive rate-limiting reaction. The thermodynamic analysis reflects that system is spontaneous, endothermic in nature. The present study showed that the activated carbon has great ability for the removal of heavy metals from aqueous

solutions and can be used for wastewater treatment because of their high adsorption capacity, easily available and economical.

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