

An Economic Removal of Ni(II) from Aqueous Solutions Using an Indigenous Adsorbent

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Abstract: Carbon slurry, dried in open air, has been used as an adsorbent material for the removal of nickel(II) from aqueous solutions at different experimental conditions in batch adsorption technique. The time required to attain equilibrium was found to be 50 minutes. The effects of initial concentration of Ni(II), particle size of adsorbent, adsorbent dose, temperature and pH on removal of nickel(II) have been studied. The removal was favored at higher pH, the pH value for maximum adsorption being 10.00. Batch adsorption kinetics has been described by the Lagergren equation. Langmuir and Freundlich isotherms were applied and both isotherms were seen to be applicable over the concentration range studied. Thermodynamic parameters indicate the endothermic nature of Ni(II) adsorption on carbon slurry.

Key Words: Adsorption, carbon slurry, isotherms, nickel(II), Thermodynamic parameters.

INTRODUCTION

In present day context water pollution is the biggest hazard for all forms of life. The environmental pollution has been the most remarkable side effect of rapid industrialization and growing population of the world. Today, almost all the things, which are most necessary to the human beings are more or less contaminated by toxic substances. Industries discharge waste water containing various pollutants such as heavy metals organic and inorganic impurities including colouring materials etc. depending on type of industries, raw materials and processing conditions used. The major Indian rivers are polluted due to the input of various types of wastes from different sources. Thus, it is a compelling necessity for the environmentalists to develop appropriate way to check the deterioration of the environment.

Nickel is 24th most abundant metal in earths crust. Its ores are basically of two types viz. sulphides and oxides. It is used in electroplating, and in a number of alloys and it is a well known catalyst also. Nickel is recognized as a micronutrient for micro-organisms and animals but not to plants. It is associated with the synthesis of vitamin B₁₂ but has been reported to be toxic at higher concentrations. In animals the toxic effect of Ni includes dermatitis "nickel-itch" and causes respiratory disorders. Out of all the compounds of nickel, nickel carbonyl is most toxic. It is a carcinogen and a half an hour are to 30 ppm of this compound is lethal to humans. Nickel(II) is present in the effluents of silver refineries, electroplating, zinc based casting and storage battery industries [1]. In India the acceptable limit of nickel in drinking water is 0.01 mg dm⁻³ and for discharge of industrial wastewater [2] it is 2.0 mg dm⁻³. At higher concentrations,

nickel(II) causes cancer of lungs, nose and bone. Dermatitis (nickel itch) is the most frequent effect of exposure to nickel objects, such as coins and costume jewelry.

The effective technologies available for the removal of metallic pollutants from water and wastewater include chemical precipitation, ion exchange, solvent extraction, reverse osmosis and adsorption [3]. Adsorption has been a very effective and economical method for removal, recovery and recycling of metals from wastewater [4]. Different conventional and non-conventional adsorbents such as red mud, coconut coirpith, sewage sludge, silica, tree fern, bone char, polymetallic sea nodule, fly ash, maple saw dust, activated carbon, modified zeolites and parthenium plant [5-20] etc. have been cited in the literature as adsorbents by several workers.

The present communication has been addressed to the removal of nickel from aqueous solutions by using carbon slurry. Batch adsorption technique has been adopted in removal and the effect of various important parameters viz. contact time and initial concentration, pH, adsorbent particle size, and temperature on removal of nickel has been discussed. The dynamics of adsorption and isotherm has also been reported. Thermodynamic parameters free energy, enthalpy, and entropy change have also been studied.

MATERIALS AND METHOD

Materials

Carbon slurry was collected from the Fertilizer Corporation of India, Gorakhpur, U.P., India and was used as an adsorbent without any pretreatment after passing through sieves to maintain desired particle sizes. Carbon slurry is generated from the scrubbers of the gasification section of ammonia plant operating on oxidation of naphtha. The particle size was measured by B.S.S. sieve size. The bulk density was measured using a specific gravity bottle at room tem-

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Table 1. Analysis of Carbon

Moisture (%)	17.37
Volatile matter (%)	24.90
Ash content (%)	2.56
Silica (%)	2.48
Bulk density (g cm^{-3})	0.238

perature. The physico-chemical characterization of carbon is shown in Tables 1-3. All the chemicals used, were of analytical reagent grade. The solutions were prepared using deionised water. Standard hydrochloric acid and sodium hydroxide solutions were used for adjusting pH of the solutions.

Method

Batch adsorption experiments were carried out by shaking 1.0 g of desired grade carbon with 50 ml of nickel solution of known concentration in a 250 ml conical flask placed in a thermostat provided with a shaking machine, at different temperatures, pH and at an agitation speed of 125 rpm. The flasks were removed after the desired contact time and the filtrates were analyzed for the remaining nickel (II) concentration. A systronics 166, Sr. No. 984 (Systronics, Naroda, Ahmedabad, India) spectrophotometer was employed. Dimethylglyoxime procedure [21] was used for the analysis.

RESULTS AND DISCUSSION

Characterization of the Adsorbent

The characterization of the adsorbent was carried out and has been presented in Tables 1-3. Table 1 shows analysis of carbon and it can be seen from this table that its ash content is 2.56% only. Other parameters have also been reported in this table. IR spectra of the adsorbent was also carried out and this provides important information regarding the adsorbent. Band positions show that it has C=C-H stretching, C=O stretching, C=C stretching, Si=O stretching and aromatic ring and C-H deforming. d values at 3.35, 2.08, 2.03, 1.54 and 1.22 Å indicate the presence of carbon(hexagonal system) and that present at 3.34, 4.26 and 1.82 Å show presence of silica(quartz low) in the adsorbent. The characterization of the adsorbent shows that these species would be responsible for removal of Ni(II) from aqueous solutions.

Effect of Contact Time and Initial Concentration

The transfer of Ni(II) by adsorption on carbon increases with time and attains a maximum value at 50 min and thereafter remains constant. On changing the initial concentration of nickel sulphate solution from 22.0 mg l^{-1} to 278.00 mg l^{-1} (Fig. 1), whereas the amount adsorbed increases from 1.0319 mg g^{-1} (93.81% removal) to 6.6498 mg g^{-1} (47.84% removal) at $25 \pm 0.5^\circ\text{C}$, pH 10.00, particle size 150 B.S.S. It is clear from Fig. (1) that with a lower initial concentration of adsorbate the amount of adsorbate attained on the solid phase is smaller than the amount attained on adsorbent when higher initial adsorbate concentrations are used. Although the percent removal was greater with lower initial concentration than with higher initial concentrations. The equilibrium was

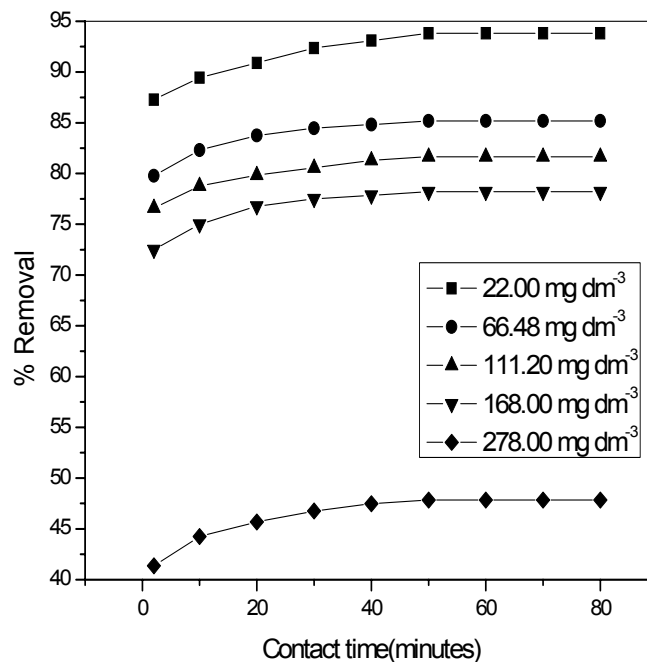


Fig. (1). Time variation of adsorption of Nickel(II) on carbon at different initial concentrations. [pH :10.00, particle size : 150 B.S.S., temperature: $25 \pm 0.5^\circ\text{C}$, agitation speed : 125 rpm].

established in 50 minutes and was independent of Ni(II) concentration in water. Similar results have been reported by other workers for other adsorbate-adsorbent system [22-24]. When the adsorbent dose was increased from 4 g l^{-1} to 36 g l^{-1} , complete removal of Ni(II) took place at initial concentration of 22.00 mg l^{-1} . The curves of Fig. (1) are clear, continuous, single and smooth indicating formation of a monolayer of adsorbate on the adsorbent surface. The higher % removal of nickel in lower concentration ranges has industrial application as in most of the effluents the lower concentration of nickel is encountered.

Effect of pH

The uptake of Ni(II) by carbon as a function of pH at initial concentration of 22.00 mg l^{-1} , 66.48 mg l^{-1} , 111.20 mg l^{-1} , 168.00 mg l^{-1} and 278.00 mg l^{-1} at temperature $25 \pm 0.5^\circ\text{C}$, contact time 50 min, adsorbent particle size 150 B.S.S. and agitation speed of 125 rpm is shown in Fig. (2). The pH range studied in each case was from 2.00 to 11.00. It is clear from the figure that for each initial concentration, value of Ni(II) the percent removal increases as pH rises and after pH 10.00, the percent removal decreases with further increase in pH value. With initial concentration of 22.00 mg l^{-1} , percent removal at pH 2.00 is 0.0999 mg g^{-1} (9.09% removal), at pH 10.00 it is 1.0319 mg g^{-1} (93.81% removal) and at pH 11.00 it is 1.0280 mg g^{-1} (93.45% removal). Similar with initial concentration of 278.00 mg l^{-1} , the percent removal at pH 2.00 is 0.6491 mg g^{-1} (4.67% removal), at pH 10.00 it is 6.6498 mg g^{-1} (47.84% removal) and at pH 11.00 percent removal again decreases upto 6.0993 mg g^{-1} (43.88% removal). This clearly shows that the optimum pH for the present adsorbate-adsorbent system is around 10.00. Analysis of the filtrate made it clear that at pH 10.0 it did not contain any precipitated form of nickel. Almost similar trends were observed for other values of initial concentration.

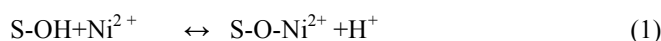
Table 2. Important Infra-red Bands of Carbon Along with their Probable Assignments

Band Position (cm ⁻¹)	Assignment
2924.35, 2849.12	C=C-H stretching
1749.59	C=O stretching
1647.36	C=C stretching
1558.62, 1591.26, 1521.97, 1508.49	Aromatic ring
1419.74, 1396.59	C-H deforming
1159.32, 1122.67, 773.52, 518.90, 418.59	Si-O stretching

Table 3. 'd' Values of Carbon (X-ray Diffraction Pattern)

d (Å°)	Possible Components
3.35	Carbon (hexagonal system)
2.08	Carbon (hexagonal system)
2.03	Carbon (hexagonal system)
1.54	Carbon (hexagonal system)
1.22	Carbon (hexagonal system)
3.34	Silica (quartz low)
4.26	Silica (quartz low)
1.82	Silica (quartz low)

It is clear from experimental data that removal increases slowly and then acquires a maxima in 120 min. Uptake of nickel is rapid in initial stages and in later stages it becomes steady. It is expected that the metal competes with protons for surface sites:



Where 'S' stands for surface. In equation it seems that during adsorption one proton is replaced by one nickel atom. Some amount of surface precipitation at the maximum removal of nickel at around 8.0 can also not be ruled out. At pH > 8.0, the Ni(II) ions get precipitated due to hydroxide anions forming a nickel hydroxide precipitate [21].

Effect of Particle Size

The removal of Ni(II) was found to increase from 2.7437 mg g⁻¹ (82.54% removal) to 2.9128 mg g⁻¹ (87.63% removal) at pH 10.00, temperature 25 ± 0.5°C, initial adsorbate concentration 66.48 mg l⁻¹ and agitation speed of 125 rpm as the adsorbent particle size was decreased from 50 B.S.S. to 300 B.S.S. (Fig. 3). B.S.S. stands for British Sieve Standards. The higher adsorption with smaller adsorbate particles may be attributed to the fact that smaller particles provide a larger surface area. The equilibrium time was found to remain unaffected by the change in adsorbent particle size. The other textural characteristics of the adsorbent have been presented elsewhere [22].

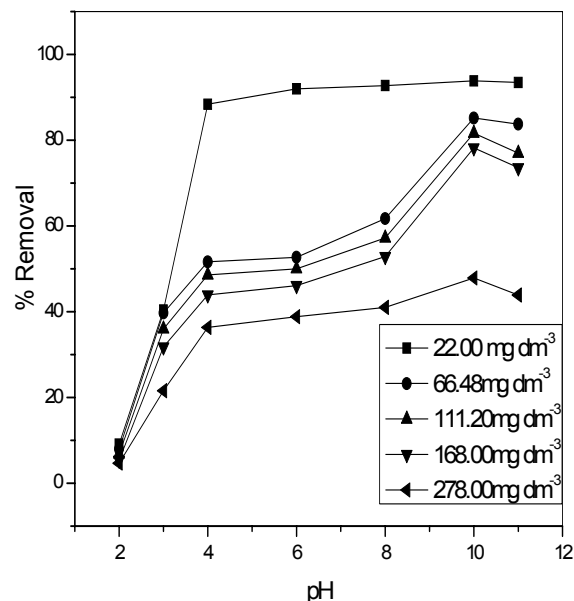


Fig. (2). Equilibrium adsorption of Nickel(II) on carbon at different pH values. [particle size : 150 B.S.S., temperature: 25 ± 0.5°C, agitation speed : 125 rpm, contact time : 90 minutes].

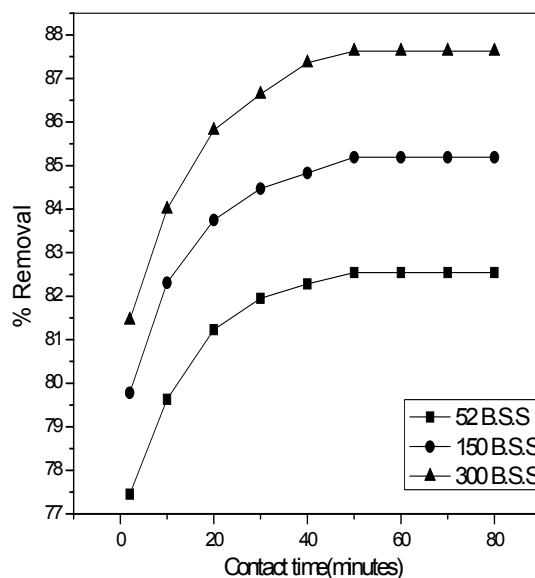


Fig. (3). Time variation of adsorption of Nickel(II) on carbon at different particle sizes. [initial concentration : 66.48 mg dm⁻³, pH: 10.00, temperature: 25 ± 0.5°C, agitation speed : 125 rpm].

Effect of Temperature

The adsorption of Ni(II) was found to increase from 6.5697 mg g⁻¹ (78.21% removal) to 7.1098 mg g⁻¹ (84.64% removal) as the temperature increased from 25 ± 0.5 °C to 75 ± 0.5°C at pH 10.00, initial adsorbate concentration 168.00 mg dm⁻³, particle size 150 B.S.S., agitation speed 125 rpm (Fig. 4). Increase in percent removal of Ni(II) with increase in temperature indicates the process to be endothermic.

Adsorption Dynamics

Adsorption dynamics gives idea about solute uptake rate which controls the residence time of solute at the solid-solution interface. Thus, study of adsorption dynamics be-

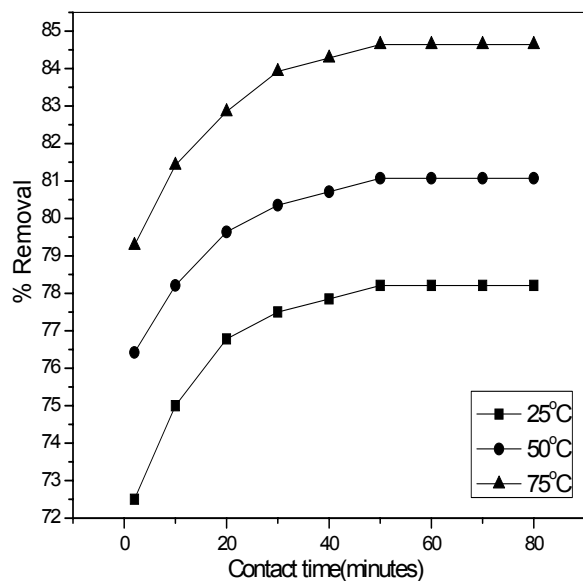


Fig. (4). Time variation of adsorption of Nickel(II) on carbon at different temperatures. [initial concentration : 168.00 mg dm⁻³, pH :10.00, particle size : 150 B.S.S., agitation speed : 125 rpm].

comes quite significant in waste water treatment. This study was done by conducting the batch adsorption of Ni(II) on carbon in a shaking water bath operated at a fixed agitation speed with different particle sizes of adsorbent, at desired temperatures as given in experimental method. A kinetic model, developed by using the Lagergren equation [25], was used which is as follows:

$$\log(q_e - q) = \log q_e - \frac{k't}{2.303} \tag{1}$$

where q_e is the amount of solute adsorbed at equilibrium per unit weight of adsorbent (mg g⁻¹), q the amount of solute adsorbed at any time t (mg g⁻¹), and k' the adsorption constant. The plots of $\log(q_e - q)$ versus t (Fig. 5) at different concentrations of Ni(II) viz. 22.00, 66.48, 111.20, 168.00 and 278.00 mg l⁻¹ are linear at 25 ± 0.5°C and pH 10.00, agitation speed of 125 rpm. A straight line is obtained for each value of initial concentration, indicating that the adsorption follows the above equation used for kinetic modelling. The slope of this plot gives the value of the adsorption constant k' . The values of k' for all the initial concentration values are similar i.e. independent initial concentration of adsorbate indicating that the system follows first order rate kinetics (Table 4).

Adsorption Isotherms

The equilibrium data for the adsorption of Ni(II) on to carbon at various temperatures and at pH 10.00 follows the rearranged Langmuir equation [26]:

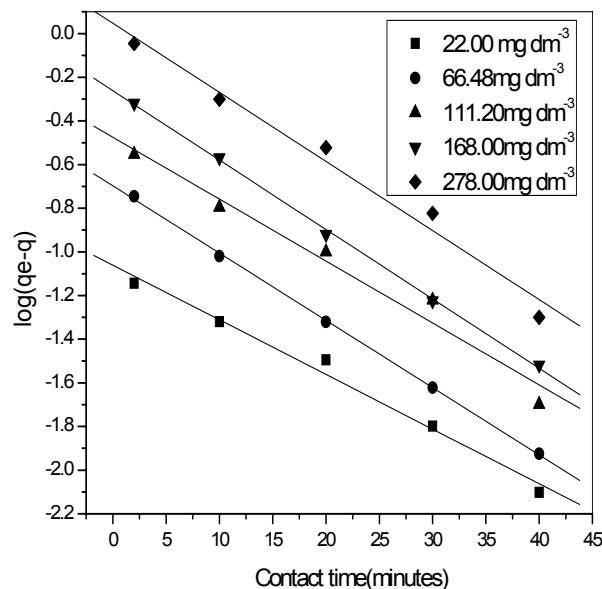


Fig. (5). Rate constant plot for adsorption of Nickel(II) on carbon at different initial concentrations. [pH :10.00, particle size: 150 B.S.S., temperature: 25 ± 0.5°C, agitation speed : 125 rpm].

$$\frac{C_e}{q_e} = \frac{1}{Q^0 b} + \frac{C_e}{Q^0} \tag{2}$$

where C_e is the equilibrium concentration (mg l⁻¹) and q_e the amount adsorbed at equilibrium (mg g⁻¹). The values of Langmuir constants Q^0 and b , which are related to adsorption capacity and apparent heat change respectively, were determined at different temperatures (Table 5) from the slopes and intercepts of the linear plot of C_e/q_e versus C_e (Fig. 6). Further, the plots of figure 6 are single and smooth. This nature of the plots indicates fitness of Langmuir’s model for the present system.

As figure 6 indicates, for given values of initial concentration, pH and particle size of adsorbent, the equilibrium concentration C_e , of adsorbate in solution decreases with increasing temperatures, i.e., the adsorption is favoured at higher temperatures. This is supported from the value of adsorption capacity and apparent heat change Langmuir constants (Q^0 and b) which increase with increasing temperature (Table 5). The applicability of the Langmuir isotherm in the present system indicates the monolayer coverage of Ni(II) on the outer surface of the carbon.

The equilibrium data for the present adsorbate-adsorbent system were analysed using Freundlich adsorption isotherm also. The general form of the Freundlich equation is as follows [27]:

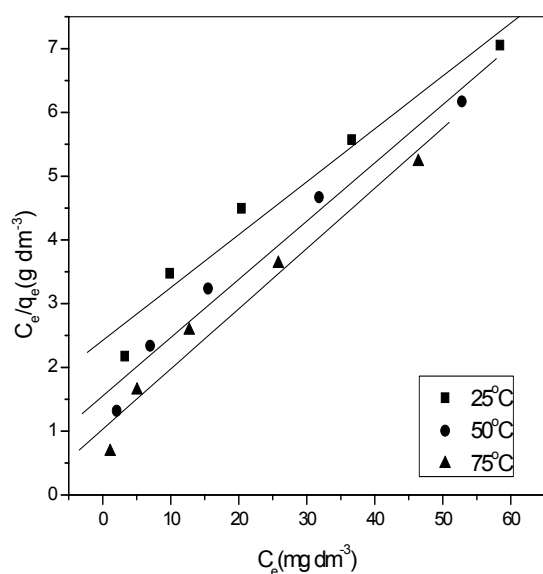
$$q_e = K_F C_e^{1/n} \tag{3}$$

Table 4. Rate Constant at Different Initial Concentrations (pH : 10.0, Temperature : 25±0.5°C, Particle size : 150 B.S.S.)

Concentration (mg dm ⁻³)	22.00	66.48	111.20	168.00	278.00
Rate constant (min ⁻¹) × 10 ⁻²	5.83	7.32	6.70	7.30	7.53

Table 5. Various Constants at Different Temperatures (pH : 10.00, Particle Size: 150 B.S.S., Contact Time: 90 minutes, Agitation speed: 125 rpm)

Parameters	Values at Different Temperature		
	25 ± 0.5°C	50 ± 0.5°C	75 ± 0.5°C
Langmuir constants			
b	0.0372	0.0576	0.0855
Q ⁰	11.2108	11.2486	11.3766
Freundlich constants			
K _F	0.0174	0.0467	0.0831
n	1.6889	2.2401	2.7488
K _c (dm ³ g ⁻¹)	0.4737	0.7576	1.4749
ΔG (kJ mol ⁻¹)	0.4425	0.1782	-0.2688

**Fig. (6).** Langmuir isotherm for the adsorption of Nickel(II) on carbon at different temperatures. [pH :10.00, particle size : 150 B.S.S., agitation speed : 125 rpm, contact time : 90 minutes].

where K_F and n are constants.

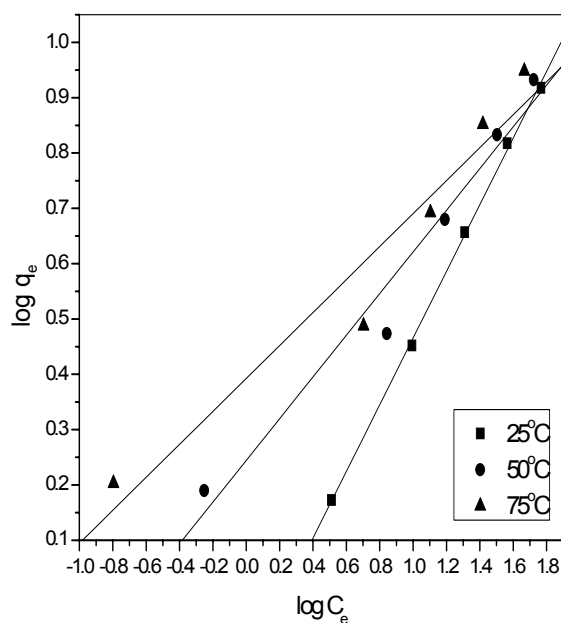
The logarithmic form of the equation is

$$\log q_e = \log K_F + 1/n \log C_e \quad (4)$$

The plot of $\log q_e$ versus $\log C_e$ gives a straight line, whose slope (equal to $1/n$) is roughly an indicator of sorption intensity and the intercept (equal to $\log K_F$) is indicator of sorption capacity. The straight line obtained (Fig. 7) indicate applicability of this isotherm as well for the concentration range studied. Values of K_F and n (Table 5) also indicate that the amount of adsorbate adsorbed per gram of adsorbent increases solution temperature which, further, proves the endothermic nature of the adsorption in the present adsorbate-adsorbent system.

Thermodynamic Parameters

Thermodynamic parameters e.g. ΔG , the free energy change and ΔH , the enthalpy change were calculated from the following equations [28]:

**Fig. (7).** Freundlich isotherm for the adsorption of Nickel(II) on carbon at different temperatures. [pH :10.00, particle size : 150 B.S.S., agitation speed : 125 rpm, contact time : 90 minutes].

$$\Delta G = -RT \ln K_c \quad (5)$$

$$\text{and } b = b'e^{-\Delta H/RT} \quad (6)$$

where K_c is equilibrium constant and can be given by

$$K_c = \frac{C_{Be}}{C_{Ac}}$$

C_{Be} is the equilibrium concentration of Ni(II) on the adsorbent (mg g^{-1}),

C_{Ac} the equilibrium concentration of Ni(II) in solution (mg l^{-1}).

T is absolute temperature and R is gas constant, b is Langmuir constant while b' is another constant.

Equation (6) can also be written as

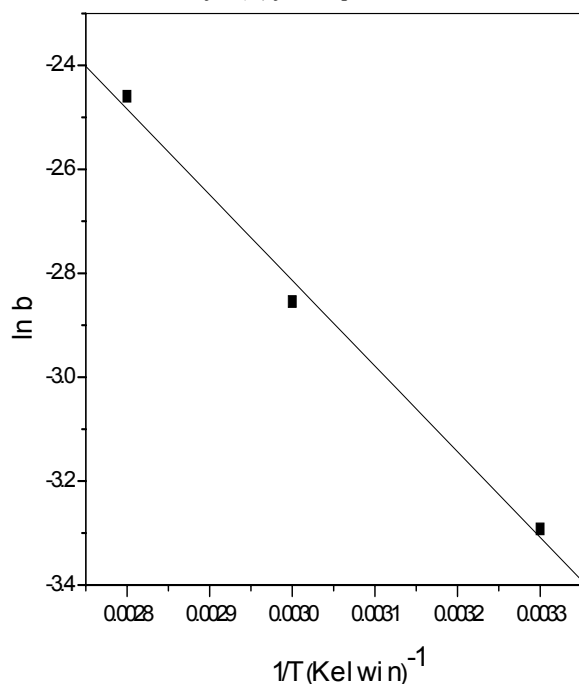


Fig. (8). Plot of $\ln b$ versus $1/T$ for enthalpy change for the adsorption of Nickel(II) on carbon at different temperatures. [pH :10.00, particle size: 150 B.S.S., agitation speed : 125 rpm, contact time : 90 minutes].

$$\ln b = \ln b' - \frac{\Delta H}{RT} \quad (7)$$

Value of ΔH was calculated from the slope of straight line obtained from the plot between $\ln b$ versus $1/T$ (Fig. 8) and it was found to be $3.3839 \text{ KJ mol}^{-1}$. Positive value of ΔH indicates that the process is endothermic. The value of ΔG has been found to decrease with rise in temperature (Table 5) and it becomes negative at $75 \pm 0.5^\circ \text{C}$ which is indicative of the fact that the adsorption process becomes more spontaneous higher temperature. Both the thermodynamic parameters support the endothermic nature of the adsorption process in the present case of study.

CONCLUSION

Carbon slurry, the waste product of naphtha based fertilizer plants, can be used as an effective adsorbent material for bringing the Ni(II) concentration in waste water to acceptable limits by maintaining the optimum conditions of pH, temperature and adsorbent dose. The data obtained in the study, can safely be used by environmentalists to devise pilot plants for abatement of Ni(II) concentration from waste waters. Using carbon slurry the adsorbent material.

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