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Studies on the adsorption of Nickel (II) ion from aqueous solution onto H_2SO_4 activated carbon prepared from the wood of *Adina cordifolia* Hook

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Abstract

An adsorption of Ni(II) ions from aqueous solution onto carbon prepared from the wood of *Adina Cordifolia* Hook activated with Conc. H_2SO_4 (ACHC).Batch mode adsorption experiments were conducted. The characteristics of the ACHC were determined by SEM, XRD, FTIR, BET analysis and pH_{ZPC}. The maximum removal efficiency of Ni(II) ion was 65.50% at pH 7. Under the chosen conditions, Experimental data obtained were fitted with linearised forms of Legergren and Ho kinetic models. The Sum of Error Squares Percentage (SSE%) for first order and second order kinetics were 1.70 & 0.17 respectively. Thus this adsorption followed second order kinetics. The equilibrium adsorption data were analyzed with four isotherm models. Order of best fitting isotherm models were found to be Freundlich>Tempkin>Dubinin Raduskevich>Langmuir. Thermodynamic parameters for such as ΔH, ΔS and ΔG were determined using Van't Hoff plots. Analysis of these values inferred that the adsorption was endothermic, spontaneous with increased randomness. The FTIR, XRD, EDX and SEM studies confirmed that adsorption was physisorption with partial chemisorption. The results in this study indicated that ACHC could be employed as an adsorbent for the removal of Ni (II) ions from an aqueous solution.

Keywords: Adsorption; *Adina Cordifolia* Hook activated carbon (ACHC); Ni(II)ion removal; Kinetics models; Isotherm studies, Thermodynamic studies; Instrumental studies.

1.0. Introduction

Heavy metals like lead, cadmium, nickel, chromium etc even at lower concentrations are hazardous to the environment due to toxicity and non–biodegradability [1]. Among heavy metals, nickel is one of the most utilized in the manufacturing process of stainless steel, super alloys, metallic alloys, coins, batteries etc. Direct exposition to nickel causes dermatitis. Some nickel compounds, as carbonyl, are carcinogenic and easily absorbed by skin [2]. Acute poisoning of Ni (II) causes headache, dizziness, nausea and vomiting, chest pain, tightness of the chest, dry cough and shortness of breath, rapid respiration, cyanosis and extreme weakness [3]. The removal of such a heavy metal from contaminated water bodies has been attempted by several scientists employing a wide variety of techniques including chemical precipitation, chemical oxidation or reduction, filtration, ion exchange, electrochemical treatment, membrane filtration, reverse osmosis and adsorption. However, most of these methods have considerable disadvantages including incomplete metal removal, high capital, operational cost and the disposal of residual metal sludge which are not suitable for small scale industries [4].

Among several chemical and physical methods, the adsorption onto activated carbon has been found to be superior to other techniques because of its capability of adsorbing a broad range of different types of adsorbates efficiently and its simplicity of design[5]. However, commercially available activated carbons are still considered expensive[6]. As a result, many researchers have studied cheaper substitutes, which are relatively inexpensive and at the same time endowed with reasonable adsorption capacity. These studies include the use of coal[7], fly ash[8-9], activated clay[10], palm fruit bunch[11], rice husk[12], hazelnut shell[13], coconut shell[14] and peat[15-16].

The objective of this study is to evaluate the feasibility of using the adsorbent prepared from *Adina Cordifolia* Hookfor the removal of Ni(II) ion from aqueous solution.

Table:1.0 Nomenclature

Nomenclature	
C_i	Liquid phase initial concentrations of the adsorbate (mg/L)
C_t	Liquid phase concentrations of the adsorbate at time “t” (mg/L)
C_e	Liquid phase concentrations of the adsorbate at equilibrium (mg/L)
V	Volume of the adsorbate solutions in liter (L)
W	Mass of the adsorbent (g)
q_e	Quantity adsorbed at equilibrium (mg/g)
q_t	Quantity adsorbed at time “t” (mg/g)
T	Time in minutes
Q_e	Amount of solute adsorbed per unit weight of adsorbent (mg/g)
C_e	Equilibrium concentration of solute in the bulk solution (mg/L)
Q_0	Langmuir adsorption efficiency
B	Langmuiradsorption energy
R_L	Langmuirseparation factor
C_0	Initial concentration of the adsorbate
K_f and n	Freundlich constants incorporating all factors affecting the adsorption capacity and intensity of adsorption respectively
B_1	Tempkinconstant related to heat of sorption (J/mol)
K_T	Tempkin equilibrium binding constant
q_D	D-R isothermtheoretical saturation capacity (mg/g)
B	D-R isothermconstant related to the mean free energy
E	D-R isotherm polanyi potential
E	D-R isothermmean free energy of adsorption
R	Gas Constant
T	Temperature (K)
k_1	Rate constant of adsorption (l/min)
k_2	Second-order constants
h	Initial adsorption rate (mg/g min)
N	Number of data points

2.0. Materials and Methods

2.1 Materials:

All the chemicals used for these experiments are analytical grade. Activated Carbon was prepared from *Adina Cordifolia* Hook.$(NH_4)_2$ Ni $(SO_4)_2$.$6H_2O$, complexing reagent used as DMG purchased from Merck company.

2.2 Preparation of Stock solution:

Ni(II) ion was determined spectrophotometrically as Ni-DMG complex. To about 10 ml of the filtered sample solution containing Ni(II) ions, 0.5ml of 0.5N HCl,0.2 mL of 0.25N Sodium citrate, one drop of 0.05N iodine and 0.8ml of 0.5% DMG were added. The absorbance of rosy red Ni-DMG complex was measured at 445 nm.

2.3 Preparation of Adsorbent:

The activated carbon was prepared from the wood of *Adina Cordifolia Hook*. The wood were cut into smaller pieces and soaked in concentrated H_2SO_4 at 1:1 ratio (weight of raw material/volume of acid) for 24 hours and activated at 160°C for 5 hours. The activated carbon was repeatedly washed with distilled water until the pH of the wash water became the pH of the distilled water (nearly 7). The carbon obtained was dried at 110°C for nearly 2 hours to remove the moisture.

The above prepared carbon was named as ACHC. The prepared carbon was kept in a desiccator for further use.

2.4 Physico - chemical characteristics of Adsorbent:

The activated carbon prepared from *Adina Cordifolia* Hook was designated as *Adina Cordifolia* Hook Carbon (ACHC). The various characteristics of the ACHC determined were collected in the Table: 2.0

Table: 2.0 Physico - chemical characteristics of ACHC

S.No.	Properties	Values
1.	Surface area (BET), m^2/g	356.45
2.	Bulk density, g/mL	0.3841
3.	Particle size (mm)	0.106 -0.212
4.	Moisture content (%)	4,23
5.	pH_{zpc}	6.78
6.	Pore volume, cm^3/g	0.388
7.	Fixed Carbon, %	73.35
8.	Pore size (Pore width), nm	2.363

2.5 Batch Adsorption procedure:

Batch adsorption studies were carried out in 250 mL iodine flasks with 50 mL of the Ni(II) ion solution of different concentrations ranging from 10 mg/L to 14 mg/L. Known amount of adsorbent was added to the solution. The flasks were agitated at a constant speed of 180 rpm. Samples were collected from the flasks at predetermined time intervals for analyzing the residual Ni(II) ion concentration in the solution. The amount of Ni(II) ions adsorbed in milligram per gram of adsorbent was determined by using the following mass balance equation:

$$q_e = (C_i - C_e)V/W$$

Where C_i and C_e are Ni(II) ion concentrations (mg/L) before and after adsorption, respectively, V (L) is the volume of adsorbate in litre and W (g) is the weight of the adsorbent in grams. The percentage of removal of Ni(II) ions was calculated using the following equation:

$$\text{Removal (\%)} = (C_i - C_e)/C_i \times 100$$

3.0. Results and Discussion

3.1 Effect of Contact time and initial concentration:

The effect of contact time on percentage removal of Ni(II) ion for different initial concentration have been shown in figure 1. Adsorption of Ni(II) ion from the solution increases with the time and finally attains equilibrium at 100,100 and 80 minutes for the initial concentrations 10,12 and 14 mg/L respectively. The percentage of removal increased with the increase in contact time and decreased with the increase of initial concentration of the Ni(II) ion. However the amount of metal ion adsorbed on the adsorbent increased with the increase of initial concentration of the Ni(II) ion solution [17]. which is depicted in figure 1 and given in table 4.

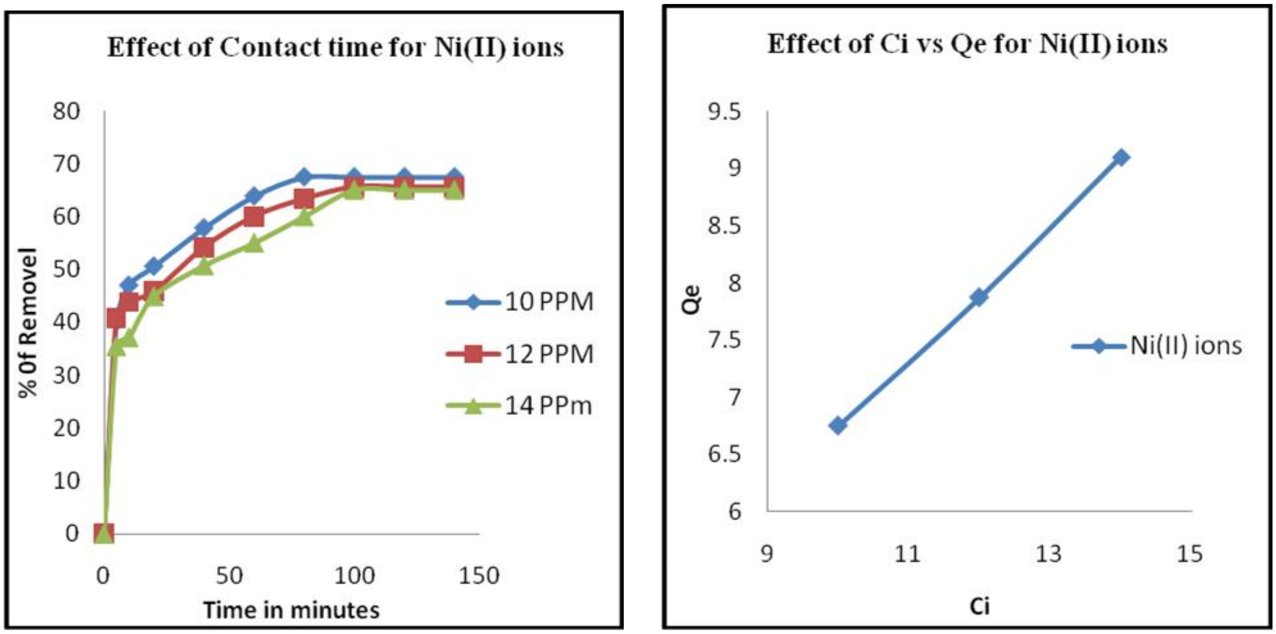


Figure: 1 Effect of Contact time and Ci vs Qe for Ni(II) ion onto ACHC

Table:4 Percentage of removal of metal ion and amount of metal ion adsorbed

C_i (mg/L)	% of Removal of metal ion at equilibrium	Adsorbed amount of Ni(II)ion at equilibrium (mg/g)
10	67.47	6.75
12	65.58	7.87
14	65.00	9.10

3.2 Effect of pH:

It is well known fact that the adsorption of heavy metal ions depends on the pH of the aqueous solution. In this experiment, the pH effect was studied in the range of 1-7. Solution pH beyond 7 was not studied as the heavy metal ions get precipitated at alkaline pH.The effect of pH on the removal of chosen metal ions onto ACHC from aqueous solutions under desired experimental conditions[18].

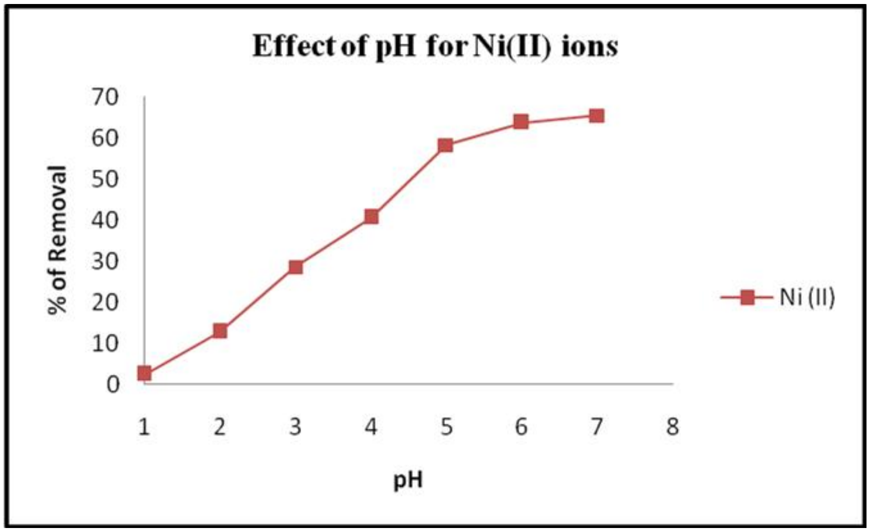


Figure: 2 Effect of pH for Ni(II) ion onto ACHC

3.3 Effect of Temperature:

It is well known that temperature plays an important role in the adsorption process. The metal ions removal increase rapidly from 305K,315K and 325K this result suggests that the experimental temperature had a greater effect on the adsorption process implying that the surface coverage increased at higher temperatures. This may be attributed to the increased penetration of metal ions inside micro pores or the creation of new active sites at higher temperatures. Thisindicates the endothermic nature of the controlled adsorption process. Similar result has been reported in the literature[19].

Table: 3 Data processing Tools

S. No.	Parameters		Formulae
1.	**Kinetic Models & SSE %**	Pseudo First order kinetics (Legergren equation)	log $(q_e - q_t)$ = log q_e - $k_1/2.303 \times t$
		Pseudo Second order kinetics (Ho equation) The initial adsorption rate h	$t/q_t = 1/k_2.q_e^2 + 1/q_e\ t$ $h = k_2 q_e^2$
		Sumoferror squares	SSE (%)= $\sqrt{\sum[(q_e)_{exp} - (q_e)_{cal}]^2 / N}$
2.	**Isotherms**	Langmuir Separation factor	$C_e/Q_e = 1/Q_0 b + C_e/Q_0$ $R_L = 1 / (1 + bC_0)$
		Freundlich	$\log Q_e = \log K_f + 1/n \log C_e$
		Tempkin	$q_e = B_1 \ln K_T + B_1 \ln C_e$
		Dubinin – Raduskevich, Polanyi potential Mean free energy of adsorption	$\ln q_e = \ln q_D - B\varepsilon^2$ $\varepsilon = RT \ln (1 + 1/C_e)$ $E = 1/(2B)^{½}$

4.0 Adsorption Kinetics:

The adsorption kinetics shows the evolution of the adsorption capacity through time and it is necessary to identify the types of adsorption mechanism in a given system. Plots of different kinetic models applied were given in the Figure: 3 and the kinetic parameters calculated were given in the Table 5. Between the first order and second order, second order kinetic model seems to best describe the above adsorption system as its R^2 values were very close to unity. Moreover, difference between qe (cal) and qe (exp) values of second order is small when compared to first order kinetic model. Statistically it is tested with the tool Sum of error squares (SSE%) [20].The Δqe and SSE % values were given in the Table: 5 from which it was concluded that second order kinetic model was more appropriate rather than first order kinetic model.

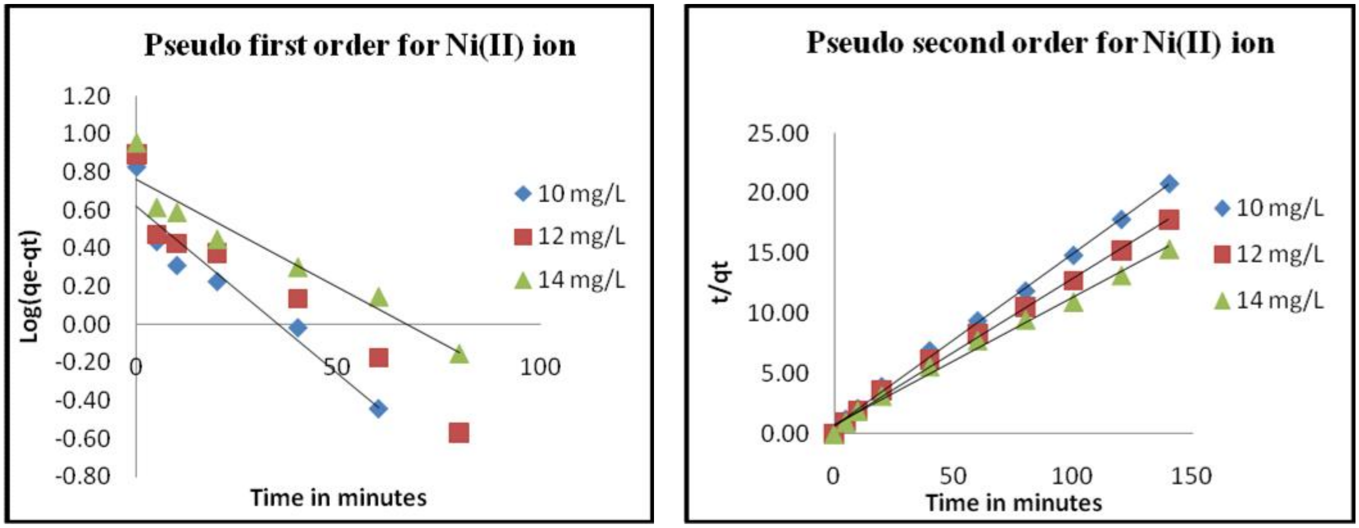


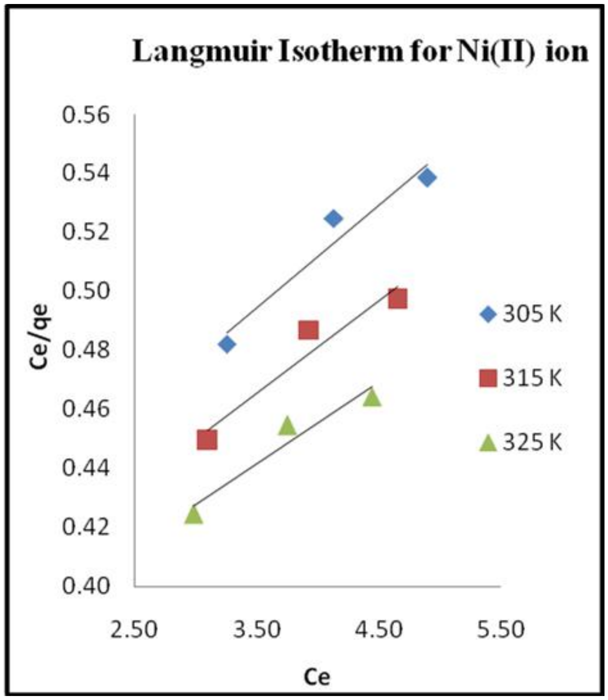
Figure: 3 Pseudo first and second order kinetics

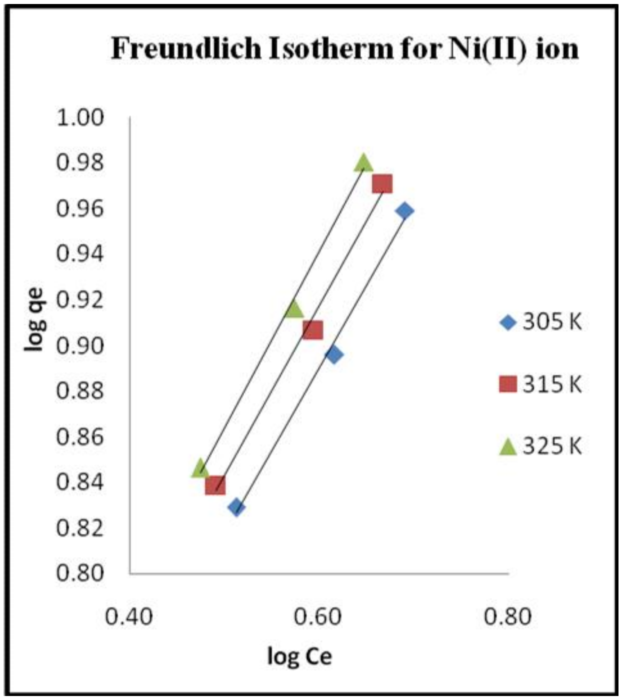
Table:5 Kinetic parameters for Ni(II) ions onto ACHC

Ci mg/L	Rate constants		$q_{e(cal)}$ mg/g		$q_{e(exp)}$ mg/g		Δq_e		R^2		(SSE %)	
	k_1 (10^{-2}) (min^{-1})	k_2 (10^{-3}) $(gmg^{-1}$ $min^{-1})$	First Order	Second order	First order	Second Order	First order	Second Order	First order	Second order	First order	Second Order
10	0.0408	3.20	4.20	6.97	06.75	06.75	2.55	0.22	0.9200	0.9900	1.70	0.17
12	0.0352	2.20	4.93	8.15	07.87	07.87	2.94	0.28	0.9400	0.9900		
14	0.0263	1.40	5.83	9.48	09.10	09.10	3.27	0.38	0.9200	0.9900		

5.0 Adsorption Isotherm Studies:

The existence of equilibrium between the liquid and solid phase is well described by adsorption isotherms. Equilibrium data collected at different temperatures were fitted in Langmuir, Freundlich, Tempkin and Dubinin-Raduskevich adsorption isotherm models [20]. These isotherms are depicted in Figure: 4. The R^2 values of these isotherm plots reveal that Freundlich isotherm well describes the present system that is the possibility of multilayer adsorption. R^2 value of Dubinin-Raduskevich isotherm is very low. In Dubinin-Raduskevich isotherm, the very low value of the constant ‘B’ related to the mean free energy of adsorption per mole of the adsorbate and the adsorption is physical in nature[21]. Results of various isotherms are presented in Table: 6.





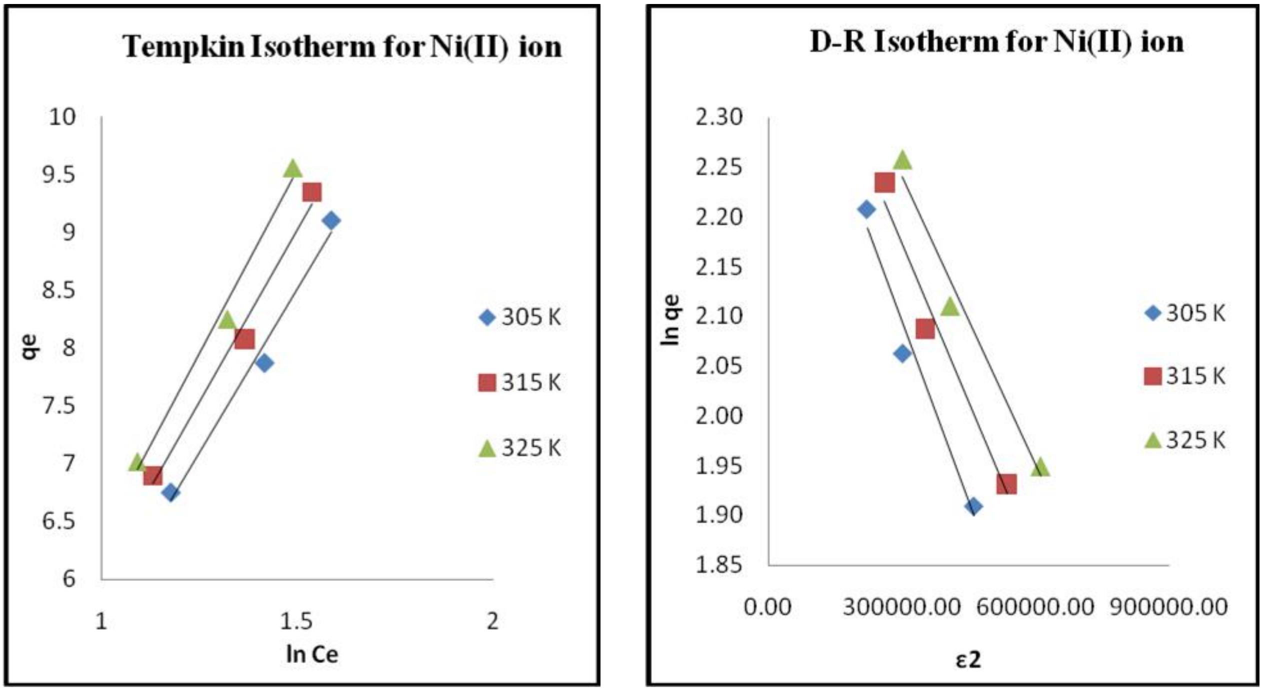


Figure: 4 Langmuir, Freundlich, Tempkin and Dubinin-Raduskevichfor the adsorption of Ni(II) ion onto ACHC

Table:6 Results of various isotherms plots for the adsorption of Ni(II) ion onto ACHC

Isotherm Models	Temp (K)	Parameters and their results			
		Q_0 (mg/g)	b	R_L	R^2
Langmuir	305	28.9	0.092	0.526	0.9300
	315	31.8	0.088	0.476	0.9200
	325	36.2	0.080	0.440	0.9300
Freundlich		K_f (mg/g^{-1})	n		R^2
	305	2.8	1.37		0.9900
	315	2.9	1.34		0.9900
	325	3.0	1.29		0.9900
Tempkin		B_1 (J/mol)	K_T (L/g)		R^2
	305	446.6	0.9825		0.9800
	315	438.8	1.0679		0.9800
	325	428.2	1.0547		0.9800
DubininRaduskevich		q_D(mg/g)	E (kJ/mol)	B × 10^{-4} (mol^2/J^2)	R^2
	305	11.65	0.7071	0.0010	0.9700
	315	12.10	0.7071	0.0013	0.9700
	325	12.50	0.7071	0.0028	0.9700

6.0 Thermodynamic study

Thermodynamic parameters like ΔH°, ΔS° and ΔG° were measured based on van't Hoff's plot.

K_d=C solid/ C liquid

$$\Delta G° = -RT \ln k_d$$

$$\text{Log } K_d = \Delta S°/(2.303R) - \Delta H°/(2.303R)T$$

Where, K_d is the equilibrium constant, ΔH° and ΔS° are the standard enthalpy and entropy changes of adsorption respectively and The values of ΔH° and ΔS° are calculated from the slopes and intercepts of the linear plot of ln K_d vs 1/T. The free energy of specific adsorption ΔG° (kJ/mol) is calculated from the following expression

$$\Delta G° = \Delta H° - T\Delta S°$$

The thermodynamically parameters calculated from van'thoff's plot are given in table 7. Negative ΔG° of adsorption indicates that the adsorption process is favorable and spontaneous in nature. The endothermic nature of adsorption is confirmed by the positive ΔH° values. The bonding between Ni(II) ions and ACHC surface is very weak, since ΔH° values are found to be in between 5.246 to 6.094kJ/mol. Positive values of ΔS° suggested good affinity of the dye towards the adsorbent and the adsorption is spontaneous in nature[22].

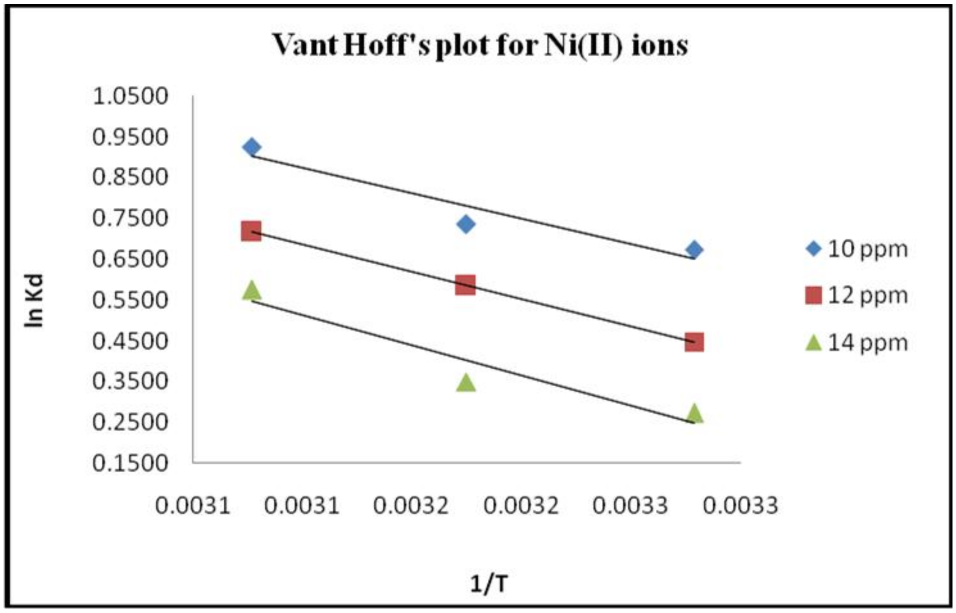


Figure 5: Thermodynamics for the adsorption of Ni(II) ions onto ACHC

Table:7 Thermodynamics parameters for the adsorption of Ni(II) ions onto ACHC

Ci mg/L	$-\Delta G^0$kJ/mol			ΔH^0 kJ/mol	ΔS^0 kJ/mol
	305K	315K	325K		
10	-1.8502	-2.0958	-2.3156	5.246	23.20
12	-1.6353	-1.8847	-2.1308	5.919	24.70
14	-1.5700	-1.8297	-2.0727	6.094	25.10

7. 0 Instrumental Analysis:

7.1 FT-IR spectrum study:

The FTIR spectrum of ACHC and Ni(II) ions loaded ACHC were shown in figure 6. The peaks positions of the above spectrum were given in table 8.

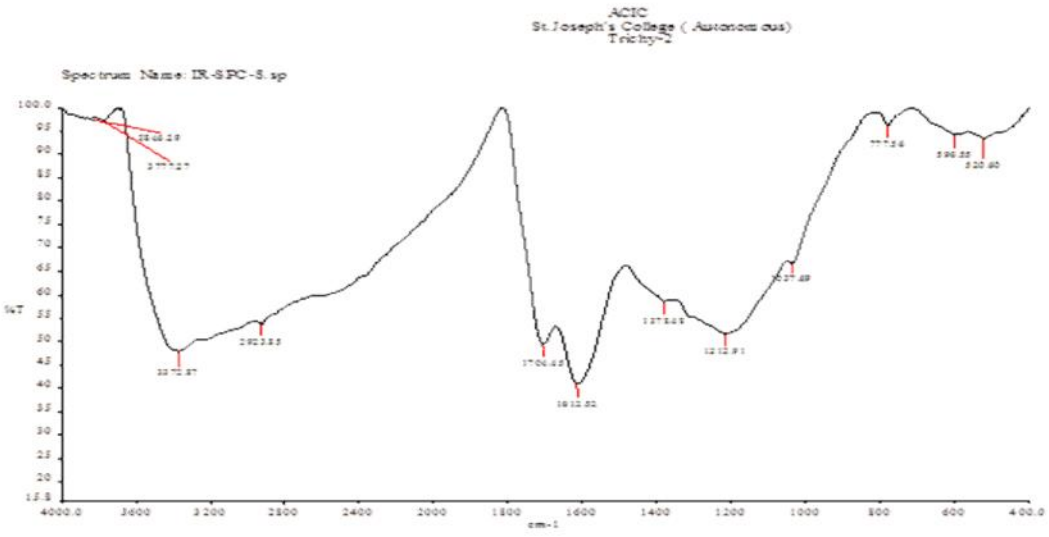
Table: 8 FTIR study of Ni(II) ions loaded ACHC

S.No.	Band position cm^{-1}		Observation
	ACHC	Ni(II)loaded ACHC	
1.	3846.29 May be due to water adsorption	3963.23	Peaks were slightly shifted
2	3777.27 May be due to water adsorption	3775.87	
3.	3372.27	3368.82	
4.	2923.85	2921.17	
5.	--------	2365.88 O-H stretch in acid group (2350-3300)	New peak Formed
6.	1704.45(1650-1730) C=O stretch in acid group	-------	Peak Disappeared
7.	1612.52(1600-1675) C=C symmetric stretch	1601.12	Peaks were slightly shifted
68.	1378.48 (1300-1410) C-O stretch in phenol	1385.21	
9.	1212.91(1020-1220) C-N stretch in amine	1213.38	
110.	1037.69(1020-1250) C-O stretch in ether or ester	-------	Peak Disappeared
11.	777.56 (750-800) C-H stretch in di substituted benzene	769.40	Peak slightly shifted

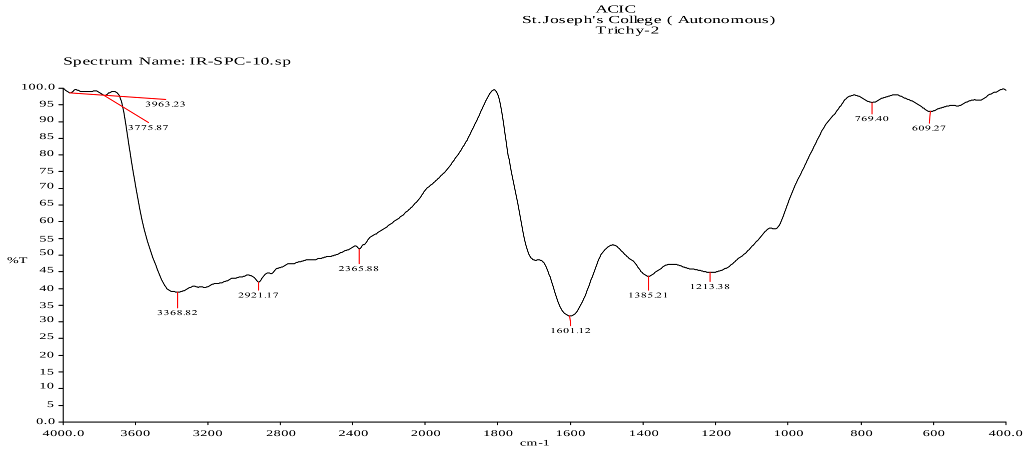
The band at 3372.87 cm^{-1} for ACHC was due to the O-H stretch vibrations and was shifted to 3368.82cm^{-1} for Ni(II) ions loaded ACHC.The bands at2923.85cm^{-1} ,1612.52 cm^{-1}1378.48 cm^{-1} 1212.91 cm^{-1} were due to the C – H ,C=C symmetric stretch C-Ostretch C-N stretch were slightly shifted to 2921.17cm^{-1},1601.12 cm^{-1},1385.21 cm^{-1} 1213.38 cm^{-1} respectively for Ni (II) ions loaded adsorbent. These results indicated the involvement of C-H,C-O,C = C, C – N functional group in adsorption process.

The characteristics adsorption peaks detected in ACHC at 1037.69 cm^{-1} due to C-O stretch was not detected in Ni(II)loaded ACHC. This infers that C-O functional group was involved in binding the Ni (II) ions. The one new peak was formed at 2365.88 cm^{-1} for Ni(II) loaded ACHC corresponding to N-H stretch which may be due to formation of new chemical bonds between the metal and adsorbent.

It is noticed that there is no change in the FTIR pattern of ACHC and Ni (II) ions loaded ACHC adsorption between the range 400-4000 cm^{-1}. Thus this FTIR spectral study supports physisorption.



FTIR of ACHC



FTIR of Ni(II) ion loaded ACHC

Figure: 6.FT-IR spectrum for before and after adsorption of Ni(II) ion onto ACHC

7.2 Energy Dispersive X-ray Spectroscopy (EDX) study:

Energy Dispersive X-ray Spectroscopy (EDX) is an analytical capability that can be coupled with several applications including Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and Scanning Transmission Electron Microscopy (STEM). EDX, when combined with these imaging tools, can provide elemental analysis on areas as small as nanometers in diameter. The impact of the electron beam on the sample produces x-rays that are characteristic of the elements present on the sample. EDX Analysis can be used to determine the elemental composition of individual points or to map out the lateral distribution of elements from the imaged area.

EDX analysis of Ni (II) ion loaded ACHC was shown in Figure 7. The well-defined peak of adsorbent loaded withNi(II) ion shows that Ni(II) ions were bound onto the ACHC surface. Peaks of certain Ni(II) ion are very small which infers the surface adsorption through Vander Walls force.

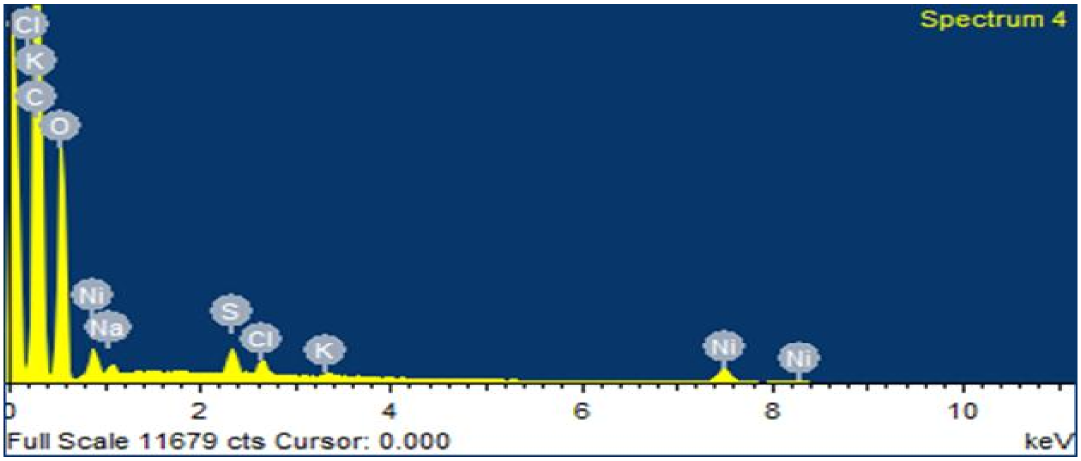


Figure: 7.EDX for adsorption of Ni(II) ion onto ACHC

7.3 SEM Analysis:

The surface morphology of the ACHC carbon was examined using scanning electron microscopy (SEM), the corresponding SEM Micrographs being obtained using at an accelerating voltage of 10 kv at 2400 x and 10kv at 3830 x magnifications (Figure 8). At such magnification, the activated carbon particle showed rough areas of surface on which micro pores and macro pores were clearly identifiable.

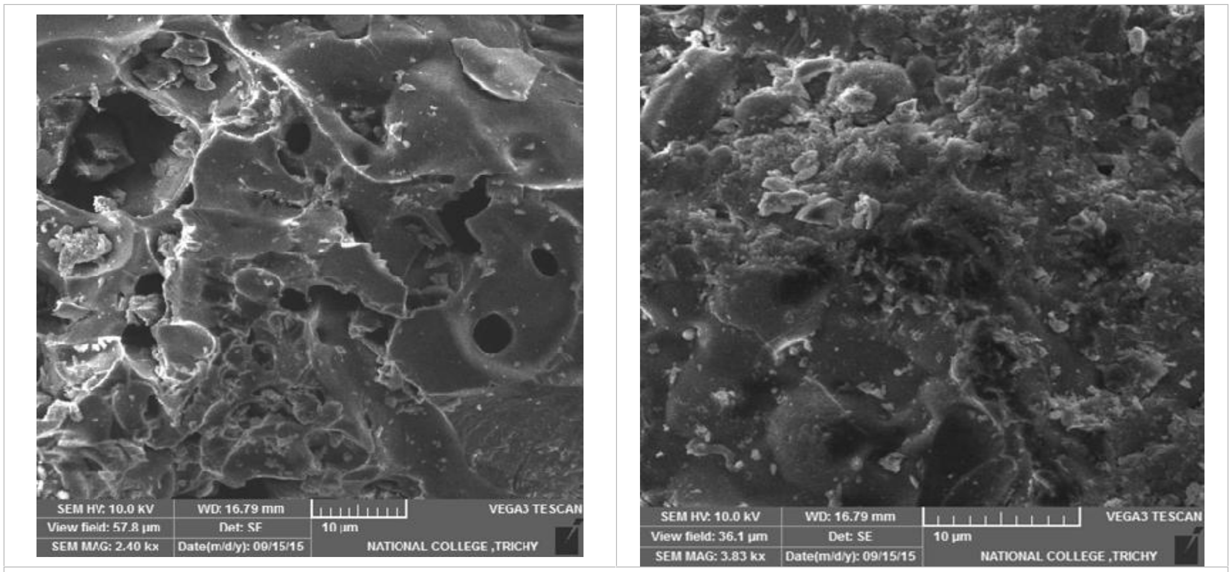


Figure: 9 SEM images of ACHC

7.4 XRD spectrum study:

X-ray Diffraction pictures of ACHC and ACHC loaded with the adsorbates are shown in figure:9.Inwhich there is no appreciable change in the before and after XRD patterns. Peak positions only slightly found to be changed. This study supports the predominant physisorption.

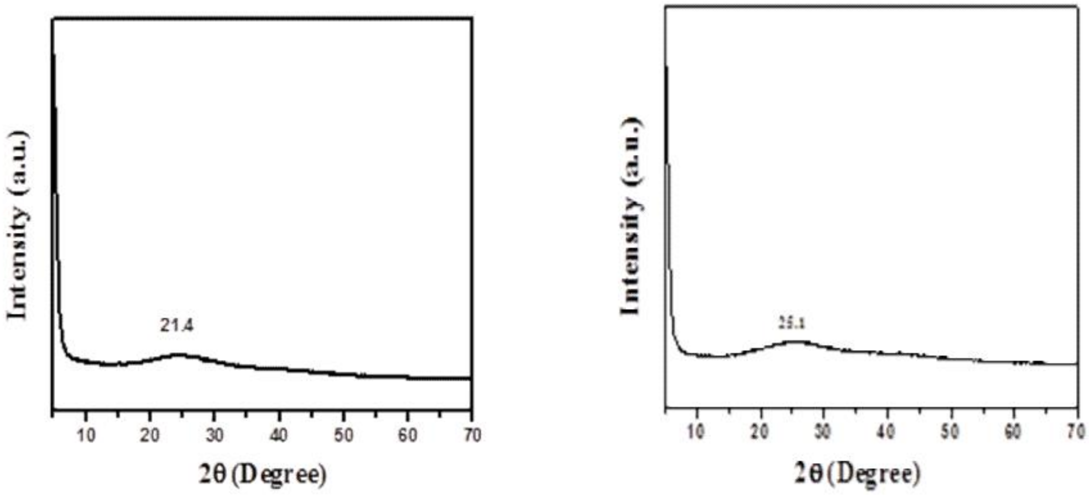


Figure: 9 XRD pattern for before and after adsorption of Ni(II) ions onto ACHC

8.0 Analysis of Isotherm:

8.1 Langmuir isotherm:

In the present study Q_0 value ranges from 28.90 to 36.20, as the temperature increases the monolayer adsorption capacity also found to increase. This kind of results was obtained in various similar studies [23].The separation factor R_L values were in between 0 to 1 which indicates the favourable adsorption.The R^2 value was very low when compared to other three isotherms.

8.2 Frendlich isotherm:

The values of n were between 1 and 10 which indicates cooperative adsorption [24]. The R^2 values were close to unity which revealed the good fitting into frendlich isotherm.

8.3 Temkin Isotherm:

B_1-Temkin constant is related to the heat of adsorption. This B_1 value increased from 446.60 to 428.20 as the temperatrue of adsorption increased. The temkin parameter K_T values give an idea about nature of adsorption [25]. In our present study the K_T values ranged from 0.9825 to 1.0547 which indicates that adsorption is physical nature. The R^2 value was low compared to frendlich isotherm.

8.4 Dubinin-Raduskevich:

The activation energy E value ranges from 0.7071 to 0.7071 and B value from 0.0010 to 0.0028 indicates the physisorption[26]. R^2 values of isotherm ranged in 0.9700.

In general the fitting data in isotherm equation were in the following order: Frendlich>Temkin>Dubinin-Raduskevich>Langmuir.

9.0. Conclusion

This study shows that ACHC can be used effectively for the removal of Ni(II) ions from aqueous solution. From the kinetic studies, it is observed that adsorption of Ni(II) ion is very rapid in the initial stage and decreases while approaching equilibrium. The equilibrium time increases with initial concentration. The percentage removal of Ni(II) ion increases with the increase in adsorbent dosage and decreases with increase in initial concentration. Experimental results are in good agreement with Freundlich isotherm when compared to other three isotherm models. Adsorption of Ni(II) ion obeys pseudo-second order equation with good correlation.Thermodynamic parameters such as ΔH^o, ΔS^o, ΔG^o values inferred that the adsorption was spontaneous, endothermic and physical nature. The instrumental analysis FTIR, XRD studies gives an idea that adsorption was physisorption with partial chemisorption. SEM pictures described the porosity of*Adina Cordifolia*HookCarbon(ACHC).

10.0. References

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