

ADSORPTION OF NICKEL FROM AQUEOUS SOLUTION BY ACTIVATED CARBON PREPARED FROM WHEAT HUSK

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ABSTRACT

Activated carbon prepared from waste wheat husk was used to remove Ni (II) from aqueous solution by adsorption. Batch adsorption studies were carried out by varying the contact time, initial Ni (II) ion concentration in the aqueous solution, adsorbent dosage, pH and temperature. Maximum adsorption was observed with the pH of 6.0, contact time of 4.0 h, adsorbent dosage of 1.50 g/150 ml and at the temperature of 30°C. It was indicated from the results that the effective removal of Nickel (II) ion was obtained by activating the carbon prepared from wheat husk which was heated to 600°C. Langmuir and Freundlich isotherms were used to describe the equilibrium nature of Ni (II) adsorption. The adsorption capacity (q_e) calculated from Langmuir and Freundlich isotherms were found to be 11.83 mg/g and 11.96 mg/g at pH 6.0 at 303 K. The removal of Ni (II) was found to be 80.1% for the initial Ni (II) concentration of 10 mg/l and adsorbent dosage of 0.50g/150ml. In the isotherm studies, the experimental adsorption data fitted well the Freundlich isotherm for the activated carbon from wheat husk. The results of the thermodynamic investigations indicate that the adsorption reactions are spontaneous ($G < 0$), slightly endothermic ($H > 0$) and irreversible ($S > 0$)

KEY WORDS: Nickel (II) removal, Activated carbon, Wheat husk, Adsorption isotherm, Thermodynamics

I. INTRODUCTION

The presence of heavy metals in the environment can be detrimental to the living species. Despite strict environmental regulations, significant quantities of heavy metals are found in the wastewaters from several industries such as non ferrous metal, electroplating, porcelain enameling, silver refining, welding, alloy manufacturing and fertilizer industry. The heavy metals are trace metals with density at least five times that of water. They are stable elements and are bioaccumulative in their nature. They include mercury, nickel, lead, arsenic, cadmium, chromium, aluminium, platinum, zinc, silver and gold (1).

Nickel is highly toxic and is present in the waste waters of industries such as paper and pulp, fertilizer, silver refining, pigments and coatings, basic steel work foundry, automobile, petroleum refining (2). The most likely sources of nickel emissions are found to be from metallurgical plants (3), engines burning fuel containing nickel additives, plating plants (4), and incineration of nickel products, metal extraction, fabrication operation and effluent from nickel-cadmium battery industries (5). The target tissues affected by nickel include skin, larynx, nasal passages and lungs. The signs and symptoms of nickel exposure include blue colored lips, cancer of lungs, nasal cavity and larynx, contact dermatitis, fever, headache, dizziness, skin rashes and vomiting (6). The incidence of lung and nasopharyngeal cancer in occupationally exposed population is due to the carcinogenic effect of nickel (7). Nickel in its ionic form can cross the cell membrane and get deposited in the nucleus. It might thus be the initiator and

promoter of cancer. The World Health Organization (WHO) recommended a maximum acceptable concentration of Ni(II) in drinking water as 0.05 mg/l per kg of the body weight per day and the inhalation should not exceed $3.5 \times 10^{-6} \text{ mg/m}^3$ (1).

Many methods like chemical precipitation (8), ion exchange (9), reverse osmosis, electro dialysis (4), membrane separation (10) and adsorption (11) are available for the removal of nickel present in the industrial effluents. Each has its own merits and demerits in application. The selection of the treatment method is based on the concentration of metal ion in the wastewater stream and the cost of the treatment (12). Currently, chemical precipitation is the most widely used technique. However, particular frustrating aspects of this method are significant sludge production, the ever-increasing cost of the landfill for the disposal and most importantly the long-term environmental consequences. These aspects result in an overall cost escalation of the process (13). Ion exchange, reverse osmosis and electro dialysis have material and operational cost (4, 8). Among these techniques, adsorption is one of the most commonly used methods due to its high efficiency and easy operation (14). In recent years, a number of adsorbents such as iron coated sand, iron coated granular activated carbon (15), modified chitin (16), fly ash (17), low grade bituminous coal (18), wool carbonizing waste (19), rice husk (20), kaolinite (12), Zeolite (21), paper mill sludge (17), bagasse (22), green algae (23, 24), bone char (25) and Lignite (26) was used in the heavy metal removal from effluents. In the present work, wheat husk was taken for the preparation of

activated carbon and its applicability in the removal of nickel ions present in the wastewater was studied. This paper reports the effect of adsorption parameters such as pH, contact time, initial concentration, and adsorbent dosage on the percentage removal of nickel (II) ions by using Activated Carbon prepared from Wheat Husk (ACWH) as the adsorbent material.

II. METHODS

A. Preparation of adsorbent

The wheat husk was washed with 1l of deionised water and rinsed with 200ml of 1% HCl. The wheat husk was dried at 30° C for 24 h. The dried wheat husk was mixed with phosphoric acid at a ratio of 1:3 (w/w) and soaked for 48 h, so that the reagents were fully adsorbed onto the raw material. The mixture was drained and dried at 108° C for 1 h. The dried husk samples were carbonized at 400° C in a muffle furnace for 1 h. The carbons prepared were washed repetitively with distilled water to recover the acid and then 1% NaHCO₃ solution to remove the residual acid. The wheat husk thus prepared was dried at 30° C for 2 h and then powdered and sieved. The powdered material remaining between 50 and 100 mesh (150 µm) was utilized for the adsorption studies. The proximate analysis of the raw material and the activated carbon properties are given in Table- 1. The adsorbent obtained is termed as 'ACWH'.

Table 1. Physical and chemical properties of adsorbent used in adsorption experiments

Surface area (m ² g ⁻¹)	1563.94
Bulk density (g cm ⁻³)	0.42
Particle size (µm)	150
Ash (%)	3.65
Carbon (%)	29.5
Moisture (1%)	9.35
Volatile (%)	57.42
Iodine number (mg g ⁻¹)	825.31
Methylene blue number (mg g ⁻¹)	280.00

B. Preparation of Adsorbate

All the reagents used were of analytical grade. Ni (II) stock solution was prepared by dissolving 4.479 g of pure nickel sulphate (NiSO₄ · 6H₂O) in 1 l of double distilled water. The pH of solution was adjusted by adding 0.1N sulphuric acid or 0.1N NaOH. Adsorption experiments were conducted by using ACWH as adsorbent.

C. Adsorption experiments using ACWH

Batch adsorption experiments were carried out at 30° C on a rotary shaker at 186 rpm using capped 250 ml Erlenmeyer flasks for various contact times. Adsorption studies involving nickel (II) ions on ACWH was conducted by taking different amounts of adsorbent like 0.15, 0.30, 0.60, 0.90 and 1.5 g for 150 ml of 10, 20, 30, 40, and 50 mg/l nickel solutions. Experiments were carried out at various pH values of nickel (II) solution adjusted with NaOH and HCl solutions using pH meter. The contact time was varied from 30 to 300 min. At the end of contact period, the reaction mixture was centrifuged at 56,000 rpm for 10 min. Nickel (II) in the supernatant was analyzed by using visible spectrophotometer using procedure given by (27) at 540nm. The percentage removal of nickel (II) was calculated from the differences between the concentrations of Ni (II) before and after adsorption.

D. Isotherm experiments

Isotherm studies were carried out in a series of 250 ml Erlenmeyer flask filled with 150 ml of Ni (II) solution having different initial concentrations with the pH of 6.0. After the equilibrium time, the solution was separated and analyzed. The initial concentrations of solutions taken for studies were 10, 20, 30, 40 and 50 mg/l, using adsorbent dosage of 1.5 g/150 ml solution. Adsorption studies were carried out at different temperatures to obtain equilibrium isotherms. The equilibrium adsorption capacity was calculated using

$$q_e = \frac{(C_0 - C_e)}{M} \times V \quad [1]$$

where q_e (mg/g) is the equilibrium adsorption capacity, C_0 and C_e are the initial and equilibrium concentrations of Ni (II) ions in solution. V (l) is the volume of the solution and M (g) is the weight of adsorbent.

III. RESULTS AND DISCUSSION

A. Effect of contact time on the percentage removal of Ni (II) ions

The effect of contact time on the percentage removal of Ni (II) on ACWH was studied at 30° C by varying the contact time from 30 to 300 min for Ni (II) solution having initial concentration from 10 mg/l to 50mg/l.

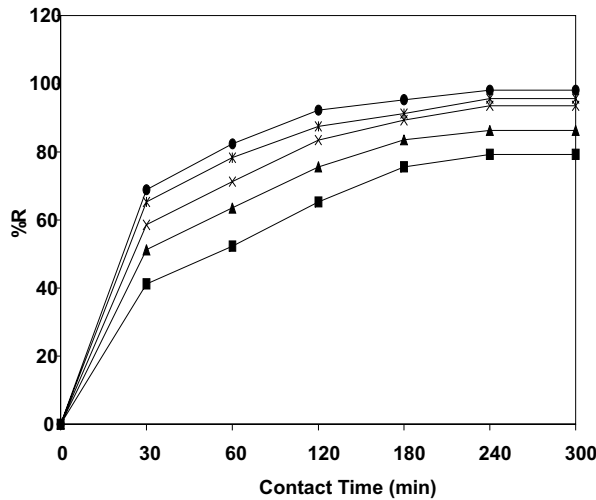


Fig. 1. Effect of Contact time on the Percentage Removal of Ni (II) ions by ACWH at different dosages (■) 0.15 g (▲) 0.3 g (X) 0.6 g (⋈) 0.9 g (●) 1.5 g

The study was made for adsorbent dosages ranging from 0.15 g to 1.5 g for 150 ml of 10 mg/l Ni (II) solution with the pH of 6.0 and at 30°C and the result is shown in the Fig 1. The percentage removal of Ni (II) ions was calculated using the formula given below

$$\% R = \frac{(C_0 - C_t)}{C_0} \times 100 \quad [2]$$

where % R is the percentage removal of Ni (II) ions in solution. The amount of Ni (II) ions adsorbed was found to increase with increase in the contact time and attained equilibrium at 4 h for all the concentrations of Ni (II) ions studied. The Ni (II) ion adsorption increases initially and approaches a constant value gradually as the equilibrium is attained. This behavior is attributed to the relatively less available adsorption sites on the surface of the ACWH as contact time increases (4).

B. Effect of pH on the percentage removal of Ni (II) ions

Adsorption experiments were carried out using Ni (II) solutions at different pH values by maintaining the adsorbent dosage as 1.50 g/150 ml Ni (II) solution at 30° C for initial concentration of Ni (II) solution ranging from 10 mg/l to 50 mg/l. The effect of pH on the percentage removal of Ni (II) ions is shown in Fig.2. The Ni (II) ion adsorption was found to increase from pH of 2.0 to 6.0, after which the metal ion uptake remained constant. The reduced uptake of metal ion at lower pH was due to the high concentration of H⁺ and free Ni (II) ions present in the solution that offered a competitive adsorption for the adsorption sites on the surface area of the ACWH. However, above the pH range of 6.0, the nickel

hydroxide species started to precipitate and thereby clogged the pores of ACWH. Similar observations were reported by (15).

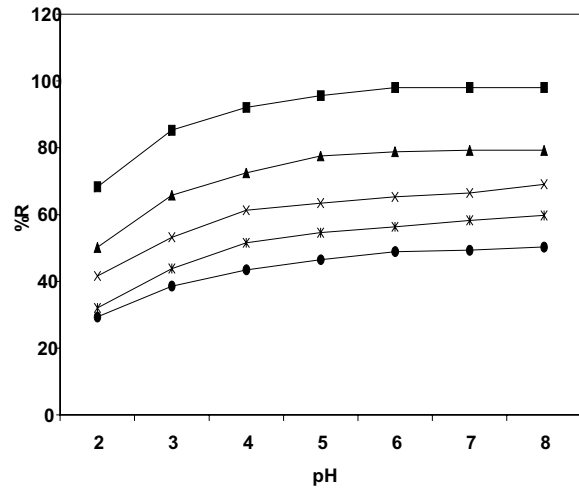


Fig. 2. Effect of pH on the Percentage Removal of Ni (II) ions by ACWH ml dosage (■) 10 mg/l (▲) 20 mg/l (X) 30 mg/l (⋈) 40 mg/l (●) 50 mg/l

The percentage removal of Ni (II) ions increased from 68.3 % to 98.1% and 29.3 % to 50.3 % for initial concentration of Ni (II) solutions 10 mg/l and 50 mg/l respectively for a fixed adsorbent dosage of 1.5 g at 30°C as the pH of the Ni (II) solution was varied from 2.0 to 8.0. This is due to the surface complexation reaction, which is influenced by electrostatic force of attraction between Ni (II) ions and surface of ACWH. Ni (II) has lower charge density, higher electro negativity of 1.8 and larger ionic radius of 0.069 nm which shows favorability to electrostatic force of attraction (28). The optimum pH was chosen to be 6.0 for further adsorption studies.

C. Effect of adsorbent dosage on the percentage removal of Ni (II) ions

Adsorbent studies were carried out using ACWH dosages ranging from 0.3 g to 1.80 g in Ni (II) ion concentration 10 mg/l, 20 mg/l, 30mg/l, 40 mg/l and 50 mg/l for the equilibrium time of 4 h at optimum pH 6.0 and at 30°C. The effect of adsorbent dosage on the percentage removal is shown in Fig. 3. It was found that the surface area available for the adsorption is limited for a specific dosage of ACWH. The reduced adsorption of Ni (II) ions at high concentration was mainly due to the unavailability of adsorption sites. It is clear from the Fig. 3 that the percentage removal can be increased from 80.1 to 98.1, if the adsorbent dosage is increased from 0.15 g to 1.5 g for 150 ml of Ni (II) solution at 10 mg/l concentration. This is due to the availability of more binding sites for complexation of nickel (II) ions (29). However, increasing the adsorbent dosage above 1.5 g had very little influence on the percentage removal which was just about 0.004 at

equilibrium for 1.8 g of ACWH. This effect may be due to the decline in the Ni (II) ion in the solution with the increase in the adsorbent dosage. Hence further addition of ACWH above 1.50 g in 150 ml solution was considered to be economically unsuitable for the removal of Ni (II) ions. At lower Ni (II) ion concentration the available sites of adsorption on the adsorbent is more, hence percentage removal was found to be high. Whereas at high concentrations, the available sites of adsorption become fewer, hence the percentage removal was found to be low.

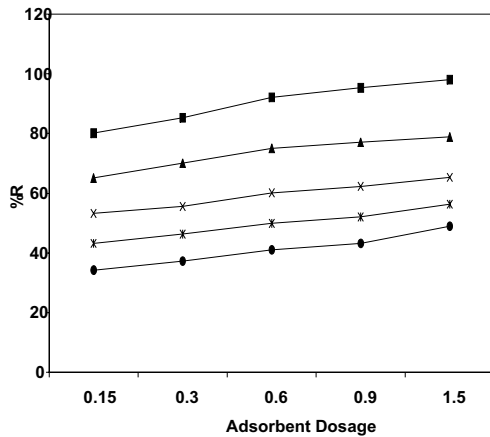


Fig. 3. Effect of Adsorbent Dosage on the Percentage Removal of Ni (II) ions at different Concentrations (■ 10 mg/l (▲ 20 mg/l (X) 30 mg/l (Ж 40 mg/l (● 50 mg/l

The adsorption density which was 6.54 mg/g for ACWH dosage of 1.5 g decreased to 5.45 mg/g for 1.8 g. This indicates that as the adsorbent dosage is increased above 1.5 g/ 150 ml of Ni (II) solution, the adsorption density got lowered. Therefore, adsorbent dosage of 1.5 g was taken as optimum for further studies.

D. Effect of initial concentration on the percentage removal of Ni (II) ions

As the initial concentration of Ni (II) ion provides an important driving force to overcome the Mass Transfer resistance between aqueous and solid phases (6), adsorption experiments were conducted using ACWH with different initial concentrations of Ni (II) ions for 4 h, at the pH of 6.0. The effect of initial Ni (II) ion concentration on the percentage removal is shown in the Fig. 4. The plot represents the percentage removal of Ni (II) ions versus the initial Ni (II) ion concentration for an equilibrium time of 4 h. The plot reveals that increasing the initial Ni (II) ion concentration from 10 mg/l to 50 mg/l decreases the percentage removal of Ni (II) ions from 80.1 % to 34.2 %. The rate of percentage removal of Ni (II) ions is higher in the beginning due to a larger surface area of ACWH being available for the adsorption of metal ions. After the adsorbed materials forms a thick layer, the capacity of the

adsorbent gets exhausted and then the uptake rate is controlled by the rate of transport of sorbate from the exterior to the interior sites of the adsorbent particles.

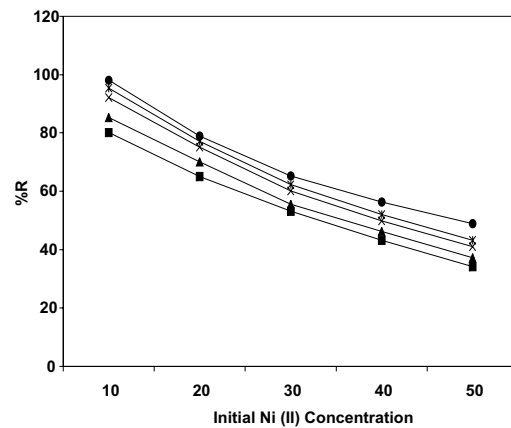


Fig. 4. Effect of Initial Ni (II) ion concentration on the Percentage Removal by ACWH at different Dosage (■ 0.15 g (▲ 0.3 g (X) 0.6 g (Ж 0.9 g (● 1.5 g

The percentage removal of Ni (II) ions is greater at lower initial concentration than at higher initial concentrations. This may be due to the fact that the surface area available for adsorption is limited for a specific dosage of activated carbon. The excess amount of Ni (II) ion in the solution is less adsorbed due to the unavailability of adsorption sites (30).

E. Effect of temperature on the percentage removal of Ni (II) ions

The effect of temperature on the adsorption of Ni (II) ions at different concentrations by ACWH at 1.50 g/150 ml is shown in Fig. 5.

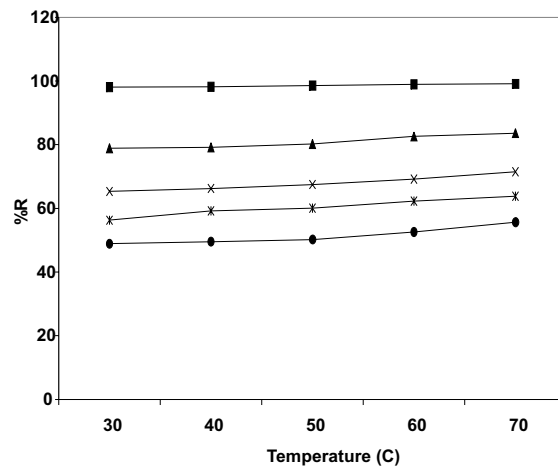


Fig. 5. Effect of Temperature on the Percentage Removal of Ni (II) ion at different Concentrations by ACWH (■ 10 mg/l (▲ 20 mg/l (X) 30 mg/l (Ж 40 mg/l (● 50 mg/l

The results reveal that the adsorption capacity increased from 10.52 mg/g to 12.26 mg/g as the temperature increased from 30°C to 70°C for an initial concentration of 20 mg/l at pH 6.0 for an optimum time of 4 h. This indicates that the adsorption of Ni (II) ions on ACWH is an endothermic process. The increase in temperature increases the mobility of the cation. The increase of adsorption capacity at increased temperature indicated that the adsorption of Ni (II) ions by ACWH might involve not only physical but also chemical sorption. This effect may be due to the fact that at higher temperatures an increase in the active sites occurs due bond rupture (24). The standard Gibbs free energy was evaluated by

$$\Delta G^\circ = -RT \ln K_c \tag{3}$$

The equilibrium constant K_c was evaluated at each temperature using

$$K_c = \frac{C_{Be}}{C_{Ae}} \tag{4}$$

where C_{Be} and C_{Ae} are the equilibrium concentration of Ni (II) ions on ACWH and solutions respectively. Van't Hoff equation was used to determine the standard enthalpy (ΔH°) and entropy (ΔS°)

$$\ln K_c = \frac{\Delta S^\circ}{R} - \frac{\Delta H^\circ}{R^2 T} \tag{5}$$

ΔH° and ΔS° were obtained from the slope and intercept of plot of $\ln K_c$ versus $1/T$ as shown in Fig. 6 and listed in Table- 2. Values of free energy changes ΔG° are negative, conforming the adsorption of Ni (II) on ACWH is thermodynamically favorable. As the temperature increases, the ΔG° value increases indicating more driving force and hence resulting in larger adsorption capacity at higher temperatures. The values of ΔH° are positive, indicating that the adsorption process is endothermic in nature. The negative values of ΔS° indicate greater order of reaction during adsorption of Ni (II) on the ACWH surface.

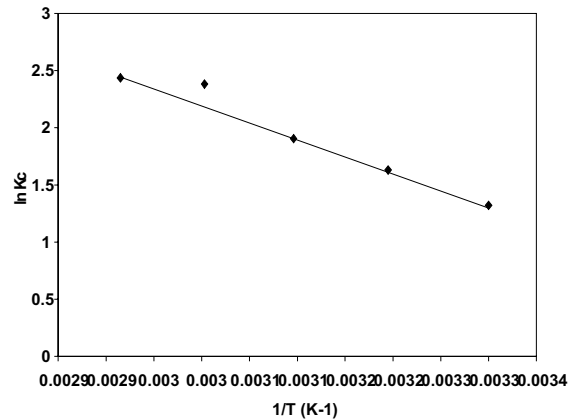


Fig. 6. Van't Hoff plot for the Adsorption of Ni (II) ions at 20 mg/l by ACWH at 1.5 g/150 ml

Table 2. Thermodynamic properties for the adsorption of Ni (II) onto ACWH

T (K)	ΔG° (kJ mol ⁻¹)	ΔH° (kJ mol ⁻¹)	ΔS° (J mol ⁻¹ K ⁻¹)
303	-3.33	+25.84	+96.28
313	-4.24		
323	-5.12		
333	-6.59		
343	-6.95		

F. Adsorption isotherm studies

The distribution of Ni (II) ions between ACWH and the Ni (II) solution, when the system is at equilibrium is used to determine the maximum adsorption capacity of ACWH. Models that are most widely used for solid-liquid adsorption are Langmuir and Freundlich models.

Langmuir isotherm

Langmuir equation, used for fitting equilibrium data is given by

$$q_e = \frac{K_L C_e}{1 + a_L C_e} \tag{6}$$

The amount of Ni (II) ion adsorbed at equilibrium per unit mass of the adsorbent is given by the above equation, where

$$a_L = \frac{K_L}{X_m}$$

The linear form of Langmuir isotherm is given by

$$\frac{C_e}{q_e} = \frac{1}{K_L} + \frac{C_e}{X_m} \quad [8]$$

where K_L (l/g) and a_L (l/mg) represent Langmuir constants. The maximum adsorption capacity in Langmuir model X_m (mg/g) is given by

$$X_m = \frac{K_L}{a_L} \quad [9]$$

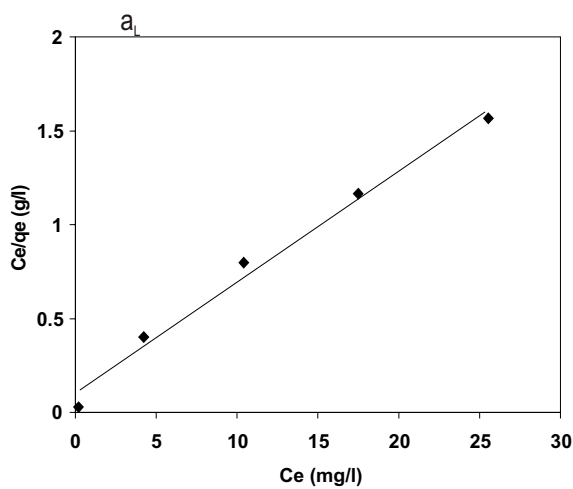


Fig.7 Langmuir Adsorption Isotherm for ACWH at 1.5 g/150 ml

The linear plot of C_e/q_e versus C_e confirms that Langmuir model can be applied for the Ni (II) ion adsorption onto ACWH. The Langmuir isotherm is shown in Fig. 7.

The Langmuir constants were calculated from the slope and intercept of this plot and are given in Table-3. The effect of isotherm shape has been considered with a view to predict whether the adsorption is favorable or unfavorable. The essential features of Langmuir isotherm can be expressed in terms of a dimensionless equilibrium parameter

$$R_L = \frac{1}{1 + a_L C_0} \quad [10]$$

Values of R_L are shown in Table- 4. for Ni (II) ion adsorption onto ACWH for various initial Ni (II) ion concentrations. Values of R_L between 0 and 1 indicate

favorable adsorption. The R_L values obtained indicate that adsorption is favorable for the entire initial Ni (II) ion concentrations studied and that it is more favorable at low initial concentration of Nickel (II) ions in the solution.

Table 3. Langmuir and Freundlich parameters for the adsorption of Ni (II) onto ACWH at 30°C ions

T (°C)	Langmuir constants				Freundlich constants			
	q_{eL} (mg g ⁻¹)	X_m	a_L (l mg ⁻¹)	R^2	q_e (mg g ⁻¹)	1/n	K_f	R^2
30	11.83	16.89	0.553	0.9874	11.96	0.246	7.380	0.9991

Freundlich isotherm

The Freundlich isotherm gives the relationship between equilibrium liquid and adsorbent capacity based on multilayer adsorption. This isotherm is widely used in adsorption studies at low concentrations of solute in aqueous medium. The expression for Freundlich equation is given by

$$q_e = K_f C_e^{1/n} \quad [11]$$

The linear form of Freundlich equation is given by

$$\ln q_e = \ln K_f + 1/n (\ln C_e) \quad [12]$$

where K_f (mg/g) and n are multilayer adsorption capacity and intensity of adsorption respectively. These constants are given in Table- 3. According to (31), values of n between 1 and 10 represent beneficial adsorption. The Freundlich adsorption isotherm is shown in Fig. 8.

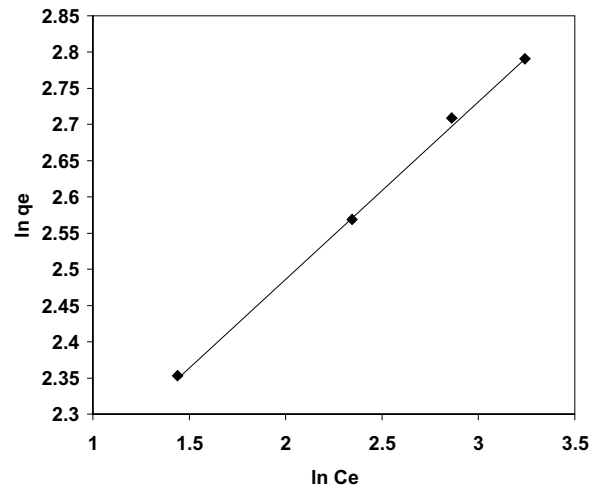


Fig. 8. Freundlich Adsorption Isotherm for ACWH at 1.5 g/150 ml

Table 4. Values of R_L and the comparison of experimental and predicted values of q_e from adsorption isotherms

q_e (mg g^{-1})	q_{e1} (mg g^{-1})	q_{ef} (mg g^{-1})	R_L
6.54	1.612	4.909	0.153
10.52	11.87	10.51	0.083
13.06	14.45	13.12	0.056
15.01	15.37	14.90	0.043
16.30	15.84	16.35	0.035

A comparison of the experimental values and predicted values from adsorption isotherms is shown in Fig. 9 and listed in Table- 4.

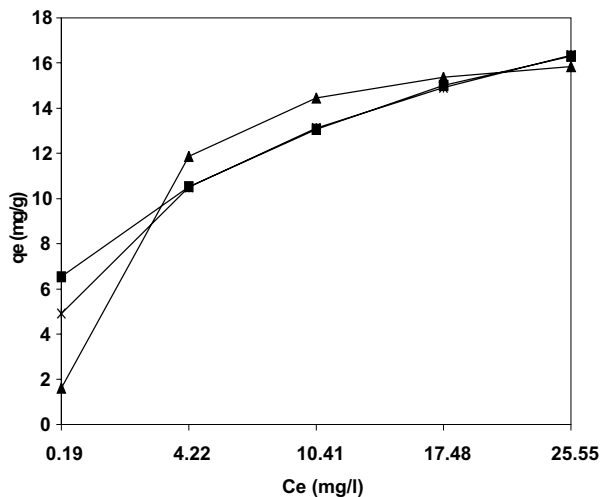


Fig.9 Experimental and predicted values of q_e from adsorption isotherm for ACWH at 1.5 g/150 ml

The error percentages between the experimental and predicted values of q_e from Langmuir model and Freundlich model are calculated using

$$\text{Error \%} = \frac{\sum_{i=1}^N (q_{e,\text{exp}} - q_{e,\text{cal}}) / q_{e,\text{exp}}}{N} \times 100 \quad [13]$$

The error percentages are found to be 7.182% and 1.613%. The Freundlich model agrees well with experimental data considering that the obtained percentage error values are lower than 2.0%. Table- 5 gives the comparison of Ni (II) removal by Activated Carbon prepared from Wheat husk (ACWH) and other adsorbents found in literature. It also provides the adsorption capacities of different adsorbents for the removal of Ni(II) ions.

Table 5. Comparison of Ni (II) removal by ACWH and other adsorbents found in literature

Adsorbent	Adsorbent capacity (mg g^{-1})	References
Fly ash	0.03	[9]
Bagasse	0.001	[9]
Carbon aerogel	12.875	[6]
Waste tea	18.42	[11]
Baker's yeast	11.40	[3]
Sheep manure waste	7.20	[30]
Turkish fly ash	1.16	[31]
ACWH	11.86	Present study

IV. CONCLUSION

The present work shows that the wheat husk activated carbon prepared by using phosphoric acid as the activating agent can be effectively employed for the adsorption of Nickel (II) ions. Nickel adsorption was found to be dependent on pH, adsorbent dosage and initial nickel ion concentration. Adsorption follows the Freundlich model. According to these results, Maximum adsorption was observed at pH of 6.0, contact time of 4.0 h, adsorbent dosage of 1.50 g/150 ml and at temperature of 30°C. This indicates that the adsorbent prepared from wheat husk can be used in the attempt for the removal of other heavy metal ions from waste water.

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