Canadian Chemical Transactions Year 2016 | Volume 4 | Issue 1 | Page 121-132

Research Article

DOI:10.13179/canchemtrans.2016.04.01.0264

Studies on Adsorption of Nickel by Activated Carbon in a Liquid Fluidised Bed Reactor

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Received: *December 10, 2015* ***Revised:*** *February 5, 2016* ***Accepted:*** *February 8, 2016* ***Published:*** *February 9, 2016*

Abstract: The work aims at adsorption studies of nickel from aqueous solution onto activated carbon using fluidised bed reactor. The experiment is run at the flowrate of 300 LPH. The parameters like pH, contact time, initial ion concentration, adsorbent dose and flow rate has been considered and their effects on adsorption of nickel are analysed. The experimental studies show that the optimal pH for removal of nickel is 6. The removal of nickel increases with time till saturation and then starts decreasing. Rate of adsorption increased with the increase of adsorbent dose. The maximum percentage removal of nickel is found to be 87.52 %. The removal percentage of nickel decreases with the increasing concentration of nickel. Langmuir and Freundlich isotherm models are applied to describe the equilibrium data and analyse nickel ion sorption from aqueous solution. Data is best fitted in Langmuir adsorption isotherm model. Maximum adsorption capacity is found to be 7.6569 mg of Ni(II) per g of GAC as adsorbent.

Keywords: Adsorption, Heavy Metals, Granular Activated Carbon, Fluidised Bed Reactor, Isotherm

1. INTRODUCTION

Pollution is one of the greatest abuses of our natural water resources. Excessive dumping of chemical waste containing heavy metals into water bodies, render the water useless for human needs. Most of the industries release untreated or partially treated effluent discharge into the nearby drains and water streams which contain heavy metals. Surface finishing and metal deposition are the important processes of electroplating industry which commonly use nickel, copper, zinc and chromium [1]. Wastewater generated from them release substantial quantity of heavy metal ions which leads to heavy metal pollution. In industrial, ground, marine, and even in treated wastewater, heavy metals are major pollutants.

Since heavy metals are non-biodegradable, they tend to accumulate in living organisms, so it is important to remove them for the protection of environmental quality and public health. Almost, all heavy

metals ingested beyond permissible quantities causes various chronic disorders in human beings. The effluents released from electroplating industries, refineries based on zinc casting and industries which produce batteries contain Ni. Nickel poisoning causes vomiting, nausea, cyanosis, headache and acute respiratory problems. In case, the concentration of nickel is high then it could lead to lung cancer. The most frequent effect of exposure to nickel is dermatitis (Ni itch) [2].

Any industry using metals faces a problem of metal disposal. So some advanced technologies are required for the treatment of heavy metal contaminated wastewater. Various conventional methods for removal of nickel include coagulation [3], ion exchange, precipitation, electro dialysis, adsorption, floatation, reverse osmosis [4, 5]. Out of these techniques, the adsorption method is widely accepted in environmental treatment applications and is more economical and efficient for removing dyes, organic pollutants and heavy metals from polluted waters [6, 7]. Of late, various low cost sorbents are used such as lignin, chitin, activated carbon, dead biomass, zeolite, clay, fly ash, algae, seaweed, orange peel, sawdust etc. [8]. But adsorption onto activated carbon has proven to be one of the most effective and reliable treatment for heavy metal removal. Granular activated carbon (GAC) is well known adsorbent. The structure of GAC is micro porous in nature which makes it good adsorbent. Its capacity for adsorption and surface reactivity is high. One of the major properties of GAC is that it can remove toxic heavy metal ions to concentration which is almost non- detectable [9, 10]. Therefore, GAC adsorption systems are considered for the present study to remove heavy metal ions from the wastewater.

Adsorption of heavy metals is often carried out in batch or continuous mode. The earlier studies on heavy metal removal limit to batch experiments or fixed bed and packed bed reactors. Fluidised bed reactor (FBR) is the current technology which is feasible and cost effective. In fluidized bed reactor fluid either liquid or gas is allowed to pass through a granular solid material at a velocity which can suspend the solid and cause it to behave as liquid. This is the process of fluidisation. The adsorption sites of absorbent could be used efficiently and the treated water can separate with the adsorbent directly in the FBR as compared to the batch adsorption process [11, 12]. Moreover, the adsorption rate is high using fluidisation than the fixed-bed adsorption process [13].

2. EXPERIMENTAL

2.1. Materials

Granular activated carbon is used as an adsorbent in the experiments and was provided by Brillex Chemical Ltd. Punjab. Stock solution of nickel was prepared by dissolving 4.479 g of $NiSO_4.6H_2O$ in 1000 ml of distilled water [14]. This stock solution was used for preparing working solution of different concentration ranging 10-50 mg/L of nickel solution.

2.2. Method

The fluidisation study was carried out in a lab-scale tapered fluidised bed reactor (FBR) having inner diameter 5cm and a height of 116 cm. The height of the tapered section is 7 cm with tapered angle

of 10 °. A weir mesh was placed on the top of FBR to prevent any escape of adsorbent from the column. The experimental setup consists of a fluidised column, water reservoir for aqueous solution of nickel, centrifugal pump and rotameter. One outlet for the effluent collection from the top of FBR has been provided for collection and recirculation of the effluent [15].

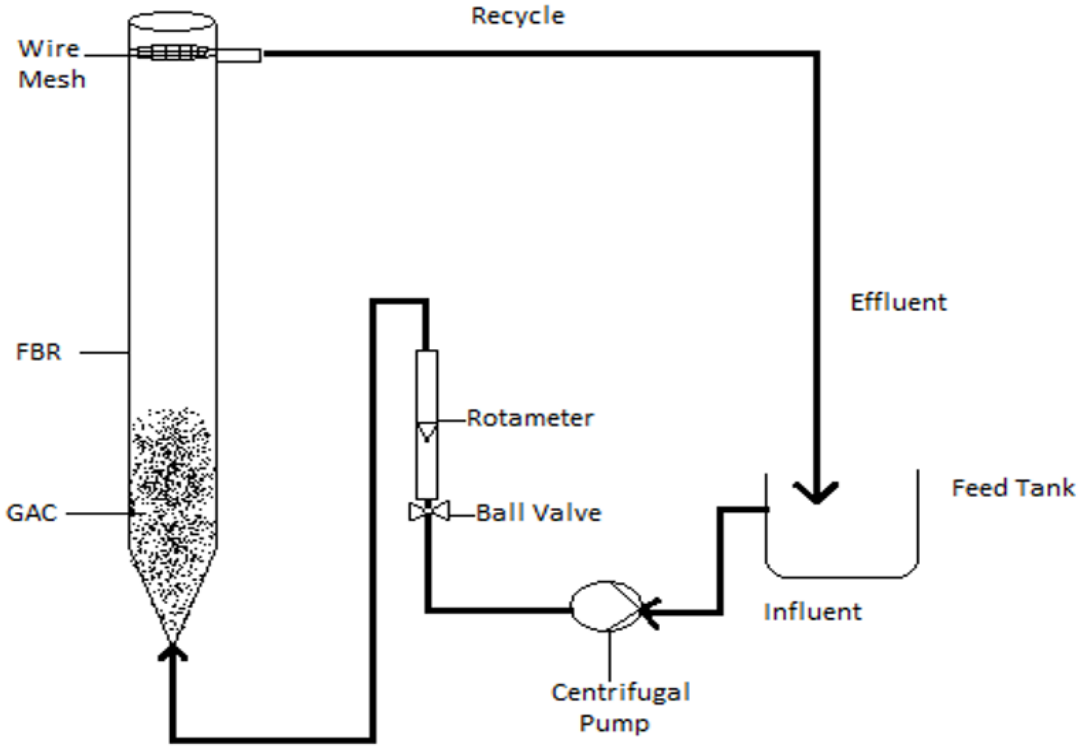

Fig. 1. Schematic diagram of lab-scale fludised bed reactor

A known weight of granular activated carbon is put into fluidised bed column. For fluidisation to occur, nickel sulphate solution was pumped into the reactor from the bottom of the column at the flowrate of 300 LPH. The sample was taken after regular interval of 60 mins.

After the adsorption the concentration of the nickel ions was determined by atomic adsorption spectrophotometer (ContrAA 700; Analytik Jena AG, Jena Germany). The percent removal of nickel ions was calculated by the following equation:

$$\%\ removal = \frac{C_o - C_i}{C_o} \times 100 \quad (1)$$

where C_0 (mg/L) is initial concentration of heavy metal, C_i (mg/L) is final concentration of heavy metal.

The adsorption capacity *Q*e (mg/g) after equilibrium was calculated by mass balance relationship equation as follows:

$$Q_e = \frac{(C_0 - C_e)}{M} V \quad (2)$$

where C_e (mg/L) is the concentration of adsorbate at equilibrium; *V* is the volume of the solution (L) and *M* is the mass of adsorbent (g) [16].

3. RESULT AND DISCUSSIONS

3.1 Effect of pH on nickel removal

To know the pH effect, the fluidisation experiment was run at pH 1 to pH 10 with initial concentration of 30 mg/L and adsorbent dose of 50 g GAC. The working solution (4L) of 30 mg/L was prepared by adding 120 mL of nickel sulphate stock solution to 3880 mL of distilled water. The percentage removal of nickel as a function of pH is shown in Fig. 2.

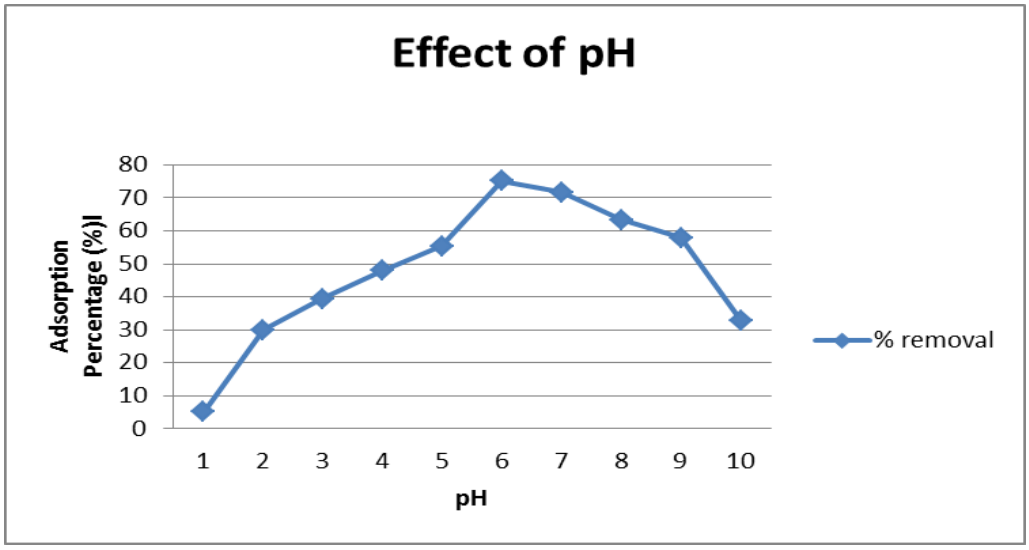

Figure 2. Effect of pH on adsorption of nickel onto GAC

The pH in the experiment was adjusted with 0.1N HCl and 0.1N NaOH The adsorption of nickel first increases with increase of pH, reaching maximum 75.23 % at pH 6 and then started decreasing when the pH is increased further. The removal of nickel at lower pH was slow possibly due to competition between hydrogen and nickel ions in the adsorption sites. At higher pH, precipitation of nickel ions forms nickel hydroxide precipitate which results in decrease in nickel adsorption [17].

3.2. Effect of time of fluidisation

The role of contact time on nickel of removal was studied, using 30 mg/L nickel ion concentration and adsorbent dose was varied between 50-100 g. The experiment in FBR was run with concentration of 30 mg/L and working volume of 4 litres. At regular interval of 1 hour, the samples were collected and analysed for nickel ion concentration.

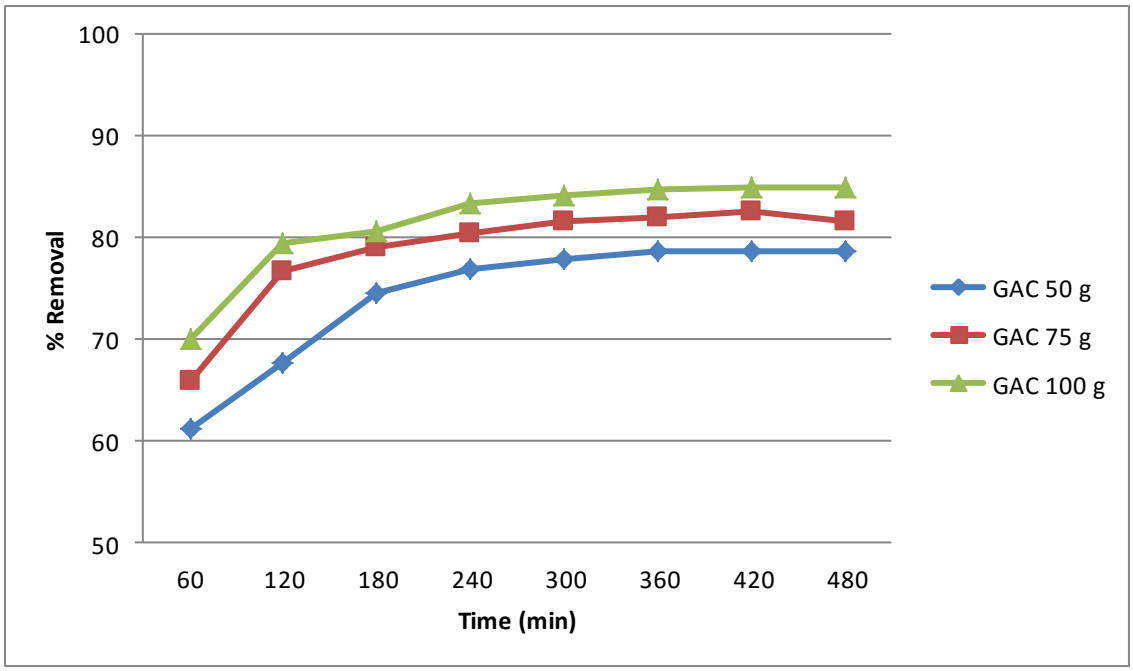

Figure 3. Effect of time on adsorption of nickel onto GAC

It was found that removal of nickel increases with time and reaches saturation in about 480 min of fluidisation. (Fig. 3). The nickel ions uptake was increases from 61.15%, 65.88% and 70 % at 60 min to 78.7%, 81.66% and 84.88% at 480 min with adsorbent dose of 50 g, 75 g and 100 g respectively. The rate of removal was high in the beginning as the number of adsorption sites was more and because of larger surface area [18]. But with the passage of time, the adsorption sites get exhausted and thus lead to decrease in removal of nickel ions.

3.3. Effect of adsorbent dose

To study the influence of adsorbent dose, adsorption of nickel was studied at room temperature at pH 6. The adsorbent dose was varied from 50-100 g in 4 L of aqueous solution and 30 mg/L of Ni (II) concentration. The effect of adsorbent dose is presented in Fig. 4.

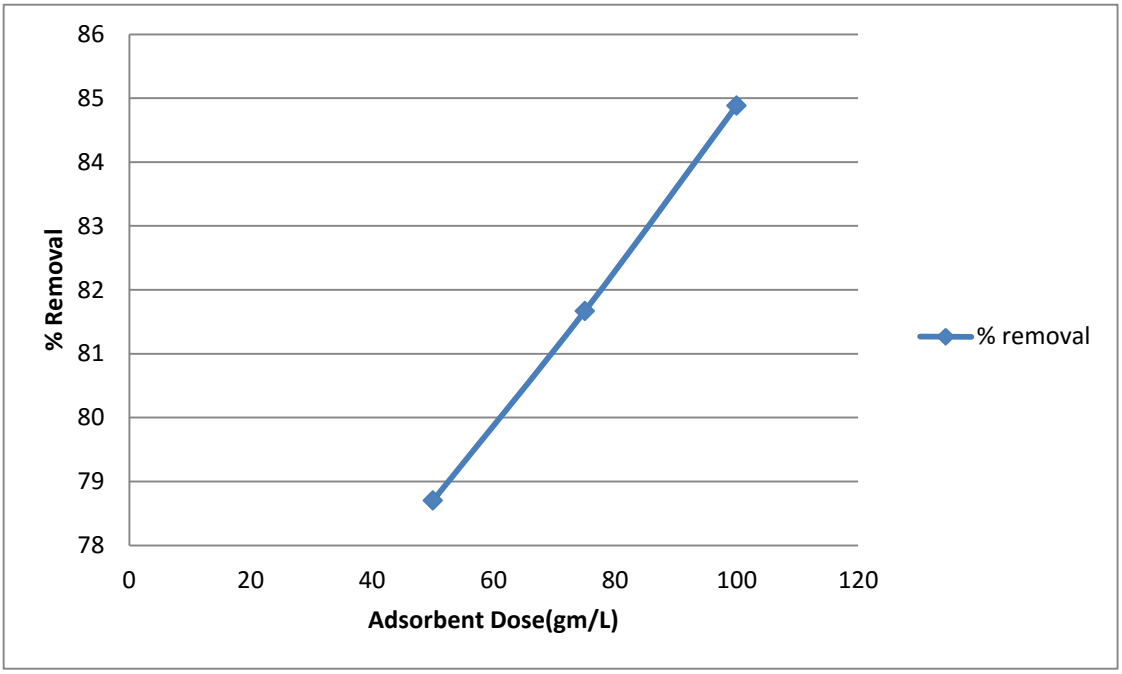

Figure 4. Effect of Adsorbent dose on adsorption of nickel onto GAC

It can be inferred that the adsorption of nickel ions increases with the increase in the granular activated carbon dose. The removal of Ni(II) ions increases from 78.7% to 84.88% when GAC was increased from 50 to 100 g.

This is due to the increase in surface area with the increase in mass of adsorbent which results in greater availability of the exchangeable sites or active sorption sites [19].

3.4. Effect of initial ion concentration of nickel

To analyse the effect of initial ion concentration, the adsorption experiments were carried out at different initial nickel ion concentrations ranging from 10 to 50 mg/L. It was revealed that, the removal percentage was high at low concentration of nickel and gradually decreased with the increasing nickel ion concentration. (Fig. 5).

The removal decreased from 81.9%, 85.8% and 87.52% to 75.96%, 76.81% and 50.78% at adsorbent dose of 50, 75 and 100 g respectively. This is due to the saturation of the exchange sites with increasing metal concentrations leading to less removal efficiency [20].

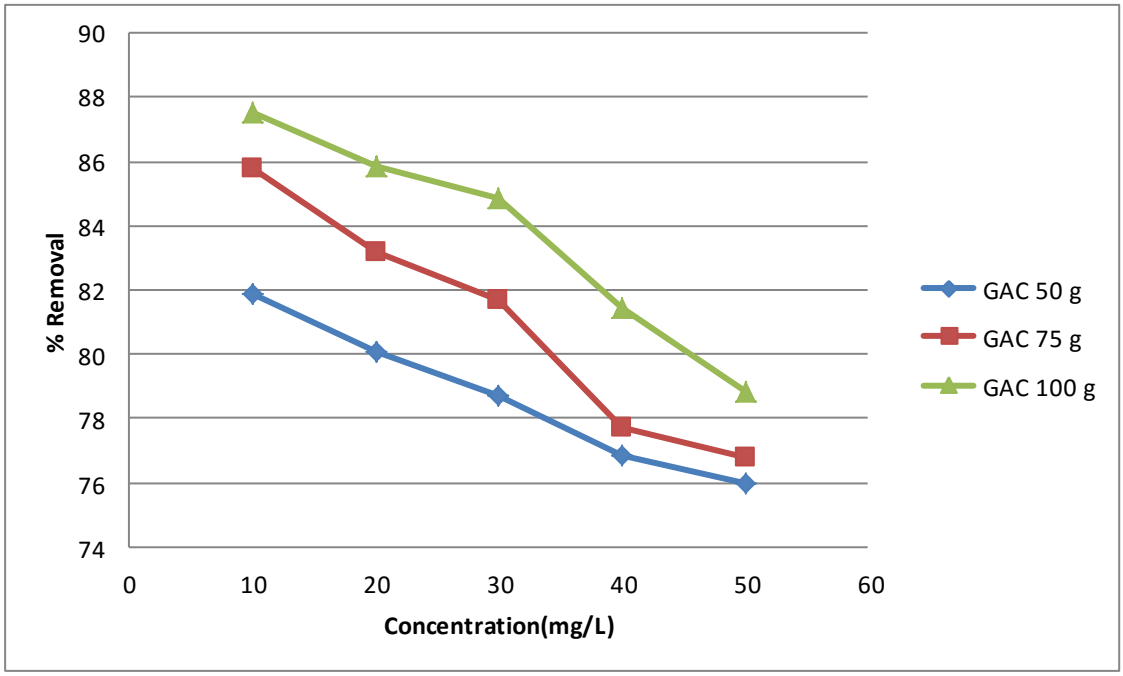

Figure 5. Effect of initial ion concentration on adsorption of nickel onto GAC

3.5. Adsorption isotherms

To analyse Ni ion sorption from aqueous solution, Langmuir and Freundlich adsorption isotherms were used. The following equation represents the of Langmuir isotherm:

$$\frac{1}{Q_e} = \frac{1}{Q} + \frac{1}{bQ}\frac{1}{C_e} \quad (3)$$

where Q is the Langmuir adsorption capacity in mg/g and b is the energy of adsorption in l/mg. the values of Q and b were computed from the slope and intercept of the graph of $1/Q_e$ Vs $1/C_e$ [21].

The Freundlich isotherm is represented by the equation:

$$\log Q_e = \log K_F + \frac{1}{n} log C_e \quad (4)$$

Where K_F is the Freundlich constant related to adsorption capacity and n is adsorption intensity. Log C_e was plotted against log Q_e from which the Freundlich constants are calculated [22]. Adsorption study data was fitted to the Langmuir (Fig. 6) and Freundlich (Fig. 7) isotherms and values of various parameters were calculated and shown in table 1 and 2.

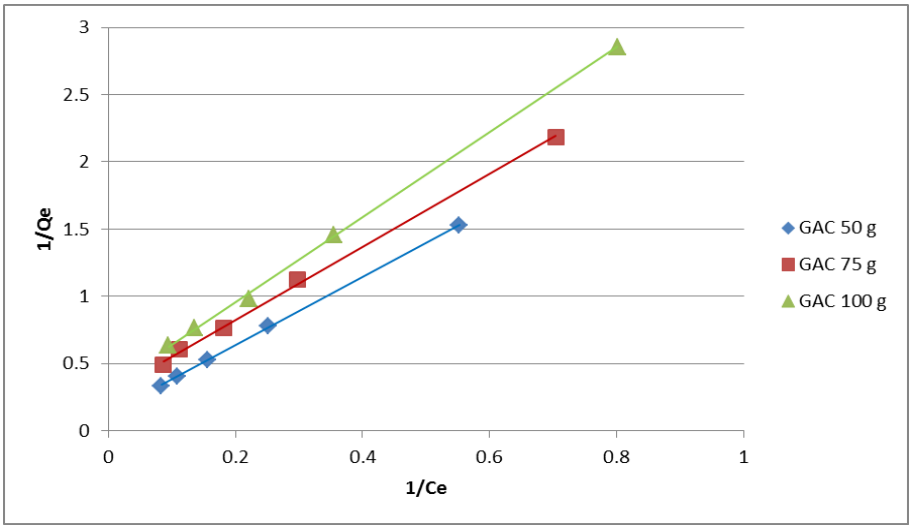

Figure 6. The Langmuir adsorption isotherms for Ni(II) by GAC

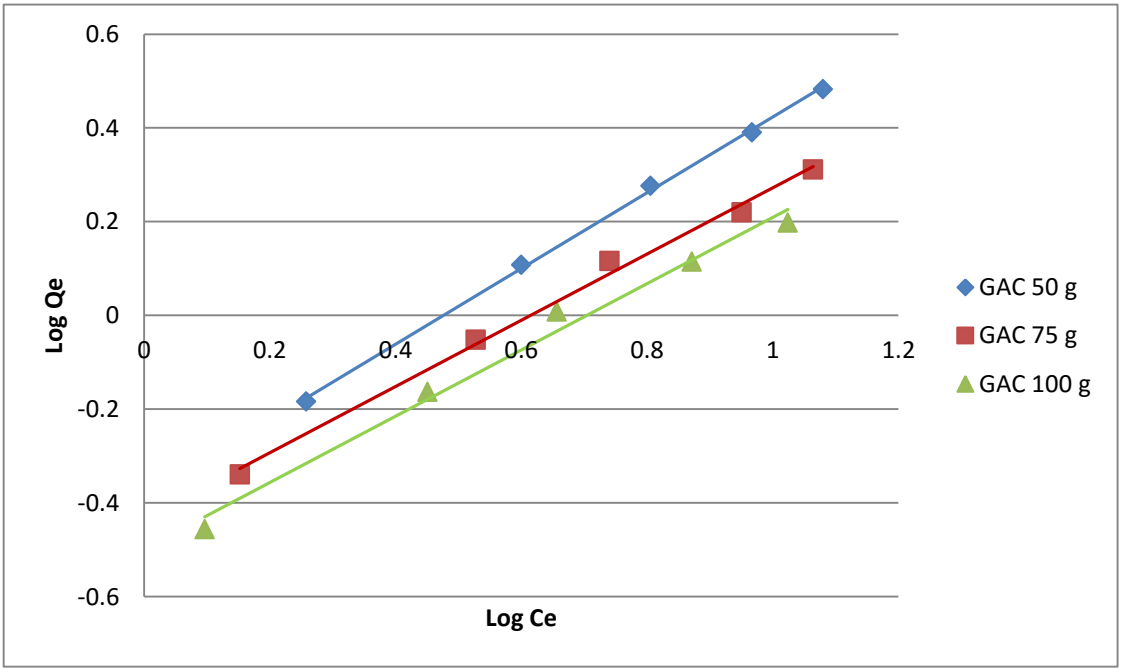

Figure 7. The Freundlich adsorption isotherms for Ni(II) by GAC

Table 1. Langmuir constants for adsorption of Ni(II) by GAC

GAC (g)	Langmuir constants			
	R^2	σ	b(L/mg)	Q(mg/g)
50	0.9996	0.00941	0.05152	7.65697
75	0.9985	0.02643	0.10422	3.53107
100	0.9994	0.02304	0.10327	3.06843

Table 2. Freundlich constants for adsorption of Ni(II) by GAC

GAC (g)	Freundlich constants			
	R^2	σ	N	K_F
50	0.999	0.00828	1.23793	0.41248
75	0.9949	0.01840	1.41623	0.36813
100	0.987	0.02951	1.41363	0.31769

The best value of R for Langmuir isotherm is 0.9996 at 50 g GAC dose. For the Freundlich isotherm also best value for R was found at adsorbent dose of 50 g (0.999). As the values are above 0.9, it indicates that data fits to both the isotherm. But it is well fitted in Langmuir isotherm. The values of *Q* and *b* were determined from the figure and were found to be 7.6569 mg/g and 0.05152 l/mg, respectively.

Adsorption process in FBR is a better technique to remove nickel as compared to other conventional methods like chemical precipitation, ion exchange, ultrafiltration, electrodialysis and adsorption by batch and packed bed experiments. The removal percentage of nickel in present study is 75.23%. The removal efficiency of other techniques is compared in the table 3.

Table 3. Various techniques used for removal of nickel.

Technique	Removal percentage	pH	Reference
Crab shell in packed bed	71.82%	4.5	[23]
Electrodeionisation	26%	NA	[24]
Immobilized sericite beads	60%	7.5	[25]
Electrolysis cell	51-67%	3-6	[26]

4. CONCLUSION

The performance of fluidised bed reactor have been investigated to remove Ni(ll) from the aqueous solution using GAC as an adsorbent. Maximum removal of 75.23% is observed at pH 6. Adsorption of Ni(II) increases with time and with increase in adsorbent dose. But with the increase in initial ion concentration of Ni(II), the rate of removal decreases. Removal of Ni(II) by adsorption on GAC follows both Freundlich and Langmuir adsorption isotherm models. It is found that with R value of 0.9996, the data best fits to Langmuir adsorption isotherm. Maximum adsorption capacity of Ni(II) per g of GAC is found to be 7.6569 mg.

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The authors declare no conflict of interest

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