Removal of Nickel from Simulated Wastewater using Pongamia Pinnata Seed Shell As Adsorbent

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Abstract: **Electroplating and metalworking industries discharge large amounts of heavy metal including nickel (Ni) ions, in their effluents have been recognized as a major problem to human health and aquatic life. Nickel exposure varies from skin irritation to damage of the lungs, nervous system, and mucous membranes. The current regulation of wastewater and drinking water standards are require contamination of heavy metal reduced up to few parts per million. Several processing techniques are available to reduce the concentrations of heavy metals in wastewater, including precipitation, flotation, ion exchange, solvent extraction, adsorption, cementation onto iron, membrane processing, and electrolytic methods. Adsorption onto activated carbon is a well-known method for removing toxic metal ions, but the high cost of activated carbon restricts its use in developing countries, so cheap and effective alternatives for the removal of heavy metals should reduce operating costs, reduce the prices of products, improve competitiveness, and benefit the environment. In this study, removal of poisonous Ni from artificially contaminated water has been investigated with the aim of detoxifying industrial effluents before their safe disposal onto land or into river waters. Low-cost natural adsorbent pongamia pinnata seed shell was used to remove Ni from synthetic wastewater. The adsorption equilibrium data correlate well with Langmuir model with regression, R2 0.925. The results showed that efficiency of pongamia pinnata seed shell for Ni ion removal was more than 95%. High adsorption capacity of the tested adsorbent makes it preferable and very attractive alternative adsorption material.**

Keywords: Adsorption, Ni, Low-cost adsorbents, Adsorption isotherms, Pongamia pinnata seed shell.

I. INTRODUCTION

Increasing industrialization throughout the world, especially in the last few decades has resulted in the production of thousands of new chemicals annually. These may be present in the water discharge of the manufacturing and industrial plants. In addition to this, increased use of various pesticides and chemical fertilizers in agriculture also contribute to the contamination of ground water. The abundance of industrial plants, Factories and other manufacturing or processing facilities creates a significant environmental hazard through emissions and wastes. Nickel may be found in waste water discharges from mining, electroplating, pigments and ceramic industries, battery and accumulator manufacturing. Nickel is toxic to a variety of aquatic organisms, even at very low concentration. An uptake of large quantities of nickel may lead to higher instances of cancer, lung embolism, respiratory failure, birth defects, asthma and chronic bronchitis, severe damage to kidney, gastrointestinal distress (e.g. nausea, vomiting, and diarrhea), allergic reactions such as skin rashes and heart disorders. Over the past few decades, environmental regulatory requirements have become more stringent because of increased awareness of the human health and ecological risks associated with environmental contaminants. [4]

A number of specialized processes have been developed for the removal of toxic metals from waste water. These include chemical precipitation, coagulation-flocculation, electro coagulation, cementation, ion exchange, membrane process, electro-flotation, concentration, adsorption, absorption, electro-deposition, reverse osmosis, solvent extraction, ion exchange process, evaporative recovery and biological treatment. The task of providing proper treatment facility for all polluting sources is difficult and also expensive, hence there is pressing demand for innovative technologies which are low cost, require low maintenance and are energy efficient. Adsorption is considered as one of the effective and economical technologies. Adsorption is considered as one of the effective and economical technology compared to others for removal of heavy metals from effluents. Natural material or certain waste from industrial or agricultural operation is one of the resources for low cost adsorbents. Generally, these materials are locally and easily available in large quantities. Therefore, they are inexpensive and have little economic Value. A fundamentally important characteristic of good adsorbents is their high porosity and consequent larger surface area with more specific adsorption sites. [6]

Pongamia pinnata trees are widely grown in Asia and also in other parts of the world. All parts of this tree have application in medicinal and other areas including the most important one the production of biofuel from Pongama pinnata seeds. Hence, large number of trees is grown at farms and availability of the Pongamia pinnata seed shell is more. The present study shows the feasibility of Pongamia pinnata using adsorbent prepared from Pongamia pinnata seed shell for removal of nickel ions from aqueous solution with parameters as pH, adsorbent dosage, concentration of metal ion and contact time. [8]

II. EXPERIMENTAL

a. Chemicals:

Laboratory grade reagents were used for heavy metal solutions; concentrated HCl and NaoH were used to adjust pH values of samples. In all experimental work, distilled demineralised water was used.

b. Adsorbent:

Pongamia pinnata seed shells was collected from Agriculture Department, Tholunase, Davangere, Karnataka. The shells were washed in 0.1N HCl solution .It was sundried for 3 days. Then the shells were dried in hot air oven for 80°C and pulverized into powder, this powder is subjected to sieve analysis in sieve shaker, to get 150µm retained powder. This powder was washed several times with distilled water to remove soluble, coloring matter, then it is sun dried and stored in air tight containers.

c. Stock solution:

Laboratory grade nickel sulphate (Nice Chemicals) of required quantity was dissolved in distilled water to prepare stock solution. 4.477grams of $NiSO_46H_2O$ was added in the 100ml of distilled water in 1000ml volumetric flask. It was dissolved by shaking and volume was made up to the mark. Nickel concentration of this was 1000mg/l. Stock solution was diluted with distilled water to obtain solutions of various concentrations.

d. Glass wares and Apparatus used:

All glass wares (Conical flasks, Pipette, measuring jar, beakers).The instruments used throughout the experiment are listed below:

- Atomic absorption spectrophotometer- GBC Avanta.
- Digital weighing balance
- Whatman filter paper
- Sieve shaker
- Jar test apparatus
- pH meter
- Glass column
- Glass wool

e. Batch mode adsorption studies:

Effect of several parameters such as pH, concentration of metal ion, concentration of adsorbent and contact time on adsorption of copper on powdered raw pongamia pinnata seed shell was studied by batch technique. All experiments were carried out at room temperature so as to avoid the heating of effluent in case this study would be applied for the pilot scale at the industry. Batch experiments were carried out at an agitation of 100 rpm, samples at predetermined time intervals were collected, filtered by whatman filter paper and remaining copper was analyzed by Atomic absorption Spectrophotometer as per standard methods. All experiments were carried out at pH values ranging from 2 to 10, the initial concentration of metal ion from 10 to 50 mg/L. Adsorbent dose of 0.6 to 3 g and particle size of 150µm and the contact time of 30min to 180min based on equilibrium conditions.

The percentage removal of toxic metal from the solution was calculated using the equation,

$$\% \text{ Removal} = \frac{\text{Ci} - \text{Cf}}{\text{Ci}} \times 100$$

Where C_i is initial concentration of toxic metal, C_f is final concentration of toxic metal.

f. Column adsorption studies:

This experiment is useful in understanding and predicting the behavior of the process. The adsorption experiments were carried out in glass column of internal dia 2.5cm and height of 50cm and that was equipped with a stopper for controlling the column flow rate. A packed column is a continuous employing process and is expected to be more efficient and economical to operate that the batch technique. The adsorption experiments were carried out in columns that were equipped with a stopper for controlling the column flow rate. Afterwards, the pH has been adjusted to 5 with H2SO4 and NaOH solutions. The sample solution was passed through the adsorption column at a flow rate of 3ml/min by gravitation. Inlet of the column was connected to the 25 liter feed bucket with the plastic tubing. The flow rate was kept constant by controlling the stopper value. The column was filled with 20 grams of pongamia pinnata seed shell adsorbent with depth of 10cm. Some glass wool was filled at two sides of the column to ensure homogenous distribution of influent solution from the top to the bottom. Before feeding the column with an influent solution containing nickel the column was run with pure distilled water for 1 hour to get wet and preserve the equilibrium between the water and adsorbent. Due to uniform distribution of solution, the pressure gradient is reduced and canalization of the sorbent is decreased. The concentration of residual individual toxic metal in the sorption medium was determined with AAS after the preparation of samples according to the standard methods.

Table 1: Physical controlled parameters in a packed bed column.

Physical controlled parameter	Value
Empty bed contact time (min)	60
flow rate (ml min-1)	3
Inflow pH	5
Initial metal ion concentration (mg L-1)	25,50,75,100
Temperature (ºC)	Room temperature
Bed height (cm)	10
Mass of adsorbent (g)	20

III. RESULTS AND DISCUSSIONS

1. Batch mode adsorption studies:

a. Effect of pH:

pH variation is one of the most important parameters controlling the uptake of toxic metals from wastewater and aqueous solutions. The studies were conducted at room temperature with an initial metal ion concentration of 50ppm and constant adsorbent dose of 3gm/300ml solution, at an agitation period of 120min.

Effect of pH on adsorption was conducted at ranges of 2, 4, 6, 8, and 10 in each solution. The percentage adsorption increases with increase in pH up to 8, and there after it decreases with further increase in pH.

Results obtained were as follows:

Table 2: Effect of pH on % removal of nickel ions by pongamia pinnata seed shell adsorbent

pH	Adsorbent Dose(grams)	Contact Time(min)	Initial Metal Ion Concentration(ppm)	Final Concentration(ppm)	% Removal
2	3	120	50	19.069	61.80
4	3	120	50	3.416	93.10
6	3	120	50	1.704	96.60
8	3	120	50	0.838	98.30
10	3	120	50	1.334	97.30

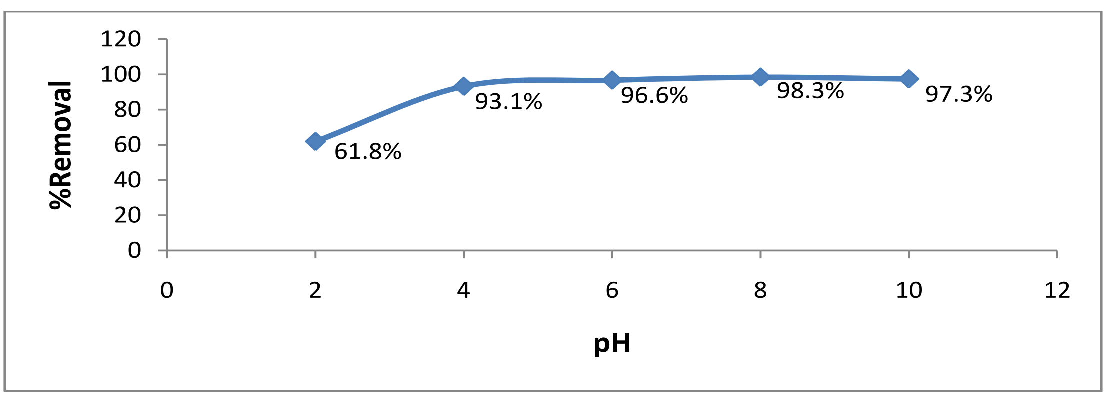

Fig 1: Effect of pH on % removal of nickel ions by pongamia pinnata seed shell adsorbent

b. Effect of initial concentration of nickel ions:

The adsorption of nickel ion on adsorbent depends on initial concentration and it can be seen that percentage removal decreases with increase in initial metal ion concentration. At lower initial metal ion concentrations, sufficient adsorption sites are available for adsorption of metal ions. However, at higher concentrations the number of metal ions relatively higher compared to availability of adsorption sites. The studies were conducted at room temperature at constant adsorbent dose of 3gm/300ml solution, with pH of 5.5 at an agitation period of 120min.

Effect of initial concentration of nickel ions was conducted at ranges of 10ppm, 20ppm, 30ppm, 40ppm, 50ppm. The percentage removal is highly effective on 10ppm initial ion concentration, after which percentage removal decreases gradually.

Results obtained were as follows:

Table 2: Effect of initial concentration of nickel ions on % removal of nickel ions by pongamia pinnata seed shell adsorbent

pH	Adsorbent Dose(grams)	Contact Time(min)	Initial Metal Ion Concentration(ppm)	Final Concentration(ppm)	% Removal
5.5	3	120	10	0.036	99.92
5.5	3	120	20	0.167	99.66
5.5	3	120	30	0.357	99.3
5.5	3	120	40	1.626	96.75
5.5	3	120	50	2.497	95

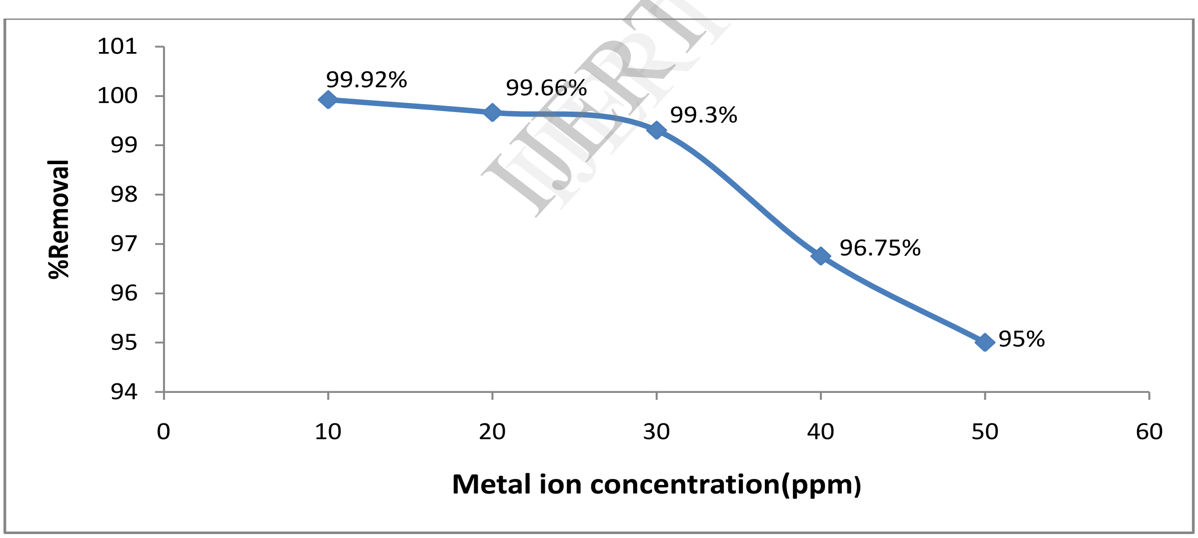

Fig 2: Effect of initial metal ion concentration on % removal of nickel ions by pongamia pinata seed shell adsorbent.

c. Effect of amount of adsorbent:

As the amount of adsorbent was increased for nickel ion removal, the available number of vacant sites for adsorption also increased. Hence adsorption of metal ion significantly increased with an increase in amount of adsorbent. The studies were conducted at room temperature with an initial metal ion concentration of 50ppm,with pH of 5.5 at an agitation period of 120min.

Effect of amount of adsorbent was conducted at adsorbent doses of 0.6g, 1.2g, 1.8g, 2.4g, and 3g respectively for 300ml of stock solution. Removal is highly effective at the dose of 3g, and the removal efficiency decreases gradually with decrease in adsorbent dosage.

Results obtained were as follows:

pH	Adsorbent Dose(grams)	Contact Time(min)	Initial Metal Ion Concentration(ppm)	Final Concentration(ppm)	% Removal
5.5	0.6	120	50	32.424	35.15
5.5	1.2	120	50	16.40	67.2
5.5	1.85	120	50	7.678	84.64
5.5	2.4	120	50	4.023	91.95
5.5	3.0	120	50	2.497	95

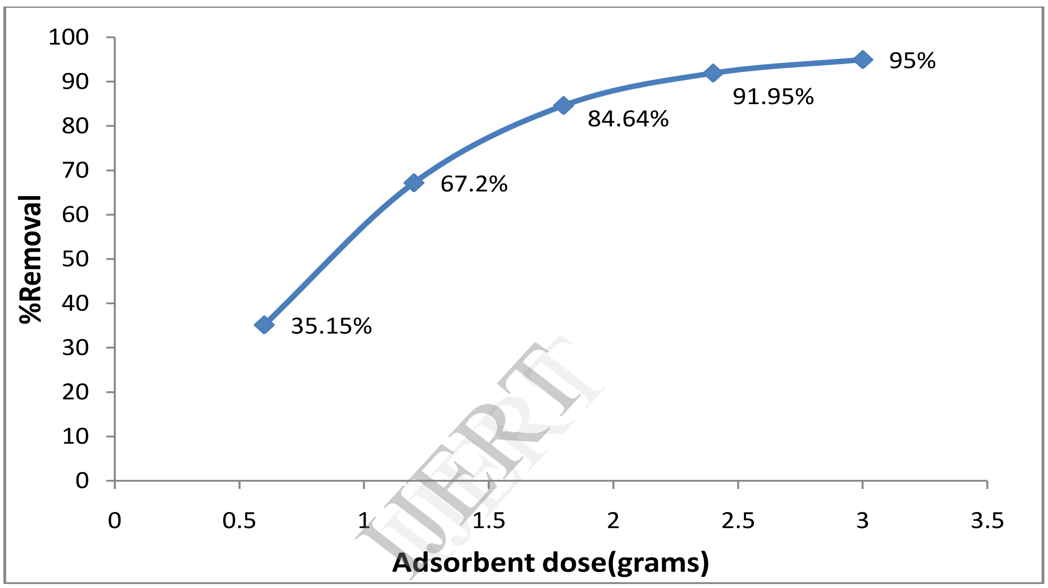

Fig 3: Effect of amount of adsorbent on % removal of nickel ions by Pongamia Pinnata seed shell Adsorbent

d. Effect of contact time:

The metal ions adsorbed on to the vacant sites of adsorbent as soon as it came in contact with it. In the beginning, large numbers of active sites were available for adsorption so the removal of nickel ions increased. As time passed the sites were filled up and they attained saturation in about 2 hours. The removal of metal ion is rapid, but then gradually decreased with time till it reaches equilibrium.

Effect of contact time was conducted at time ranges of 30min, 60min, 90min, 120min, 180min. Removal is highly effective in the range of 30 to 60min beyond which efficiency decreases with increase in contact time.

Results obtained were as follows:

pH	Adsorbent Dose(grams)	Contact Time(min)	Initial Metal Ion Concentration(ppm)	Final Concentration(ppm)	% Removal
5.5	0.6	30	50	32.424	96.44
5.5	1.2	60	50	16.40	95.76
5.5	1.85	90	50	7.678	95.25
5.5	2.4	120	50	4.023	95.28
5.5	3.0	180	50	3.0	93.37

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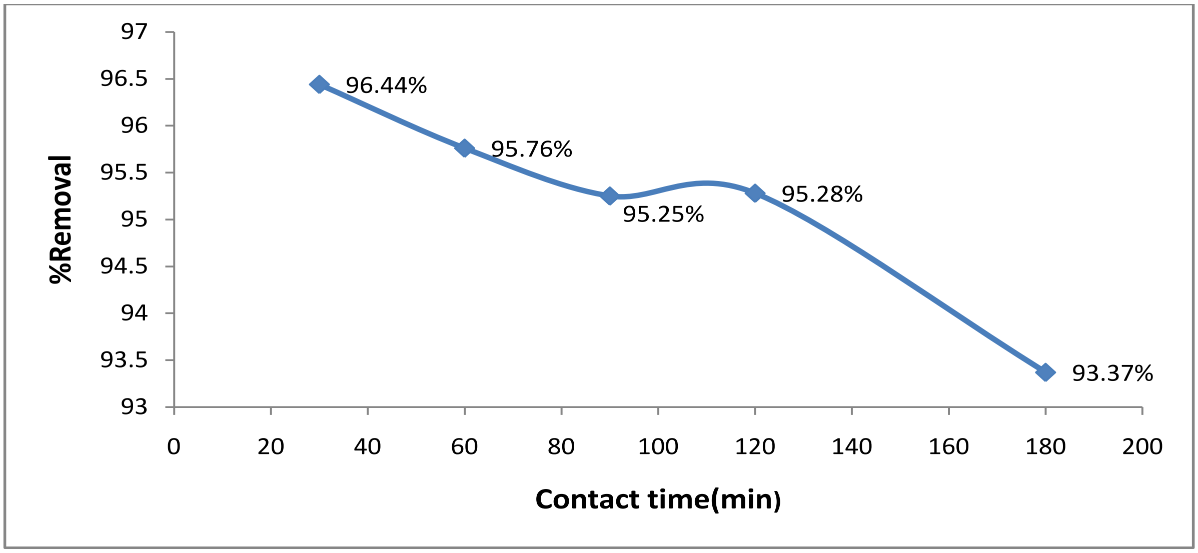

Fig 4: Effect of contact time on % removal of nickel ions by Pongamia Pinnata seed shell Adsorbent

e. Adsorption Isotherms:

To understand the effect of various factors on adsorption, different adsorption isotherms are available in literature. The adsorption isotherm provides a relationship between the concentration of metal ions in solution and the amount of metal ions adsorbed onto the adsorbent when both phases are at equilibrium. The shape of an isotherm can usually predict to know whether the adsorption is favorable or unfavorable. For the present study, the langmuir adsorption isotherm tested to fit the experimental data. The Langmuir adsorption isotherm is based on an assumption of monolayer coverage of the adsorbate on the surface of adsorbent. The Langmuir adsorption isotherm was used to describe the observed sorption of copper ions and is as shown by the following equation.

$$\frac{C_e}{q_e} = \frac{1}{b\theta} + \frac{C_e}{\theta}$$

Where, θ is the measure of adsorption capacity (mgg-1) under the experimental conditions and b is a constant related to the energy of adsorption. Following figure shows the Langmuir isotherm for copper adsorption.

Adsorption capacity measurement:
The absorption capacity of the adsorbent was measured using following relation;

$$q = \frac{V(C_i - C_f)}{M}$$

Where, q is the absorption capacity, V is volume of sample, Ci is initial concentration, Cf is final concentration and M is the amount of adsorbent used.

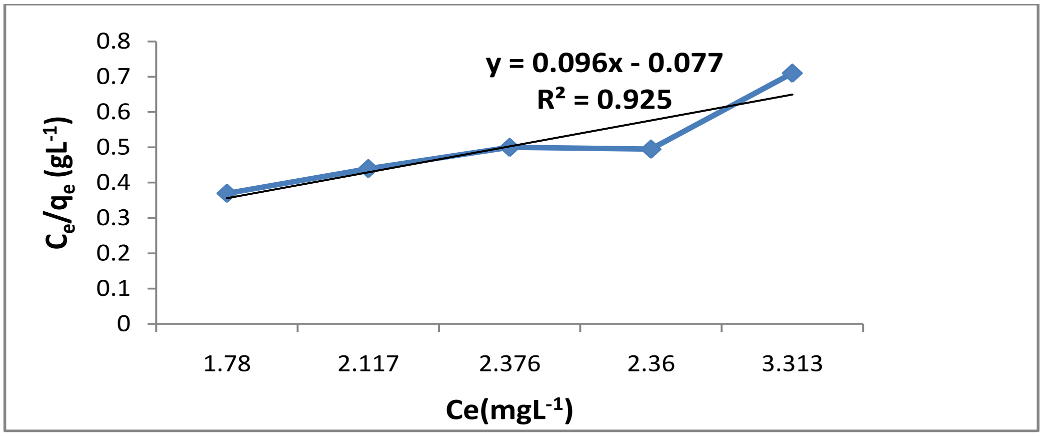

Fig 5: Langmuir adsorption isotherm for adsorption of nickel ion onto Pongamia Pinnata seed shell Adsorbent.

The value of R^2 are used to select the best adsorption isotherm model. Correlation coefficient (R^2) for Langmuir adsorption isotherm is 0.925.

The essential feature of the Langmuir equation can be given in terms of a dimensionless separation parameter R_L. The values of constants indicate favourable conditions for adsorption. Langmuir type model presupposes homogeneity of the adsorbing surface and no interactions, involving uniform energies of adsorption on the surface and no transmigration of metal ion in the plane of the surface. R_L is given as

$$R_L = \frac{1}{1 + bC_i}$$

Where, b is the Langmuir constant, C_i is the initial concentration. R_L indicates the shape of the isotherm ($R_L>1$, unfavourable, $R_L=1$ linear, $0<R_L<1$ favourable, $R_L<0$ irreversible). For the present study the value of R_L is 0.011 which indicates that adsorption is favorable.

2. COLUMN STUDIES

The dynamic behavior of a packed bed column is described in terms of breakthrough curve. A plot of effluent concentration versus time is referred as breakthrough curve. The breakthrough profile of nickel ions adsorption on Pongamia Pinnata seed shell adsorbent for a given flow rate and bed height are shown in following figures.

a. Initial metal ion concentration = 25 ppm
Depth of adsorbent = 10cm
Flow rate = 3 ml/min
Adsorbent dosage = 20gm
pH = 5

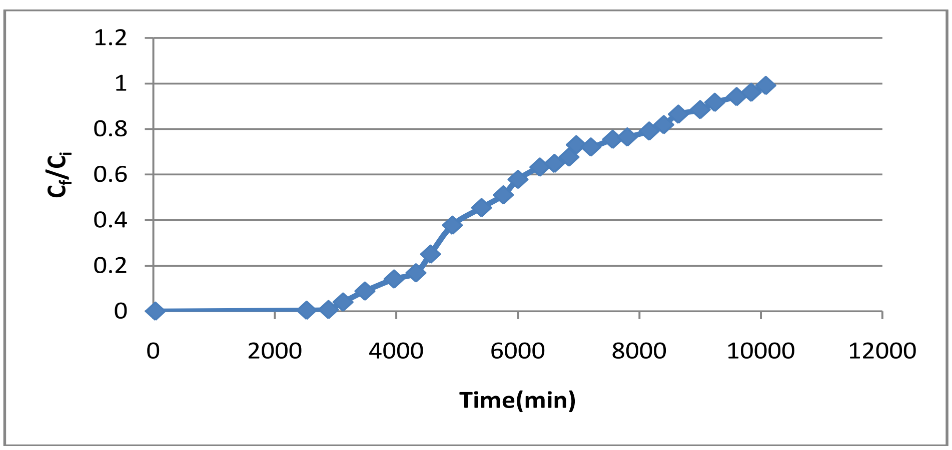

Fig 6: Breakthrough curve for adsorption of nickel at initial metal ion concentration of 25ppm.

b. Initial metal ion concentration = 50ppm
Depth of adsorbent = 10cm
Flow rate = 3 ml/min
Adsorbent dosage = 20gm
pH = 5

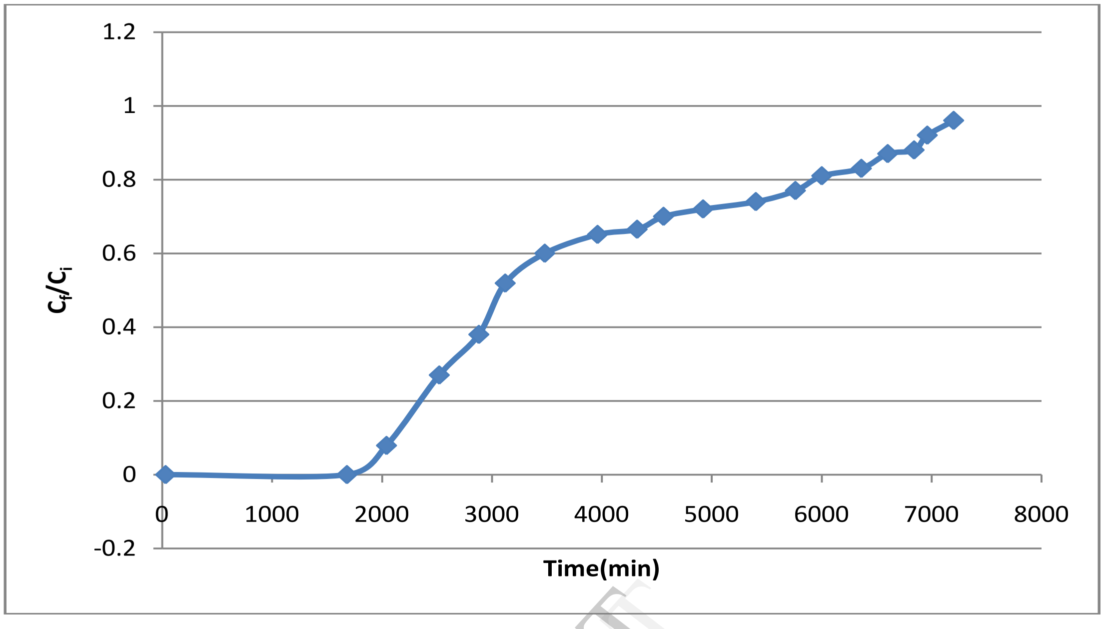

Fig 7: Breakthrough curve for adsorption of nickel at initial metal ion concentration of 50ppm.

c. Initial metal ion concentration = 75 ppm
Depth of adsorbent = 10cm
Flow rate = 3 ml/min
Adsorbent dosage = 20gm
pH =5

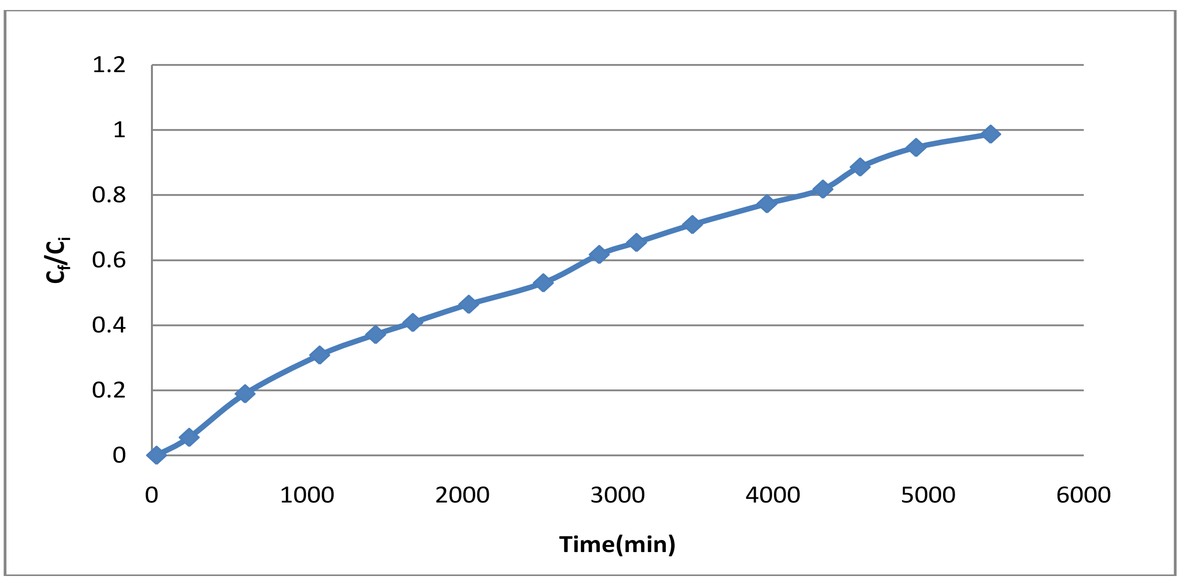

Fig 8 : Breakthrough curve for adsorption of nickel at initial metal ion concentration of 75ppm.

d. Initial metal ion concentration = 100 ppm
Depth of adsorbent = 10cm
Flow rate = 3 ml/min
Adsorbent dosage = 20gm
pH =5

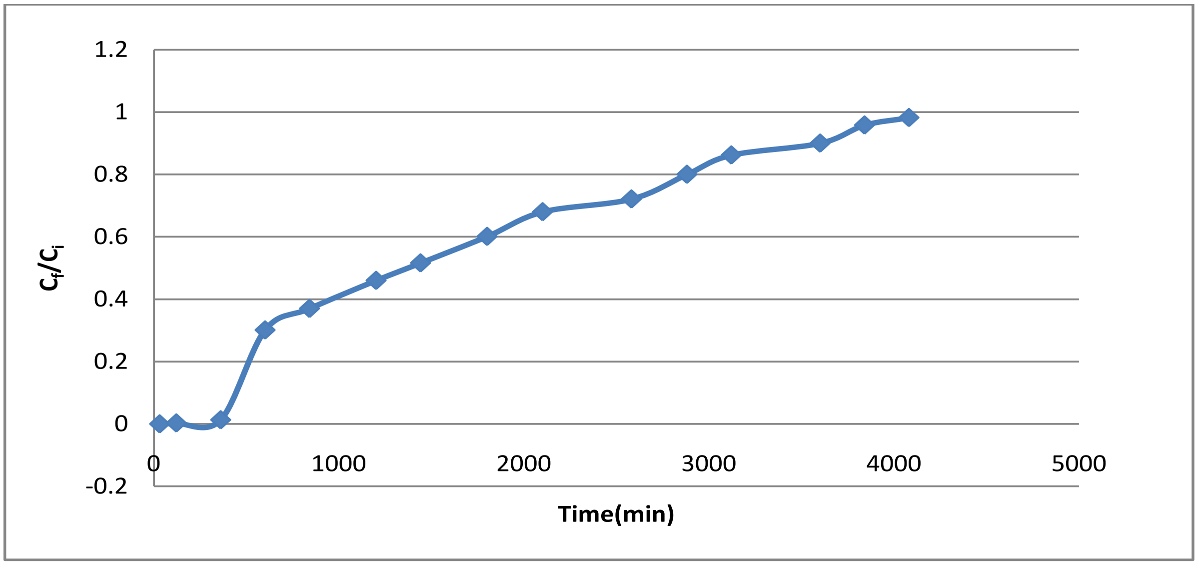

Fig 9: Breakthrough curve for adsorption of nickel at initial metal ion concentration of 100ppm.

IV. CONCLUSION

The Pongamia Pinnata seed shell is a cheap and effective adsorbent for the removal of nickel ions from wastewater without requiring any pretreatment. Activation of the adsorbent is not required hence, only drying and size reduction costs are considered. Experimental results showed that maximum removal of nickel ion by Pongamia Pinnata seed shell at optimum parameters were pH 8 with adsorbent dose of 3g/300ml, contact time of 30 to 60minutes and 10ppm initial metal ion concentration. Finally to increase the efficiency in removing toxic metals from Industrial effluents, this adsorption process can be used in line wherever an individual metal ion is generated in the waste water.

The present study investigated the performance of the column in removing nickel ions from aqueous solutions. The results showed that the potential use of Pongamia Pinnata seed shell for the adsorption of nickel from aqueous solution.. The Pongamia Pinnata seed shell adsorbent may reduce the level of nickel upto 99%. From results, it is suggested that this adsorbing material can be effectively used for the removal of heavy metal ions from any industrial effluent.

As the pH of industrial wastewater varies, the applicability of this adsorbent to remove the toxic metals at different pH needs further research. Furthermore, there is a need to explore the effect of different operating parameter such as pH, adsorbent dose, loading volume and mechanism of action. The best model to explain the metal adsorption behavior by the sorbent was the Langmuir's isotherm model and according to its results, the adsorption is a single-layer process.

Further research is required to utilize this adsorbent for maximum removal of toxic metals. Different modes of operation can be tried for maximum adsorption of toxic metals. Also behavior of the adsorbent need to be tested with real industrial effluents where different types of toxic metals are present and analyzing the adsorption capacity.

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