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Removal of Copper from Simulated Wastewater Using Pongamia Pinnata Seed Shell as Adsorbent

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Abstract
The adsorption process is being widely used by various researchers for the removal of toxic metals from waste streams and activated carbon has been frequently used as an adsorbent. Despite its extensive use in the water and wastewater treatment industries, activated carbon remains an expensive material. In recent years, the need for safe and economical methods for the elimination of toxic metals from contaminated waters has necessitated research interest towards the production of low cost alternatives to commercially available activated carbon. Presence of copper in the water used for various activities affects living beings in its own way depending on its concentration. Considering the effects of high dose of copper concentration on humans and animals, it is important to remove this metal ion from industrial effluent before discharge to environmental stream. In this regard Pongamia pinnata seed shell is proven to be an effective and promising adsorbent. Removal of copper ion from aqueous and industrial waste water depends on pH, concentration of metal ion, concentration of adsorbent, contact time and agitation. Equilibrium adsorption data was tested for the Langmuir equation. Adsorption isotherm studies indicated that Langmuir model fits better for the present case.
Keywords: Adsorption, copper ion, *Pongamia pinnata* seed shell, adsorption isotherm.

I. INTRODUCTION

Environmental pollution is currently one of the most important issues facing humanity. It was increased exponentially in the past few years and reached alarming levels in terms of its effects on living creatures. The presence of toxic metals in the environment specifically in various water resources is of major concern because of their toxicity, non-biodegradable nature and threat to human, animal and plant life. Electroplating, painting, metallurgical, mining, textile, municipal incineration and chemical manufacturing industries all generate toxic toxic metals of various levels.[1] Stringent regulations have been made by different countries for safe discharge standards of these effluents.

Copper ions and other toxic metal ions enter into water and soil from different wastewater sources and thus pollute the water streams and soil. A number of crops and aquatic plants take up these heavy metals and accumulate within them. When humans consume food and water with copper concentration exceeding the permissible limits then problems like nausea, gastrointestinal disturbance, vomiting, liver or kidney damage etc is likely to be caused. [3] Researchers worldwide have developed a number of methods such as chemical precipitation, oxidation or reduction, ion exchange, filtration, membrane separations, electrochemical treatment, reverse osmosis, evaporation recovery, coagulation etc., for removing the toxic metals from industrial effluents before discharging into aquatic streams. [2] The conventional treatments produce toxic chemical sludge and in turn disposal or treatment becomes costly and not ecofriendly. Adsorption is considered as one of the effective and economical technology compared to others for removal of toxic metals from effluents. Activated carbon is widely used in many industries as an effective adsorbent for toxic metal removal. The cost of activated carbon is high. Usually industries strive for low cost methods for waste water treatment and hence may opt for low cost adsorbents. Though researches reported shows varieties of low cost adsorbents, all adsorbents are not available easily throughout the world. Hence, considering the availability of materials in particular region, the adsorbents may be suitably selected to cater the needs of that region.[6]

All parts of the *Pongamia pinnata* tree have application in medicinal and other areas including the most important one the production of biofuel from *Pongamia pinnata* seeds. Hence, large numbers of trees are grown at farms and availability of the seed shells is more. The present study shows the feasibility of using adsorbent prepared from *Pongamia pinnata* seed shell for removal of copper ions from simulated wastewater with parameters as pH, concentration of adsorbent, concentration of metal ion and contact time. [5]

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 copper sulphate (Nice Chemicals) of required quantity was dissolved in distilled water to prepare stock solution.3.927grams of $CuSO_45H_2O$ was added in the 100ml of distilled water in 1000ml volumetric flask. It was dissolved by shaking and the volume was made up to the mark. Copper concentration of this solution 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/L 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. The sample solution was passed through the adsorption column by gravitation. The flow rate was kept constant by controlling the stopper value. Inlet of the column was connected to the 25 liter feed bucket with the plastic tubing. Afterwards, the pH has been adjusted to 5 with H_2SO_4 and NaOH solutions. 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 copper 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. So, adsorbent particles quickly participate in practice and absorb. It increases the absorption rate. 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 copper 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

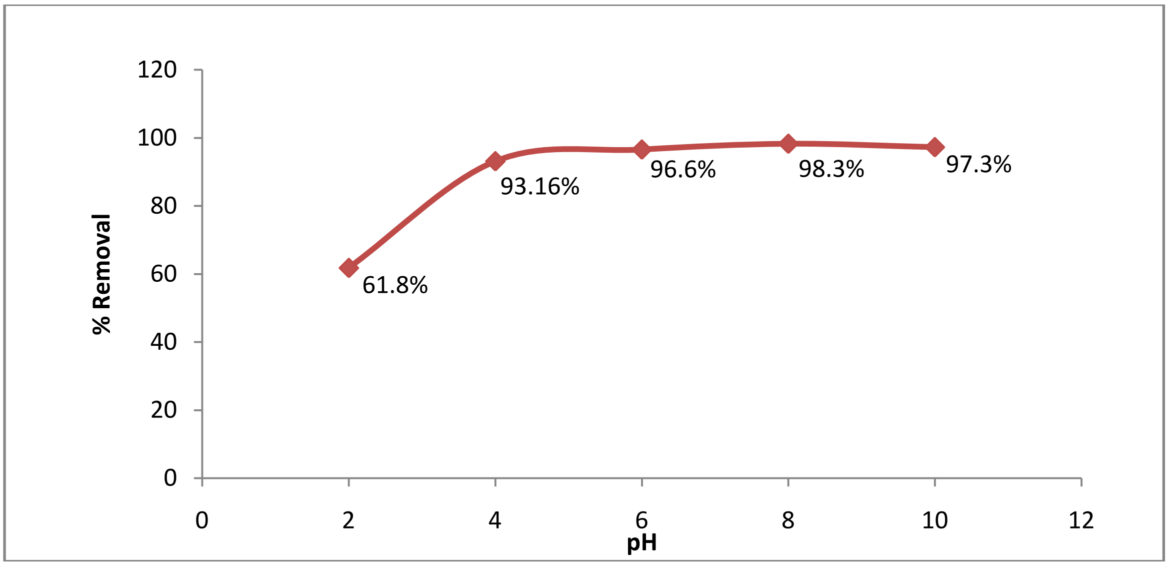

Fig 1: Effect of pH on % removal of copper ions by pongamia pinnata seed shell adsorbent.

b. Effect of initial concentration of copper ions:

The adsorption of copper 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 copper 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 3: Effect of initial metal ion concentration on % removal of copper 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	0.025	99.95
5.5	3	120	20	0.030	99.94
5.5	3	120	30	0.161	99.00
5.5	3	120	40	0.630	98.00
5.5	3	120	50	1.354	97.30

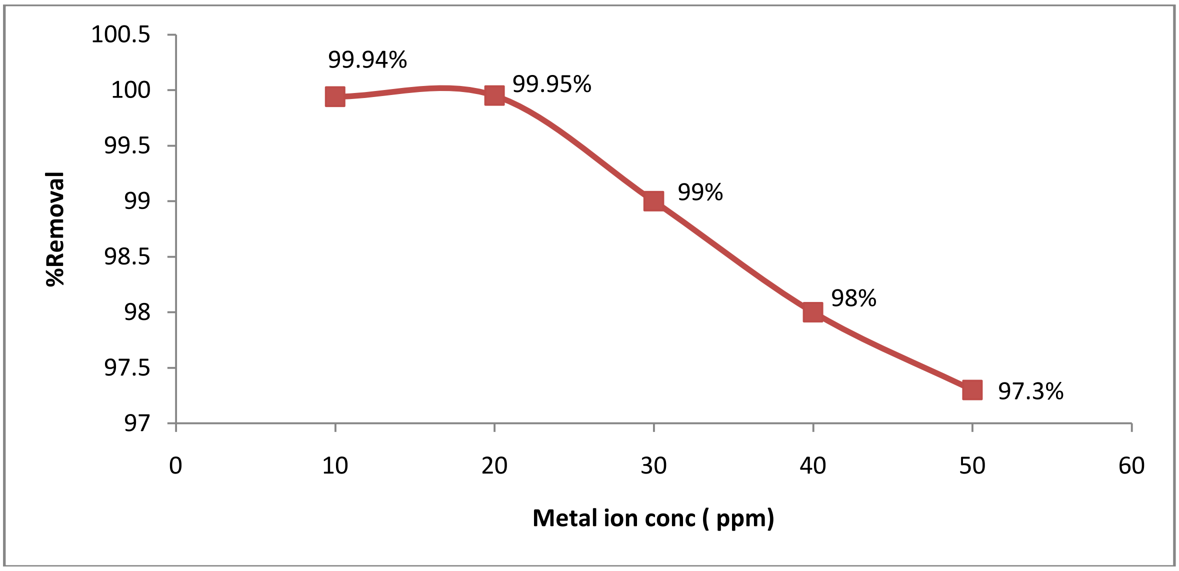

Fig 2: Effect of initial metal ion concentration on % removal of copper ions by pongamia pinnata seed shell adsorbent.

c. Effect of amount of adsorbent:

As the amount of adsorbent was increased for copper 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:

Table 4 : Effect of amount of adsorbent on % removal of copper 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	0.6	120	50	15.46	69.07
5.5	1.2	120	50	5.10	89.70
5.5	1.85	120	50	1.72	96.50
5.5	2.4	120	50	1.57	96.80
5.5	3.0	120	50	1.35	97.30

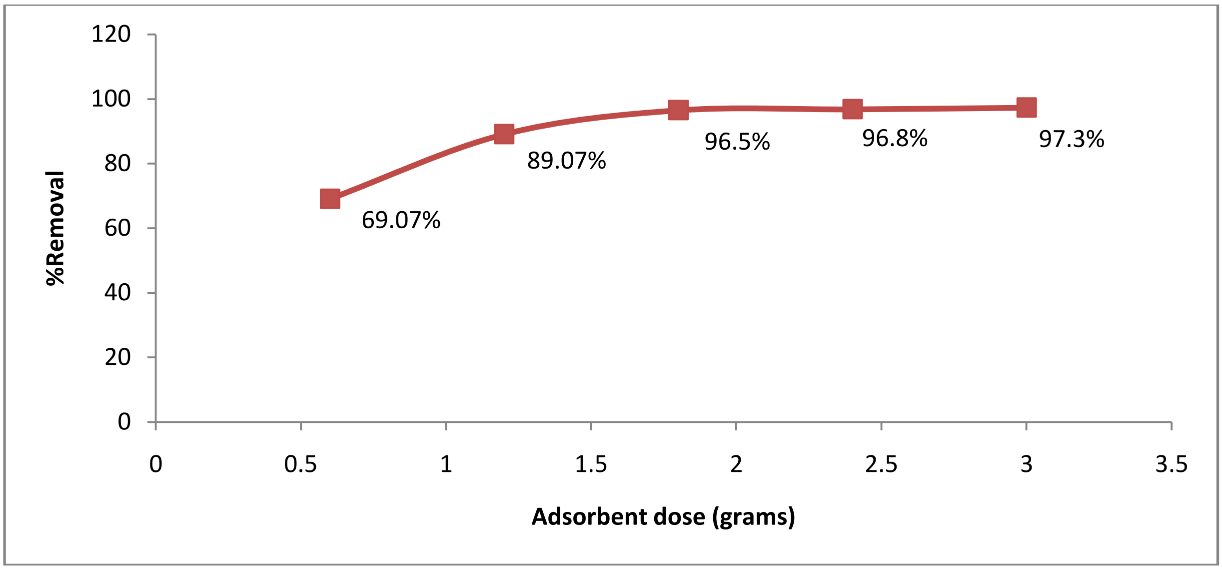

Fig 3: Effect of amount of adsorbent on % removal of copper ions by Pongamia Pinnata seed shell Adsorbent.

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 copper ions increased. As time passed the sites were filled up and they attained saturation. The removal of metal ion is rapid, but then gradually decreased with time till it reaches equilibrium. The studies were conducted at room temperature with an initial metal ion concentration of 50ppm, with pH of 5.5 at constant adsorbent dose of 3gm/300ml solution.

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 follow

Table 5: : Effect of contact time on % removal of copper 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	0.6	30	50	0.339	99.30
5.5	1.2	60	50	0.414	99.17
5.5	1.85	90	50	0.992	98.00
5.5	2.4	120	50	1.278	97.40
5.5	3.0	180	50	2.183	95.60

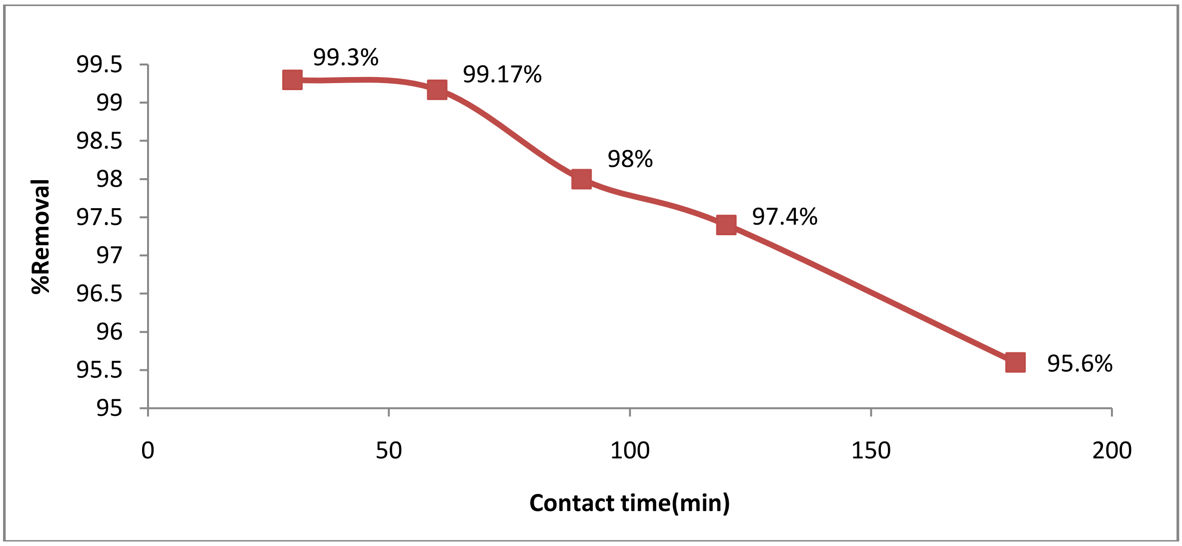

Fig 4: Effect of contact time on % removal of copper ions by Pongamia Pinnata seed shell Adsorbent.

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.

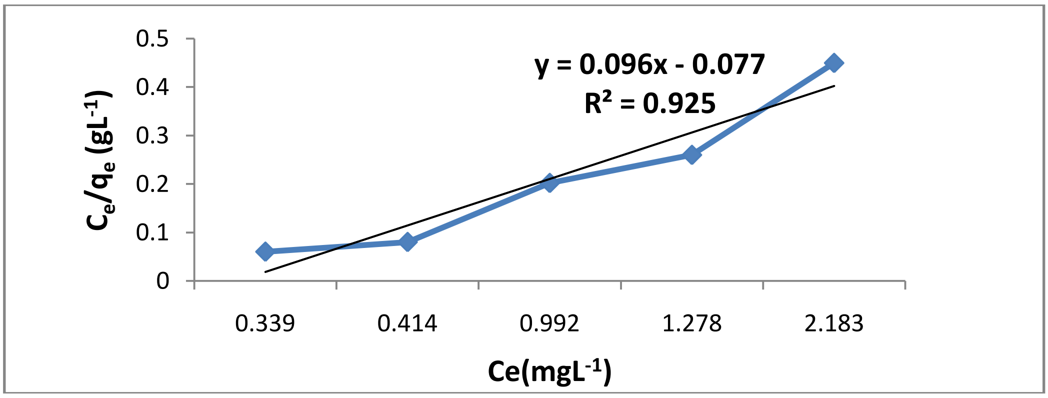

Fig 5: Langmuir adsorption isotherm for adsorption of copper ion onto Pongamia Pinnata seed shell Adsorbent.

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 copper 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

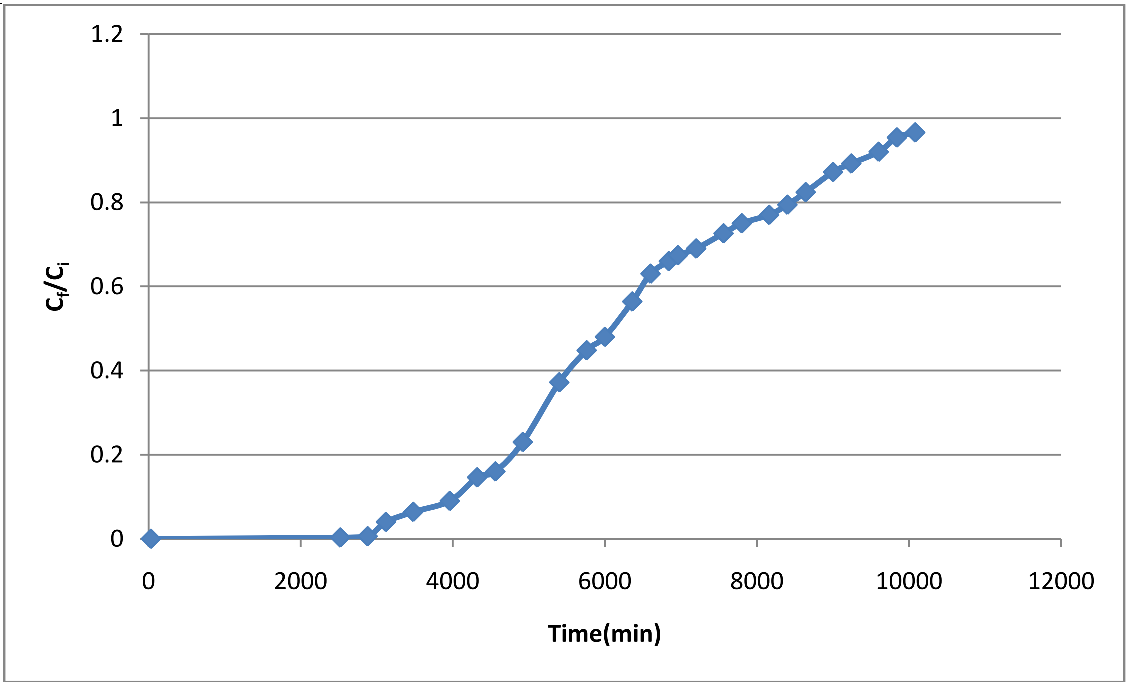

Fig 6: Breakthrough curve for adsorption of copper at initial metal ion concentration of 25ppm.

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

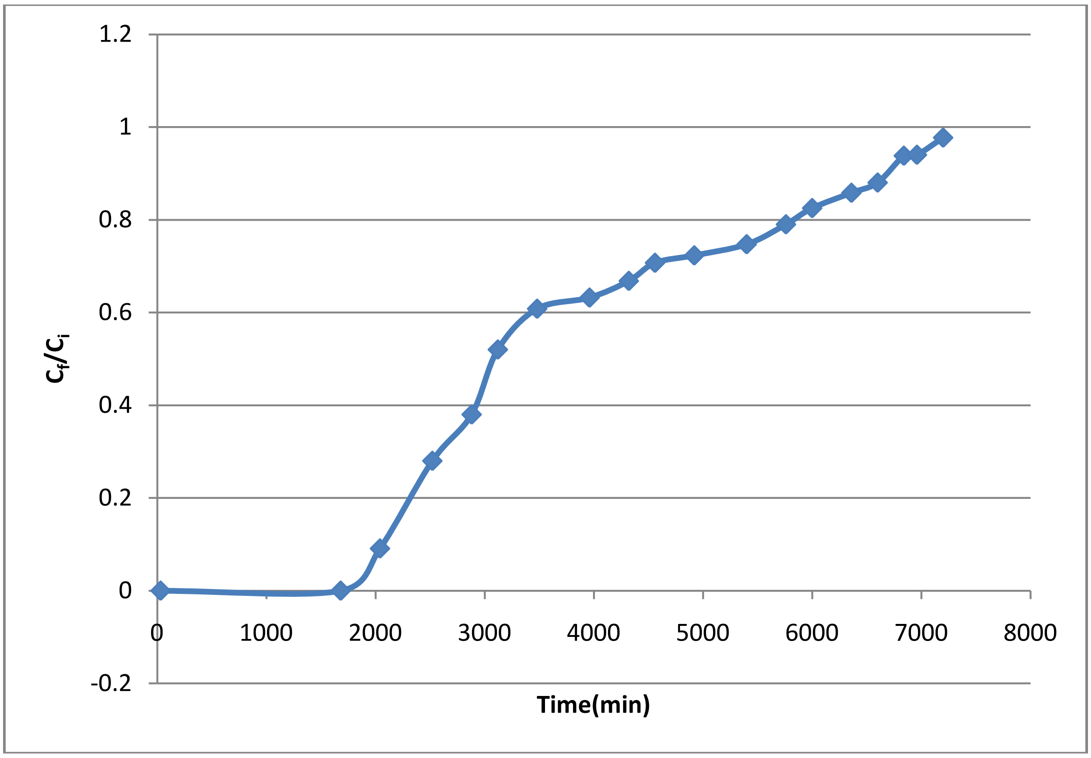

Fig 7: Breakthrough curve for adsorption of copper at initial metal ion concentration of 50ppm.

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

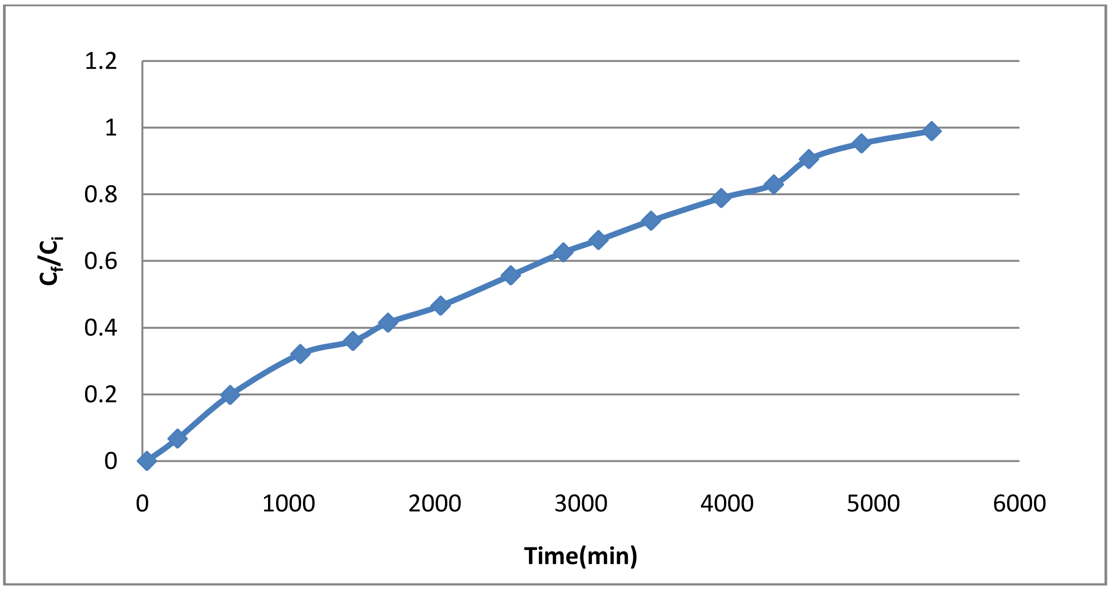

Fig 8 : Breakthrough curve for adsorption of copper at initial metal ion concentration of 75ppm.

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

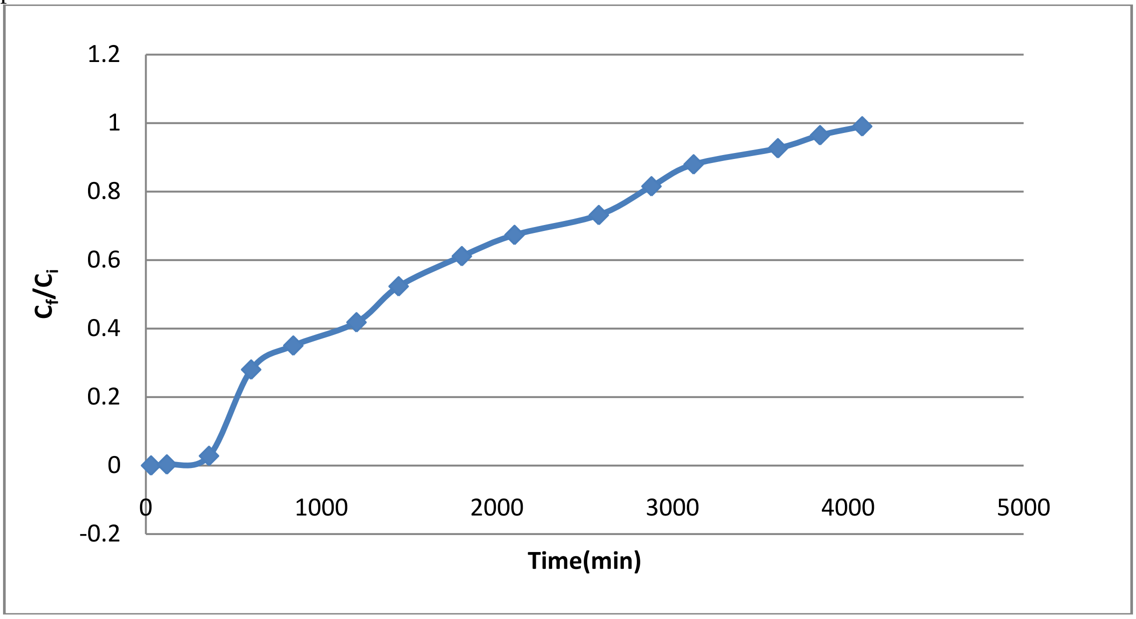

Fig 9 : Breakthrough curve for adsorption of copper at initial metal ion concentration of 100ppm.

IV. CONCLUSION

The Pongamia Pinnata seed shell is a cheap and effective adsorbent for the removal of copper ions from waste water 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 copper 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 adsorption isotherm followed the Langmuir model due to the fact that it shown a good fitting to the experimental data, according to its results, the adsorption is a single-layer process.

The continuous down flow process designed in this study for adsorption in order to remove the copper ions has a good potential to remove copper from wastewater samples in practical application. It is concluded that Pongamia Pinnata seed shell may act as an efficient adsorbing material for the adsorption of copper ions from simulated solution. The Pongamia Pinnata seed shell adsorbent may reduce the level of copper more than 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. Furthermore, there is a need to explore the effect of different operating parameter such as pH, adsorbent dose, loading volume and mechanism of action.

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

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