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Kinetic Modeling of Continuous Column study for the removal of Cu (II) and Fe (II) using orange peel as an adsorbent

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Abstract : This paper deals with the removal of Cu (II) and Fe (II) in continuous column mode operation which involves the study effect of flow rate and effect of bed height at a fixed metal ion concentration of 100 ppm for the adsorption of Copper and Iron on orange peel adsorbent. The equilibrium adsorption capacity of orange peel for Copper and Iron was examined by various models like isotherm modelling, Kinetic modelling and thermodynamic modelling studies. The results prove that the biosorption capacity of orange peel for Copper and Iron are endothermic, efficient, and very rapid in nature. The overall efficiency of the orange peel was studied and optimized the above parameters on the basis of the results of batch mode experimentation work. Various standard isotherm models like Thomas model, BDST (Bed Depth Service Time) model and Adam-Bohrat models are verified for the column mode study. BDST model equation has been fitted with higher R^2 values of 0.973 and 0.984 for Cu (II) and Fe (II) when compared to Adam-Bohart model and Thomas model.

Key words : Effect of flow rate, Effect of bed height, Biosorpion capacity, Bed Depth Service time model, Adam-Bohart model, Thomas model.

1. Introduction

The contamination of surface waters by heavy metal ions has become a serious ecological issue and health problem due to their toxic effect even in low concentrations. Heavy metals are of special concern because they are non-degradable and thus persistent. Heavy metal ions such as cobalt, copper, chromium, nickel, palladium, lead, zinc are detected in the waste streams from the mining operations [1], tanneries [2], electronics [3], electroplating, batteries [4] and petrochemicals [5] industries has major effects on the human and aquatic life [6]**.**Water pollution remains a major problem in the environment due to the development of urbanization and industrialization which have contributed to the large scale of pollution for both human and aquatic life. The wastewater is discharged into the streams. Wells, rivers and other water bodies without proper treatment. The pollution depreciates the land values, increases the municipal cost, operational cost and cause adverse biological and human health effects. Heavy metals are non-biodegradable in nature and cause and their presence in the water streams leads to bioaccumulation in living organisms causing health problems to animals, plants and human life [7]. Industrial effluents containing enormous quantities of inorganic and organic chemical wastes, which are steadily become more difficult to treat by on-going conventional methods[16-21]. A number of conventional treatment technologies such as Chemical precipitation, ion exchange, electro dialysis, membrane separations, reverse osmosis, and solvent extraction and adsorption have been considered for treatment of wastewater contaminated with organic substances. Among them adsorption is found to be the most effective method [8]. Adsorption is found to be superior to any other treatment methods because of the simplicity of design, ease of operation, capability for adsorbing a broad range of different types of adsorbate concentrations efficiently. Commercial activated carbon is regarded the most effective material for controlling the organic load [5]. Many conventional methods have been developed for the removal of heavy metals from effluents such as sedimentation, ion exchange, membrane filtration, electrochemical processes, chemical precipitation, reverse osmosis and solvent extraction. But these methods are expensive or inefficient when the concentrations of metals are low (below 100ppm) and there is generation of large quantities of wastes. Development of eco-friendly, efficient and low - cost processes is the need of the hour and in this aspect, adsorption is a versatile technology with the advantages of high efficiency and selectivity for adsorbing metals in low concentrations, recycling of the adsorbent and minimization of the sludge generation [9,10].

Batch adsorption tests/studies provide information on adsorption equilibrium characteristics and adsorption kinetics, which is important in determining the effectiveness of the adsorbent in removing solute from solution. However, batch operations are not often economical in practice and the data obtained from these is not sufficient to give accurate scale up data required in the design of industrial adsorption columns. Therefore, column studies have to be performed, whereby the most important parameter to be determined is the column breakthrough curve, which determines the operating life span of the fixed adsorbent bed [11].All the design models of fixed bed columns are based on determining the breakthrough curves for the specific system. The breakthrough curve depicts the time or volume of the effluent treated versus the effluent concentration or dimensionless concentration, C/C_o, at different bed heights; hence it describes the performance of any fixed bed column. The characteristic shape of this curve will depend on the equilibrium between the solid and liquid phase, based on the kinetic adsorption process, which is divided into four stages; that is, diffusion in bulk fluid, external mass transfer, intra particle diffusion and micropore diffusion. From this curve it is possible to determine the time the adsorbent material will be able to sustain removing a specified amount of solute from solution before it needs regeneration or replacement, this period of time is called the service time of the bed. Figure 1 can be used to describe how the breakthrough curve is established in a fixed bed column. Solution at solute concentration, $C_o$enters the column and a concentration gradient is established within a finite zone that is the adsorption or mass transferzone. In this zone the concentration of solution changes from C_o to C_e, where C_e is close to zero. The loading of solute on the solid (adsorbent) also increases within the mass transfer zone until the solid is completely saturated. Since the system is a dynamic one, the mass transfer zone moves steadily from the influent end of the column to the outlet end. The below figure shows how the mass transfer zone moves along the column, from column , where the adsorbent is not yet in contact with the solution, to column where the adsorbent is completely exhausted after time T_c. Time T_b represents the breakthrough point, where tracesof solute start to be detected in the effluent from the column outlet.

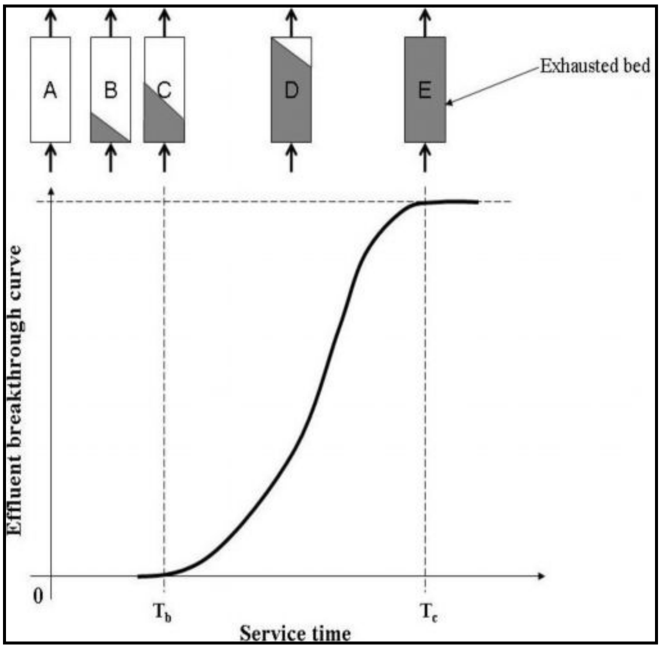

Figure 1 Break through curve concept

2. Materials and Methods

2.1 Chemicals used

All the chemicals used in this experiment were in their pure form. The stock solution of Cu(II) was prepared in laboratory by dissolving 1 gram of copper salt in 1000 ml of deionized water. The same stock solution is used for various experimental runs. The pH value of the solution is adjusted by the addition on 0.1N HCl and 0.1 N NaOH solutions.

2.1.1 Preparation of adsorbent by chemical treatment

The orange peel was collected from the local juice shops of Dehradun and then it is washed thoroughly by tap water and followed by rinsing with deionized water. Then the peel was treated with 0.1 N HNO_3solutions for the protonation purpose for 5 hours. Then the peel was dried in sunlight for 5 days and followed by drying in oven at 105°Cfor 3 hours to remove the moisture completely. Thus in the last stage the peel was crushed, ground and passed through 1 mm sieve to get uniform sized adsorbent.

2.1.2 Instruments used for Adsorbent / biosorbent characterization

A standard pH meter was used for the adjustment of pH of the simulated metal ion solution. After the adsorption process the final concentration of the solution was measured by standard Atomic Absorption Spectroscope (AAS) equipment.

2.1.3 Column Studies

Continuous flow operation experiments were conducted in a transparent cylindrical plastic (PVC) column having dimensions of (4 cm internal diameter and 100 cm height). A 20 mesh size stainless sieve was attached to the bottom of the column. Five side valves are provided at a distance of 12 cm from the bottom of the column. It was marked as 12 cm, 24 cm, 36 cm, 48 cm and 60 cm from the bottom as shown in fig 3. A submersible pump of 40 W is used to pump the liquid from the bottom of the column at the desired flow rates (10ml/min, 20ml/min and 30ml/min). The 0.8μm mesh is used to hold the adsorbent particles of 1 mm size. A small globe valve is used to control the flow simulated waste water up the column to meet the suitable residence time. The solution pH is adjusted to 6 for copper and 5 for Iron metal ions. Various parameters in the column study are considered which involves the bed height of 12 cm, 24 cm and 36 cm. The metal ion concentration was varied from 100 ppm to 250 ppm. The flow rates of simulated waste water are varied from 10 ml/min to 30 ml/min.Samples were collected from the exit of the column at different bed heights at different intervals of time until the achievement of equilibrium. The final concentration of waste water is measured by using standard atomic absorption spectrometer equipment at various time intervals. The breakthrough curves are plotted by taking C_e/C_oon y axis and time interval (t) on x- axis. Various adsorption models are studied for predicting the adsorption behaviour. Fig 2 indicates the packed bed column in operation for the collection of the samples from different outlets.

2.1.3.1 Study of bed heights (weight of the adsorbent) and volumetric flow rate

The design of the packed bed column has been studied at a bed height of 12cm, 24cm, and 36cmwith the flow rates varying from 10, 20, 30 ml/min with an initial concentration varying from 100 to 250 ppm. The main design of the column involves the study of break through curves experimentally by plotting C_e/C_oVs t until C_e/C_o becomes 1. The design involves the study of various kinetic parameter models such as Thomas model, BDST model, and Adam –Bohart model.

Figure 2 Packed bed column in operation

2.1.3.2 Parameters & Dimensions of the packed bed column

Weight of the adsorbent added 50g, 100g, and 150g respectively (for 12 cm -50g; for 24 cm -100 g; and for 36 cm -150 g)

Inner Diameter of the column: 4cm
Total height of the column =100cm
Submersible pump used for sending the effluent into the column = 40 Watts.
Initial Metal Con of the metal ions Cu and Fe (C_o) = 100 ppm
Effect of volumetric flow rate -10, 20, 30 ml/min
Effect of (bed height) -12cm, 24cm, 36cm

Fig 3 Experimental setup of Continuous flow column

2.1.4 Desorption and regeneration methodologies

The desorption and regeneration methodologies were conducted to check the possibilities of the reuse of adsorbent and possible recovery of concentrated heavy metals. The regeneration process involves the extensive washing of orange peel in 0.2 M HCl solution. The reaction was allowed to takes place for 30 min at a rotational speed of 150-180 rpm in an orbital shaker followed by washing with water to remove the traces of metal ions[12].

3 Results and Discussion

3.1 Mass balance equation for the control volume of the bed

Assumptions

- The bed consists of spherical adsorbents and all the adsorbents are systematically packed so that the interstitial velocity is assumed to be constant
- The resistance to heat and mass transfer both inside and outside the sorbent pellet, can be important, depending on the operational conditions, one of them is sometimes neglected in favour of the other. The models usually assume that the local rate of adsorption is instantaneous compared to transport processes.
- The physical process of adsorption is so fast relative to other slow steps (diffusion within the solid particle)

- Fluid is in plug flow, Dilute solution, Isothermal
- The mass balance equation for the bulk flow is through the bed, neglecting radial dispersion.
- Pore diffusion phenomenon is neglected in Homogeneous Surface Diffusion Model.

The mass balance equation for the bulk flow in the bed neglecting the radial dispersion is given by

$$-D_L\frac{\partial^2 c}{\partial z^2} + v\frac{\partial c}{\partial z} + c\frac{\partial v}{\partial z} + \frac{\partial c}{\partial t} + \left(\frac{1-\epsilon}{\epsilon}t\right)\frac{\partial q_p}{\partial t} = 0 \qquad \_\_\_\_1$$

for constant superficial velocity the equation becomes,

$$-D_L\frac{\partial^2 c}{\partial z^2} + v\frac{\partial c}{\partial z} + \frac{\partial c}{\partial t} + \left(\frac{1-\epsilon}{\epsilon}\right)\frac{\partial q_p}{\partial t} = 0 \qquad \_\_\_\_2$$

Where $D_L$is the axial dispersion coefficient, υ is the interstitial velocity in bed, zis the axial distance variable, tis the time, ϵis the bed porosity and qis the total sorbed phase concentration of all the pellets.

The initial conditions are given by the equations

$$c = 0 \quad z = 0 \quad t = 0 \qquad \_\_\_\_\_\_3$$

The boundary conditions at both ends of the column are given by Equations

$C = C_o \quad Z = 0 \; t > 0$ ______ 4

$$\frac{\partial c}{\partial z} = 0 \quad z = L \quad t > 0 \qquad \_\_\_\_\_\_\_5$$

The results obtained from Mat lab simulation are shown in fig 4 which indicates the nature of the graph plotted between equilibrium concentrations(mg/l) on Y-axis vs time (hrs) on X-axis

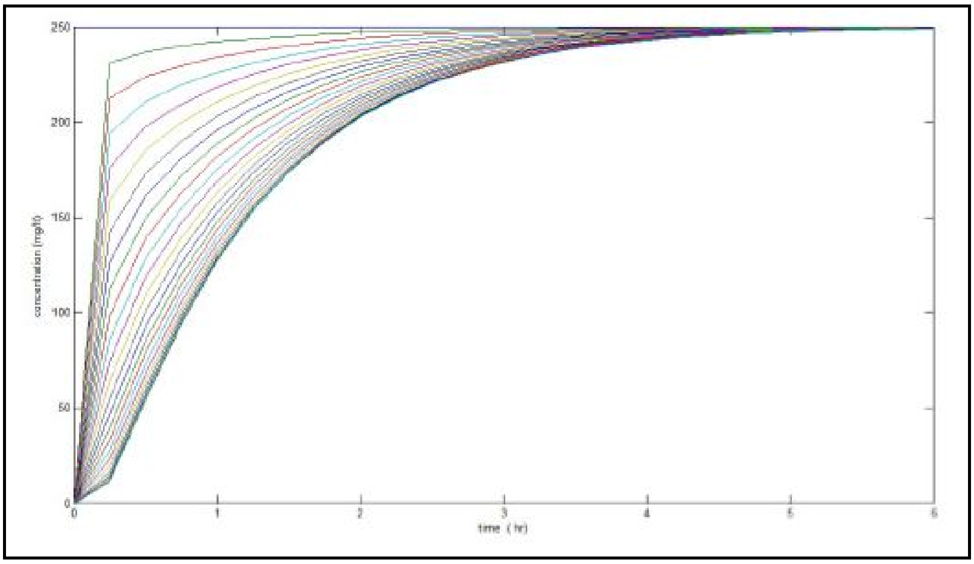

Fig 4 Simulation result for packed column: Expected nature of graph

3.2 Column mode operation results for the removal of copper and iron

Various process parameters of the column mode adsorption are considered and the effectof these parameters on adsorption was studied and the results are expressed in terms of Break through curves.

3.2.1 Effect of bed height

The adsorption of copper and iron in the packed bed column is largely dependent on the bed height (12, 24 and 36 cm of bed height were considered) which is directly proportional to the quantity of orange peel in the column.The adsorption breakthrough curves were obtained by varying the bed heights at a flow rate of 10 mL/min, an inlet metal ion concentration of 250 mg/L at a temperature of 30°C and pH 6 for copper and 5 for Iron. Faster breakthrough curves were observed for a bed height of 12 cm, while the slowest breakthrough curve was observed at a bed height of 36 cm as shown in the fig 5 for Cu (II) and fig 6 for Fe (II) using orange peel as an adsorbent. More number of binding sites will be available for the metal ion to attach which will eventually lead to the attainment of a higher bed capacity. Additionally an increased bed height resulted in more contact time being available for the metal ion to interact with the adsorbent This phenomena has allowed the metal ion molecules to diffuse deeper into the adsorbent. Subsequently, the percentage of dye removal increased when the bed height was increased [11].

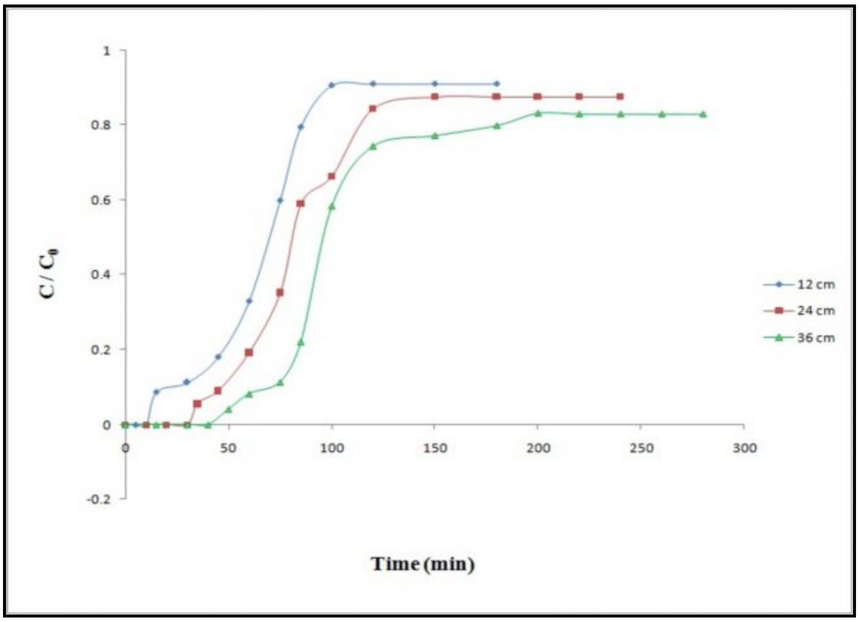

Figure 5 Effect bed height for copper ion adsorption on orange peel

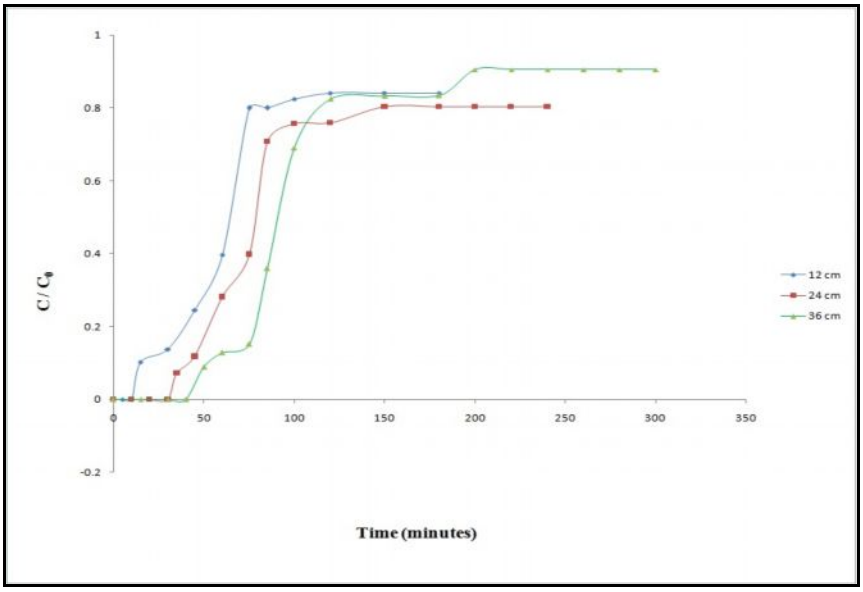

Figure 6 Effect bed height for iron ion adsorption onto orange peel

3.2.2 Effect of flow rate

The effect of flow rate on copper and iron metal ion adsorption by orange peel was investigated by varying the flow rate of the waste water inlet solution from 10 to 30 mL/min while maintaining the initial concentration and bed depth at 250 mg/L and 36 cm respectively along with temperature of 30°C and pH 6 for copper and 5 for Iron solution is maintained. Result indicates that a quicker breakthrough was achieved for higher flow rates and the slowest breakthrough curve was observed for the lowest flow rate as shown in fig 7 and 8 for Cu (II) and Fe (II) respectively. At lower flow rates, the contact time between the metal ions and adsorbent is greater which results in a slower breakthrough curve. Conversely for the higher flow rate the solution will leave the bed before the equilibrium can be reached. This will result in a decreasing amount of metal ion being adsorbed [13].

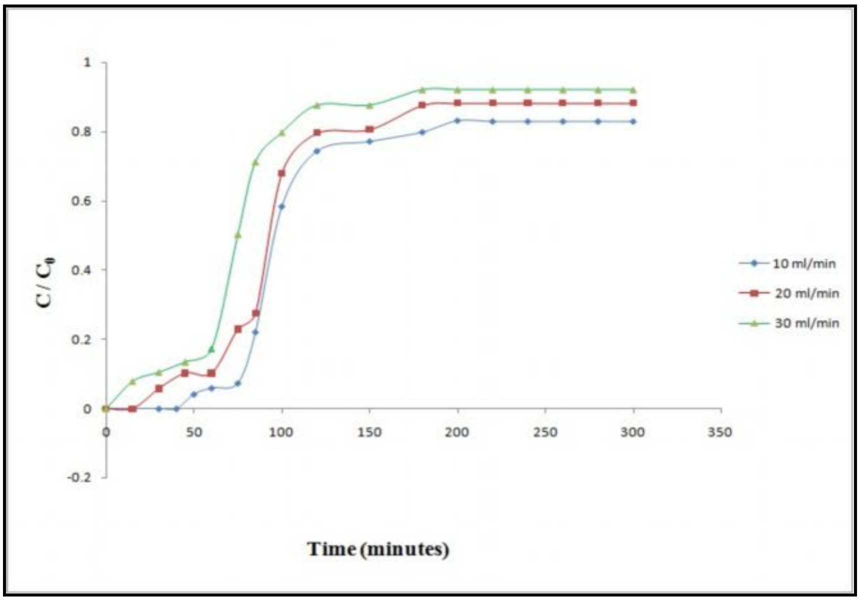

Figure 7 Effect flow rate for the adsorption of copper ion on orange peel

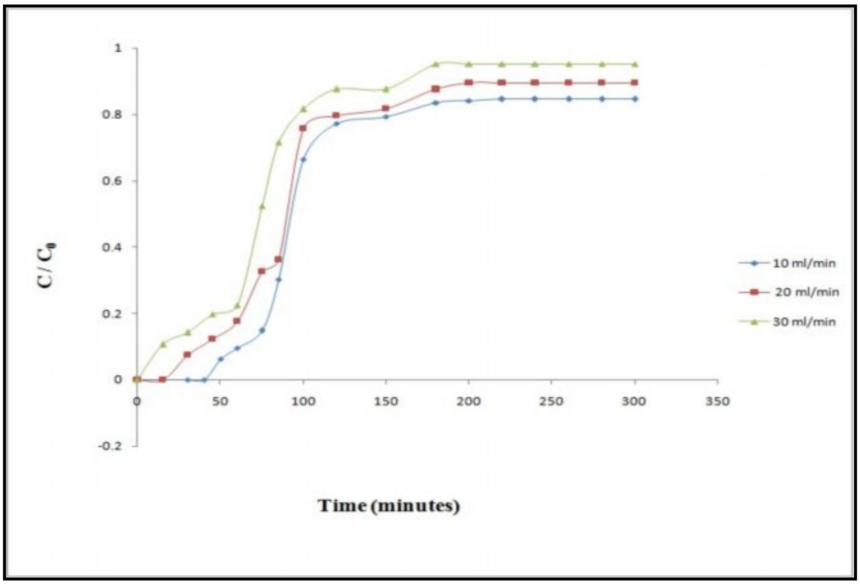

Figure 8 Effect flow rate on the adsorption of iron by orange peel

3.2.3 Effect of Initial metal ion concentration

The effects of the three initial metal concentrations (100 mg/L, 200 mg/L and 250mg/L) on the adsorption process at a constant flow rate of 10 mL/min and fixed bed height of 36 cm are shown in Fig 9 and

10 respectively for Cu (II) and Fe (II) at a temperature of 30°C ,pH 6 for copper and5 for Iron solution. It can be deduced that at a lower inlet concentrations a slower breakthrough curve and the highest treated volume will be obtained. The slow transport of metal ions onto orange peel was due to the lower concentration gradient and resulted in a slower breakthrough curve. Conversely at higher concentration of metal ions has shown a higher driving force for the metal ions to overcome the mass transfer resistance in the liquid phase. Consequently the quick saturation of the available binding sites for metal ions has caused the breakthrough time to decrease with the increasing inlet metal concentration. Apart from achieving a quicker breakthrough curve, the adsorption capacity of the bed also increased simultaneously with the increase of initial metal ions concentration [13].

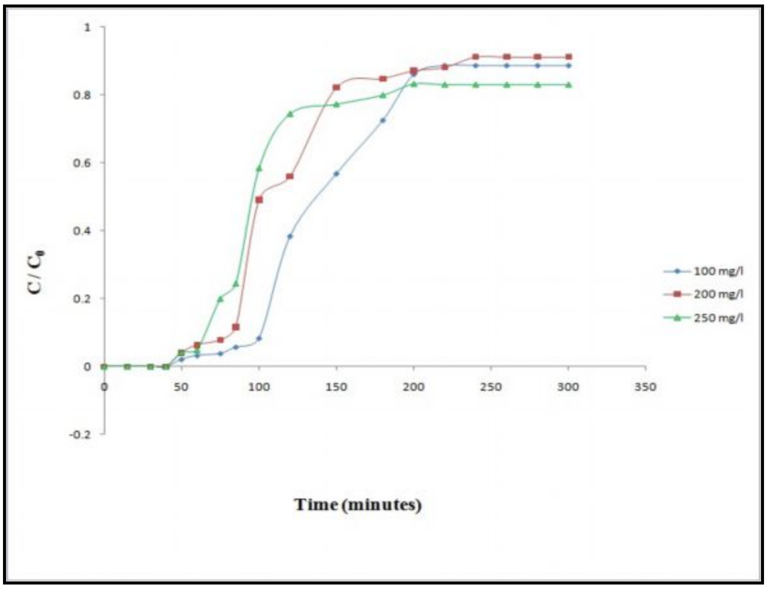

Figure 9 Effect initial metal ion concentration on adsorption of copper by orange peel

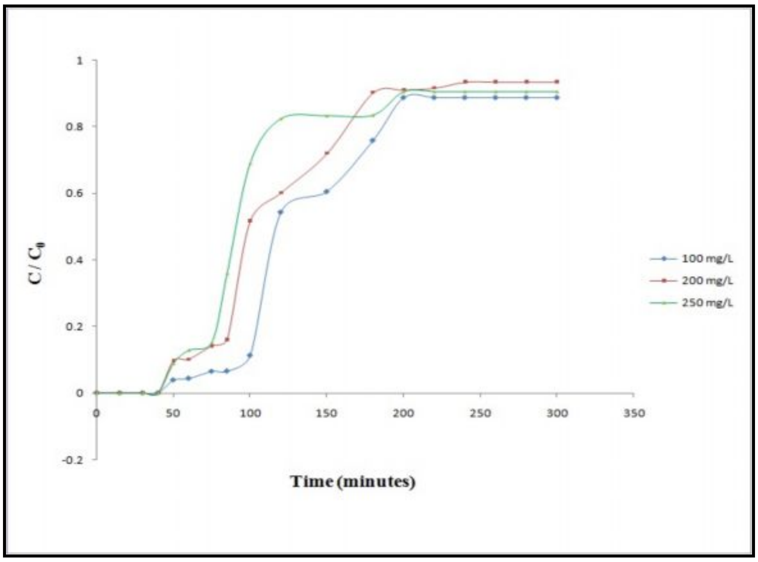

Figure 10 Effect initial metal ion concentration on adsorption of iron by orange peel

3.3 Interpretation of the Column study models

3.3.1 Thomas model

Thomas developed a model for adsorption processes in which external and internal diffusion limitations are not present. The linearized form of the Thomas model can be expressed as

$$ln\left(\frac{C_0}{C_t}-1\right)=\frac{kq_eW}{Q}-ktC_0$$

________ 6

where k is the Thomas rate constant (L/min. mg), q_e is the equilibrium metal ion uptake (mg/g), C_o is the inlet concentration (mg/L), C_t is the effluent concentration at time t (mg/L), W is the mass of adsorbent (g), Q is the inlet flow rate (L/min) and t is the flow time (min). The value of C_o/C_t is the ratio of inlet to outlet concentrations. A linear plot of ln($\frac{C_o}{C_t}$ -1) against time (t) was plotted as shown in the fig 12 and 13 for Cu (II) and Fe (II) to determine the values of q_e and k from the intercept and slope of the plot respectively. From the graph the value of k is 7 x 10^{-5} L/mg min for copper and that of iron is 3 x 10^{-5} L/mg min. The value of q_e was found to be 8.92 mg/g for copper and 9.56 mg/g for iron [11]. The value of k is inversely proportional to increase in bed height, increase in metal ion concentration and increase in flow rate. This can be analysed from the graph at any interval of time.

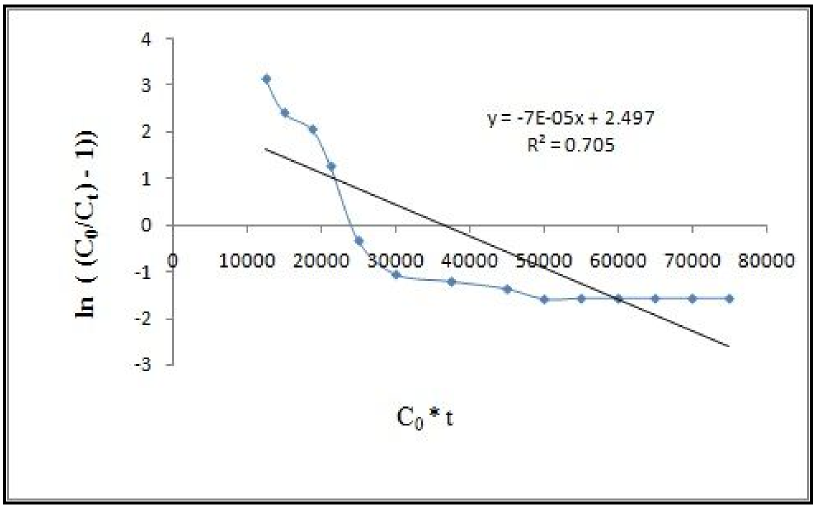

Fig 12: Thomas plot for the removal of Cu (II)

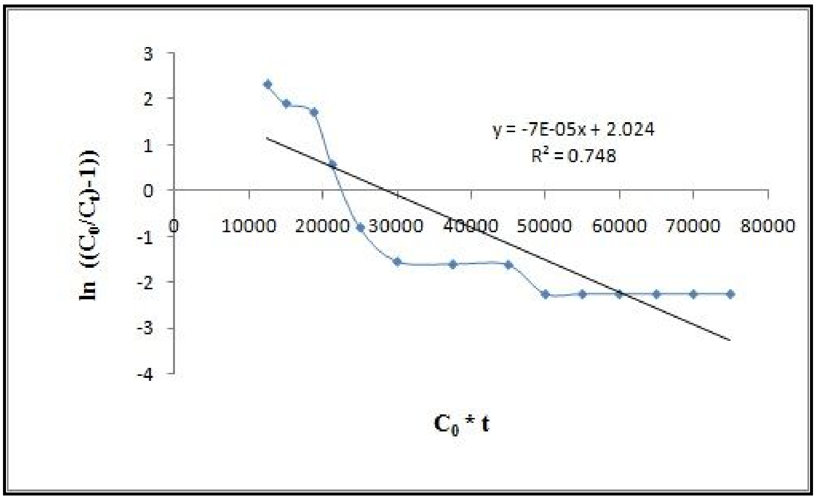

Fig 13: Thomas model for the removal of Fe (II)

3.3.2 Adam -Bohart model

Generally, the Adam-Bohart model is used to describe the initial part of the breakthrough curve. The expression is given by

$$ln\left(\frac{C_t}{C_0}\right) = kC_0t - \frac{kN_0Z}{F}$$

_______7

Where, C_o is the inlet dye concentration (mg/L), $C_t$is the effluent concentration (mg/L), k is the kinetic constant (L/mg min), F is the linear velocity (flow rate / column section area, cm/min), Z is the bed depth of the column (cm) and $N_o$is the saturation concentration (mg/L). A linear plot of ln (C_t/C_o) against time (t) was plotted as shown in fig 14 and 15 for Cu (II) and Fe (II) respectively and the values of k and $N_o$were determined from the slope and intercept of the plot respectively [14]. The graph indicates the slope value of (k) for copper which is equal to $4x10^{-5}$ L/mg min and that of iron it is $3x10^{-5}$L/mg min. The value of N_o for copper is 38.8 mg/L and that of iron is 41.0 mg/L

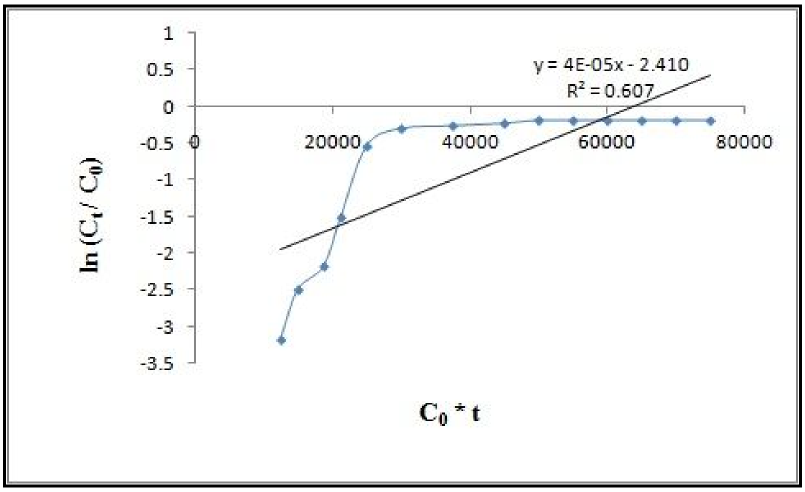

Fig 14: Adam –Bohart model for Copper

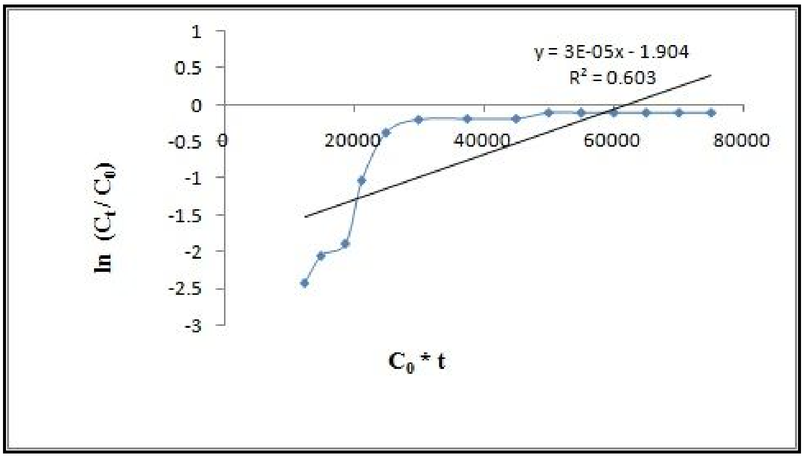

Fig 15: Adam –Bohart model for Iron

3.3.3 Bed Depth Service Time (BDST) Model

BDST model is used to predict the bed capacity by utilizing the different breakthrough values. The modified version of the equation used to describe the model is given by eq. 8

$$t = \frac{N_0Z}{C_0F} + \frac{1}{kC_0}ln\left(\frac{C_0}{C_t} - 1\right)$$

________ 8

Where t is the time (mins), N_o is the adsorption capacity (mg/L), C_o is the inlet concentration of metal ion solution in (mg/L), F is the linear velocity across the column (cm/min), Z is the bed depth (cm), k is the rate constant of BDST model (L/mg.min) and $C_t$is the effluent concentration (mg/L). A plot of t versus Z for Cu (II) and Fe (II) was expected to yield a linear curve through which N_o and k could be evaluated, from the slope and Y- Intercept respectively. The experimental results indicate the value of N_o is 8.73 mg/L for copper and 12.13 mg/L for iron. The value of k is 0.000317 L/mg min for copper and 0.000138 for L/mg min for iron which almost matches with the experimental values[15].

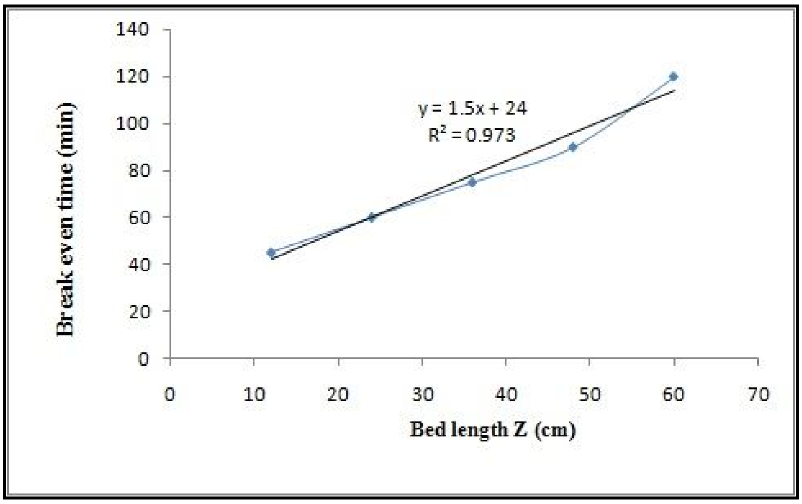

Fig 18: BDST model for Cu (II)

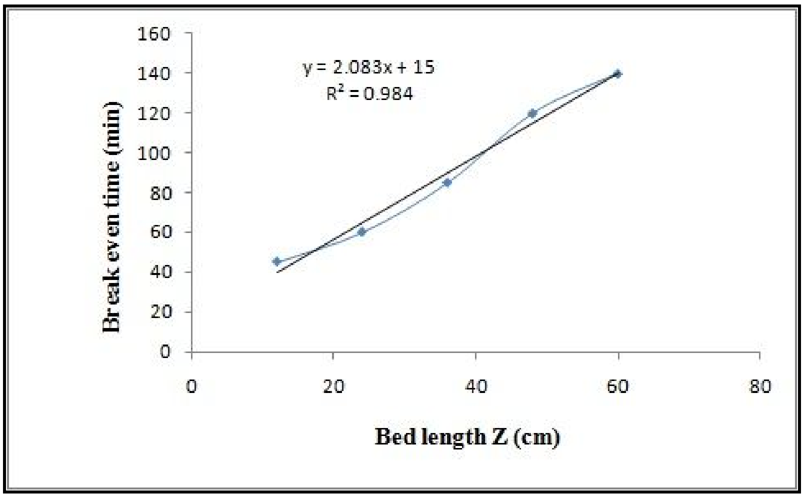

Fig 19: BDST model for Fe (II)

4 Conclusions

According to results obtained from fixed bed column studies, the adsorption of heavy metals from solution was affected by operational conditions such as flow rate and bed height. Slower flow rates gave better removal efficiencies and capacities compared to faster ones and longer bed heights also resulted in greater adsorption efficiencies due to an increase in residence time and available adsorption sites. Moreover the breakthrough time increased with the increase in bed height and decrease of flow rate. The kinetic modeling of column studies has been studied by Adam –Bohart model, Thomas model, BDST model.BDST model equation has been fitted with higher correlation / regression coefficient (R^2) values of 0.973 and 0.984 for Cu (II) and Fe (II) when compared to Adam-Bohart model and Thomas model. From the above conclusions it was predicted that the potential of natural orange peel as a low cost material for the removal of iron and copper from synthetic metal solutions was treated as a best adsorbent having high % removal of metal ions as well as high equilibrium capacity and these studies can also be extended to other metals.

Conflict of Interests

The authors declare that they have no conflict of interests.

Declaration by the Authors

The facts and views in the manuscript are ours and we are totally responsible for authenticity, validity and originality etc. I / We undertake and agree that the manuscripts submitted to your journal have not been published elsewhere and have not been simultaneously submitted to other journals. I/We also declare that manuscripts are our original work and we have not copied from anywhere else.

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