Adsorption of Ferrous and Ferric Ions in Aqueous and Industrial Effluent onto *Pongamia pinnata* Tree Bark

M. Mamatha, H. B. Aravinda, E. T. Puttaiah, S. Manjappa

Abstract—One of the causes of water pollution is the presence of heavy metals in water. In the present study, an adsorbent prepared from the raw bark of the *Pongamia pinnata* tree is used for the removal of ferrous or ferric ions from aqueous and waste water containing heavy metals. Adsorption studies were conducted at different pH, concentration of metal ion, amount of adsorbent, contact time, agitation and temperature. The Langmuir and Freundlich adsorption isotherm models were applied for the results. The Langmuir isotherms were best fitted by the equilibrium data. The maximum adsorption was found to 146mg/g in waste water at a temperature of 30°C which is in agreement as comparable to the adsorption capacity of different adsorbents reported in literature. Pseudo second order model best fitted the adsorption of both ferrous and ferric ions.

Keywords—Adsorption, Adsorption isotherms, Heavy metals, Industrial effluents.

I. INTRODUCTION

THE growth in the industrial sector for the last few decades is the major cause for the wide varieties of environmental pollution. Heavy metal pollution is one of the important concerns of the day. Heavy metals found in the effluent are not easily degradable and hence adversely affect the soil and water dependents. Different activities such as mining, electroplating, tanneries, metallurgical, textile, painting, car-radiator manufacturing [1]-[3] generates various heavy metals in the effluent. Stringent regulations are made by various countries for proper disposal of these effluents [4]. Extensive literature on the removal of heavy metals shows that much work is carried throughout the world to reduce the concentrations of heavy metals in waste water.

Many conventional physical, chemical and biological methods such as chemical precipitation [5], electrode deposition [6], membrane separations [7], evaporation, solvent extraction, ion exchange [2], [8], coagulation, cementation, biological treatment, reverse osmosis [9], accumulation by aquatic macrophytes [10] etc. are available. Some of these

methods are not economically viable and also have the problem of precipitate formation during treatment which in turn ends up with disposal problems. In this regard adsorption is one of the promising methods for removal of heavy metals. Varieties of materials are used as adsorbents with and without pretreatment such as polymers [11], [12], zeolites [13], [14], lignite [15], waste tires [16], algae [17], crab shells [18], lignin [19], fly ash [20], [21], activated carbon from various agricultural residues [22]-[27], tree barks [8], [28]-[30] for removing various heavy metals from aqueous and waste water.

The waste water based on type of industry contains varying levels of toxic metal ions such as lead, copper, cobalt, cadmium, chromium, nickel, mercury, arsenic, zinc, iron, manganese etc. Such heavy metals in water exhibit specific damaging effects on flora and fauna if the allowable limits are exceeded [28]. The present paper highlights the need and method for removal of iron from waste water. The presence of iron in drinking water supplies is objectionable for a number of reasons. Under the high pH conditions existing in drinking water supply, ferrous ions are unstable and precipitates as insoluble ferric compounds, which settles out as rust colored silt. Such water often tastes unpalatable even at low concentrations (0.3mg/L) and stains laundry and plumbing fixtures. The iron that settles out in the distribution system gradually reduces the flow of water.

The physiological functioning of iron involves controlled oxidation reduction reaction. Free iron is toxic because it can chemically catalyze the oxidation of lipids and other biomolecules. Specific binding proteins control extracellular transport and intracellular storage. Three quarters of the typical 4g body content of iron is in hemoglobin, myoglobin and iron containing enzymes. Virtually all of the rest is in storage and transport proteins. The other problems that result from iron toxicity include anorexia, oligura, diarrhea, hypothermia, diphasic shock, metabolic acidosis, and even death. In some cases, vascular congestion of the gastrointestinal tract, liver, kidneys, heart brain, adrenals, and thymus may also occur. Impotence may also occur in young men and amenorrhea in young women. These problems occur due to iron loading in the anterior pituitary [31]. Some rivers are found to be polluted by the presence of iron due to industrial activity in the surrounding area [32], [33].

The primary objective of this study is to determine the adsorption capacity of raw *Pongamia pinnata* tree bark for iron removal from aqueous and waste water. *Pongamia pinnata* also known as Karanja, the seeds of this tree are used for biofuel production. It is commonly found throughout the Philippines along the sea shore. It also occurs in the

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Mascarene Islands and in tropical Asia, across Malaya to Australia, and in Polynesia. Presently the bark of this tree is used for making strings and ropes. The bark contains a bitter alkaloid, soluble in alcohol and water, also an acid resin of a greenish brown color, soluble in ether. The alcoholic extract is composed of a substance analogous to quinovin together with sugar. The watery extract contains much mucilage, which is gelatinized by ferric chloride. A decoction of the bark gives a blue black color with iodine solution and tannins are absent in this bark. The bark is also useful for bleeding piles [34]. There are thousands of *Pongamia pinnata* trees grown in and around Davangere (study area), Karnataka, India.

II. MATERIALS AND METHODS

A. Adsorbent Preparation

Pongamia pinnata bark was peeled off from the cut branches of the tree in the study area, Davangere, Karnataka, India. The raw bark was Sun dried for 3 days. Then the bark was dried at 80°C for six hours in hot air oven and cut into 2 to 3 inch pieces. Dried bark was powdered in pulverizer. The powder was washed several times with double distilled water to remove solubles, coloring matter and again dried in hot air oven at 60°C for 8 hours. The powdered bark was sieved (Indian Standard Sieve) and various fractions of adsorbent was separately stored in air tight containers. The particle diameter of the various sieve fractions are as in Table I.

TABLE I
AVERAGE PARTICLE SIZE OF ADSORBENT ANALYZED BY INDIAN STANDARD SIEVES

Sl.No.	Sieve Size	Average particle size (µm)
1	2.36mm-1mm	1680
2	1mm-600µ m	800
3	600µm-300µ m	450
4	300 µm-150µ m	225
5	150 µm-75µ m	112.5

mm =millimeter, µm =micro meter.

B. Stock Solution

Analytical grade Ferrous Ammonium Sulfate $Fe(NH_4)_2(SO_4)_2.6.H_2O$ and Ferric Ammonium Sulfate $FeNH_4(SO_4)_2.12H_2O$ (Nice Chemicals) were used to prepare stock solution. Double distilled water was used to prepare stock solution. Solutions of required concentrations were prepared using stock solution. 0.1N HCl and 0.1N NaOH solutions were used to adjust the pH of the solutions. Industrial effluent (before treatment) from a local industry was collected, filtered using Whatman no.1 filter paper and stored in air tight containers.

C. Batch Adsorption Studies

Batch adsorption studies were conducted to determine the equilibrium time required to reach saturation. Adsorption kinetics were carried out using separately 100ml of Ferrous Ammonium Sulfate and Ferric Ammonium Sulfate solutions of 25 to 100ppm, pH in the range 2 to 9, 2.5 to 10g/L adsorbent of average particle size 225µm, a contact time up to 60min, agitation from 100 to 200rpm and temperature of

30°C. The parameters were varied one at a time and samples collected at predetermined intervals, filtered, centrifuged and analyzed by UV-VIS Spectrophotometer (Systronics) as per standard methods [35]. The amount of ferrous or ferric ion uptake by the bark was found. All experiments were carried out in duplicate and mean values are presented. The maximum deviation was 5.0%. The percentage removal of heavy metal from the solution was calculated using (1),

$$\%\mathrm{Re}moval = \frac{C_o - C_i}{C_o}X100 \tag{1}$$

where C_0 is initial concentration of heavy metal, C_i is final concentration of heavy metal. The adsorption capacity q_e (mg/g) after reaching equilibrium was calculated using (2).

$$q_e = (C_o - C_e)\frac{V}{W} \tag{2}$$

where V is the volume (L) of solution and W is the mass (g) of adsorbate used.

III. RESULTS AND DISCUSSION

A. Characterization of Adsorbent

A scanning electron microscope (SEM) was used to examine surface of the *Pongamia pinnata* bark. Fig. 1 shows the SEM photograph of the bark. There holes an caves type openings on the surface of the bark and would increase the surface area available for adsorption. Table II shows the proximate analysis of the *Pongamia pinnata* adsorbent used in the present study.
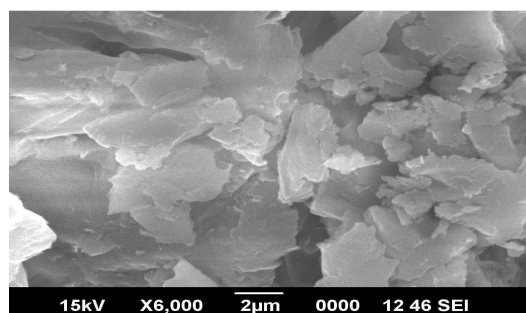

Fig. 1 SEM photograph of *Pongamia pinnata* bark

TABLE II
PROXIMATE ANALYSIS OF *PONGAMIA PINNATA* TREE BARK

Sl.No.	Property	Result
1	Bulk density (kg/m³)	410
2	Moisture content (% Wt.)	8.4
3	Ash content (%Wt.)	14.1
4	Volatile matter (%Wt.)	7.43
5	Fixed Carbon (% Wt.)	83.2
6	Matter soluble in water (%)	1.51
7	Matter soluble in acid (%)	0.56

kg=kilogram, m=meter, % Wt=Percentage weight

The spectral analysis of *Pongamia pinnata* tree bark shows the presence of 8 compounds namely, 3-methoxy- (3",4"-dihydro-3"-hydroxy-4"-acetoxy)-2",2"-dimethylpyrano-(7, 8:5",6")-flavone, 3-methoxy-(3",4"-dihydro-4"- hydroxy-3"-acetoxy)-2",2"-dimethylpyrano-(7,8:5",6")-flavone, caryophyllene oxide, obovatachalcone,8-hydroxy-6-methoxy-3-pentyl-1H-isochromen-1-one,6,7,2,2-dimethylchromono-8,γ,γ,-dimethylallylflavanone,isolonchocarpin, ovaliflavanone A [36].

All the above mentioned compounds can be decomposed through biological degradation by various microorganisms. The decomposition products are either gases or molecules that can be recycled by other microorganisms or plants generating no negative effects on the environment. Thus the bark used for decontaminating industrial effluents can be degraded by natural means [28].

B. Effect of pH

The effect of pH on the adsorption of ferrous and ferric ions is as shown in Fig. 2. The pH of solution determines the surface charge of the adsorbent, the degree of ionization and speciation of the adsorbate. Batch equilibrium studies were carried at different pH values in the range of 2 to 8. It was observed from figure that maximum removal of ferrous or ferric ions was in the range of pH 5.8 to 6.4. The removal of metal ions was constant above pH 7. This was due to the formation of metal hydroxides which precipitated in solution. This show that a strong force of interaction between the ferrous or ferric ions and *Pongamia pinnata* bark that H^+ ion could influence the absorption capacity. The interaction is more at pH 5.8 due to the competence of acidic H^+ ion with metal cation for the absorption sites. [24].
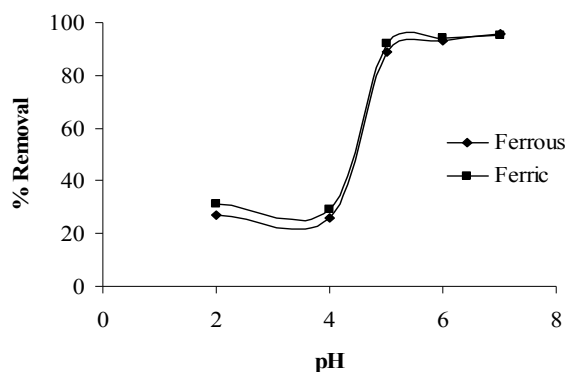

Fig. 2 Effect of pH on adsorption onto *Pongamia pinnata* bark
Conditions: Initial adsorbate concentration 100ppm; adsorbent concentration 10g/L; contact time 1hr; agitation 200rpm

C. Effect of Initial Metal Ion Concentration

The effect of initial metal ion concentration is as shown in Fig. 3. From the figure it is evident that the metal uptake mechanism is dependent on initial heavy metal concentration. The metal ions are adsorbed onto the active sites of the adsorbent hence as the metal concentration increases the specific sites are saturated and no further adsorption occurs [15]. Batch experiments with varying initial metal ion concentration from 25ppm to 100ppm were carried out keeping other parameters constant. It was observed that metal ion uptake was more with a solution of 25ppm and was observed that increase in metal ion concentration decreased adsorption.
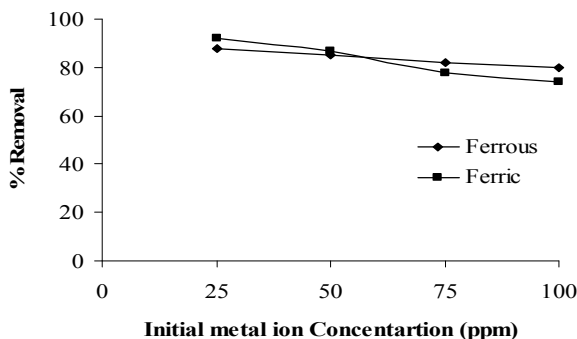

Fig. 3 Effect of Initial metal ion concentration on adsorption onto
Pongamia pinnata bark
Conditions: pH 5.8; adsorbent concentration 10g/L; contact time 1hr; agitation 200rpm

D. Effect of Adsorbent Concentration

As the number of active sites available for the adsorption of metal ion increases, the adsorption percentage also increases. This occurs as the concentration of adsorbent increases. In the present case, batch study with adsorbent concentration ranging between 2.5g/L to 10g/L keeping all other parameters constant was conducted. The results are as shown in Fig. 4. It shows that 10g/L of adsorbent concentration adsorbs the maximum metal ion in both cases.
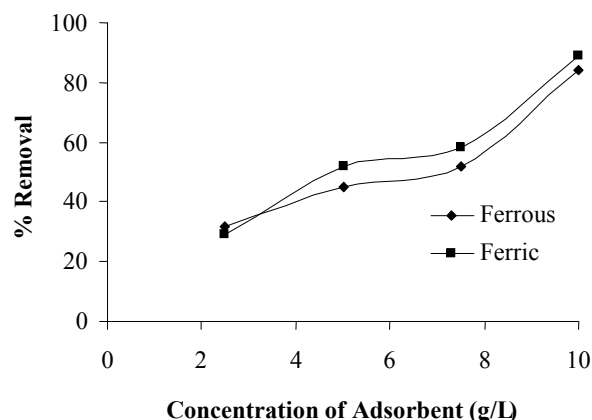

Fig. 4 Effect of concentration of adsorbent on adsorption onto
Pongamia pinnata bark
Conditions: pH 5.8; Initial adsorbate concentration 100ppm; contact time 1hr; agitation 200rpm

E. Effect of Contact Time

The removal of ferrous and ferric ions from the stock solution increases with time and attains equilibrium value in about 40 to 50min. Initially within 10 to 15min the uptake of metal ions from bark is rapid, but gradually decreases with time till it reaches equilibrium. In the beginning the number of

active sites available for adsorption is more hence metal uptake increases but later on the available active sites reduce and metal ions need to compete for the vacant sites. Fig. 5 shows that at the start of the adsorption the percent removal of metal ions is more and later reaches a constant value where upon no further adsorption was observed.
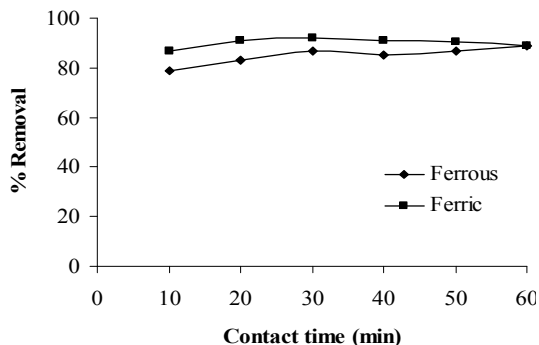

Fig. 5 Effect of contact time on adsorption onto *Pongamia pinnata* bark
Conditions: Initial adsorbate concentration 100ppm; adsorbent concentration 10g/L; pH 5.8; agitation 200rpm

F. Effect of Agitation and Temperature

In all batch experimental studies conducted a constant agitation of 200rpm was maintained keeping all other parameters constant. To study the effect of agitation batch experiments were conducted with agitation speeds maintained at 100rpm, 150rpm, and 200rpm. Fig. 6 shows that an agitation of 200rpm showed higher uptake of ferrous or ferric ions onto adsorbent. The contact between the adsorbent site and metal ion is more when agitation speeds are high. All experiments were conducted at 30°C.
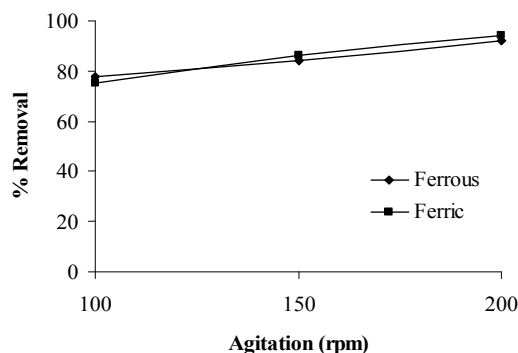

Fig. 6 Effect of agitation on adsorption onto *Pongamia pinnata* bark
Conditions: pH 5.8; Initial adsorbate concentration 100ppm; adsorbent concentration 10g/L; contact time 1hr

G. Adsorption Isotherms

From all the batch experiments carried out optimum parameters selected were pH of 5.8, initial metal ion concentration of 100ppm, adsorbent concentration of 10g/L, contact time of 10 to 60min, temperature of 30°C, and agitation of 200rpm. The adsorption data was correlated with the Langmuir and the Freundlich isotherm model equations.

The Langmuir isotherm is valid for monolayer adsorption onto a surface with a finite number of identical sites. It is based on assumption of adsorption homogeneity, such as equally available adsorption sites, monolayer surface coverage and no interaction between adsorbed species [8]. According to the Langmuir adsorption isotherm, the adsorption process can be expressed as (3)

$$q_e = \frac{q_m C_e}{K_d + C_e} \tag{3}$$

on rearrangement of (3) to linear form as (4)

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{K_d}{q_m} \tag{4}$$

where C_e (mg/L) is the equilibrium concentration of ferrous or ferric ions in solution, q_e (mg/g) the adsorption capacity at equilibrium, q_m (mg/g) the maximum adsorption capacity and K_d is the effective dissociation constant. The values of q_m and K_d estimated from Figs. 7 (a) and (b) for adsorption of ferrous were 100mg/g and 0.1 respectively. For ferric ion adsorption the corresponding values were 125mg/g and 1.4 respectively. The correlation coefficient of the Langmuir isotherm (R^2) was 0.9995 for ferrous and 0.9960 for ferric ions.
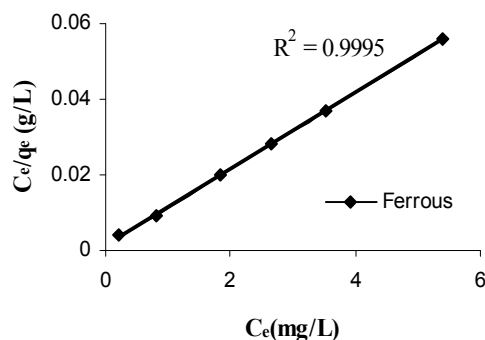

Fig. 7 (a) Langmuir adsorption isotherm of ferrous ion in aqueous solution adsorbed onto *Pongamia pinnata* bark
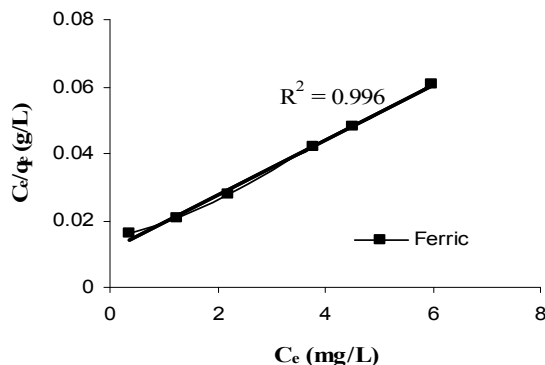

Fig. 7 (b) Langmuir adsorption isotherm of ferric ion in aqueous solution adsorbed onto *Pongamia pinnata* bark

In the Freundlich equation, the empirical relationship whereby it is assumed that the adsorption energy of ferrous or ferric ions binding to a site on an adsorbent depends on the adjacent sites available for adsorption and it is a multilayer adsorption process. The Freundlich isotherm is expressed as (5):

$$q_e = k_f \left(C_e \right)^{\frac{1}{n}} \tag{5}$$

on rearrangement to linear form (6):

$$\ln q_e = \ln K_f + \frac{1}{n} C_e \tag{6}$$

where q_e (mg/g) is the adsorption capacity at equilibrium, C_e (mg/L) the equilibrium concentration of ferrous or ferric ions in solution, K_f and n are the physical constants of the Freundlich adsorption isotherm. The K_f and n values are the indicators of the adsorption capacity and adsorption intensity respectively. The slope and the intercept of the linear Freundlich equation are equal to 1/n and ln K_f respectively as in Figs. 8 (a) and (b). The constants n and K_f obtained from Fig. 8 (a) for ferrous ions were 37.73 and 91.89 respectively. For ferric ion adsorption the corresponding values from Fig. 8 (b) were 3.041 and 56.14 respectively. The correlation coefficient of the Freundlich isotherm (R^2) was 0.9966 for ferrous and 0.9854 for ferric ions.
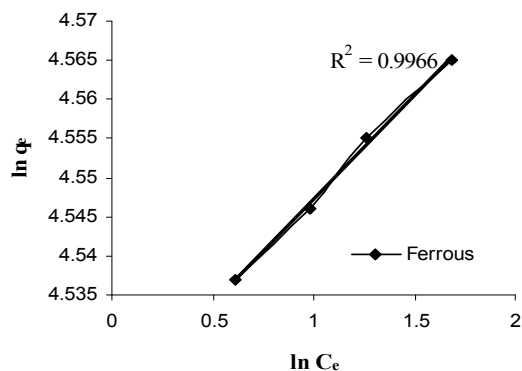

Fig. 8 (a) Freundlich adsorption isotherm of ferrous ion in aqueous solution adsorbed onto *Pongamia pinnata* bark
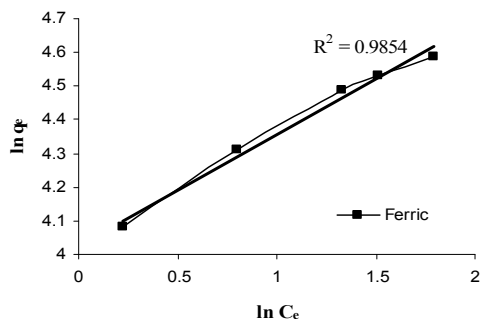

Fig. 8 (b) Freundlich adsorption isotherm of ferric ion in aqueous solution adsorbed onto *Pongamia pinnata* bark

Comparing both of the correlation coefficients obtained, it shows that the Langmuir isotherms suits well for the obtained adsorption data than Freundlich isotherm.

H. Adsorption Kinetics

The kinetics involved in the adsorption of ferrous and ferric ions on to *Pongamia pinnata* were studied based on three models namely, the pseudo first, pseudo second order and Elovich model. For the adsorption of solute from liquid solutions, pseudo first order model is most widely used. This model is as below,

$$\ln(q_e - q_t) = \ln q_e - K_1 t \tag{7}$$

where q_e is the mass of metal ion adsorbed at equilibrium (mg/g), q_t is the mass of metal adsorbed at time t (mg/g), K_1 is the first order reaction rate constant (min^{-1}). Plots of ln (q_e-q_t) vs. t indicate the application of the first-order kinetic model for ferrous and ferric ions and are shown in Figs. 9 (a) and (b).
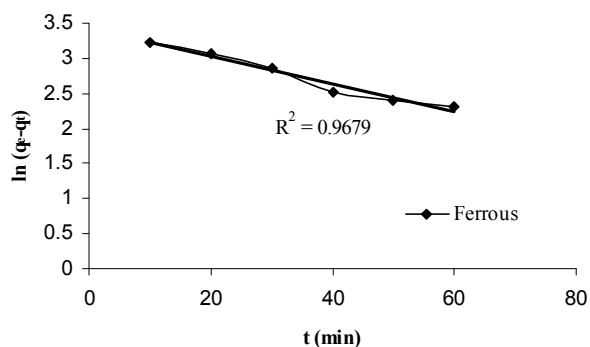

Fig. 9 (a) First order kinetic model for ferrous ion adsorption onto *Pongamia pinnata* bark
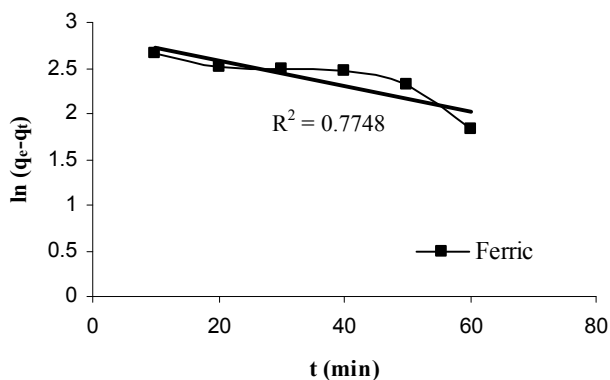

Fig. 9 (b) First order kinetic model for ferric ion adsorption onto *Pongamia pinnata* bark

Based on equilibrium adsorption capacity pseudo second order model is as shown in the form

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \tag{8}$$

where K_2 is the second order reaction rate constant (mg/g min). A plot of t/q_t against t is a linear relation for the second order kinetics to be applicable to that model as shown in Figs. 10 (a) and (b).
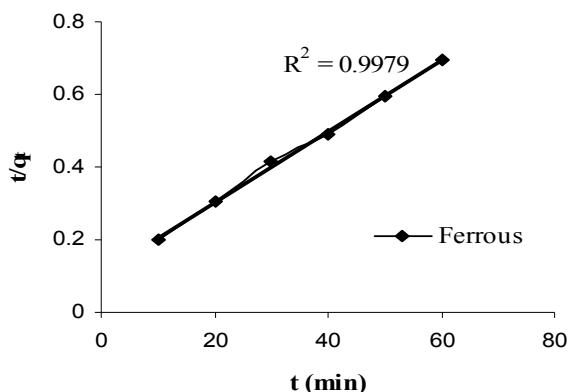

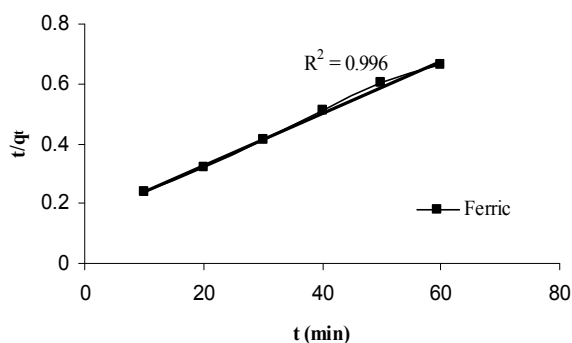Fig. 10 (a) Second order kinetic model for ferrous ion adsorption onto *Pongamia pinnata* bark

Fig. 10 (b) Second order kinetic model for ferric ion adsorption onto *Pongamia pinnata* bark

Elovich model for the adsorption is expressed as

$$q_t = \alpha + \beta \ln t \qquad (9)$$

A plot of q_t against ln t gives a linear relationship for the applicability of the simple Elovich kinetic model as shown in Figs. 11 (a) and (b).
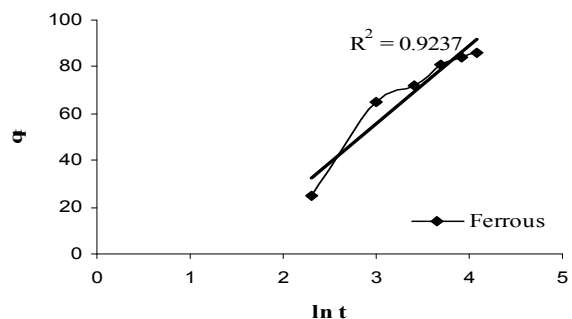

Fig. 11 (a) Elovich kinetic model for ferrous ion adsorption onto *Pongamia pinnata* bark
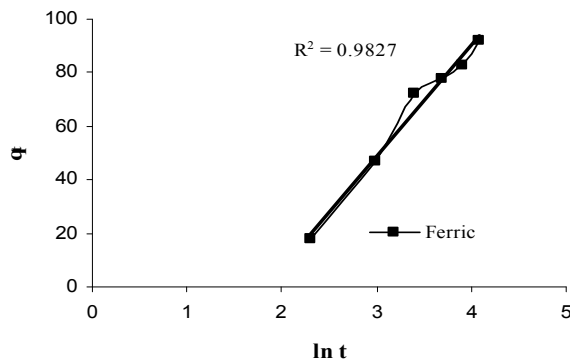

Fig. 11 (b) Elovich kinetic model for ferric ion adsorption onto *Pongamia pinnata* bark

Comparing all the above models the parameters obtained are reported in Table III. Based on linear regression ($R^2 > 0.99$) values, the kinetics of ferrous and ferric ion adsorption on *Pongamia pinnata* can be described well by pseudo second order kinetics.

TABLE III
PARAMETERS OF KINETIC MODELS

Metal ion	First order		Second order		Elovich		
	R^2	K_1	R^2	K_2	R^2	α	β
Ferrous	0.96	0.02	0.99	9.97×10^{-4}	0.92	-44.1	33.2
Ferric	0.77	0.01	0.99	6.76×10^{-4}	0.98	-74.9	41.2

I. Mechanism of Adsorption

The mechanism of adsorption of metal ion onto adsorbent follows three steps viz. the movement of metal ion from bulk liquid onto the surface of adsorbent, pore diffusion and intra particle transport. The overall rate of the adsorption is governed by the slowest step of all. For the batch adsorption mode, pore and intra particle diffusion are rate limiting whereas in case of continuous adsorption mode, film diffusion is more likely the rate limiting step. The adsorption rate parameter which controls the batch process for most of the contact time is intra particle diffusion. The possibility of intra particle diffusion resistance affecting adsorption was explored by using the intra particle diffusion model as

$$q_t = k_i t^{1/2} + I \qquad (10)$$

where, k_i is the intra particle diffusion rate constant, a plot of q_t vs. $t^{1/2}$ for ferrous and ferric ions are as shown in Fig. 12 (a) and (b). Values of I are as reported in Table IV. I values give an idea about the thickness of boundary layer. The larger the intercept the greater is the boundary layer effect.

TABLE IV
KINETIC PARAMETERS AND BOUNDARY LAYER FOR ADSORPTION

Metal ion	K_i	I	R^2
Ferrous	9.38	18.156	0.927
Ferric	11.87	1.867	0.978

The deviation of straight lines from the origin as shown in Figs. 12 (a) and (b) may be because of the difference between

the rate of mass transfer in the initial and final steps of adsorption. Further, such deviation of straight line from the origin indicates that the pore diffusion is not the rate controlling step.
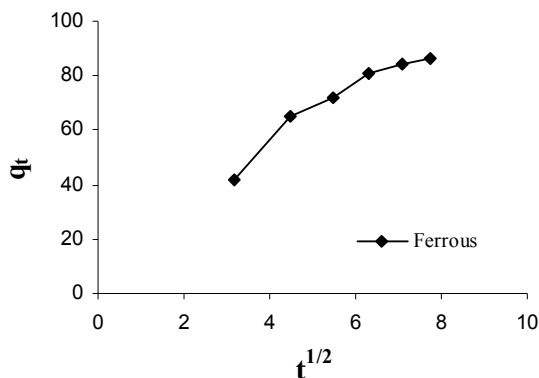

Fig. 12 (a) Mechanism of particle diffusion for ferrous ion adsorption onto *Pongamia pinnata* bark
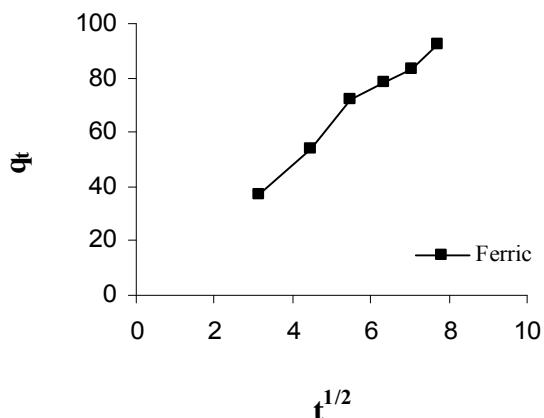

Fig. 12 (b) Mechanism of particle diffusion for ferric ion adsorption onto *Pongamia pinnata* bark

From Figs. 12 (a) and (b), it may be seen that there are two separate regions the first portion is attributed to the bulk diffusion and second portion to the intraparticle diffusion. The values of $k_{i,1}$ and $k_{i,2}$ are obtained from slopes of the two straight lines are 15.06 and 9.79 for ferrous and 13.23 and 3.53for ferric ions.

J. Adsorption of Iron from Industrial Effluent

Raw industrial effluent without treatment was collected from a local wire drawing industry. After filtration, batch experiments were carried out using 100ml of effluent each with 10g/L adsorbent, temperature of 30°C and agitation of 200rpm. The pH of solution was varied from 2 to 7. Fig. 13 shows the effect of pH on the adsorption of iron by *Pongamia pinnata* bark. Maximum removal of iron was observed in the pH range 5 to 6. Above pH 8 precipitate was observed. The percentage iron removal was low compared to synthetic solution as the effluent consisted of other competing ions such as copper, lead and nickel.
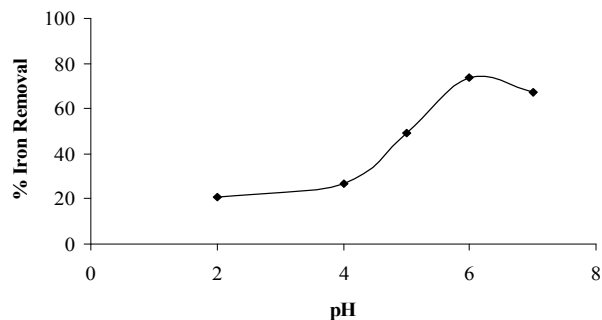

Fig. 13 Effect of pH on adsorption of iron from industrial effluent onto *Pongamia pinnata* bark
Conditions: adsorbent concentration 10g/L; contact time 1hr; agitation 200rpm

IV. CONCLUSION

Pongamia pinnata seeds are widely used for biofuel production, hence in recent years this tree is widely grown in the fields. Thus the availability of the *Pongamia pinnata* tree bark is more. Also the adsorbent preparation does not involve the activation process of the bark hence the preparation is simple and economical. It was concluded from the present study that *Pongamia pinnata* tree bark can be used effectively for the removal of iron from aqueous and industrial effluents. The adsorption data were better fitted by Langmuir model than Freundlich isotherm model. An optimum of 5 to 6 pH, 10g/L of adsorbent, initial metal ion concentration of 100ppm, with a contact time of 50min and agitation 200rpm was chosen from the experiments conducted. Pseudo second order model best fitted the kinetics. In case of industrial effluent, the decrease in the percent removal of iron was due to the interference of other metal ions present. In this regard, *Pongamia pinnata* tree bark can be effectively used as a viable and economic adsorbent for removal of iron from aqueous and industrial effluents.

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