

## Research Article

**Adsorptive Removal of Acid Red 1, from Aqueous Solutions by Preformed Floccs**Nijagala Munilakshmi<sup>\*a</sup>, Muduru Srimurali<sup>a</sup> and Janakiram Karthikeyan<sup>a</sup><sup>a</sup>Department of Civil Engineering, S.V. University, A.P, India

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**Abstract**

Aqueous Acid Red 1 dye was decolorized by adsorption onto preformed floccs of Aluminium Sulphate, Ferric Chloride and Ferrous Sulphate. To understand the potentiality of preformed floccs, experiments were conducted to determine equilibrium time, optimum dose of adsorbent, optimum pH, adsorption isotherm and intraparticle diffusion. The corresponding results showed that excellent colour removal of Acid Red 1 can be achieved with preformed floccs at pH of 4 than 10. The maximum adsorption capacity of Ferric chloride, Aluminium Sulphate and Ferrous Sulphate floccs was 250mg/g, 32.26mg/g and 6.623mg/g respectively. The FTIR analysis was done to know the type of chemical bonds involved in the colour removal. The isothermal equilibrium sorption data fitted well into the Langmuir Isotherm. Kinetic studies revealed that the interaction is of chemical nature. Pore diffusion seems to be the rate limiting step.

**Keywords:** Adsorption, preformed floccs, Aluminium Sulphate, Ferric Chloride, Ferrous Sulphate

**1. Introduction**

Synthetic dyes are known to cause immediate visible pollution in effluent water aside from contamination by interfering with light which may retard photosynthesis and biota growth (Benadjema et.al, 2011). Dyes containing azo-aromatic groups are highly dispersible pollutants. These effluents discharged from various industries (e.g. textile, paper, leather, food, etc.) are known to be toxic, carcinogenic, mutagenic and their release in the environment is a considerable source of non-aesthetic pollution since the existence of low concentrations of dyes is clearly visible. Thus the removal of these colored compounds from wastewater is an important target from the environmental point of view (Lara abramian et.al., 2009). Treatment processes such as physical, physicochemical, chemical and biological methods have been investigated and proved to be effective but to varying degree of success. Colour removal by physical process is negligible while the ability of biological processes for decolorization of dye effluents is ambiguous, differing and divergent, perhaps due to toxic nature of dye chemicals. Adsorption and chemical treatment processes have shown promise as a practical and economic process for treatment of textile waste; especially for colour removal.

Adsorption has been reported as an efficient method for the removal of different toxic pollutants in dye waste effluents. Activated carbons are widely used as powerful adsorbents for most pollutants in waste water (Sharkawy

et.al. 2007). Even though colour removal by adsorption onto activated carbon is highly efficient, the overlying cost of the activated carbon and associated problems of regeneration and reuse warrants search for alternative low cost adsorbents. Low cost adsorbents like materials derived from industrial waste materials, agricultural wastes, minerals, coal, soil, etc. have been widely investigated for textile dye colour removal (Gupta et.al. 2003). Treatment with chemicals like Aluminium Sulphate, Ferrous Sulphate, Ferric Chloride, Calcium chloride, Copper Sulphate, Sulphuric and hydrochloric acid etc., either alone or in combination for removal of colour from individual dye wastes, as well as composite mill waste were investigated (Venkatamohan et.al., 1999). Among several methods investigated for colour removal from textile dyestuffs, coagulation and adsorption appears to be better suited; however each method has its own merits and demerits. Combining the merits of both these methods, the present investigation was conducted to remove C.I. Acid Red 1 dye colour employing preformed floccs of Aluminium Sulphate, Ferric Chloride and Ferrous Sulphate. Scope of the study includes kinetics and isothermal equilibrium studies, effect of adsorption process parameters such as pH and adsorbent dose on colour removal, also chemical groups that are involved in the adsorption process. This study will be useful for further applications in designing the batch adsorption process for the decolorization of dye wastewaters.

**2. Materials and Methods***2.1. Glass ware and chemicals*

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All the glass ware used in this study is of 'Pyrex' quality and analytical grade (AR) chemicals were used throughout the study. Thermo UV VIS Spectrophotometer of Evolution 201 Model was used for taking transmittance and absorbance readings.

## 2.2. Adsorbent

Preformed flocs of Aluminium Sulphate, Ferric Chloride and Ferrous Sulphate were used as adsorbent.

## 2.3. Adsorbate

Acid Red 1 (Azo) was used as adsorbate. Stock solution of C.I. Acid Red 1 (Azo) was prepared by dissolving 50mg in 1000ml distilled water.

## 2.4. Analysis and Characterization

The maximum wavelength of Acid Red 1 (530nm) was observed using Thermo make UV VIS Spectrometer of Evolution 201 Model. SEM (Scanning electron microscopy) analysis was carried out on the adsorbents, to study its surface texture before and after Acid Red 1 Adsorption. Fourier Transform Infrared (FTIR) (FTIR-2000 Perkin Elmer) analysis was applied on the free dye and the dye adsorbed preformed flocs to know the surface functional groups involved in colour removal, where the spectra was recorded from 4000 to 400  $\text{cm}^{-1}$ . All analyses were done in accordance with standard methods (Clescerl et.al., 1998).

## 3. Experimental Procedure

Varying doses of coagulants were dissolved in distilled water and was maintained at different pH conditions varying from 2 to 13 and observed for floc density. Floc density of preformed flocs was measured in terms of turbidity, and the dose and pH at which highest turbidity was recorded, were adopted as favorable dose and favorable pH. Agitated, non-flow batch sorption studies were conducted by bottle point method using reagent bottles of 250ml capacity. To a 100ml of test dye solution (pH adjusted to respective pH of preformed floc) of 50mg/l concentration taken in the reagent bottle, premeasured quantity of preformed floc was added and the resultant mixture was agitated in a horizontal shaker at a rate of 125 rpm for varying time intervals of 1, 3, 5, 7, 9, 12, 15, 30, 45 and 60 minutes. The bottles were withdrawn from the shaker at designated time intervals and the reagent bottles were kept undisturbed for 4 hours for sedimentation, at the end of which, samples were withdrawn by carefully pipetting out 10ml portion and are analyzed for colour content remaining in the test dye solution. The time at which maximum removal of colour takes place and no further significant difference in colour removal occurs from that time, was taken as the equilibrium contact time and used in all further studies. Equilibrium Isothermal studies were conducted by adding varying doses of preformed flocs such as 100, 200, 300,

400, 500, 600, 700, 800, 900 and 1000mg/l to the test dye solution and contacted for equilibrium time. The residual colour was analyzed with a Spectrophotometer by measuring OD/% T at respective maximum wavelength (530nm) of dye solution and computing concentrations from the calibration curve.

## 4. Results and Discussion

Favorable dose and Favorable pH of coagulants are presented in Table 1. All subsequent experiments were performed by employing favorable dose at favorable pH.

**Table.1** Favourable pH and Favorable dose of coagulants

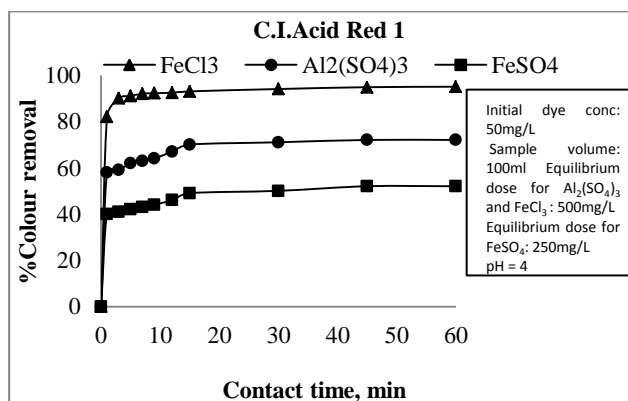
Coagulant	Favorable pH		Favorable dose, mg/100ml
	Acidic Medium	Basic Medium	
Aluminium Sulphate ( $\text{Al}_2(\text{SO}_4)_3 \cdot 16 \text{H}_2\text{O}$ )	4	10	1000
Ferric Chloride( $\text{FeCl}_3$ )	4	10	1000
Ferrous Sulphate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ )	4	10	500

### 4.1. Kinetics of colour adsorption

The length of the time required to reach equilibrium of sorption reaction is important to determine the capacity of the sorbent with respect to solute removal. Thus, the results of the kinetic studies are the basis for all additional batch studies. Sorption kinetics also influence the shape of the adsorption profile, fast kinetics will result in a steep profile (Venkatamohan et.al., 1997).

#### 4.1.1. Effect of contact time

The influences of contact time vis-à-vis kinetics of colour removal by preformed flocs are presented in Fig. 1 and Fig.2 at pH of 4 and pH of 10. It may be observed from figures that the rate of colour removal was rapid initially. The rate leveled off gradually and then attained a more or less constant value (equilibrium) beyond which there was no significant increase in colour removal. The time required to attain equilibrium was 15 minutes.



**Fig.1.** Effect of contact time on colour removal from acid red 1 aqueous solution at pH=4.

As per Fig.1, Excellent colour removal of 95 % was observed with Ferric chloride preformed flocs, Moderate colour removal of 72% was observed with Aluminium Sulphate preformed flocs and low colour removal of 52% was observed with Ferrous Sulphate preformed flocs at pH of 4.

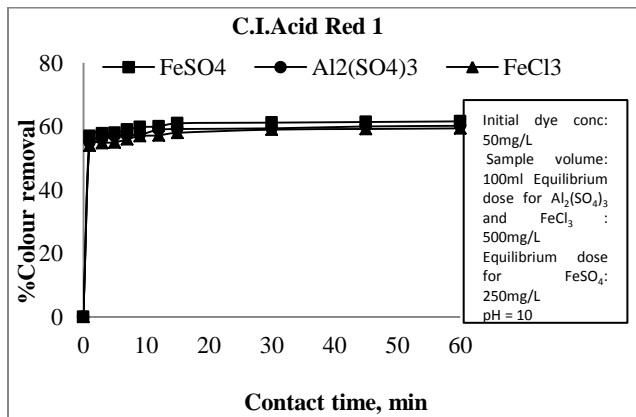


Fig.2. Effect of contact time on colour removal from acid red 1 aqueous solution at pH=10.

As per Fig.2, Moderate colour removal of 61% was observed with Ferrous Sulphate preformed flocs, 59% with Aluminium Sulphate preformed flocs and 59% with Ferric Chloride preformed flocs at pH of 10.

4.2. Equilibrium Isothermal adsorption by preformed flocs

Equilibrium isothermal adsorption studies were conducted by varying the amount of floc dose from 100 to 1000 mg/l and the results are presented graphically as % color removal at different doses of flocs for C.I. Acid Red 1, at pH=4 and pH=10, as shown in Fig.3 and Fig. 4.

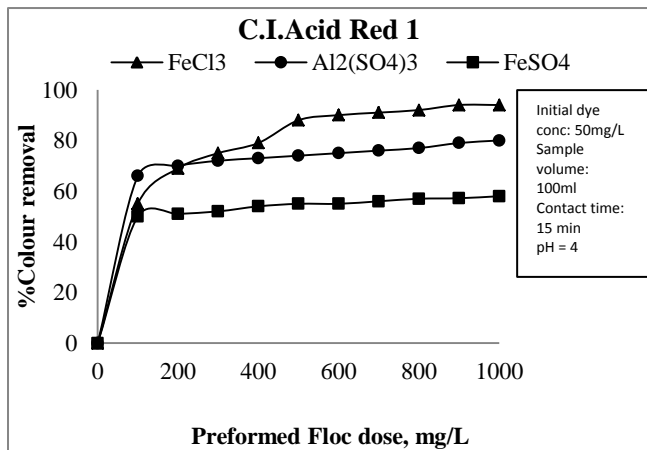


Fig.3. Response of C.I. Acid Red 1 to preformed flocs at different dosages (pH=4)

From Fig.3, it was observed that C.I. Acid Red 1 dye, Azo, responded favorably to preformed flocs of all coagulants and the colour removal range is decreasing in the order of

coagulant Ferric chloride, Aluminium Sulphate and Ferrous Sulphate at pH of 4. Excellent colour removal of 95 % was observed with Ferric chloride preformed flocs, Good colour removal of 80% was observed with Aluminium Sulphate preformed flocs and Moderate colour removal of 58% was observed with Ferrous Sulphate preformed flocs at pH of 4.

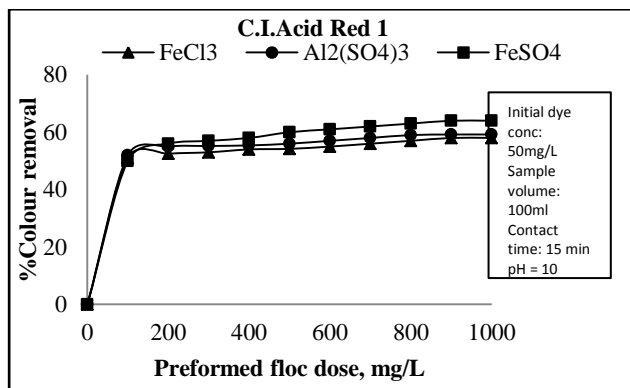


Fig.4. Response of C.I. Acid Red 1 to preformed flocs at different dosages (pH=10)

From Fig.4, it shows that C.I. Acid Red 1, responded moderately to preformed flocs of all flocs and the colour removal range is decreasing in the order of coagulant Ferrous Sulphate, Aluminium Sulphate and Ferric Chloride at pH of 10. Moderate colour removal of 65% was observed with Ferrous Sulphate preformed flocs, 59% with Aluminium Sulphate preformed flocs and 58% with Ferric Chloride preformed flocs at pH of 10

4.3. Equilibrium study

Adsorption isotherms are mathematical models that describe the distribution of the adsorbate species among liquid and adsorbent, based on a set of assumptions that are mainly related to the homogeneity/ heterogeneity of adsorbents, the type of coverage and possibility of interaction between the adsorbate species. Adsorption data are usually described by adsorption isotherms, such as Langmuir, Freundlich and Temkin isotherms. These isotherms relate dye uptake per unit mass of adsorbent,  $q_e$ , to the equilibrium adsorbate concentration in the bulk fluid phase  $C_e$ .

4.3.1. The Langmuir isotherm

The Langmuir model (Weihuaet.al., 2013) is based on the assumption that the maximum adsorption occurs when a saturated monolayer of solute molecules is present on the adsorbent surface, the energy of adsorption is constant and there is no migration of adsorbate molecules in the surface plane.

The linear form of Langmuir isotherm is given as;

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{q_m K_L C_e}$$

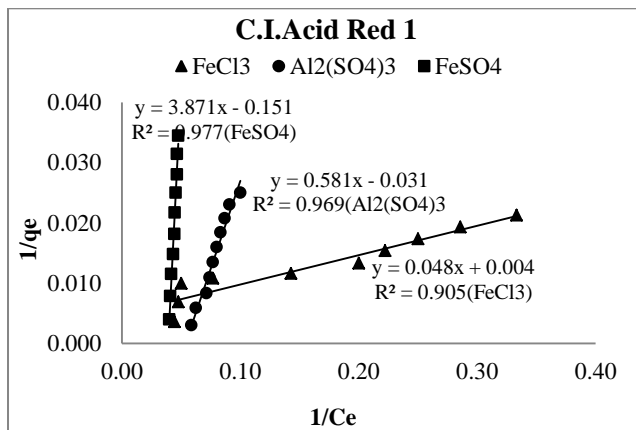
**Table.2.** Isotherm Model constants and Correlation Coefficients for Adsorption of Dye from Aqueous Solution

Adsorbent	Langmuir isotherm			Freundlich isotherm		
	q <sub>m</sub> (mg/g)	K <sub>L</sub> (L/mg)	R <sup>2</sup>	K <sub>F</sub> (mg of color/g of flocs)	n	R <sup>2</sup>
Aluminium Sulphate	32.26	0.0533	0.969	0.369x10 <sup>-2</sup>	0.25	0.936
Ferric Chloride	250	0.0833	0.905	25.58	1.698	0.789
Ferrous Sulphate	6.63	0.039	0.977	5.3x10 <sup>-14</sup>	0.09	0.942

**Table.3.** Comparison between the Adsorption Rate constants, q<sub>e</sub>, and correlation co-efficients associated with Pseudo-first-order and Pseudo-second-order equations

Coagulant	Pseudo-first-order equation			Pseudo –second-order rate equation			
	K <sub>1</sub> min-1	q <sub>e</sub> mg/g	R <sup>2</sup>	K <sub>2</sub> gmg <sup>-1</sup> min <sup>-1</sup>	q <sub>e</sub> mg/g	R <sup>2</sup>	h mgg <sup>-1</sup> min <sup>-1</sup>
Aluminium Sulphate	0.0461	2.576	0.841	0.0855	62.5	1.0	333
Ferric Chloride	0.076	6.98	0.922	0.05	100	1.0	500
Ferrous Sulphate	0.119	46.77	0.910	0.004	100	1.0	40

Where q<sub>m</sub> and K<sub>L</sub> are the Langmuir constants, representing the maximum adsorption capacity for the solid phase loading and the energy constant related to the heat of adsorption respectively. The plot between 1/q<sub>e</sub> and 1/C<sub>e</sub> is as shown in the Fig.5.



**Fig.5.** Langmuir Adsorption Isotherm

From Fig.5, it can be concluded that the isotherm data fits the Langmuir equation well, as it is straight line [8].

4.3.2. The Freundlich isotherm

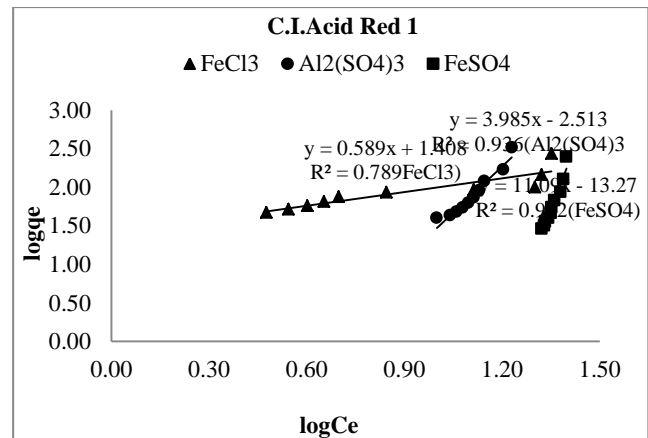
The Freundlich isotherm model (Srichandana et.al, 2012) is an empirical relationship describing the adsorption of solutes from a liquid to a solid surface and assumes that different sites with several adsorption energies are involved. Freundlich adsorption isotherm is the relationship between the amounts of dye adsorbed per unit mass of adsorbent, q<sub>e</sub>, and the concentration of the dye at equilibrium, C<sub>e</sub>.

$$q_e = K_F C_e^{1/n}$$

The logarithmic form of the equation is

$$\log q_e = \log K_F + \frac{1}{n} \log C_e$$

Where k<sub>F</sub> and n are the Freundlich constants. K<sub>F</sub> and n are the indicators of the adsorption capacity and adsorption intensity, respectively. The ability of Freundlich model to fit the experimental data was examined. Plot between logC<sub>e</sub> and logq<sub>e</sub> was drawn to generate the intercept value of k<sub>F</sub> and the slope of n as shown in the Fig. 6.



**Fig.6.** Freundlich Adsorption Isotherm

The values of sorption capacities and coefficient of correlation (R<sup>2</sup>) for various sorbents and equilibrium models are as shown in the Table 2.

From Table 2 above, The values of coefficient of correlation (R<sup>2</sup>) was obtained as 0.969,0.905 and 0.977 for Aluminium Sulphate, Ferric Chloride and Ferrous Sulphate giving a best fit for Langmuir equation compared to Freundlich Isotherm. The monolayer capacity (q<sub>m</sub>) and adsorption energy K<sub>L</sub> calculated from the linear plot are given in the same table.

It follows from the data that the equilibrium adsorption of dye onto Aluminium Sulphate, Ferric chloride and

Ferrous Sulphate preformed flocs follows Langmuir isotherm model, which reflects the formation of a monolayer of sorbate over a homogeneous surface of uniform energy and that the adsorbed layer is unimolecular (Hameedet.al. 2008).

It may also be observed that the isotherm fits of ferrous Sulphate and Aluminium Sulphate have negative intercepts which indicates that the removal is good at lower concentrations, but not as good, at higher concentrations.

4.4. Kinetic Study

In order to investigate the controlling mechanism of adsorption processes such as mass transfer and chemical reaction, the pseudo-first-order and pseudo-second-order equations are applied to model the kinetics of dye adsorption onto preformed flocs (Senthilet.al. 2009). The pseudo-first-order rate equation is given as

$$\text{Log}(q_e - q_t) = \text{Log}(q_e) - \left(\frac{k_1}{2.303}\right)t$$

Where  $q_t$  and  $q_e$  are the amount of color adsorbed (mg/g) at time  $t$ , and at equilibrium respectively and  $k_1$  is the rate constant of the pseudo-first-order adsorption process ( $\text{min}^{-1}$ ). To determine the correlation coefficients, graph was drawn between  $\log(q_e - q_t)$  and time as shown in the Fig.7.

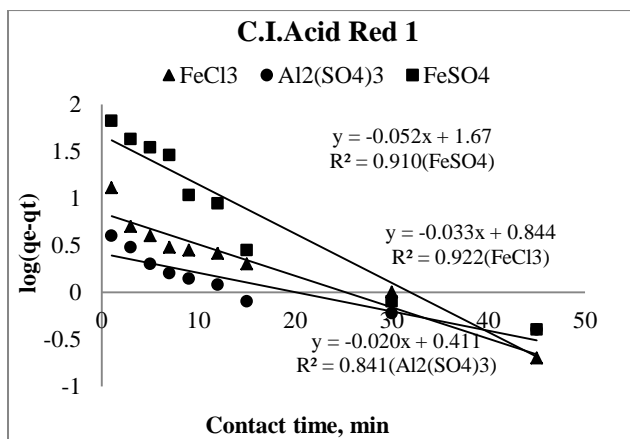


Fig.7. Pseudo-first-order Reaction for dye Adsorption onto preformed flocs

The pseudo-second order Kinetics is expressed as:

$$\frac{t}{q_t} = \frac{1}{h} + \frac{t}{q_e}$$

Where  $h = k_2 q_e^2$  ( $\text{mg g}^{-1} \text{min}^{-1}$ ) can be regarded as initial adsorption rate as  $t \rightarrow 0$  and  $k_2$  is the rate constant of pseudo-second-order adsorption ( $\text{g mg}^{-1} \text{min}^{-1}$ ).

The graph between  $t/q_t$  versus  $t$  was drawn as shown in the Fig.8, to determine the values of  $q_e$ ,  $k$  and  $h$  from the slope and intercept of the plot.

Adsorption rate constants  $q_e$  and correlation coefficients from pseudo-first-order and pseudo-second-order plots are as shown in the Table 3.

From the Table.3, it was observed that pseudo-second-order model yields very good straight lines ( $R^2=1$ ) for all coagulants as compared to the plot of pseudo-first order. It is clear that adsorption of dye onto preformed flocs follows second order kinetic model, which relies on the assumption that chemisorption may be the rate-limiting step.

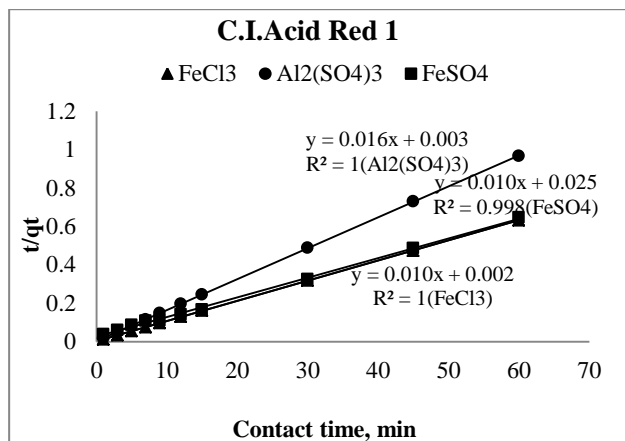


Fig.8 Pseudo-second-order Reaction for Dye Adsorption onto preformed floc

4.5. Sorption Mechanism

In order to gain insight into the mechanisms and rate controlling steps affecting the kinetics of adsorption, the kinetic experimental results were fitted to the Weber's intraparticle diffusion effects (Sri Chandana et.al. 2012). The results were analyzed by the intraparticle diffusion model to elucidate the diffusion mechanism, which model is expressed as

$$q_t = k_{id} t^{1/2} + C$$

Where  $q_t$  is the amount of color adsorbed per gram of adsorbent,  $t$  is the time of uptake,  $C$  is the intercept and  $k_{id}$  is the intraparticle diffusion rate constant ( $\text{mg/g min}^{1/2}$ ). The time dependence of uptake of color of Acid Red 1 dye by preformed flocs is portrayed in Fig.9. The obtained plots were of general characteristic shape, where the initial curved portion may be attributed to boundary layer diffusion effects; while the final linear portion is due to the intraparticle diffusion effects (Sri Chandana et.al. 2012). Since the straight line did not pass through the origin it can be said that the intraparticle diffusion alone is not the rate controlling step.

4.6. Characterization of Sorbent

The FTIR spectrum of dye before adsorption is as shown in Fig.10. The FTIR spectrum of Aluminium Sulphate, Ferric Chloride and Ferrous Sulphate (loaded sorbents) are as shown in Fig.11, Fig.12 and Fig.13.

The IR spectra were recorded for the Acid Red 1 dye molecule in dilute aqueous solution and the flux after filtration and drying the residue after loading with

Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>.16H<sub>2</sub>O and their spectra are reproduced as shown in Fig.10 and Fig.11. Respectively. The Acid Red 1 dye exhibited three bands at 3361 cm<sup>-1</sup> (-NH stretching), 1636 cm<sup>-1</sup> (-O-H bending) and 1097 cm<sup>-1</sup> (C-O stretch). The IR Spectrum of flux after loading with Aluminium Sulphate showed three bands at 3467 cm<sup>-1</sup>, 1645 cm<sup>-1</sup> and 1140 cm<sup>-1</sup>. The bands were observed at higher frequencies when compared to the free dye. It is an abnormal observation. Usually the absorption stretching bands should have been recorded at lower wave number.

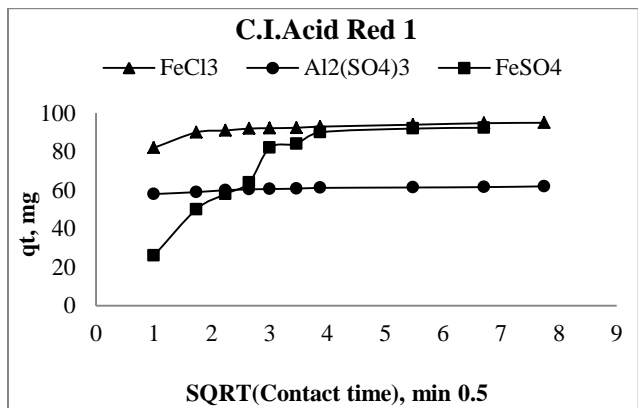


Fig.9. Intraparticle diffusion plot for sorption of Acid Red1 onto preformed flocs of Aluminium Sulphate, Ferric chloride and Ferrous Sulphate

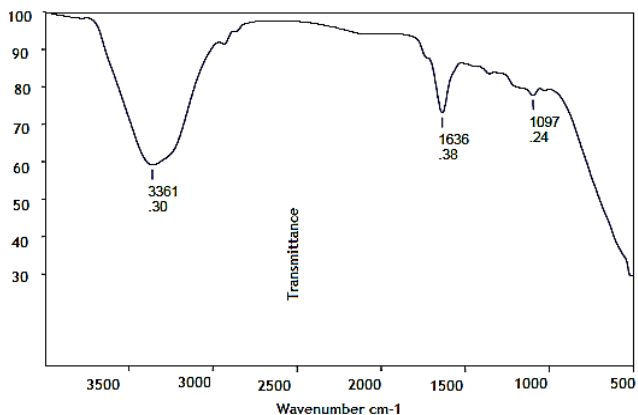


Fig.10. FTIR spectrum of Free dye Acid Red 1

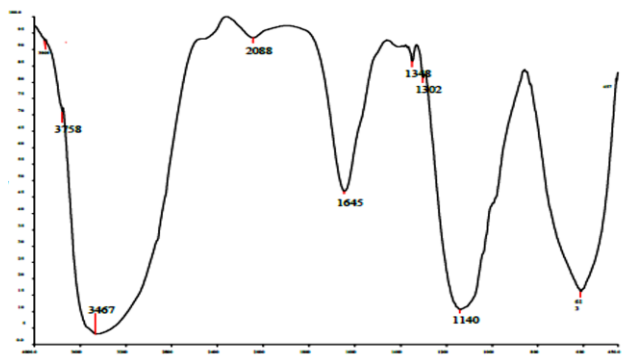


Fig.11. FTIR spectrum of preformed flocs of Aluminium Sulphate Preformed flocs (loaded)

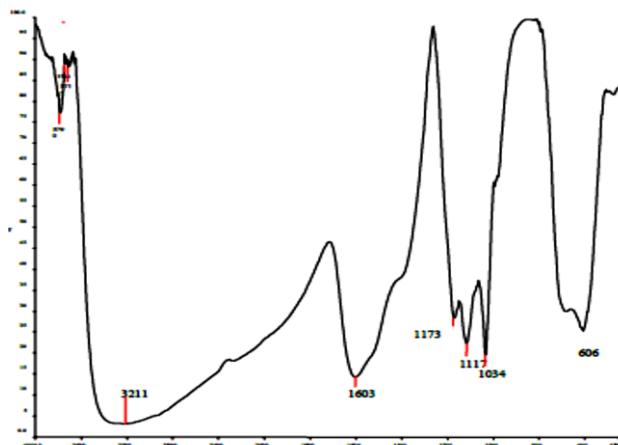


Fig.12. FTIR spectrum of preformed flocs of Ferric Chloride (loaded)

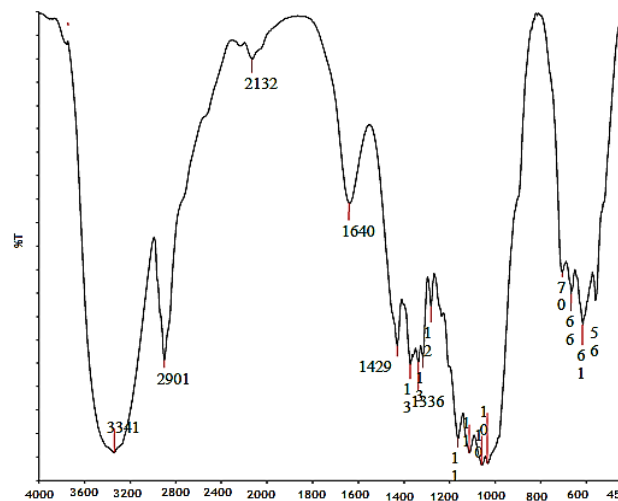


Fig.13. FTIR spectrum of preformed flocs of FeSO<sub>4</sub> (loaded)

From Fig.12, the IR spectrum of flux after loading with FeCl<sub>3</sub> showed the bands at 3790(-OH stretch), 3211(N-H stretch), 1603 (C=C stretch), 1173 (C-C stretch), 1117(C-C stretch) and 1034 (C-N stretch)cm<sup>-1</sup>. The stretching frequency of -OH band is lowered by -150 cm<sup>-1</sup> and -OH bending band is also decreased by -33 cm<sup>-1</sup> when compared to the frequencies of free dye. It indicates that Fe<sup>3+</sup> metal is interacting strongly with the dye leading to weakening of -OH bond.

It is observed that colour removal is maximum (>90%) in the case of FeCl<sub>3</sub> adsorbent. The IR spectrum of floc after loading with FeSO<sub>4</sub> as shown in Fig.13, exhibited the -OH band at slightly lower frequency (-20 cm<sup>-1</sup>) at 3341cm<sup>-1</sup>, when compared to free dye -OH band. There is no much difference in the bending frequency of -OH group. Similar observations were observed with Julid et.al, 2006.

Fig.14 and Fig.15 shows the SEM images of Adsorbent before and after dye sorption showing monolayer formation (Laila et.al. 2012).

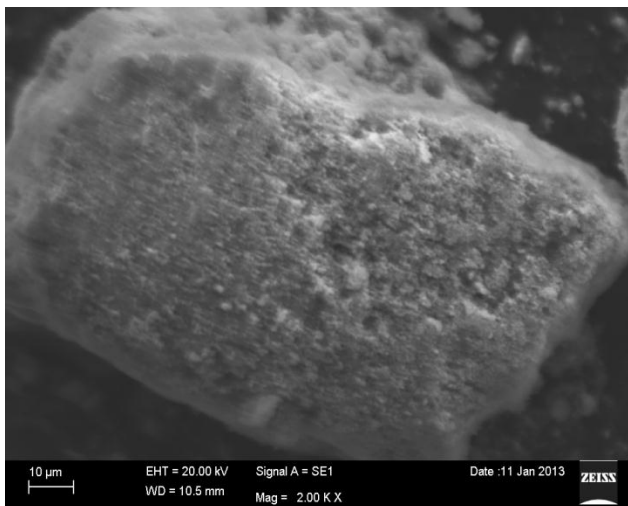


Fig.14.SEM image of un-loaded adsorbent

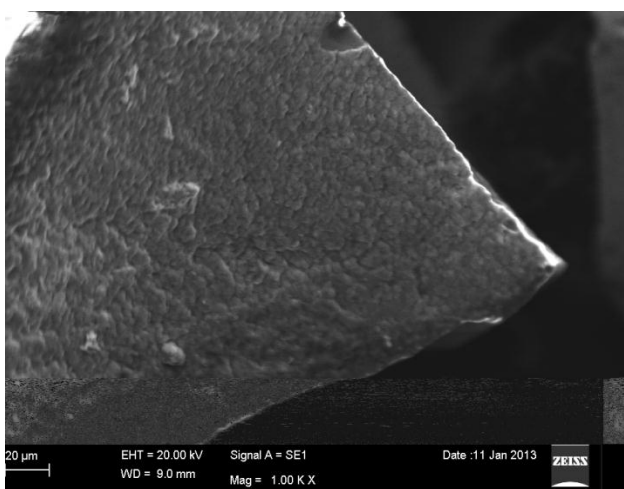


Fig.15. SEM image of loaded adsorbent

**5. Conclusions**

As evidenced in this study, Acid Red1 dye aqueous solution can be decolorized by adsorption onto preformed flocs. Excellent colour removal can be achieved with preformed flocs of Ferric Chloride, Aluminium Sulphate and Ferrous Sulphate at pH of 4 than 10. Colour removal decreased in the order of coagulants Ferric Chloride, Aluminium Sulphate and Ferrous Sulphate at pH of 4. The data fitted well with Langmuir isotherm and Pseudo second order equation stating monolayer formation and chemisorption is involved in the colour removal.

**List of symbols**

- $q_e$ - Solid phase concentration at equilibrium (mg/g).
- $q_t$  -average solid phase concentration at time t in min (mg/g)
- $k_1$  - pseudo-first order rate constant
- $k_2$ - pseudo-second order rate constant
- $k_{id}$  -intraparticle diffusion rate coefficient

- $C_e$  and  $1/n$  -empirical constant related to the magnitude and driving force of adsorption
- $K_L$  -Langmuir constant
- $q_m$  -Maximum amount of adsorption corresponding to complete monolayer coverage.

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