

Evaluation of the Adsorption Kinetics and Equilibrium for the Potential Removal of Congo Red Dye From Aqueous Medium by Using a Biosorbent

Abstract:

Activated Mangifera Indica Saw Dust (AMSD) has been investigated as low cost and eco-friendly biosorbent prepared for the removal of Congo red (CR) in aqueous solution. Various adsorption parameters such as effect of contact time, adsorbent dose, effective pH and initial concentration of Congo red on the adsorption capacity of the adsorbent were studied. The adsorbent was also characterized by FTIR, XRD and SEM Analysis. The equilibrium data followed both Langmuir and Freundlich isotherm. The kinetic parameters of adsorption such as Lagergren Pseudo first order, Pseudo second order have also been studied. These studies suggest that the AMSD Could be used as a low cost alternative for waste water treatment for dye removal.

Keywords : Adsorption, kinetics, Isotherm, Congo red, XRD, SEM.

1. INTRODUCTION :

Water pollution with toxic compounds is of major concern for human health as well as for the environmental quality[1]. The wide use of synthetic dyes in textile dyeing, paper and pulp and other industrial application has caused large scale development of dye industries and consequently has produced large volume of dye waste water[2]. Due to ever growing demands in textile, synthetic organic dyes are widely used for dyeing textile fibers such as cotton and polyester, Approximately 10,000 different dyes and pigments are used for textile industries and over 7×10^5 tons of waste produced annually. Considering volumes discharged and effluent composition, water from textile industry was declared as one of the major source of wastewater in Asian Countries. Discharge of such effluents impacts color to receiving streams and affect its aesthetic value[3]. The Textile dyeing industry consumes large quantities of water at its different steps of dyeing, finishing etc. Processes, due to large volume of water consumption, the Production of huge Volume of wastewater is inevitable. Generally, the wastewater from Printing and dyeing unit in a textile plant is rich in color, containing residues of dyes and chemicals. Dye wastewater is characteristically high in

30 organic content, high salt content and low in biodegradation[4]. Many pollutants in
31 wastewater from dye manufacturing, printing and textile industries are color left by dyes.

32 The discharge of dye bearing waste water into natural streams or on land has
33 created significant concerned as the dye impact toxicity and impedes light penetration and
34 thus upsets the biological activity[5]. Color is one of the most important hazards in industrial
35 effluents because the presence of dyes in water reduced light penetration, precluding the
36 photosynthesis of aqueous flora. Besides that some dyes may cause Allergy, dermatitis skin
37 irritation and cancer to human in addition to being mutagenic[6]. the industrial effluents
38 discharge from dyeing and printing industries are highly colored with a large amount of
39 suspended organic solid. These effluents are discharge into rivers make water unfit for
40 domestic, agricultural and industrial purposes. Many dyes and their break down Product may
41 be toxic for living organism; therefore removals of dyes are most important aspects of waste
42 water treatment before discharge[7].

43 Traditionally, biological, physical and chemical method has been tested for dye
44 removal. Biological Process is probably the most in expensive one but many dyes are poorly
45 biodegradable due to their complex structure and xenobiotic properties moreover many dyes
46 are toxic to microorganism, causing direct destruction of their catalytic capabilities. Reverse
47 osmosis and electrochemical coagulation is complex and generally not feasible on a large
48 scale due to economic consideration advanced oxidation processes ($\text{CH}_2\text{O}_2/\text{UV}$, O_3) have the
49 potential to eliminate organic carbon of dye wastewater; however, they are too expensive.
50 Chemical coagulation needs loading of chemical coagulant and optimal Operation condition
51 such as PH and coagulant dose[8]. Adsorption is an effective and simple method for dye
52 removal.

53 In adsorption Process activated carbon is the most popular and widely used dye
54 adsorbent due to their high specific Surface area and high removal efficiency for most of
55 dyes. In spite of this it suffers from few disadvantages such as activated carbon is quite
56 expensive and its regeneration Produced additional effluent and results considerable loss of
57 the adsorbent [9]. Thus use of several low cost adsorbents has been studied by many
58 researchers. They have studied the feasibility of using low cost material such as waste orange
59 peel,[10] banana pith,[11] neem leaf powde,[12] bagasse fly ash,[13] yellow passion fruit
60 peel,[14] oil palm fire-based activated carbon,[15] corncob activated carbon[16] teak leaf,
61 maize corn and babool tree bark carbon,[17] pumpkin seed hull,[18] and ground egg shell
62 waste,[19] as adsorbent for removal of dyes in aqueous solutions.

The objective of the present communication is to evaluate the adsorption kinetics and equilibrium for the removal of Congo red (CR) dye using Activated mango saw dust (AMSD). Mango tree is a plant belongs to the family anacardiace (mangifera indica) is an ever green plant, which is extensively cultured in India.²⁰

2. EXPERIMENTAL DETAILS

2.1. Materials and Method:

2.1.1. Preparation of adsorbent :

The mango saw dust was collected from the local saw mill. Mango saw dust was cleaned, thoroughly washed with distilled water and then dried in an oven. Now it was further treated with hydrogen peroxide (100 volumes) for about 24 hours to remove all adhering organic particle and dried at 110⁰C for one hour in a vacuum oven. Finally, granules of activated saw dust thus obtained were stored in vacuum desiccators until required.

2.1.2. Preparation of stock Solution :

Congo red (CR) (Direct Red 28, C.I.22120, azo dye) supplied by Loba Chemical Ltd., (India) were used as adsorbate without further purification. The stock solutions (1000 mg/L) were prepared by dissolving accurately weighed quantity of dye (CR) in double distilled water. Experimental solutions of the desired concentration were obtained by successive dilutions.

2.2. Adsorption Studies :

The adsorption experiment were carried out in a batch process by using aqueous solution of Congo red (CR) with activated mango saw dust (AMSD) the experiments were conducted to observe the effect of contact time, adsorbent dose, PH and initial concentration of Congo red at equilibrium condition for the concentration of dye over range 10 mg/L to 90 mg/L at a fixed pH. In each experiment an accurately weighed amount of AMSD was added to 50 ml of Congo red solution in 100 ml stopper bottle and the mixture were agitated in mechanical shaker for a given length of time at constant temperature 27⁰C

The adsorbent was separated from solution by centrifugation. The absorbance of the supernatant solution was estimated to determine the residual concentration. Residual dye concentration was determined using absorbance values measured before and after the treatment at $\lambda_{\text{max}} = 500 \text{ nm}$ by spectrophotometer (Systronic model-2206) with a quartz cell of 1 cm path length. The experiments were carried out at initial PH values ranging from 2 to 10. The initial PH of solution was controlled by addition of 0.1 M HCl and 0.1 M NaOH solutions. Kinetics of adsorption was determined by analyzing adsorptive uptake of the dye

from aqueous solution at different time intervals. Two main system variables, initial dye concentration and contact time, were varied to investigate their effect on adsorption kinetics.

The FTIR, X-ray diffraction (XRD), Scanning Electron Microscope (SEM) studies of the adsorbent were carried out at SICART, V.V Nagar (Gujarat).

3. RESULTS AND DISCUSSION

3.1. Adsorbent Characterization : For structural and morphological characteristics F.T.I.R., X.R.D., and SEM of adsorbent AMSD were used.

3.1.1. F.T.I.R. Analysis:

FTIR spectra were obtained for activated mango saw dust before and after the adsorption process shown in fig.1 & 2 respectively. The I.R. absorption band and corresponding possible groups are shown in table-1. The Main functional groups involved in adsorption process were found to be =C=O , -COOH , R-OH and Ar-NH_2 , R-NH_2 groups. The broad intense absorption peaks detected at 3422 cm^{-1} and $3421.\text{cm}^{-1}$ of AMSD before and after Congo red (CR) adsorption could be assigned to O-H stretching vibration of hydroxyl group. The bands observed at about $2923.\text{cm}^{-1}$ and $898.\text{cm}^{-1}$ could be assigned to the -CH group stretching.

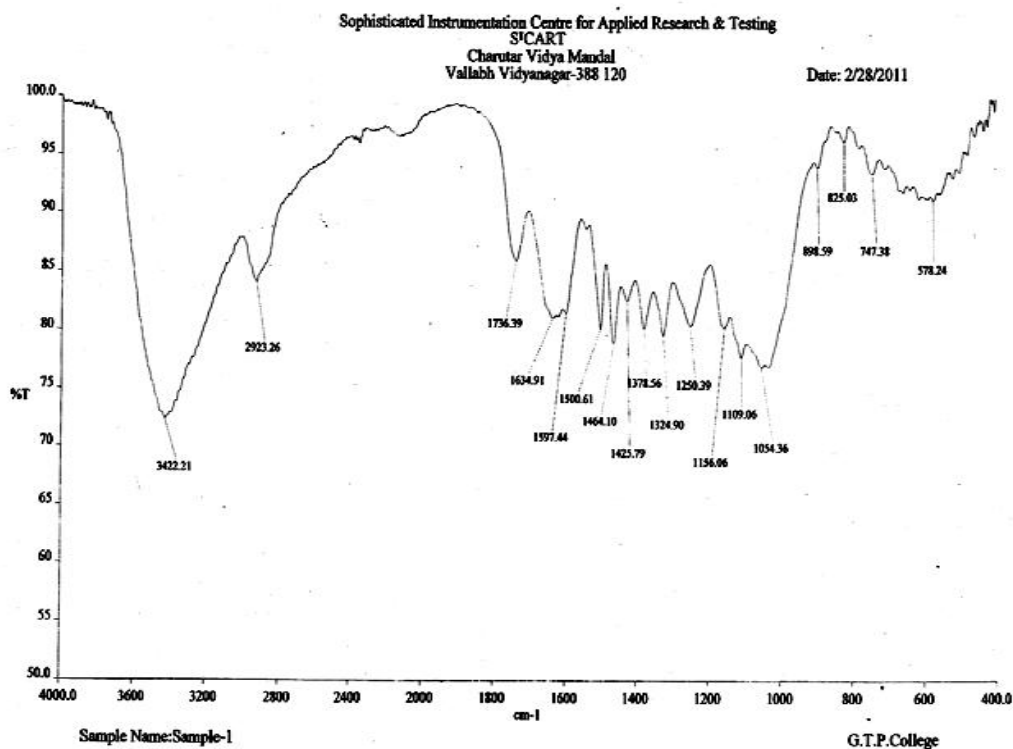


Fig-1: FTIR Spectra of AMSD before adsorption of Congo red.

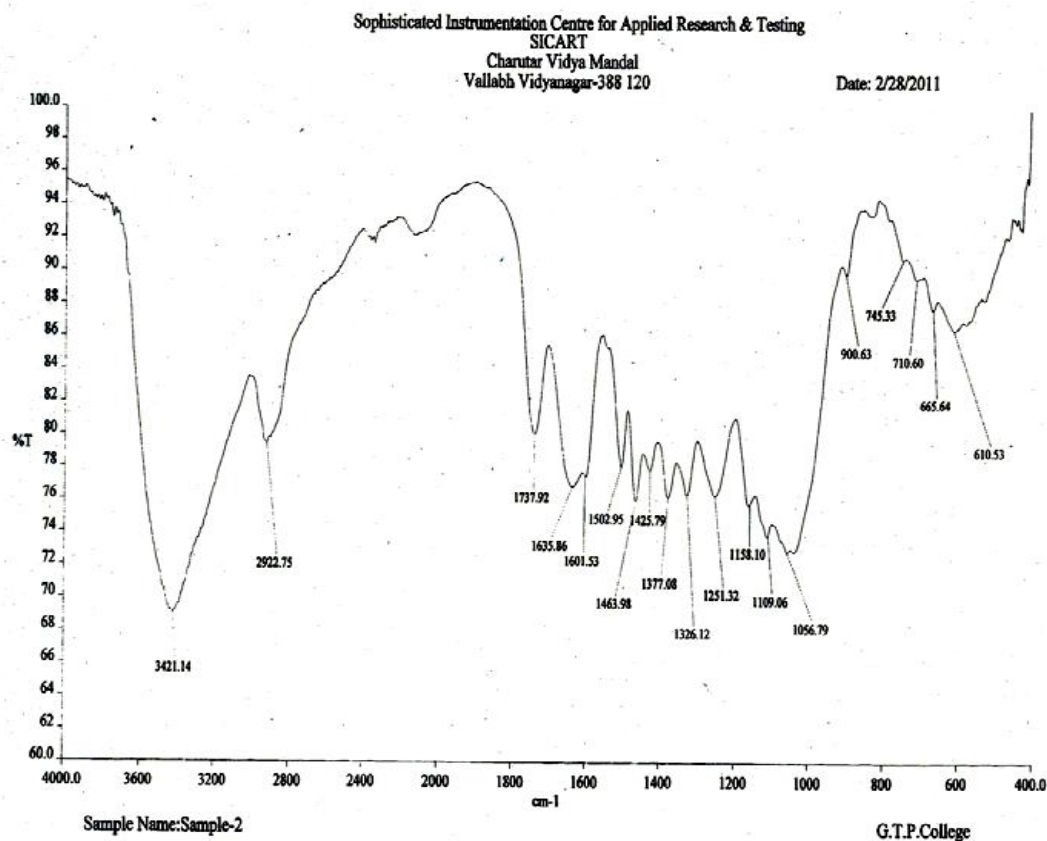


Fig-2: FTIR Spectra of AMSD after adsorption of Congo red.

The peak located at 1634cm^{-1} and 1736cm^{-1} is characteristic of carbonyl group stretching from aldehyde and ketones. The band appears at $1054\text{-}1109\text{cm}^{-1}$ is due to C-O or C-N group Stretching. The absorption peak at 1250 cm^{-1} indicates presence of C-O stretching in alcoholic or ester group. While the absorption peaks at 1464 , 1500 and 1597 cm^{-1} and bending vibration band at $747\text{-}898\text{ cm}^{-1}$ are mainly due to the C=C bond ring stretching (Phenyl ring). The involvement of these functional groups in dye adsorption process can be judged from change in frequency of absorbing groups (fig.1 and 2).

Fig.1 and 2 shows presence of several functional groups for binding Congo red on activated mango saw dust surface.

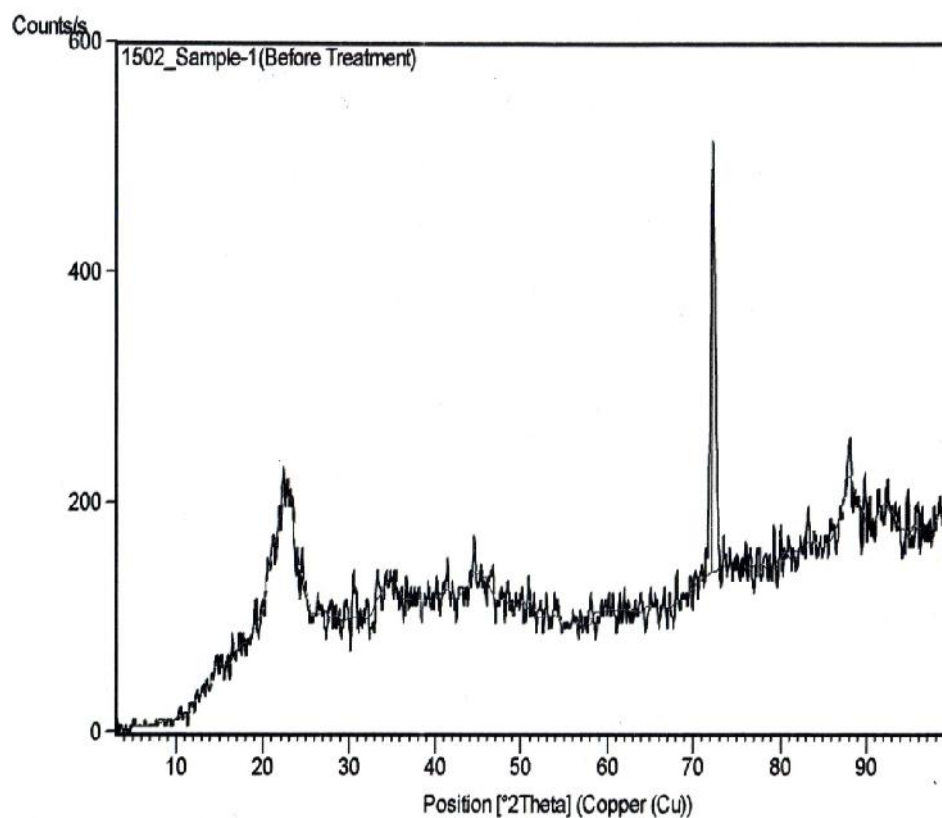
Table-1: I.R. Adsorption bands and corresponding possible groups.

Adsorption band (Cm^{-1}) before adsorption	Adsorption band(Cm^{-1}) after adsorption	Functional Groups
3422	3421	-OH,-NH ₂
2923,	2922	-CH
1736	1737	-C=O
1054-1109	1056-1109	-C-O or-C-N
1634	1635	-COO ⁻ , -C=O
1500-1597	1502-1601	=C=C=
747-898	747-898	=C=C= (ring structure)
1250	1251	-C-N

3.1.2 XRD analysis:

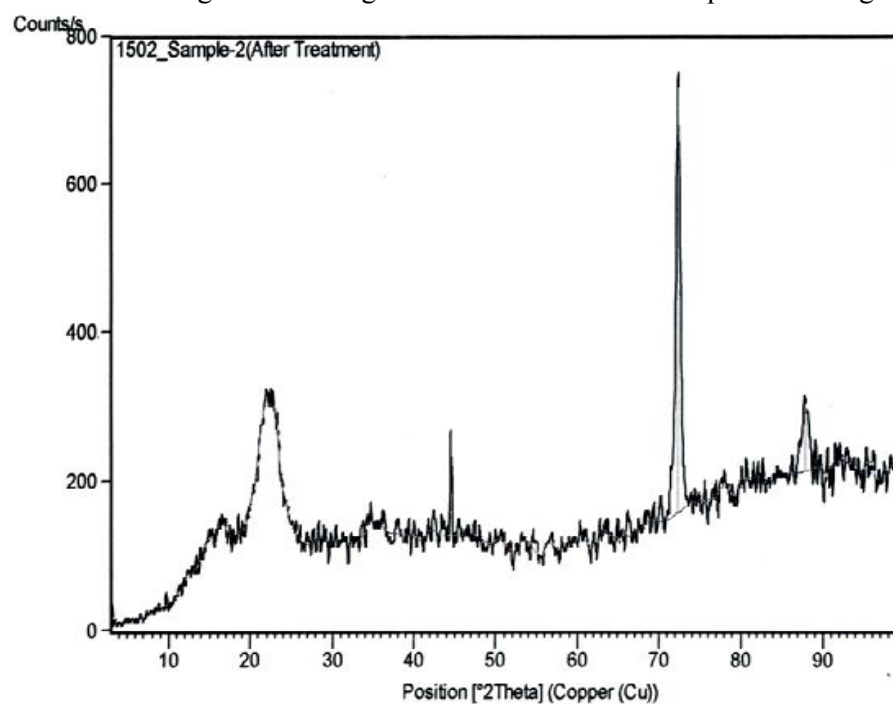
The XRD diagrams of activated mango saw before and after the adsorption process are shown in fig.3 and 4 and table 2 and 3 respectively. The XRD pattern of the adsorbent shows typical spectrum of cellulose material having main and secondary peaks. The Main peak is taken as indicative of the presence of highly organized crystalline cellulose, while secondary peak is a measure of polysaccharide structure (21).

In present study XRD Pattern of adsorbent before treatment showed spectrum having main peak at 2θ of 72.2877 , while the XRD spectrum of adsorbent after treatment show three main peaks of 2θ of 44.539 , 72.3548 , 87.9629 , respectively.



141

142 Fig-3: XRD diagram of AMSD before adsorption of Congo red.



143

144 Fig:-4 XRD diagram of AMSD after adsorption of Congo red.

Table-2 :XRD peak position of AMSD before adsorption of Congo red.



SOPHISTICATED INSTRUMENTATION CENTRE FOR APPLIED RESEARCH & TESTING (SICART)						
MOTA BAZAR - VALLABH VIDYANAGAR - 388 120						
No.	Visible	Dataset Name	Start pos. [°2Th.]	End pos. [°2Th.]	Step [°2Th.]	Measured Date/Time
1	TRUE	1502_Sample-1(Before Treatment)	3.025	99.975	0.05	2/15/2011 15:12
No.	Pos. [°2Th.]	d-spacing	Significance	Rel. Int. [%]	Height	FWHM [°2Th.]
1	72.2877	1.306	6.3488	100	376.09	0.72

Table-3: XRD peak position of AMSD after adsorption of Congo red.

SOPHISTICATED INSTRUMENTATION CENTRE FOR APPLIED RESEARCH & TESTING (SICART)						
MOTA BAZAR - VALLABH VIDYANAGAR - 388 120						
No.	Visible	Dataset Name	Start pos. [°2Th.]	End pos. [°2Th.]	Step [°2Th.]	Measured Date/Time
1	TRUE	1502_Sample-2(After Treatment)	3.025	99.975	0.05	2/15/2011 15:20
No.	Pos. [°2Th.]	d-spacing	Significance	Rel. Int. [%]	Height	FWHM [°2Th.]
1	44.5839	2.03238	1.2161	15.65	143.19	0.246
2	72.3548	1.30604	3.4917	100	571.75	0.3936
3	87.9629	1.10926	0.846	51.8	89.83	0.96

3.1.3.SEM Analysis:

Scanning electron microscopy (SEM) has been a primary tool for characterizing the surface morphology and fundamental physical properties of the adsorbent surface it is useful for determining the particle, shape, porosity and appropriate size distribution of adsorbent, scanning electron micrographs of adsorbent before and after treatment are shown in fig. 5 and 6, respectively.

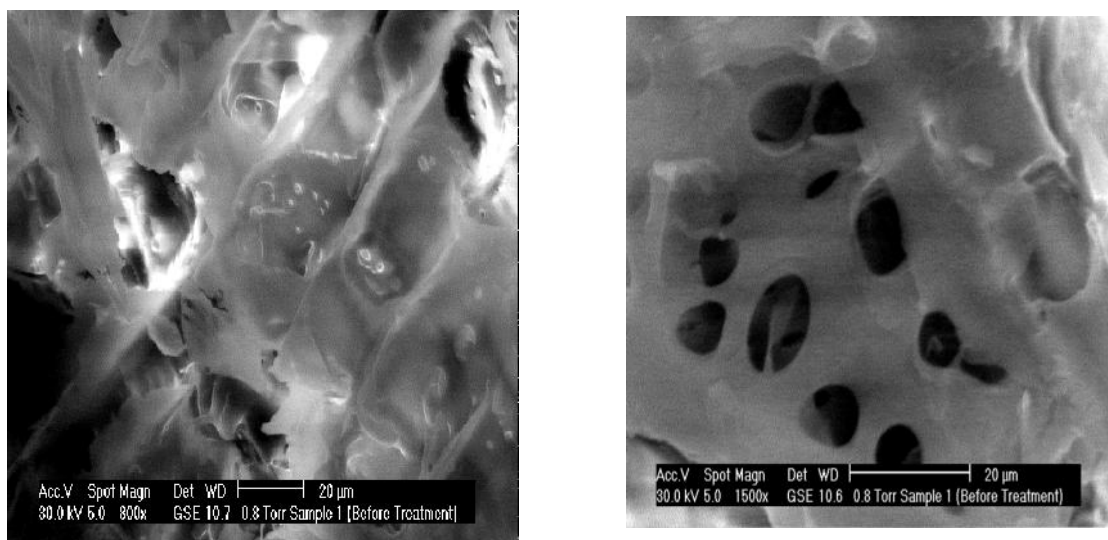


Fig-5: SEM micrograph of AMSD before adsorption of Congo red.

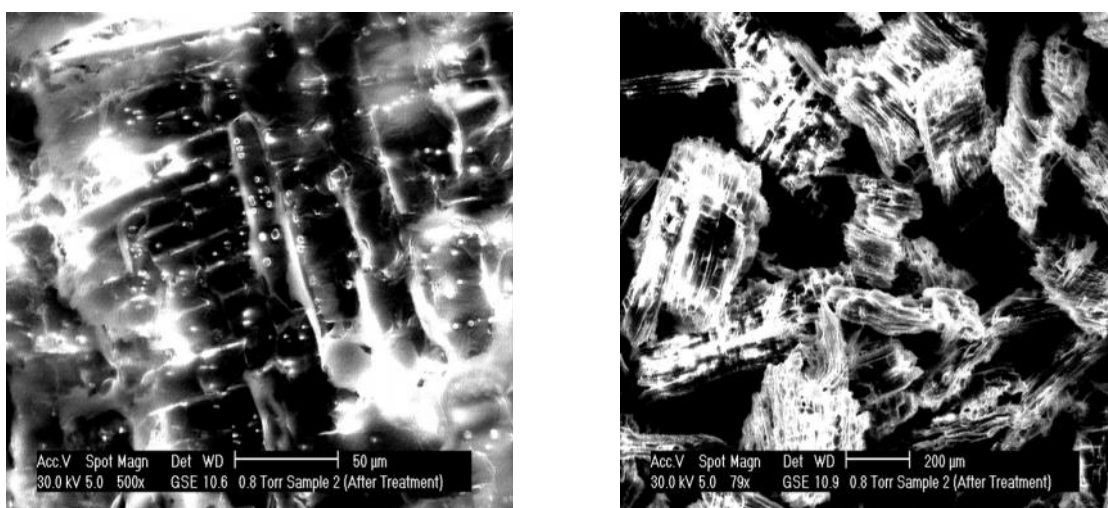


Fig-6: SEM Photo graph of AMSD after adsorption of Congo red.

From fig.-6, it is clear that activated mango saw dust has considerable numbers of pores before treatment where there is a good possibility for dye to be trapped and adsorbed into these pores while fig.6 shows SEM picture of activated mango saw after treatment the fig.6 show very distinguished dark spots which can be taken as a sign of effective adsorption of dye molecule in the cavities and pores of this adsorbent.

3.2 Adsorption Studies:

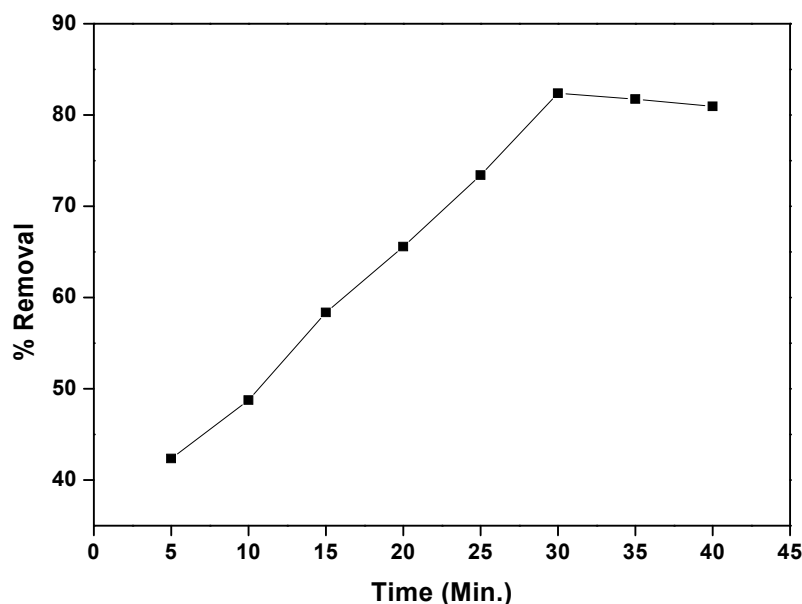
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177 **Effect of Operational Parameters:**

178

179 **3.2.1. Effect of contact time:**

180 The effect of contact time on the removal of Congo red (CR) was investigated using 70 mg/L
 181 initial concentration of CR solution with 2 g/L AMSD at pH 6. The extent of removal of CR
 182 by AMSD was found to increases, reach a maximum value with increase in contact time
 183 shown in fig.-7 based on these results, 30 min for AMSD was taken as the equilibrium time
 184 in adsorption experiments.



185

186 **Fig.-7:** Effect of Contact time on % Removal of Congo red (Initial Conc. of Congo red
 187 7×10^{-5} M) adsorption dose 2 gm/L. and pH 6.)

188

189 **3.2.2. Effect Adsorbent dose:**

190 The effect of adsorbent dose on the removal of CR was studied using 70 mg/L initial
 191 concentration of CR solution. With varying adsorbent dose of AMSD from 0.5 to 4 g/L. at
 192 PH 6 and contact time 30 min. The % removal of CR increases as the adsorbent dose
 193 increases due to increase in total number of exchange sites as shown in fig.8. When adsorbent
 194 dose increases reach a maximum value (88.46%) at 2 g/L. Adsorbent dose. Based on these
 195 results 2 g/L dose of AMSD was taken as the equilibrium dose.

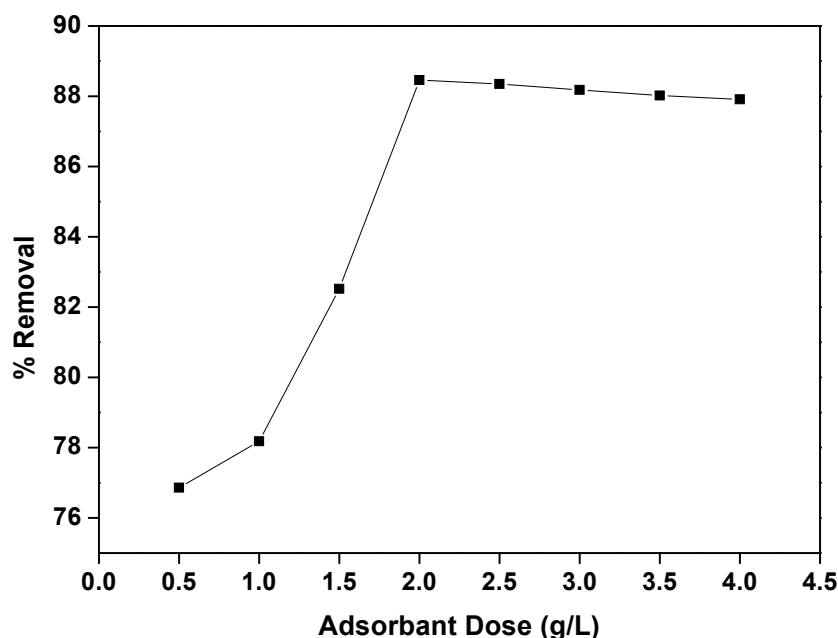


Fig.-8: Effect of adsorbent dose on % Removal of Congo red
(Initial conc. of Congo red 7×10^{-5} M) contact time 30 Min. and pH 6.

3.2.3 Effect of pH:

The PH is one of the most important factors controlling the adsorption of dye on to adsorbent to determine the optimum pH conditions for the adsorption of Congo red over AMSD the effect of pH on removal of CR was observed over entire pH range 2.0 to 10. The studies were conducted at a fixed initial concentration of CR 70 mg/L, adsorbent dose 2 g/L and contact time 30 min. The results obtained are presented in fig.-9. Which describe maximum removal of CR (84.80%) was take place at pH 6. Hence, all the succeeding investigation was performed at pH 6.

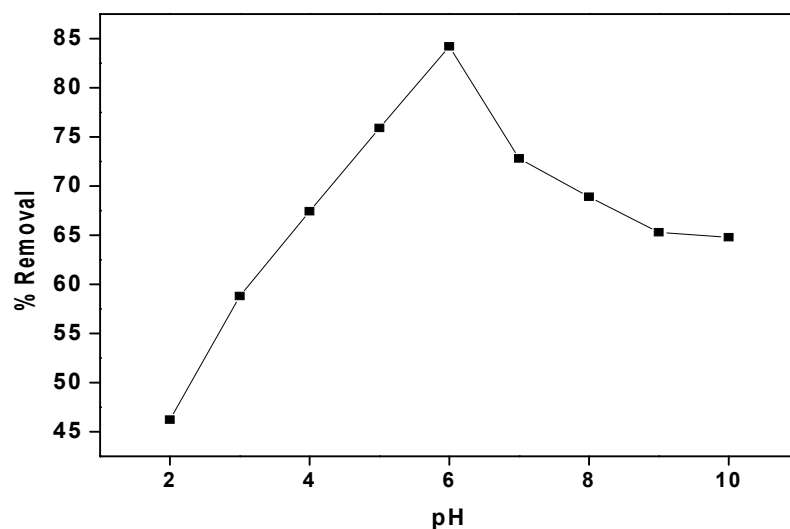


Fig.-9: Effect of pH on % Removal of Congo red (Initial conc. of Congo red 7×10^{-5} M) adsorption dose 2 gm/L., contact time 30 min.)

3.2.4. Effect of initial adsorbate concentration:

The effect of initial concentration of adsorbate (CR) on the amount adsorbed (q) is shown in fig.10. While the effect of initial concentration of adsorbate (CR) on Removal of Congo red is shown in fig.11. The initial concentration of Congo red were changed in order to determine proper Congo red adsorption keeping the adsorbent dose 2 g/L, contact time 30 min and pH 6 fig.10. Show that the amount adsorbed dyes increases with increases in dye concentration. While fig.11 showed that % removal decreases with an increase in the dye concentration. It is also note that the rate of removal of dye is faster at lower concentration and decreases with increasing concentration. It is apparent from the fig.11 that with increasing concentration of dye (adsorbate) from 10 mg/L to 90 mg/L the % removal decreases from 86.02 to 43.94% for AMSD.

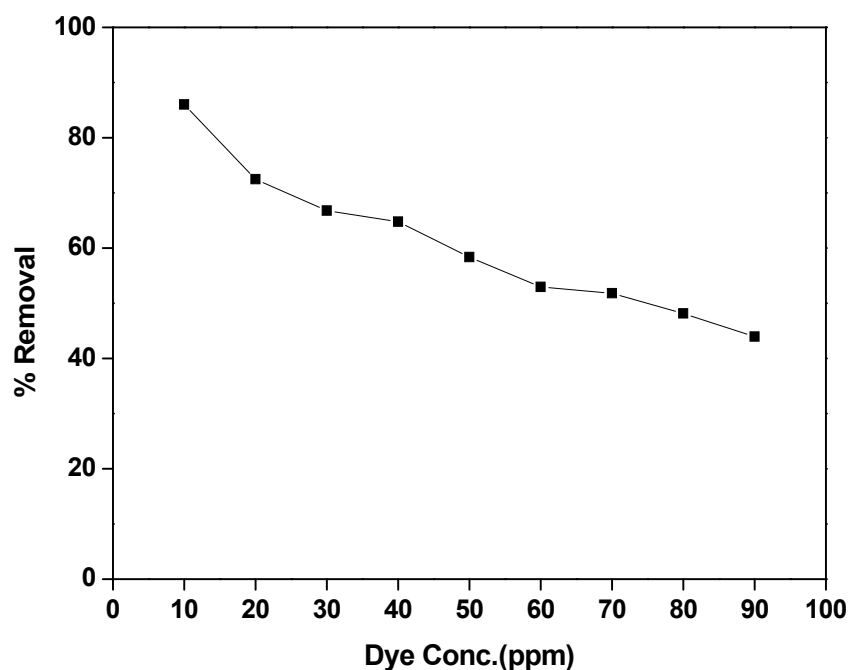


Fig.-10: Effect of Initial dye Concentration on % Removal of Congo red (Initial conc. of Congo red 7×10^{-5} M) adsorption dose 2 gm/L. and pH 6.)

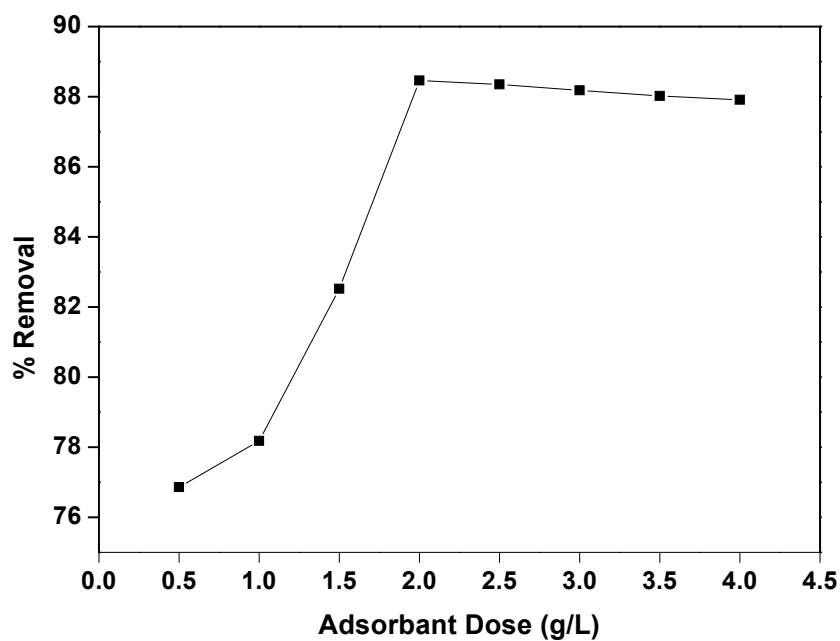


Fig.-11: Effect of Initial dye Concentration on adsorption of dye. (adsorption dose 2gm/L. contact time 30 min. and pH 6.)

3.6. Adsorption isotherms:

The distribution of dye between the liquid phase and the adsorbent is a measure of the position of equilibrium in adsorption process and can be generally expressed by Langmuir and freundlich isotherms.

The adsorption studies were conducted at a fixed initial concentration of Congo red with varying adsorbent dose. The equilibrium data were analyzed by Langmuir and freundlich isotherms.

3.6.1. Langmuir isotherm :

The Langmuir model (22) assumes uniform energies of adsorption on to the surface and no transmigration of adsorbate in the plane of the surface. The Langmuir isotherm is represented by the following equation.

$$\frac{C_e}{q_e} = \frac{1}{a b} + \frac{C_e}{b} \quad \text{-----} \quad (\text{Eq. 1})$$

Where as C_e is concentration of dye solution (mg/L) at equilibrium and q_e is the amount of CR adsorbed per unit mass of adsorbent (mg/g). a and b are Langmuir constant the constant ' a ' signifies the adsorption capacity (mg/g) and b is related to the energy of adsorption (mg/L).

The linear plot of C_e / q_e v/s C_e shows that adsorption follows Langmuir isotherm (fig-12) values of a and b were calculated from the slope and intercept of linear plots respectively and presented in table 4.

Table-4: Langmuir Constants

Concentration of Congo red (mg/L)	Langmuir Constant		
	$a(\text{mg/g})$	$b (\text{L/mg})$	R^2
70	40	0.275	0.9680

In present investigation the value of adsorption capacity (a) was 40 mg per gram of AMSD for CR. The applicability of the Langmuir isotherm suggested monolayer coverage of CR on the surface of AMSD.

The essential characteristics of the Langmuir isotherm can be expressed by dimension less constant called equilibrium parameter R_L define by equation

$$R_L = \frac{1}{(1 + b C_i)} \quad \text{————— (Eq. 2)}$$

Where b is Langmuir constant and C_i is the initial dye concentration (mg/L). R_L values obtained using equation (2) for 70 mg/L CR concentration was 0.069.

According to the value of R_L the isotherms shape and whether it is favorable or not may be interpreted as

R_L Values	Adsorption
$R_L > 1$	unfavorable
$0 < R_L < 1$	Favorable
$R_L = 0$	Irreversible

In present study the value of R_L is less than 1 and greater than zero show that adsorption of CR on AMSD is favorable.

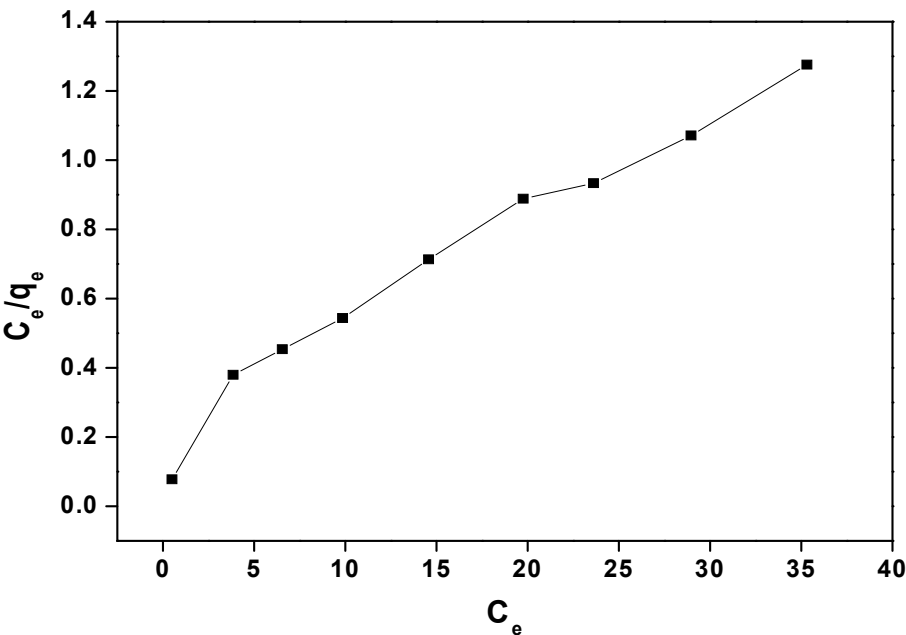


Fig. 12: Langmuir plot for absorption of Congo red by AMSD. (adsorption dose 2gm/L. contact time 30 min. and pH 6.)

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276 3.6.2. Freundlich isotherm:

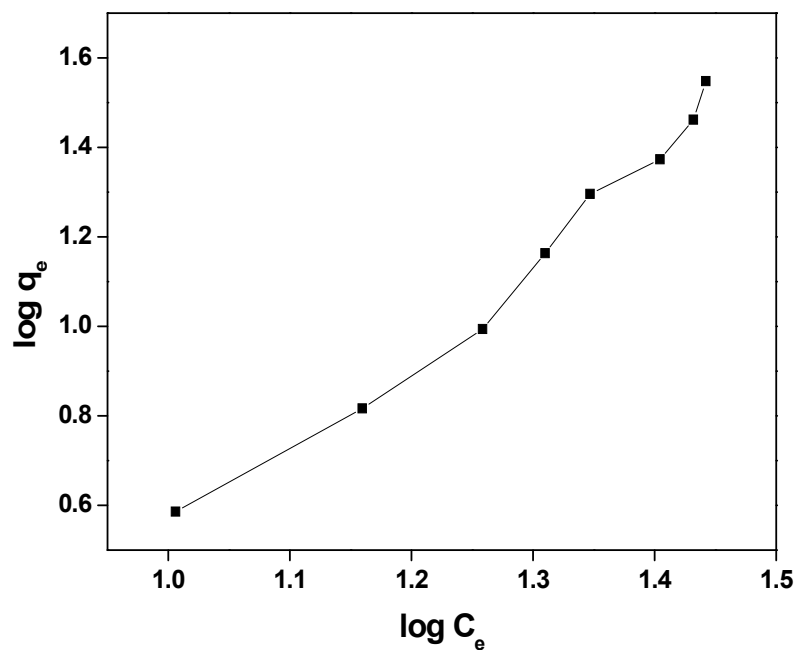
277 The freundlich isotherm (23) is an empirical equation assuming that the adsorption process
278 take place on heterogeneous surface and adsorption capacity is related to the concentration of
279 adsorbate.

280 The linear form of the freundlich equation is as follows.

281

$$282 \log q_e = \log K + \frac{1}{n} \log C_e \quad \text{—————} \quad (\text{Eq. 3})$$

283 Where log k is a measure of the adsorption capacity and n is an indicator of adsorption
284 intensity. The freundlich coefficient 1/n which should have values in the range of 0 to 1 for
285 favorable adsorption. A plot of log q_e v/s log C_e gives a slope of 1/n and intercept of log K
286 shown in fig-13. The value of and K were calculated from slope and intercept respectively
287 and are given in table 5.



288

289 **Fig.-13: Freundlich plot for adsorption of Congo red by AMSD**
290 (adsorption dose 2gm/L. contact time 30 min. and pH 6.)

291

292

293

294

Table-5: Freundlich Constant

Concentration of Congo red (mg/L)	Freundlich Constant		
	K	1/n	R ²
49	5.623	0.40	0.7818

4. Adsorption Kinetics:

In order to investigate the mechanism of adsorption two kinetic models were tested. a pseudo first order and a pseudo second order reaction.

4.1. Pseudo first order model :

Lagergren's rate equation is most widely used for the adsorption of adsorbate from a solution. The first order lagergren's rate equation is expressed as follows :

$$\log(q_e - q_t) = \log q_e - K_1 t \quad \text{—————} \quad (\text{Eq. 4})$$

Where q_e and q_t (both in mg/gm) are the solute amounts adsorbed per unit mass of adsorbent at equilibrium at time t in min. respectively K_1 (min^{-1}) is the first order rate constant.

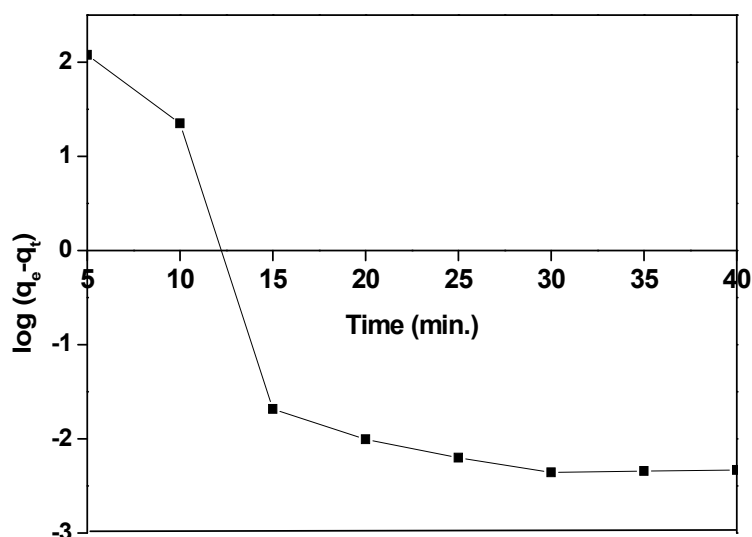
The values of K_1 and q_e were calculated from the slope and the intercept of the plot of $\log (q_e - q_t)$ vs t respectively (fig.14) the calculated values of K_1 and q_e (cal) and q_e (exp) are summarized in table 6.

Table-6: Kinetic model values for adsorption of Congo red on to AMSD.

Concentration of Congo red (mg/L)	q_e (exp) (mg/g)	Pseudo first order constant		
		q_e (cal) (mg/g)	K_1	R ²
70	33.73	50.12	0.2366	0.5254

In present investigation the value of K_1 q_e (cal) and q_e (exp) was 0.2366 min^{-1} , 50.12 mg/g and 33.73 mg/g respectively. The experimental q_e values is in agree with calculated ones,

318 obtained from the linear plots. This shows that the adsorption of CR on AMSD is a Pseudo
319 first order kinetics.



320

321 **Fig.-14:** Lagergren first order plot for adsorption of Congo red by AMSD.

322 (Initial conc. $7 \times 10^{-5} \text{M}$, adsorption dose 2gm/L. contact time 30 min. and pH 6.)

323 4.2. Pseudo second order model :

324 Pseudo second order equation based on equilibrium adsorption is expressed as

$$325 \quad \frac{1}{q_t} = \frac{1}{K_2 q_e} + \frac{t}{q_e} \quad \text{Eq. (5)}$$

326 Where K_2 (g/mg.min) is the rate constant of second order adsorption If second order
327 kinetics is applicable, the plot of t/q_t v/s t should show a linear relationship q_e and K_2 can be
328 determined from the slope and intercept of the plot. The Linear plots of t/q_t v/s t (fig-15).

329 The calculated values of K_2 and q_e (cal) are summarized in table-7.

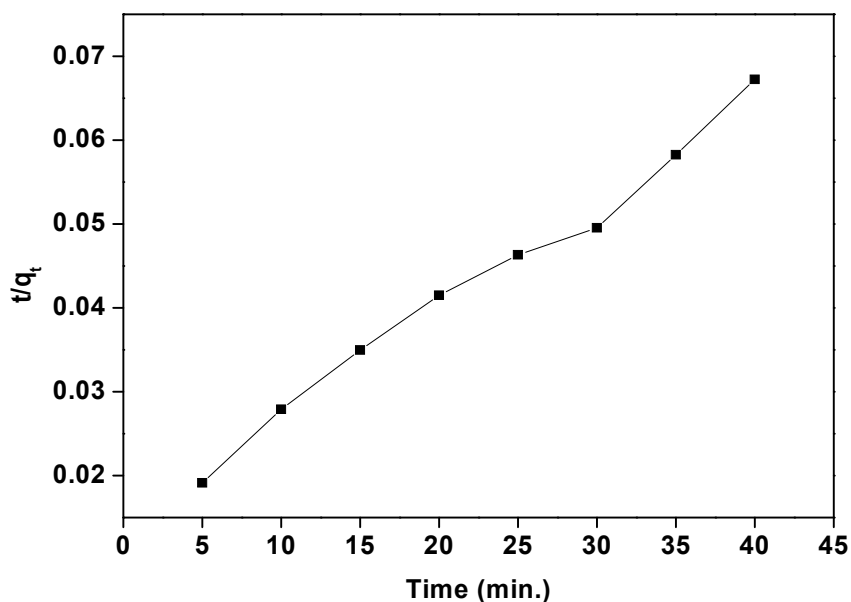
330 Table-7: Kinetic model values for adsorption of Congo red on to AMSD.

331

Concentration of Congo red (mg/L)	q_e (exp)(mg/g)	Pseudo second order		
		q_e (cal)(mg/g)	K_2	R^2
70	33.73	0.00128	0.0125	0.9938

332

333 In present investigation the values of K_2 and q_e was 0.0125 min^{-1} and 0.00128 respectively.
 334 The experimental q_e values do not agree with the calculated ones, obtained from the linear
 335 plots. This shows that the adsorption of CR on to AMSD is not a Pseudo second order
 336 kinetic.



337 **Fig.-15:** Pseudo second order adsorption. of Congo red on AMSD.
 338 (Initial conc. $7 \times 10^{-5} \text{ M}$, absorption dose 2 gm/L . contact time 30 min . and pH 6)
 339

340 5. Conclusions:

341 The Adsorption of Congo red is depends on contact time, adsorbent dose, pH and initial
 342 concentration of dye solution. From the results it was concluded that maximum adsorption of
 343 CR from aqueous solution occurred at pH 6. Adsorption was increased with increasing
 344 adsorbent dose and decreases with increase in initial concentration of CR. Kinetics and
 345 isotherm studies revealed that AMSD can be effectively employed for the adsorption of CR.
 346 The experimental data are correlated reasonably well by the Langmuir and freundlich
 347 adsorption isotherm the value of $RL < 1$ obtained indicates that applicability of Langmuir
 348 adsorption isotherm. The whole adsorption process was well described by Pseudo first order.
 349 The result of this study indicates that AMSD can be successfully used for the adsorption of
 350 Congo red from aqueous solution.

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