

# Preparation of Activated carbon from Cassia alata seeds for the scavenging of Anionic dye Alizarin Red S

<sup>1</sup>G. Ushanandhini<sup>1</sup>, <sup>1\*</sup>K. Arivalagan, <sup>2</sup>S. Sivanesan  
<sup>1</sup>Ph.D Research Scholar, <sup>1\*</sup>Assistant Professor, <sup>2</sup> Dean and Professor  
<sup>1</sup>PG&Research Department of Chemistry  
Govt. Arts College for Men, Nandanam, University of Madras, India

## Abstract

Activated carbon has been utilized for the adsorption of anionic dye Alizarin Red S (ARS) from the aqueous solution in a batch method to investigate high removal percentage. The influential parameters of variables such as system pH, temperature, ARS dye concentration, adsorbent dosage, and contact time on ARS adsorptive removal percentage was investigated. Isotherm models, Kinetics and thermodynamics were analyzed for fitting the experimental results. The adsorption process of the present study follows a pseudo second-order kinetic model. Langmuir and Freundlich isotherm models investigated. The proposed adsorption process fit with Langmuir isotherm model. Gibbs free energy (negative value), High enthalpy (positive value) and high entropy values shows the removal process is feasible, endothermic and spontaneous in nature.

**Keywords-** Activated carbon; adsorption; Alizarin Red S; isotherm; thermodynamics

## I. Introduction

Production of synthetic dyes by the industries has been resulted in an enormous increase of color in the wastewater. This wastewater contains various toxic and biorefractory organic compounds. These compounds were discharged into the environment [1]. These compounds affect the life of aquatic animals and plant also affects humans. Dyes are carcinogenic in nature [2]. Alizarin Red S is a widely used anthraquinone dye and it is mainly derived from madder plant roots. It is a natural pigment synthesized first. Alizarin Red S is an oxidant reagent so contact with heat and moisture is not advisable [3]. Alizarin Red S is highly soluble in water. It is used as a staining agent in textile industries. It belongs to the category of the most headstrong and long-lasting pollutants. So the present study focussed on efficient and simple technique for their economic and safe removal. Biodegradation and oxidation methods for the removal of dyes provide many toxic carcinogen as by products [4]. Among various techniques for the removal of dyes from wastewater, adsorption is an effective classical technique widely used in the water treatment to remove various dyes. This technique is based on adsorption of dye molecule onto the surface of an activated carbon with high surface area [5]. Many adsorbents have been employed in adsorption process to remove the toxic organic substances. In recent days Activated carbon is mostly used as an adsorbent in industrial process. This adsorbent has homogeneous surface with macropores [6]. The surface characteristics of activated carbon is mainly depends on the porosity, carbonization temperature and activation method. In the present work activated carbon was derived from the seeds of Cassia alata plant. The aim of this proposed study is focused on investigation and examination of adsorption capacity of activated carbon towards the adsorptive removal of ARS from an aqueous solution.

## II. Experimental

### 2.1 Materials and Methods

The stock solution of Alizarin Red S was prepared with the concentration of 1000mg L<sup>-1</sup>. Double distilled water is used to prepare this stock solution. Working solutions was prepared by diluting the stock

solution. pH of the working solution was measured with digital pH meter. UV-Visible spectrophotometer (systronics 2203, Ahmedabad, India) was used to carry out the adsorption studies. The suspensions of adsorbent and adsorbate were mixed using an orbital shaker at 220 rpm. The chemical substances including Hydrochloric acid and Sodium Hydroxide, Acetone, Alizarin Red S were purchased from Sigma Aldrich (Chennai) and used as such.

Cassia alata plant seeds were collected from our campus. The thinly powdered seeds were put into a muffle furnace at 450°C for 5 hours. The carbonized sample was impregnated in 1:1 H<sub>2</sub>SO<sub>4</sub> solution for 12hrs. The suspension was filtered and customized samples were washed several times with distilled water until the pH of the washings turn to neutral. Finally, the modified activated carbon was dried in an electrical oven at 105°C for 12hrs. The modified activated carbon was crushed to have a uniform particle size and utilized for the adsorption experiments and stored in a plastic container for further use.

## 2.2 Dye uptake capacity measurement

Batch mode experiments were conducted to examine the removal of ARS by prepared activated carbon. About 60 mL of a known concentration of dye solution and 0.2g of the adsorbent was taken in a 250 ml glass-stoppered flask at 25°C and the mixture was stirred on a mechanical shaker at 220 rpm for 60 min. The samples were withdrawn during stirring at preset time intervals, and the adsorbent was separated from the solution by centrifugation (Research Centrifuge, Remi Scientific Works, Mumbai) at 3000 min<sup>-1</sup> for 5 min. The absorbance of the supernatant solution was estimated to determine the residual dye concentration, and was measured before and after treatment. The dye removal percentage can be calculated as follows,

$$(\%) \text{ of dye removal} = \frac{(C_0 - C_e)}{C_0} \times 100 \quad \dots (1)$$

where C<sub>0</sub> and C<sub>e</sub> (mgL<sup>-1</sup>) are the initial and equilibrium concentrations of the dye in solution [7]. Isothermal studies were conducted with different amount of adsorbate (60mg/l to 90mg/l). All experiments were carried out twice, and the concentrations given are average values. The effect of pH was observed by studying the adsorption of dye over the pH range from 3 to 9. The pH of the dye solution was adjusted by using NaOH or HCl solution and a pH meter. The sorption studies were carried out at different temperatures (25°, 35° and 45°C). This is used to determine the effect of temperature. The amount of adsorption at time t, q<sub>t</sub> (mg/g), was calculated using the following formula:

$$q_t = \frac{(C_0 - C_t)V}{W} \quad \dots (2)$$

Where C<sub>t</sub> (mgL<sup>-1</sup>) is the liquid phase concentrations of dye at any time, C<sub>0</sub> (mgL<sup>-1</sup>) is the initial concentration of the dye in solution. V is the volume of the solution (L) and W is the mass of dry adsorbent (g). The amount of equilibrium adsorption, q<sub>e</sub> (mg/g), was calculated using the formula,

$$q_e = \frac{(C_0 - C_e)V}{W} \quad \dots (3)$$

Where, C<sub>0</sub> and C<sub>e</sub> (mgL<sup>-1</sup>) are the liquid-phase concentrations of dye initially and at equilibrium.

## III. RESULTS AND DISCUSSION

### 3.1 FTIR

In the FTIR spectrum of ARS supported Activated carbon derived from cassia alata seeds is shown in fig.1. The intense sharp peak 3741 cm<sup>-1</sup> is largely due to -OH stretching vibration of -OH group. So the activated carbon still carries surface residual -OH groups. It is also supported by its C-O vibration at 1255 cm<sup>-1</sup>. Presence of C=C also evident by the peak at 1524 cm<sup>-1</sup>. Presence C=O groups is evident by their stretching vibration at 1699 cm<sup>-1</sup>. The aromatic ring skeletal vibration of ARS occurred around 1600 cm<sup>-1</sup>. The sharp peaks at 1524 cm<sup>-1</sup> is due to -CH<sub>2</sub> bending or vibration mode. The peaks at 1150 cm<sup>-1</sup> and is assigned to -SO<sub>3</sub> vibration of ARS [8].

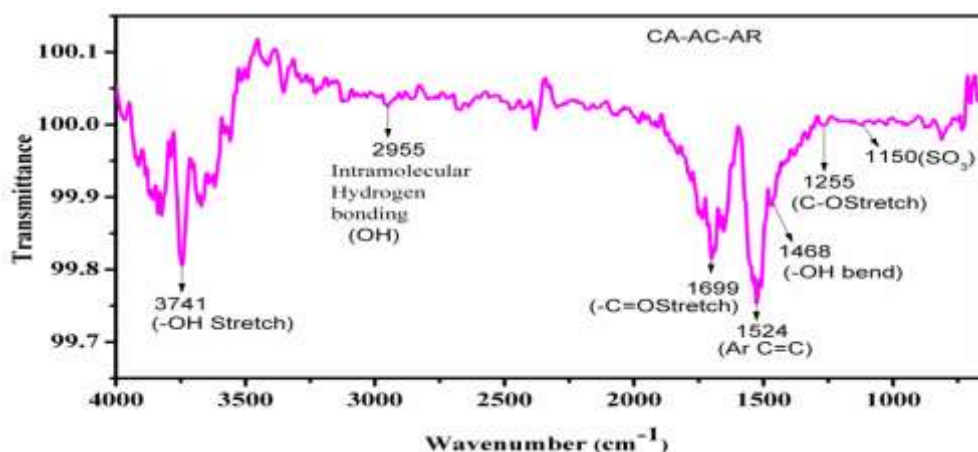


Fig. 1 FTIR spectra of ARS supported activated carbon

### 3.2 SEM

SEM image of prepared activated carbon (fig.2) showed honey comb or flower like structure with macropores on its surface. The well developed pores at the surface of the prepared adsorbents showed the possibility for anionic dyes to be adsorbed onto the surface of the pores.

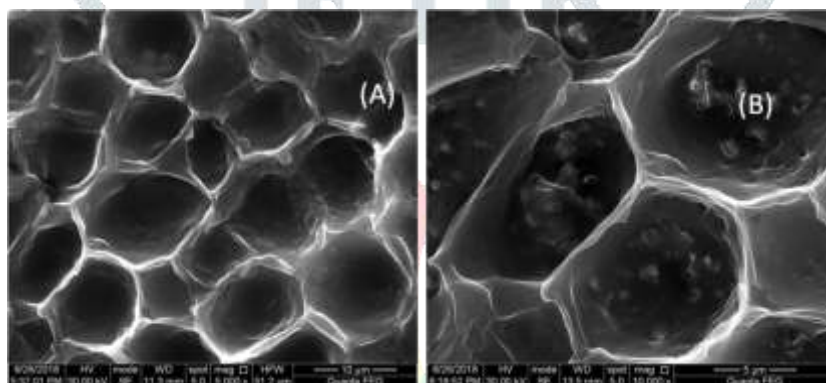


Fig.2. Scanning electron microscopy image of (a) activated carbon, (b) ARS supported activated carbon.

### 3.3 Effect of system pH

It has been observed that pH is one of the key parameter which plays an important role that not only affects the degree of ionization of dyes [9] also affects surface charge of an adsorbent, structure of the entire dye molecule, dissociation of functional groups on the active sites of the adsorbent and the extent of rate of adsorption [10]. So the adsorption of dye mainly depends on the pH value of the dye solution. In other words adsorption capacity was controlled by system pH. Considering the initial and final pH values of the solution, the initial pH has more influence than the final pH value. Hence the pH dependent adsorptive removal of ARS dye using adsorbents such as CA-AC, was studied by varying the pH from 2-10 with 60mg of initial concentration of dye solution at room temperature. The plot obtained from percentage of dye removal Vs pH is shown in fig.3. From the observation maximum removal of AR by CA-AC was 93% at pH 6. When the pH of the system has been increased, concentration of hydroxyl ions will also be increased and thus a negatively charged AC surface is not favourable for the adsorption of ARS due to its strong electrostatic repulsion. In alkaline medium, there is a competition between excess hydroxyl ions with the chosen anionic dye molecule at the adsorption sites. On the other hand, with raise in pH protonation decreases, which result into retardation of adsorption and diffusion thereby [11, 12].

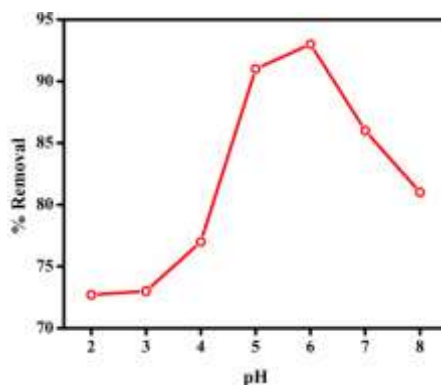


Fig.3 Effects of pH for the removal of ARS by CA-AC at RT

### 3.4 Effect of adsorbent dose

The adsorbent dose is also one of the important parameter. Adsorbent dosage plays significant role in the process of adsorption. Because adsorption is a surface phenomenon thus alters the adsorption capacity at known concentration of dye solution [13]. Fig.4 shows the effect of adsorption of ARS dye as a role of an increase in the dosage of CA-AC. Adsorption studies were conducted by changing the amount of the adsorbent dosage ranging from 0.1g to 1g with a fixed concentration of dyes 60mg/L.

When the adsorbent dosage increased from 0.2g to 0.3g the percentage of removal of dye increased from 87% to 94.1%. Further increase of adsorbent dose (0.2g to 0.6g) the percentage of removal not varied much. Therefore 0.2g was optimum dosage chosen for all further studies.

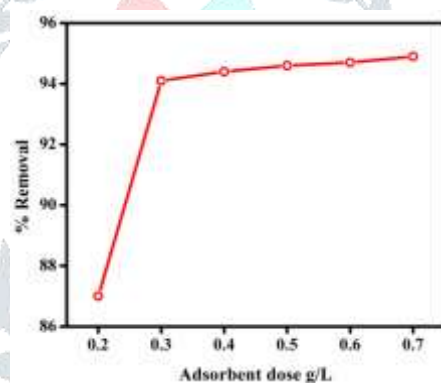


Fig. 4 Effects of Adsorbent dose for the removal of ARS by CA-AC at RT

### 3.5 Effect of concentration of dye

Adsorption kinetics is also dependent on initial dye concentration. It is also a one of the main factor that affects the adsorption kinetics [14]. The effect of initial concentration of dyes during adsorption is shown in fig.5. The initial concentrations of ARS dye varied from 60, 70, 80 and 90 mg. The effect of removal of ARS dyes using CA-AC studied at room temperature and contact time 60min. The percentage removal of ARS decreased with increase in the concentration of dye. By increasing the concentration of ARS from 60 to 90mg the percentage of removal decreased from 94.5 to 85.6%. Hence at low concentration of adsorbate, numerous active sites are available on the prepared adsorbents. So at low concentrations, the dye molecules are easily adsorbed on the surface of adsorbents. But, at high concentrations of adsorbate there is a demand of active sites on the surface of adsorbents, thus percentage of removal is fewer [15].



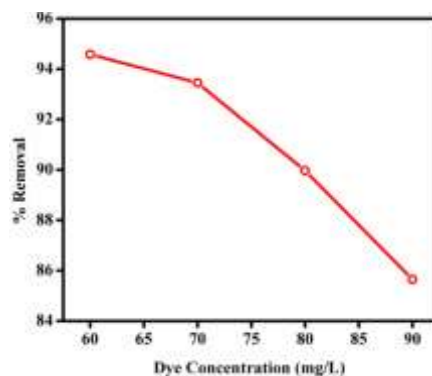


Fig.5 Effects of initial concentration of dye for the removal of ARS by CA-AC at RT

### 3.6 Effect of Contact time

Among various factors, contact time is also an effective factor on adsorption efficiency. At room temperature the percentage removal of dyes was investigated by varying the agitation time. The effect of adsorption of AR was investigated, by changing the contact time intervals as 20 to 60 minutes with the increment of 10 minutes and they were shown in fig.6. At 20 minutes of contact time CA-AC removed 92.8% of AR and the removal percentage increased about 97.5% with the 60minutes of contact time. Equilibrium achieved at 40 minutes of contact time and 96.9% of AR dye removed at equilibrium. The percentage removal capacity of the adsorbent gradually exhausted with increase in time after equilibrium. It is due to remaining vacant pores or active sites became difficult to be occupied by the dye particles. This is because of repulsion between the solute molecules on bulk solution and the solid.

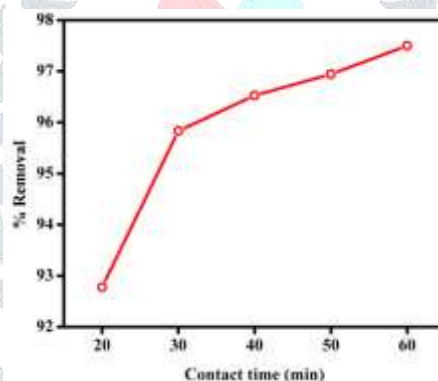


Fig.6 Effects of time for the removal of ARS by CA-AC at RT

### 3.7 Effect of Temperature

The temperature of the system is clearly an important factor on the adsorption rate. The effect of temperature is the key factor to evaluate the thermodynamic parameter. The efficiency of CA-AC onto ARS was analyzed in the temperature range 25°C to 45°C with the increment of 10°C. The results were shown in fig. 7. From the figure it is understood that the removal of dyes increases with increase in temperature.

In the case of AR percentage of removal increased from 86.33% to 93.1%, with the temperature range 25°C to 45°C. It was found that increase of temperature in the removal of ARS was slightly affected.

It may be concluded that at optimum temperature the kinetic energy of the adsorbent molecules has been sufficiently increased and thus it adsorbs easily onto the surface of adsorbent resulting maximum removal percentage. Above optimum temperature it weakens the physical force of attraction between the adsorbent surface and dye molecules. At high temperature the dye molecules moves with high speed and reduces the interaction time of adsorbent and adsorbate. Thus the percentage of removal decreases. Further this increase of temperature influences the desorption process.

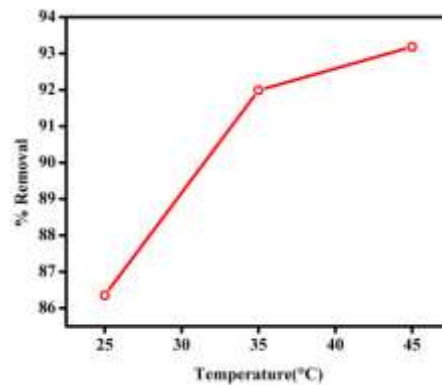


Fig.7 Effects of temperature for the removal of ARS by CA-AC

### 3.8 Isotherm, Kinetic and Thermodynamic Modelling studies for removal of anionic dyes

#### 3.8.1 Isotherm Studies

Isotherm studies [16, 17] are most important to predict how the activated carbon will adsorb the dye molecules. It provides the information about the adsorption capacity of the Activated carbon. Langmuir adsorption isotherm model implies that the adsorption occurs onto identical homogenous surface within the adsorbent. Langmuir adsorption isotherm is based on physical hypothesis. Langmuir adsorption isotherm can be estimated by plotting a graph of  $1/q_e$  vs  $1/C_e$ . The values of  $q_m$  and  $K_L$  can be determined from the linear plot of  $1/q_e$  vs  $1/C_e$ . It can be expressed as the following equation [18, 19]

$$\frac{1}{q_e} = \frac{1}{q_m K_L C_e} + \frac{1}{q_m} \quad \dots (4)$$

In the above equation  $q_e$  represents the amount of dye adsorbed (mg/g),  $C_e$  is the equilibrium concentration of the adsorbate ( $\text{mgL}^{-1}$ ), and  $q_m$  and  $b$  are Langmuir constants related to the maximum adsorption capacity (mg/g) and energy of adsorption (L/mg). A dimensionless separation factor ( $R_L$ ), defined by Weber and Chakravorti, can be used to predict the affinity between the adsorbate and adsorbent.  $R_L$  is calculated by the following equation [20, 21].

$$R_L = 1 / (1 + K_L C_0) \quad \dots (5)$$

The  $R_L$  values indicates the type of adsorption as either unfavourable ( $R_L > 1$ ), linear ( $R_L = 1$ ), favourable ( $0 < R_L < 1$ ), or irreversible ( $R_L = 0$ ). The Langmuir isotherm parameters for the adsorption of AR by CA-AC shown in fig.8. The correlation coefficient ( $R^2$ ) values were found from the plots is 0.976. The significant characteristics of a Langmuir isotherm model known as equilibrium factor ( $R_L$ ), also known as dimensionless separation factor can also be calculated. The  $R_L$  value for the adsorption of ARS by CA-AC is 0.0191 respectively.

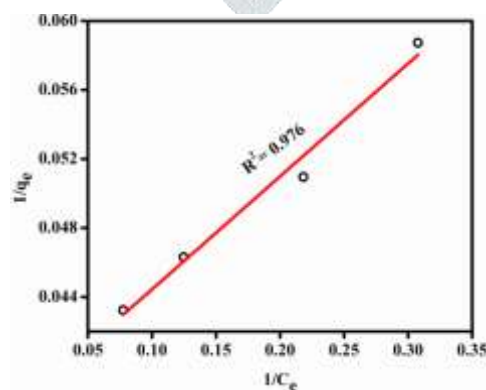


Fig. 8 Langmuir plot for the removal of ARS by CA-AC

According to the calculated dimensionless separation factor ( $R_L$ ), it is determined that the adsorptive removal of ARS dye by CA-AC was favourable under the conditions maintained in this study.

Freundlich isotherm is based on empirical equation. The logarithmic form of Freundlich isotherms equation is,

$$\log q_e = \log K_F + \frac{1}{n \log C_e} \quad \dots (6)$$

Where  $q_e$  is the amount of dye adsorbed per unit mass of the adsorbent (mg/g),  $C_e$  is the equilibrium liquid-phase concentration of the dye (mg/L), the  $K_F$  represent quantity of dye adsorbed in mg/g for unit concentration of dye and  $1/n$  is a measure of adsorption density. Values of  $n > 1$  represent the favourable adsorption condition. The linear plot of  $\log q_e$  vs.  $\log C_e$  is shown in fig.9, obtained straight line with slope of  $1/n$  and intercept of  $\log K_f$ . The  $K_f$  and  $n$  values were calculated from the intercept and slope of the plot.

The limitation of Freundlich isotherm is the quantity of dye molecules adsorbed increases indefinitely with concentration of dyes in the solution. But this isotherm is not used to predict the saturation of adsorbent during adsorption process. So it assumes that multilayer adsorption occurs at the surface of adsorbent. Thus infinite surface coverage predicted by Freundlich model using the mathematical equation. However, the calculated correlation coefficient values from Freundlich plots for the adsorption of ARS by CA-AC are 0.944 respectively.

By comparing the correlation coefficient values of Langmuir and Freundlich models the adsorption of CA-AC better fitted with the Langmuir model.

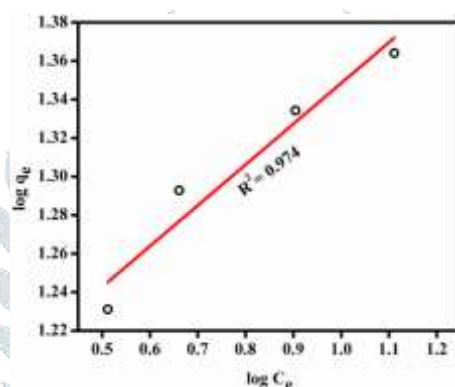


Fig.9 Freundlich plots for the removal of ARS by CA-AC

Table.1. Isotherm parameters

Langmuir parameters				Freundlich parameters		
$q_m$ (mg/g)	$K_L$ (L/mg)	$R_L$	$R^2$	$n$	$K_f$	$R^2$
27.02	0.5693	0.0191	0.976	4.7393	13.677	0.944

### 3.9 ADSORPTION KINETICS MODELLING STUDIES

The calculated adsorption kinetic results of the present study follows Lagergren's kinetic equations including pseudo-first order [22] and pseudo second order models [23]. The kinetic study values are calculated on the basis of influence of contact time on the removal of anionic dyes chosen. The pseudo first-order rate expression is given as,

$$\ln q_e - q_t = \ln q_e - k_1 t \quad \dots (7)$$

Where,  $q_e$  and  $q_t$  are the amounts of dye adsorbed on the adsorbent (mg/g) at equilibrium and at contact time  $t$ .  $k_1$  is the rate constant of first order adsorption ( $\text{min}^{-1}$ ). The slope and intercepts of plots of  $\log (q_e - q_t)$  vs  $t$  were used to determine the first order constant  $k_1$ . The pseudo second-order kinetic model is expressed as,

$$t/q_t = 1/k_2 q_e^2 + t/q_e \quad \dots (8)$$

Where  $k_2$  is the rate constant of the pseudo second order adsorption (g/mg/min). The plot of  $t/q_t$  vs  $t$  gives a linear relationship, from which  $q_e$  and  $k_2$  can be determined from the slope and intercept of the plot. Pseudo first order and second parameter values are listed in Table.2 and shown in fig 10 and 11.

The adsorption process of ARS by CA-AC provides the correlation coefficient value 0.971 for pseudo-first order kinetic model and 0.999 for Pseudo-second order kinetic model. The pseudo-first order kinetic model does not fit well at any contact time.

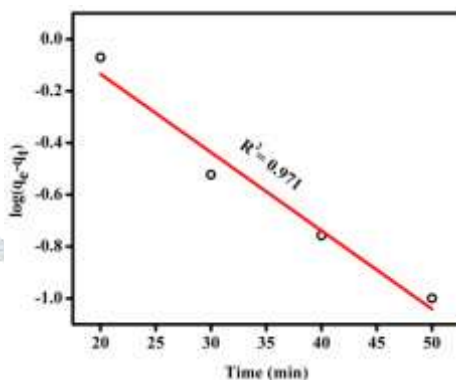


Fig.10 Pseudo first order plots for the removal of ARS by CA-AC

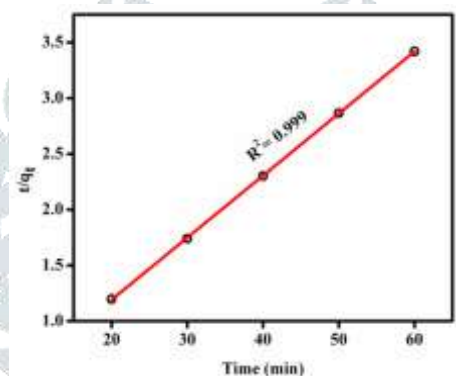


Fig.11 Pseudo second order plots for the removal of ARS by CA-AC

Table.2 Pseudo first and second order kinetic parameters

Pseudo first order				Pseudo second order		
$q_e(\text{exp})$	$K(\text{ad})$	$q_e(\text{cal})$	$R^2$	$K_2$	$q_e(\text{cal})$	$R^2$
17.55	0.0690	2.95	0.971	0.039	18.18	0.999

### 3.10 THERMODYNAMIC MODELING STUDIES

The adsorption thermodynamic parameters [24], gives the information of the energy changes associated with adsorption. These parameters are significant in adsorption process for understanding the effect of temperature. Different thermodynamic parameters such as change in standard free energy ( $\Delta G^0$ ), enthalpy change ( $\Delta H^0$ ), and entropy change ( $\Delta S^0$ ), were determined by using the following equations,

$$K_d = \frac{q_e}{C_e} \quad \dots (9)$$

$$\Delta G = -RT \ln K_d \quad \dots (10)$$

$$\ln K_d = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \quad \dots (11)$$



Where,  $K_d$  is the distribution coefficient,  $T$  is the temperature and  $R$  is the gas constant ( $8.3145\text{J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$ ) respectively.  $\Delta S^0$  and  $\Delta H^0$  were calculated from the slope and intercept of Van't Hoff plots of  $\ln K_d$  vs  $1/T$  shown in fig.12 and the values are listed in table.3 respectively.

The different adsorption thermodynamic parameters values are useful to recognize the mechanism (either physical or chemical) involved in adsorption process. From the observations the negative free energy change ( $\Delta G^0$ ) implies that adsorption is feasible and spontaneous and thus minimum activation energy needed at the given temperature. It is observed that when  $\Delta G^0$  values decreases with increase in temperature and thus the adsorption process has been inversely proportional to the system temperature. It is observed that higher temperature more favourable for the adsorption process. This confirms that the process was highly spontaneous with the low  $\Delta G^0$  values. Meanwhile, when the negative  $\Delta G^0$  values are very close to each other, indicates that spontaneity of adsorption process is independent of the system temperature. When the negative  $\Delta G^0$  values are not close to each other, we can conclude that the spontaneity of the adsorption process is dependent on temperature. In the adsorption process the temperature plays two major effects. As the temperature increases the rate of diffusion of the dye molecules between the boundary layer and in the internal pores of activated carbon increases, leads to minimum viscosity of the aqueous dye solution. In addition to this it also alters the equilibrium adsorption capacity of the selective adsorbent and adsorbate.

In the present study the positive value of  $\Delta H^0$  shows that the adsorption process of chosen anionic dye with AC is endothermic. It is already reported that the positive values of  $\Delta H^0$  is due to more interaction between preadsorbed water molecules and the AC than the interactions between the dyes and AC. As a result, the adsorption process of dyes onto AC is not only influenced by the interaction of adsorbent and adsorbate, but also influenced by the solvent-adsorbate and solvent-adsorbent interactions. Many researchers reported that the  $\Delta H^0$  values varying from 2.1- 20.9 kJ/mol then the adsorption is physical adsorption.

The entropy value determines whether the adsorption process occurs faster or slower compare to any individual reaction. The positive  $\Delta S^0$  values confirms that there is a good interaction of chosen anionic dye with AC and thus increase in the degree of randomness or disorderness at solid-liquid interface and implies the favourable conditions for the adsorption process. Negative  $\Delta S^0$  implies that randomness decreases at solid liquid interphase during adsorption process.

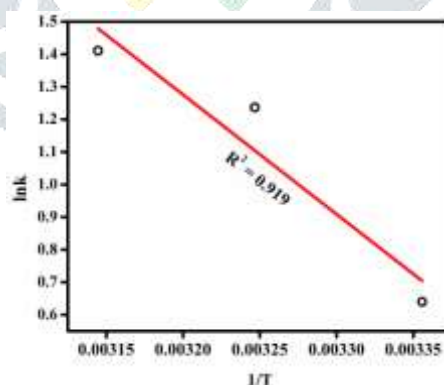


Fig. 12 Thermodynamic plots for the removal of ARS by CA-AC

Table.3 Thermodynamic values of CA-AC and GM-AC from Van't hoff plot

Adsorbate	Temperature (K)	$\Delta G^0$ (KJ/mol)	$\Delta H^0$ (KJ/mol)	$\Delta S^0$ (J/mol/k)
CA-AC-AR	298	-1.5836	70.27	54.57
	308	-3.0014		
	318	-3.7310		

#### IV. CONCLUSION

In this research the cheap adsorbent such as Cassia alata seeds based activated carbon was prepared. Sulphuric acid was used as an activating agent. The modified activated carbon using sulphuric acid was utilized for the investigation of removal of anionic dye namely Alizarin Red S from aqueous media.

FTIR showed the presence of various functional groups on the surface of activated carbon after adsorption of chosen anionic dye. SEM images showed well defined pores. These pores conclude the maximum capacity of prepared adsorbents on the adsorption of various pollutants over the surface of it. The percentage of colour removed increase with increasing adsorbent dosage, increase with increasing contact time and varied with dye solution pH. Optimum contact time for equilibrium to be achieved is found to be 40-60 min. The adsorption rates increases with increasing temperatures. The adsorption process of better fits to Pseudo second-Order Kinetic model. The equilibrium data of the present investigation fits well to the Langmuir Isotherm model. Results indicated monolayer adsorption. Thermodynamic parameters reveal that physical adsorption along with some electrostatic adsorption occurs between the adsorbent and adsorbate. The physical adsorption process is spontaneous and endothermic. This was concluded from sign of Gibbs free energy and enthalpy. Findings of the present study CA-AC can be represented as an effective adsorbent for the removal ARS dyes from an aqueous solution.

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