Preparation Of Cationic Resin Using Cashew Nut Sludge For Highly Efficient Removal Of Acid Red 18 Dye

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Abstract- *The extraction of cationic resin from agro-Industrial waste (cashew nut sludge) by solvent extraction method and it was appliedfor adsorption of acid red 18 dye. Various parameters are influenced for optimizing the dye removal efficiency such as initial solution pH, initial dye molecule concentration, resin dosage, contact time, and temperature. The maximum dye removal was found of pH 3, initial dye concentration 20 mg/l, resin dose 0.12 g/100 ml, contact time in 45 min, and temperature at 328 K of 85.45 %, 98.18 %, 98.24 %, 99.18%, 99.54 % respectively.The adsorption process was well fitted with pseudo second order kinetic modeland Langmuir isotherm model. Thermodynamics parameters were found the negative values of Gibb's energy ΔG, ΔH (45.27 kJ/mol) and ΔS (19.27 J/mol K) expressed process is spontaneous and endothermic nature.*

Keywords- Acid red 18, Cashew nut sludge, cationic resin, Isotherms, Kinetics.

I. INTRODUCTION

Industrial azo dyes are mainly affect the environment, plant, and human health in terms of allergenic, carcinogenic, mutagenic, and teratogenic which discharged from various industries such as cosmetic, textile, pharmaceutical, paper, leather, pulp, tanning, and food industries [1]. Approximately more than 10000 dyes are available commercially,and about1 million tons are generating annually [2].60-70 percent of azo dyes contributing worldwide which attributed highly carcinogenic and mutagenic [3].Therefore, the treatment of azo dye (acid red 18) is important to prevent from deleterious effects on humanbeings and environment.

Various techniques are available for effluent treatment such as biological process, ion exchange, membrane filtration, coagulation and chemical precipitation, and adsorption. Adsorption isa preferabletechnique for treatment of effluent due to cost effective, high adsorption capacity, reusability, easily recoverable[4-6].Nowadays, researchers focusing wastewater treatment with various adsorbents, are activated carbon, zeolite, polystyrene, and resins for the removal of dye from aqueous solution [7-8].Conventional adsorbents are unable to achieve adsorption quickly. Recently, theresinhas been satisfyingthat the alternativefor the removal of dyes[8].

Agricultural industriesare generating huge amounts ofwaste all the time. Mostly, these wastes are being utilized as animal feed ordeposition on the dry land. Agricultural waste mainly containsrich in sugars, minerals, and proteins. The cashew nut cultivation is increasingevery year correspondingly cashew nut sludgealso increasing, this sludge is adeposit on landfill[9].Cashew nut shell liquid (CNSL) is the by-product of the cashewnut Industry. The CNSL can be extracted from the upper portion of cashew nut,and it isusing in variousapplications. The shall liquid has broad applications in automobile Braking, Paint manufacturing, Varnish, compound and foundry resin [10-12]. Different plants are producingvarious types of resin.

The cashew shall liquid contains alkenyl phenolic compounds which can beseparated into various compounds like cardanol, cardol, and 2-methylcardol [13]. The cardanol is one of the main compounds for the synthesis of cation exchange resin. The cashew nut shell contains 85 % of defeated cashew nut sludge to cardanol [14-15].Various methods are availablefor extraction of CNSL from Cashew nut sludge (CNS), such as Oil bath method, drum roasting method, open pan roasting method, cold extrusion method, kiln method, expeller method, subcritical water extraction, Soxhlet extraction, two-step extraction, supercritical CO_2extraction, and solvent extraction method[16-19]. The solvent extraction method is asimple and profitable technique where extraction may getupto 98% of oil[17, 20-21].

In this present work, mainlyfocus onpreparation and development of cationic resin from CNS by solvent extraction method. The extracted resin was analyzed using various characterization, FTIR, TGA-DTA, FESEM for morphological image and DSC to study the thermal decomposition of extracted resin. Finally, extractedcationic resin was utilizedforhighly efficient removal of Acid Red 18 dye. Also, studied adsorption kinetics, isotherms, and thermodynamics.

II. MATERIALS AND METHODS

2.1.Raw Material and chemicals

All the chemicalswere usedin this study are analytical reagent grade (AR). Propanol, methanol, diethyl ether was obtained from Merck, India. Acid Red 18 dye was obtained from Sigma-Aldrich, India. Fig.2(a)showsthat the Cashew nut sludge (CNS) got from the cashew nut industry at Cuddalore, Tamil Nadu, India. The chemical properties of acid red 18 dye as shown in Table 1.

Table 1: Chemical properties of Acid red 18

Parameters	Value
Chemical name	Acid red 18
Chemical formula	$C_{20} H_{11} N_2 Na_3 O_{10} S_3$
Molecular wt (g.mol^{-1})	604.48
λ_{max} (nm)	506

2.2.Pretreatment of Sludge

The sludge pretreatment was executed to enhance the activity of cashew nut sludge. 20 ml of 0.1 N alkali solution has been accounted for soaking of 5 g of well-furnished raw materials around 12 h. The above solution wasrepeatedly washedwith distilled water untilreach pH 7. Afterwards, filter and drythe sample at 60 ºC for overnight.Fig.2(b)shows the extraction of cationic resin from AIW.

Fig 2 (a): Cashew nut sludge (CNS) Fig 2 (b): Extracted resin from CNS

Fig 2: Raw material and final product

2.3.Extractionof cationic resin

The extraction of cationic resin techniquewas adopted from our previous studies[22] as following procedure. The pretreated sample was stored at 4ºC to ignore the compoundsdegradation. The stored sample was added in a propanol solution at 50ºC for 12 hoursin a soxhlet extractor apparatus. Similarly, the mixture was soaked with n-hexane at 60ºC for 12 hours. Then, 15 g of samplewas soaked in water and n-hexanesolution 1:5(w/w) for stirring at 30ºC for 2 h. The dried samples was ground with mortar and pestle to make afine powder. The fine residue was added to a 50 ml of 70% ethanol solution, which passed through a 60 mesh sieve followed by a 50 ml of 0.1 M NaOH for 5 min. Finally, thesample was filtered and dried using freeze drier. The product was stored and utilized for characterization and adsorption studies. The schematic diagram of cationic resin extraction as shown in Fig.1.

FESEM morphology images obtained from Nova Nano SEM/FEI model with beam landing energy 50 V and resolution is 1.4 nm at 1 kV without beam declaration.

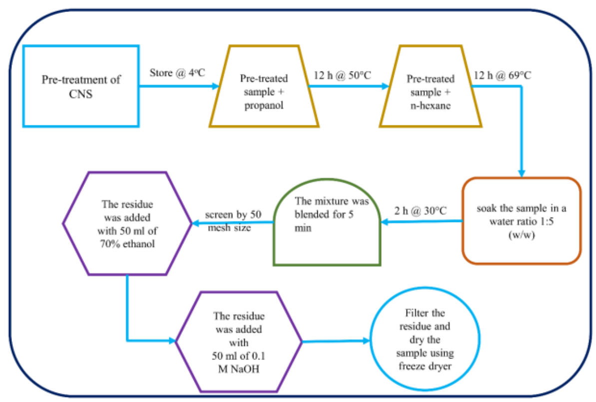

Fig 1: Schematic diagram for extraction of resin from CNS

2.4.Adsorption studies

Before the adsorption study, calibration graphwas drawnby aknown concentration of acid red 18 dye. The adsorption experiment was done using a batch adsorption technique.Various parameters were considered as a significant role in batch adsorption such aspH, initial concentration, resin dose, contact time, and temperature respectively. The experiment was carried out using 100 ml ofadsorbate solutions in 250 ml of beaker,initial pH (3-12) was adjusted either adding a 0.1N HCl or0.1 N NaOH solution, initial dye concentration (20-100 mg/l), resin dose (0.02-0.2 g/100 ml), contact time (15-180 min), and temperature (298-328 K). Rotating speed of orbital shaker was maintained 120 rpm throughout the experiments. After the experiments, the solution was centrifugedat 5000 rpm for 10 min. Finally, the supernatant was taken to measure theconcentration of dye using absorbance at 506 nm.

The removal percentage (% R) dye concentration and the adsorption capacity at equilibriumq_e (mg/g) were calculatedusing the following equations,

$$R\% = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (1)$$

$$q_e = \frac{(C_0 - C_e) \times V}{M} \quad (2)$$

$$q_t = \frac{(C_0 - C_t)\, V}{M} \quad (3)$$

Where, C_0 and C_e are the initial and equilibrium concentration of theacid red 18 dye (mg/l), C_t is the equilibriumdye concentration at time t (min),and V is the volume of solution (in ml), M is the mass of adsorbent (in mg/100 ml).

2.5. Adsorption isotherm

The adsorption isotherm is assistance to determine uptake of the dye solution on provided adsorbent dose at equilibrium. The three isotherms were chosen in thisstudy such as Langmuir, Freundlich, and Tempkin isotherm.
The Langmuir isotherm equation[23]

$$\frac{c_e}{q_e} = \frac{1}{K_L q_{max}} + \frac{c_e}{q_{max}} \quad (4)$$

The separation factor (R_L) is important characteristics of Langmuir isotherm; it can be written as,

$$R_L = \frac{1}{(1 + K_L C_0)} \quad (5)$$

The Freundlich isothermequation[24]

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

The Tempkin isothermequation[25]

$$q_e = \beta \ln K_T + \beta\, C_e \quad (7)$$

Where,

$$\beta = \frac{RT}{b} \quad (8)$$

$K_L$and q_{max} are Langmuir isotherm constant (L/mg) and the maximum adsorption capacity of the adsorbent; R_L presents the type of isotherm;linear (R_L=1), irreversible (R_L=0), favorable (0< R_L<1), and unfavorable (R_L >1). q_e is the dye concentration at equilibrium onto the adsorbent (mg/g); C_0 (mg/l) is the highest concentration of dyesolution. C_e is the dye concentration at equilibrium insolution (mg/l).$K_F$is the Freundlich isotherm constants (L/mg), and n is the adsorption intensity. K_T (dimensionless) and b are the Temkin isotherm constant. R is the universal gas constant 8.314 (J/mol K).

2.6. Adsorption kinetic

Adsorption kinetic is the critical study to determine uptake rate of the solute concentration. It controls the residence time of adsorbent uptake at the solid solution interface. Lagergren's pseudo first order kinetic equation and Ho's pseudosecond order kinetic equation were analyzedto study the time dependence of adsorption process[26].
Pseudo-first-order kinetic equation

$$\log(q_e - q_t) = \log q_e - k_1 t \quad (9)$$

Pseudo-second-order kinetic equation

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (10)$$

Where, q_t is the amount of dye uptake (mg/g) at equilibrium time t (min), $k_1$and k_2 are the pseudo-first-order rate constant (min^{-1}), and the pseudo-second-order rate constant (g/mg min).q_t cal refers to the calculated values.

2.7. Adsorption thermodynamics

The thermodynamic study was employed to know the adsorbent affinity of the adsorbate. Acid red 18 dye adsorption onto resin was calculated using the below equations[6],

$$\Delta G = \Delta H - T\Delta S \quad (11)$$

$$\ln K_C = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (12)$$

Where, K_c = q_e/C_e and q_e are the dye concentration atequilibrium onto the resin dose (mg/L), R is the universal gas constant (8.314 J/mol K), and C_e is the concentration of acid red 18 dye at equilibrium (mg/L).

III. RESULTS AND DISCUSSION

3.1.Field Emission-Scanning Electron Microscope (FESEM)

The FESEM of morphology images as shown in Fig.3 for extracted resin from CNS at different magnification. Our previous research has been reportedin detail about temperature profile, mass loss, functional group[22] of extracted resin.Fig 4 shows that presents of amorphous phase with clear pores which helpful to adsorb dye molecule within that pore size.

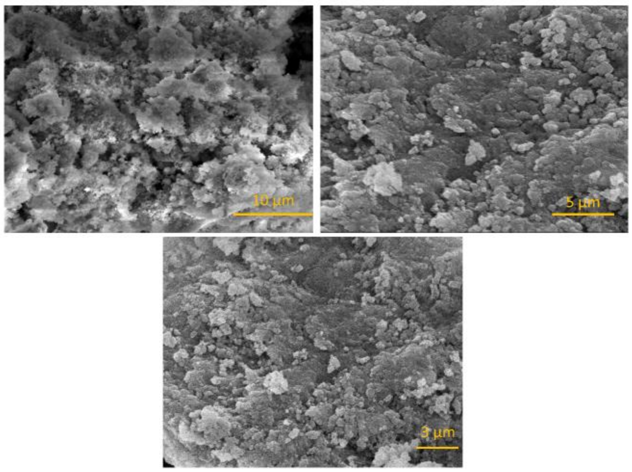

Fig 3: FESEM images of extracted resin from CNS at different magnification

3.2.Adsorption studies

To study the effect of pH is the most significant parameter for control thedye adsorption to un-adsorbed dye molecules. The effect of pH result is shown in Fig.4 (a). The effect of pH was studied at 298 K, 20 mg/L acid red 18 initial dye concentration, contact time 60 min and resin dose 0.1 g/100 ml. The pH was varied from 3-12for investigatingthe uptake adsorbate at equilibrium. The results show that the removal percentage reached up to 85.45% at pH 3.After pH 3 the removal percentage was started to decrease. Hence, the optimized pH for maximum removal is 3.

To investigate the effect of resin dose on dye removal also acrucial parameter for adsorption study. The initial dye concentration 20 mg/l, temperature 298 K, pH 3, contact time 60 min werekept at constant. Fig.4(b) shows that the dye removal percentage increased from 85.45% to 98.24% of 0.1–0.12 g/100 ml of resin dissolved in 100 mL acid red 18 dyesolutions. Beyond 0.12 g/100 ml of resin, the removal percentage of dye is remainedsame. Thus, the adsorbent dose was fixed 0.12 g/100 ml for the subsequent experiments.

The effect of contact time is directly related to the adsorption studies. The contact time and concentration were employed in the range of 15-180 min and 20-100 mg/l respectively. The initial dye concentration 20 mg/L, temperature 298 K, pH 3, and resin dose 0.12 g/100ml were kept constant.Fig.4(c)shows that contact time is corresponding the concentrations results. It is demonstratedthat the removal of acid red 18 dye achieved amaximum of 99.18% on 20 mg/Lat 45 min. The adsorption experiment was further continued upto 180 min, though after 45 min the removal percentage was unchanged. Similarly, 40, 60, 80, 100 mg L^{-1}also reached highest removal percentage at 45 min. Based on obtained results, thesubsequent experiment was followed the contact time was at 45 min.

The effect of initial concentration of acid red 18on dye removal percentage as shown in Fig.4 (d).Contact time 45 min, temperature 298 K, pH 3, and resin dose 0.12 g/100 ml were kept constant.The initial dye concentration was varied from 20 to 100 mg/l. Fig.4 (d) shows that irrespective of the dye concentration where increasing dye concentration with decrease the removal percentage. After 20 mg/l,the removal percentage started slowly decreasing from 98.18% to 93.03%.Therefore, 20 mg/lwas achieved the maximum percentage of dye removal is 98.18%.

The effect of temperature onremoval of acid red 18 as shown in Fig.4(e). The temperature was investigated between298-328 K. Contact time 45 min, pH 3, resin dose 0.12 g/100 ml, and initial dye concentration 20 mg/lwere kept constant. The obtained results show while increasing the temperature dye removal also increasing prominently. This removal attributed to the surface activation and pores enlargement. After 298 K temperature, the removal was slowly increased from 98.18% to 99.54 % at 328 K.

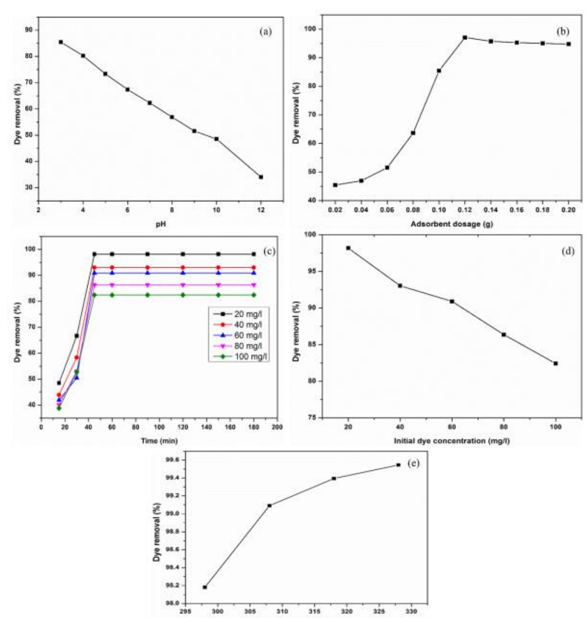

Fig 4: The different parameters and removal percentage of acid red 18 dye (a) the effect

of pH, (b) the effect of resin dose, (c) the effect of contact time, (d) the effect of concentration, (e) the effect of temperature.

3.4. Adsorption isotherm

The Langmuir isotherm provides the interaction of dye molecules and solid surface at aconstant temperature. Fig. 5 (a) shows that the plots of C_e/q_evs. $C_e$present linear correlation coefficients of (R^2) 0.959 at 298 K. The higher value of correlation coefficients indicate that the isotherm is monolayer and uniformly distributed on the solid surface.q_{max} and $K_L$were obtainedfrom the slope and intercept of the straight lines of the plot C_e/q_evs.C_e and the calculated values are given in Table 2. The adsorption capacity of acid red 18 on resin is (q_{max}) 38.91 mg g^{-1}298 K. Also, the separation factor R_L (dimensionless) also calculated from the Langmuir isotherm constant is 0.41 at 298 K as shown in Table 2. This result demonstrates that extracted resin is favorable for the adsorption of acid red 18 dye. Therefore, Langmuir isotherm is preferable for this sorption process.

Table 2: DifferentIsotherm model of acid red 18 by cationic resin.

Langmuir				Freundlich			Temkin		
q_{max} (mg/g)	K_L (L/mg)	R_L	R^2	K_F((mg/g (L/mg)$^{1/n}$)	n	R^2	b	K_T (L/mg)	R^2
38.91	1.322	0.41	0.959	0.417	5.675	0.492	0.373	6.773	0.909

Fig. 5 (b) shows that the Freundlich adsorption isotherm plot of ln$q_e$vs. ln C_e.This isotherm explains about heterogeneous process between thedye concentration and resin surfaces. The K_F(Freundlich constants) and n represent the adsorption capacity, and intensity was determined from the linear plot of ln q_e vs. ln C_e.Table 2 shows that the obtained values of K_F and n. K_F value is 0.417 mg g^{-1} (L/mg)$^{1/n}$and n value is 5.675at 298 K. The value of n>1 is indicated that the process is favorableof acid red 18 dye on theresin surfaces. The correlation coefficient values (R^2=0.692). According to R^2values, the correlation valuewere not fitted with the Freundlich adsorption isotherm.

The Tempkin isotherm explains the interaction between adsorbate and adsorbent,and it provides that the adsorption is linear or non-linear. K_T and b values were determined from the slope and intercept of the plot $q_e$vs. ln $C_e$as shown in Fig. 5 (c). The K_T is the binding constant (L/mg) at equilibrium state and corresponding to the highest binding energy and b is the heat of adsorption. Tempkin constant b and $K_T$were tabulated in Table 2. Based on the R^2 (0.909)value Tempkin isotherm is not fitted.

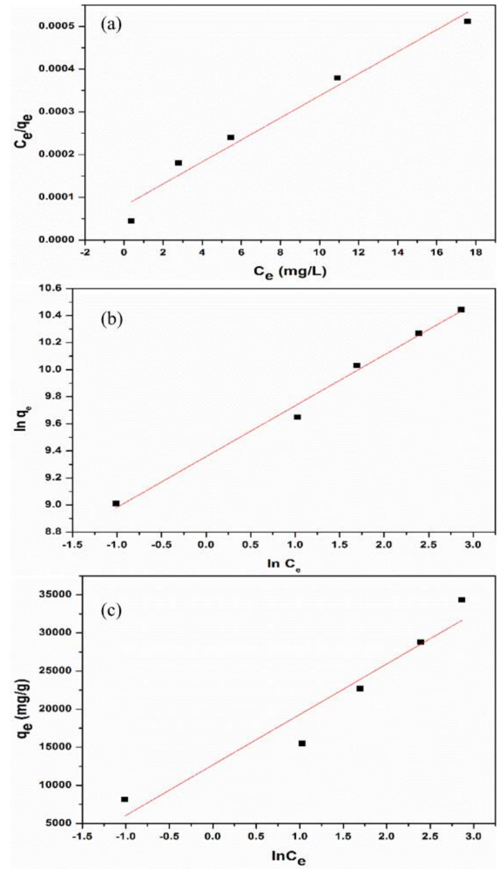

Fig 5: The adsorption isotherms study of acid red 18 dye onto cationic resin. (a) Langmuir isotherm, (b) Freundlich isotherm, (c) Tempkin isotherm.

3.5. Adsorption kinetics

The adsorption kinetic study was performed to predict the rate of adsorption which plays a major role in themodeling of the process. The experimental data of acid red 18 adsorptions on resin were fitted using the pseudo-first-order [27]and pseudo-second-order kinetic models [28]. A plot of ln (q_e-q_t) vs. t is a linear relationship, for the sorption of acid red 18 dye to theextracted resin as shown in Fig. 6 (a). In Table 4shows that the values of k_1 and q_e cal is determined from the slope and intercept of ln (qe-qt) vs. t. The correlation coefficient R^2(0.736) was found from the first-order kinetic model.

Table 3: Different Kinetic studies of acid red 18 onto cationic resin

Model	Parameters	20 mg/l
Pseudo-first-order	k_1 ($\times 10^{-2}$ min^{-1})	1.076
	q_e cal (mg/g)	90.74
	R^2	0.736
Pseudo-second-order	k_2 (g/mg min)	0.001
	q_e cal (mg/g)	142.18
	R^2	0.998

Fig. 6 (b) shows the plot of t/q_t vs t. The k_2 and $q_e$cal were obtained from the slope and intercept in the plot as shown in Table 4. The R^2 value determinedfor this model is0.998. The R^2 value is higher than thepseudo-first-orderkinetic model.Thus the first-order kinetic model did not fit. Therefore,experimental results concluded that the kinetics of acid red 18adsorptionson cationic resinperfectly fit with the pseudo-second-order kinetic model. Also, suggeststhat the chemisorption was controllingthis adsorption process.

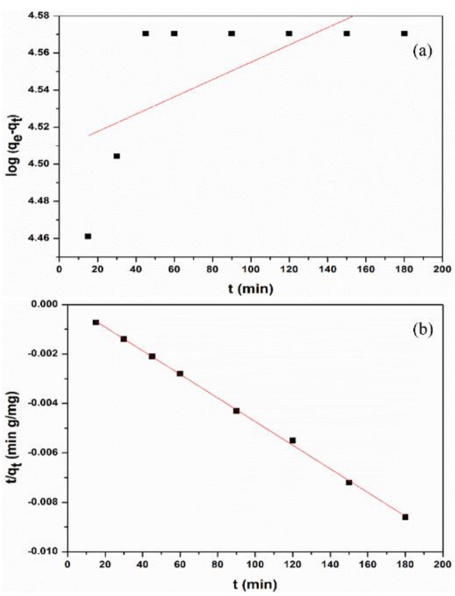

Fig 6: Adsorption kinetic studies on cationic resin. (a) Pseudo-first-order kinetic, (b) Pseudo-second-order kinetic.

3.6. Adsorption thermodynamics\

Fig. 7 shows thatthe plot of ln K_0vs. 1/T. The enthalpy (ΔH) and entropy(ΔS) changes were observed from the slope and intercept of the plot ln K_0vs. 1/T as shown in Table 4. Thepositive value of ΔH (45.27 kJ/mol^1) suggests that the acid red 18 dye is a chemisorption process and endothermic nature. The positive value of ΔS (19.27 J/mol K) represents that the redistribution of energy between dye solution and resin dose.

Table 4: Thermodynamic study of acid red 18 onto cationic resin

Temperature (K)	ΔG^0 ($kJ.mol^{-1}$)
298	-9.88
308	-12.01
318	-13.44
328	-14.69
ΔH^0 (kJ/mol)	45.27
ΔS^0 (J/mol K)	19.27

The negative values of ΔG (Table 4)suggesting that the adsorption of acid red 18 is spontaneous. From the above discussioncirculationof energy among the dye moleculesis increasedwith increasing the adsorptioncapacity due to a positive value of ΔS. Therefore, randomness is increasing continuouslyduring the adsorption process. The adsorption occurres spontaneously at normal and as well high temperatures as ΔH > 0 and ΔS > 0.

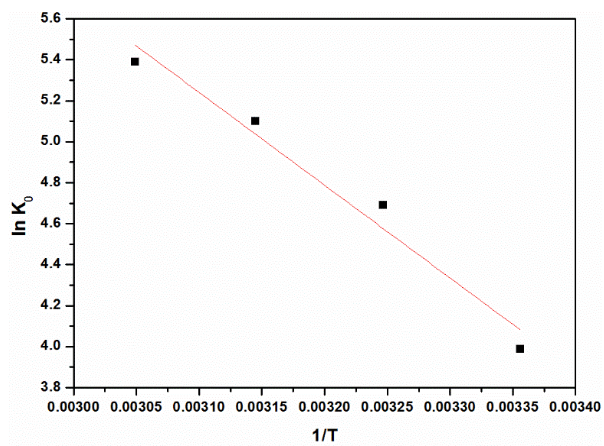

Fig 7: Thermodynamic studies of acid red 18 dye.

IV. CONCLUSION

Cationic resin from cashew nut sludge was extracted successfully by solvent extraction method and characterized using FESEM analysis. The cationic resin was utilized for removal of acid red 18 dye from aqueous solution. Adsorption process was optimized using different parameters and the maximum removal efficiency was found at solution pH of 3, initial dye concentration 20 mg/l, resin dose 0.12 g/100 ml, contact time in 45 min and temperature at 328 K of 85.45 %, 98.18 %, 98.24 %, 99.18%, 99.54 % respectively.The adsorption od acid red 18 well fitted with pseudo second order kinetics model R^2>0.998, and Langmuir isotherm model R^2>0.959. Also studied thermodynamic constant, ΔG secured negative values, ΔH (45.27 kJ/mol) and ΔS (19.27 J/mol K) represents the spontaneous and endothermic nature for acid red 18 dye on the cationic resin.

V. ACKNOWLEDGMENTS

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The authors declare no conflict of interest.

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