Membrane Water Treatment, *Vol. 9, No. 2 (2018) 79-85*
DOI: https://doi.org/10.12989/mwt.2018.9.2.079

Acid green-25 removal from wastewater by anion exchange membrane: Adsorption kinetic and thermodynamic studies

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(Received October 26, 2016, Revised April 16, 2017, Accepted June 3, 2017)

Abstract. In this work, batch adsorption of anionic dye acid green-25 (AG-25) from aqueous solution has been carried out at room temperature using anion exchange membrane (DF-120B) as a noval adsorbent. The effect of various experimental parameters such as contact time, membrane dosage, ionic strength and temperature on the adsorption of dye were investigated. Kinetic models namely pseudo-first-order, pseudo-second-order, Elovich, liquid film diffusion, Bangham and modified freundlich models were employed to evaluate the experimental data. Parameters like adsorption capacities, rate constant and related correlation coefficients for every model are calculated and discussed. It showed that adsorption of AG-25 onto DF-120B followed pseudo-first-order rate expression. Thermodynamic study indicates that adsorption of AG-25 onto DF-120B is an exothermic and spontaneous process.

Keywords: adsorption; Acid Green-25; anion exchange membrane; kinetics; Freundlich kinetic model; thermodynamic; exothermic process

1. Introduction

Wastewater containing large quantity of dyes are produced in several industries namely textiles, paper, tanneries, clothing, printing, paint, etc. (Shen *et al.* 2011). Unluckily, most of dyes wastewater has undesirable color, carcinogen and toxicant. If discharged into the aquatic environment above the level that the nature can eliminate, it would be dangerous to aquatic creatures and humans (Errais *et al*. 2011). In general, dyes can be classified anionic (direct, acid and reactive dyes), cationic (basic dyes) and non-ionic (disperse dyes and vat dyes) (Zheng *et al.* 2015). Among them, azo dyes (anionic) with existence of nitrogen–nitrogen double bonds are considered to be the largest and most versatile class of organic dyes (Gao *et al*. 2013, *Zheng et al*. 2015), but most difficult is to be degraded due to their complicated aromatic structure and poor biodegradability (Feng *et al*. 2010). Therefore, it is essential to introduce convenient method for the wastewater treatment.

Various method such as precipitation, coagulation, membrane filtration, electrochemical, ion exchange, chemical oxidation and adsorption (Ayad and Abu El-Nasr 2012, Zheng *et al*. 2015, Li *et al*. 2016), are used for the removal of dyes from wastewater. Adsorption process has advantages over other techniques used for the removal of dyes from wastewater because of its low generation of residues and the possibility of its adsorbent being recycled and reused (Ayad and Abu El-Nasr 2012). Several effective, selective, and cheaper adsorbent materials were used for dyes removal from wastewaters such as banana pith (Maghraby and Taha 2014), pumice powder (Cifci and Meric 2016), modified zeolite (Alver and Metin 2012), porous composite membrane (Lin *et al*. 2015), coriolus versicolor (Sathain *et al.* 2012), Turkish coal powder (Khataee *et al.* 2013), morus albal leaf powder (Khan *et al.* 2015), organo-bentonite (Koswojo *et al.* 2010), Chitosan/Alumina Composite (Zhang *et al*. 2011) and ion exchange membranes (Khan *et al.* 2016, Khan *et al*. 2015, Labanda *et al*. 2011, Xing *et al*. 2012, Zhang *et al.* 2015).
In recent years, commercially available anion exchange resins have shown excellent adsorption capacity for the removal of reactive dyes (Greluk and Hubicki 2011, Shuang *et al*. 2012, Greluk and Hubicki 2013). The anion exchange resins reported were in the form of particle packed-bed operations have certain disadvantages as slow pore diffusion, low accessible flow rate, high pressure drop and flow channeling. For the removal of above mentioned

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ISSN: 2005-8624 (Print), 2092-7037 (Online)

limitations, AEMs were used instead of resin particles to remove the anionic reactive dyes from water (Chiu *et al.* 2009, Khan *et al.* 2016, Khan *et al*. 2015). Using macroporous membrane system can not only solve the technical problems of packed-bed operation but it also exhibits the ability of scale-up by simple stacking more membranes together or using a large membrane area. Morover, ion exchange membranes can also used in a lot of separation processes (Palaty and Bendova 2010, Tanaka 2010, Koter *et al*. 2011). Thus, IEMs become a good choice of adsorbent for industrial uses.

In our previous work, commercial AEM EPTAC has been reported for removal of anionic dye Congo red (CR) dye from aqueous solution (Khan *et al*. 2015) whereas membranes B1, BIII and DF-120B were used for removal of methyl orange (MO) dye from aqueous solution (Khan *et al.* 2016). In the present study, commercial anion exchange membrane DF-120B was used for the removal of anionic dye acid green-25 (AG-25) from aqueous solution. The effect of several parameters such as contact time, membrane dosage, ionic strength and temperature on the removal of dye from aqueous solution was investigated in batch mode. Various kinetic models such as pseudo-first-order, pseudo-second-order, Elovich, liquid film diffusion, Bagham and modified Freundlich model were applied to the experimental data and the rate of kinetics parameters were interpreted and compared. Thermodynamic parameters like change in Gibb's free energy, enthalpy and entropy for adsorption of AG-25 onto DF-120B were determined at different temperatures.

2. Experimental

2.1 Adsorbent

The commercial anion exchange membrane DF-120B was provided by Tianwei Membrane Com. Ltd, Shandong, China. The ion exchange capacity (IEC) and water uptake (WR) of DF-120B membrane are 0.83 mmol/g and 74.2 % respectively (Khan *et al*. 2016). It was used as adsorbents for removal of anionic dye AG-25 from water. The membrane DF-120B was conditioned with 1 M HCl and NaOH to withdraw impurities from its surfaces before the experiments.

2.2 Adsorbate

The molecular formula of acid green-25 dye is $C_{28}H_{20}N_2Na_2O_8S_2$. Its molecular weight is 622.57 g/mol. The stock solution of dye was prepared by dissolving its appropriate amount in deionized (DI) water. The working solutions were prepared by further diluting the stock solution with DI water to provide appropriate concentration. Chemical structure of AG-25 is shown in Fig. 1.

2.3 Adsorption

Batch adsorption studies of AG-25 dyes were carried out by immersing anion exchange membrane DF-120B into known volume and concentration of AG-25 dye solution at

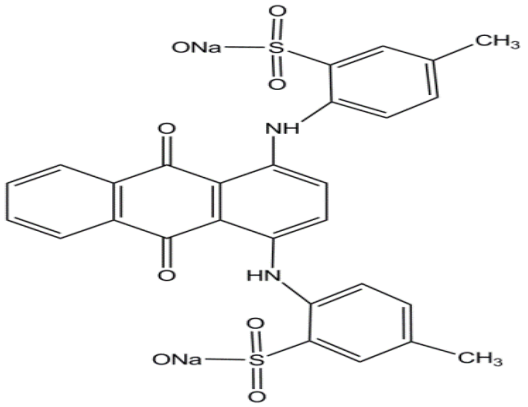

Fig. 1 Chemical structure of Acid Green-25

room temperature. The bottles were shaked at constant speed of 120 rpm and the concentration of AG-25 was measured using UV/VIS spectrophotometer (UV-2550, SHIMADZU) and related calibration curves were obtained at wavelength of 605 nm for AG-25 dye. The amount of AG-25 adsorption onto anion exchang membrane DF-120B at time t, was calculated by below relation (Gong *et al.* 2013).

$$q_t = \frac{C_o - C_t}{W} \times V \quad (1)$$

where Co and Ct are the concentrations of anionic dye AG-25 at initial stage and at time t respectively. Similarly V and W are volume of AG-25 aqueous solution and weight of adsorbent (DF-120B) respectively.

3. Results and discussion

3.1 Effect of contact time

The effect of contact time on the percentage removal of anionic dy AG-25 from aqueous solution by anion exchange membrane (DF-120B) was investigated keeping membrane dosage (0.1 g), concentration of dye (50 mg/L), volume of solution (40 ml) and stirring speed (120 rmp) consant at room temperature and the percentage removal of dye is shown in Fig. 2. It can be seen that the uptake of dye was very fast in the start and then slow down and reached its equilibrium stage after 24 hrs. It is concluded that at initial stage a lot of vacant active sites are present on membrane surface for adsorption of dye which become saturated with the passage of time until equilibrium established and the remaining active sites on the anion exchange membrane surface are hard to be occupied due to repulsive forces between solute molecules on the solid and bulk phase.

3.2 Effect of membrane dosage

The influence of membrane dosage on the percentage removal of AG-25 from aqueous solution was studied keeping the other conditions constant and results are represented in Fig. 3.

It can be seen that the percentage removal of AG-25 is

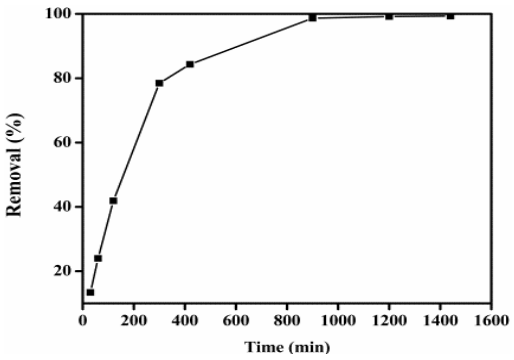

Fig. 2 Effect of contact time on the adsorption of AG-25 onto DF-120B membrane, Temperature=25 ^{o}C, Membrane mass=0.1 g, Area=2×2 cm^2, Initial concentration of dye=50 mg/L

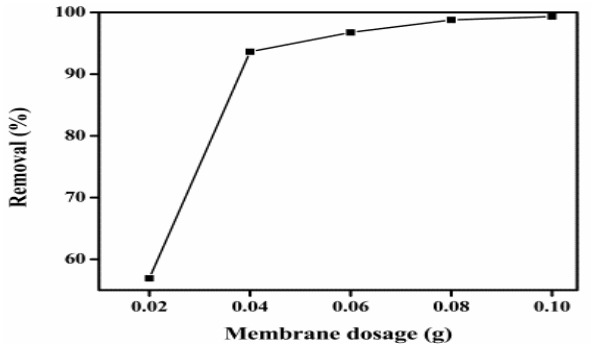

Fig. 3 Effect of membrane dose on the adsorption of AG-25 onto DF-120B membrane, Adsorption time= 24 hour, dye solution volume=40 ml, Conc.=50 mg/L, Temperature=25^{o}C

found to be increased with increasing the membrane dosage. This increase in adsorption of AG-25 dye was due to the fact that the available adsorption sites on the surface of the AEM increases with increasing the membrane dosage. It can be seen that the removal of AG-25 was increased with increasing dosage of membrane and then it becomes almost unchange with further increasing the membrane dosage. The maximum removal of AG-25 was achieved by using 0.1 g of membrane dosage and no significant change beyond this amount was attained. Therefore, 0.1 g of DF-120 was selected as an optimum amount and was used in further experiments to get better results. The observed two stage-dependent adsorption behaviour have also been reported in the literature (Gong *et al.* 2013).

3.3 Effect of ionic strength

The effect of ionic strengh on the removal of AG-25 from aqueous solution was investigated by addition of different amounts of sodium chloride to the dye solution because the ionic strength is an important parameter that control both electrostatic and nonelectrostatic intractions between the dye and the membranes surface. The removal of AG-25 dye is found to be decreased with increasing the concentration of salt as shown in the Fig. 4. This could be due to the competition between the AG-25 anions and Cl-

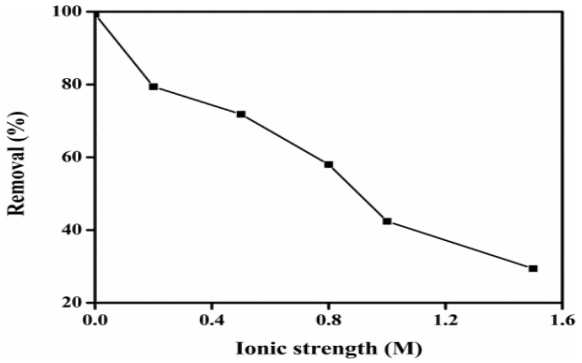

Fig. 4 Effect of ionic strength on the adsorption of AG-25 onto DF-120B membrane, Temperature=25^{o}C

Table 1 Pseudo-first-order, pseudo-second-order and Elovich model rate

Pseudo-first-order				Pseudo-second-order			Elovich model		
$q_{e\,(exp)}$	$q_{e(cal)}$	$k_1\times10^{-3}$	R^2	q_e	$k_2\times10^{-3}$	R^2	α	β	R^2
39.72	44.70	5.5	0.990	50.86	2.12	0.989	2.01	0.10	0.974

Constants (q_e:mg/g k_1:(min^{-1}); k_2:g/mg.min; α: mg/g.min; β: g/mg)

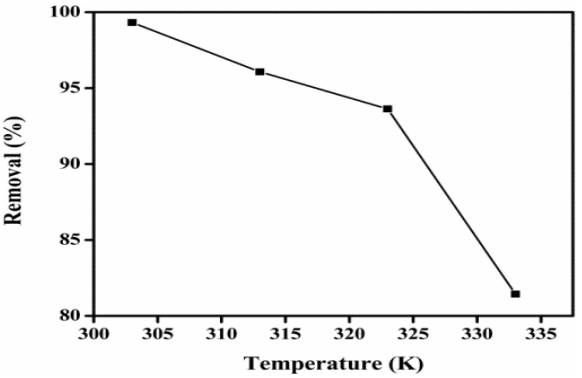

Fig. 5 Effect of temperature on the adsorption of AG-25 onto DF-120B membrane

for the active sorption sites (Wang and Chu 2011). Hence, the adsorption efficiency of the DF-120B is significantly influenced by the ionic strength of the aqueous liquor.

3.4 Effect of temperature

The removal of AG-25 from aqueous solution with temperature was studied keeping all other parameters such as contact time, membranes dosage, stirring speed, solution volume and concentration constant and obtained results are shown in Fig. 5.

It has been observed that the removal of AG-25 is found to be decreased from 98.3% to 81.4% with increasing the temperature from 293K to 333K. The decrease in adsorption of AG-25 with increasing the temperature is associated to the decrease in the surface activity. This depicts that adsorption of AG-25 onto DF-120B is an exothermic process.

3.5 Adsorption kinetics

3.5.1 Pseudo first order

The linearized form of pseudo-second kinetic model is expressed as (Zhang *et al.* 2012)

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (2)$$

where q_e and q_t are amounts of dye adsorbed at equilibrium and time t repectively and k_1 (/min) is the rate constant of pseudo-first-order adsorption model. The plot of log(q_e-q_t) vs time for pseudo-first-order model is given in Fig. 6. The value of K_1 was calculated from slope whereas q_e was calculated from intercept of this plot and given in Table 1. This plot is linear and coefficient of regression is 0.990 showing that pseudo-first-order model was fitted well and more likely to explain the rate process.

3.5.2 Pseudo second order model

The linearized form of pseudo-second kinetic model is expressed as (Zhang *et al.* 2012)

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (3)$$

where k_2 (g/mg. min) is the rate constant of pseudo-second-order model. The graphical representation of pseudo-second-order model is shown in Fig. 7. The value of adsorption capacity (q_e) and rate constant were determined from slope and intercept of linear plot and are given in Table 1. Moreover, the correlation cofficient (R^2=0.989) which is less than pseudo-first order showed that experimental data was not fitted well to the pseudo-second-order model.

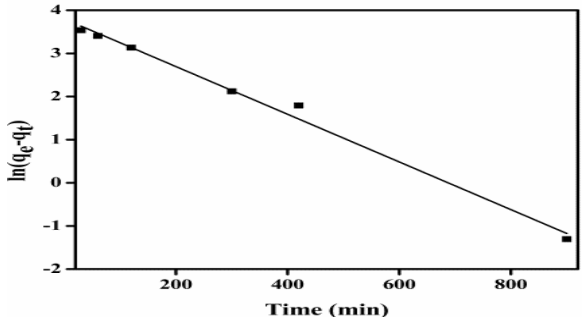

Fig. 6 Pseudo-first-order kinetics for adsorption of AG-25 onto DF-120B membrane

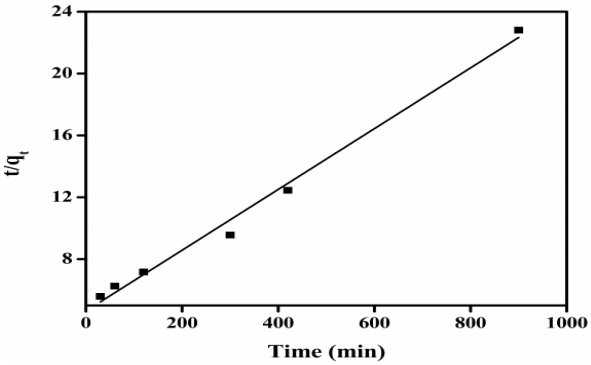

Fig. 7 Pseudo-second-order kinetics for adsorption of AG-25 onto DF-120B membrane

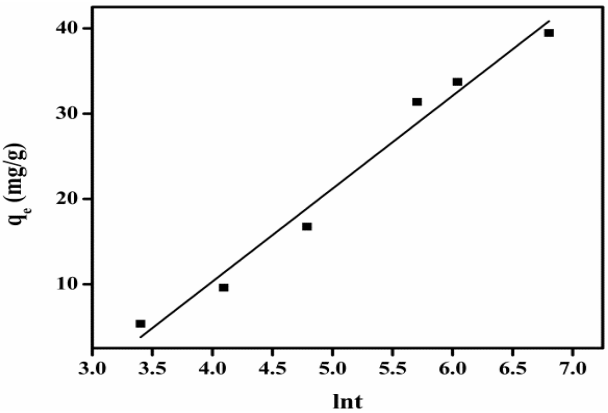

Fig. 8 Elovich model for adsorption of AG-25 on DF-120B membrane

3.5.3 Elovich model

The Elovich kinetic model is interesting model to describe the activated chemisorption for any adsorption system and it can be expressed as (Belaid *et al.* 2013).

$$q_t = \frac{1}{\beta}\ln(\alpha\beta) + \frac{1}{\beta}\ln t \quad (4)$$

where α and β are Elovich constants and α is considered as initial sorption rate (mg/g.min) and β is related to the extent of surface coverage and activation energy for the chemisorption. The plot of q_t vs lnt for Elovich model is given in Fig. 8. The values of α and β are determined from intercept and slope of linear plot of q_t vs lnt and are given in Table 1. The value of correlation cofficient (R^2) was 0.974 lower than that of pseudo-second-order model.

3.5.4 Liquid film diffusion model

The liquid film model is expressed as (Liu *et al.* 2013).

$$Ln(1-F) = -K_{fd}t + C_{fd} \quad (5)$$

where K_{fd} is liquid film diffusion rate constant and F=q_t/q_e. The attained results of this model for adsorption of AG-25 onto anion exchange membrane DF-120B are quite similar to pseudo-first order model. Thus, the liquid film diffusion model and pseudo-first order model are equivalent.

3.5.5 Bangham equation

Bangham equation is given as (Rahmani-Sani *et al.* 2015)

$$\log\log\left(\frac{C_o}{C_o - q_t m}\right) = \log\left(\frac{k_o m}{2.303V}\right) + \alpha \log t \quad (6)$$

where C_o is the initial dye concentration (mg/L), V is volume of solution (mL), q_t is amount of dye adsorbed (mg/g) at time t, m is weight of adsorbent used (g/L). The α and k_o (mL/g/L) are the constants of Bangham equation. The plot of loglog(C_o/C_o-q_tm) vs logt is a straight line with correlation coefficient of 0.907 and is given in Fig. 9. The values of α and k_o were calculated from slope and intercept and are given in Table 2. The double logarithmic plot did not give linear curves for AG-25 removal by DF-120B

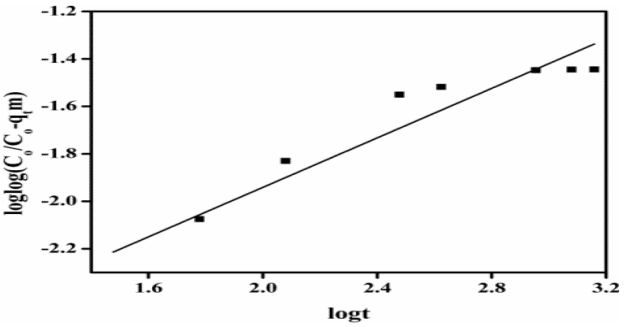

Fig. 9 Bangham model for adsorption of AG-25 on DF-120B membrane

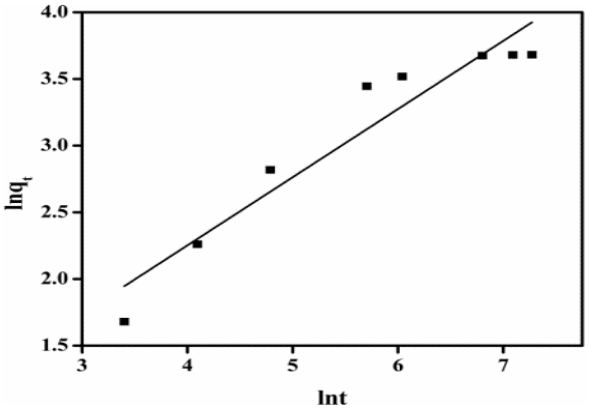

Fig. 10 Modified Freundlich model for adsorption of AG-25 on DF-120B membrane

Table 2 Modified Freunlich equation and Bangham equation rate

Modified Freundlich equation			Bangham equation		
m	k	R^2	k_o	α	R_2
1.96	0.025	0.905	0.96	0.52	0.907

Constants (k : L/g.min; k_o: mL/g/L)

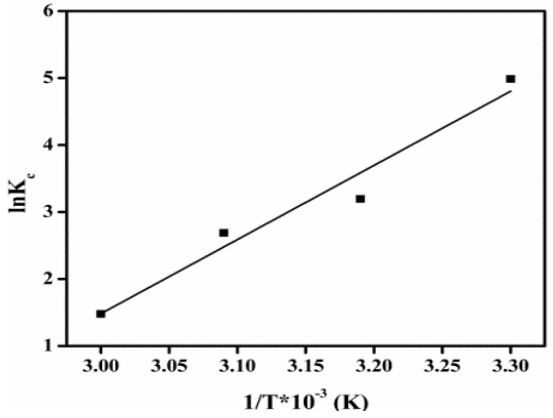

Fig. 11 Plot of 1/T vs $\ln K_c$ for adsorption of AG-25 on DF-120B membrane

Table 3 Thermodynamic parameters for adsorption of AG-25 onto DF-120 membrane

ΔHº (KJ/mol)	ΔSº (J/mol)	ΔGº (KJ/mol)			
		303K	313K	323K	333K
-91.99	-263.68	79.90	82.50	85.30	87.90

indicating that the diffusion of dye into pores of the membrane is not the only rate controling step (Khan *et al.* 2015). It can be concluded that both film and pore diffusion were important to different extent in the removal AG-25 from aqueous solution.

3.5.6 Freundlich equation

The modified freundlich eaquation was orignally developed by Kuo and Lotse (Khan *et al.* 2015).

$$q_t = kC_o t^{1/m} \tag{7}$$

where q_t is amount of adsorbed dye (mg/g) at time t, k is apparent adsorption rate constant (L/g.min), C_o is the initial dye concentration (mg/L), t is the contact time (min) and m is the Kuo-Lotse constant. The values of k and m were used to evaluate the effect of dye surface loading and ionic strength on the adsorption process.

Linear form of modified Freundlich equation given as

$$\ln q_t = \ln(kC_o) + \frac{1}{m}\ln t \tag{8}$$

The graphical representation of modified Freundlich model is given in Fig. 10. The parameters *m* and *k* were determined from slope and intercept and are given in Table 2. The correlation coefficient values were 0.905.

3.6 Adsorption thermodynamics

Thermodynamic parameters show the feasibility and spontaneity of adsorption process. The parameters namely change in Gibb's free energy (ΔG^o), enthalpy (ΔH^o) and entropy (ΔS^o) were determined from given equations

$$\ln Kc = \frac{\Delta S^o}{R} - \frac{\Delta H^o}{RT} \tag{9}$$

$$K_c = \frac{C_a}{C_e} \tag{10}$$

$$\Delta G^o = \Delta H^o - T\Delta S^o \tag{11}$$

where K_c, C_a, C_e, R, and T are the the equilibrium constant, amount of dye (mol/L) adsorbed on the adsorbent per litre (L) of the solution at equilibrium, equilibrium concentration (mol/L) of dye in solution, general gas contant (8.31 J/mol.K) and absolute temperature (K) respectively. Similarly ΔG^o, ΔH^o and ΔS^o are the change in Gibb's free energy (KJ/mol), enthalpy (KJ/mol) and entropy (J/mol.K) respectively. The plots of $\ln K_c$ Vs 1/T for adsorption of AG-25 is shown in Fig. 11. The adsorption change in enthalpy (ΔH^o) and entropy (ΔS^o) were determined from slope and intercept of linear Vant Hoff's plot and are given in Table 3. The values of Gibb's free energy (ΔG^o) were positive at all the temperatures studied. It might be because of intraction between adsorbent and adsorbate, with unbalanced competition imputed to heterogeneity of membrane surface and system got energy from external source at higher temperatures.

The nagative value of enthalpy (ΔH^o) indicates that the adsorption of AG-25 onto DF-120B is exothermic process.

Similarly the negative values of entropy (ΔS^o) represents decrease in randomness at the dye-membrane interface during the adsorption of AG-25 onto DF-120B.

4. Conclusions

The adsorption of anionic dye acid green (AG-25) from aqueous solution onto anion exchange membrane (DF-120B) was investigated at room temperature. The results showed that AG-25 was successfully removed from aqueous solution by membrane DF-120B. The removal of AG-25 dye was enhanced with contact time and membrane dosage whereas deceased with ionic strength and temperature. Adsorption kinetics showed that the experimental data fitted well to the pseudo-first-order model with highest R^2 value than all the kinetic models applied. Moreover, thermodynamic study indicates that the adsorption of AG-25 onto DF-120B was exothermic process. The negative value of ΔS^o suggested that the randomness decreases between dye-membrane interface during adsorption process. Thus the anion exchange membrane DF-120B is an excellent adsorbent for anionic dye AG-25 removal from wastewater and can be employed for the wastewater management at industrial level with proper scale up.

Acknowledgments

The authors are highly thankful to CAS-TWAS President's fellowship for Ph.D. programs for financial support.

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