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Research Paper

Adsorption modeling of alizarin yellow on Biosorbent casuarina equisetifolla

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Abstract-*Adsorption studies of alizarin yellow from an aqueous solution were carried out on casuarina equisetifolla. Casuarina equisetifolla was subjected to various pretreatments. Various parameters such as pH, dosage amount, and contact time were studied to evaluate the adsorption behavior. Langmuir, Freundlich, Temkin, Harkin-Jura, and Halsey isotherm models were used to explain the experimental findings. The Freundlich model was best fitted, pointing out multilayer adsorption. Thermodynamic parameters such as ΔG◦, ΔH◦, and ΔS◦ were also calculated, which showed that a spontaneous and favorable reaction occurred for casuarina equisetifolla. The positive values of ΔH◦ for biosorbent indicate that the adsorption mechanism was endothermic. From the Arrhenius equation, parameter E was equal to 19.1414kJ mol ⁻1, which corroborated the temperature dependency of the rate of adsorption.*

Keywords: Adsorption, alizarin yellow, casuarina equisetifolla, Langmuir, Freundlich, Temkin, Harkin-Jura, Halsey etc.

Introduction

Discharge of untreated or partially treated wastewaters and industrial effluents into natural ecosystems poses a serious problem to the environment [1]. Among effluents, dye-polluted water from textile and dyestuff industries is one of the most difficult waters to treat because of the synthetic and complex aromatic molecular structure of dyes, which makes them more stable and difficult to biodegrade [2]. It has been estimated that 10%-15% of dyes are lost in the effluents during the dyeing process [3]. Physicochemical processes such as electro coagulation, ozonation, photo catalysis, membrane filtration, and adsorption have been employed for the treatment of dye containing wastewaters [4]. Among these technologies, adsorption is considered an efficient technology that involves phase transfer of dye molecules onto the adsorbent, leaving the clear effluents behind. Adsorption is a cheap method for removing dyes using low-cost adsorbents such as wood cynodon dactylon and biosorbents[5] .Several research groups treated dye-polluted water by employing the adsorption principle[6] used rubber seed shells to remove Methylene blue from aqueous solutions). In addition, papaya seeds have been used as adsorbents for the adsorption of Methylene blue [7]. Acid-treated activated carbon was another adsorbent for dyes[8]. Researchers have also looked at the surface chemistry of activated carbons in order to interpret dye adsorption [9].

In the present work, alizarin yellow dye was removed by using *cynodon dactylon*. Alizarin yellow is a mordant dye, suitable for the dyeing of wool and nylon [10]. It usually exists as a sodium salt. In its pure form, it is a rust-colored solid[11]. Its molecular formula is C 13 H 8 N 3 NaO_5 (Na salt) (Fig1), its molecular mass is 309.21 g mol $^{-1}$ and its λmax is 370 nm. It is a slightly brown powder soluble in cold water. It causes irritation in the eyes, skin, digestive tract, and respiratory tract. This study also includes the calculation of equilibrium parameters and kinetics and a comparison of treated and untreated cynodon dactylon for adsorption of alizarin yellow. In addition, Langmuir, Freundlich, Temkin, Harkin-Jura, and Halsey isotherms were studied in detail.

Experimental work

Instrumentation

The pH was adjusted with a digital pH meter (Jenway Model 3320) using HCl (0.1 mol L −1) and NaOH (0.1 mol L −1). Alizarin yellow was estimated with a UV/VIS spectrophotometer (Labomed UVD 3500) at λmax 370 nm.

Preparation of adsorbent

Casuarina equisetifolla, collected from the Tranquebar coastal area, was crushed with laboratory-scale crushers, powdered with a disk pulverizer, and sieved to 0-63 mesh (ASTM). The powdered adsorbent was washed, dried at 105 ◦ C for 10 h in an oven, and stored in high-density polythene (HDPE) bags. The proximate analysis of the coal was carried out by using standard methods (ASTM D 5142-90). Powdered adsorbent was soaked in HCl (0.1 mol L −1) for 24 h, followed by filtering and washings with distilled water. Afterwards, it was dried in an oven at 105 ◦ C for 10 h and stored in HDPE bags.

Chemicals

All chemicals used during experimental work were of analytical grade and were used as such without purification. Alizarin yellow (Fluka), HCl (E. Merck 11.6 M). Double distilled water was used for the preparation of all types of solution and dilution when required.

Instrumentation

Balance ER-120A (AND), Electric grinder (Kenwood), pH meter HANNA pH 211 (with glass electrode), UV/VIS spectrophotometer (Labomed, Inc. Spectro UV-Vis double beam UVD = 3500).

Standard Solutions

1.0 g of Alizarin yellow was taken in 1000 mL measuring flask and dissolved in double distilled water, making volume up to the mark. This was 1000 ppm stock solution of dye. Standard solutions of dye were prepared by successive dilution of stock solution.

Adsorption Experiments

The adsorption studies were carried out at 30 ± 1 °C. pH of the solution was adjusted with 0.1 N HCl. A known amount of adsorbent was added to sample and allowed sufficient time for adsorption equilibrium. Then the mixture were filtered and the remaining dye concentration were determined in the filtrate using (Spectro UV-Vis Double Beam UVD-3500, Labomed.Inco) at λ_{max} = 370 nm.The effect of various parameters on the rate of adsorption process were observed by varying mesh size of adsorbent, contact time, t , initial concentration of dye C_O , adsorbent amount , initial pH of solution and temperature. The solution volume (V) was kept constant 50 mL).The dye adsorption (%) at any instant of time was determined by the following equation:

Dye adsorption (%) = (C_O – C_e) × 100/ C_O

Where C_O is the initial concentration and C_e is the concentration of the dye at equilibrium. To increase the accuracy of the data, each experiment was repeated three times and average values were used to draw the graphs.

Isotherm studies

A series of experiments Were carried out for isothermal and kinetic study of casuarina equisetifolla adsorption of alizarin yellow dye.Langmuir (eq :1) ,Freundlich(eq :2) ,Temkin(eq :3) ,Harkin-Jura (eq :4),Halsey(eq :5),Redlich-peterson(eq :6) and ,Dubinin-Kaganer-Radushkevich(DKR) (eq :7) were plotted by using standard straight-line equations and corresponding parameters were calculated from their respective graphs.

$Ce/X = 1/K*K_L + Ce/ K$ ----------------------- (1)

$\log qe = \log K_F + 1/n \log Ce$ ---------------------- (2)

$qe = K_T \ln Ce + b_T$ ------------------------ (3)

$1/qe2 = B/A - 1/A \log Ce$ ------------------------ (4)

$\ln qe = 1/n \ln K – 1/n \ln Ce$ ------------------------- (5)

$qe = K_R Ce / (1+b_R Ce\beta)$ ------------------------- (6)

$\log qe = \log Xm – \beta\varepsilon^2/2.303$ ----------------------- (7)

Ce is the equilibrium concentration of the adsorbate (mg/L) and X is the amount of adsorbate adsorbed (mg/g). K_L indicates monolayer adsorption capacity (mg/g),K is the Langmuir equation constant(L/mg),K_F and 1/n are constants for a given adsorbate and adsorbent at a particular temperature and bT (KJ/mol) is adsorption potential of the adsorbent . K_T is the Temkin isotherm constant and 1/A is the external surface area for the Harkin –Jura isotherm.$K_{R,}$ $b_{R,,}$β are Redlich Peterson constants. Xm is maximum sorption capacity; β is mean sorption energy and ε sorption potential in DKR isotherms

Results and Discussion

In order to find the appropriate conditions of particle size of the adsorbent, adsorbent dose, concentration of dye, contact time, pH, stirring speed, and temperature for the adsorption of Alizarin yellow on Casuarina equisetifolla, various experiments were conducted. The results of these experiments were as followed.

Characterization of adsorbent

The adsorbent analysis revealed that it had high moisture content and volatile matter. Ash content was also appreciable. Results are illustrated in Table 1.It was determined by proximate and ultimate analysis as follows:

Proximate analysis

Moisture

About 1g of finely powdered air-dried adsorbent sample is weighed and taken in a crucible. The crucible is placed inside an electric hot-air oven and heated at 100-105'C for 1hour. It is then taken out, cooled in a desicator and weighed. From this, the percentage of moisture can be calculated as follows:

*Percentage of moisture = (loss in weight of adsorbent / weight of air dried adsorbent taken)*100*

Volatile matter

The crucible with moisture free adsorbent sample is covered with a lid and placed in an electric muffle furnace, heated at 905-945'C for seven minutes. It is then taken out, cooled in a desicator and weighed. From this, the percentage of volatile matter can be calculated as follows:

*Percentage of volatile matter = (loss in weight of adsorbent / weight of dried adsorbent taken)*100*

Ash content

The crucible with residual adsorbent sample is placed in an electric muffle furnace, heated without lid at 650-750'C for 30 minutes. It is then taken out, cooled in a desicator and weighed. From this, the percentage of ash content can be calculated as follows:

*Percentage of ash = (weight of ash left / weight of dried adsorbent taken)*100*

Fixed carbon

The fixed carbon content can be calculated from the following equation

Percentage of = 100- % of (moisture+ volatile matter + ash)

Ultimate analysis of Sulphur

A known quantity of adsorbent sample is burnt completely in a Bomb calorimeter. During this process sulphur is converted in sulphate, which is extracted with water. The extract is then treated with $BaCl_2$ solution so that sulphates are precipated as $BaSO_4$.The precipitate is filtered, dried and weighed. From the weight of $BaSO_4$ obtained, the sulphur present in the adsorbent was calculated as follows.

$$\underset{(32)}{S} \xrightarrow{2O_2} SO_4^{2-} \xrightarrow{BaCl_2} \underset{(233)}{BaSO_4}$$

Percentage of sulphur in adsorbent = (32 weight of $BaSO_4$ obtained) / (233* weight of dried adsorbent taken)*

Results are given in table: 1

Effect of Mesh Size

The effect of adsorbent's mesh size was studied in the range of 0-200 microns mesh size (0-63, 63-125, 125-200) for checking the maximum adsorption of Alizarin yellow, and the smallest mesh size (0-63) was shown to be best for adsorption, as particles with smallest size presents a larger surface area and the results are shown in Fig. 2. Mesh size is inversely related with particle size. As the mesh size is larger, the size of particle is accordingly decreased which results in more surface area available for adsorption.

Effect of Adsorbent Dose

The effect of variation in the adsorbent amount on the process adsorption of Alizarin yellow was studied, with different adsorbent amount in the range of 50-200mg. The results obtained are shown in Fig. 3. From Fig. 3, it is observed that adsorption increases with increasing amount of Casuarina equisetifolla dose. Maximum removal was 89.75 % for dye dose of 25 ppm. The increase in adsorption with increase in amount of Casuarina equisetifolla dose is due to the fact that more surface area is available for adsorption or in other words more active sites are available.

Effect of Initial Dye Concentration

Initial dye concentration was one of the effective factors on adsorption efficiency. The percentage of Alizarin yellow adsorption was studied as a function initial dye concentration of in the range of 10-60 ppm. The results obtained are present in Fig.4. The percentage adsorption increases with increase in initial concentration of the dye for Casuarina equisetifolla. It was observed that adsorption yield increased with increase in initial concentration of the dye. Minimum adsorption was 60.41 % for 60ppm concentration to maximum adsorption value 93.29% for 10 ppm concentration of dye solution. This may be due to available active sites and increase in the driving force of the concentration gradient, as an increase in the high initial concentration of the dye.

Effect of Contact time

Contact time was one of the effective factors in adsorption process. The percentage of Alizarin yellow adsorption was studied as a function of contact time in the range of 30-180 minutes. The results obtained were presented in Fig. 5. It was observed that with the increase of contact time, the percentage adsorptions also increased. Minimum adsorption was 60.55 % for time 30 minutes to maximum adsorption value 92.98 % for the time 180 minutes for 25 ppm initial concentration of dye solution. The adsorption characteristic indicated a rapid uptake of the dye. The adsorption rate however decreased to a constant value with increase in contact time because of all available sites was covered and no active site available for binding.

Effect of pH

The pH of the aqueous solution was clearly an important parameter that controls the adsorption process. The percentage of Alizarin yellow adsorption was studied as a function of pH in the range of 1-5. The results obtained were shown in Fig. 6. The minimum adsorption was 0.54% at pH 5.0 and maximum adsorption was 97.69% at pH 1.0 for 25 ppm initial concentration of dye solution. This might be due to the weakening of electrostatic force of attraction between the oppositely charged adsorbate (Alizarin yellow) and adsorbent (Casuarina equisetifolla) that ultimately resulted in the reduced % age adsorption.

Effect of Temperature

Temperature has an important effect on the rate of adsorption. The percentage of Alizarin yellow adsorption was studied as a function of temperature in the range of 30-60 °C. The results obtained were present in Fig. 7. It was observed that adsorption yield increase with increase in temperature. The minimum adsorption was 93.25 % at 30 ° C and maximum adsorption was 96.51 % at 60°C for 25 ppm initial concentration of dye solution. The increase in adsorption at high temperature because molecules move with great speed and strong interaction was available for dye anions with adsorbent material.

Adsorption isotherm

Isotherm parameters, evaluated from the linear plots of equations (1-7) are illustrated in Table 2a, 2b, 2c,(Fig:8-14).The K_L value for the Langmuir isotherm, ie. 3.424658mg/g, indicated the high adsorption capacity of biosorbent toward alizarin adsorption. The R^2 (correlation coefficient) value of 0.9741indicated that the Langmuir isotherm is good for explaining the alizarin yellow adsorption. The R^2 value calculated for the Freundlich isotherm was found to be 0.992, indicating that the experimental data can be explained by the Freundlich isotherm. The Kf (ultimate adsorption capacity) value as calculated from the Freundlich isotherm was 3.715352.The Temkin equation was also good to explain the experimental data ,with an R^2 value 0.962.bT (heat of sorption) was calculated from the Tempkin plot as 3.594KJ/mol ,indicating moderately strong cohesive forces between alizarin yellow and biosorbent. A value less than 8 indicates a weak interaction between the adsorbent and adsorbate (Anwar et al.,2010).The Harkin –Jura expression of the value of the correlation coefficient was 0.950,providing good suitability for the experimental data of Alizarin yellow on casuarina equisetifolla. Halsey's expression of the value of the correlation coefficient was 0.983, providing a better fit for the experimental data of Alizarin yellow on casuarina equisetifolla. The Harkin-Jura and halsey equations were more suitable to explain the multi layer adsorption of the adsorbate on adsorbent [11]. The R^2 value calculated for the Redlich-peterson isotherm was found to be 0.916, indicating that the experimental data can be explained by the Redlich-peterson isotherm. The β value as calculated from this isotherm was 1.592. The R^2 value calculated for the DKR isotherm was found to be 00.967, indicating that the experimental data can be explained by the DKR isotherm poorly. The β value as calculated from this isotherm was 0.987987.

Thermodynamic parameters

Thermodynamic parameters such as standard Gibbs free energy (ΔG^0),Enthalpy (ΔH^0) and entropy (ΔS^0) were also calculated using equations 8 and 9 and the results obtained are illustrated in table-3a,3b (Fig:15).

$$\Delta G^0 = -RTlnK \quad \text{----------------------------8}$$

$$lnKc = (\Delta S^0/R)-(\Delta H^0/RT) \quad \text{--------------------------9}$$

Here, K denotes the distribution coefficient for the adsorption. R is the universal constant and T is the absolute temperature in Kelvin. The negative value of the ΔG^0 at the studied temperature range indicated that the sorption of alizarin yellow on sorbent was thermodynamically feasible and spontaneous. The increase in the value of ΔG^0 with temperature further showed the increase in feasibility of sorption at the elevated temperature for casuarina equisetifolla. In other words, sorption is endothermic in nature .The positive value of ΔH^0 for casuarina equisetifolla showed that the sorption was endothermic. The positive value of ΔS^0 showed an increased randomness at the solid alizarin yellow solution interface during the adsorption of alizarin yellow, reflecting the affinity of casuarina equisetifolla for alizarin yellow.

Arrhenius equation

Activation energies for adsorption of alizarin yellow on adsorbent was calculated using the Arrhenius equation (eq10),plotted in Fig 16 and tabulated in table 4.The activation energy obtained(Table 4) in this case ,indicate that physical forces are involved in the sorption mechanism and sorption feasibility.

Arrhenius equation

$$\text{Log } K = \text{Log } A - (E_A / 2.303 \quad RT) \quad \text{------------- 10}$$

Kinetic study

A linear trace for the plot of log (qe-q) Vs time (Fig: 17, 18, 19, 20) shows that the adsorption kinetics follow pseudo second order kinetics. Pseudo first order, Elovich and intraparticle diffusion kinetics studies were carried out (Table 5a, 5b, 5c).

pseudo second order

$$\text{Log } (q_e-q_t) = \log q_e - (K1t / 2.303) \quad \text{----------------- 11}$$

Conclusion

From the present study, it is concluded that Casuarina equisetifolla is a good adsorbent for the removal of the dyes from aqueous media. Optimum conditions for the removal of Alizarin yellow with Casuarina equisetifolla are: 0.6 g of adsorbent, dye concentration 25 ppm, at 30 °C, with 60 minutes contact time, 300 rpm agitation speed and at pH 1.0.

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Table: 1

Parameters	Moisture	Volatile matter	Ash	Fixed C	S
Values	2.1	8.5	3.2	80.6	1.1

Table2a

c0	%	ce	c0-ce	qe	ka	Ka C0	ce/qe	logce
10	93.29	0.671	9.33	3.11	4.6344	46.3438	0.2158	-0.1733
20	86.7	2.66	17.34	5.78	2.1729	43.4586	0.4602	0.4249
30	74.89	7.533	22.47	7.49	0.9942	29.8248	1.0059	0.8770
40	70.59	11.76	28.24	9.41	0.8001	32.0027	1.2499	1.0706
50	65.52	17.24	32.76	10.92	0.6334	31.6705	1.5788	1.2365
60	60.41	23.75	36.25	12.08	0.5086	30.5178	1.9661	1.3757

Table2b

log qe	log ce/qe*-1	ceβ	1/ce+1	ε2	1/qe2
0.4927	0.6660	0.529837	2.4903	5.2883	0.1034
0.7619	0.3370	4.746916	1.3759	0.6470	0.0299
0.8744	-0.0025	24.8962	1.1327	0.0987	0.0178
0.9737	-0.0969	50.62017	1.0850	0.0423	0.0113
1.0382	-0.1983	93.01807	1.0580	0.0202	0.0084
1.0821	-0.2936	154.9433	1.0421	0.0108	0.0069

Table2c

Langmuir parameters	K_L = 3.424658	q0 = 13.5135	b_L = 0.253425	R^2 =0.974
Freundlich parameter	K_F = 3.715352	n =2.673797		R^2 = 0.992
Dubinin-kaganer-Radushkevich parameters	**β** = 0.987987	b = 0.469	q0 =2.944422	R^2 = 0.967
Redlich peterson parameters	β= 1.592	K_R = 0.8833	b_R = 0.003534	R^2 = 0.916
Tempkin parameters	k_T= 5.658	b_T = 3.594		R^2 = 0.962
Harkin-jura parameters	A= 40	B = 1.6		R^2 = 0.983
Halsey parameters	N= 2.673797	K = 4.59084		R^2 = 0.992

Table3a

T,K	c0	%	Ce	c0-ce	ka
303.15	50	93.25	3.375	46.625	4.604938
313.15	50	94.54	2.73	47.27	5.771673
323.15	50	95.49	2.255	47.745	7.05765
333.15	50	96.51	1.745	48.255	9.217765

Table3b

∆G0	∆H0	∆S0	Log 10 Ka	1/T
-3.84896	22.59363	76.58857	0.663224	0.003299
-4.56389			0.761302	0.003193
-5.25005			0.84866	0.003095
-6.15211			0.964626	0.003002

Table4

Adsorbent	Log A	Ea (KJ/mol)
Casuarina equisetifolla	3.955	19.1414

Table5a

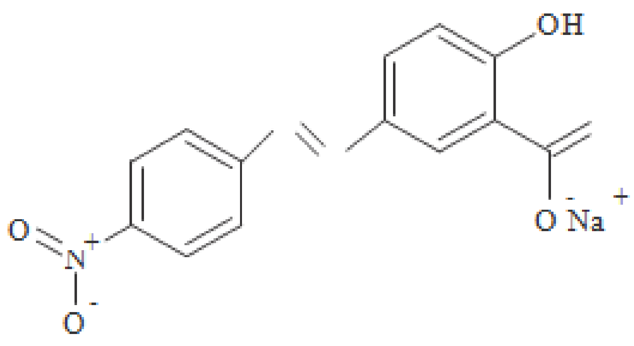
Time, in	%	C0	Ce	ct	qt
30	60.55	10	3.9	6.055	2.018
60	66.34	10	3.4	6.634	2.211
90	71.56	10	2.8	7.156	2.385
120	75.00	10	2.5	7.5	2.500
150	87.56	10	1.2	8.756	2.919
180	92.98	10	0.7	9.298	3.099

Table 5b

t/qt	qe-qt	Log (qe-qt)	t1/2	1/qt	1/t	Ln t
14.864	1.382	0.1404033	5.477226	0.495458	0.033333	3.401197
27.133	1.189	0.0750601	7.745967	0.452216	0.016667	4.094345
37.731	1.015	0.0063234	9.486833	0.419229	0.011111	4.49981
48.000	0.900	-0.0457575	10.95445	0.4	0.008333	4.787492
51.393	0.481	-0.3175541	12.24745	0.342622	0.006667	5.010635
58.077	0.301	-0.5219147	13.41641	0.32265	0.005556	5.192957

Table5c

I Order	K1 = 0.009212	Qe = 1.409169	R2=0.900
II Order	K2 = 0.008383	Qe = 3.521127	R2=0.967
Intra particle diffusion	Kp = 0.135	C = 0.819	R2=0.930
Elovich model	B = 0.347	A = 0.819	R2=0.988



Structure of alizarin yellow (Na salt).

Fig 1

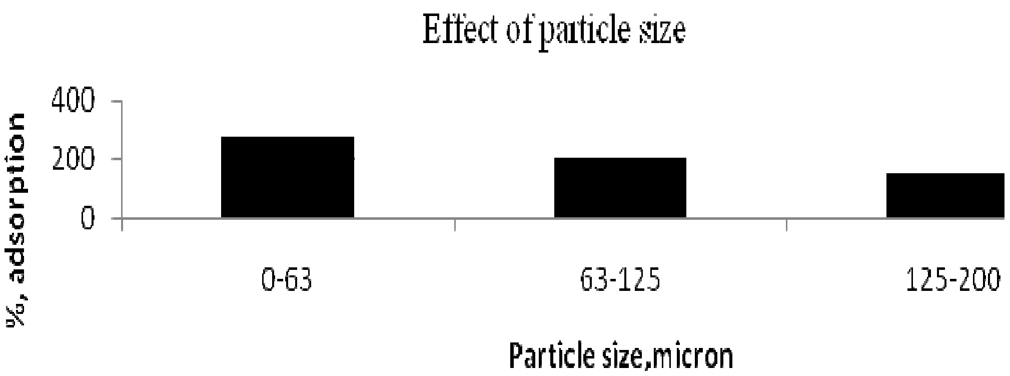


Fig 2

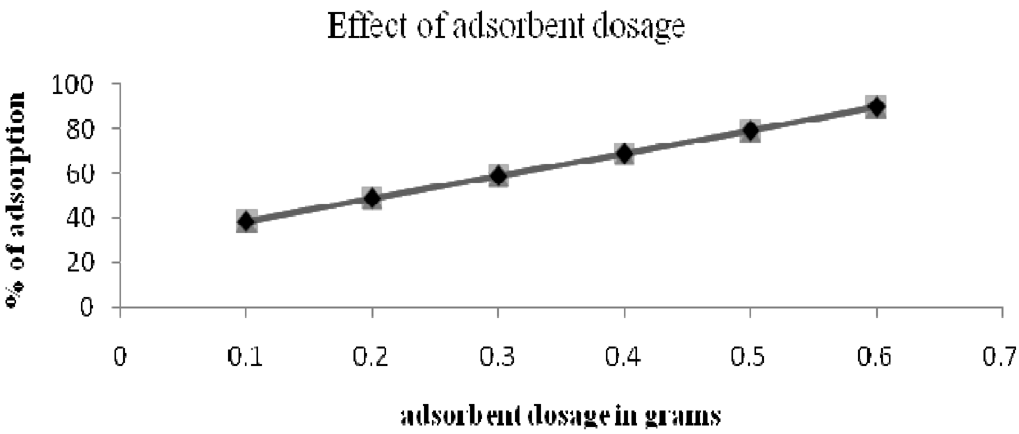


Fig 3

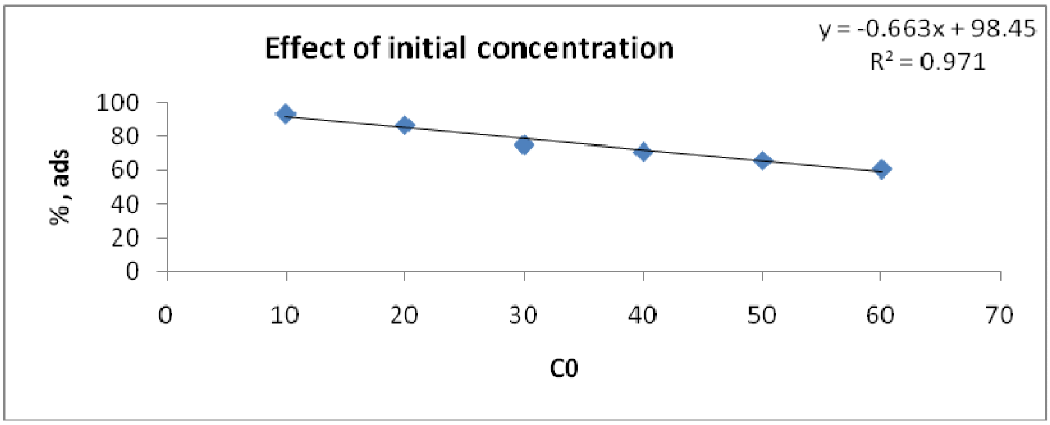


Fig 4

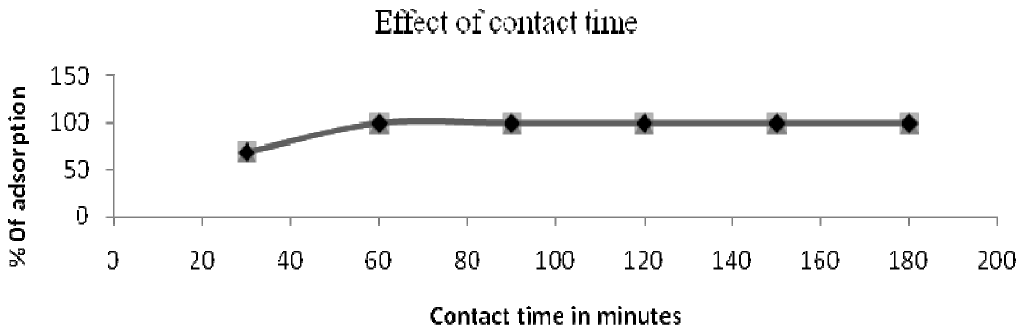


Fig 5

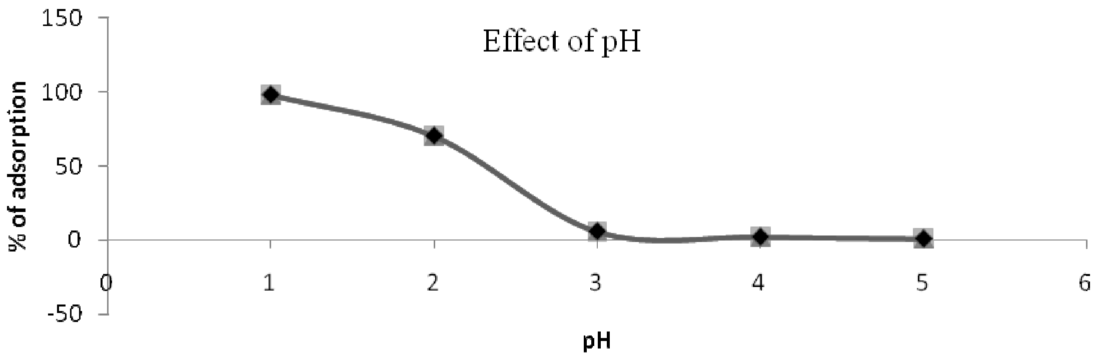


Fig 6

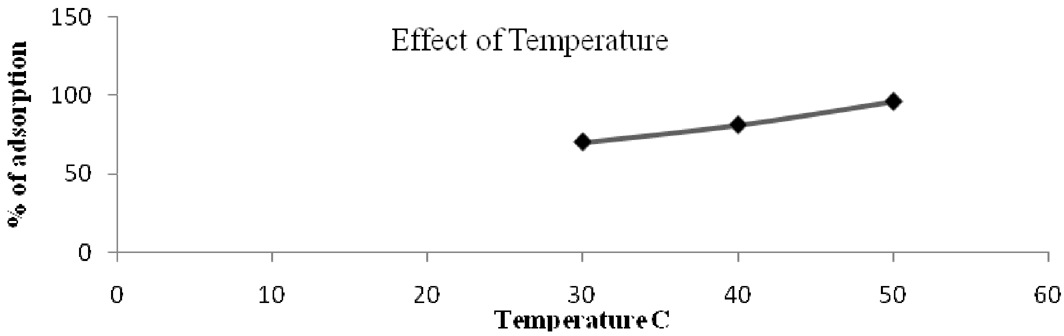


Fig 7

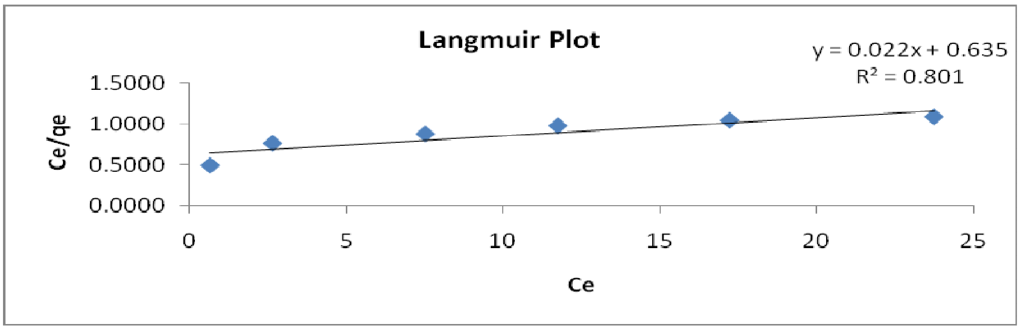


Fig 8

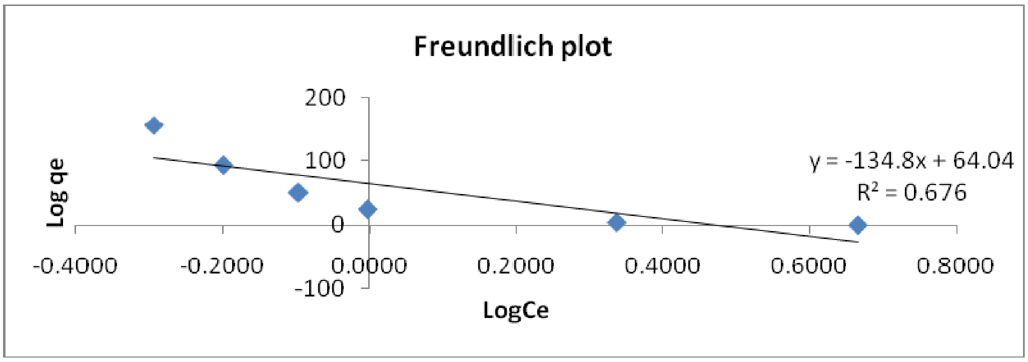


Fig 9

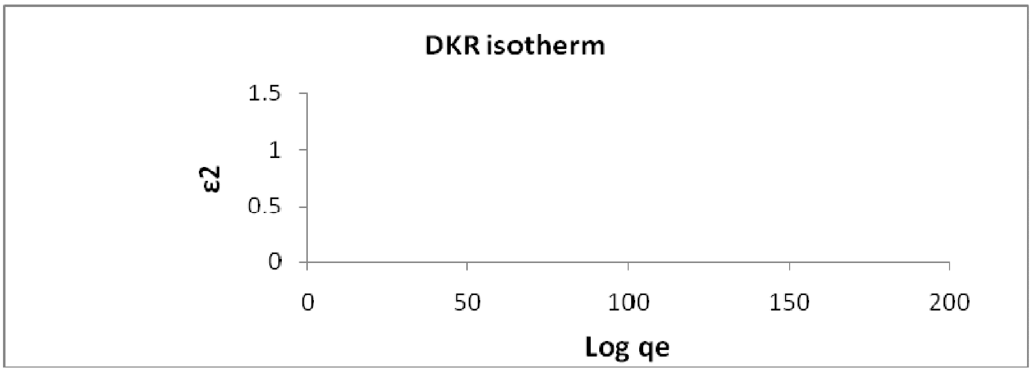


Fig 10

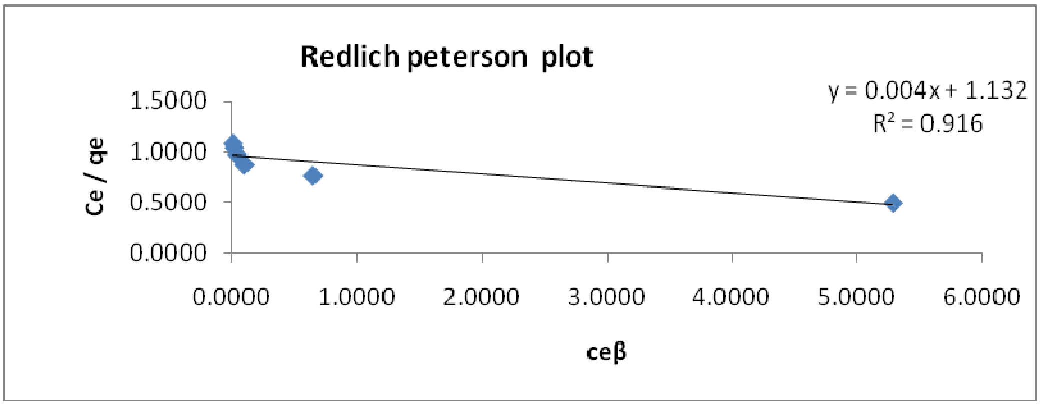


Fig 11

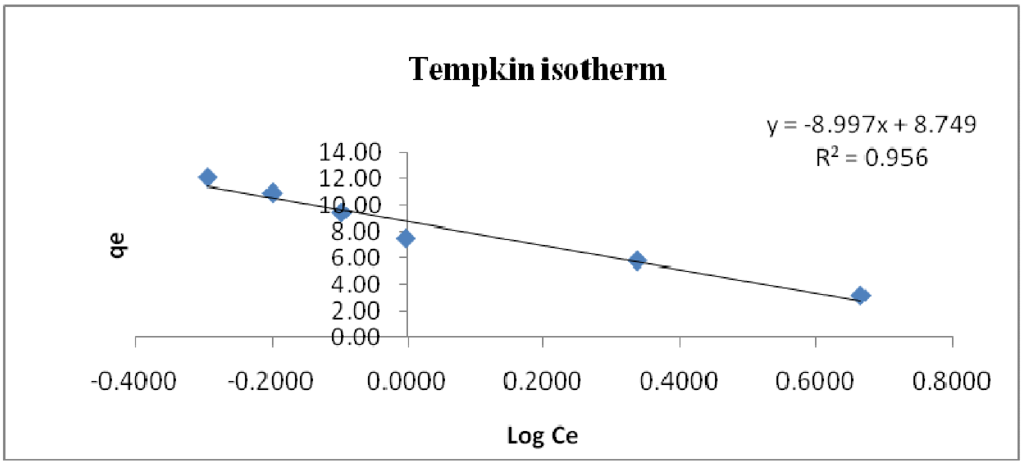


Fig 12

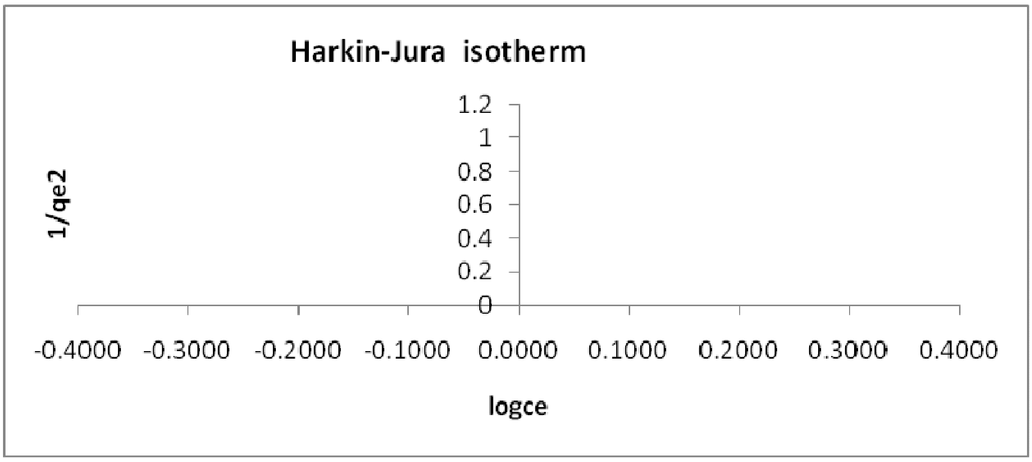


Fig 13

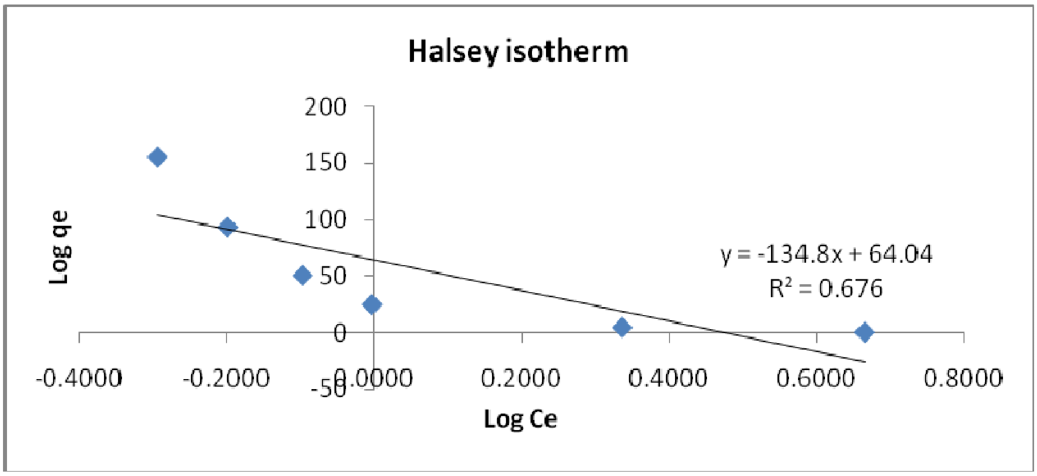


Fig 14

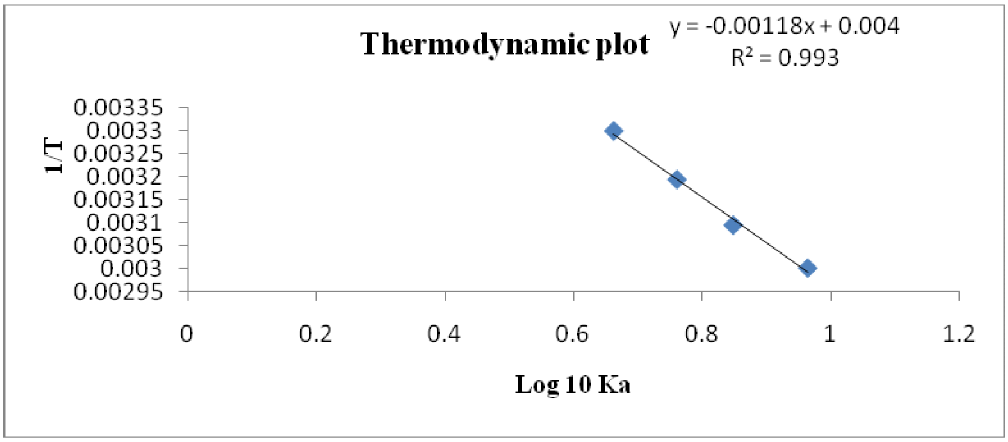


Fig 15

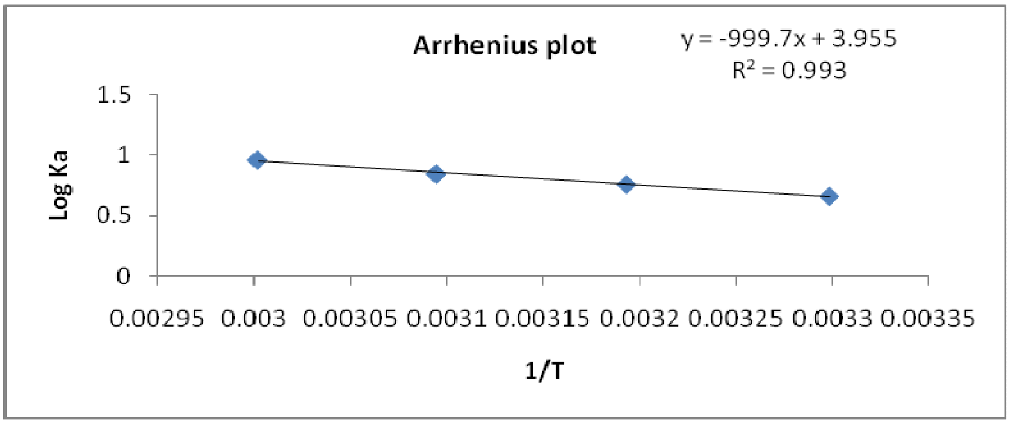


Fig 16

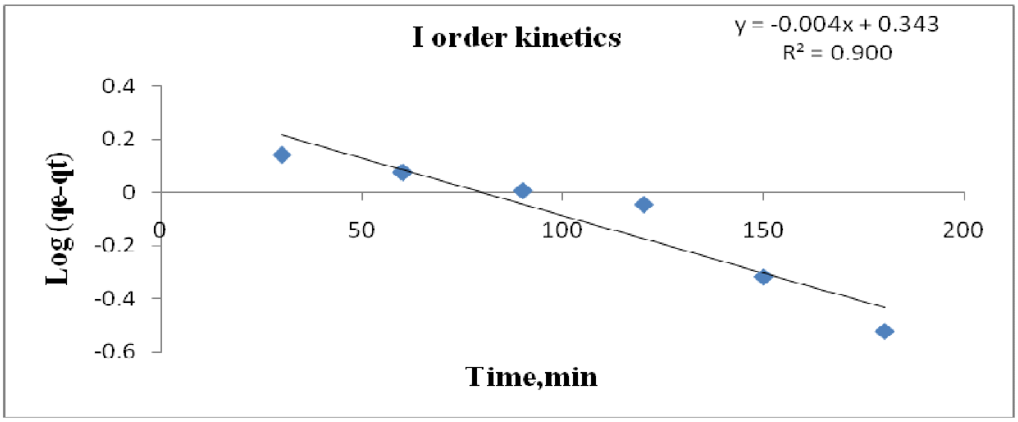


Fig 17

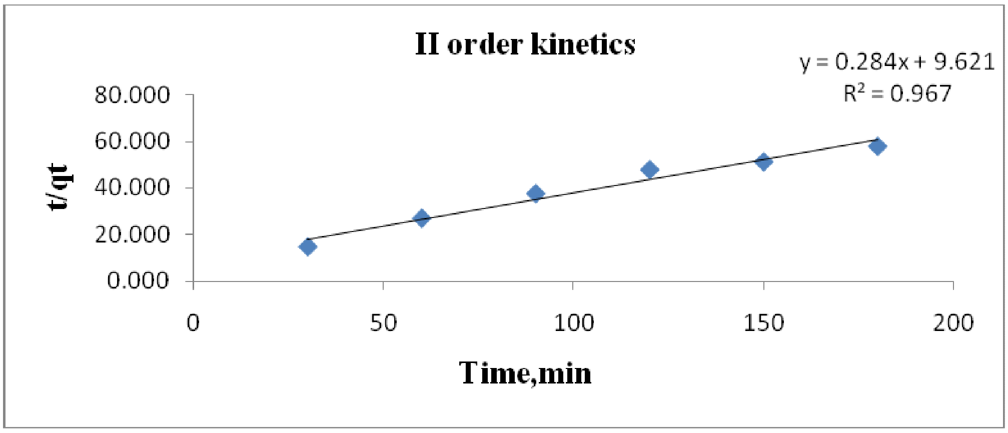


Fig 18

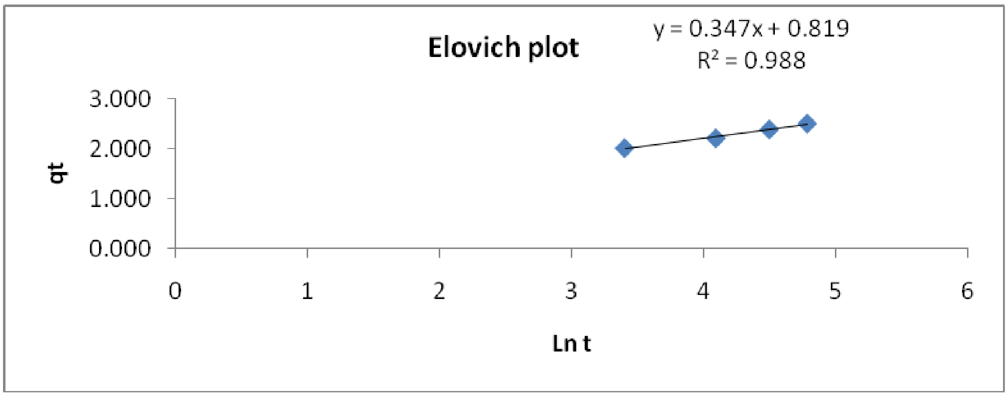


Fig 19

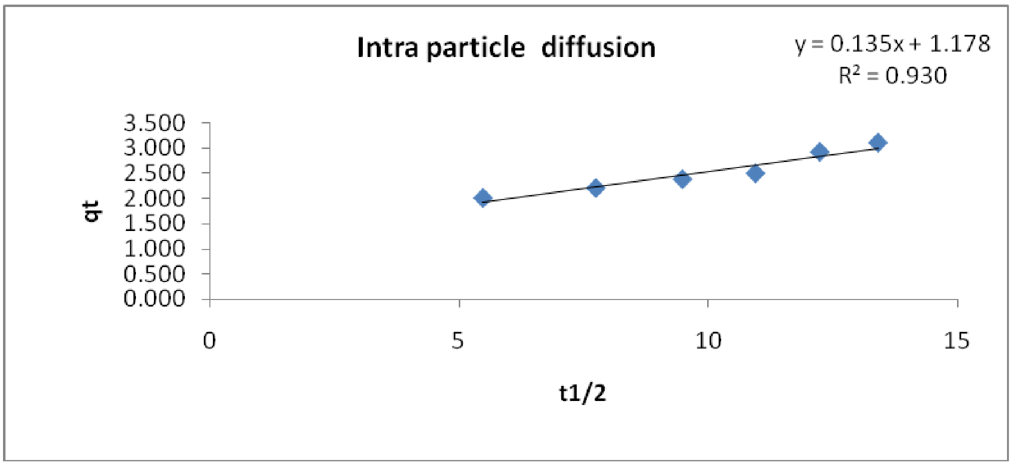


Fig 20