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Adsorption of Acid Blue 92 from Aqueous Solution using an Activated Carbon Prepared from *Sterculia quadrifida* Seed Shell Waste

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Abstract

Studies on removal of Acid Blue 92 from aqueous solution using activated carbon prepared from Sterculia Quadrifida seed shell waste as an adsorbent have been carried out. The influences of initial Acid Blue 92 concentration (20, 40 & 60 mg/L), temperature (30 °C, 45 °C and 60 °C) and pH have been reported. Adsorption of Acid Blue 92 is highly pH dependent and the results are obtained at the optimum pH of 6.5 for the removal of dye. Kinetic parameter such as Pseudo first order, the Elovich model and the Pseudo second order was also discussed. A probable explanation was offered to account for the results of isotherms studies such as Langmuir, Freundlich and Temkin adsorption isotherms.

Keywords: *Sterculia Quadrifida seed shell waste, Adsorption of Acid Blue 92, Optimum pH, Adsorption kinetics, Isotherm studies.*

1. INTRODUCTION

Colour or dye being one of the recalcitrant, persist for long distances in flowing water, retards photosynthesis, inhibit growth of aquatic biota by blocking out sunlight and utilizing dissolved oxygen. Some dyes may cause allergic dermatitis, skin irritation, cancer and mutation in man. The methods of colour removal from industrial effluents include coagulation, floatation, biological treatment, hyper filtration, adsorption and oxidation. Among these options, adsorption is most preferred method and activated carbon is most effective adsorbent widely employed to treat wastewater containing different classes of dyes, recognizing the economical drawback of commercial activated carbon.

Many investigators have the feasibility of using inexpensive alternative materials like Jatropha Curcus seed shell, Delomix regia seed shell, Ipomea Carnia stem (Karthikeyan *et al.* 2008), Fly-ash (Nagamaik *et al.* 2003), Wollastonite (Jambulingam *et al.* 2007), Olive stones (Lopez, 1984), almond shells (Linares-Solano *et al.* 1980), apricot and peach stones (Nasser and Geundi, 1991), maize cob (Bousher *et al.* 1991), linseed straw (Kadirvelu *et al.* 2000), saw dust (Srinivasan *et al.* 2000), rice hulls (Rengaraj *et al.* 1999), cashew nut hull, cashew nut sheath (Banerjee *et al.* 1976), coconut shells and husks (Niraus *et al.* 2000), eucalyptus bark (Balasubramanian and Muralisankar, 1987), linseed cake, tea waste ash (Mittal and Venkobachar, 1989). Beside these other source of activated carbon is sulfonated coal (Lucchesi and Machio, 1983), tyre coal dust (Lambert *et al.* 1997), activated bauxite, cement kiln dust (Al-Qodah, 2000), Share oil ash (Xu *et al.* 1997) and ground sunflower stalk(Karthikeyan *et al.* 2008), Feronia limonia swingle shell (Karthikeyan *et al.* 2012) , Moringa oleifera fruit shell waste (Sumithra *et al.* 2014) etc., as the carbonaceous precursors for the removal of dyes from waste water.

In the present investigation the adsorption of Acid Blue 92 on activated carbon prepared from

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Sterculia Quadrifida seed shell waste by carbonization with phosphoric acid activation process. The kinetic and equilibrium adsorption data obtained ware utilized to characterize the sample prepared. The amount and rates of adsorption of Acid Blue 92 using above activated carbon from water were then measured. Three simplified kinetic models including Pseudo first order equations, Pseudo second order equations and Elovich equations were used to describe the adsorption process(Karthikeyan *et al.* 2010).

2. EXPERIMENTAL PROCEDURE

2.1 Adsorbent

Sterculia Quadrifida shells were collected from various places in Erode city, Tamil Nadu, India. They were cut into small pieces, dried in sunlight until all the moisture was evaporated. The dried Material was used for the preparation of activated carbons using physical and chemical activation methods. The materials to be carbonized were soaked with phosphoric acid for a period of 24 hours. After impregnation, the product was washed with large volume of water to remove the free acid, and then it was dried at 160 °C for 2 hours by using air oven finally activated at 800 °C and powdered.

The Nitrogen adsorption-desorption isotherms of activated carbons were measured using a gas sorption analyzer (NOVA 1000, Quanta Chrome corporation) in order to determine the surface area and the total pore volume. The surface area was calculated using the BET equation.

2.2 Batch adsorption studies

The batch adsorption studies were performed at 30 °C. A predetermined amount of adsorbent is mixed with known initial concentration of Acid Blue 92 solution and agitated for desired time. The adsorbent and the adsorbate were separated by filtration and the filtrate was analyzed for residual Acid Blue 92 concentration spectrophotometrically.

The amount of Acid Blue 92 adsorbed in mg/L at time t was computed by using the following equation.

$$q_t = \frac{C_o - C_t}{m_s} \times V \qquad (1)$$

where, C_o and C_t are the Acid Blue 92 concentration in mg/L initially and at given time t, respectively, V is the volume of the Acid Blue 92 solution in ml and m_s is the weight of the activated carbon. The percentage of removed Acid Blue 92 ions (R %) in solution was calculated using equation.

$$\% \text{ Removal} = \frac{C_o - C_t}{m_s} \times 100 \qquad (2)$$

The initial concentration of Acid Blue 92, pH and temperature was investigated by varying any one parameters and keeping the other parameters constant.

2.3 Kinetic Models

In order to investigate the mechanism of adsorption and potential controlling steps such as mass transport, several kinetic models were tested including the Pseudo first order kinetic model, the Elovich model and the Pseudo second order kinetic model for a batch contact time process, where the rate of sorption of dye on to the given adsorbent is proportional to the amount of dye sorbed from the solution phase.

2.3.1 Pseudo First Order Kinetic Model

A simple kinetic analysis of adsorption, the pseudo first order kinetics and its integrated form, is given by Lagergren (Akkaya and Ozer, 2005).

$$\frac{dq_t}{dt} = k_L (q_e - q_t) \qquad (3)$$

$$\log(q_e - q_t) = \log q_e - \frac{k_L}{2.303} t \qquad (4)$$

where k_1 is the pseudo first order rate constant. A plot of log (q_e-q_t) Vs time enables calculation of the rate constant k_1 and q_e from the slope and intercept of the plot.

2.3.2 Elovich Model

The Elovich or Roginsky–Zeldovich equation is generally expressed as follows

$$\frac{dq_t}{dt} = \alpha \exp(\beta q_t) \qquad (5)$$

On integrating this equation for the boundary conditions,

$$q_t = \frac{1}{\beta} 1n(\alpha\beta) + \frac{1}{\beta} \ln t \qquad (6)$$

where the initial dye adsorption rate (mg/g) and desorption constant (g/mg) respectively are obtained from the slope and intercept of linear plot of q_t vs ln t.

2.3.3 Pseudo Second Order Kinetic Model

To describe the dye adsorption, the modified pseudo second order kinetic equation is expressed as

$$\frac{dq_t}{dt} = k_2 (q_e - q_t)^2 \quad (7)$$

where, k_2 is the pseudo second order rate constant. A plot of t/q_t Vs t enables calculation of the rate constant k_2 which in turn is used to calculate the initial sorption rate h as follows

$$\frac{t}{q_t} = \frac{t}{k_2 q_e^{\,2}} + \frac{1}{q_e} t \quad (8)$$

2.4 Isotherm Models

2.4.1 Langmuir Isotherm

The Langmuir model was developed based on the assumption of the formation of a monolayer of the adsorbate species onto the surface of the adsorbent. It has also been assumed that the surface sites are completely energetically homogeneous. But in the true sense, the adsorbent surface is energetically heterogeneous. The study of the Langmuir isotherm is essential in assessing the adsorption efficiency of the adsorbent. This study is also useful in optimizing the operating conditions for effective adsorption. In this respect, the Langmuir isotherm is important, though The Langmuir and the rearranged Langmuir equation are given below

.

$$\frac{1}{q_e} = \frac{1}{Q_o b} \cdot \frac{1}{C_e} + \frac{1}{Q_o} \quad (9)$$

$$\frac{C_e}{q_e} = \frac{C_e}{Q_o} + \frac{1}{Q_o b} \quad (10)$$

Where, q_e = The amount of dye removed at equilibrium, mg/g.

C_e = The equilibrium concentration of dye, mg/L

Q_o = The Langmuir constant, related to the adsorption capacity, mg/g and

b = The Langmuir constant, related to the energy of adsorption, L/mg.

The essential characteristics of Langmuir isotherm can be expressed in terms of dimension less constant separate ion factor or equilibrium parameter, R_L which is defined by $R_L = 1/1+b\ C_o$, where C_o is the initial dye concentration (mg/L) and b is the Langmuir constant (L/mg). The parameter indicates the shape of isotherm as follows

R_L Value	Type of Isotherm
$R_L > 1$	Unfavourable
$R_L = 1$	Linear
$0 < R_L < 1$	Favourable
$R_L = 0$	Irreversible

2.4.2 Freundlich Isotherm

The linear form of Freundlich equation (Sumanjit and Prasad, 2001) is represented as

$$\log \frac{x}{m} = \log k_f + \frac{1}{n} \log C_o \quad (11)$$

where 'x' is the amount of Acid Blue 92 adsorbed in mg. 'm' is the weight of adsorbent (g), C_e is the residual concentration of Acid Blue 92 at equilibrium in mg.

K_f and 1/n are Freundlich constants related to the adsorption capacity and adsorption intensity respectively and are evaluated by least square fitting of the data by plotting log x/m vs log C_e with a slope of 1/n and intercept of log k_f.

2.4.3 Tempkin adsorption isotherm

Tempkin adsorption isotherm model was used to evaluate the adsorption potentials of the SQAC for Acid Blue 92. The derivation of the Tempkin isotherm assumes that the fall in the heat of adsorption is linear rather than logarithmic, as implied in the Freundlich equation. The Tempkin isotherm has commonly been applied in the following form

$$q_e = \frac{RT}{b} \ln AC_e \quad (12)$$

The Tempkin isotherm can be simplified to the following equation:

$$q_e = \beta \ln \alpha + \alpha \ln C_e \quad (13)$$

where β = (RT)/b, T is the absolute temperature in Kelvin and R is the universal gas constant,

8.314 J (mol K)$^{-1}$. The constant b is related to the heat of adsorption[24]. The adsorption data were analyzed according to the linear form of the Tempkin isotherm equation.

3. RESULTS & DISCUSSION

3.1 Optimum pH

The adsorption behavior of the Acid blue 92 on to activated carbon was studied over a wide range of pH 2 to 10 is represented in Fig. 1. The uptake of Acid Blue 92 decreased when the solution pH was increased from 2 to 10. The uptake of Acid Blue 92 by activated carbon was increased from pH 2 to 4 and then decreased. The interaction between the adsorbate and adsorbent is affected by pKa of dye as well as the isoelectric point (pH PZC) of the adsorbent16. The pKa value of Acid Blue 92 is 3.2. Below the isoelectric point (pH PZC = 5.6), the electrostatic repulsion between the adsorbed molecules is minimized, resulting in a maximum adsorption. At pH below 5.6, the surface of the adsorbent may acquire a positive charge leading to an increased anionic dye adsorption due to electrostatic attraction. At pH above the PZC the adsorbent surface acquires a negative charge, which repels the negative anionic dye molecules. On increasing the pH, the added NaOH increases the ionic strength, which also makes more competition for adsorption sites, hence, the adsorption decreases.

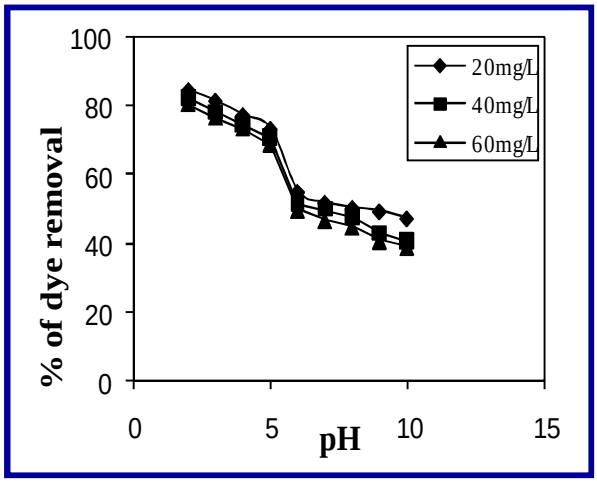

Fig. 1: Influence of pH on Acid Blue 92 adsorption

3.2 Adsorption kinetics

3.2.1 Effect of initial concentration on rate parameters

Acid Blue 92 dyes were carried out by using various concentrations of 20, 40 & 60 mg/L at P^H 6.5 at reaction temperature of 30 °C. The rate constant and the rate parameters at different initial dye concentration presented in the Table 1 shows that the rate constants increase with increase in initial dye concentration.

Table 1. Rate parameters for the adsorption of Acid Blue 92 onto SQAC seed shell waste activated carbon at various initial concentrations

Conc., mg/L	Pseudo First Order		Elovich			Pseudo Second Order			
	k_L, min^{-1}	R^2	β, g/ min	α, mg/g/ min	R^2	q_e, mg/g	k_2, g/mg/ min	h, g/ mg/ min	R^2
20	0.2330	0.7298	0.1750	1.569	0.9786	26.66	0.038	3.681	0.9931
40	0.0177	0.9616	0.1123	2.450	0.9869	42.01	0.062	0.912	0.9993
60	0.0186	0.9833	0.0822	2.543	0.9899	56.17	0.368	0.859	0.9985

3.2.2 Effect of initial concentration on rate parameters

Acid Blue 92 dyes were carried out by using various concentrations of 20, 40 & 60 mg/L at P^H 6.5 at reaction temperature of 30 °C. The rate constant and the rate parameters at different initial dye concentration presented in the Table 1 shows that the rate constants increase with increase in initial dye concentration.

From the analysis the data reveals that the influence of the initial concentration of Acid Blue 92

was little persuade on the Pseudo first order rate constant. In the case of Elovich model the obtained data reveals that the initial sorption rate (α) was increases for 20 mg/L, 40 mg/L and 60 mg/L simultaneously. Then the (β) desorption constant was decreases with the increase in concentration.

In the case of Pseudo second order the amount of dye adsorbed at equilibrium q_e increase simultaneously for 20 mg/L, 40 mg/L and 60 mg/L concentrations. Then the rate constant k_2 was increases for all the three concentrations. The initial adsorption rate (h) gradually decreases.

When increasing the initial dye concentration for 20 ppm, 40 ppm and 60 ppm the Acid Blue 92 the linear regression co-efficient (R^2) was very closer or equal to 1 for Pseudo second order kinetic model, where as the Pseudo first order and Elovich kinetic models the R^2 values was comparatively low than the Pseudo second order. Hence the relatively higher R^2 values indicate that the model successfully describes the kinetics of Acid Blue 92 adsorption (Fig. 1a, 1b, 1c).

3.2.3 Effect of temperature on kinetic rate parameters

The data obtained from experiment at various temperatures viz 30, 45 and 60 °C presented in the Table 2, the rate constant values K_L for Pseudo first order decreases with increase in temperature.

In the case of Elovich model the obtained data reveals that the initial sorption rate (α) was 1.1477, 1.0338 & 1.3114 for 30 °C, 45 °C and 60 °C temperature simultaneously. The desorption constant (β) increases with increase in temperature. In the case of Pseudo second order kinetic modal at the different temperature 30 °C, 45 °C and 60 °C, the amount of dyes adsorbed at equilibrium (q_e) and rate constant (k_2) decreases with increase in temperature. Then the initial adsorption rate (h) increases with increase in amount of dyes adsorbed at equilibrium (q_e).

When increasing the temperature 30 °C, 45 °C and 60 °C the adsorption of Acid Blue 92 on to SQAC, comparing the R^2 value obtained for Pseudo first order, Elovich and Pseudo second order, the relatively higher R^2 values obtained only for the Pseudo second order. Hence the Pseudo second order equation fits good correlation co-efficient (Fig. 2a, 2b, 2c) .

3.2.4 Effect of particle size on kinetic rate parameters

The experiments were conducted at different particle size viz 75-180, 180-280 and 280-355 microns, the rate constant were calculated and the results are presented in the Table 3.

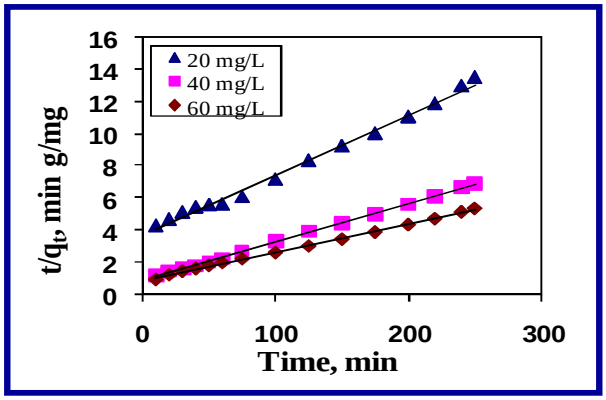

Fig. 1a: Effect of Initial Dye Concentration on Pseudo Second Order Plot for Acid Blue 92 Adsorption

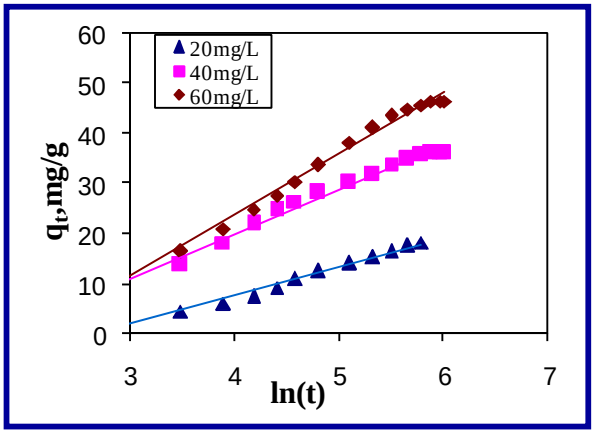

Fig. 1b: Effect of Initial Dye Concentration on Elovich Plot for Acid Blue 92 Adsorption

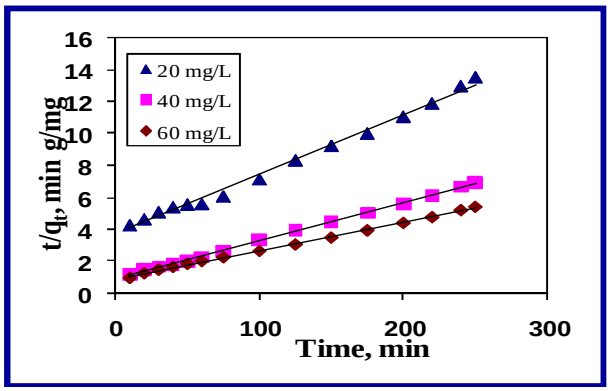

Fig. 1c: Effect of Initial dye concentration on pseudo second order plot for acid blue 92 adsorption

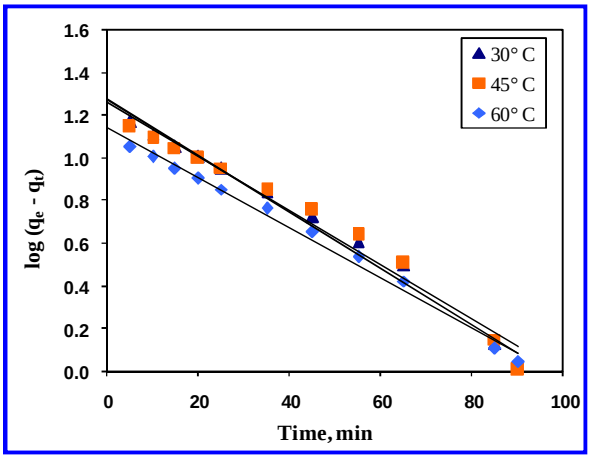

Fig. 2a: Effect of temperature on Pseudo first order plot for Acid Blue 92 adsorption

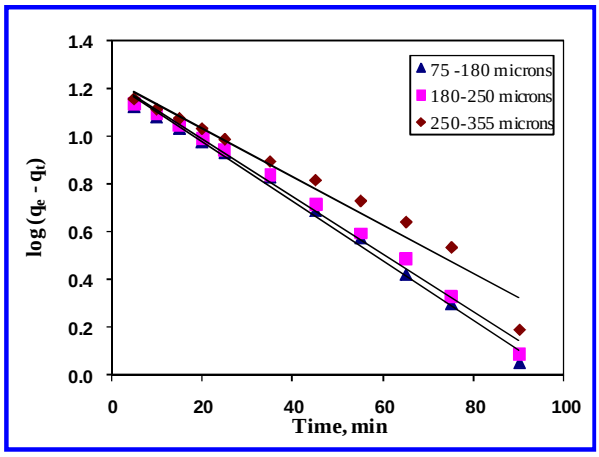

Fig. 3a: Effect of Particle size variation on Pseudo first order for Acid Blue 92 adsorption

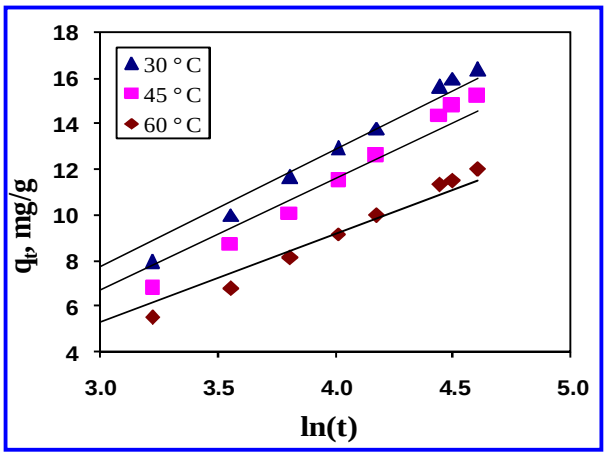

Fig. 2b: Effect of temperature on Elovich plot for Acid Blue 92 Adsorption

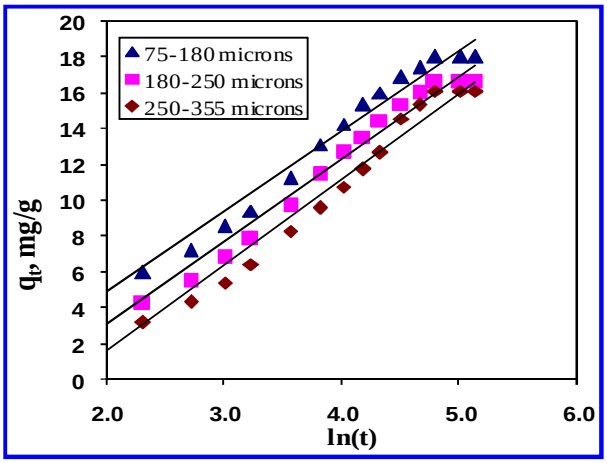

Fig. 3b: Effect of particle size variation Elovich plot for Acid Blue 92 adsorption

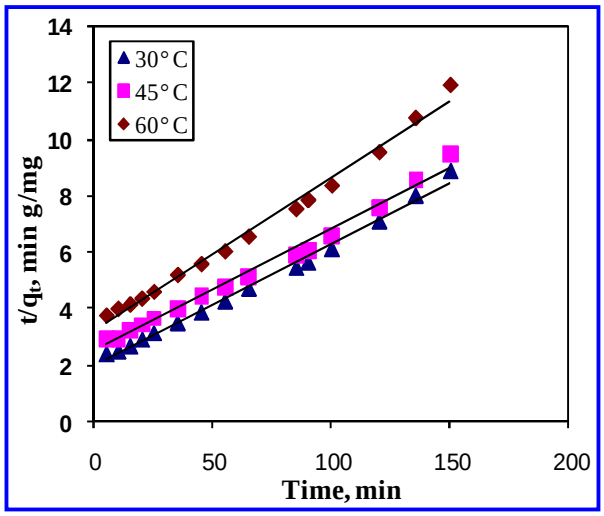

Fig. 2c: Effect of Temperature on Pseudo second order plot for Acid Blue 92 adsorption

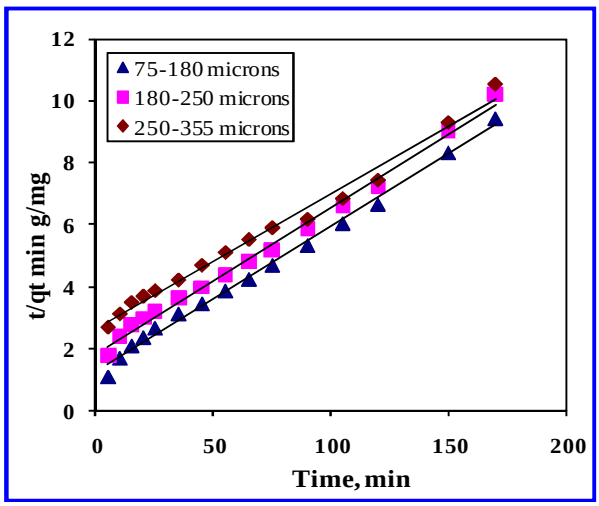

Fig. 3c: Effect of particle size variation on Pseudo second order plot for Acid Blue 92

Table 2. Rate parameters for the adsorption of Acid Blue 92 onto SQAC at various Temperatures

Temp. °C	Pseudo First Order		Elovich			Pseudo Second Order			
	k_L, min^{-1}	R^2	β, g/ min	α, mg/ g/ min	R^2	q_e, mg/g	k_2, g/ mg/ min	h, g/ mg/ min	R^2
30	0.0306	0.9823	0.194	1.147	0.9801	23.201	0.0933	1.991	0.9931
45	0.0294	0.9764	0.205	1.033	0.9681	23.094	0.0748	2.504	0.9891
60	0.0269	0.9909	0.260	1.311	0.9708	18.518	0.0907	3.212	0.9909

Table 3. Rate parameters for the adsorption of Acid Blue 92 onto SQAC at various Particle sizes

Particle size microns	Pseudo First Order		Elovich			Pseudo Second Order			
	k_L, min^{-1}	R^2	β, g/ min	α, mg/g/ min	R^2	q_e, mg/g	k_2, g/ mg / min	h, g/ mg/min	R^2
75 – 180	0.0285	0.9933	0.2231	1.831	0.9723	21.0970	1.2456	1.8037	0.9927
180 –250	0.0278	0.9923	0.2170	1.2293	0.9739	21.3219	1.7694	1.2431	0.9944
250 – 355	0.0232	0.9643	1.2293	1.5063	0.9666	23.0946	0.0705	2.6564	0.9880

Pseudo first order kinetic models, the rate constant values decreases with increase in particle size simultaneously. The amount of dyes adsorbed at equilibrium (q_e) for the Pseudo second order increases with increase in practical size as well as the initial adsorption rate (h) was 1.8037, 1.2431 & 2.6564 for all the three different particle size. For the Elovich kinetic modals the initial sorption rates (α) increases with increase in particle size. The desorption constant (β) was 0.2231, 0.2170 & 1.2293. The results of the sorption of Acid Blue 92 on to SQAC at various particle size, from the obtained data's the linear regression co-efficient values R^2 obtained for the Pseudo first order, Elovich and Pseudo second order, the relatively higher R^2 values obtained only in the Pseudo second order kinetic model. Hence the Pseudo second order fits good correlation (fig. 3a, 3b, 3c).

3.3 Adsorption kinetics

Isotherms are the equilibrium relation between the concentration of the adsorbate on the solid phase and in the liquid phase.

The adsorption isotherms of the Acid Blue 92 on activated carbon at three different temperatures of solutions are represented in (fig. 4a, 4b, 4c).

The experimental equilibrium adsorption data were analyzed using three adsorption isotherm models such as Langmuir, Freundlich and Tempkin models. The values in Table 2 show that the experimental data were more suitable to the Langmuir model than to the Freundlich and Tempkin models. This is in accordance with the correlation coefficients (R^2) as a goodness of fit criterion.

As seen from Table 2 the values Qo of maximum sorption capacity, corresponding to monolayer coverage of the binding sites available in the sorbent, was obtained at 30 °C. In the range 30 °C to 60 °C and increase in temperature does not affect significantly adsorption of Acid Blue 92 on SQAC. The lower value of Qo at 60 °C may be attributed to the large molecular size of dye which inhibits adsorption; the increase in temperature enhances the molecules dye diffusion in the complex porous structure of activated charcoal.

Table Constants of Langmuir, Freundlich and Tempkin adsorption isotherms of Acid Blue 92 with various temperatures

Temp. °C	Langmuir Isotherm			Freundlich Isotherm			Tempkin Isotherm	
	Q_0, mg/g	b, L/mg	R^2	1/n	K_f, mg/g	R^2	β_T	R^2
30	111.17	0.075	0.9935	0.4725	13.79	0.9696	20.16	0.9638
45	92.93	0.096	0.9930	0.4291	14.55	0.9492	16.50	0.9768
60	82.22	0.119	0.9941	0.3903	15.37	0.9385	14.22	0.9726

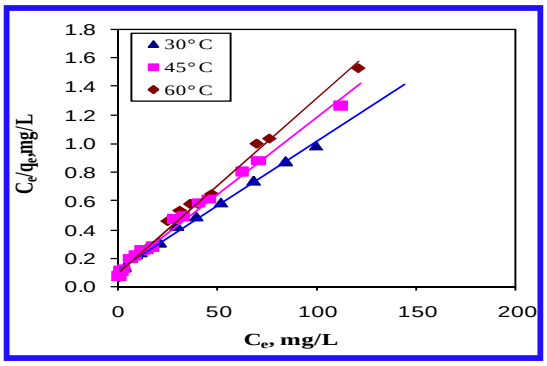

Fig. 4a: Effect of temperature variation on Langmuir plot for Acid Blue 92 adsorption

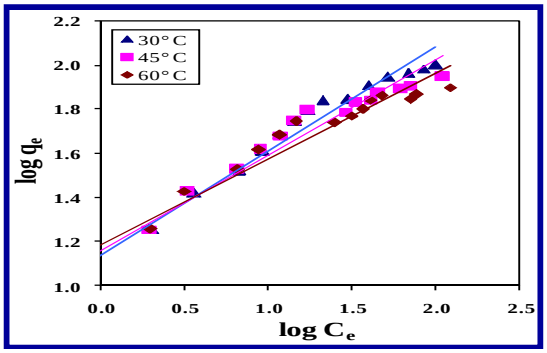

Fig. 4b: Effect of temperature variation on Freundlich plot for Acid Blue 92 adsorption

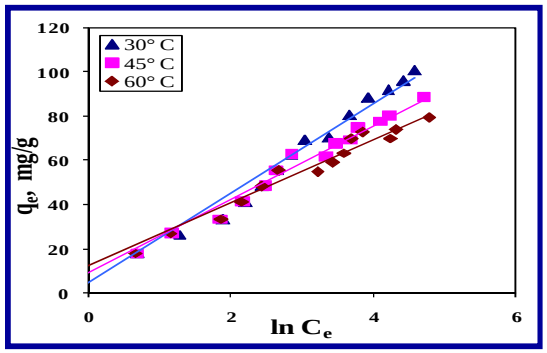

Fig. 4c: Effect of temperature variation on Temkin plot for Acid Blue 92 adsorption

The small values of Langmuir constant b L/mg (increased with the increasing temperature in the range 30 °C to 60 °C) may suggest a weaker binding between Acid Blue 92 and the carbon surface. The molecular properties of Acid Blue 92, which is a voluminous charged organic molecule consisting of polar and non-polar portions, call for a complex adsorption process. The dyes sulphonic acid groups are susceptible to electrostatic interactions with the adsorbent surface, whereas, non-polar portions play an important role in hydrophobic interactions (vander Waals bonds).

4. CONCLUSIONS

In the present study adsorption of Acid Blue 92 on activated *Sterculia Quadrifida* seed shell waste carbon have been investigated. The data obtained through supports that the *Sterculia Quadrifida* seed shell waste carbon is an effective low cost adsorbent for the removal of Acid Blue 92 from aqueous solution.

The Pseudo second order provides a best fit description for the adsorption of the Acid Blue 92 on to *Sterculia Quadrifida* relative to Elovich and Pseudo first order equations. The Pseudo second order was consider the most appropriate due to high correlation co-efficient when compared to Pseudo first order and Elovich equations. The experimental equilibrium adsorption data were analyzed using three adsorption isotherm models such as Langmuir, Freundlich and Tempkin models. The experimental data were more suitable to the Langmuir model than to the Freundlich and Tempkin models. This is in accordance with the correlation coefficients (R^2) as a goodness of fit criterion.

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