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Batch Adsorption Studies on Treatment of Textile Industry Effluent using Bamboo and Green Coconut shell Activated Carbon

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Abstract. The use of activated carbon (AC) as a strong adsorbent was commonly used. This work deals with the study of textile industry wastewater (dye) adsorption on AC produced from Bamboo and Green coconut shell through chemical activation method. The effect of various process parameters such as the amount of adsorbents, initial concentration of dye, pH, contact time, temperature and agitation speed on percentage removal of dye on the activated carbon i.e. Bamboo activated carbon (BAC) and Green Coconut Shell Activated carbon (GCSAC) batch adsorption tests were investigated. Testing was performed for quantitative description of Langmuir and Freundlich equilibrium adsorption isotherms. Based on the experimental findings, the equilibrium was observed in 6 hours. Langmuir isotherm was the best suited for the adsorption of dye, with a maximal adsorption capacity of 142.86 mg / gm. The removal of dye by the use of BAC has been found to be greater than GCSAC.

1. Introduction

Owing to the widespread and growing use of a variety of dyes, contamination from waste water has become a serious environmental threat in recent years. In many industries including textiles, rubber, plastics, printing, leather, cosmetics and others, dyes are commonly used in the manufacture of coloured goods as well. It is estimated that approximately 3% of these colours, which are produced from most of textiles, are dumped into the water system [1]. The ecological structure of the receiving surface water potential is impaired by the waste water, and so the groundwater supplies are definitely disrupted. The majority of dyes used are stable to light and are not biodegradable in the textile industry.

It is important to process it before discharge in the atmosphere to reduce the risk of contamination from this waste. In order to remediate wastewater dyes, various processes have been investigated including photo-catalytic degradation using UV / TiO_2, sound degradation, biologic Fenton treatment, biodegradation, chemical biological integration process, electrochemical process, adsorption process, degradation process with chemical coagulation-flocculation, etc. It has proven to be the most efficacious among the different techniques for its remediation adsorption technique [2-3]. Adsorption is a process of unit activity which refers to the fixation of molecules to the surface. This is because certain solids preferably adsorb another solution on their surfaces from the solution. Especially with different adsorbents, dyes are removed [4].

A variety of these adsorbents were investigated for removal, including the orange peel, the neem seed, the olive cakes, the date palm, the olive shell, the coal etc. In comparison to other adsorbents, however, activated carbon had important removal properties and it is a flexible, high porosity, high

surface adsorbent. Selectively the activated charcoal has been used for removing dye, commonly used in the textile field, among different forms of dyes extracted [5]. The purpose of this study is therefore to evaluate the impact of the process parameters such as the amount of adsorbents (0.5, 1, 1.5, 2, 2.5 g/L), initial concentration of dye (200, 400, 600, 800 mg/L), pH (3, 5, 7, 9, 11), contact time (1, 2, 3, 4, 5, 6, 7, 8 Hrs.), Agitation speeds (100, 200, 300 rpm) and temperature (30, 40, 50 ^{0}C) on removal capacity of dye. However, compared with other conventional adsorbents the adsorption activity of BAC for removal of dye, water has been less reported.

2. Experimental

2.1. Preparation of adsorbents:

The materials necessary for the preparation of the AC (bamboo and green coconut shell) were collected in the local region. To extract soil, dried and crushed seving through a 60-mesh sieve, bamboo and green coconut shell have been washed. The chemical activation agent phosphoric acid (H_3PO_4) was used to generate AC. All Chemicals were used analytical (AR) grade and the glassware was washed with distilled water. The carbonization of the materials was done at 200ºC for two hours and allowed to cool at room temperature. A 250 g sample of crushed bamboo and green coconut shell was combined with a fixed volume of H_3PO_4. A bamboo/H_3PO_4 and a green coconut shell/H_3PO_4 mixture were used and kept in the stainless steel container. The container was put in a muffling oven at a temperature of 600^{0}C, reached progressively within 3 hours and was held for 45 minutes. BAC and GCSAC were washed after cooling down until pH 7. In an oven at 100 ° C, the samples were then dried overnight to minimize any moisture content. In an airtight bottle the prepared BAC and GCSAC were stored. Figure 1 shows the flow of processing of raw materials to AC.

Figure 1. Flow of processing of raw materials to AC

2.2. Characterization of adsorbents:

BAC and GCSAC are the two adsorbents used in the present investigation. Adsorbents used for their characterization, such as bulk density, particle size, surface area and proximate analysis, provide physico-chemical characteristics. Physico-chemical characteristics of the adsorbents are presented in Table-1, BAC bulk density is noted to be lower than GCSAC.

Table 1. Characteristics of the BAC and GCSAC

Sr.No	Properties	Adsorbent Types	
		BAC	GCSAC
	Typical Properties		
1	Specific surface area ($m^2\,g^{-1}$)	316.20	291.80
2	Total pore volume ($cm^3\,g^{-1}$)	0.27	0.31
3	Average pore diameter (A)	34.95	42.72
4	Bulk density ($g\,cm^{-3}$)	0.60	0.71
	Proximate analysis (wt. %)		
1	Moisture	9.39	10.70
2	Volatile matter	25.57	26.77
3	Fixed carbon	63.37	60.77
4	Ash content	1.67	2.09
	Ultimate analysis		
1	C	61.74	58.74
2	H	1.37	2.24
3	N	5.63	3.07
4	S	9.33	9.99
5	O (by difference)	21.96	21.96

2.3. Experimental Procedure:

2.3.1. Batch adsorption studies:
The tests were performed for the adsorption of the dye using the BAC and GCSAC as adsorbent. Adsorption studies are performed through observation of different important parameters such as namely, amount of adsorbents (0.5, 1, 1.5, 2, 2.5 g/L), initial concentration of dye (200, 400, 600, 800 mg/L), pH (3, 5, 7, 9, 11), contact time (1, 2, 3, 4, 5, 6, 7, 8 Hrs.), Agitation speeds (100, 200, 300 rpm) and temperature (30, 40, 50 ^{0}C). The experimental adsorption studies were conducted using a technique of a completely mixed reactor batch (CMBR). At 200 rpm and 30^{0} C, the flasks were agitated in an isothermal shaker to achieve a balance. The time was observed for equilibrium of 5 hours.

The experimental data were therefore calculated up to 8 hours to ensure maximum equilibrium was achieved. For two other flasks containing the same initial dye and the same activated carbon dose were accompanied by identical procedures; however the thermodynamic experiments were studied from 40 to 50^{0}C. The samples were centrifuged for 10 minutes at 1500 rpm in order to avoid interferences from being caused due to carbon fine material and the left-hand concentration in the overnatant solution was analyzed using the UV visible spectrophotometer by monitoring the changes in absorption at a maximum wavelength The dyes were taken from each dyes dye solution at a predetermined time interval [6]. Using the corresponding UV-visible spectrophotometers, dye concentrations in the sample solutions were determined before and after adsorption. It was measured using the normal calibration curve to the dye concentration [7]. In equivalent conditions each test was duplicated and mean values for additional calculations were used. The adsorption value at equilibrium qe (mg / g) was determined accordingly:

$$q_e = \frac{(C_0 - C_e)V}{X} \qquad (1)$$

Where, qe = Adsorbed dye per unit adsorbent mass (mg/g), C_0 = Initial concentration of dye (mg/L), C_e = Final concentration of dye (mg/L), V = Volume of Solution (ml), X = Dose of dry adsorbent used (gm). For the adsorption experiments, fixed amount of adsorbents Bamboo Activated carbon (BAC) and Green coconut shell Activated carbon (GCSAC) was added to 250 mL of dye in each run at definite pH. In a fixed time interval the suspension was adsorbed. A sample was taken using a pipette at various intervals.

3. Result and Discussion

3.1. Influence of operating parameters
The evaluation in the design of all adsorption systems is of vital importance for various operational parameters such as adsorbent dosage, dye, contact time, temperature, pH, agitation speed, etc . This effect was investigated for the removal by BAC and GCSAC adsorbents of various dyes.

3.1.1. Effect of Adsorbent dose
A significant parameter is the adsorbent dose since this defines the adsorbent potential for the initial adsorbate stage. Effect of BAC and GCSAC on dye removal by keeping some further experimental condition consistent at an initial dyes concentration of 200 mg/L was examined in adsorbent doses. Figures 2 and 3 demonstrate the effect of the adsorbent dose on dye removal. The results showed that the removal by percentage has increased to a 2 g/L adsorbent dose and remains virtually constant from Figures 2 and 3. This could result from an improvement in the adsorption dose that increases the adsorption region, which enhances the availability of more sites for adsorption. At low concentrations, equilibrium was found to be reached more easily. Therefore, the adsorbent BAC and GCSAC adsorption dose was chosen 2 g/L for all the adsorption experiments
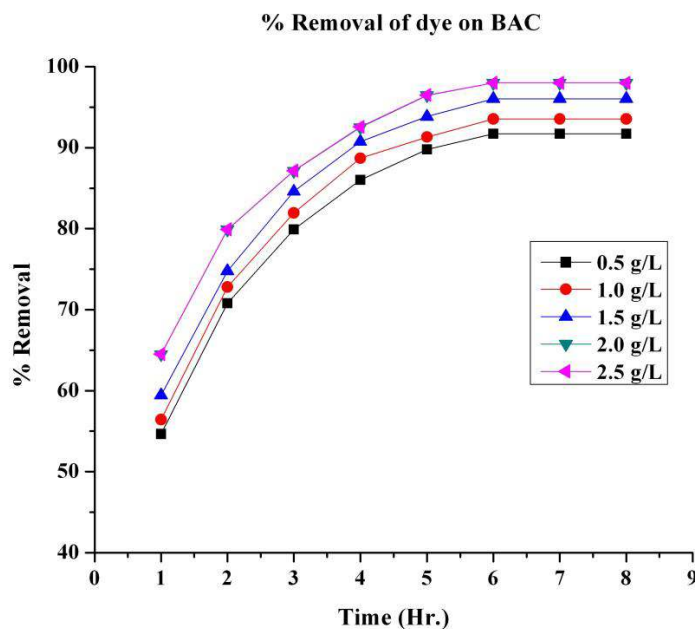

**Figure 2.** Adsorbent (BAC) dose effect on dye removal
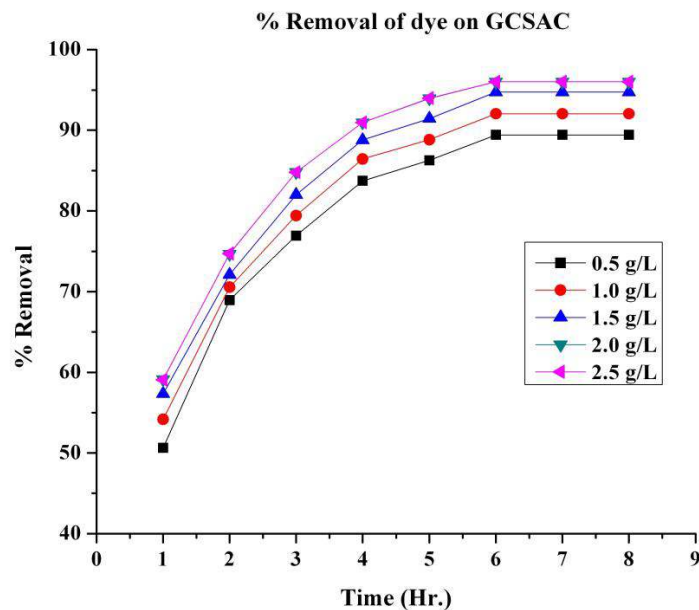
Figure 3. Adsorbent (GCSAC) dose effect on dye removal

3.2.2. Effect of initial concentrations of dye and contact time

At different initial dye levels (200, 400, 600, 800 mg/L) at different time intervals, the experimentation was carried out at 30^0C, with agitation speed at the pH 7 and at a fixed adsorbent dose (2 g/L). The dye removal in various concentrations on BAC and GCSAC is shown in Figure 4 and 5 respectively. The efficiency of the waste water treatment adsorbent is indicated by rapid absorption of contaminants and the establishment of equilibrium in a short time. Even the initial adsorbent or pollutant concentration has an important function since the adsorbent mass will adsorb a fixed amount of solvent only. The further solution or effluent is concentrated, the less effluent amount that can be purified by a specific mass of adsorbent [8]. The fractional adsorption is low in high concentration levels.
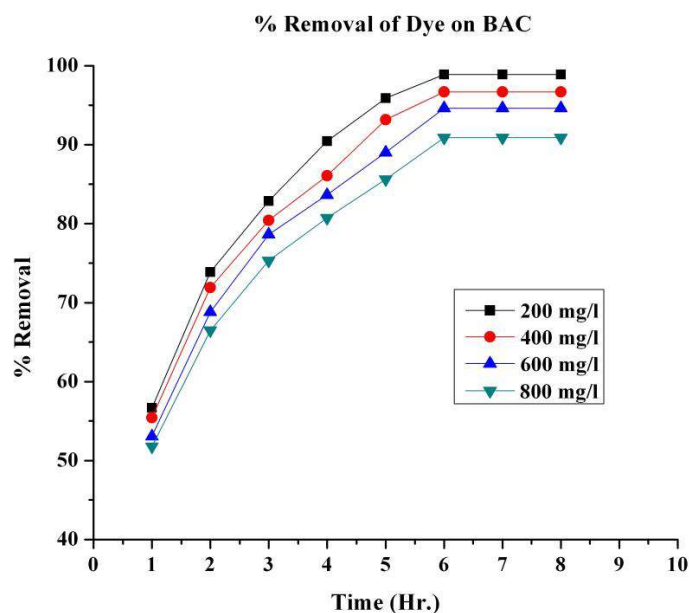
Figure 4. Initial concentration effect and time of contact with dye on BAC for percentage removal
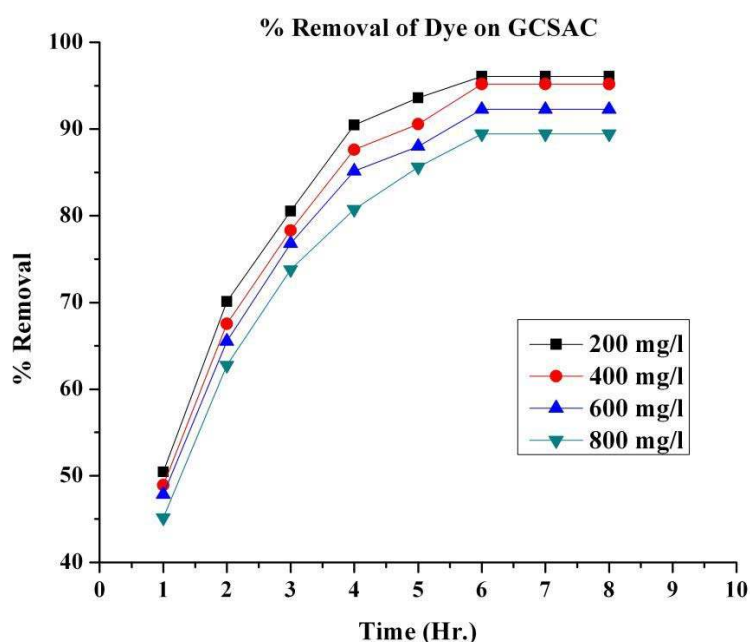
Figure 5. Initial concentration effect and time of contact with dye on GCSAC for percentage removal

   It has been observed from Figures 3 and 4 that the removal of the dye is rapid for the first 04 hours and is slower and eventually saturated. A fast adsorption on the outside, followed by slower adsorption on the pores may explain this. Due to the fact that at lower concentrations almost all the colouring molecules were easily assorbed to the outer surface, but further increase in the initial dye concentration, since the initial dye concentration increases from 200 up to 800 mg/L, dye balance was declining from 98.90 to 90.90 %, and dye removal decrease from 96.07 to 89.44 %. The time of equilibrium was found to be independent of the initial concentration of dye. However, it takes 6 hours to reach equilibrium. To ensure complete equilibrium, experimental data were calculated up to 8 hours. The findings show that the removal percentage increases with an initial concentration reduction. However, with an increase in the initial dye concentration in all situations, dye uptake per adsorption unit weight was found to increase.

3.1.3. Effect of pH
Dyes-containing wastewater will be discharged at a different pH; therefore the effect of pH on dye degradation is necessary to analyse. In the whole of the adsorption process and especially in terms of adsorption capability, the pH of the dye solution affects the surface load of the adsorbent, the level of ionisation of the material in the solution and the dissociation of the working groups on the adsorption's active site [9]. It is well known that, due to the presence of H+ ions, the adsorbent is well adsorbed by the anion to lower pH values. Cations are adsorbed at high pH values because of negative adsorption surfaces. Experiments were performed at different pH values ranging from 3, 5, 7, 9 and 11 in order to examine the influence of pH in the removal percentage. The analysis consisted of an agitation by using a water bath shaker at 30^0 C of 250 ml of a dye solution of 50 mg /l with various pH values (3-11). At 200 rpm, 8 hours were disturbed which was more than enough time for equilibrium. The pH was calibrated using the pH metre and with the solutions 0.1 N N NaOH and 0.1 N HCl. The percentage dye removal in pH function is shown in Figures 6 and 7. At higher pH, dye removal is favourable. With pH rising from 3 to 5, the removal of dye is negligible. The removal of the dye increases significantly above pH 5. The maximum removal of dye was observed to be at pH 11. However there is sharp increase in dye uptake for dye from pH 7 to 9 and then after there is little increase in dye uptake as the pH is further increased from 9 to 11. Therefore the best pH range for

adsorption of dye was from 7 to 11. There were also findings showing that dye adsorbent effects on BAC and GCSAC of contact time and pH were studied. No effect of pH on equilibrium time, which remained around 6 hrs, was observed.
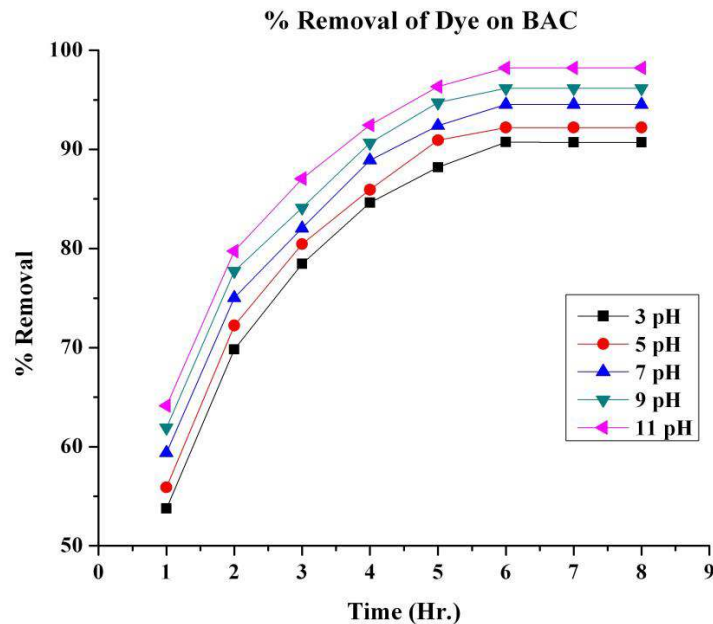

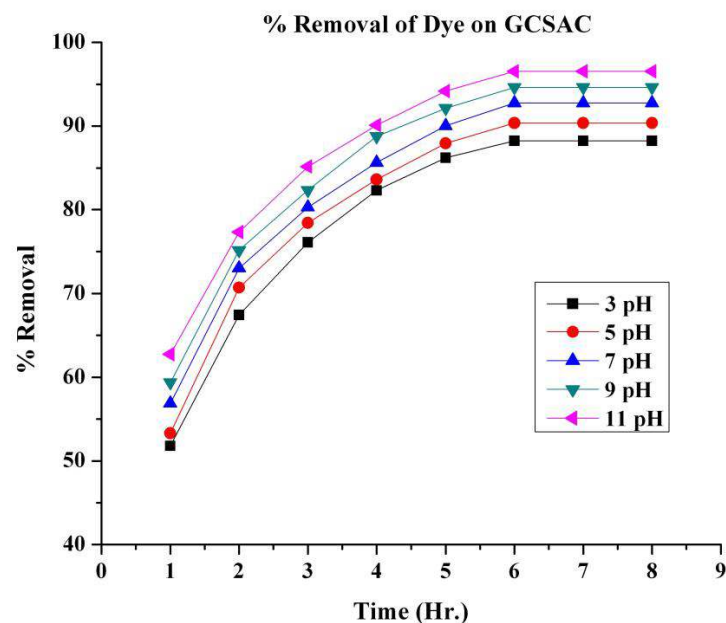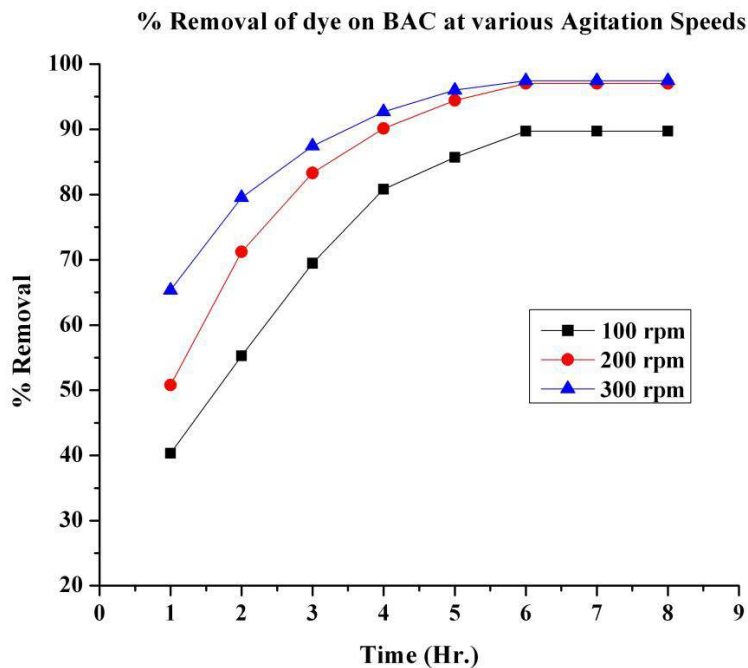**Figure 6**. pH effect and contact time on BAC for percentage removal

Figure 7. pH effect and contact time on GCSAC for percentage removal
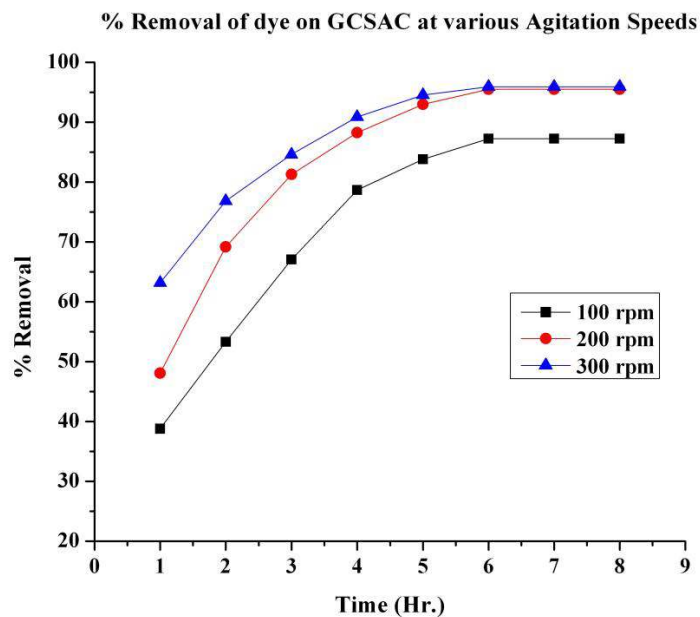
3.1.4. Effect of Agitation speed
Agitation is an useful characteristic for the delivery of solutions in bulk solutions and for the creation of external border films during sorption [10,11]. A broad range of agitation speeds (100, 200, 300 rpm) has been tested in order to ensure that concentration, pH and temperature, along with other

parameters, are constant during the process. Figures 8 and 9 display graphically the findings obtained. The equilibrium dye in solid phase was influenced by low turbulent speed from the Figures-8 and 9. These findings suggest that adsorption of dye increases from 89.75 to 97.45% for BAC adsorption, while dyes adsorption of GCSAC increases from 87.24 to 95.95% with rotational speed increases from 100 to 300 rpm. Even so, the absorption of dye is not significantly hampered by increasing the agitation speed from 200 to 300 rpm. In relation to a low speed agitation, however, higher speed equilibrium was achieved.


**Figure 8.** Agitation speed effect and contact time on dye removal on BAC

Figure 9. Agitation speed effect and contact time on dye removal on GCSAC

3.1.5. Effect of Temperature

The study of influence of temperature on the rate of adsorption is helpful in the determination of increase or decrease in the extent of adsorption and in computing the corresponding activation energies. Several researchers documented adsorption decreases with temperature increases [12,13]. Adsorption experiments were performed in various temperatures (30^0C, 40^0C and 50^0C) at the initial 200 mg/L dye concentrations at pH 7 and 200 rpm.
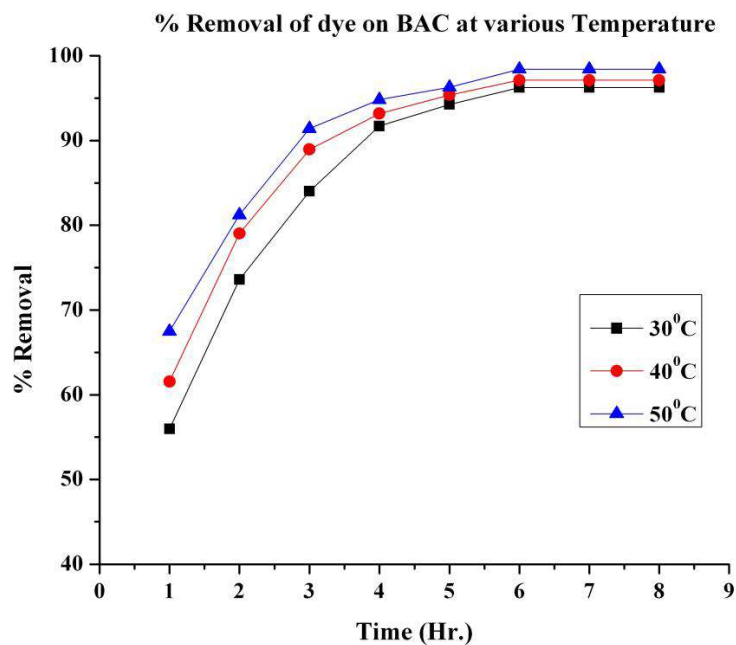
**Figure 10.** Temperature effect and contact time on BAC dye removal
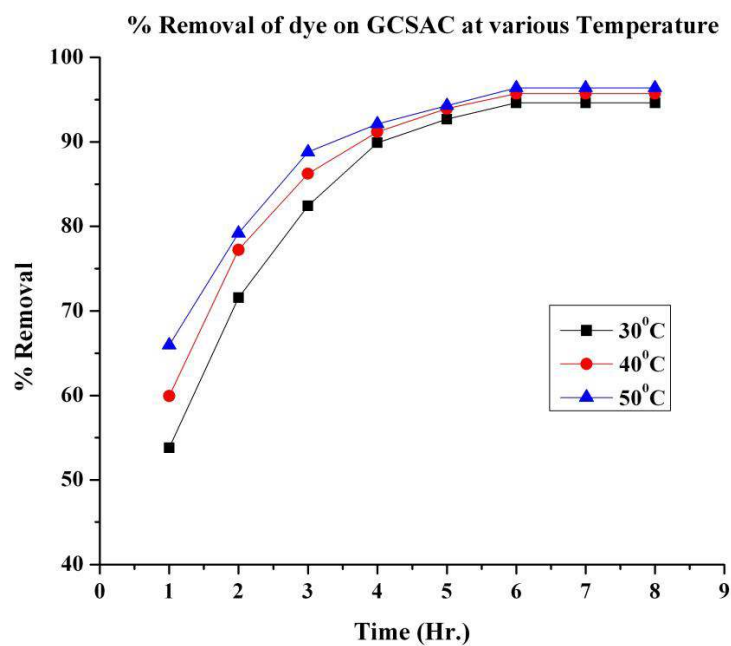
Figure 11. Temperature effect and contact time on GCSAC dye removal

Figures 10 and 11 demonstrate graphically the outcomes of dye absorption experiments with various adsorbents (BAC and GCSAC) at different temperatures (30^0C, 40^0C and 50^0C) and it shows that the BAC and GCSAC adsorbent on removal of dyes is increasing as temperature is rising. The percentage removal of by BAC adsorbent increases from 96.25 to 98.44 % where as it increases from 94.64 to 96.39 % in case of GCSAC as adsorbent with the rise of temperature from 30 to 50^0C at 200 mg/l. There is negligible uptake of dye with increase in temperature from 30 to 50^0C in view of this all the experiments in batch mode were carried out at 30^0C. From these figures, it is apparent that the adsorbed species take up relatively quickly at the initial stages of the adsorption process, which is slow in the later stage and ultimately saturated [14]. Furthermore, these curves are smooth, continuous and saturation-focused.

3.2. Isotherm results:

3.2.1. Langmuir Isotherm results:
Equilibrium data were analysed with Langmuir's model to absorb dye onto BAC and GCSAC. The product with unique adsorption (C_e / q_e) in relation to the equilibrium concentration (C_e) has a linear relationship shown in Figures-12-(a, b , c) and 13-(a, b , c) with correlation factors (R^2) > 0.99 which confirm that the Langmuir model has the same adsorption energy as BAC and GCSAC adsorbents. Form the graph, the slope was found which gives us the value of $1/(q_{max}K_L)$. The equation is y = mx + c. The y-axis intercept gives the $1/q_{max}$= m. Maximum q_m monolayer dye capacity obtained with Langmuir model for dyeing BAC adsorbent is 142.86 mg / gm and 125 mg / gm dye with GCSAC adsorbent at 300^C. Table 2 and 3, represent the Langmuir Models Isotherm constants for adsorption of dye on BAC and GCSAC.
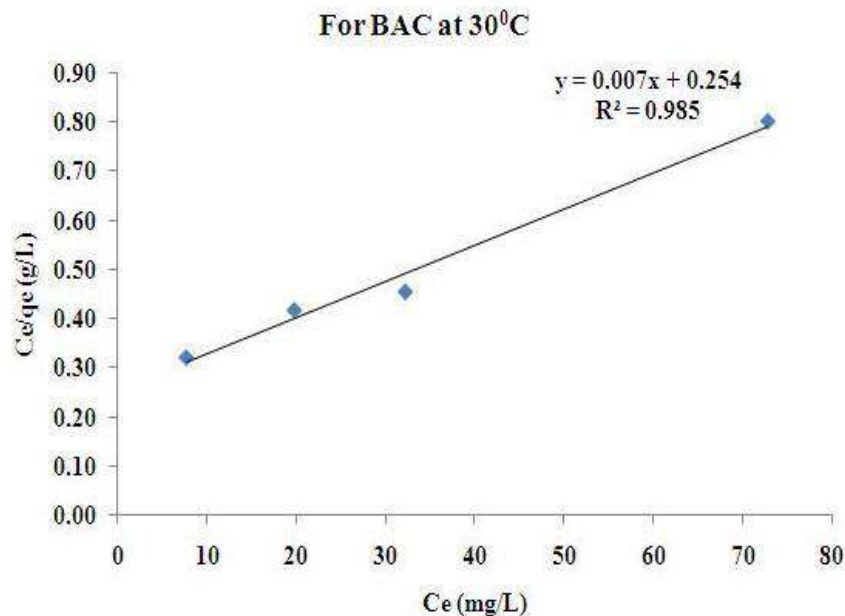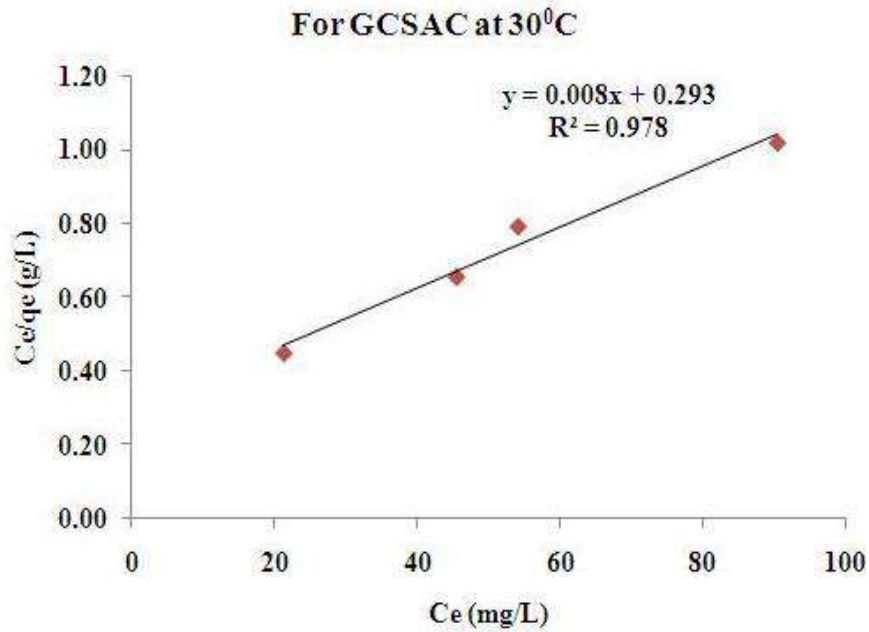

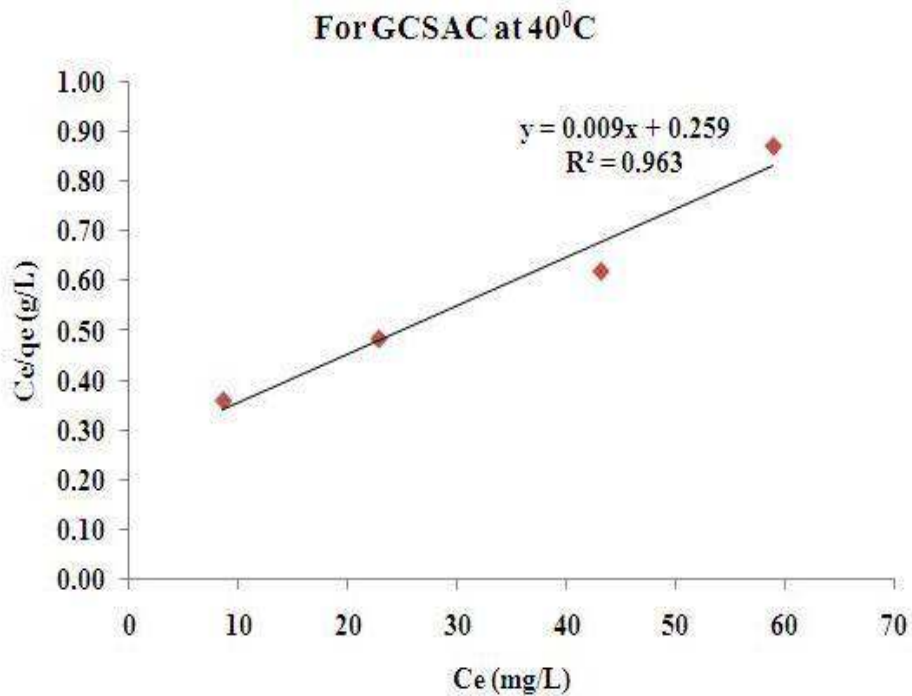**Figure 12 (a).** Langmuir Isotherm plot for dye on BAC at 30^0C
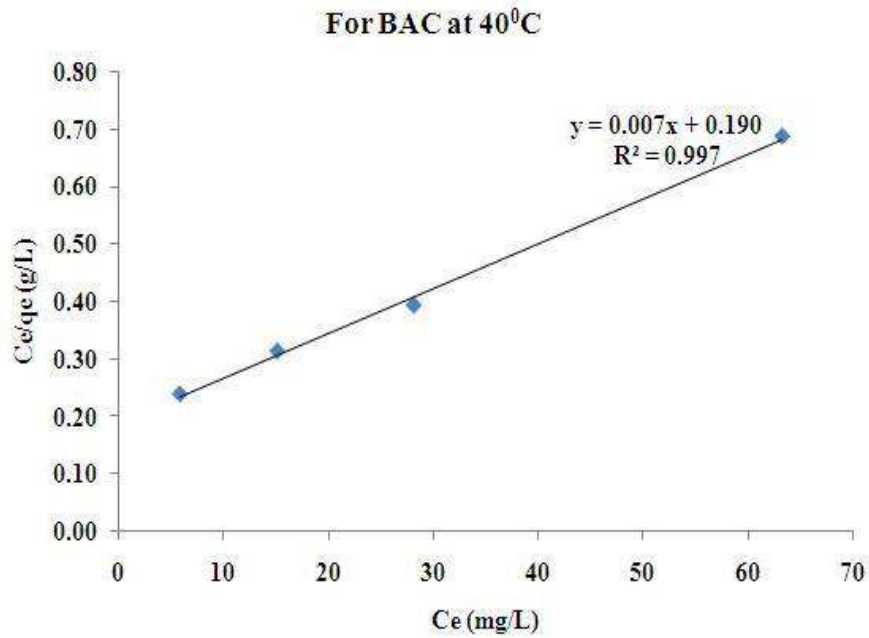
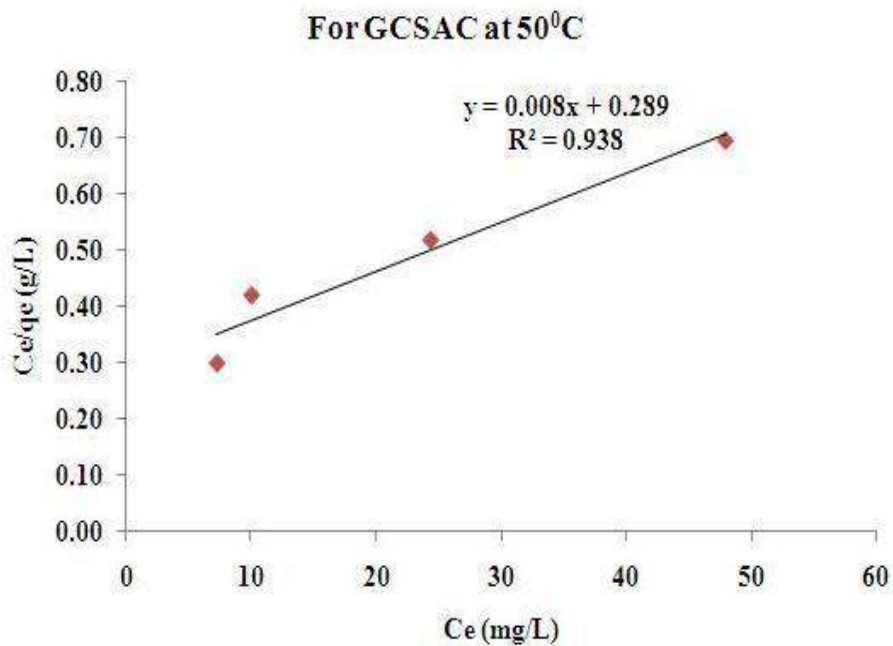**Figure 12 (b).** Langmuir Isotherm plot for dye on BAC at 40^0C\
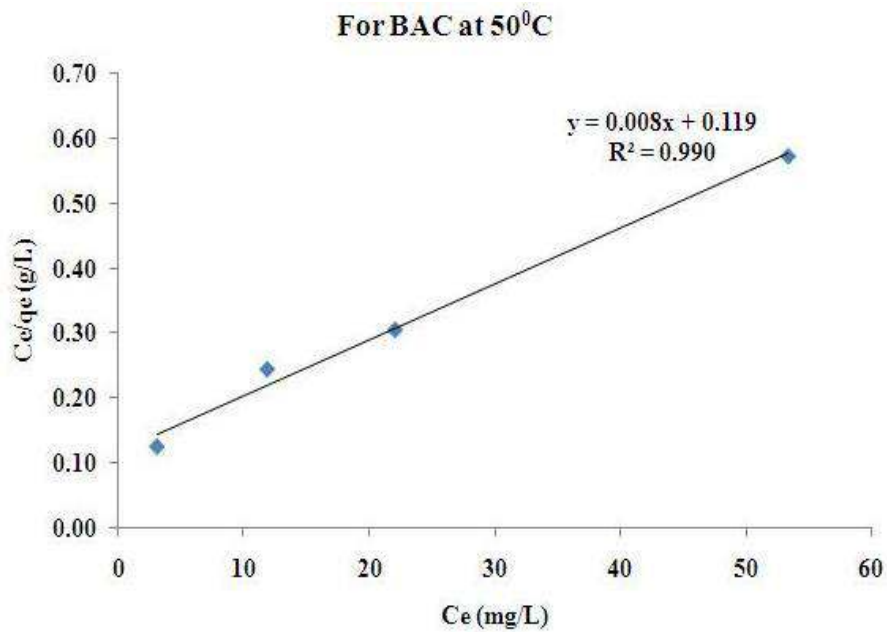
Figure 12 (c). Langmuir Isotherm plot for dye on BAC at 50^0C

Figure 13 (a). Langmuir Isotherm plot for dye on GCSAC at 30^0C

Figure 13 (b). Langmuir Isotherm plot for dye on GCSAC at 40^0C

Figure 13 (c). Langmuir Isotherm plot for dye on GCSAC at 50^0C

Table 2: Langmuir Models Isotherm constants for adsorption of dye on BAC

| Parameters for the Langmuir adsorption isotherm onto BAC		
Temperature (^{0}C)	Constants	Magnitude
	K_L, [L/mg]	0.024
	q_{max} [mg/g]	142.86
	R^2	0.985
	K_L, [L/mg]	0.029
	q_{max} [mg/g]	142.86
	R^2	0.997
	K_L, [L/mg]	0.055
	q_{max} [mg/g]	125
	R^2	0.99

Table 3. Langmuir Models Isotherm constants for adsorption of dye on GCSAC

Parameters for the Langmuir adsorption isotherm onto GCSAC		
Temperature (^{0}C)	Constants	Magnitude
	K_L, [L/mg]	0.027
	q_{max} [mg/g]	125
	R^2	0.978
	K_L, [L/mg]	0.026

q_{max} [mg/g]	111.11
R^2	0.963
$K_{L,}$ [L/mg]	0.026
q_{max} [mg/g]	125
R^2	0.938

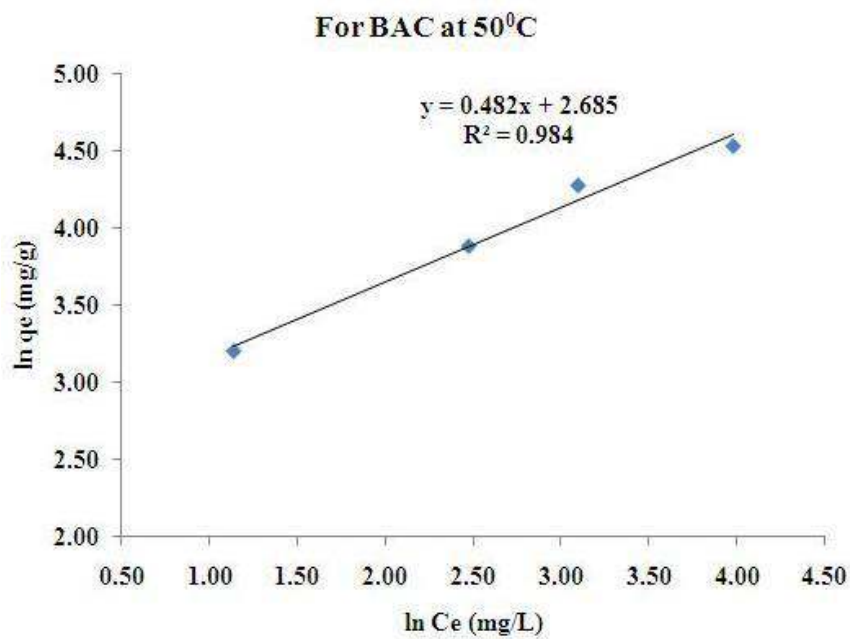### 3.2.2. Freundlich Isotherm results

The data on equilibrium for the dye adsorption to the BAC and GCSAC have been evaluated with the Freundlich equation. Figures-14-(a,b,c) and 15-(a,b,c) represents the plot of ln q_e versus ln C_e, This line is straight, with an interception value of ln K_F and 1 n slop, presented in Tables 4 and 5. R^2 obtained from the Freundlich model shows that the experimental data can fit into the Freundlich model and n >1 suggests a favorable adsorption mechanism for dye on BAC and GCSAC. On the basis of the R^2 value, however, the Freundlich isotherm seems less successful than Langmuir isotherm models.
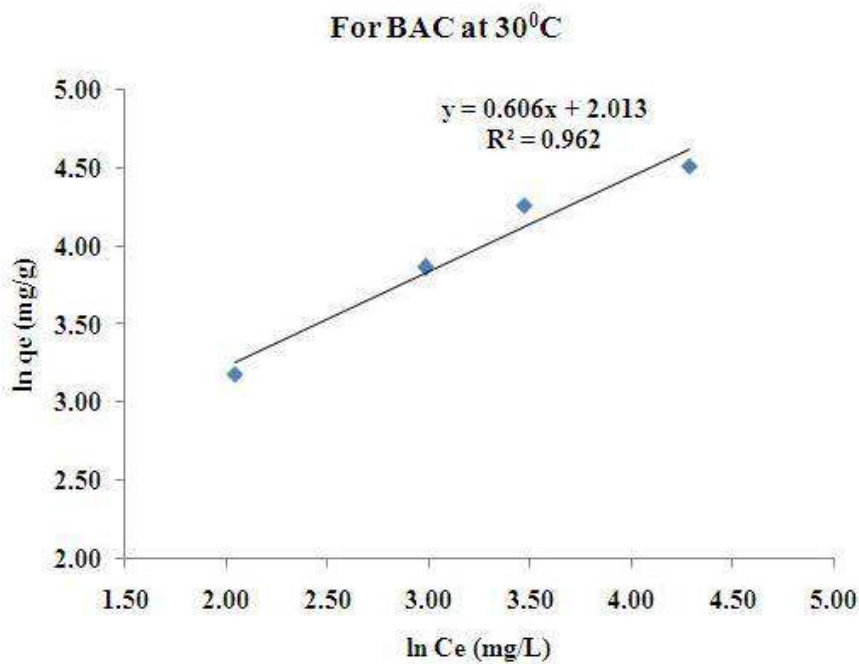
Figure 14 (a). Freundlich Isotherm plot for dye on BAC at 30^0C

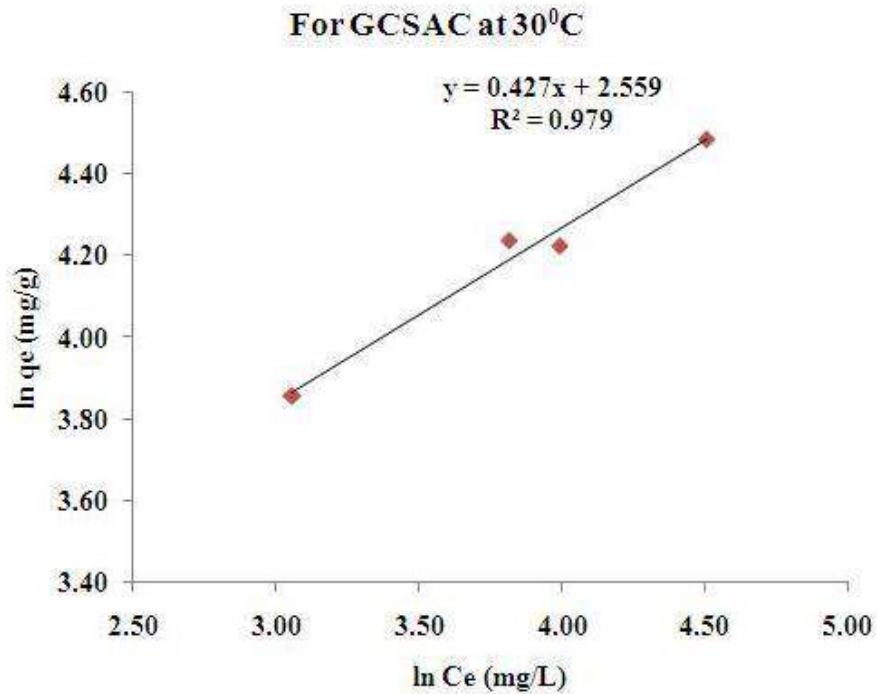**For BAC at 40⁰C**

$y = 0.567x + 2.267$
$R^2 = 0.968$
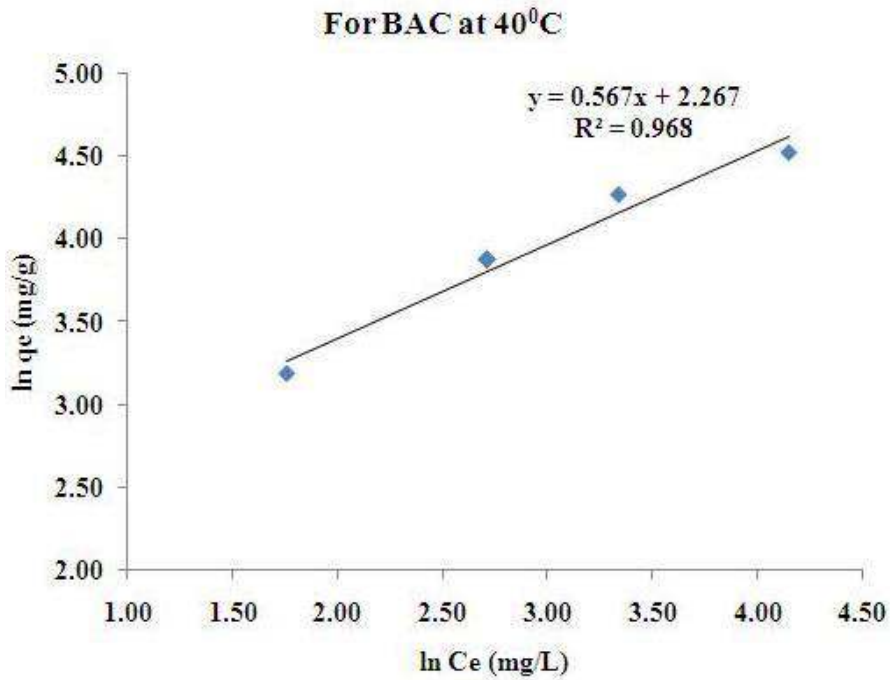
Figure 14 (b). Freundlich Isotherm plot for dye on BAC at 40^{0}C

For BAC at 50⁰C

$y = 0.482x + 2.685$
$R^2 = 0.984$

Figure 14 (c). Freundlich Isotherm plot for dye on BAC at 50^{0}C

For GCSAC at 30⁰C

$y = 0.427x + 2.559$
$R^2 = 0.979$

Figure 15 (a). Freundlich Isotherm plot for dye on GCSAC at 30^0C

For GCSAC at 40⁰C

$y = 0.573x + 1.991$
$R^2 = 0.960$
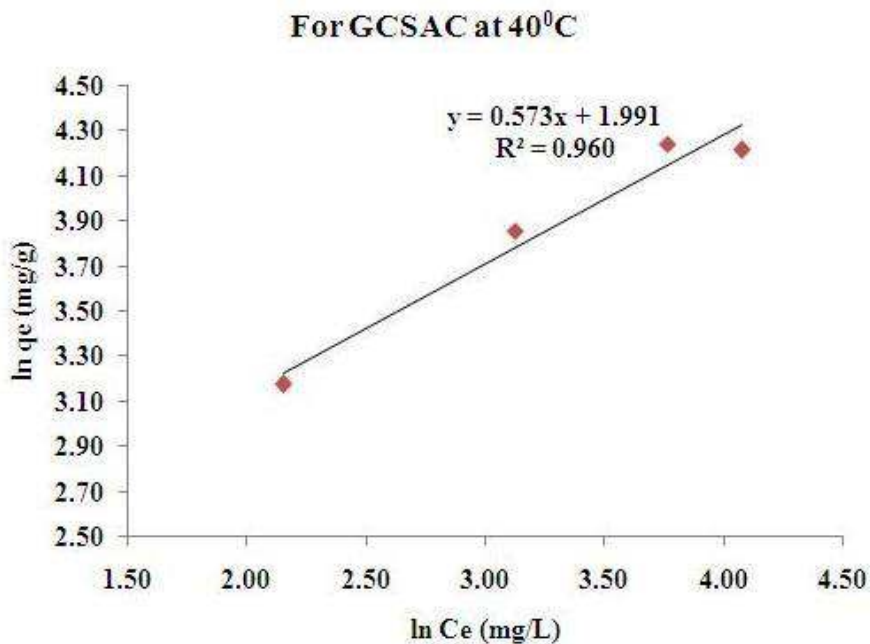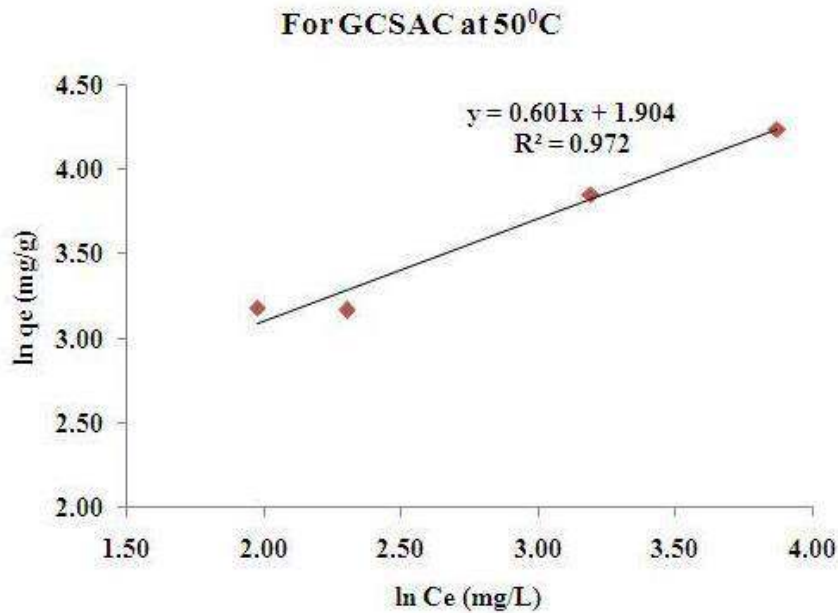
Figure 15 (b). Freundlich Isotherm plot for dye on GCSAC at 40^0C

Figure 15 (c). Freundlich Isotherm plot for dye on GCSAC at 50^0C

Table 4. Freundlich Model Isotherm constants for adsorption of dye on BAC

Parameters for the Freundlich adsorption isotherm onto BAC		
Temperature (^{0}C)	Constants	Magnitude
	K_F, [mg g-1] [mg^3 / g]$^{1/n}$	6.76
	n	1.65
	R^2	0.96
	K_F, [mg g-1] [mg^3 / g]$^{1/n}$	8.77
	n	1.76
	R^2	0.97
	K_F, [mg g-1] [mg^3 / g]$^{1/n}$	13.72
	n	2.07
	R^2	0.98

Table 5. Freundlich Model Isotherm constants for adsorption of dye on GCSAC

Parameters for the Freundlich adsorption isotherm onto GCSAC		
Temperature (^{0}C)	Constants	Magnitude
	K_F, [mg g-1] [mg^3 / g]$^{1/n}$	12.96
	n	2.34
	R^2	0.98
	K_F, [mg g-1] [mg^3 / g]$^{1/n}$	6.54

n	1.75
R^2	0.96
K_F, [mg g-1] [mg^3 / g]$^{1/n}$	6.74
n	1.66
R^2	0.97

4. Conclusion

The use of BAC as an adsorber is clearly shown to be much more effective than GCSAC in this research work. That can be used effectively to absorb dye from the aqueous solution at 98.90%. During the investigation process, the various parameters observed indicate that the total adsorption process is significantly influenced by the amount of adsorbents, initial concentration of coloration, pH , contact time, agitation speeds and temperature. Test results were adapted for Langmuir as well as Freundlich isotherms. Langmuir is stronger than the Freundlich Isotherm with a maximum adsorption potential of 142.86 mg/gm, demonstrated by adsorption constant and regression factor.

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