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ORIGINAL ARTICLE

Studies on the Removal of Methylene Blue Dye Using Activated *Hibiscus sabdariffa* Stem Nano Carbon

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ABSTRACT
The research of the present work was to investigate the removal of methylene blue dyes from aqueous solution by using Activated Hibiscus Sabdariffa Stem Nano Carbon (AHSNC). Generally, dyes are organic compounds used as colouring products in chemical, textile, paper, printing, leather, plastics and various food industries. The need for the treatment of dye contaminated the waste water passed out from the industry. In this study, Hibiscus Sabdariffa Stem was studied for its potential use as an adsorbent for removal of a cationic dye methylene blue. The various factors affecting adsorption, such as initial dye concentration, contact time, adsorbent dose and effect of temperature, were evaluated. The experimental data were fitted into the pseudo-second order kinetic model. The equilibrium of adsorption was modeled by using the Langmuir and Freundlich isotherm models. The objective of the present work suggests the AHSNC may be utilized as a low cost adsorbent for methylene blue dye removal from aqueous solution.
Key words: *Activated Hibiscus Sabdariffa Stem Nano Carbon (AHSNC); Methylene blue; Adsorption isotherm; Kinetics; Equilibrium models.*

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INTRODUCTION

Dyes are widely used, generally in the textiles, plastics, paper, leather, food industry to color products. In process of washing and finishing coloured products, waste water contaminated with dyes is generated. The contaminated waste waters are hazardous, which is a great threat to environment [1-3]. Dye contamination in wastewater causes problems in various ways: the presence of dyes in water, even in very low quantities, is highly visible and undesirable; color interferes with penetration of sunlight into waters; retards photosynthesis; inhibits the growth of aquatic biota and interferes with gas solubility in water bodies. These materials are the complicated organic compounds and they resist against light, washing and microbial invasions [4-7]. The need for the treatment of dye contaminated waste water arose from the environmental impact [8]. Activated minerals are one of the most popular adsorbents used for the removal of toxic substances from waste water. This could be related to their extended surface area [9]. The major use of Activated Hibiscus Sabdariffa Stem Nano Carbon is in solution purification and for the removal of colour, odors and other unpleasant impurities from liquids, water supplies and vegetable and animal oils.

In recent years it has been increasingly used for the prevention of environmental pollution and antipollution laws have increased the sales of low-cost activated minerals for control the of air and water pollution. Various techniques like precipitation, ion exchange, chemical oxidation and adsorption have been used for the removal of toxic pollutant from, wastewater. Methylene blue (MB) is selected as a model compound for evaluating the potential of AHSNC to remove dye from aqueous solution.

MATERIALS AND METHODS

Adsorption studies

Methylene blue (MB) was employed as an adsorbate in the adsorption experiments. Adsorption from the liquid phase was carried out to verify the nature the porosity and the capacities of the samples. An

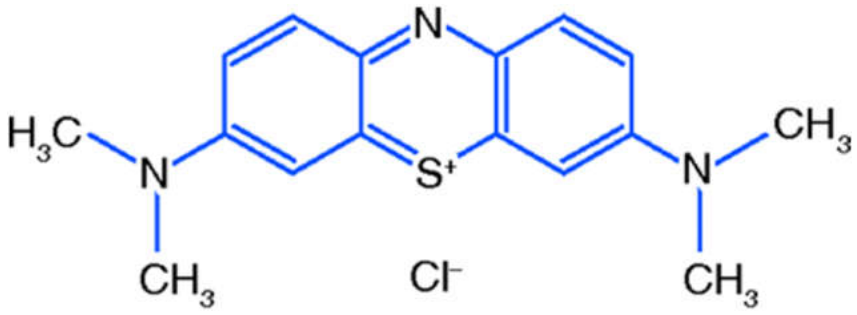
aqueous solution with a concentration of 50-250 mg/L was prepared by mixing an appropriate amount of MB with distilled water adsorption experiments were conducted by placing 0.025 g of the AHSNC samples and 50 ml of the aqueous solution in a 250 ml of glass-stoppered flask. The flask was then put in a constant-temperature shaker bath with a shaker speed of 150 rpm. The isothermal adsorption experiments were performed at 30 ±2°C.

Preparation of adsorbent materials

The Hibiscus Sabdariffa Stem collected from agricultural area nearby Thiruvarur districts was carbonized with concentrated Sulphuric acid and washed with water and activated around 1100°C in a muffle furnace for 5 hrs the it was taken out, ground well to fine powder and stored in a vacuum desiccators.

Preparation of adsorbate

Methylene blue was chosen in this work because of its strong adsorption onto solids and it recognized usefulness in characterizing adsorptive material Methylene blue is employed to evaluate the adsorption characteristics of carbon. A known weight of 1000 mg of MB was dissolved in about one litre of distilled water to get the stock solution.



Structure of Methylene Blue

Batch equilibrium method

The adsorption experiments were carried out in a batch process at 30, 40, 50 and 60˚ C. A known weight of AHSNC was added to 50 ml of the dye solutions with an initial concentration of 50 mg/L to 250 mg/L, which is prepared from 1000 mg/L of methylene blue stock solution. The contents were shaken thoroughly using a mechanical shaker with a speed of 150 rpm. The solution was then filtered at present time intervals and the residual dye concentration was measured.

RESULT AND DISCUSSIONS

Characteristics of the adsorbent

Activated Hibiscus Sabdariffa Stem Nano Carbon is an effective adsorbent for the abatement of many pollutant compounds (organic, inorganic, and biological) of concern in water and wastewater treatment. Most of the solid adsorbents possess micro porous fine structure, high adsorption capacity, high surface area and high degree of surface, which consists of pores of different sizes and shapes. The wide usefulness of AHSNC is a result of their specific surface area, high chemical and mechanical stability. The chemical nature and pore structure usually determines the sorption activity. The physico-chemical properties of the chosen adsorbent are listed in Table 1.

Table 1-Characteristics of the Adsorbent

Properties	AHSNC
Particle size(mm)	0.030
Density (g/cc)	0.1035
Moisture content (%)	0.1634
Loss in ignition (%)	0.030
pH of aqueous solution	4.8

Effect of contact time and initial dye concentration

The effect of contact time on the amount of dye adsorbed was investigated at 1000 mg/L concentration of the dye (Fig. 1). It is observed that the percentage removal of dye increases rapidly with an increase in contact time initially, and thereafter, beyond a contact time of about 40 min, no noticeable change in the percentage removal is observed the percentage removals after 40 min were 85%. Therefore, the optimum contact time is considered to be 40 min. this is also the equilibrium time of the batch adsorption experiments, since beyond a contact time of 40 min, adsorption is not changed. The rapid removal of dye is observed at the beginning of the contact time due to the percentage of large number of binding sites available for adsorption. The experimental results of adsorptions at different concentrations (50 to 250mg/L) collected in Table 2 observed that percent adsorption decreased with increase in initial dye concentration, but the actual amount of dye adsorbed per unit mass of AHSNC increased leads to increase in dye concentration. This means that the adsorption is highly dependent on initial concentration of dye. At lower concentration, the ratio of the initial number of dye molecules to the available surface area is low. Subsequently, the fractional adsorption becomes independent of initial concentration. However, at high concentration the available sites of adsorption become less and hence the percentage removal of dye is dependent upon initial concentration [10, 11].

Effect of adsorbent dosage

The adsorption of the methylene blue dye on AHSNC was studied by varying the adsorbent dose (25–125 mg/50ml) for 50 mg/L of dye concentration. The percentage of adsorption increased with increases in the AHSNC concentration, which is attributed to increased carbon surface area and the availability of more adsorption sites [12, 13]. Hence, all studies were carried out with 0.025g of adsorbent /50 ml of the varying adsorbate solutions.50, 100, 150, 200 and 250. The results obtained from this study are shown in Fig. 2. The amount of MB adsorbed per gram reduced with increase in the dosage of AHSNC. This reveals that the direct and equilibrium capacities of MB are functions of the activated AHSNC dosage.

Effect of solution pH

The solution pH is one of the most important factors that control the adsorption of dye on the sorbent material. The adsorption capacity can be attributed to the chemical form of dye in the solution at specific pH. In addition, due to different functional groups on the adsorbent surface, which become active sites for the dye binding at a specific pH the effect of adsorption can vary substantially. Therefore, an increase in pH may cause an increase or decrease in the adsorption, resulting different optimum pH values dependent on the type of adsorbent. To examine the effect of pH on the % removal of MB dye , the solution pH were varied from 2.0 to 10.0 by adding acid and base to the stock solution This increases may be due to the presence of negative charge on the surface of the adsorbent AHSNC that may be responds for the dye binding. However, as the pH is lowered, the hydrogen ions compete with dye for the adsorption sites in the adsorbent AHSNC, the overall surface charge on the particles become positive and hinds the binding of positively charged dye. On other hand, decrease in the adsorption under pH >6.3 may be due to occupation of the adsorption sites by OH^- ions which retard the approach of such dye further toward the adsorbent AHSNC surface. From the experimental results, the optimum pH range for the adsorption of the MB dye is 2.0 to 6.5 shown in Fig.3.

Adsorption isotherms

Langmuir isotherm

The theoretical Langmuir isotherm is used for adsorption of a solute from a liquid solution as monolayer adsorption on a surface containing a finite number of identical sites. Therefore, the Langmuir isotherm model was chosen for estimation of the maximum adsorption capacity corresponding to complete monolayer coverage on the adsorbent surface. The Langmuir non-linear equation is commonly expressed as follows:

$$C_{eq}/Q_{eq} = 1/Q_m b + C_{eq}/Q_m \quad \text{..... (1)}$$

Where C_{eq} is the equilibrium concentration of adsorbate in the solution (mg/L), Q_{eq} is the amount adsorbed at equilibrium (mg/g), Q_m and b are Langmuir constants related to adsorption efficiency and energy of adsorption, respectively. The linear plots of Ceq/ Q_{eq} vs. C_{eq} suggest the applicability of the Langmuir isotherms. The values of Q_m and *b* were calculated from slope and intercepts of the plots are given in Table 3. From the results, it is obvious that the value of adsorption efficiency Q_m and adsorption energy *b* of the AHSNC increases on increasing the temperature. The values can conclude that the maximum adsorption corresponds to a saturated monolayer of adsorbate molecules on adsorbent surface with endothermic nature of adsorption [14, 15]. To confirm the favorability of the adsorption process, the separation factor (R_L) was determined and given in Table 4.The values were established to be between 0 and 1 and confirm that the ongoing adsorption process is favorable [16].

The Freundlich isotherm

The Freundlich isotherm model is the earliest known equation describing the adsorption process. It is an empirical equation and can be used for non-ideal sorption that involves heterogeneous adsorption. The Freundlich equation was employed for the adsorption of methylene blue dye on the adsorbent. The Freundlich isotherm was represented by the following equation.

$$\log Q_e = \log K_f + 1/n \log C_e \quad \text{.....(2)}$$

Where Q_e is the amount of methylene blue dye adsorbed (mg/g), C_e is the equilibrium concentration of dye in solution (mg/L), and K_f and n are constants incorporating the factors affecting the adsorption capacity and intensity of adsorption, respectively. Linear plots of log Q_e versus log C_e shows that the adsorption of methylene blue obeys the linear plots of log Q_e versus log C_e shows that the adsorption of methylene blue dye obeys the Freundlich adsorption isotherm. The values of K_f and *n* are given in Table 4 shows that the increase of negative charges on the adsorbent surface makes electrostatic force like Vanderwaal's between the AHSNC surface and dye ion. The molecular weight and size either limit or increase the possibility of the adsorption of the dye onto adsorbent. However, the values clearly show the dominance in adsorption capacity.

The intensity of adsorption is an indication of the bond energies between dye and adsorbent, and the possibility of slight chemisorptions rather than physisorption [17, 18]. However, the multilayer adsorption of methylene blue through the percolation process may be possible. The values of *n* are less than one, indicating the physisorption is much more favorable [19].

Effect of temperature

To study the effect of temperature on the adsorption of dye adsorption by AHSNC, the experiments were performed at temperatures of 30, 40, 50, 60°C. As it was observed that, the equilibrium adsorption capacity of MB onto AHSNC was found to increase with increasing temperature, especially in higher equilibrium concentration, or lower adsorbent dose because of high driving force of adsorption. This fact indicates that the mobility of dye molecules increased with the temperature. The adsorbent shows the endothermic nature of adsorption. The adsorption capacity of the AHSNC increased with increase of the temperature in the system from 30° to 60°C. Thermodynamic parameters such as change in free energy *(* $\Delta G°$) (kJ/mol), enthalpy ($\Delta H°$) (kJ/mol) and entropy ($\Delta S°$) (J/K/mol) were determined using the following equations.

$$K_0 = C_{solid}/C_{liquid} \quad \text{..... (3)}$$

$$\Delta G° = -RT \ln K_0 \quad \text{..... (4)}$$

$$\log K_0 = \Delta S°/ (2.303R) - \Delta H°/ (2.303RT) \quad \text{..... (5)}$$

Where K_o is the equilibrium constant, C_{solid} is the solid phase concentration at equilibrium (mg/L), C_{liquid} is the liquid phase concentration at equilibrium (mg/L), *T* is the temperature in Kelvin, and *R* is the gas constant. The $\Delta H°$ and $\Delta S°$ values obtained from the slope and intercept of Van't Hoff plots are given in Table 5.The values of $\Delta H°$ is the range of 9 to 17 kJ/mol, indicate the physisorption. The results show that physisorption is much feasible for the adsorption of methylene blue. The positive values of $\Delta H°$ show the endothermic nature of adsorption which governs the possibility of physical adsorption [19, 20]. Because in the case of physical adsorption, while increasing the temperature of the system, the extent of dye adsorption increases, there is no possibility of chemisorption. The negative values of $\Delta G°$ (Table 5) show that the adsorption is highly favorable and spontaneous. The positive values of $\Delta S°$ (Table 5) show the increased disorder and randomness at the solid solution interface of methylene blue with AHSNC adsorbent. The enhancement of adsorption capacity of the activated AHSNC at higher temperatures was ascribed to the enlargement of pore size and activation of the adsorbent surface.

Adsorption kinetics

The study of adsorption dynamics describes the solute up take rate and evidently this rate controls the residence time of adsorbate uptake at the solid-solution interface .The kinetics of MB dye adsorption on

the AHSNC were analyzed using pseudo second-order[21] Elovich [22] and intra-particle diffusion[23] kinetic models. The conformity between experimental data and the model predicted values was expressed by the correlation co- efficient (γ) and the values are close or equal to 1. A relatively high correlation coefficient (γ) value indicates that the pseudo second-order model successfully describes the kinetics of MB dye adsorption.

The pseudo second- order equation

The pseudo second-order adsorption kinetic rate equation is expressed as

$$dq_t/d_t = k_2(q_e-q_t)^2 \text{ ……..(6)}$$

Where: k_2 is the rate constant of pseudo second- order adsorption (g mg/min). For the boundary conditions t = 0 to t= t and $q_t = 0$ to $q_t = q_t$ the integrated form of Eq. (6) becomes:

$$1/(q_e - q_t) = 1/q_e + K_2t \text{ …….. (7)}$$

This is the integrated rate law for a pseudo second-order reaction. Equation (7) can be rearranged to obtain Eq.(8),which has a linear form:

$$t/q_t=(1/k_2q_e^2)+ ((1/q_e)t \text{ ……..(8)}$$

If the initial adsorption rate (h)(mg $g^{-1}min^{-1}$) is :

$$h=k_2q_e^2 \text{ …….. (9)}$$

Equation (8) and (9) becomes,

$$t / q_t = 1 / h + 1 / q_e\, t \text{ ……. …(10)}$$

The plot of (t/q_t) and t of Eq. (10) gives a linear relationship from which q_e and k_2 can be determined from the slope and intercept of the plot, respectively. The pseudo-second order rate constants K_2, the calculated h values, and the correlation coefficients (γ) are summarized in Table (5).At all studied initial MB dye concentrations, the straight lines with extremely high correlation co-efficient (>0.99) were obtained. From table 5, the values of the rate constant k decrease with in increasing initial MB dye concentration for AHSNC. This is shows that the sorption of MB dye on AHSNC follows pseudo second order kinetic model [24, 25].

The Elovich equation

The Elovich model equation is generally expressed as

$$dq_t / d_t = \alpha \exp(-\beta q_t) \text{ ……..(11)}$$

Where; α is the initial adsorption rate (mg g^{-1} min^{-1}) and β is the desorption constant (g/mg) during any one experiment. To simplify the Elovich equation [22]. Assumed $\alpha\beta t >> t$ and by applying boundary conditions $q_t = 0$ at t= 0 and $q_t = q_t$ at t = t Eq.(11) becomes:

$$q_t = 1/\beta \ln(\alpha\beta) + 1/\beta \ln t \text{ …….. (12)}$$

If MB dye adsorption fits with the Elovich model, a plot of q_t vs. ln(t) should yield a linear relationship with a slope of $(1/\beta)$and an intercept of $(1/\beta)\ln(\alpha\beta)$. The Elovich model parameters α, β, and correlation coefficient (γ) are summarized in table 5. The experimental data such as the initial adsorption rate (α) adsorption constant (β) and the correlation co-efficient (γ) calculated from this model indicates that the initial adsorption (α) increases with temperature similar to that of initial adsorption rate (h) in pseudo-second–order kinetics models. This may be due to increase the pore or active site on the AHSNC adsorbent.

The intra particle diffusion model

The intra-particle diffusion model used here refers to the theory proposed by Weber and Morris [23] based on the following equation for the rate constant:

$$q_t = k_{id}\, t^{(1/2)} + C \text{……..(13)}$$

Where k_{id} is the intra-particle diffusion rate constant (mg/g/min) and C is the constant. Since the rate limiting step is intra-particle diffusion, the graph drawn between (q_t) (mg/g) verses square root of the contact time ($t^{1/2}$) yields a straight line passing through the origin [23]. The slope of the will give the value of the intra-particle diffusion coefficient (k_{id}) and correlation coefficient (γ) indicate the fitness of this model. The value of C gives an idea about the thickness of the boundary layer. The intercept value indicates that the lines were not passing through origin, there are some other process affect the adsorption. But the correlation coefficient (γ) value is very high, so that the intra-particle diffusion takes place along with other process that may affect the adsorption. The values are given in table 5.

Desorption studies:

Desorption studies help to elucidate the nature of adsorption and recycling of the spent adsorbent and the dye. If the adsorbed dye can be desorbed using neutral pH water, then the attachment of the dye molecule of the adsorbent is by weak bonds. The effect of various reagents used for desorption studies. The results indicate that hydrochloric acid is a better reagent for desorption, because we could get more than 90% removal of adsorbed dyes. The reversibility of adsorbed dyes in mineral acid or base is in agreement with the pH dependent results obtained. The desorption of dye molecules by mineral acids and

alkaline medium indicates that the dye was adsorbed onto the AHSNC through physisorption as well as by chemisorptions mechanisms.

TABLE: 2. EQUILIBRIUM PARAMETERS FOR ADSORPTION OF MB DYE ONTO AHSNC ADSORBENT

M_0	Ce (Mg / L)				Qe (Mg / L)				Removal %			
	30°C	40°C	50°C	60°C	30°C	40°C	50°C	60°C	30°C	40°C	50°C	60°C
50	1.0215	0.8876	0.7732	0.7023	97.957	98.225	98.454	98.595	97.957	98.225	98.454	98.595
100	4.836	4.2689	3.5050	2.9576	190.3283	191.46	192.99	194.08	95.164	95.731	96.495	97.042
150	10.820	9.556	8.343	7.271	278.3606	280.89	283.31	285.46	92.787	93.629	94.438	95.153
200	21.048	19.446	8.343	16.092	357.9046	361.11	383.31	367.82	89.476	90.277	95.829	91.954
250	32.844	30.816	17.763	26.929	434.3130	438.37	464.47	446.14	86.863	87.674	92.895	89.228

TABLE: 3. LANGMUIR AND FREUNDLICH ISOTHERM PARAMETER FOR ADSORPTION OF MB ONTO AHSNC

Temp. (°C)	Langmuir Parameters		Freundlich Parameters	
	Q_m	b	K_f	n
30°C	503.99	0.1435	97.358	2.3246
40°C	503.53	0.1642	104.144	2.3643
50°C	594.67	0.1670	109.695	1.9797
60°C	501.58	0.2246	119.058	2.4248

TABLE: 4. DIMENSIONLESS SEPERATION FACTOR (R_L) FOR ADSORPTION OF MB ONTO AHSNC

(C_i)	Temperature °C			
	30°C	40°C	50°C	60°C
50	0.1223	0.1086	0.1070	0.0818
100	0.0651	0.0574	0.0565	0.0426
150	0.0444	0.0390	0.0384	0.0288
200	0.0337	0.0295	0.0291	0.0218
250	0.0271	0.0238	0.0234	0.0175

TABLE: 5. THERMODYNAMIC PARAMETER FOR THE ADSORPTION OF MB ONTO AHSNC

(C_0)	ΔG°				ΔH°	ΔS°
	30°C	40°C	50°C	60°C		
50	-9749.3	-10443.9	-11154.3	-11770.0	-10.788	67.822
100	-7505.9	-8093.6	-8903.0	-9664.5	-14.554	72.627
150	-6434.9	-6993.9	-7605.1	-8242.1	-11.834	60.229
200	-5391.8	-5798.9	-8416.9	-6744.5	-15.437	69.262
250	-4758.3	-5105.3	-6903.3	-5853.5	-11.045	52.517

TABLE: 6. THE KINETIC PARAMETERS FOR THE ADSORPTION OF MB ONTO AHSNC

C_0	Temp °C	Pseudo second order				Elovich model			Intraparticle diffusion		
		q_e	K_2	γ	h	α	β	γ	K_{id}	γ	C
50	**30**	100.59	0.0051	0.9913	51.692	27348	0.2013	0.9912	0.0527	0.9903	1.8951
	40	100.72	0.0053	0.9934	53.618	68379	0.2107	0.9933	0.0502	0.9924	1.9006
	50	100.82	0.0055	0.9905	56.197	17903	0.2206	0.9904	0.0477	0.9895	1.9058
	60	101.01	0.0055	0.9916	55.769	13216	0.2169	0.9915	0.0485	0.9906	1.9052
100	**30**	195.17	0.0027	0.9907	103.618	97105	0.1070	0.9906	0.0510	0.9897	1.8854
	40	196.30	0.0027	0.9918	104.230	13451	0.1082	0.9917	0.0501	0.9908	1.8893
	50	197.70	0.0028	0.9909	109.741	24410	0.1105	0.9908	0.0486	0.9899	1.8957
	60	198.96	0.0028	0.9930	109.818	16716	0.1077	0.9929	0.0496	0.9920	1.8968
150	**30**	285.73	0.0018	0.9911	147.010	9945	0.0718	0.9910	0.0520	0.9901	1.8726
	40	288.04	0.0019	0.9904	153.556	16202	0.0730	0.9903	0.0507	0.9894	1.8790
	50	290.63	0.0019	0.9904	159.290	20548	0.0731	0.9903	0.0501	0.9894	1.8844
	60	292.16	0.0016	0.9923	133.388	68079	0.0780	0.9922	0.0467	0.9913	1.8878
200	**30**	367.72	0.0014	0.9906	182.606	6967	0.0541	0.9905	0.0538	0.9896	1.8535
	40	370.87	0.0013	0.9917	185.688	85666	0.0542	0.9916	0.0532	0.9907	1.8584
	50	374.22	0.0014	0.9928	189.947	10433	0.0542	0.9927	0.0526	0.9918	1.8635

	60	377.38	0.0014	0.9919	202.709	18752	0.0553	0.9918	0.0510	0.9909	1.8709
250	**30**	446.47	0.0011	0.9943	218.035	62698	0.0438	0.9942	0.0547	0.9933	1.8390
	40	450.65	0.0011	0.9944	217.231	54820	0.0431	0.9943	0.0552	0.9934	1.8420
	50	455.36	0.0010	0.9930	209.305	30275	0.0413	0.9929	0.0571	0.9920	1.8422
	60	458.52	0.0011	0.9937	222.886	539490	0.0423	0.9936	0.0553	0.9927	1.8496

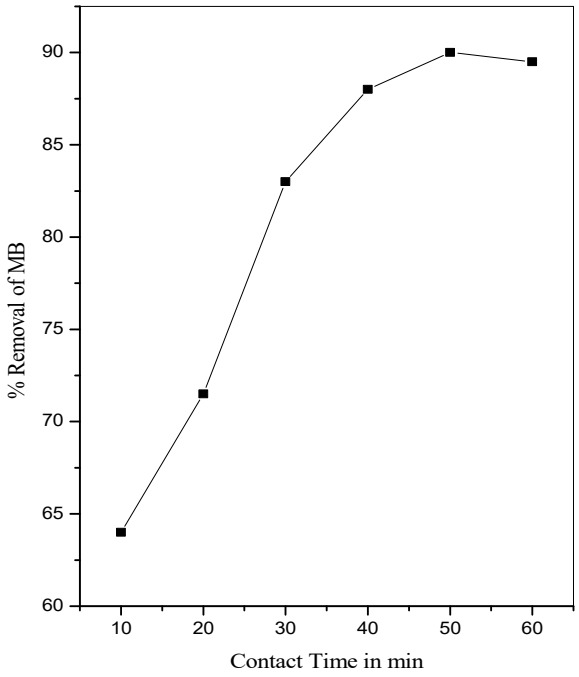
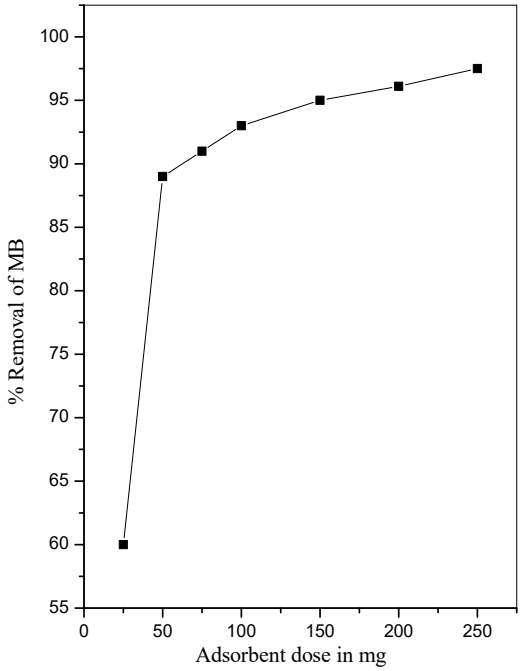
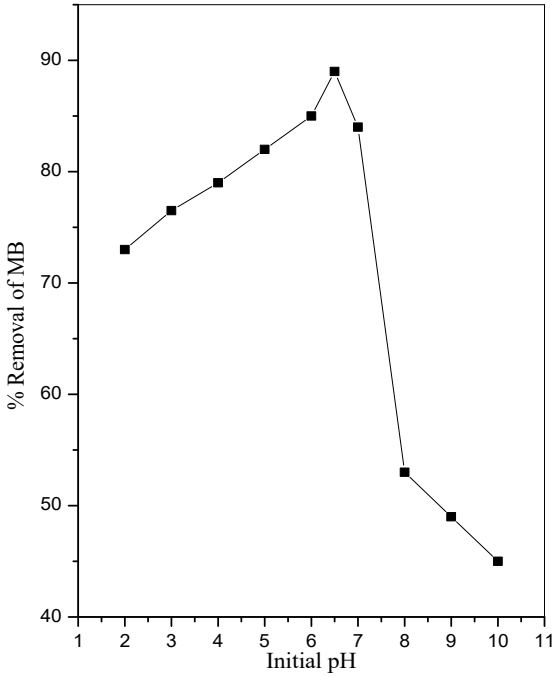


Fig:1- Effect of Contact Time on the Removal of MB Dye
[MB]=50 mg/L;Temprature 30°C;Adsorbent dose=25mg/50ml



Fig;2- Effect of Adsorbent dose on the removal of MB Dye
[MB]=50mg/L;Contact Time 50min;Temprature 30^{0}C



Fig;3- Effect of Initial pH on the removal of MB Dye
[MB]=50 mg/L;Temprature 30°C;Adsorbent dose=25mg/50ml

CONCLUSION

The present study has shown the effectiveness of using AHSNC in the removal of methylene blue dye from aqueous solutions. Activated Hibiscus Sabdariffa Stem Nano Carbon in different forms has a great role in modern life to clean environment. Hibiscus Sabdariffa Stem can be good precursors for producing highly porous Activated Hibiscus Sabdariffa Stem Nano Carbon by simple preparative methods. An adsorption test has been carried out for industrial pollutants (methylene blue) under different experimental conditions in batch mode. The adsorption of methylene blue was dependent on adsorbent surface characteristics, adsorbent dose, methylene blue concentration, time of contact and temperature. A study of the kinetic models on sorption showed that sorption fitted the pseudo second- order kinetics model. The ΔG^0, ΔH^0, and ΔS^0 reveal the favorability of adsorption. The thermodynamic parameters suggested that the adsorption on AHSNC was a spontaneous and endothermic process.

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