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Equilibrium, Adsorption Isotherm and Kinetic Studies on the Removal of Methylene Blue Dye from Aqueous Solution onto Senna Occidentalis Seeds Activated Carbon

Gulumbe, N.S.[1*] Bello, K.A.[1] Yakubu, M.K.[1] Abdulazeez, A.Y.[2]
1.Department of Polymer and Textile Engineering, Faculty of Engineering, Ahmadu Bello University, Zaria – Nigeria
2.Department of Textile Technology, Collage of Science & Technology, Kaduna Polytechnic, Kaduna State – Nigeria

Abstract

This paper studied the adsorption potentials and efficiency of low cost activated carbon produced from *Senna occidentalis* seeds for the removal of Methylene Blue dye in aqueous solutions. Batch adsorption experiments were carried out to investigate the effects of variables such as initial dye concentration, carbon dosage, contact time, pH and temperature to the process. Similarly, adsorption isotherms consisting Langmuir, Freundlich and Temkin were employed and found Langmuir Adsorption model fitted best. However, Kinetic models consisting pseudo first-order, pseudo second-order and intra-particle diffusion were also used to investigate the kinetic nature of the process, and found that pseudo second-order model better explained the process.

Keywords: Equilibrium, Adsorption Isotherm, Kinetic Studies, Activated Carbon and Methylene Blue Dye
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1. Introduction

The environmental and health issues associated with residual colour (which is basically due to dyes and pigments) in textile effluents have posed a major challenge to environmental scientists as well as the textile colouration processors (Joshi *et al*., 2004).

Large scale production and the widespread application of synthetic dyes by various industries such as the textile, leather, tanning, food, cosmetics, paper printing, plastics etc. lead to severe contamination of surface and groundwater sources in the vicinity of dye industries (Ali *et al*., 2010).

Synthetic dyes often have complex aromatic molecular structures that make them more difficult to naturally biodegrade. However, their disposal in water bodies exhibit adverse effect to the aquatic and human life by creating eutrophication mutagenic, carcinogenic effects, dysfunction of the organs, also having effect on aquatic ecosystem by preventing sunlight from reaching into the water bodies (Sharifi *et al*., 2018).

Some of the available methods for dyes removal or degradation include; aerobic biodegradation (Liu *et al*., 2018), membrane nano-filtration (Oatley-Radcliffe *et al*., 2017), photocatalytic degradation (Giwa *et al*., 2016), adsorption (Okoli *et al*., 2015), electro-coagulation (Nandi and Patel, 2013), ozonation (Wijannarong *et. al*., 2013), precipitation (Gupta *et. al*., 2012), electrochemical (Jain *et al*., 2004) etc.

Many studies have been undertaken for the removal of pollutants by using variety of materials (used as adsorbents) ranging from agricultural waste or agro-by-products such as; coir pith (Namasivayam and Kavitha, 2002), date seeds (Yakubu *et al*., 2008), rice husk (Sharma and Janveja, 2008), mango seed shell (Akpen *et al*., 2011), groundnut shell (Mohammed *et al*., 2015), tamarind seed (Okoli *et al*., 2015), aloe vera leaves shell (Omidi *et al*., 2017); biomass such as cellulose dust (Sharifi *et al*., 2018) to varieties of clays (Vimonsesa *et al*., 2009).

Activated carbon is extensively used as an adsorbent due to its high level of effectiveness, but it is expensive and has high usage costs. This has motivated many environmental scientists to embark on exploration for inexpensive and locally available adsorbents so that the process can become economically viable. In this study, the use of Senna Occidentalis Seeds Activated Carbon (SOSAC) as an adsorbent for the removal of a MB dye from aqueous solution was carried out. Senna occidentalis is a cheaply available plant occurring as shrub that is regarded as weed or not important. The plant is said to be found throughout Nigeria (especially in the northern part) along major highways, growing abundantly on waste lands (Gulumbe *et al*. 2019).

2. Materials and Methods

The major materials used in this study are; Senna occidentalis seeds which was obtained from Birnin Kebbi local government area of Kebbi State, Nigeria, from which the adsorbent was prepared; Methylene blue dye which was purchased from Cardinal Scientific Supplies, Zaria, Nigeria, from which the adsorbate solution was prepared. Other materials used include zinc chloride, hydrochloric acid, sodium hydroxide, double-distilled water etc. All other chemicals used were of analytical reagent grade.

2.1 Preparation of Adsorbent

The adsorbent used in this study was adopted from the already prepared SOSAC with its preparatory methods been reported in the earlier work (Gulumbe *et al*., 2019). The intention of this research is to evaluate and confirm the adsorption potentials of SOSAC as reported in the earlier research.

2.2 Preparation of Adsorbate

Methylene blue (MB) dye of commercial-grade purchased from Cardinal Scientific Supplies was used as received to prepare a stock solution. This is done by dissolving 1 g of the powdered MB dye into 1 litre of distilled water to obtain stock solution of 1 g/L concentration. The following specifications concerning the dye were obtained before use: Name – Basic Blue 9, C.I No. – 52015, chemical formula – $C_{16}H_{18}ClN_3S$, molecular weight – 319.85 g/mol and λmax – 670 nm.

However, the experimental solutions in the desired concentrations were prepared through subsequent dilutions of the stock solution.

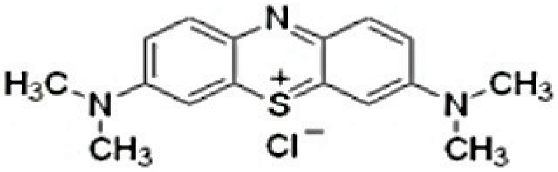

Figure 1: Chemical Structure of Methylene Blue Dye

2.3 Equilibrium Adsorption Studies

Batch adsorption process was employed in these studies, and in each adsorption process, 100 ml of dye solution with a known concentration (in mg/L) was added to a mass (in g) of activated carbon in a 250 ml clean beaker at a room temperature (of about 30 ^{0}C), and the mixture was stirred on a magnetic stirrer at 100 rpm. The samples were withdrawn from stirring setup at a predetermined time intervals, allowed to settle for 10–15 minutes and the adsorbent was separated from the solution by the help of a micropipette. The absorbance of the supernatant solution was estimated to determine the residual dye concentration, and was measured before and after treatment with the help of spectrophotometer at the maximum wavelength of 670 nm.

The adsorption parameters varied include; initial MB dye concentration (10–60) mg/L; contact time (20–180) minutes; pH (3–11)**;** temperature at (303, 313 and 323) K and carbon dosage (0.5–2.5) g. The pH of the dye solution was adjusted by using 0.1M NaOH or 0.1M HCl solution and a pH meter.

The parameters variation was to determine their effects to the equilibrium adsorption, the adsorption isotherm as well as kinetics of MB dye adsorption onto SOSAC.

The amount of MB dye adsorbed at time, t, q_t (mg/g) as well as the percentage dye removed (%) were calculated using the eq. 1 and 2 respectively:

$$q_t = \frac{(C_o - Ct) \times V}{M} \qquad (1)$$

$$R\,(\%) = \frac{(C_o - Ce)}{C_o} \times 100 \qquad (2)$$

where; C_0 and C_t are the initial concentration of the dye in solution (mg/L) and equilibrium concentrations of the dye in solution at time, t (mg/L), V is the volume of the solution used (L) and M is the mass of the adsorbent (g).

2.4 Adsorption Isotherms

There are several isotherm equations available for analysing experimental adsorption equilibrium parameters and the most common being the Langmuir, Freundlich and Temkin models. Meanwhile, the mathematical relationship between the amount of MB dye adsorbed (q_t) and its equilibrium concentration at time, t (C_t), was evaluated using Langmuir, Freundlich and Temkin adsorption isotherms.

2.4.1 Langmuir Adsorption Isotherm

The Langmuir isotherm model is based on the assumption that there is a finite number of active sites which are homogeneously distributed over the surface of the adsorbent These active sites have the same affinity for adsorption of a mono molecular layer and without interaction between the adsorbed molecules.

The Linear form of the Langmuir equation can be expressed as eq. 3 (Langmuir, 1916; Langmuir, 1918):

$$C_t/q_t = 1/K_L q_m + 1/q_m \times C_t \qquad (3)$$

where; q_t is the amount of dye adsorbed (mg/g), C_t is the equilibrium concentration of the adsorbate at time, t (mg/L), while q_m and K_L are Langmuir constants that stand for maximum adsorption capacity (mg/g) and energy of adsorption (L/mg). When adsorption obeys the Langmuir equation, a plot of C_t/q_t against C_t should give a straight line graph with a slope of 1/qm and intercept of $1/K_L \times q_m$.

However, another very important characteristic of Langmuir isotherm is called Langmuir Dimensionless Separation Factor (R_L) which can be obtained from the eq. 4:

$$R_L = 1/(1 + K_L C_0) \quad (4)$$

where; C_0 is the highest initial dye concentration (mg/L)

The R_L value normally indicates the type of adsorption either it is favourable (i.e. $0<R_L<1$), unfavourable (i.e. $R_L > 1$), feasible or linear (i.e. $R_L = 1$) or irreversible (i.e. $R_L = 0$).

2.4.2 Freundlich Adsorption Isotherm

The Freundlich isotherm model is the earliest known relationship describing the non-ideal and reversible adsorption, which can be applied to multilayer adsorption, on the basis of an assumption concerning the energetic surface heterogeneity. The Freundlich model is an empirical equation based on the distribution of solute between the solid phase and aqueous phase at equilibrium. The Freundlich model is used for heterogeneous surfaces but can describe adsorption data over a restricted range only (Ng *et. al*., 2002).

The linear form of Freundlich equation can be expressed as eq. 5 (Freundlich, 1906):

$$\ln q_t = \ln K_f + 1/n \ln C_t \quad (5)$$

where; q_t is the amount of dye adsorbed at any time (mg/g), C_t is the equilibrium concentration at any time (mg/L), K_f and n are Freundlich constants related to maximum adsorption capacity (mg/g) and surface heterogeneity (dimensionless). The values of K_f and 1/n can be obtained respectively from intercept and slope of plot of the $\ln q_t$ against $\ln C_t$.

2.4.3 Temkin Adsorption Isotherm

Temkin adsorption model assumes that the fall in the heat of sorption is linear rather than logarithmic, as implied in the Freundlich equation. The appropriate isotherm according to Temkin is shown in the eq. 6 (Tempkin and Pyzhev, 1940):

$$q_e = (RT)/b_T \ln k_T + (RT)/b_T \ln C_e \quad (6)$$

$$B_1 = RT/b_T \quad (7)$$

where; b_T indicates the adsorption potential of the adsorbent, k_T is the equilibrium binding constant (L/mg) and B_1 is the Temkin heat of sorption (J/mol) which can be obtained from eq. 7.

3. Results and Discussion

3.1 Effect of Initial MB Dye Concentration

The result for the effect of initial concentration of MB dye to its removal onto the activated carbon was presented in Fig. 2. The concentration of MB solution was varied from the ranges of 10–60 mg/L at 1.5 g/100 mL carbon dosage and solution pH of 7.3. It can be seen from Fig. 2 that percentage MB dye removal increased with increased in initial adsorbate concentration, and similar was reported by other researchers (Akpen *et. al*., 2011). The highest removal recorded was at 93.6%.

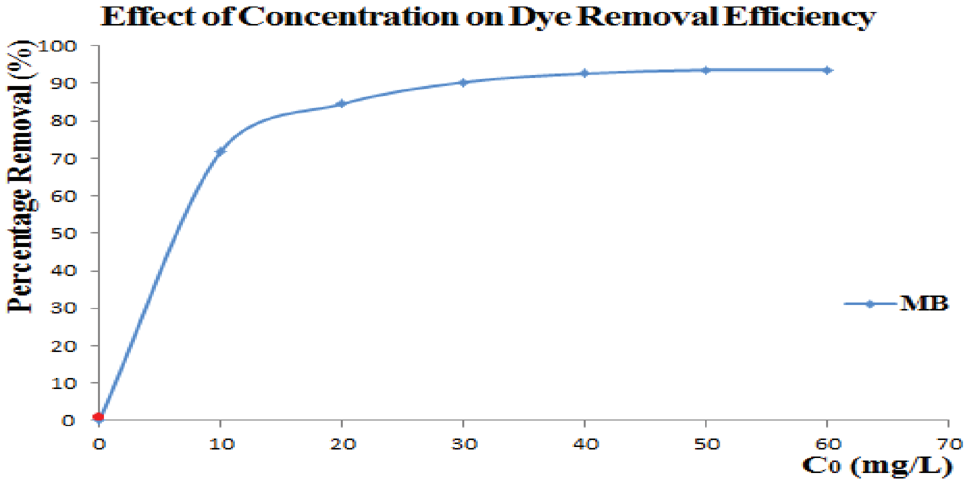

Fig. 2: Effect of Initial Dye Concentration on the Percentage MB Dye Removal onto SOSAC at 1.5g Carbon Dose and Contact Time of 60 minutes.

3.2 Effect of Contact Time

The result for the effect of contact time is presented in Fig. 3 which clearly shows the extent of adsorption is rapid in the initial stage and becomes slow in later stage till saturation was reached. It is chiefly because of the saturation of the active sites on the SOSAC, which do not permit furthermore adsorption to occur. This can be explained by the fact that initially, the quantity of sites on the surface is very large which allows adsorption to take place very easily. But with the passage of time, the active sites get saturated thereby reducing the rate at which adsorption occurred.

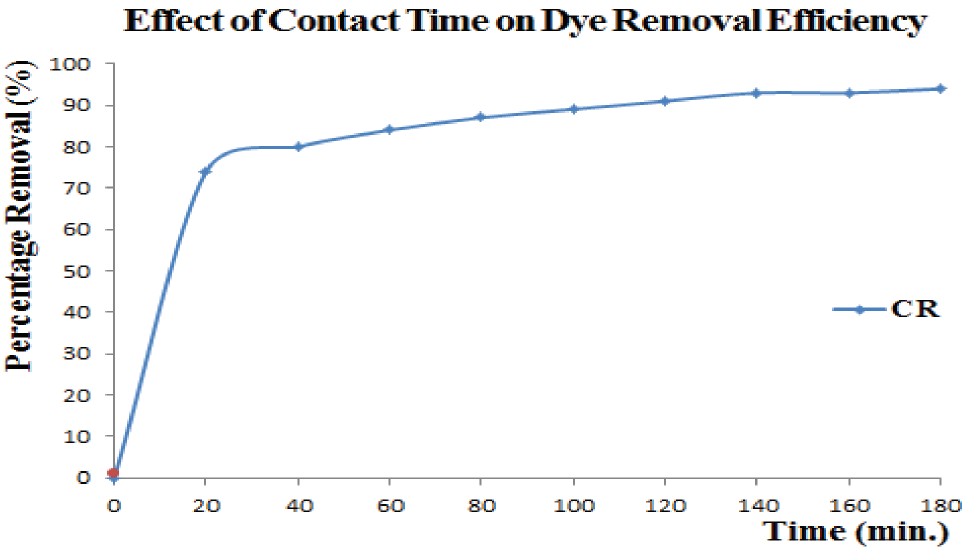

Fig. 3: Effect of Contact Time on the Percentage MB Dye Removal onto SOSAC at 2g Carbon Dose and 50 mg/L Dye Concentration.

3.3 Effect of SOSAC Dosage

The result for variation of percentage removal of MB with adsorbent doses of 0.5, 1, 1.5, 2 and 2.5 g at dye concentration of 50 mg/L is presented in Fig.4. The figure revealed that an increase in the quantity of SOSAC results in a corresponding increase in the amount of MB dye removed. This increase in removal efficiency with simultaneous increase in adsorbent dose is due to the increase in surface area and hence more sites were available for adsorption of the dye. Similarly, it is clear that the SOSAC has achieved at least 93% removal of MB dye from the aqueous solution. However, 1 g/100 ml of SOSAC was chosen to be the optimum carbon dose under this study. This finding is in line with the result that has been reported (Akpen *et. al*., 2011).

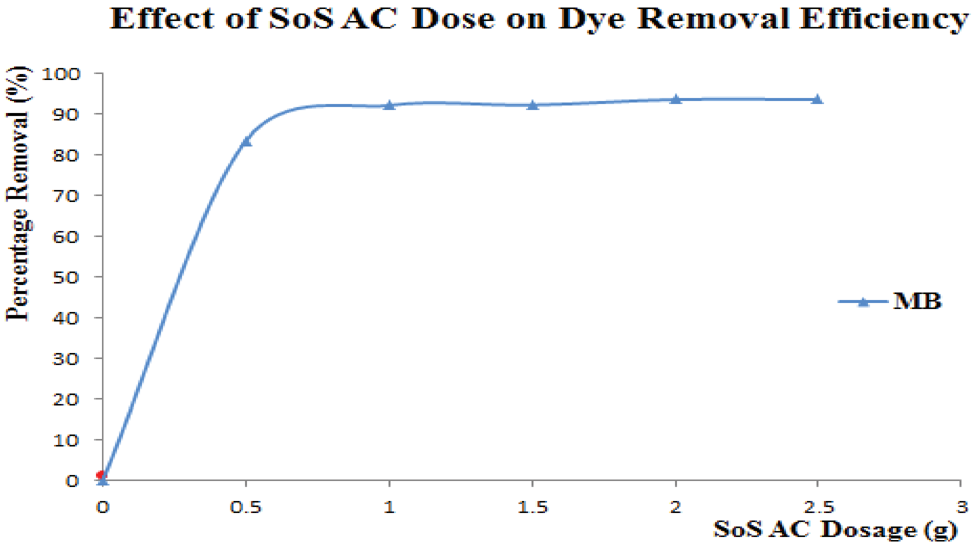

Fig. 4: Effect of Adsorbent Dosage on the Percentage MB Dye Removal onto SOSAC at 50 mg/L Dye Concentration and 60 minutes Contact time.

3.4 Effect of Initial pH

The effect of initial pH on dye solution for the MB dye removal was investigated by varying the pH from 3 to 11 and presented in the Fig. 5. SOSAC has achieved maximum removal of MB dye at pH = 11 and decreased down the pH Scale as we see in Fig 5. In fact adsorption was found to increase with increase in pH of solution. The adsorption of these positively charged dye groups on the adsorbent surface is primarily influenced by the surface charge on the adsorbent which in turn is influenced by the solution pH. The result showed that availability of negatively charged groups at the adsorbent surface is necessary for the adsorption of basic dyes to proceed which we see at pH – 3 is almost minimum as there is a net positive charge in the adsorption system due to the presence of H_3O^+. Thus as the pH increased, more negatively charged surface was available thereby facilitating greater dye removal.

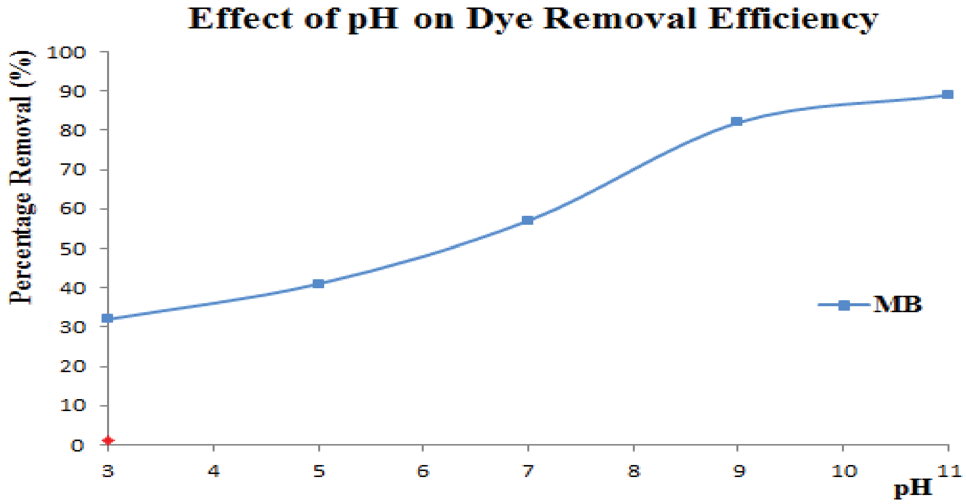

Figure 5: Effect of pH on the Percentage MB Dye Removal onto SOSAC at 1 g Carbon Dose, 30 mg/L Dye Concentration and 40 minutes Contact time.

3.5 Effect of Temperature

The effect of temperature on the percentage MB dye removal from solution with initial concentration of 20 mg/L at dye solution pH and varied temperatures of 30, 40 and 50 °C has been determined. It has been established that the percentage MB dye removal increased with increased in temperature and the result is presented in the Fig. 6. The increase in the percentage MB dye removal may be as a result of increase in the mobility of the large dye molecule with temperature. An increasing number of molecules may also acquire sufficient energy to undergo an interaction with active sites at the surface. Furthermore, increasing temperature may produce a swelling effect within the internal structure of the activated carbon enabling large dyes to penetrate further.

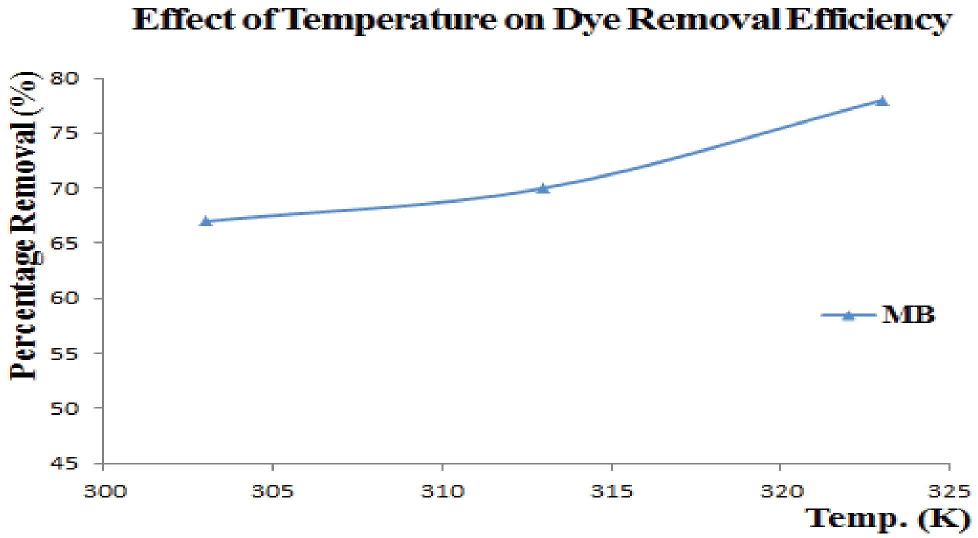

Figure 6: Effect of Temperature on the Percentage MB Dye Removal onto SOSAC at 0.5g Carbon Dosage, 20 mg/L Dye Concentration and 40 minutes Contact time.

3.6 Adsorption Isotherms

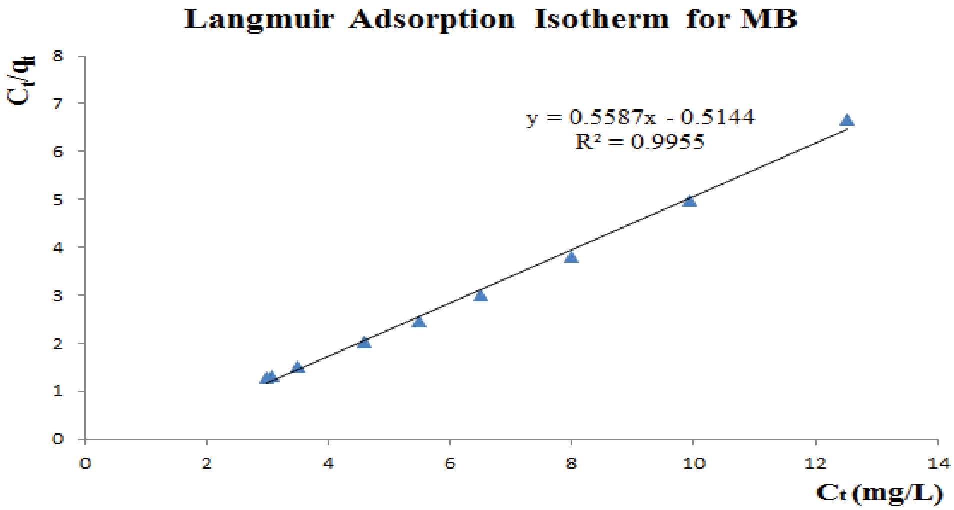

Figure 7: Langmuir Isotherm Plot for Adsorption of MB Dye onto SOSAC

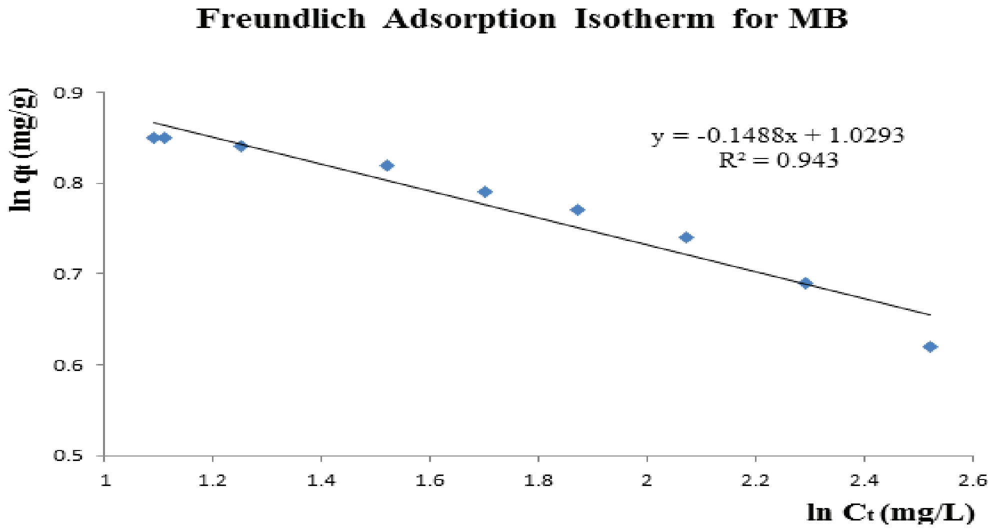

Figure 8: Freundlich Isotherm Plot for Adsorption of MB Dye onto SOSAC

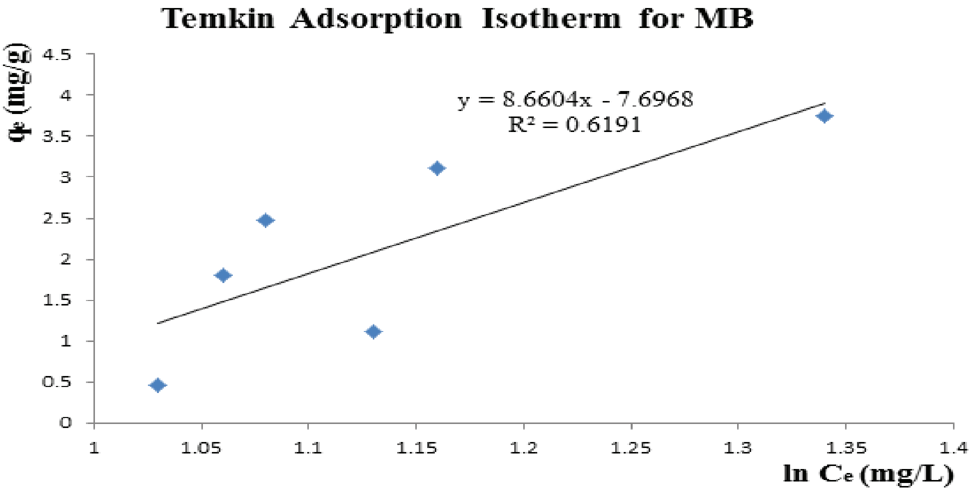

Figure 9: Temkin Isotherm Plot for Adsorption of MB Dye onto SOSAC

The plots of Langmuir, Freundlich and Temkin isotherm are shown from the Figs. 7–9 and the isotherm constants including the correlation coefficient (R^2) obtained are presented in the Table 1. The results have clearly indicated that the adsorption of MB dye onto SOSAC fits well with the Langmuir model and hence, suggests monolayer and chemical adsorption.

Table 1: Langmuir, Freundlich and Temkin isotherm constants for the adsorption of MB dye onto SOSAC at 303K

Models	**Parameters**	**Methylene Blue Dye**
Langmuir	K_L (Lmg^{-1})	1.09
	q_m (mg/g)	1.79
	R_L	0.01
	R^2	0.9955
Freundlich	K_f (Lg^{-1})	2.80
	1/n	0.148
	N	6.72
	R^2	0.9430
Temkin	K_T ($L.mg^{-1}$)	2.43
	b_T (J/mg)	286.08
	B_1 ($J.mol^{-1}$)	8.66
	R^2	0.6191

3.7 Kinetic Studies

3.7.1 Pseudo First-order Kinetic Model

In this study pseudo-first-order, pseudo-second–order, and intra-particle diffusion were applied to examine the controlling mechanism of dye adsorption from aqueous solution. Lagergren's first order rate equation is the earliest known to describe the adsorption rate based on adsorption capacity.

The linear form of Lagergren's first order equation is as in the eq. 8 (Lagergren, 1898):

$$\ln (q_e - q_t) = \ln q_e - K_1 t \quad (8)$$

where; q_e is the amount of dye adsorbed onto the adsorbent at equilibrium (mg/g), q_t is the amount of dye adsorbed onto the adsorbent at any time t (mg/g), and K_1 (min^{-1}) is the rate constant of the pseudo first-order adsorption which can be calculated from the slope of the linear plot of ln (qe–qt) against t which gives K_1 as slope and q_e as intercept.

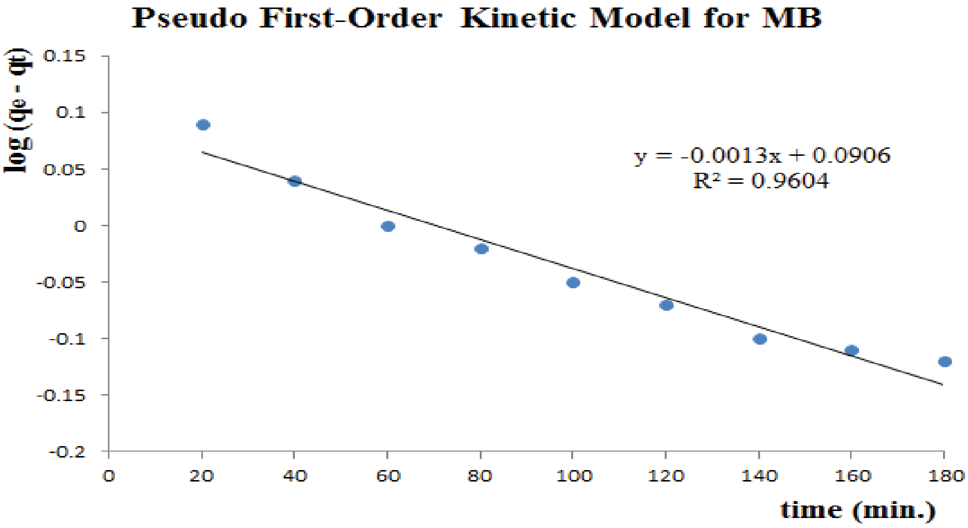

Figure 10: Pseudo-first Order Kinetics Model Plot for Adsorption of MB Dye onto SOSAC

3.7.2 Pseudo Second-order Kinetic Model

Ho *et al*., (2000) proposed a second order model for the sorption of divalent adsorbate ions or molecules onto peat particles based on the adsorption capacity of the adsorbents with the goal of differentiating the kinetics of a second-order rate expression based on the adsorbent concentration from models which are based on the solute concentration and represent a pseudo second-order rate expression.

The linear form of the pseudo second-order model can be as given by Ho *et al*., (2000):

$$t/q_t = 1/K_2 qe^2 + 1/q_e (t) \quad (9)$$

where; K_2 ($gmg^{-1}min^{-1}$) is the rate constant of the pseudo-second-order adsorption, q_e is the amount of dye adsorbed on the adsorbent at equilibrium (mg/g), and q_t is the amount of dye adsorbed on the adsorbent at any time, t (mg/g). K_2 ($gmg^{-1}min^{-1}$) can be calculated from the slope and intercept of the plot of t/qt against t.

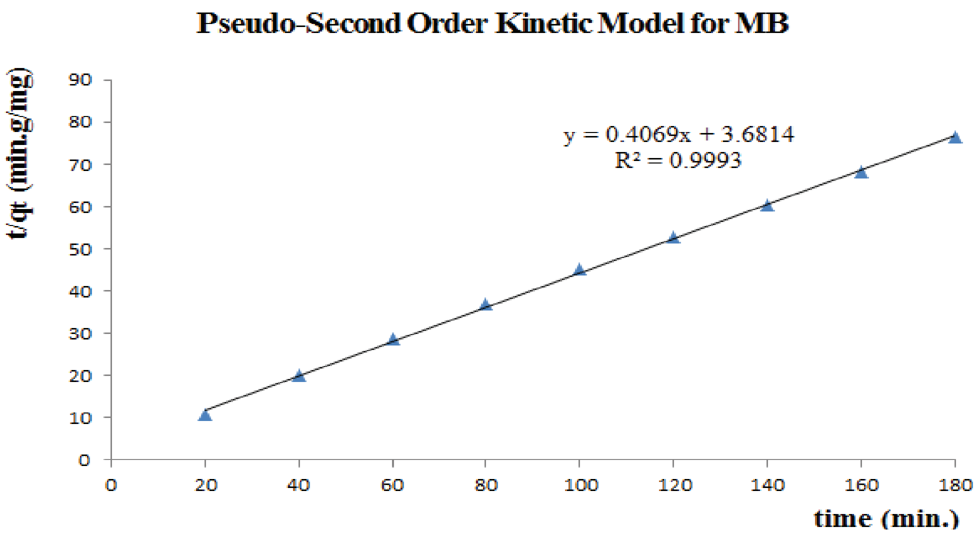

Figure 11: Pseudo Second-order Kinetics Model Plot for Adsorption of MB Dye onto SOSAC

3.7.3 Intra-particle Diffusion Model

The intra-particle diffusion model as put by Weber and Morris, (1962) is applied to describe the competitive adsorption involved between a liquid-solid system, which they believed that the adsorption of a fraction of solute onto the particle surface depends on; diffusivity of the solute within the particle and the particle radius.

The logarithmic form of the intra-particle diffusion model is given as eq. 10 (Weber and Morris, 1962):

$$\log q_t = \log k_{id} + 0.5 \log t \quad (10)$$

where; qt is the amount of dye adsorbed onto the adsorbent at time, t (mg/g), C_{id} is the intercept, and K_{id} is the intra-particle diffusion rate constant (mg.g^{-1} min^{-1}).

The plot of log q_t against 0.5 log t should yield a straight line with a positive intercept for intra-particle diffusion controlled adsorption process. k_{id} is determined from the intercept of the plot.

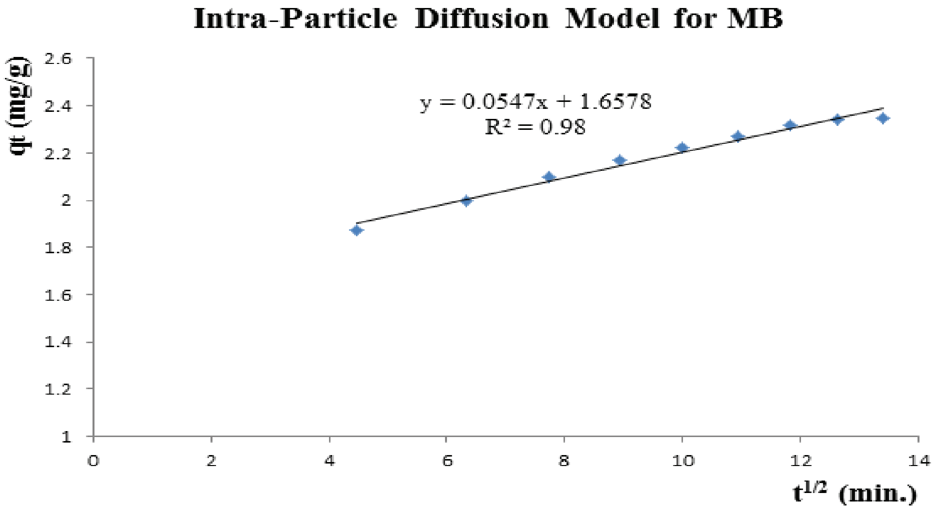

Figure 12: Intra-particle Diffusion Kinetics Model Plot for Adsorption of MB Dye onto SOSAC

Table 2: Constants from Kinetic Studies of MB Dye Adsorption onto SOSAC at 303K

Models	Parameters	Methylene Blue Dye
Pseudo-first Order	K_1 (min^{-1})	1.30 × 10^{-3}
	q_e (mg/g)	1.10
	R_2	0.9604
Pseudo-second Order	K_2 (g.mg^{-1}min^{-1})	4.49 × 10^{-2}
	q_e (mg/g)	2.46
	R^2	0.9993
Intra-particle Diffusion	K_{id} (mg.min^{-1}g)	0.055
	C_{id} (mg/g)	1.66
	R^2	0.9801

4. Conclusion

Removal of MB dye from aqueous solutions by batch adsorption process using SOSAC has been experimentally studied and the following conclusions were reached;

1. The percentage of MB dye removed increases with increase in adsorbent dosage, contact time and temperature while, varied with pH of the dye solution. The higher the pH, the greater the removals of MB dye by adsorption. The adsorption of these positively charged dye groups on the adsorbent surface is primarily influenced by the surface charge on the adsorbent which in turn is influenced by the solution pH.
2. Optimum contact time for equilibrium was achieved at around 160 min. It is basically due to saturation of the active sites which did not allow further adsorption. Optimum adsorbent dose for the dye is 1.5g/100 ml. It is obvious that with increasing amount the active sites for adsorption of the dyes increases which results in an increase in removal efficiency. The decrease in adsorption capacity with an increase in the adsorbent concentration could be ascribed to the fact that some of the adsorption sites remained saturated or not really involved during the process and agglomeration of activated carbons as a result all the surface area is not available for adsorption process. The optimum pH value and temperature were found to be at 11 and 50 ℃ respectively.
3. Langmuir was found among the three isotherm models tested to be the best that described the adsorption process while pseudo second-order when compared with pseudo first-order and intra-particle diffusion kinetic models was found to be the best suitable model that described the kinetic process of the adsorption.

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