LAUTECH Journal of Civil and Environmental Studies DOI: 10.36108/laujoces/9102/20(0270)
Volume 3, Issue 1; September 2019

Adsorption of Methylene Blue from Aqueous Solution using Microwave-Assisted $BaCl_2$ Modified Activated Carbon Produced from Mango Seed Shell

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Abstract

The presence of methylene blue (MB) in wastewater is a major concern in the environment due to its low biodegradability and harmful effect on man. The adsorption of MB from aqueous solution onto activated carbon (AC) prepared from mango seed shells sourced locally in Ogbomoso Township was investigated in this study. The AC was prepared from mango seed shell by activating with $BaCl_2$ at impregnation ratio I.R (1:2 w/w), microwave frequency (600 Hz), and time (30 min) before being carbonized at 500 °C for 20 min. Batch adsorption experiment was carried out at 25 °C to study the effect of contact time and initial dye concentration on MB adsorption. The equilibrium kinetics and adsorption mechanism were investigated. The pseudo first order, high correlation coefficients, *R,* (0.9491 and 0.9907) for initial concentrations of 10 and 15 mg/l, respectively, was very suitable to describe the kinetic characteristics of the MB adsorption on to mango seed shell activated carbon, Weber and Morris intra-particle model with *R* of 0.9907, is very fit to describe the diffusion mechanism of the adsorption process. The result implied that mango seed shell was suitable as an adsorbent material for adsorption of MB.

Keywords: Activated carbon, Mango seed shell, Methylene blue, Optimization

List of Notations

C_0 (mg/L) is the initial concentrations methylene blue solution

t is time,

Ce (mg/L) is the concentration of the methylene blue solution at equilibrium time,

V (L) is the volume if the solution

M (g) is the mass of the adsorbent

q_e (mg/g) is the amounts of adsorbate adsorbed at equilibrium

q_{cal} (mg/g) is the amounts of adsorbate adsorbed calculated from graph

q_t (mg/g) is the amounts of adsorbate adsorbed at any time t

K_1 (h^{-1}) is the adsorption rate constant

K_2 (g/mg.h) is the rate of second-order adsorption

K_{id} ($mg/gh^{½}$) is the intra particle diffusion rate constant.

C is constant for the boundary layer property

Introduction

Dyes are widely used in most manufacturing industries to improve the aesthetics of their product in terms of colour presentation for consumers' choices. Many of the organic dyestuffs are harmful to human beings and toxic to microorganisms (Ashraf, *et al.,* 2019). The presence of these colored compounds in wastewater impede light penetration to the bottom of the water stream (Ashraf, 2016). Cases of increase in the biological oxygen demand (BOD) which cause lack of dissolved oxygen to sustain aquatic life in water stream due to the presence of dyes have been recorded (Al-Qodaha, *et al.,* 2007). Methylene blue (MB) is a cationic dye that is used commonly for coloring and dyeing cotton, wool, and silk (Vargas, *et al.,* 2011). The presence of MB water stream is very detrimental to humans as well as other terrestrial and aquatic and animals (Yi and Zhang, 2008).

Therefore, the treatment of effluents containing such dyes is of great interest due to its harmful impacts on receiving waters. Many treatment processes have been applied for the removal of dyes from wastewater such as, chemical coagulation/flocculation, ozonation, oxidation processes, ion exchange, reverse osmosis, adsorption/precipitation processes and ultra-filtration (Amuda and Alade, 2006) and (Zhu, *et al.,* 2007). However, adsorption on activated carbon has been chosen as the best because of its efficiency (Alabi *et al.,* 2019).

Adsorption has been considered as an attractive method to remove MB from aqueous solutions due to its low cost, simplicity of design, ease of operation, (EL Alouani, *et al.,* 2018). Several adsorbents such as plant wastes (Gupta, *et al.,* 2011), silica gel (Samiey and Toosi, 2010), clay (Tahir and Rauf, 2006), activated carbon (Altenor, *et al.,* 2009) and rice husk (Safa and Bhatti, 2011) have been studied for adsorption of methylene blue from aqueous solutions. Among the above mentioned adsorbents, AC is a major MB removal adsorbent due to its large specific surface area, low density, chemical stability, suitability for large scale production, variety of structural forms, and the ability to modify the pore structures (Okeowo, *et al.,* 2017).

The search for less economical adsorbent has led to the increasing use of agricultural residue in the preparation of activated carbon in which mango seed shell is of no exception. Mango (*Mangifera Indica*) is a very common tropical fruit usually found in Nigeria. Its trees reach 35-40 m in height (Kittiphoom, 2012). The Mango seed is generally discarded in the environment and it constitute a major waste in the tropical rainforest zone of Nigeria (Kwaghger, *et al.,* 2012). The resulting volume of wastes due to the high consumption of Mango, constitute environmental nuisance of high degree of health impacts (Okonogi, *et al.,* 2007). This work was aimed at using activated carbon (AC) produced from mango seed shell to remove MB from water and evaluate some adsorption parameters such as kinetics and transfer mechanism in order to understand the adsorption process between the selected dye (MB) and the activated carbon developed.

Methodology

Chemicals and reagents

The main raw materials used for this study were mango seed shell. The MB ($C_{16}H_{18}N_3ClS$, MW of 319.85 g/mol) supplied by Merck was used as the adsorbate and while BaCl2 was supplied by Sigma-Aldrich. These reagents ($BaCl_2$ and methylene blue), were all of analytical grade.

Preparation of Adsorbent

The mango seed shell was sourced from LAUTECH Teaching and Research Farm, Ogbomoso, Oyo State. The mango seed shell was sorted to remove the stones then washed with distilled water to remove other impurities (Bullut and Tez, 2007). It was later sun-dried for 72 h. and then oven-dried at 105 ºC for 6 h. to constant weight (Tossou, *et al.*, 2019). Thereafter, it was milled to reduce its size and increase its surface area (Bullut and Tez, 2007) before activation and carbonization as stated in previous studies (Okeowo, *et al.,* 2017).

The mango seed shell precursor (50 g) was mixed with 100 ml of 0.1 M $BaCl_2$ in a beaker based on the impregnation ratio 1:2 (w/w) for 24 h (Üner *at al.,* 2016). The mixture was then heated in a Microwave oven set at 600 Hz for 30 mins to aid the extent of activation. The excess $BaCl_2$ solution was boiled off and the mixture was oven-dried at 110 °C for 2 h (Gumus and Okpeku, 2015), before being washed with distilled water to remove the activant and then oven-dried to constant weight. The $BaCl_2$–modified mango seed shell precursor (8 g) was used to fill about 70% of crucibles volume and then charged into the muffle furnace at 500 °C and 20 min (Okeowo, *et al.,* 2017). The carbonised $BaCl_2$–modified mango seed shell produced was cooled in a desiccator before being crushed, washed and screened to the desired size of 200 µm.

Preparation of MB solution

Stock solution of concentration of 50 mg/L was produced by dissolving 50 mg of methylene blue in 1 L of distilled water (De Castro *et al.,* 2018). Desires serial dilution was carried out appropriately and calibration curve was generated from the prepared concentration of 10-50 mg/L and was done by plotting absorbance against concentration (Figure 1). This curve was used to determine the concentration of methylene blue solution at equilibrium time, subsequently.

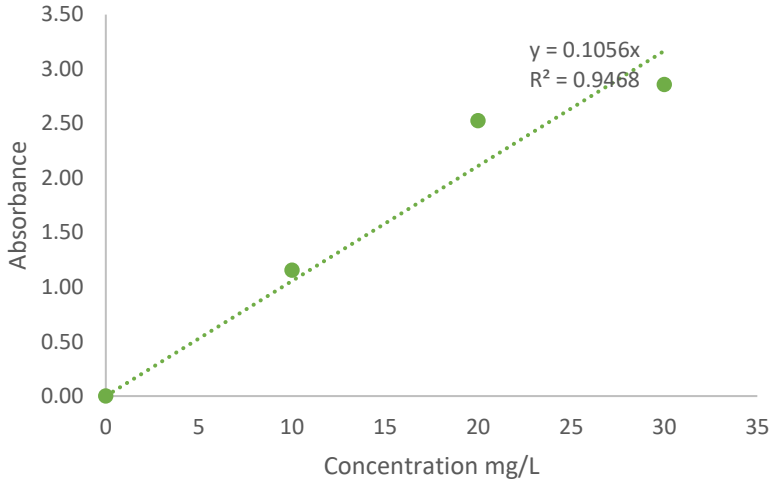

Figure 1: Calibration plot of methylene blue solution at 645 nm

Adsorption Experiment

The adsorptive capacity of the activated carbon was determined by placing 100 mg of the activated carbon in contact with 10 mL of 10 mg/L of methylene blue solution for 24 hours at 25 °C room temperature. The concentration of dye remaining in the solution was determined at 645 nm with UV/V spectrophotometer. Equation 1 was used to calculate the amount of methylene blue adsorbed from each solution.

$$q_e(mg/g) = (C_0 - C_e) \times \frac{V}{M} \qquad (1)$$

Where C_0 (mg/L) is the methylene blue solution at initial time (t = 0), Ce (mg/L) is the concentration of the methylene blue solution at equilibrium time, V (L) is the volume if the solution and M (g) is the mass of the adsorbent and q_e (mg/g) is the adsorption of the methylene blue.

The effects of contact time, and initial dye concentration (10-20 mg/L) on adsorption capacity of mango seed shell activated carbon was determined. The dye solution (100 ml) of a known concentration was poured into a 250 ml conical flask, and mixed with the mango seed activated carbon (1.0 g). The flask and its content were placed on a rotary shaker and agitated at constant speed of 150 rpm, for 8 h at room temperature. Each flask was withdrawn from the shaker at predetermined time

intervals of 30 mins. The dye solution was decanted and its absorbance was then measured using UV-spectrophotometer at 645 nm wavelength (Üner *et al.,* 2016).The actual concentrations of the unadsorbed dye solutions were deduced from the standard curve.

Kinetic models

The kinetic data were analyzed using Pseudo-First Order (PFO) and pseudo-second order (PSO) models, expressed in equations 2 and 3 (Ejikeme, *et al.,* 2014), in order to understand the dynamics of the adsorption reactions with respect to the order of the rate constant.The plot of $ln(q_e - q_e)$ against t gave a slope of K_1 and an intercept of lnq_e (Alabi *et al.,* 2019). The linear plot of t/q_t against t gave $1/q_e$ as the slope and $1/K_2q_e^2$ as the intercept (Alabi *et al.,* 2019) which was used to predict the behaviour of the adsorption process.

$$\ln(q_e - q_e) = lnq_e - K_1 t \quad (2)$$

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + t/q_e \quad (3)$$

Where q_e and q_t (mg/g) are the amounts of adsorbate adsorbed at equilibrium and any time t (hr.) respectively, and K_1 (h^{-1}) is the adsorption rate constant. K_2 (g/mg.h) is the rate of second-order adsorption.

Mass Transfer mechanism

Various adsorption cases indicates that solute uptake varies almost proportionally with t½ rather than with the contact time, t and the model is expressed in Eqn (4). K_{id} and C were obtained as slope and intercept (mg/g) of the plot of q_t versus t ½. Value of C gives the idea about the thickness of boundary layer and the larger the intercept, the greater the contribution of the surface sorption in the rate controlling step (Ejikeme *et al.,* 2014).

$$q_t = K_{id}\, t^{1/2} + C \quad (4)$$

Where K_{id} is the intra particle diffusion rate constant ($mg/gh^{½}$).

Results and Discussions

Effect of initial Concentration on MB adsorption

The effect of initial dye concentrations of 10 mg/L and 15 mg/L on the prepared mango shell activated carbon was determined in this study and the results indicated that the equilibrium adsorption capacity (q_e) decreased with increasing initial dye concentration for the first 7 h., but increases for the next one hour. (Figure 2). The low adsorption capacity at the initial high concentration was due to the overload concentration of the dye molecules on the adsorbent surface sites which disturbed the adsorption process. The initial dye concentration plays an important role which provide the driving force to overcome the resistance to mass transfer of the dye between the aqueous and solid phases. Therefore, at higher initial concentration, more ions compete for the available sites on the surface of adsorbent, hence, more adsorbate were adsorbed due to high pressure that forced the ions inner the pores of the adsorbents (Idris, *et al.,* 2011).

Effect of Contact Time on MB adsorption

The result revealed that there was rapid uptake of adsorbate at the initial stage of the contact time for both concentrations (Figure 2) (Küncek and Sener, 2010). This may be linked to higher number of adsorption sites that were available at the initial contact time of the experiment, and after a lapse of time the number of active sites became less and the adsorbent became crowded thus impeding the movement of the adsorbate (Kennedy, *et al.,* 2007). It was observed that the adsorption capacity of 15 mg/L has increased to 1.28-1.35 mg/g, at contact time between 450 and 480 mins, while that of 10

mg/L increased slightly from 1.25-1.28 mg/g. Similarly, the q_e of the mango seed shell activated carbon, increased as the time increased and this is similar to the trends reported from the studies of Üner *et al.,* (2 016)., for the removal of MB using waste water melon rind.

The maximum qe (1.28 and 1.35 mg/g)obtained in this study were higher than 0.199, 0.51, 0.80, 0.84, 1.10, 1.17, 1.21 and 1.33 mg/g of MB adsorbed on acid-bound iron oxide magnetic nanoparticles, corncob based activated carbon, fly ash, Chrome sludge, eggshell and eggshell membrane polyacrylic, living biomass, fir wood based activated carbon and almond shell-activated carbons (Mak and Chen 2004; Tseng *et al.,* 2006; Woolard *et al.,* 2003; Lee, *et al.,* 1996; Tsai, *et al.,* 2006; Fu and Viraraghavan, 2000; Wu, and Tseng 2008; and Aygun, *et al.,* 2003, respectively).

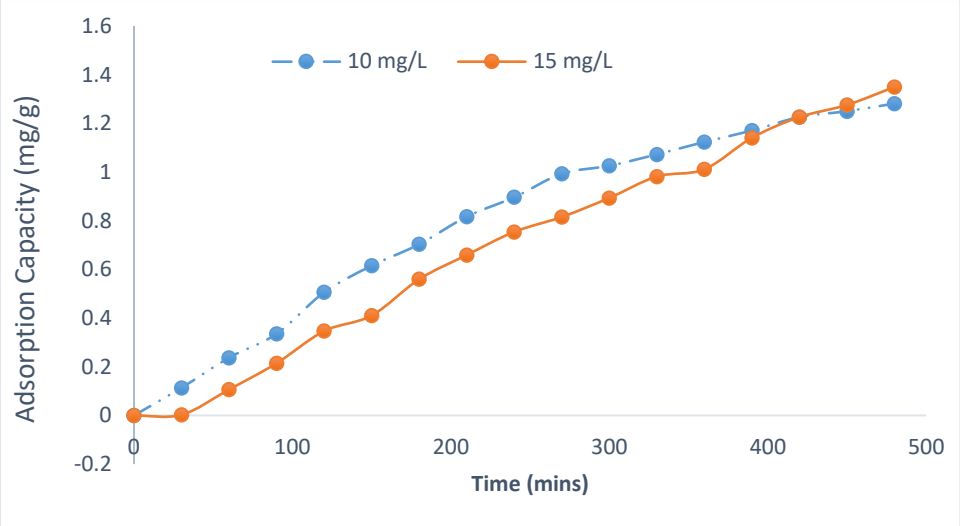

Figure 2: The effect of contact time and initial dye concentration on the adsorption of MB dye onto AC from mango seed shell

Kinetics Studies

Pseudo first order

The plots of ln (q_e-q_t) versus t (Figure 3) gave straight lines with values of K_1 and q_{cal} were calculated from the slopes (K_1) and intercepts (ln q_e) of the plots respectively, and are presented in Table 1. The plots showed high correlation coefficients, *R,* (0.9491 and 0.9907) for initial concentrations of 10 and 15 mg/l, respectively. The low difference between the calculated ($q_{e(cal)}$) and the experimental ($q_{e(exp)}$) adsorption capacity as well as higher values of *R* suggested that the pseudo first order was very suitable to describe the kinetic characteristics of the MB adsorption on to mango seed shell activated carbon (Gupta and Abdul Rafe, 2013).

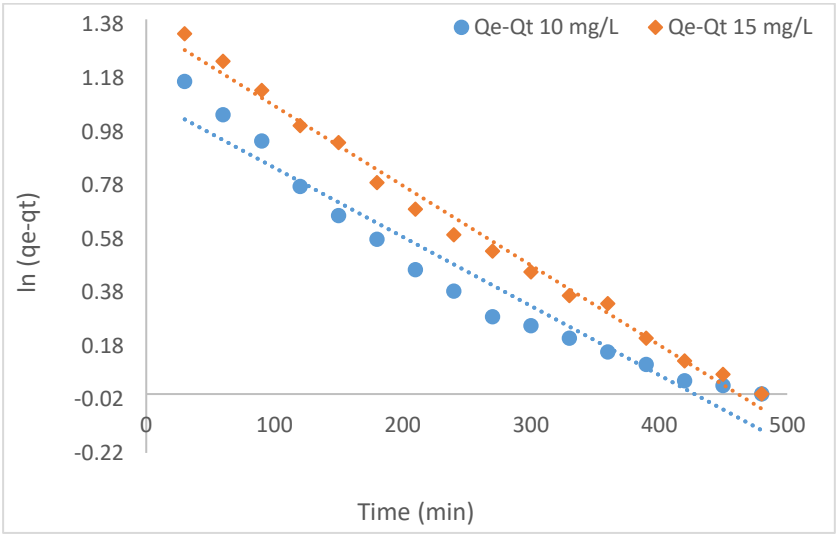

Figure 3: Plot of $\ln(q_e - q_t)$ against time for the adsorption of MB onto AC

Pseudo second order

The values of 'K_2' and q_e (Table 1) for the pseudo-second order model were calculated from the intercepts ($1/kq_e^2$) and slopes ($1/q_e$) of the plots of t/q_t vs. t (Fig. 4). The R^2 values for initial concentrations of 10 and 15 mg/L were 0.1848 and 0.8213 respectively. The low R^2 values for both concentrations indicate that adsorption characteristics of MB onto the mango seed shell activated carbon does not fit well to the pseudo second order model even though the calculated q_e of the high concentration was in agreement with experimental q_e (Gupta and Abdul Rafe, 2013).

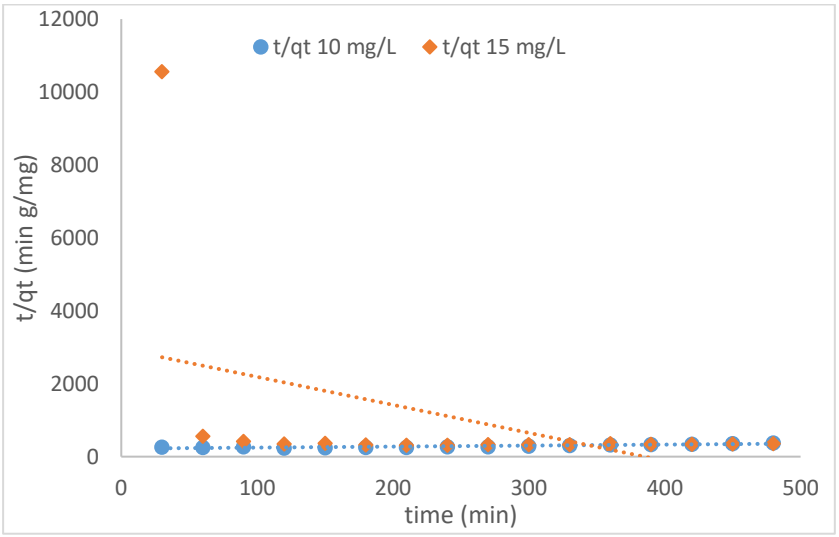

Figure 4: Plot of t/q_t against time for the adsorption of MB onto mango seed shell activated carbon

Elovich Model

The plot of q_t against ln t (Figure 5) provides a linear relationship which α and β are determined from the slope and intercept of the plot. The initial adsorption rate (α) and the desorption constant (β) increased from 0.0105 to 0.0136 mg/g min and 1.9231 to 2.1222 g/mg as the concentration decreases from 15 to 10 mg/L (Table 1). The R^2, of the Elovich models are 0.9655 and 0.9221 respectively as compared to result of Li *et al*. (2013), which reported R^2 lesser than 0.9752. This high correlation

coefficient value suggested that this model is applicable to fit the kinetic experimental data. Similarly, it has higher correlation coefficient at low initial concentration.

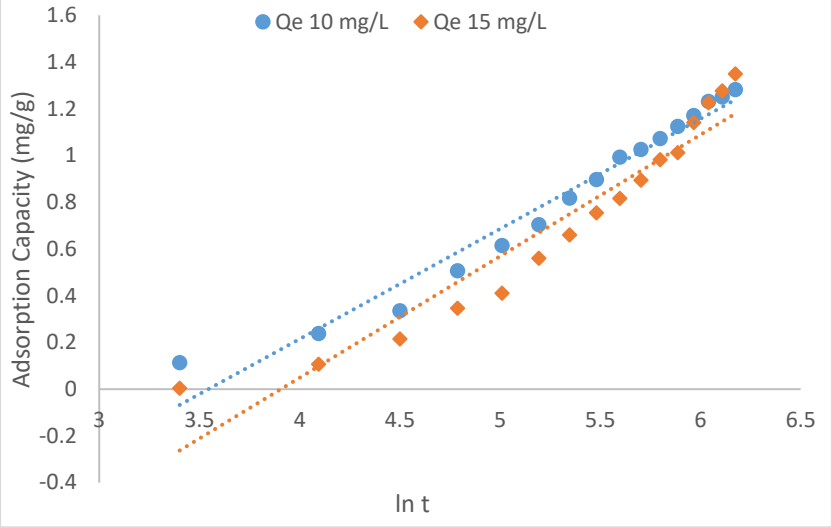

Figure 5: Plot of q_e against ln t for the adsorption of MB onto AC

Table 1: Parameters and Correlation Coefficients (R) obtained for the Kinetic models

	Constants	Methylene Blue Concatenations	
		10 mg/L	15 mg/L
	$q_{e(exp)}$ (mg/g)	1.2813	1.3494
Pseudo first order	$q_{e(Cal)}$ (mg/g)	3.0153	3.9539
	K_1 (h^{-1})	0.0026	0.003
	R^2	0.9491	0.9907
Pseudo second order	$q_{e(Cal)}$ (mg/g)	-0.1303	3.6311
	K_2 (g/mg.h)	0.0199	1.3×10^{-3}
	R^2	0.1848	0.8213
Elovich	α (mg/g min)	0.0136	0.0105
	β (g/mg)	2.1222	1.9231
	R^2	0.9655	0.9221

Adsorption mechanism

Weber and Morris intra-particle diffusion resistance model was used to determine the influence or identifying the adsorption mechanism, and predicting the rate controlling step in adsorption of phenol onto the activated carbon. The values of k_i were calculated from slopes (k_i) of the plots of q_t vs. $t^{0.5}$ (Figure 6) and are presented in Table 2. The plot shows that the linear plot did not pass through the origin which indicated that the intra particle diffusion was not only the rate controlling step and the boundary layer diffusion controlled the adsorption (Kushwaha, *et al.,* 2014) (Figure 6). The value of C increased from -0.5658 to -0.3155 with decrease in concentration from 10 to 15 mg/L (**Table 2)**. The *R* of the two concentrations was the same (0.9907) and this high value indicated that the model is very fit to describe the diffusion adsorption stage of MB through the solution to the external surface of the mango seed shell activated carbon.

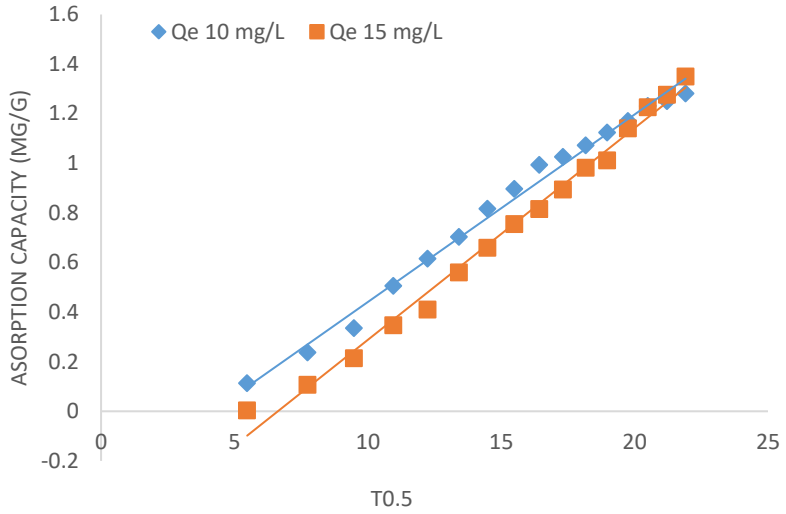

Figure 6: Plot of q_t against $t^{0.5}$ for the adsorption of MB onto AC

Table 2: Weber and Morris Intraparticle Diffusion constants

Methylene Blue Concatenations (mg/L)	K_i	C	R^2
10	0.0756	-0.3155	0.9909
15	0.0854	-0.5658	0.9909

Conclusion

The activated carbon was produced by chemical activation of mango seed shell, successfully in this study. Batch adsorption experiments showed that the adsorption of MB onto AC was dependent on contact time and initial dye concentration. The pseudo-first-order model, pseudo-second-order model, and Elovich model were applied to study the kinetic of the adsorption. The results illustrated that the adsorption of MB onto the produced AC fit better to the pseudo-first order. The intra-particle diffusion model showed that pore diffusion is not the sole rate- controlling step and that boundary layer diffusion controlled the adsorption to some degree. It can be deduced that mango seed shell is a good starting material in the production of activated due to its high yield and good adsorption capacity.

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