Kinetic Studies of Methylene Blue Adsorption on to Activated Carbon Prepared from Plantain Pod (Musa paradisiac)

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Author’s contribution

This work was carried out by the author and has read through the final manuscript.

ABSTRACT

The mechanism of adsorption for methylene blue from aqueous solution using an activated carbon prepared from plantain pod was studied at varying time. The adsorption reached at equilibrium in 90 minutes of contact time. The kinetic data were fitted to the pseudo first-order, pseudo second-order, and intra-particle diffusion models. The pseudo first and second order rate constants were found to be 0.0152 min\(^{-1}\) and \(4.22 \times 10^{-5}\) g/mg.min with correlation coefficient of 0.930 and 0.931 using dye concentration of 75 mg/l. Isotherm studies were also conducted by increasing the mass of adsorbent. The experimental data obtained at temperature of 30ºC were fitted in to Langmuir, Freundlich, Tempkin and Dubinin-Raduskevich adsorption isotherm models respectively. The correlation coefficient (\(r^2\)) of Langmuir, Freundlich, Tempkin and Dubinin-Raduskevich adsorption isotherm models were found to be 0.993, 0.991, 0.999, and 0.986 respectively. Based on Dubinin-Raduskevich adsorption isotherm model, mean free energy was determined to be 0.03535 J/mol, < 8 the adsorption process can be described as physisorption.

Keywords: Plantain pod; activated carbon; methylene blue dye; adsorption; kinetics.

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1. INTRODUCTION

Waste water effluent from dyeing or textile industries on both land and water bodies can be a serious threat to the environment. Effluent from these industries when released into other water bodies; has negative effect on human health due to their turbidity level, toxic nature. [1,2] and non–biodegradable nature. Organic colorants are mainly used in textile, cosmetics and paper industries and among the organic dyes employed in manufacturing processes; methylene blue is the most widely used. There have been various technological methods: such as reverse osmosis, electro chemical coagulation, nano filtration and adsorption used to treat waste water. Among these methods; is the adsorption process which has gained considerable interest in recent times due to the high level of treatment and degree of reliability which can meet stringent environmental emission standards [3,4]. The method of adsorption is reported to be feasible and economically viable for treatment of effluent from industrial processes [5,6]. Several researchers have attempted the use of agricultural waste and plant materials; that are not useful; to prepare activated carbon to remove reasonable amount of dye from industrial effluent. These plant materials are renewable; thus, the low cost of activated carbon production is highly attractive, compared to conventional materials such as wood, coal etc. Sourcing for low cost adsorbent will contribute to sustainability of the environment since these constitutes disposal problems. Thus, it is an advantage using waste to product approach; a cleanup excise to the environment and would offer benefit for commercial purposes in the future [7]. Some of the waste materials used in the recent past are: palm shell [8] silkworm [9] Nigeria bamboo [10] orange waste [11] fluted pumpkin stem waste [12,13] raphia palm fruit endocarp [14] wild cocoyam [15] coconut fibre [16] waste of rapeseed [17] maize cob [18] and snail shell [19].

Several experimental studies have been conducted on the isothermal batch adsorption of model waste water (methylene blue dye) using different activated carbons; waste weed [20] sindora siamensis seed and canarium sublatum guillaumin fruit [21], agrowaste [22], banana empty fruit bunch and delonix regia fruit pod [23]. Despite the numerous studies on the adsorption methylene blue dye, the use of activated carbon prepared from plantain pod is limited. The plantain pod (Musa paradisiac) is a waste obtained from plantain fruit bunch. Plantain is a stable food in the southern part of Nigeria. It is in abundance, grows or cultivated and contains iron; recommended food for the elderly, either cooked or made in to powdered form for preservation purposes. The pod is the waste after the edible part has been removed. Depending on the size of the plantain, the pod could be as long as 2 m to 3 m, with an oval shape. The disposal of this waste constitutes an environmental problem.

The exploration of waste materials is inexhaustible in Nigeria; being a developing country with the confronting waste management problems. Therefore there is need to employ available low cost biomass materials for the production of activated carbon for waste water treatment systems. The purpose of this study is to use activated carbon from plantain pod for kinetic and isotherm for adsorption of methylene blue. Three kinetic models- pseudo first order, pseudo second order and intraparticle diffusion models are used to identify the potential rate controlling steps in this work. Whilst Langmuir, Freundlich, Tempkin and Dubinin- Raduskevick models were used for the isotherm studies.

2. EXPERIMENTALS

2.1 Materials and Methods

The materials used for the preparation of activated carbon include: plantain pod, (Musa paradisiac); obtained from Amassoma farmers, laboratory grade phosphoric acid, oven, Muffle furnace, thermometer and filter paper, The plantain pod was washed thoroughly with warm water and sun dried for 10 min and pulverized into smaller sizes. The sundried samples were oven dried at 120ºC for 12 hours to ensure complete loss of the water content or to a minimum.

2.2 Carbonization and Chemical Activation

The dried material was carbonized using a laboratory Muffle furnace at 400ºC for 2 hours. The material was allowed to cool, ground and sieved to 300 µm size particles. This process was repeated at 500ºC. The carbonized samples at temperatures of 400ºC and 500ºC were soaked in 20% H₃PO₄ solution for 24 hours. It was then washed with distilled water to remove all traces of acid and activated at 600ºC, 700ºC
and 800°C for 30 minutes in a muffle furnace. The activated carbon samples were characterized and stored in a tight container for further adsorption processes.

### 2.3 Characteristics of Activated Carbon

The characteristics properties of the activated carbon samples were studied using methods described earlier by the author [24]. The adsorbate (methylene blue) was used to construct calibration curve using varying concentrations. The calibration curve for methylene blue concentration versus absorbance is shown Fig. 2.

### 2.4 Batch Adsorption Studies

The adsorption study of the activated carbon was conducted using a fresh prepared methylene blue of 75 mg/L concentration and placed in a shaker with a dosage of 20 mg. at room temperature. The effect of contact time was performed with the same dye concentration of 75 mg/l by varying the time. The solution was withdrawn at predetermined time intervals ranging from 30, 60, 90, 120 and 150 min respectively. The equilibrium dye concentrations were estimated from the absorbance recorded at wavelength between 450 nm to 530 nm, using a spectrophotometer (JASCO UV/Vis-550 model)
and the calibration curve. The effect of adsorbent dosage on the adsorption of methylene blue dye on the prepared activated carbon, was also conducted by varying the dosage from 30 mg – 60 mg. The amount of dye adsorbed and percentage were determined with equations 1 and 2.

\[ q_e = \frac{(C_0 - C_e) \times V}{m} \]  \hspace{1cm} (1)

\[ q_t = \frac{C_0 - C_t}{C_0} \times 100 \]  \hspace{1cm} (2)

Where

- \( C_0 \) = Initial dye concentration (mg/L), \( C_e \) = equilibrium concentration (mg/l), \( C_t \) = Final dye concentration at time, \( t \) (mg/L), \( q_e \) = Amount of dye adsorbed at equilibrium \( q_t \) = Amount of dye adsorbed at equilibrium time, \( m \) = Mass of adsorbent (mg), \( V \) = Volume of dye solution (ml).

### 3. RESULTS AND DISCUSSION

#### 3.1 Characterization of Activated Carbon from Plantain Pod

Table 1 shows the properties of the activated carbon produced from plantain pod (Musa paradisiac) at carbonization temperature of 400°C and 500°C. It is clear that temperature has great effect on porosity, pore volume and bulk density. Increasing the activation temperature from 600°C to 700°C reduces the moisture content from 0.42 to 0.30 and showed increase to 0.60 when temperature was further increased to 800°C. All other properties; ash content, porosity, pore volume and bulk density increased with increase in temperature. High ash content in activated carbon can reduce the overall activity [25]. They report that a good activated carbon contains ash content ≤ 8. The activated carbon in this study has ash content between 2.07 -1.67, which is less than 8. Similarly, the pore volume and porosity increased with temperature and these values (0.35 – 0.45) pore volume; are comparable to work reported in literature [25] with activated carbon from Bamboo. The bulk density increased and it is higher compared to values found in work by others [25]. Similar results were obtained with samples carbonized at 500°C but the moisture content decreased and remained constant; with further increase in temperature. In all cases, higher values were obtained with samples carbonized at 500°C.

| Carbonization temperature (°C) | 400   | 500   |
|--------------------------------|-------|-------|
| Activation temperature (°C)    | 600   | 700   | 800   | 600   | 700   | 800   |
| Moisture content               | 0.42  | 0.30  | 0.60  | 1.10  | 0.40  | 0.40  |
| Ash content                    | 2.07  | 1.74  | 1.67  | 4.70  | 2.21  | 2.15  |
| Porosity                       | 0.18  | 0.20  | 0.23  | 0.23  | 0.24  | 0.25  |
| Pore volume                    | 0.35  | 0.40  | 0.45  | 0.45  | 0.49  | 0.50  |
| Bulk density                   | 1.98  | 1.98  | 1.99  | 1.99  | 2.00  | 2.01  |

#### 3.2 Effect of Dosage and Contact Time

Adsorption is strongly influenced by the dose of the adsorbent. In Fig. 2 it is clear that with increase in dosage the adsorption also increased. An initial adsorption of 37.39% was observed which increased to 40.88% and linearly increased to 49.39 % with an increase in dosage. Further increase in dosage can still increase adsorption because optimum quantity was not achieved even with 0.6 g. This may be due to available adsorption sites.

The effect of contact time on methylene blue adsorption on to activated carbon was also studied and is presented in Fig. 4. Adsorption increases with the increase in stirring time up to 120 min. As can be seen, the rate of adsorption increased rapid with most of the dye being adsorbed within the first 120 min. The percentage adsorbed increased to 67.48%, thereafter the rate of adsorption tend to be slow. This may be due to saturation of sites, which does not allow further adsorption to take place.
3.3 Kinetic Studies

Kinetic models have been proposed by many research groups to elucidate the mechanism of adsorbate adsorption. The rate and mechanism of adsorption is controlled by various factors such as physical and chemical properties of the adsorbent, the ambient temperature of the medium, the solution pH, and the nature of adsorbate. These kinetic models are useful for the design and optimization of effluent treatment process. In this study, the adsorption of methylene blue onto activated carbon prepared from plantain pod (*Musa paradisiaca*), was performed with the following kinetic models: Pseudo first-order kinetic model, Pseudo second-order kinetic model, intra particle diffusion model to determine the rate controlling step.

3.3.1 Pseudo first-order kinetics model

The pseudo-first order kinetic model was proposed and the integrated linear form is given in equation 3

$$\log \left( q_e - q_t \right) = \log q_e - \frac{k_1}{2.303} t$$

(3)
$k_1$ is the first order kinetic constant (min$^{-1}$), $t =$ time (min), $q_e =$ amount adsorbed at equilibrium, $q_t =$ amount adsorbed at equilibrium time. The plot of $\log (q_e - q_t)$ versus $t$ (See Fig. 5) gives a straight line and the rate constant $k_1$ and $q_e$ adsorptive capacity can be determined from the slope and intercept. From the graph, $k_1$ was found to be 0.0152 min$^{-1}$ and adsorptive capacity of 0.7516 mg/g, with coefficient of determination, $R^2 = 0.9302$. The calculated and experimental $q_e$ values showed high deviation with coefficient of determination value of $R^2 = 0.930$ (See Table 2) which suggests that the adsorption data fitted poorly to pseudo first-order kinetics.

### 3.3.2 Pseudo second-order kinetics model

The adsorption mechanism can also be described by the pseudo second-order kinetic model. The linearized form of the pseudo second-order kinetic model is given in equation 4

$$
\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t
$$

(4)

The plot of $\frac{t}{q_t}$ versus $t$ in Fig. 6 gives a straight line and the rate constant $k_2$ and adsorption capacity $q_e$ were determined from the slope and intercept. The initial adsorption rate $h$ (mg/g/min) is given as; $h = k_2 q_e^2$, whilst $k_2 =$ second order kinetic rate constant (g/mg.min). The pseudo second order constant $k_2$ was found to be $4.22 \times 10^{-5}$ g/mg.min with coefficient of determination $R^2 = 0.931$. The calculated adsorptive capacity for the pseudo second order model was higher 166.6 mg/g compared to pseudo first order model with slight increase in the coefficient of determination. The initial adsorption rate obtained was found to be 0.913. The results suggest that pseudo second order describes the adsorption of methylene blue on to PPAC much better than pseudo first order model.

### 3.3.3 Intra particle diffusion model

In the batch mode adsorption process, initial adsorption occurs on the surface of the adsorbent. In addition, there is also the possibility of adsorbate to diffuse into the interior pores of the adsorbent. Report in literature [26] suggested the following kinetic model to investigate if the adsorption is intra-particle diffusion.

$$
q_t = k_d t^{1/2}
$$

(5)

Where

$K_d =$ diffusion constant $t =$ time of contact.
Table 2. Results of kinetic parameters for adsorption of methylene blue onto PPAC, solution concentration = 75 mg/L

| $q_e$ exp(mg/g) | Pseudo first order | $q_e$ cal(mg/g) | Pseudo second order | Intra particle diffusion |
|----------------|-------------------|----------------|---------------------|-------------------------|
| $q_e$ cal(mg/g) | $K_1$ (min$^{-1}$) | $R^2$          | $K_2$ (g/mg.min)    | $h$ (mg/g.min)          | $R^2$ | $k_d$ | $R^2$ |
| 140.2          | 0.7516            | 0.0152         | 166.6               | 4.22x10^{-5}           | 0.9130 | 0.9312 | 7.1506 | 0.9765 |

Fig. 6. Pseudo Second-order kinetics model of methylene blue on activated carbon, size of particle = 300 µm
The plot of $q_e$ versus $t^{1/2}$ gives a linear plot that does not pass through the origin, presented in above Fig. 7. Similar results have been found in literature [27] and they attributed the deviation from origin to the variation of mass transfer in the initial and final stages of adsorption. The deviation indicates that pore diffusion is the only controlling step. The high coefficient of determination, $R^2 = 0.976$; is a good agreement that pore diffusion plays a major role in the adsorption of methylene blue on the activated carbon prepared from plantain pod. The intra particle diffusion rate was found to be 7.1506 g/mg. min. Similar findings were reported for the adsorption of basic red 29 on to EAAC with intra particle diffusion rate of 10.539 g/mg.min and $R^2 = 0.9847$ with concentration of 75 mg/L solution [28].

3.4 Isotherm Studies

The extent of adsorption was estimated using Langmuir, Freundlich, Dubinin-Rudushkevich and Tempkin models. Adsorption isotherm is the basic requirement for designing any adsorption system. Isotherm expresses the relation between the amounts of adsorbate removed from the aqueous solution by unit mass of adsorbent at constant temperature [29].

3.4.1 Langmuir isotherm

This model was originally developed to describe the adsorption of gas on to solid surface. It represents one of the theoretical treatments of non- linear sorption and suggests that uptake occurs on a homogeneous surface by monolayer sorption without interaction between adsorbed molecules. The model also assumes uniform energies of adsorption onto the surface and no transmigration of the adsorbent occurs [30]. The Langmuir isotherm is expressed as:

$$ q_e = \frac{Q_o b_L C_e}{1 + b_L C_e} $$

(6)

The linear form is given in equation 7

$$ \frac{C}{q_e} = \frac{1}{Q_o b_L} + \frac{C_e}{Q_o} $$

(7)

Where

- $C_e$ = equilibrium concentration
- $q_e$ = amount adsorbed at equilibrium
- $b_L$ = obtained from the intercept and $1/Q_o$ from the slope of the plot.

The essential characteristics of Langmuir isotherm can be expressed by a dimensionless separation factor $R_L$ (the value indicates favourability of the adsorption process) given in equation 8.
\[ R_L = \frac{1}{1 + b_L C_0} \]  

\( R_L = 1 \) Unfavourable, \( R_L = 1 \) linear, \( R_L = 0 \) irreversible, \( C_0 = \) initial concentration of methylene blue, \( b_L = \) Langmuir constant related to the energy of adsorption (L/mg). The quantity \( Q_o \) which is a constant related to the adsorption capacity, was found to be 71.42 mg/g, \( b_L = 0.041 \) (L/mg), determined from the slope and intercept of plot \( C_e/q_e \) versus \( C_e \) illustrated in Fig. 8. The coefficient of determination was found to be 0.993 while the value of \( R_L = 0.245 < 1 \), indicating a favourable adsorption process with high R² value, showing a monolayer coverage.

### 3.4.2 Freundlich adsorption isotherm

This model is the most popular model for a single solute system, based on the distribution of solute between the solid phase and aqueous phase at equilibrium. It is originally empirical in nature but was later interpreted as bio-sorption to heterogeneous surfaces or surfaces supporting sites of varied affinities and it is widely used for experimental data [31]. The Freundlich isotherm model is given as

\[ q_e = k_f C_e^n \]  

\( q_e \) is the amount adsorbed at equilibrium (mg/g), \( C_e \) is equilibrium concentration of the adsorbate, \( k_f \) is the measure of adsorption capacity and \( n \) is the adsorption intensity. The \( k_f \) and \( n \) values can be determined from the intercept and slope of plot with Log \( q_e \) versus \( C_e \) and the value was found to be \( k_f = 22.23 \) (mg g⁻¹ L⁻¹/²) and \( 1/n = -1.314 \) and \( n = -0.761 \) with coefficient of determination, \( R^2 = 0.991 \).

\[ \ln q_e = \log k_f + \frac{1}{n} \ln C_e \]  

### 3.4.3 Tempkin isotherm

This model proposed that the fall in the heat of adsorption is linear rather than logarithmic as stated in Freundlich expression. The heat of sorption of all the molecules in the layer would decrease linearly with coverage due to sorbate/sorbent interaction. The Tempkin adsorption isotherm is given in equation 11 [32]

\[ q_e = \frac{RT}{b_T} \ln \left( a_T C_e \right) \]  

The Linear form of the Tempkin isotherm model is given as

\[ q_e = \frac{RT}{b_T} \ln a_T + \frac{RT}{b_L} \ln C_e \]  

Where \( b_T \) and \( a_T \) can be determined from the slope and the intercept from the plot of \( q_e \) versus \( \ln C_e \) shown in Fig. 10, \( T = \) temperature and \( R = \) universal gas constant (8.314 J/K mol⁻¹).

\( b_T \) is the Tempkin constant related to heat of sorption (J/mg) which was found to be \( -11.95 \) J/mg while \( a_T \) is the equilibrium binding constant corresponding to the maximum binding energy (L/g) = 0.01089 L/g and coefficient of determination was 0.999.

### 3.4.4 Dubinin-Radushkevich isotherm

The D.R model is reported to be more general than Langmuir and Freundlich isotherms. It is used to determine the apparent energy of adsorption, the characteristic porosity of the adsorbent toward the adsorbate and does not assume a homogeneous surface or constant sorption potential [33]. The sorption mean free energy is the energy required to transfer one mole of the sorbate from infinity in solution to the surface of solid. The magnitude of the sorption mean free energy \( E \) is widely used for estimating the type of adsorption [34,35], if it is physisorption or chemisorption. The equation describing this relationship is given in equation 13.

\[ q_e = q_D e^{-E \varepsilon^2} \]  

The linear form of Dubinin-Radushkevich isotherm is given in equation 14.

\[ \ln q_e = \ln q_D - BE \varepsilon^2 \]  

\( q_D \) is the theoretical saturation capacity obtained from the intercept and \( B \) the constant related to the adsorption energy given as;

\[ E = 1/2B^{1/2} \]
obtained from the slope of ln q_e versus ε^2 shown in Fig. 11, which is the Polanyi potential given as:

\[ \varepsilon = RT \ln \left( 1 + \frac{1}{C_e} \right) \]  

(15)

The energy of activation as proposed by Dubinin-Radushkevich isotherm shows that the adsorption process in this study is physiosorption in nature; since the activation energy was found to be 0.03536 KJ/mol whilst the mean free energy of adsorption per mole of adsorbate was 4.0 \times 10^{-4} (mol^2/J^2). The theoretical saturation capacity was found to be 84.56 mg/g. Reports found in literature revealed that the mean sorption energy < 8 kJ/mol indicates physical sorption; 8-16 kJ/mol is chemical ion – exchange and above > 40 kJ/mol favours chemisorption mechanism [36]. From the results, all the models tested, Langmuir, Freundlich, Tempkin and D-R isotherms fitted well to the experimental data for the adsorption of methylene blue onto activated carbon from plantain pod. The isotherm constants are presented in Table 3. Similar results have been recorded in literature [37].

![Langmuir adsorption isotherms for the adsorption of methylene blue on to PPAC at a temperature of 30ºC and adsorbent size of 300 µm](image1)

**Fig. 8.** Langmuir adsorption isotherms for the adsorption of methylene blue on to PPAC at a temperature of 30ºC and adsorbent size of 300 µm

![Freundlich adsorption isotherms for the adsorption of methylene blue on to PPAC at a temperature of 30ºC and adsorbent size of 300 µm](image2)

**Fig. 9.** Freundlich adsorption isotherms for the adsorption of methylene blue on to PPAC at a temperature of 30ºC and adsorbent size of 300 µm
Table 3. Results of adsorption isotherm for the adsorption of methylene blue onto PPAC at temperature =30ºC

|                  | Langmuir       | Freundlich | Tempkin          | Dubinin-Raduskevich |
|------------------|----------------|------------|------------------|---------------------|
|                  | $q_e$ (mg/g)   | $b_L$ (L/mg) | $R_L$ | $R^2$ | $k_l$ (mg$^{1-1/2}$L$^{1/2}$g$^{-1}$) | $n$ | $R^2$ | $b_L$ (J/mg) | $a_T$ (L/g) | $R^2$ | $q_o$ (mg/g) | $B$ (mol$^2$J$^2$) | $E$ (J/mol) | $R^2$ |
| $71.42$          | $0.041$        | $0.245$    | $0.993$          | $22.23$             | $-$          | $0.9480$ | $-11.95$ | $0.010$ | $0.999$ | $0.8456$ | $4.0\times10^{-4}$ | $0.0353$ | $0.9860$ |
|                  |                |            |                  |                     |              | $0.761$  | $9$      |          |                |          |             |                |              |

Fig. 10. Tempkin adsorption isotherm for the adsorption of methylene blue on to PPAC at a temperature of 30ºC and adsorbent size of 300 µm
Fig. 11. Dubinin - Radushkevich Isotherm for the adsorption of methylene blue on to PPAC at a temperature of 30ºC and adsorbent size of 300 µm

4. CONCLUSION

1. Activated carbon was produced from plantain pod (Musa paradisiac for the adsorption of methylene blue from aqueous solution and also proved that it is effective in removing the methylene blue, uptake up to 68.69%.

2. The characteristics properties of the prepared activated carbon increased with increase in temperature except the ash content.

3. The pseudo second-order kinetic model described the adsorption better than pseudo first-order model and intra particle diffusion model indicates that pore diffusion play a major role for the adsorption of methylene blue by PPAC.

4. All the isotherms fitted well to the experimental data > 0.9 but the Langmuir model is more appropriate to explain the nature with high correlation coefficient (mono layer) compared to Freundlich. Temkin model also fitted better with a correlation coefficient of 0.999, for all the models tested. The mean free energy was found to be 0.03536 kJ/mol, indicating physisorption adsorption process.

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COMPETING INTERESTS

Author has declared that no competing interests exist.

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