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Adsorption of Methylene Blue from Aqueous Solution Using Steam-Activated Carbon Produced from *Lantana camara* Stem

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Abstract

This study investigated the adsorption of Methylene Blue (MB) present in wastewater onto the activated carbon produce from *Lantana camara* stem. The agricultural material (*Lantana camara* stem) was carbonized at 300˚C for 2 h, ground and steam-activated. The Steam-Activated *Lantana camara* (SALC) stem carbon was characterized using Scanning Electron Microscope (SEM) and Fourier Transform Infrared (FTIR) Spectrophotometry before and after adsorption. Batch model experiments were conducted at 20˚C to study the effects of pH, agitation time, adsorbent dosage and initial concentration of methylene blue. The equilibrium adsorption isotherms and kinetics were investigated. The FTIR bands at 3500, 2500, 2196 and 1682 cm^{-1} were shifted to 3646.3, 3030, 2822, 1709.05 cm^{-1} after MB adsorption. Similarly, the Scanning Electron Microscopy (SEM) analysis showed that the average pore size on the activated carbon was 20 µm. The Methylene Blue (MB) uptake increased with the increase in pH. Similarly, the dye adsorption increased as contact time increased, and reached equilibrium at 60 minutes. The removal of the dye increased when the dosage was increased from 0.5 - 2.0 $g \cdot L^{-1}$, at different dye concentrations (50 - 200 $mg \cdot L^{-1}$). The percentage removal decreased with increasing initial dye concentration for SALC. The adsorption isotherm data fitted well to the Freundlich isotherm ($R^2 = 0.989$) while the experimental data fitted very well to the pseudo-second-order kinetic model ($R^2 = 0.99$). This study suggests that adsorbent prepared from *Lantana camara* stem can be used effectively for the adsorption of methylene blue in wastewater.

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Keywords

Adsorption, *Lantana camara,* Methylene Blue, Steam Activation

1. Introduction

Dyes are important hazardous substances found in textile industry, food industry, pharmaceutical industry, paper industry and plastics industry. Their presence in water bodies reduces light penetration and this consequently thwarts the photosynthesis of aqueous flora [1] [2]. Similarly, this makes the water objectionable for drinking. Dyes in water stream causes allergy, dermatitis, skin irritation, which, at extreme cases, provoke cancer and mutation in humans [3]. Furthermore, the colour and the non-biodegradable nature of the spent dye baths constitute serious environmental problems.

Various treatment methods for the remove dyes from wastewater have been investigated and these methods can be classified as chemical coagulation/flocculation, ozonation, oxidation processes, chemical precipitation, ion exchange, reverse osmosis, and ultra-filtration [4]. However, most of these methods for the removal of dyes from dye containing wastewater have serious restrictions such as high cost, formation of hazardous by-products or intensive energy requirements [5] [6]. Therefore, the development of efficient, low-cost and environmentally-friendly technologies to reduce dye content in wastewater is extremely necessary.

Adsorption is rapidly gaining prominence among the treatment technologies and [7] has noted that adsorption can produce high-quality water while also being a process that is economically feasible. The physical characteristics of the adsorbents such as surface area, porosity, size distribution, density and surface charge have high influence in the adsorption process [8]. As a result, there has been a great interest in developing new adsorbent materials with diverse compositions, properties and functionalities. Although commercial activated carbon is the most widely used adsorbent for dye removal, it is too expensive [9]; consequently, numerous low-cost alternative adsorbents have been proposed including chemically modified sugarcane bagasse lignin [10], pistachio hull waste [11], coffee husk-based activated carbon [12], pine cone [13], rice husk [14], synthetic calcium phosphates [15], natural untreated clay [16], pillared clays [17], and swelling clays [18].

The purpose of this work is to investigate convenient and economic method for basic dye (methylene blue) removal from wastewater by adsorption on an abundantly available adsorbent, but in this case, adsorption of methylene blue dye onto steam activated carbon from *Lantana camara* stem (SALC). Batch studies involving process parameters such as the effect adsorbent dosage, initial dye concentration, pH of solution, and contact time, were carried out. Equilibrium and kinetic analysis were conducted to determine the factors controlling the rate of adsorption, the optimization of various parameters in dye recovery and to find out the possibility of using this material as low-cost adsorbent for dye removal. The experimental data obtained were analysed using isotherm models such as Langmuir and Freundlich isotherm models.

2. Materials and Methods

2.1. Preparation of Adsorbent

The natural low cost material, *Lantana camara*, was obtained from Stadium Area, Ogbomosho, and OyoState, Nigeria. The stems were separated from the seed and flower and were extensively washed with tap water to remove soil and dust particles. It was then sundried, and further crushed into smaller size. The *Lantana camara* was carbonized using an electric furnace at 300˚C for 2 h. The carbonized sample was ground into granular form and the carbon was activated using steam activating reactor. The resultant activated carbon was oven-dried at 105˚C for 2 h and cooled to room temperature in a desiccator. It was then ground with mortar and pestle and sieved to 2 mm mesh size. It was later stored inside a desiccator to avoid moisture absorption. The Infrared spectroscopic analysis was conducted on the Steam-Activated *Lantana camara* Stem (SALCS) to investigate the functional groups present on its surface using Fourier Transform Infrared Spectrometer (Perkin-Elmer Spectrum GX, Kuala Lumpur, Malaysia). The morphologies of the steam activated carbon were characterized using a scanning electron microscope (SEM, JEOL JSM-6480 LV).

2.2. Adsorption Studies

The adsorb ate was prepared by dissolving 1 g of methylene blue powder in small quantity of distilled water in 1000 ml volumetric flask. More distilled water was added to make up to the mark. It was then shaken vigorously for five minutes to ensure complete dissolution and homogeneity; this makes the stock solution of concentration 1000 mg/L. Different concentrations were prepared by serial dilution [19]. Batch adsorption method was employed to determine the effects of contact time, adsorbent dose, dye concentration and pH solution.

100 ml of (50 mg/L) dye solution was poured into a conical flask with adsorbent dosage 0.5 g and place inside a shaker (environmental orbital shaker, Deneb Instruments). The samples were withdrawn from the shaker at predetermined time intervals and the dye solution was separated from the adsorbent by the help of a micropipette.

The absorbance of the solution was then measured. The dye concentration was measured after 50, 100, 150, and 200, until the equilibrium was reached. To study the effects of initial pH of the solution on the adsorption process, 100 ml of 50 mg/L dye solution was poured into a conical flask with adsorbent dosage of 1 g. The pH of the dye solutions was adjusted with dilute HCl (0.05 N) or KOH (0.05 N) solution by using a pH meter (EUTECH Instrument, pH 510). 10 ml of dye solution was prepared taking dye and the pH of solution was changed from 3 to 10.

The adsorbent dosage was varied as 0.5, 1.0, 1.5, and 2.0 g/150ml. Effect of concentration 100 ml of dye solution was prepared in conical flask with dye concentration 50 mg/L and adsorbent dose (1 g/L) and place the shaker. The temperature was maintained at 20˚C. The final dye concentration readings were taken at 50, 100, 150, and 200. Dye concentration was estimated, spectrophotometrically, at the wavelength corresponding to maximum absorbance, λ max using a spectrophotometer (JASCO UV/Vis-550). The samples were withdrawn from the shaker at predetermined time intervals (30, 45, 60 and 120 min) and the dye solution was separated from the adsorbent by the help of a micropipette. The absorbance of the solution was then measured. The dye concentration was measured after 50, 100, 150 and 200, until the equilibrium was reached. The q_e is expressed as

$$q_e = \frac{\left(C_e - C_o\right)V}{M} \tag{1}$$

where, q_e = amount of dye adsorbed per unit mass of adsorbent (mg/g), C_o = initial dye concentration (mg/L), C_e = final dye concentration (mg/L), V = volume of dye solution and M = mass of adsorbent (g/L).

3. Results and Discussion

3.1. Characterization of Adsorbent

The FTIR spectra of SALC were taken before and after the adsorption of MB to ascertain the possible involvement of the functional groups on the surface of SALC in the adsorption of MB (**Figure 1** and **Figure 2**). The broad band at 3500 - 2500 cm^{-1} can be attributed to -OH stretching vibration while the band at 2196 cm^{-1} can be assigned to the C≡C and/or C≡N stretching vibrations. The peak at 3500 cm^{-1}was strong and broad due to a carbonyl group stretching vibrations superimposed on the side of hydroxyl group band, which has been reported to occur at 3500 - 2500 cm^{-1}. The peaks at 1709.05 - 1682 cm^{-1} may be attributed to C=O stretching, -CH bending vibrations of carbonyl compound, respectively. The bands at 3030 cm^{-1} and 2822 cm^{-1} can be linked to C-H stretching **Figure 3** reveals that some new bands appeared, some remained and some were shifted after MB sorption. The bands at 3500, 2500, 2196 and 1682 cm^{-1} were shifted to 3646.3, 3030, 2822, 1709.05 cm^{-1} after MB adsorption. This is an indication that OH, C-H, C=C and C=O, carbonyl group could be involved in the adsorption of MB onto SALC.

Plates 1(a) and (b) shows the SEM micrographs of SALC samples before and after dye adsorption. The SALC exhibits a caves-like, uneven and rough surface morphology. The surface of dye-loaded adsorbent, however, shows that the surface of SALC is covered with dye molecules: (a) fresh SALC and (b) dye adsorbed SALC.

3.2. Effect of Solution pH on Dye Adsorption onto SALC

The pH of the dye solution plays an important role in the whole adsorption process and particularly on the adsorption capacity [20]. The effect of each individual solution pH (3 - 11) on the equilibrium uptake capacity of

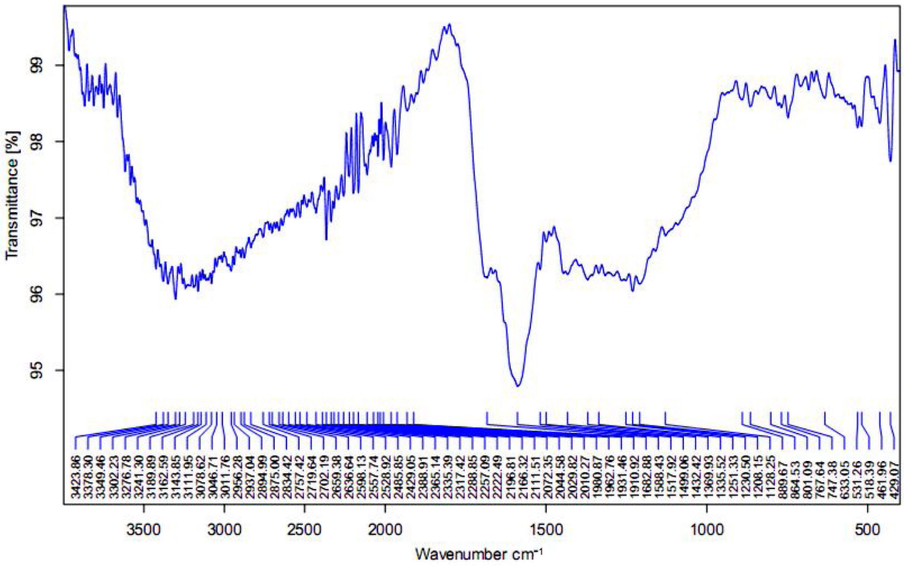


Figure 1. FTIR characterization of SALCS before MB adsorption.

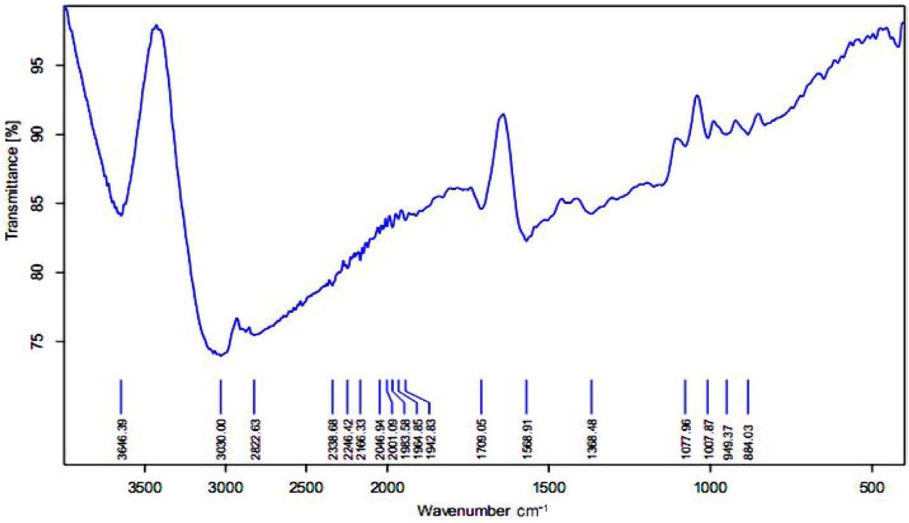


Figure 2. FTIR characterization of SALC after MB adsorption.

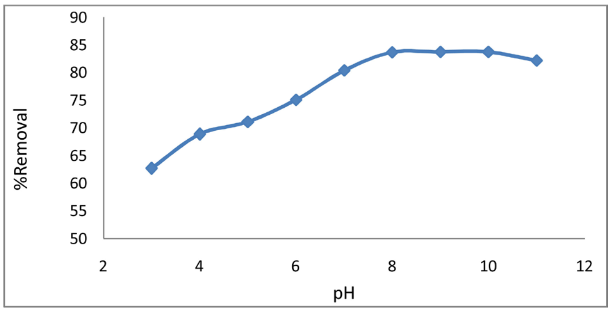


Figure 3. Effect of pH as a function of percentage removal of MB.

MB dye was studied at different initial dye concentrations (50 - 200 mg·L^{-1}) at room temperature. The dye uptake was found to increase with an increase in pH (**Figure 4**). At lower pH, the surface charge may be positive,

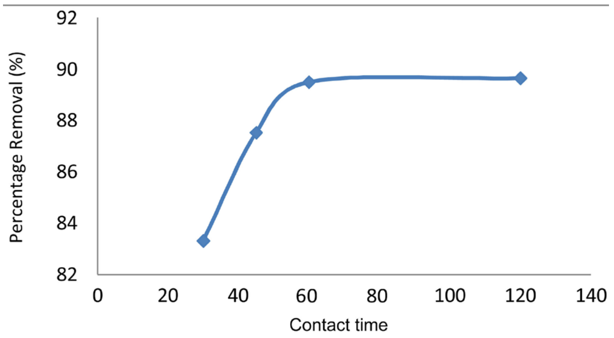


Figure 4. Effect of contact time as a function of percentage removal of MB.

thus making (H^+) ions compete effectively with cationic dyes causing a decrease in the amount of dye adsorbed [21]. And at higher pH, the surface of SALC may be negatively charged which enhance the positively charged on MB dye through electrostatic force attraction [22]. Similar observations were reported for adsorption of dyes indicating that the adsorbent has a net positive charge on its surface [23] [24].

3.3. Effect of Contact Time on Dye Adsorption onto SALC

The adsorption of MB dye at initial concentration of 50 $mg \cdot L^{-1}$was studied at different contact time (30 - 120 min). The dye adsorption uptake was increased as contact time increased, and reaches equilibrium at 60 minutes (**Figure 5**). The result suggests that, adsorption takes place rapidly at the initial stage on the external surface of the adsorbent followed by a slower internal diffusion process, which may be the rate determining step [25] [26]. In addition, the fast adsorption at the initial stage may be due to the fact that a large number of surface sites are available for adsorption but after a lapse of time, the remaining surface sites are difficult to be occupied. This is because of the repulsion between the solute molecules of the solid and bulk phases, thus, taking long time to reach equilibrium [27].

3.4. Effect of Adsorbent Dose on Efficiency of Dye Removal onto SALC

The solid/solution ratio is an important factor determining the capacity of adsorbent in a batch adsorption. The effect of adsorbent dosages on the percentage removal of MB dye has been shown in **Figure 6**. It followed the predicted pattern of increasing percentage adsorption as the dosage was increased [28]. This is probably because of the resistance to mass transfer of dye from bulk liquid to the surface of the solid, which becomes important at high adsorbent loading in which the experiment was conducted [29].

It can be clearly seen that the removal of MB dye increased with increasing the amount of SALC. However the amounts of adsorbed dye per unit weight (Q_e) of the SALC decreased with increasing the solid/solution ratio (**Figure 6**). The removal of the dye increased when the dosage was changed from 0.5 - 2.0 $g \cdot L^{-1}$, at different dye concentrations (50 - 200 $mg \cdot L^{-1}$). As expected, at constant initial concentration of dye, increasing the sample dose provides a greater surface area and larger number of adsorption sites and hence enhancement of dye uptake [30]. The primary factor explaining this characteristic is that adsorption sites remain unsaturated during the adsorption reaction whereas the number of sites available for adsorption site increases by increasing the adsorbent dose [31].

3.5. Effect of Initial Dye Concentration on Efficiency of Dye Removal onto SALC

The effect of the initial dye concentration on the dye adsorption capacity was investigated in the concentration range of 50 - 200 mg/L^{-1} at room temperature without changing the initial pH of the medium. The results represented in **Figure 7** shows that the percentage of removal decreased with increasing initial dye concentration for SALC. The lower uptake at higher concentration resulted from an increased ratio of initial adsorption number of moles of the dye to the available surface area; hence fractional becomes dependent on initial concentration. The initial dye concentration provides an important driving force to overcome the mass transfer resistance of the dye between the aqueous and solid phases. Therefore, at higher initial dye concentration, the number of

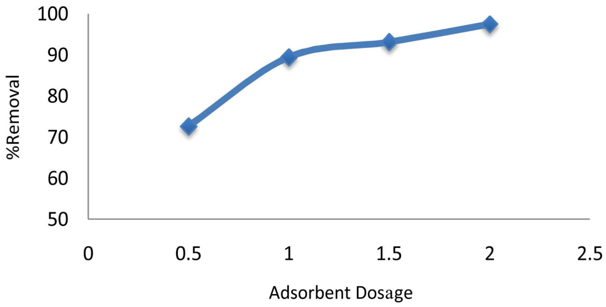


Figure 5. Effect of adsorbent dosage as a function of percentage removal of MB.

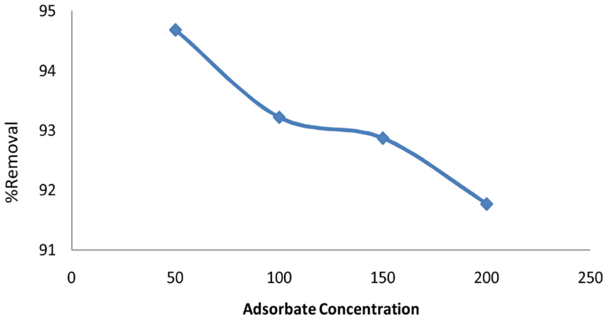


Figure 6. Effect initial dye concentrationsas function of percentage removal of MB.

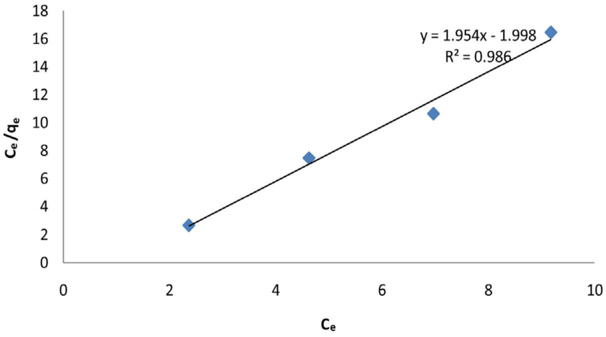


Figure 7. Langmuir adsorption isotherm for the adsorption of MB onto SACL.

ions competing for the available sites on the surface of SALC was high, hence, resulting in higher MB adsorption capacity [27]. Similar results were also reported by other researchers [32].

3.6. Adsorption Isotherms

The adsorption isotherm indicates how the adsorption molecules distribute between the liquid phase and the solid phase when the adsorption process reaches an equilibrium state. The analysis of equilibrium adsorption data, by fitting them to different isotherm models, is an important step to find the suitable model that can be used for design purpose [33]. Adsorption isotherm study was carried out on two well-known isotherms, Langmuir and Freundlich. The applicability of the isotherm equation was compared by judging the correlation coefficients, R^2.

3.6.1. Langmuir Isotherm

The linear form of Langmuir's isotherm model plotted is given by the following equation.

$$\frac{C_e}{Q_o} = \frac{1}{Q_{ob}} + \left(\frac{1}{Q_o}\right) C_e \tag{2}$$

where: C_e is the equilibrium concentration of the adsorbate (MB) (mg/L), Q_o is the amount of adsorbate adsorbed by unit mass of adsorbate (mg/g), Q_{ob} are Langmuir constant related to monolayer adsorption capacity and affinity of adsorbent towards adsorbate respectively [34].

When C_e/Q_o was plotted against C_e straight line with slope $1/Q_o$ was obtained (**Figure 8**), indicating that the adsorption of the Methylene Blue (MB) onto SALC follows the Langmuir isotherm. The Langmuir constant "*b*" and "Q_o" were calculated from this isotherm and their values are given in **Table 1**. Conformity of the experimental data to Langmuir isotherm model indicates the homogenous nature of SALC similar observations were reported on the adsorption of Cd onto *Crysophyllum albidum* shell [29]. The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless equilibrium parameter (R_L) [35], which is defined in Equation (3) as.

$$R = \frac{1}{1 + bC_0} \tag{3}$$

where: Q_{ob} is the Langmuir constants and C_e is the highest dye concentration (mg/L).

The value of R_L indicates the type of the isotherm to be either unfavourable ($R_L > 1$), linear ($R_L = 1$), favourable ($0 < R_L < 1$) or irreversible ($R_L = 0$) (**Table 2**). Value of R_L was found to be 1 which confirmed that the adsorbent prepared from the *Lantana camara* stem is favourable for adsorption of MB under conditions used in this study [36].

3.6.2. Freundlich Isotherm

The well-known logarithmic form of Freundlich model used in this is given by the following equation.

$$\log q_e = \log K_f + \left(\frac{1}{n}\right) \log C_e \tag{4}$$

where: q_e is the amount adsorbed at equilibrium (mg/g), C_e is the equilibrium concentration of the adsorbate (MB) and K_f and n are Freundlich constants; n given an indication of how favourable is the adsorption capacity of the adsorbent [37].

K_f can be defined as the adsorption or distribution coefficient and represents the quantity of methylene blue onto SALCS for a unit equilibrium concentration. The slope $1/n$ ranging between 0 and 1 is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its, value gets closer to zero [38]. Value of $1/n$ below one indicates a normal Langmuir isotherm while above one is indicative of cooperative adsorption [39]. The plot of $\log q_e$ verse $\log C_e$ gives straight lines with slope "$1/n$" (**Figure 9**), which shows that the adsorption of MB also follows the Freundlich isotherm. Accordingly, Freundlich constants (K_f and n) were calculated and recorded in **Table 1**.

Table 1 shows the value of the parameter of the two isotherms and related correlation coefficients. The Freundlich model yields a somewhat better fit ($R^2 = 0.989$) than the Langmuir model ($R^2 = 0.986$). The value of $1/n$ is 0.751 (**Table 1**) which indicates favourable adsorption [40] [41].

The adsorption capacities of SALC were compared to previously reported works on the adsorption capacities

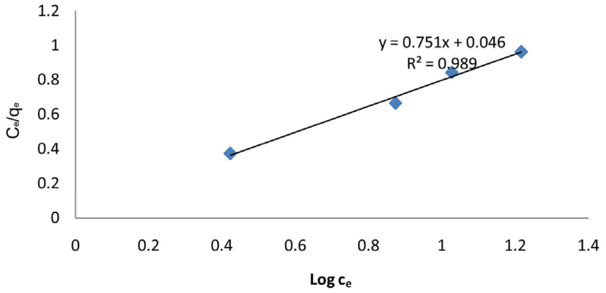


Figure 8. Freundlich adsorption isotherm for the adsorption of MB onto SALC.

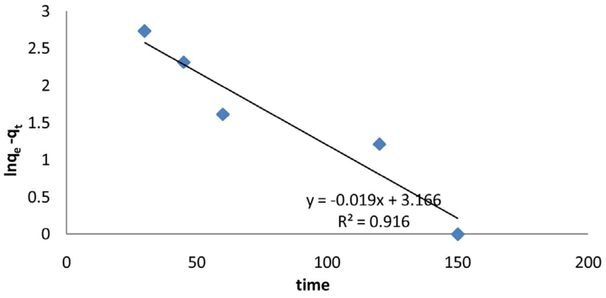


Figure 9. Plot of $\ln(q_e - q_t)$ against time for the adsorption of MB onto SALC.

Table 1. Isotherm characteristics parameters of Langmuir and Freundlich isotherm constants for MB onto SALCS.

Langmuir isotherm constants			Freundlich isotherm constants		
Q_o [(mg/g) (L/mg)]	b	R^2	$1/n$	K_f [(mg/g) (L/mg)]	R^2
19.84	0.548	0.986	0.751	1.111	0.989

Table 2. K_R values for different isotherm shapes.

Values of K_R	Type of isotherm
$K_R > 1$	Unfavourable
$K_R = 1$	Linear
$0 < K_R < 1$	Favourable
$K_R = 0$	Irreversible

Source: [35].

of various low-cost adsorbent. The experimental data of the present study was found to be higher than those of walnut bark (15.1 mg/g) and yellow passion fruit (16 mg/g) and lower than those of rice husk (4.59 mg/g), cherry saw dust (39 mg/g), sugarcane bagasse (34.2 mg/g) and banana peel (20.8 mg/g) (**Table 3**).

3.7. Kinetics Studies

It is important to be able to predict the rate at which contaminants is removed from aqueous solution in order to design adsorption treatment plant. In order to investigate the mechanism of adsorption and potential rate controlling steps such as mass transfer and chemical reaction, the kinetics of MB sorption onto SALC was investigated using two different models: the pseudo-first-order kinetic [42], and the pseudo-second-order kinetic [43] models. The conformity between experimental data and the model predicted values was expressed by the correlation coefficients (R^2, values close or equal to 1 and SSE = sum of square of errors) [41]. A relatively high R^2 value indicates that the model successfully describes the kinetics of MB adsorption [44].

The rate constant of adsorption was determined from the pseudo-first-order equation given by

$$\ln\left(q_e - q_t\right) = \frac{1}{n} q_e - K_t \tag{5}$$

where: q_e and q_t are the amounts of methylene blue adsorbed (mg/g) at equilibrium and at time (min), respectively and K_1 the rate constant of adsorption (2 h). Values of K_1 were calculated from plots of $\ln\left(q_e - q_t\right)$ vs t at different concentrations of methylene blue (MB). The correlation coefficient values are higher than 0.90, the experimental q_e values also agree slightly different of about (4.7%) with the calculated ones, obtained from the linear plots (**Figure 10** and **Figure 11**). This shows that the adsorption of MB onto SALC is pseudo second order kinetics.

Sum of square of errors is a test of kinetic models. Beside the value of R^2, the applicability of both kinetics models were verified through the sum of error squares (SSE, %). The adsorption kinetics of MB on adsorbent prepared from *Lantana camara* stem (SALC) was tested at different initial concentration. The validity of each

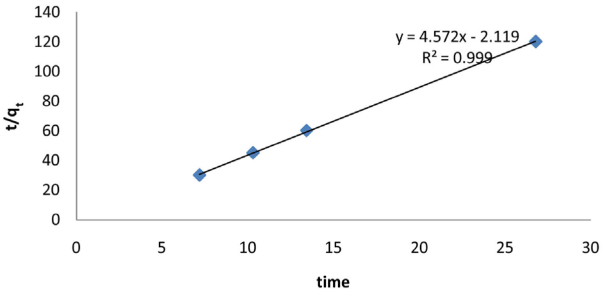
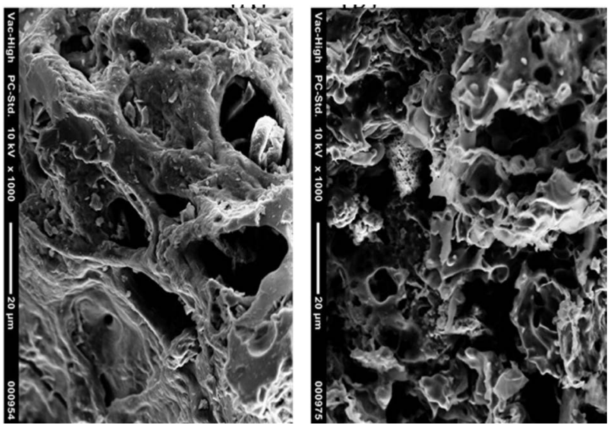


Figure 10. Plot of t/q_t against time for the adsorption of MB onto SALC.



(a) (b)

Figure 11. Scanning electron microscope of (a) fresh SALCS and (b) dye adsorbed SALCS.

Table 3. The natural forms of agricultural waste and its dye adsorption capacities.

Adsorbents	Adsorption capacities (mg/g)	References
Banana peel	20.80	[20]
Cherry saw dust	39.00	[45]
Rice husk	40.59	[46]
Sugarcane bagasse	34.20	[47]
Walnut bark	15.10	[48]
Yellow passion fruit	16.00	[49]
Lantana camera stem	19.84	This work

model was determined by the sum of error squares (SSE, %) given by;

$$\mathrm{SSE}(\%)=\frac{\sqrt{\sum\left(q_e-q_e\mathrm{cal}\right)^2}}{N} \tag{6}$$

where: N is the number of data points:

The higher the value of R^2 and the lower the value of SSE, the better the goodness of the fit. **Table 4** lists the calculated results. It was found that the adsorption kinetics of MB on adsorbent prepared from *Lantana camara* stem can be described by the second-order kinetic model.

4. Conclusion

Lantana camara stem like other agricultural waste can be used in the treatment process of dyes in wastewater.

Table 4. Comparison of the pseudo-first-order and pseudo-second-order adsorption rate constants, calculated experimental q_e values for different initial concentrations of MB.

Initial conc. (mg/L)	Q_e (mg/g)	First order kinetics model			SSE (%)	Second order kinetics model			SSE (%)
		K_1 (min^{-1})	$q_e$cal (mg/g)	R^2		K_2 (g/mg/mm)	$q_e$cal (mg/g)	R^2	
50	12.34	0.077	13.52	0.98	0.60	0.0053	13.83	0.99	0.48
100	24.77	0.082	26.00	0.91	0.57	0.0026	27.49	0.99	0.95
150	37.00	0.061	37.12	0.98	0.04	0.0018	42.49	0.99	1.45
200	47.00	0.058	46.82	0.98	0.69	0.0012	55.25	0.99	1.98

The adsorption capacity is dependent on pH solution, contact time, adsorbent dosage and adsorbate concentration. Maximum percentage MB removal (85%) was attained at 60 minutes. The adsorption capacity of SALC increased with the increase in pH and initial MB concentration. The Langmuir maximum adsorption capacity showed a slight increase. The Freundlich constant (n) revealed that the adsorption process was favourable and that the adsorption of MB into SALC was dominated by chemisorption. The kinetic modelling of adsorption of MB onto SALC followed the pseudo-second-order kinetic model. The value of the maximum adsorption capacity, Q_o (19.84 mg/g) was comparable with the values observed for other adsorbents reported in the earlier studies. These preliminary studies suggest that adsorbent prepared from *Lantana camara* stem can be used effectively for the adsorbent of MB in wastewater.

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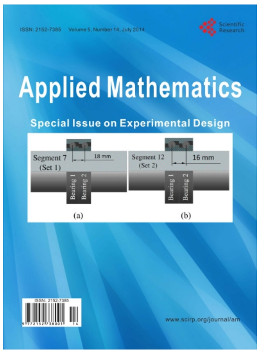
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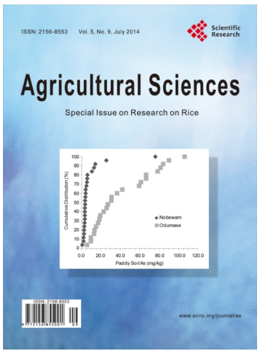
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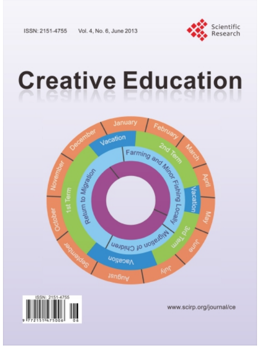
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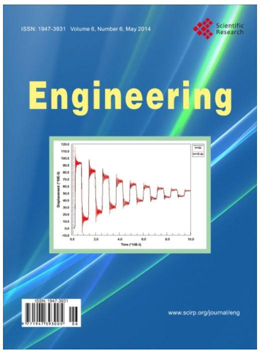
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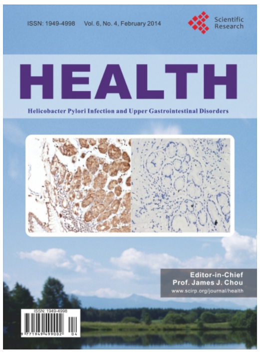
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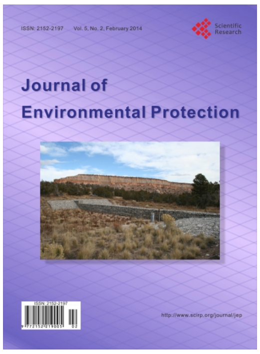
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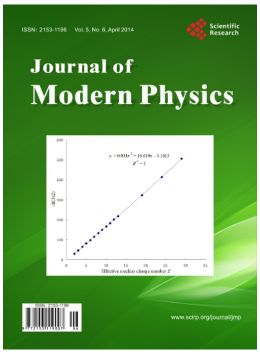
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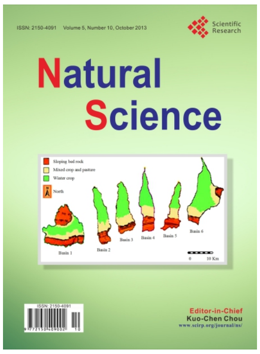
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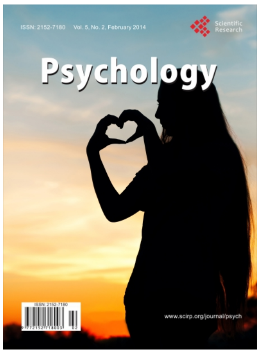
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