
Adsorption of Malachite Green Dye Using Orange Peel

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To cite this article:

Abdussalam Zhul-quarnain, Iwuzor Kingsley Ogemdi, Ilesanmi Modupe, Emuobosa Gold, Ekpunobi Emmanuel Chidubem. Adsorption of Malachite Green Dye Using Orange Peel. *Journal of Biomaterials*. Vol. 2, No. 2, 2018, pp. 31-40. doi: 10.11648/j.jb.20180202.12

Received: October 17, 2018; **Accepted:** November 2, 2018; **Published:** November 27, 2018

Abstract: High level of dyes production and their widespread use in many applications generate colours wastewaters which cause severe water pollution. The pollutant contributes to high suspended solids (SS), chemical oxygen demands (COD), biochemical oxygen demands (BOD), heat, colour, acidity, basicity and other soluble substances. The presence of these dyes in aquatic systems poses heavy risks to human health. Therefore, removal of such dyes from water bodies may be considered an interesting and important research activity. This study shows that orange peel can be used as a suitable adsorbent for the removal of malachite green dyes from solutions. This is a work on the removal of Malachite green dye from their solution with the use of orange peel. The orange peels sourced locally. Proximate analysis done on the adsorbent revealed that the orange peels had 13.25% Moisture content, 5.68% Ash content and 4.7% Carbon content. FTIR technique was also used to identify the functional groups and organic compounds inherent in the orange peels. The adsorption isotherm models used were Henry, Langmuir I, Langmuir II, Langmuir III, Langmuir IV, Freundlich, Temkin and Dubinin-Radushkevich. The result from the models shows that Henry isotherm model fits better for the adsorption of the dye with Orange peels. Results obtained showed that adsorption followed second order kinetics. Thermodynamic data for enthalpy (ΔH) for the adsorption of the dye shows that adsorption was endothermic. The entropy result indicates that there is an increase in randomness at the solid liquid interface. Free energy change shows that adsorption for the dye at temperatures of 298, 323, 343 and 373 were spontaneous and feasible.

Keywords: Malachite Green Dye, Orange Peel, Adsorption Isotherm, Proximate Analysis, FTIR

1. Introduction

Dyes are widely used in industries such as textiles, rubber, paper, plastics, cosmetics, etc., to color their products. The dyes are invariably left as the major waste in these industries. Due to their chemical structures, dyes are resistant to fading on exposure to light, water and many chemicals and, therefore, are difficult to be decolorized once released into the aquatic environment. Many of the organic dyes are hazardous and may affect aquatic life and even the food chain. Release of these dyes in water stream is aesthetically undesirable and has serious environmental impact. Due to intense color they reduce sunlight transmission into water hence affecting aquatic plants,

which ultimately disturb aquatic ecosystem; in addition they are also toxic to humans [1].

Among the various known forms of pollution, water pollution is of great concern since water is the prime necessity of life and extremely essential for the survival of all living organisms. Indeed, it is a part of life itself, since the protoplasm of most living cells contains about 80% of water. It is worth noting that only 0.02% of the total available water on the earth is immediately available for use in the form of rivers, lakes and streams. However, years of increased industrial, agricultural and domestic activities have resulted in the generation of large amount of wastewater containing a number of toxic pollutants, which are polluting the available fresh water continuously. With the realization that pollutants present

in water adversely affect human and animal life, domestic and industrial activities, pollution control and management is now a high priority area. The availability of clean water for various activities is becoming the most challenging task for researchers and practitioners worldwide [2].

Dyes are coloured substances that can be applied to various substrates (textile materials, leather, paper, hair) from a liquid in which they are completely, or at least partly, soluble. Man has made use of dyes since prehistoric times, and in fact, the demand and the usage of dyes have continuously increased. However, the presence of dyes even in trace quantities is very undesirable in aqueous environment as they are generally stable to light and oxidizing agents, and are resistant to aerobic digestion [3].

Many industries often use dyes and pigments to color their products. Most dyes are inert and non-toxic at the concentration discharged into the receiving water; however, they impart color undesirable to the water user. Color removal from textile effluents is a major environmental problem because of the difficulty to treating such streams by conventional physicochemical and biological treatment methods. Liquid-phase adsorption has been shown to be an effective way for removing suspended solids, odors, organic matter, and oil from aqueous solutions [4].

Some of the conventional methods of color removal from industrial effluents include ion exchange, activated carbon adsorption, membrane technology and coagulation. Amongst all, the sorption process by activated carbon has been shown to be one of the most efficient methods to remove dyes from effluents.

Dyes are an important class of pollutants, and can even be identified by the human eye. Disposal of dyes in precious water resources must be avoided, however, and for that various treatment technologies are in use. Among various methods adsorption occupies a prominent place in dye removal. Adsorption techniques are widely used to remove

certain classes of pollutants from waters, especially those that are not easily biodegradable [5].

There have been attempts by researchers to explore the adsorption potential of non-conventional, naturally-occurring agricultural residues in dye removal from effluents. In India alone more than 400 million tonnes of agricultural residue is generated annually, which includes rice husk, bagasse, stalk, coir pith etc. Exploring application of the agricultural residues for use as adsorbents can provide suitable alternatives for the removal of spent dyes from industrial effluents. Dyes represent one of the problematic groups. Currently, a combination of biological treatment and adsorption on activated carbon is becoming more common for removal of dyes from wastewater. Although commercial activated carbon is a preferred sorbent for color removal, its widespread use is restricted due to high cost [6, 7].

Dyes production industries and many others industries which used dyes and pigments generated wastewater, characteristically high in colour and organic content. Presently, it was estimated about 10,000 of different commercial dyes and pigments exist and over 7×10^5 tones are produced annually worldwide [8].

An Adsorption Isotherm is a mathematical equation, which describes the relationship between pressure (p) of the gaseous adsorbate and the extent of adsorption at any fixed temperature, is called adsorption isotherm [9].

The extent of adsorption is expressed as mass of the adsorbate adsorbed on one unit mass of the adsorbent.

Thus, if Xg of an adsorbate is adsorbed on mg of the adsorbent, then

$$\text{Extent of adsorption} = \frac{x}{m}$$

Various adsorption isotherms are commonly employed in describing the adsorption data as shown in Table 1.

Table 1. Isotherm Models [10].

S/N	ISOTHERM MODEL	EQUATION	X	Y	SLOPE	INTERCEPT
1	HENRY	$q_e = KC_e$	C_e	q_e	K	0
2	LANGMUIR I	$\frac{C_e}{q_e} = \frac{1}{q_m K} + \frac{C_e}{q_m}$	C_e	$\frac{C_e}{q_e}$	$\frac{1}{q_m}$	$\frac{1}{q_m K}$
3	LANGMUIR II	$\frac{1}{q_e} = \frac{1}{q_m K C_e} + \frac{1}{q_m}$	1	$\frac{1}{q_e}$	$\frac{1}{q_m K}$	$\frac{1}{q_m}$
4	LANGMUIR III	$q_e = q_m + \frac{q_e}{K C_e}$	$\frac{q_e}{C_e}$	q_e	$\frac{1}{K}$	q_m
5	LANGMUIR IV	$\frac{q_e}{C_e} = q_m K + q_e K$	q_e	$\frac{q_e}{C_e}$	K_L	$q_m K$
6	FREUNDLICH	$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e$	$\ln C_e$	$\ln q_e$	$\frac{1}{n}$	$\ln K_F$
7	TEMKIN	$q_e = B \ln K + B \ln C_e$	$\ln C_e$	q_e	$B = \frac{1}{2a}$	$B \ln K$
8	DR	$\ln q_e = \ln q_D - \beta \varepsilon^2$	ε^2	$\ln q_e$	B	$\ln q_D$

The aim of this study was to determine the adsorption capacity of Orange peel on Malachite green dye.

2. Method

2.1. Apparatus and Equipment

The apparatus used include: conical flasks, beakers; stirrer,

stop watch; spatula, measuring cylinder, volumetric flask, filter papers; weighing balance [mettle, AE 166 model], sample plates, sample bottles, syringe (10 and 5mls), wash bottles, crucible, desiccator, muffle furnace, UV/Vis Spectrophotometer [UV752PEC medical USA] and Thermostat water bath [DK 424 technad U.S.A model], Scanning Electron Microscopy, Fourier transform infrared

spectrophotometer (FT-IR), Oven [search tech instrument DHG-9053A]

2.2. Reagents Used

Distilled water, Hydrochloric acid, Sodiumhydroxide and Malachite green dye. All the chemicals used, were of analytical grade. And all the glass ware were washed and dried before and after each experiment.

2.3. Sourcing of Sample

The Orange peels were locally sourced and were ground with the aid of a milling/grinding machine till it became powdery.

2.4. Preparation of Stock Solution

2.5 g of the Dye (Malachite green) was weighed with an electronic weighing balance, and then 500ml of distilled water was weighed using a volumetric flask.

Serial dilution

To prepare 0.1g/L: 10ml was pipetted out from the stock solution and put into a 50ml volumetric flask. The volume was made up to the mark with de-ionized water. The same procedure was applied in the preparation of 0.2g/L, 0.3 g/L, 0.4 g/L and 0.5 g/L.

2.5. Proximate Analysis

2.5.1. Determination of Ash Content

5g of the sample (ground Orange peel) was weighed into a crucible. The crucible containing the sample was placed in a muffle furnace and was allowed to ash at 550°C. The sample was then removed from the furnace and allowed to cool in a desiccator then weighed immediately. The weight of the residual ash was then calculated as percentage Ash content as follows;

$$\text{Percentage Ash} = \frac{\text{Weight of Ash} \times 100}{\text{Weight of original sample}}$$

2.5.2. Determination of Moisture Content

5g of the sample (ground Orange peel) was weighed into a crucible. The samples were put into an oven at 105°C and heated for 3 hours. The dried samples were put into desiccators, allowed to cool and reweighed. The process was reported until constant weight was obtained. The difference in weight was calculated as a percentage of the original sample.

$$\text{Percentage (\%)} \text{ moisture} = \frac{W_3 - W_1 \times 100}{W_2 - W_1}$$

W_1 =Weight of empty crucible

W_2 =Weight of crucible and sample before burning

W_3 =weight of crucible and ash

2.5.3. Carbon Content / Volatile Solid

5g of the wet sample (ground Orange peel) was weighed into a crucible and placed in a muffle furnace at 700°C for

3hrs. The sample was then cooled in a desiccator after burning and weighted. Percentage carbon content was calculated as follows;

$$\text{Percentage (\%)} \text{ carbon content} = \frac{W_3 - W_1 \times 100}{W_2 - W_1}$$

W_1 =Weight of empty crucible

W_2 =Weight of crucible and sample before burning

W_3 =weight of crucible and ash

2.6. FT – IR Spectra Experiment

The surface functional groups of the sample (ground Orange peel) were identified using Fourier transform infrared spectroscopy (FT-IR). The analysis was carried out by mixing 1g of powdered orange peel with standard reagent and stirred thoroughly; the mixture was compressed under a very high pressure in a special die at 15,000 pounds per square inch to form a small disk of about 1cm and 1.2mm thick. The disk was transparent to IR radiation and analyzed directly. The FT-IR spectra of the sample was recorded between 3500 and 1000 cm^{-1}

2.7. Procedures Used for the Adsorption of the Malachite Green Dye from Solution Using ORANGE Peel

Equilibrium studies

3g of the raw adsorbent was weighed into different sample plates each containing 50ml of varying concentrations of 1g/L, 2g/L, 3g/L, 4g/L, 5g/L and 6g/L of the dye solution. The mixture was stirred thoroughly with a stirrer and allowed to stand for 1hr 30minutes, after which it was filtered. The filtrate was then analyzed using a UV spectrophotometer.

Kinetics Study Experiment

10g/L of the dye solution was prepared and then 15ml of the solution was drawn from it and put into the sample plate containing 3g of the raw adsorbent. The mixture was stirred and allowed to stand. At the time intervals of 5, 10, 30, 60, 90, and 120mins, while other parameters were kept constant. The solutions were then filtered. The filtrate was again analyzed for the dye content concentration and the results obtained were used for kinetic study.

Thermodynamics Study Experiment

Adsorption of the dye from its aqueous solution was investigated over the temperature range of 25°C, 50°C, 70°C and 100°C. 60ml of the dye was withdrawn from the stock solution. This was made up to 200ml with distilled water to produce a concentration of 3g/L. 50ml of the prepared solution was then put into different sample plates containing 3g of the adsorbent and each of them were stirred for even distribution. These sample plates were placed in a thermostat water bath set at temperatures of various degrees. The sample plates were allowed to stand in the bath for 1 hour, 30 minutes and after which samples were drawn from them and filtered for analysis.

3. Result

The result of the proximate composition of the Orange peel is given below:

Table 2. Table showing the result of the proximate composition of the Orange peel.

Parameter	Concentration
% Moisture content	13.25%
Ash content	5.68%
Carbon content	4.7%

The result of the Fourier Transformed Infrared Spectroscopy of the Orange peel is given below:

Table 3. Table showing the FTIR result of the Orange peel.

S/N	Wavelength (cm ⁻¹)	Functional group	Compounds
1	810.5564	C-Cl	Aliphatic Chloro C-Cl stretch
2	862.2151	C-Cl	Aliphatic Chloro compound C-Cl stretch
3	1278.716	RNH ₃	Primary amine NH bend
4	1400.972	H-C=CH	Ethene C=C anti-symmetric stretch
5	1617.011	R-NH ₂	Secondary amine NH bend
6	1864.459	R-COOR	Cyclic ester COO stretch
7	1998.430	R-S-C=N	Thiocyanate SCN anti-symmetric stretch
8	2046.426	RCOO	Carboxylic acid CO stretch
9	2458.507	R-C≡N	Nitrile CN anti-symmetric stretch
10	2554.576	R-C≡N	Nitrile CN anti-symmetric stretch
11	2658.547	CH ₂	Methylene CH symmetric stretch
12	2784.330	CH ₃	Methyl CH symmetric stretch
13	2866.735	CH ₂	Methylene CH symmetric stretch
14	2978.803	CH ₃	Methyl CH symmetric stretch
15	3056.479	RCHOH	Primary alcohol OH stretch
16	3183.124	RCHOH	Primary alcohol OH stretch
17	3367.023	R ₂ CHOH	Secondary alcohol OH stretch
18	3475.009	R-NH ₂	Secondary amine NH bend
19	3593.989	R ₃ CHOH	Tertiary alcohol OH stretch
20	3700.514	R ₃ CHOH	Tertiary alcohol OH stretch
21	3813.971	R ₃ CHOH	Tertiary alcohol OH stretch

The result of the Isotherm models of the dye adsorption by the Orange peel is given below:

Table 4. Table showing the Isotherm parameters for the Malachite green dye removal.

Isothermal Model	Equation of graph	R ² value
LANGMUIR I	$y = 3.488x + 0.171$	0.0105
LANGMUIR II	$y = 0.195x - 7.816$	0.7612
LANGMUIR III	$y = 0.000x + 0.010$	0.0426
LANGMUIR IV	$y = 58.44x + 5.171$	0.776
FREUNDLICH	$y = 0.925x + 1.297$	0.7461
TEMKIN	$y = 0.011x + 0.086$	0.7461
DR	$y = -1x10^{-8}x - 1.483$	0.7535
HENRY	$y = 4.759x + 0.002$	0.8012

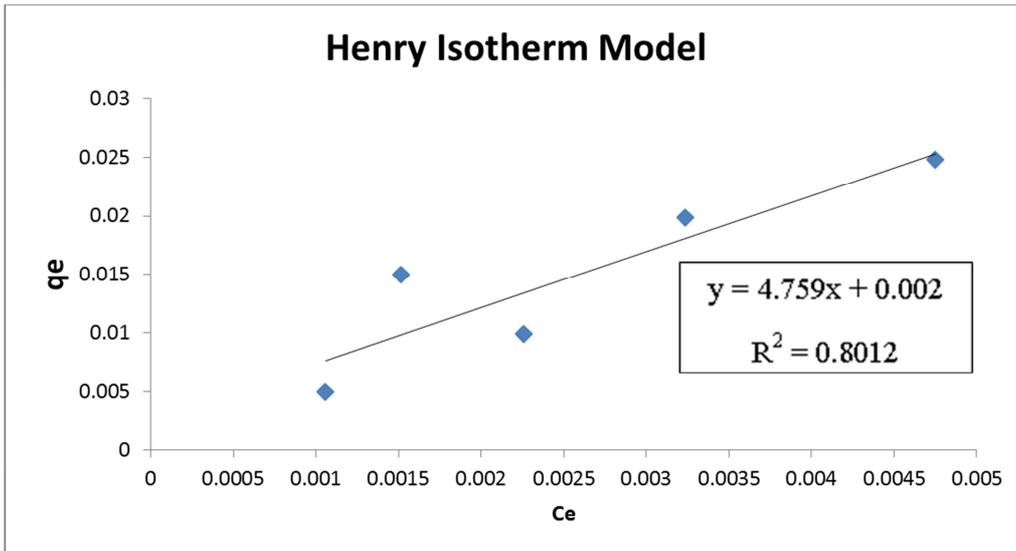


Figure 1. Henry Isotherm plot for Malachite green dye adsorption using Orange peel.

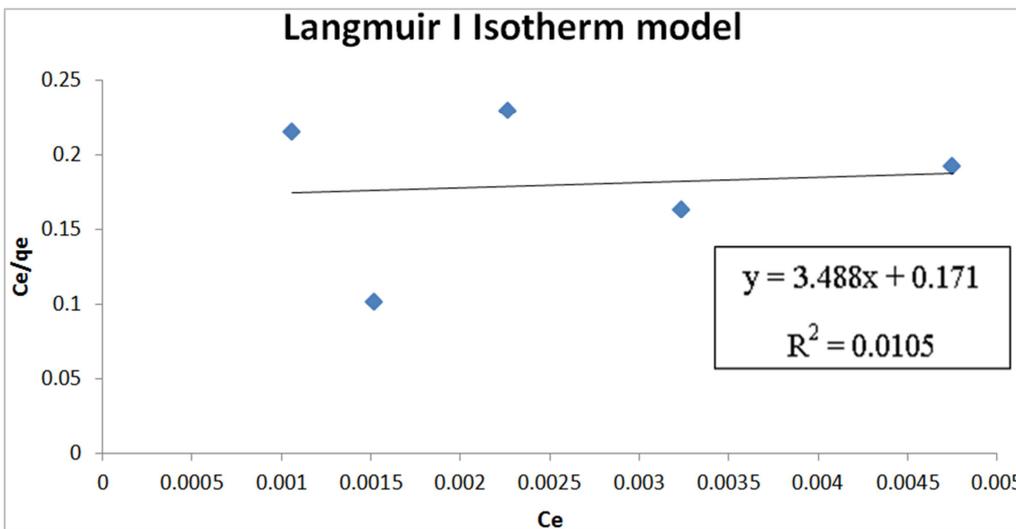


Figure 2. Langmuir I Isotherm plot for Malachite green dye adsorption using Orange peel.

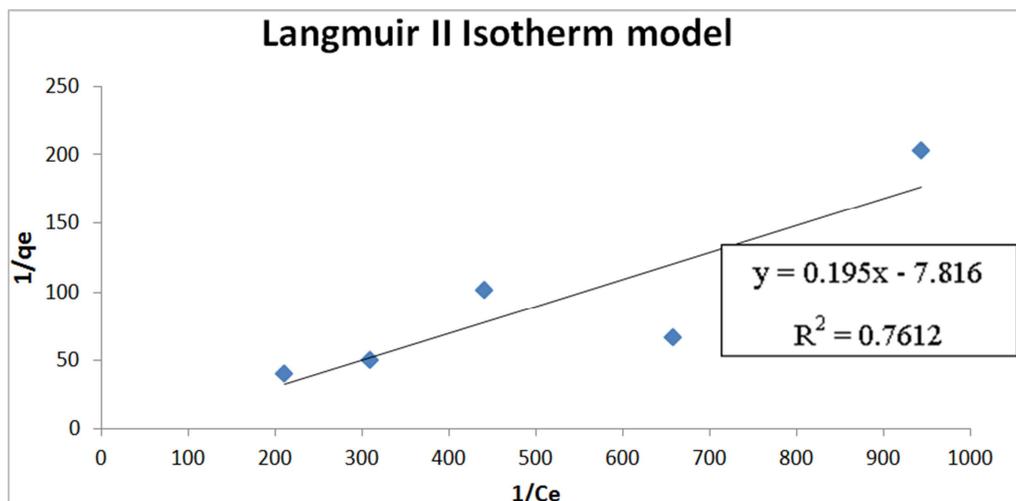


Figure 3. Langmuir II Isotherm plot for Malachite green dye adsorption using Orange peel.

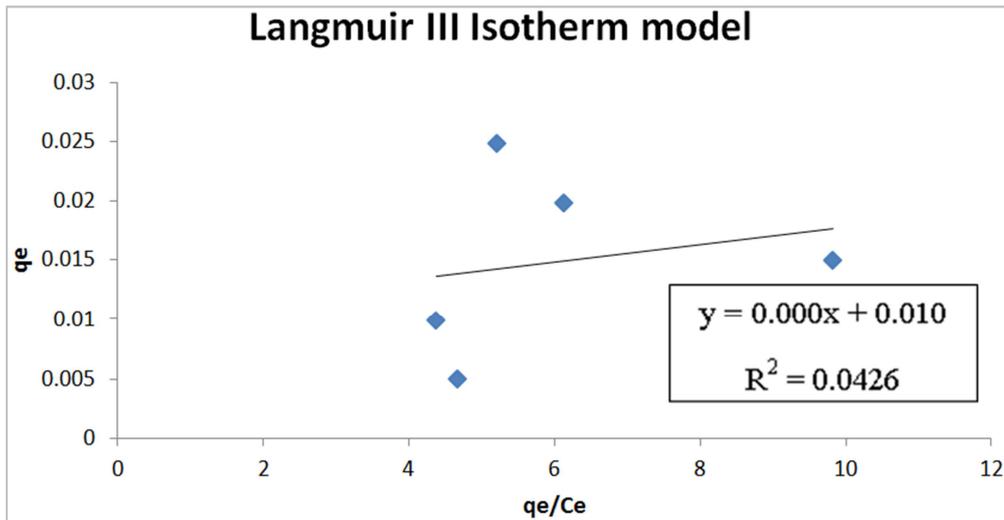


Figure 4. Langmuir III Isotherm plot for Malachite green dye adsorption using Orange peel.

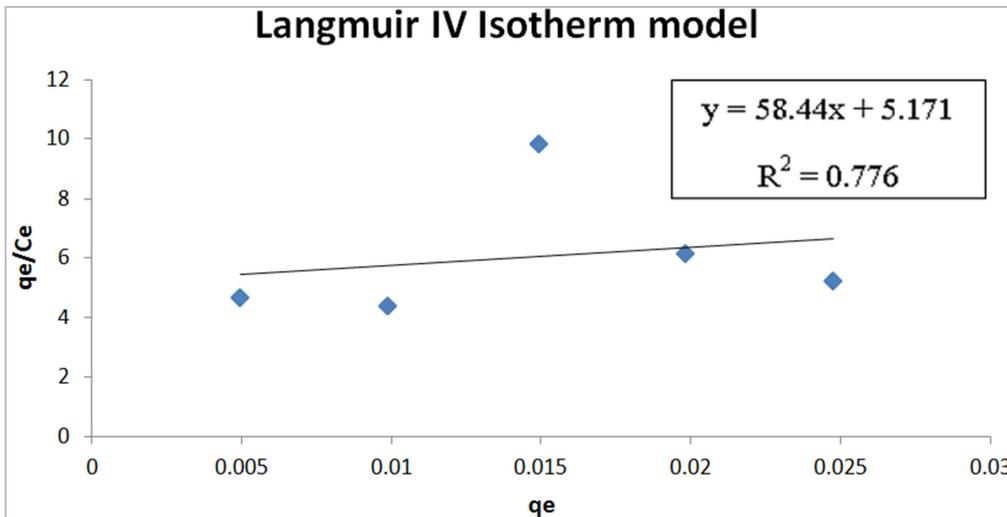


Figure 5. Langmuir IV Isotherm plot for Malachite green dye adsorption using Orange peel.

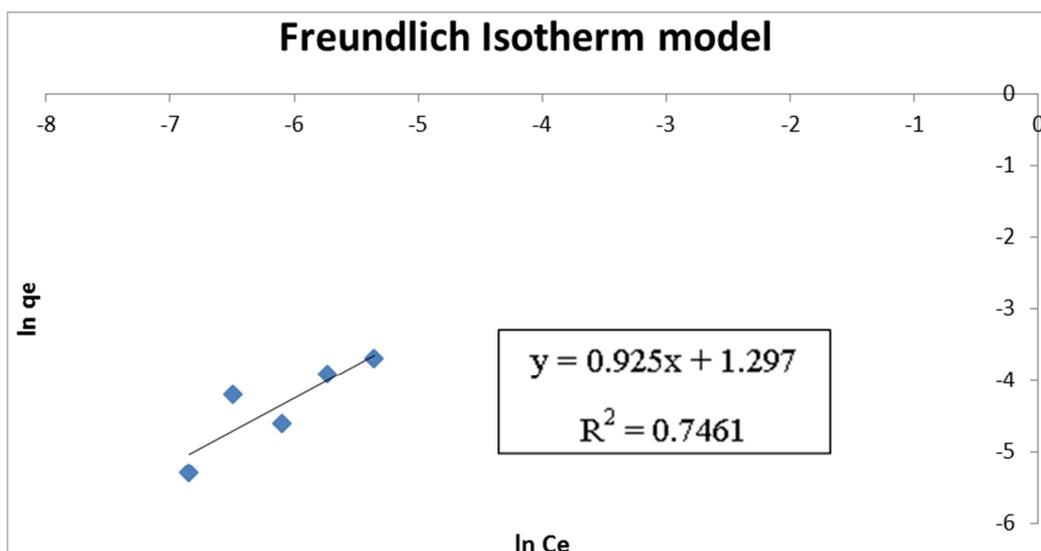


Figure 6. Freundlich Isotherm plot for Malachite green dye adsorption using Orange peel.

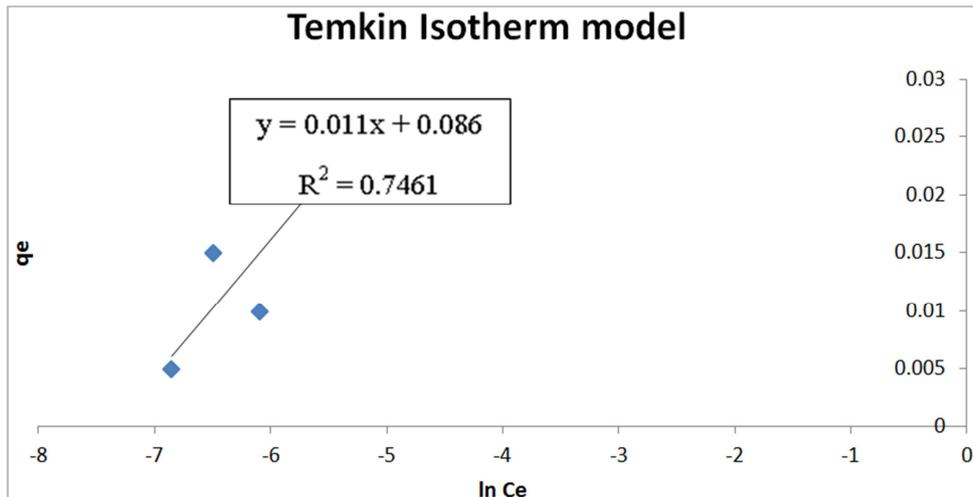


Figure 7. Temkin Isotherm plot for Malachite green dye adsorption using Orange peel.

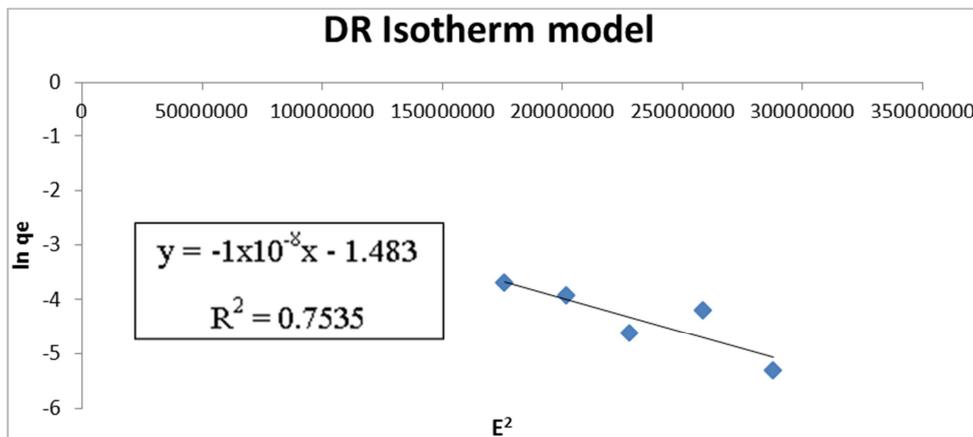


Figure 8. Dubinin-Radushkevich (DR) Isotherm plot for Malachite green dye adsorption using Orange peel.

The result of the Kinetic study of the dye adsorption by the Orange peel is given below:

Table 5. Kinetic parameters for different models for Orange peel.

KINETIC PARAMETERS FOR ADSORPTION						
S/N	MODELS	SLOPE	INTERCEPT	R ²	q _e	K
1.	Lagergren 1 st Order	0.0531	- 7.0869	0.0614	8.36×10^{-4}	0.0531
2.	Pseudo 2nd Order	20.216	1.1458	1.0	0.0495	356.682
3.	Weber-Morris	5×10^{-5}	0.049	0.5155		

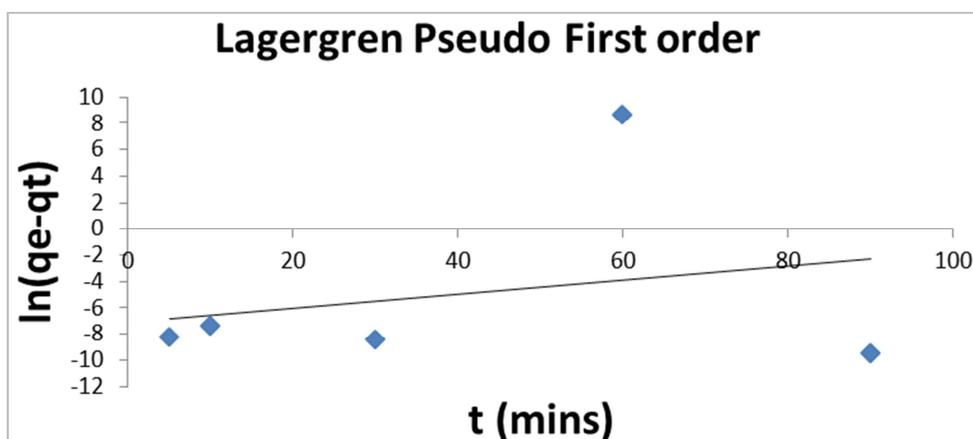


Figure 9. Lagergren pseudo 1st order plot for Malachite green dye adsorption using Orange peel.

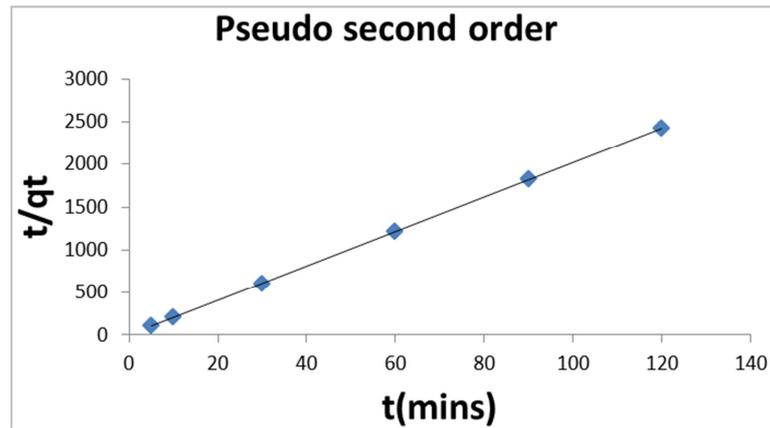


Figure 10. Pseudo second order plot for Malachite green dye adsorption using Orange peel.

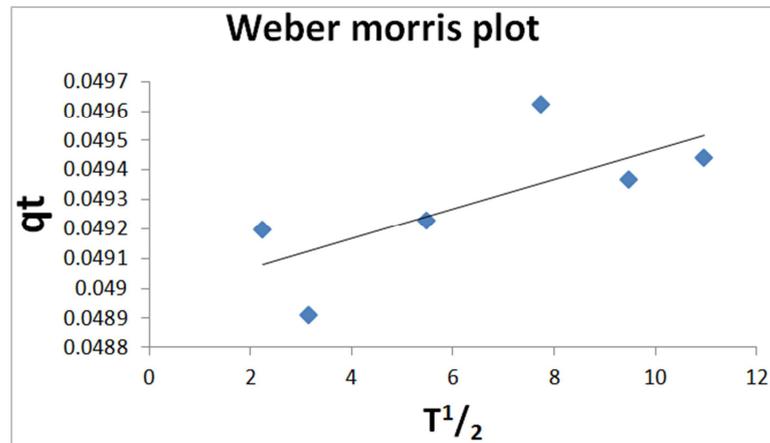


Figure 11. Weber-Morris plot for Malachite green dye adsorption using Orange peel.

The result of the Thermodynamic study of the dye adsorption by the Orange peel is given below:

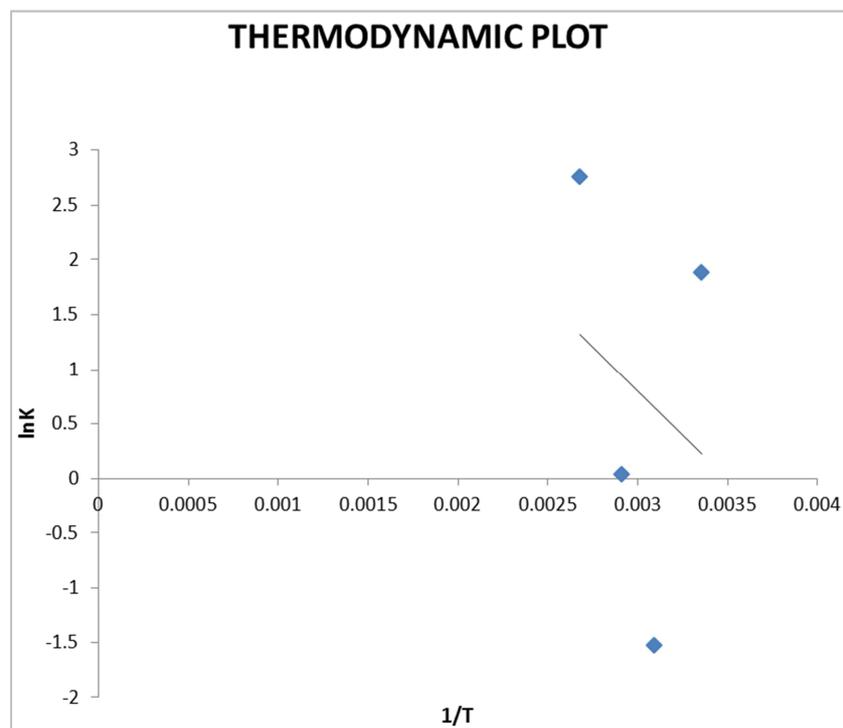


Figure 12. Thermodynamic plot for Malachite green dye adsorption using Orange peel.

Table 6. Thermodynamic Parameters.

TEMPERATURE (K)	ΔG (kJ/mol)	ΔH (kJ/mol)	ΔS (J/mol/K)
298	-553.026	13526.878	47.248
323	-1734.226		
343	-2679.186		
373	-4096.626		

4. Discussion

Table 2 shows that the Orange peels used for this analysis contains 13.25% moisture content, 5.68% Ash content and 4.7% carbon content. This implies that Orange peels contains some vital nutritional content

The FTIR technique is an important tool which is instrumental in the determination of functional groups and organic compounds inherent in the Orange peel. Result of the FTIR spectra is shown in table 3 above. From the table of results, the peak value at 810.5564cm^{-1} and 862.2151cm^{-1} were assigned to C-Cl stretch of chloro compounds. The peak value around 1278.716cm^{-1} , 1617.011cm^{-1} and 3475.009cm^{-1} were assigned to NH stretch of primary and secondary amines respectively. The absorption around 1400.972cm^{-1} was due to C=C of ethene compound. The absorption around 1864.459cm^{-1} was assigned to COO stretching vibration of ester compound whereas the medium band at 2046.426cm^{-1} was assigned to CO stretching vibration of carboxylic compound while the peaks around 1998.430cm^{-1} , 2458.507cm^{-1} , 2554.576cm^{-1} were due to SCN stretch of Thiocyanate compound and CN anti-symmetric stretch of Nitrile compounds. The peak values around 2658.547cm^{-1} , 2784.330cm^{-1} , 2866.735cm^{-1} , 2978.803cm^{-1} were found to correspond to CH symmetric stretch of Methylene and Methyl compounds respectively. The broad band around 3056.479cm^{-1} , 3183.124cm^{-1} , 3593.989cm^{-1} , 3700.514cm^{-1} , 3813.971cm^{-1} were assigned to OH hydroxyl compounds of primary, secondary and tertiary alcoholic compound present in the Orange peel.

According to table 4, the separation factor of the various Langmuir models (R_L) ranged between -1 to 2. To confirm the favorability of adsorption process, the dimensionless equilibrium parameter (R_L) was used. The R_L value of Langmuir I and II model is greater than zero but less than 1 and this means that the process of the isotherms is unfavorable. The R_L value of Langmuir III model is greater than 1; this means the process of adsorption is unfavorable, while the R_L value of Langmuir IV is less than zero. The values obtained for Langmuir I and II is similar to the result obtained by Okafor, *et al.*, 2012. Comparing the Langmuir coefficients of determination (R^2) values between the four different Langmuir models used, Langmuir II has the highest value (0.7612), which means its better fitted than others. The R^2 values of Langmuir isotherm model when compared with other isotherm models, Henry isotherm model had the highest R^2 value (0.8012) which makes it the best fitted isotherm that best describes the isotherm adsorption data. The D-R model (Dubinin and Radushkevich, 1947) was used

to estimate the apparent energy of adsorption. A plot of $\ln q_e$ versus ε^2 gave a linear plot (figure 8) where β and q_e were obtained from the slope and intercept respectively. Similarly, the β value obtained was then used to estimate the mean free energy of adsorption (E). The value of E was found to be 7kJ/mol as shown in table 4 Since E is less than 8kJ/mol, it suggests the adsorption mechanism is physical in nature. Similar result was obtained by Olugbenga and Mohn, 2011 in their experiment on the adsorptive removal of synthetic textile dye using cocoa pod husk.

The result of the kinetic study of adsorption of Malachite green dye onto Orange peel carried out is shown in table 5 and figure 9, 10, and 11. The pseudo first order equation (Lagregren and Svenska, 1898) was used to test the adsorption data. A plot of $\ln(q_e - q_t)$ against t resulted in a graph with negative slope (figure 9). K and q_e were calculated from the slope and intercept, respectively. A plot of t/q_t against t according to figure 10 represented the pseudo second order that was also used to test for the suitable order that best describes the adsorption of Malachite green dye with Orange peel. Comparing the three methods, it can be seen that the pseudo 2nd order has the highest value of R^2 close to 1. Hence, it better describes the adsorption of Malachite green dye using Orange peel unlike the other two methods (1st Order and Weber Morris). Also, looking at the K_{ad} value for the 1st order and 2nd order in table 5, it can be seen that the 2nd order (-0.0235) had the highest value. This indicates that the 2nd order describes better the adsorption of Malachite green dye using Orange peel.

The result gotten from the thermodynamic study as shown in figure 12 and table 6 shows the effect of temperature on adsorption of Malachite green dye. The plot indicates that as temperature increases, the adsorptive capacity also increases. Three thermodynamic parameters were considered to characterize the adsorption process: the enthalpy (ΔH), Gibb's free energy (ΔG) and entropy (ΔS). Due to the transfer of a mole of dye from the solution onto solid-liquid interface, the positive value of ΔH , 13526.878kJ/mol indicates that the adsorption process is endothermic in nature. The positive value of standard entropy change, ΔS , 47.248J/mol/K shows increase in randomness at the solid/solution interface occurring in the affinity of the adsorbent towards the Malachite green molecule. Values of free energy change, ΔG for Malachite green adsorption was negative as shown in table 6, indicating that the adsorption is spontaneous and feasible.

5. Conclusion

Environmental and Climatic problems have been tackled

and are still being tackled presently. Chemists and Scientists generally are in constant research for means by which these pollutants can be reduced. The reduction of these pollutants would encourage healthy living and a wide range of good tidings to mankind. The presence of these dyes in aquatic systems poses heavy risks to human health. Therefore, removal of such dyes from water bodies may be considered an interesting and important research activity. This study shows that orange peel can be used as a suitable adsorbent

for the removal of malachite green dyes from solutions.

This research showed that orange peel fits better for the adsorption of malachite green dye in the Henry isotherm model and that the reaction is an endothermic one as well as a spontaneous and feasible one. It is hereby recommended that orange peels are good adsorbents for the removal of malachite green dyes from aqueous solutions. Therefore, should be employed for the removal of dyes in the environment, as it can be easily gotten even at a low cost.

Appendix

q_e	Amount absorbed at equilibrium (mg/g)
C_e	Equilibrium concentration (mg/l).
q_m	Monolayer adsorption capacity (mg/g).
K	Equilibrium constant in (l/mg),
n	Freundlich exponent
K_F	Freundlich constant in (l/g)
B	$B=1/2a = RT/b$ where a represents the Temkin surface energy
$\beta\epsilon$	DR constant in mol^2/kJ^2 related to the adsorption energy E
q_D	theoretical monolayer saturation capacity in (mg/g)
ϵ	Polanyi potential, equal to $RT \ln(1 + 1/C_e)$.
K_1	Pseudo-first order rate constant in (min^{-1})
K_2	Pseudo-second order rate constant in (g/mg/min)
q_t	Amount in (mg g^{-1}) adsorbed at time t
k_{ID}	$k_{ID}(\text{mg/g/min}^{0.5})$ is the intraparticle diffusion rate constant
I	Intercept which reflects the boundary layer effect/ thickness

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