**American Journal of Chemical Engineering**
2018; 6(6): 126-134
http://www.sciencepublishinggroup.com/j/ajche
doi: 10.11648/j.ajche.20180606.12
ISSN: 2330-8605 (Print); ISSN: 2330-8613 (Online)

# Kinetic Modelling of the Biosorption of Methylene Blue onto Wild Melon *(Lagenariasphaerica)*

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**To cite this article:**
Okon Ebi Ebi, Ademiluyi Falilat Taiwo, Adepoju Tunde Folorunsho. Kinetic Modelling of the Biosorption of Methylene Blue onto Wild Melon *(Lagenariasphaerica)*. *American Journal of Chemical Engineering*. Vol. 6, No. 6, 2018, pp. 126-134.
doi: 10.11648/j.ajche.20180606.12

**Received**: June 6, 2018; **Accepted**: June 26, 2018; **Published**: January 10, 2019

**Abstract:** The Kinetics of the Biosorption of Methylene Blue onto LagenariaSphaerica as adsorbent was studied and the plant was characterized to check it suitability to be used as adsorbent for the treatment of aqueous waste water. The LagenariaSphaerica used was sliced into small sizes, sun dried for 14 days and later oven dried at 105°C for 2 hours to archive bone dryness, it was ground and sieved to various particle sizes and Batch adsorption process was employed. For the study. The effect of process parameters such as Effect of contact time, adsorbent dose, initial concentration of methylene Blue and particle size were investigated. The highest dye uptake stood at 90.1% after 24 hours using an initial dye concentration of 100 ppm and adsorbent dosage of 0.3 g. The experimental data obtained was fitted into adsorption isotherms such as Langmuir and freundlich isotherm model. It was described using linear regression method. Pseudo-first order, pseudo- second order and intra-particle diffusion kinetic models was used to analyze the Experiment. The data best fitted in intra-particle diffusion model and Freundlich isotherm. Considering the result obtained, it can confidently be said that Lagenariasphaerica can be used as an adsorbent for the removal of dye from aqueous solution.

**Keywords:** Biosorption, Methylene Blue, *Lagenariasphaerica*, Adsorbent

## 1. Introduction

Many industries use dyes to colour their final products and their discharge into natural waters causes severe problems because there are toxic to aquatic life and damage the aesthetic nature of the environment. Adsorption is the most widely used separation techniques in the treatment of wastewater. Stirred batch adsorbers are often used in adsorption studies because they offer a number of advantages in comparison to fixed bed systems [1-4]. The most used adsorbent presently is activated carbon which is quite an expensive material [25]. The need to source for a less expensive material as adsorbent became important. Biosorption is said to be the adhesion of molecules of gas, liquid, or dissolved solids to a surface. This process creates a film of the biosorbate (the molecules or atoms being accumulated) on the surface of the adsorbent. Similar to surface tension, adsorption is a consequence of surface energy. In a bulk material, all the bonding requirements (be they ionic, covalent, or metallic) of the constituent atoms of the material are filled by other atoms in the material. Natural plantsare very promising in wastewater treatment because they represent a cheap source of adsorbent, as they are readily available in large quantities, and it has been shown that they display a high metal binding capacity [10-14]. Dyes usually have complex aromatic molecular structures which make them more stable and difficult to biodegrade. Many dyes may cause allergic dermatitis, skin irritation, dysfunction of kidney, liver, brain, reproductive and central nervous system [17-18].

Organic dyes are an integral part of many industrial effluents and demand an appropriate method to dispose them off. There are various conventional methods of removing dyes including coagulation and flocculation, oxidation or ozonation and membrane separation. [9]. Dyes fines application in industries such as textiles, rubber, plastics, printing, leather, cosmetics, etc., and also in production of coloured products. About $7x10^5$ dyes are produced annually in the global market. It is estimated that about 2% of these dyes are discharged into the water system with most of the sources generated from textile industries [28-31].

Various physical, chemical and biological methods have been used for the treatment of dye containing waste water. Physical adsorption technology which is by activated carbon has gain favour in recent years because it is favourable for high stable dye and it is economically feasible when compared with other methods [19-23]. However, activated carbon is very expensive and is not easily regenerated.

The textile dyes are classified into anonic, nonionic and cationic dyes. The major anionic dyes are the direct, acid and reactive dyes[27], and the most problematic ones are the brightly coloured, water soluble reactive and acid dyes (they cannot be removed through conventional treatment systems). The major non-ionic dyes are disperse dyes that does not ionized in the aqueous environment, and the major cationic dyes are the azo basic anthraquinone disperse and reactive dyes, etc. The most problematic dyes are those which are made from known carcinogens such as benidine and other aromatic compounds (i.e. anthroquinone-based dyes are resistant to degradation due to their fused aromatic ring structure). Some disperse dyes have good ability to bioaccumulation, and the azo and nitro compounds are reduced in sediments, other dye-accumulating substrates to toxic amines.

The organic dyes used in the textile dyeing process must have a high chemical and photolytic stability, and the conventional textile effluent treatment in aerobic conditions does not degrade these textile dyes, and are presented in high quantities into the natural water resources in absence of some tertiary treatment. The discharge of MB is a great threat for both toxicological and aesthetical reasons impede light penetration and are toxic to supply food chain for organisms [28].

*Lagenariasphaerica* is an herbaceous climber in the Cucurbitaceae family. It can be described as aPerennial, woody rootstock, Stems annual, robust, trailing, angular, hairless and up to 10 m long or longer. Often completely leafless when fruiting. Leaves rather rigid, shaped like ahand, margins toothed, 50-180 mm long, both surfaces roughly and shortly hairy, upper surface dark green, lower surface paler; foetid when crushed. Leaf stalks 20-80 mm long, with two lateral apical yellow-green glands [32]. It is commonly known as the Wild melon. The plant is found in low-lying areas from the Eastern Cape of South Africa to East Africa and West Africa. They may grow along river floodplains or up into the canopy of riparian forests. They may also be found in coastal dune vegetation. These plants produce large white flowers which attract many insects. The melon bottle gourd is green and flecked with white. The fragrant flowers open around 7:00 a.m., and close in the afternoon. The Mature fruits are poisonous, bitter-tasting, and release strong-smelling volatile compounds. It is observed that the structure of *Lagenariasphaerica* is cellulose based and the surface of cellulose in contact with water is negatively charged [24-25]. Methylene Blue used in this study will dissolve to give the cationic solution and this will undergo attraction on approaching the anionic *Lagenariasphaerica* structure. On this note, it is expected that the cationic Methylene Blue will have a strong sorption affinity for *Lagenariasphaerica.*

For the study, the effect of process parameters such as Effect of contact time, adsorbent dose, initial concentration of methylene Blue and particle size were investigated. The experimental data obtained was fitted into adsorption isotherms such as Langmuir and freundlich isotherm model. It was described using linear regression method. Pseudo-first order, pseudo- second order and intra-particle diffusion kinetic models was used to analyze the data.

# 2. Materials and Methodology

### 2.1. Preparation of Lagenaria Sphaerica

*LagenariaSphaerica*used in this work was gathered from IkotAkpaden, AkwaIbom State. The plant fruit was gathered into a clean plastic bag. They were washed with clean water to remove sand and other impurities, it was then sliced and sun dried for 10 days and later oven dried at 105°C for 2 hours to achieve constant weight (bone dryness), and afterwards ground with a grinding mill. *LagenariaSphaerica*powder were sieved and were of particle size 0.18 mm to 0.45 mm. This was to allow for shorter diffusion path, resulting in a higher rate of biosorption [25].

### 2.2. Preparation of Aqueous Solution

A stock solution of Methylene dye with a concentration of 1000 mg/L (1 g of Methylene Blue in 1 litre of distilled) was prepared. All other working solutions in the batch adsorption process was obtained from the stock solution by diluting with distilled water. The concentrations of the dye in the solutions was determined with a UV-visible Spectrophotometer at 665 nm wavelength [26]

### 2.3. Bulk Density

The bulk density of the adsorbent was determined by washing a 250 ml empty cylinder, clean and dried in an oven. The weight of the cylinder was weighed, then filled with 100 g of the biosorbent untapped to avoid breaking up the agglomerate and the weight was also taken and the bulk density calculated using the formula;

$$Bulk\ density = \frac{w_2 - w_1}{v} \quad (1)$$

Where $W_1$ = weight of empty cylinder
$W_2$ = weight of cylinder filled with sample
$V$ = volume of cylinder.

### 2.4. PH

The pH was determined using the standard method ASTMD 3838-80 [28]. 1 g of *LagenariaSphaerica*sample was introduced into 100 ml of distilled water in a conical flask. The mixture was stirred for 1 hour, the pH value was read using pH meter.

### *2.5. Iodine Number*

A Standard Iodine solution was prepared using 10 ml of 0.1 M Iodine solution in a conical flask.1 drop of Starch solution was added to it which turned pale yellow colour of Iodine Solution Blue. Titration of the formed solution was done with 0.100 M Sodium Thiosulphate till it becomes Colorless. Burette reading corresponds to blank reading (B).0.2g of *lagenariasphaerica* was introduced into 30 ml of 0.1 M Iodine solution in a flask. The flask was agitated for 24 hours and then filtered. The filtrate was collected in a dry flask and then 1 g of the filtrate was titrated againstStandard Sodium Thiosulphate solution using starch as indicator. Burette reading corresponds to (A). The Iodine number was then calculated using:

$$Iodine\ Number = \frac{\mathrm{B-S}}{\mathrm{B}} \times \frac{\mathrm{V}}{\mathrm{W}} \times M \times 126.91 \tag{2}$$

WhereM = Molarity $\left\{\frac{\mathrm{mol}}{\mathrm{dm^3}}\right\}$
B = Blank reading (ml)
S = volume of Thiosulphate used (ml)
V = volume of iodine used. (ml)
W = weight of sample used (g/kg)

### *2.6. Moisture Content*

A small amount of the sample was put in a petri-dish or crucible, covered with a lid and weighed using a weighing balance. The crucible was placed in the hot air oven at 105°C with its lid removed and dried for 3.0 hrs. The crucible was taken out, immediately covered with the lid, cooled in a dessicator and weighed. The process was continued until constant weight was achieved.

$$M = \frac{100(B-F)}{B-G} \tag{3}$$

Where: Weight of empty petri-dish (G)
Weight of empty petri-dish + sample (before heating) (B)
Weight of empty petri-dish + moisture free sample (after heating) (F)
% of moisture content (M)

### *2.7. Ash Content*

The crucible was ignited in the muffle furnace at 750°C for 1.5 hours. The crucible was placed in the dessicator, cooled to room temperature and weighed. 1gm of the sample was put in the crucible and the crucible was placed back in the muffle furnace at 750°C for 1.5 hours. The crucible was taken out of the furnace, placed in the dessicator, cooled to room temperature and weighed.

$$A = \frac{100(B-F)}{B-G} \tag{4}$$

Where: Weightof empty silica crucible (G)
Weight of empty crucible + sample (before heating) (B)
Weight of empty crucible + ash (after heating) (F)

### *2.8. Biosorption Experiment*

The biosorption studies for evaluation of *LagenariaSphaerica*for removal of Methylene Blue from aqueous solutions was carried out in triplet using the batch biosorption procedure [25]. For these experiments, fixed amount of adsorbents (100 mg to 1000 mg) was placed in a 250 ml conical flasks containing 30 ml of Methylene Blue solutions with initial concentrations of 100 ppm which wereagitated for a suitable time of 10, 20, 70, 100, and 120 minutesat room temperature. Subsequently, in order to separate the adsorbents from the aqueous solutions, the flasks were centrifuged at 200 rpm for 10 minutes. The final concentrations of the dye remaining in the solution were determined by UV-visible spectrophotometer. The amount of the dye sorbed and percentage of removal of dye by the biosorbent were calculated by applying the Eqns. (2.5-2.6), respectively:

$$q = \frac{(C_o - C_f)}{m} v \tag{5}$$

$$\%\ Removal = \frac{(C_o - C_f)}{m} . 100 \tag{6}$$

where $q$ is the amount of dye sorbed by the biosorbent (mg/g); $C_o$ is the initial dye concentration put in contact with the biosorbent (mg/L), $C_f$ is the final concentration (mg/L) after the batch biosorption procedure, $V$ is the volume of aqueous solution (L) put in contact with the biosorbent and m is the mass (g) of biosorbent.

## 3. Result and Discussion

### *3.1. Physicochemical Properties*

The physicochemical properties of *Lagenariasphaerica* are showed in the Table 1. The pH of the adsorbent observed was 8.74 as showed in Table 1 below which shows that it is alkaline. When the pH of solution is higher, the surface becomes negatively charged. MB is a cationic dye. Due to electrostatic force of attraction molecules of MB are adsorbed on the surface of adsorbent. Similar trends were observed in literature [33]. The particle size used for the experiment ranged from 0.18 mm to 0.45 mm as showed in Table 4.1 below. This particle size was chosen to reduce the Diffusion path and enhance the adsorption process. Literature has it that, the smaller the particle size, the smaller the diffusion path, the faster the adsorption process [25]. Iodine numberwhich is a measure of the micropore content of the adsorbent, was observed to be 1150 as showed in Table 1. Basically, iodine number is a measure of the iodine adsorbed in the pores and, as such, is an indication of the pore volume available in the adsorbent of interest. Typically, water treatment carbons have iodine numbers ranging from 600 to 1100 (Anon). It can be said that the adsorbent follows the trend. The moisture content measure stood at 5% which is relatively low. Literature has it that the lower the moisture content the more suitable the adsorbent. The bulk density observed was 0.34 g/ml as showed in Table 1. Bulk density is another important physical parameter, especially when an adsorbent product is to be investigated for its filterability. This is because it determines the mass of the adsorbent that can be contained in a filter of given solid capacity and the amount of treated liquid that can be retained by the filter cake [7]. The ash content observed was 22.8% as showed in Table 1. The Ash content, indicates the particle density is relatively small and that the biomaterial should be an excellent raw material for adsorbents. The lower the ash value; the better the biomaterial for use as adsorbent, but a high ash value reduces its efficiency [5-6]. The values of the physico-chemical parameters of *lagenariasphaerica* are in the range with those reported in the literature for Biosorbent.

***Table 1.*** *Physico-Chemical property lagenariasphaerica.*

| Properties | Values |
|---|---|
| pH of Adsorbent | 8.74 |
| Particle size (mm) | 0.18 to 0.45 |
| Iodine Number | 1150 |
| Moisture content | 5% |
| Bulk Density(g/ml) | 0.34 |
| Ash content | 22.8% |

### *3.2. Langmuir Adsorption Isotherm*

$$\frac{c_{eq}}{q_{eq}} = \frac{1}{bq_{max}} + \frac{1}{q_{max}} c_{eq} \qquad (7)$$

It is known that $K_L$ = the constant related to the energy of adsorption (Langmuir Constant). $R_L$ value indicates the adsorption nature to be either unfavourable if $R_L$>1), linear if $R_L$ =1, favourable if 0< $R_L$<1 and irreversible if $R_L$=0 [8]. $q_{max}$ is the maximum specific uptake corresponding to the site saturation. From the data calculated in table 2, the $R_L$ is greater than 0 but less than 1 indicating that Langmuir isotherm is favourable, $K_L$ (Langmuir isotherm constant) is 0.014 L/mg, $q_{max}$ is 27.03 mg/g, $R_L$ (the separation factor) is 0.42 indicating that the equilibrium sorption was favourable and the $R^2$ value is 0.96 as shown in Figure 1a, proving that the sorption data fitted well to Langmuir Isotherm model.

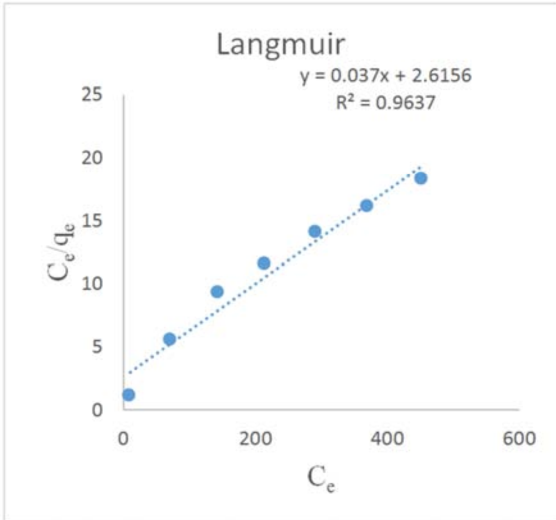
### *3.3. Freundlich Adsorption Isotherm*

$$\log q_{eq} = \log k_f + \frac{1}{n}\log c_{eq} \qquad (8)$$

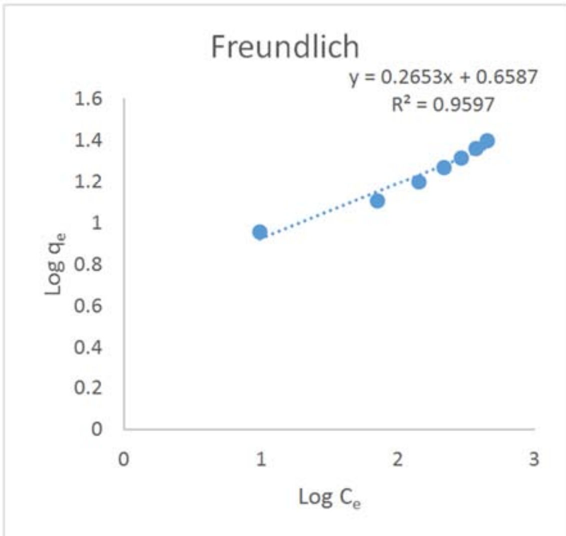
Where $C_{eq}$ is the equilibrium concentration (mg/l), $q_{eq}$ is the amount adsorbed (mg/g) and $K_F$ and n are constants incorporating all parameters affecting the adsorption process, such as adsorption capacity and intensity respectively [15-16]. If 1/n = 1 then the partition between the two phases are independent of the concentration. If the value of 1/n is below one it indicates a normal adsorption. On the other hand, 1/n being above one indicates cooperative adsorption [8]. However, $K_f$ and n are parameters characteristic of the sorbent-sorbate system, which must be determined by data fitting and whereas linear regression is generally used to determine the parameters of kinetic and isotherm models. Specifically, the linear least-squares method and the linearly transformed equations have been widely applied to correlate sorption data where 1/n is a heterogeneity parameter, the smaller 1/n, the greater the expected heterogeneity. This expression reduces to a linear adsorption isotherm when 1/n = 1. If n lies between one and ten, this indicates a favourable sorption process [8]. From the data in table 2, $k_f$ is 4.56 mg/g, that value of 1/n = 0.25 while n= 3.9 indicating that the sorption of methylene blue unto *Lageneriasphaerica*is favourable and the $R^2$ value is 0.96 as shown in Figure 1b.

***Table 2.*** *Langmuir, Freundlich Isotherm constants for the adsorption of methylene blue unto Lageneriasphaerica.*

| Langmuir Isotherm | Value | Freundlich Isotherm | Value |
|---|---|---|---|
| qmax (mg/g) | 27.03 | 1/n | 0.25 |
| RL | 0.42 | n | 3.9 |
| KL(L/mg) | 0.014 | Kf (mg/g) | 4.56 |
| R2 | 0.96 | R2 | 0.96 |




Subfigure (A)




Subfigure (B)

***Figure 1.*** *Langmuir and Freundlich isotherm plot for the Biosorbent of Methylene Blue Dye onto Lagenariasphaerica is shown in Subfigure (A) and (B) respectively.*

### *3.4. Scanning Electron Microscopy (SEM)*

Scanning electronic microscopy (SEM) study is one of the most popular, primary, and widely used characterization techniques applied for the study of surface properties and morphology of biosorbent material. Moreover, SEM study also tells about porosity and texture of biosorbent material. [6]. Figure 2shows that *Lagenariasphaerica*powder has small cavities on surface and has a porous texture that may provide large surface for the adsorption of the dye molecules.

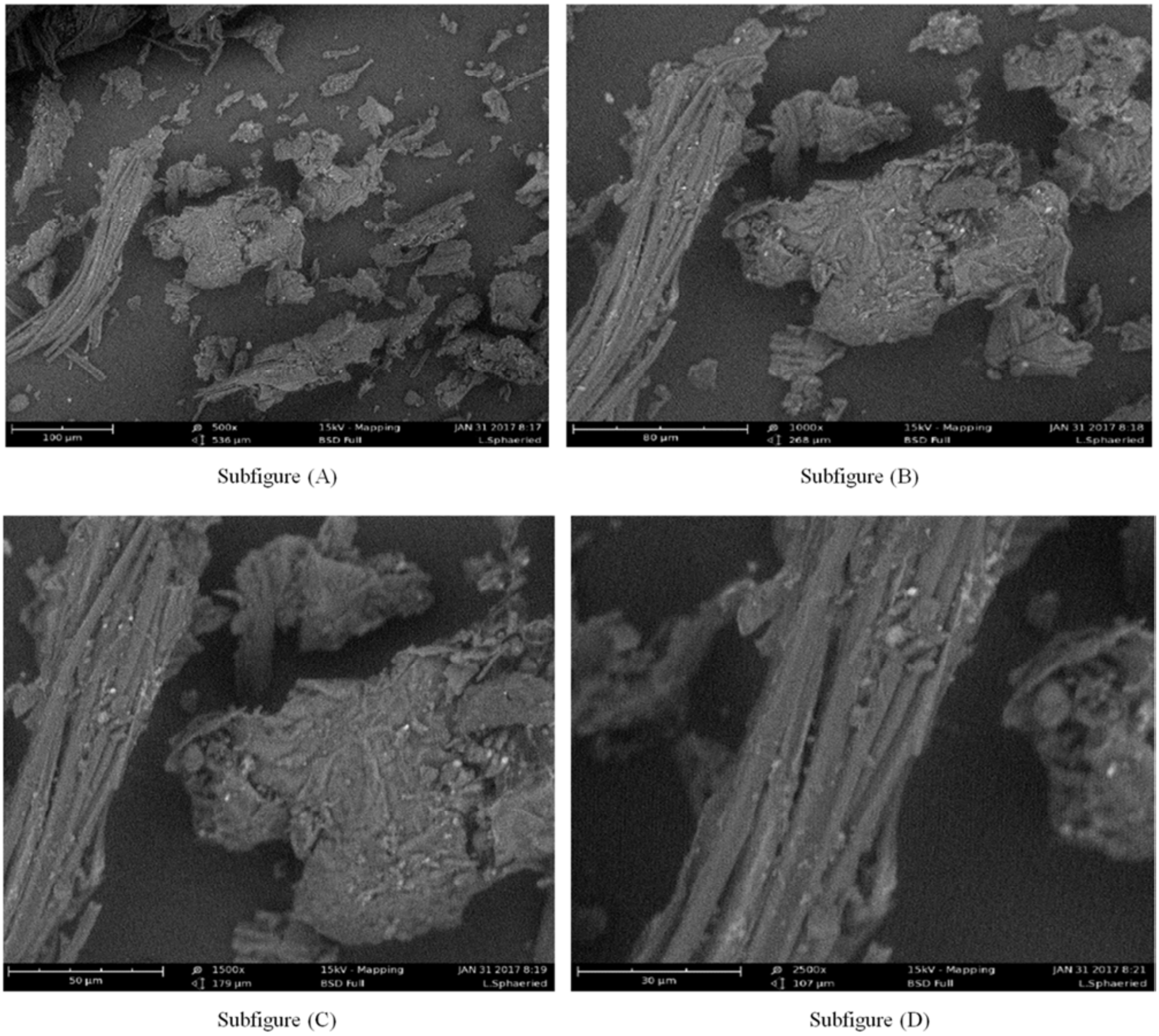



***Figure 2.*** *Scanning electron microscopy of Lagenariasphaericabiosorbent: (A) 500x; (B) 1000x; (C) 1500x; (D) 2500x.*

### *3.5. Effect of Particle Size*

The Experimental results for the sorption of Methylene Blue dye onto *Lagenariasphaerica*are shown in Figure 3. The result show that the % removal of the dye from the aqueous solution increases from 33.9% to 70.2% with the decrease of the adsorbent particle size ranging from 0.45mm to 0.18mm. This clearly shows that the smaller the particle size, then for a given mass of *Lagenariasphaerica,* more surface area is made available for adsorption to take place and the number of active sites is increased. Consequently, it is very clear that adsorption equilibrium capacity is dependent on the particle size. From kinetics, it can be said that methylene blue sorption on different particle sizes of *Lagenariasphaerica* follows the Intra- particle diffusionmodel. Figure 6a shows the applicability of the Intra- particle diffusion model for the systems of methylene blue with *Lagenariasphaerica*for particle sizes ranging from 0.18 mm to 0.45 mm. The correlation coefficient $R^2$ for the Intra- particle diffusion model were all higher than 0.991 for all the systems studied while the correlation coefficient for Pseudo second order and pseudo-first order were all higher than 0.980 and 0.740 for all systems studied, respectively.

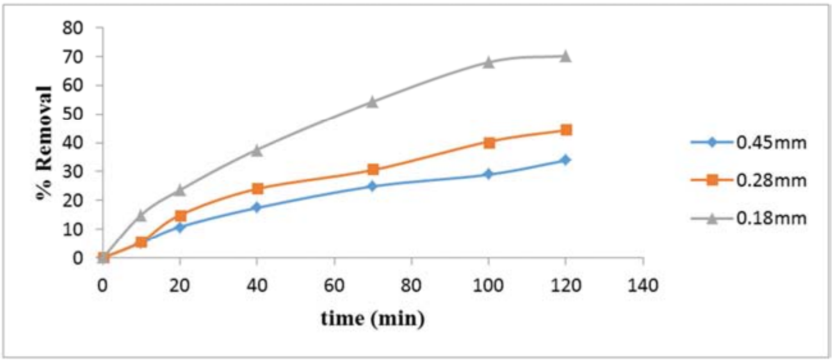



***Figure 3.*** *A plot of % removal Methylene Blue against time for different particle sizes constant dye concentration of 100 ppm and constant adsorbent dosage of 0.3 g at different time intervals.*

### *3.6. Effect of Adsorbent Dosage*

The effect of adsorbent dosage was studied in contact time of 2 hours. It was observed that the % removal of methylene blue dye from the aqueous solution increased from 42.8% to 77.2% as the adsorbent dosage was increased from 0.1 g to 0.5 g as shown in Figure 4. Kinetics shows that the difference in adsorbent dosage follows the pseudo-second order rate equation with the highest $R^2$ value of 0.9997 as seen in Figure6b.

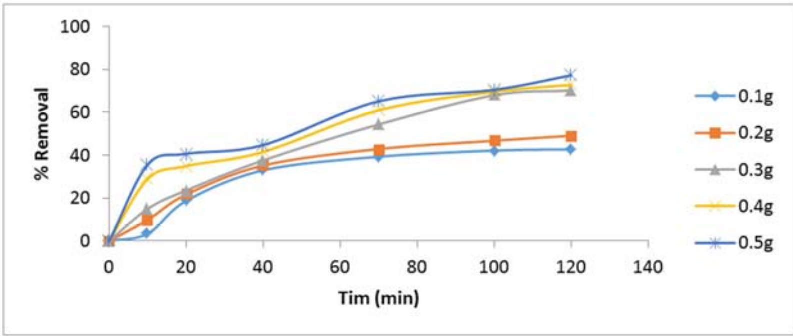



***Figure 4.*** *A plot of % removal Methylene Blue against time for different adsorbent dosage at constant dye concentration of 100 ppm and at for different time intervals.*

### *3.7. Effect of Initial Dye Concentration*

The influence of initial dye concentration on the adsorption of methylene blue dye on *Lagenariasphaerica*was studied. The % removal of methylene blue dye from the solution dropped from 70.211% to 49.154% as the initial concentration was increased from 100 ppm to 500 ppm as shown in Figure 5. This clearly shows that the higher the initial concentration of the solution, the lower the adsorbance for a given as of the adsorbent. The kinetics of methylene blue sorption on *Lagenariasphaerica*follows the intra-particle diffusion model where the highest $R^2$ was 0.995 and value for all the systems under studies was higher than 0.95 as shown in Figure6c.

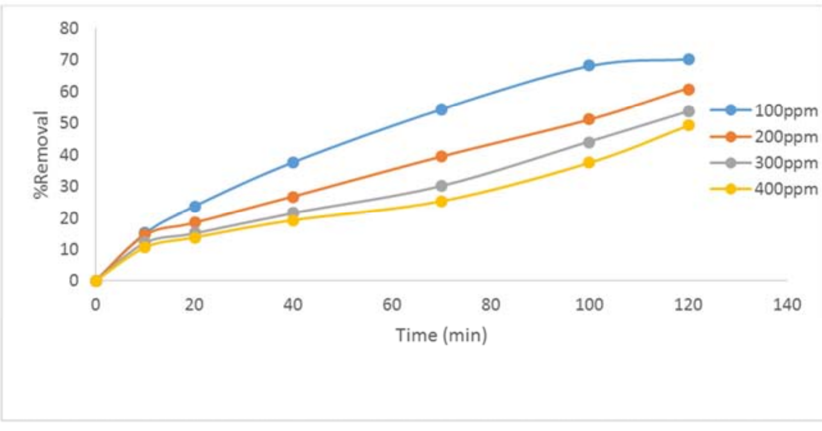
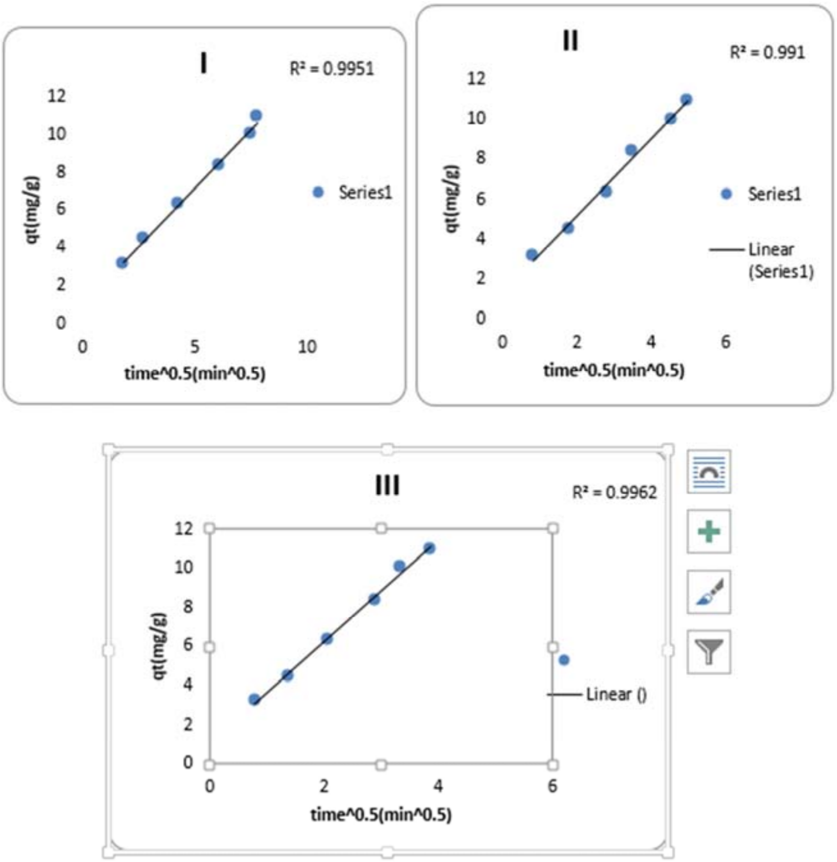
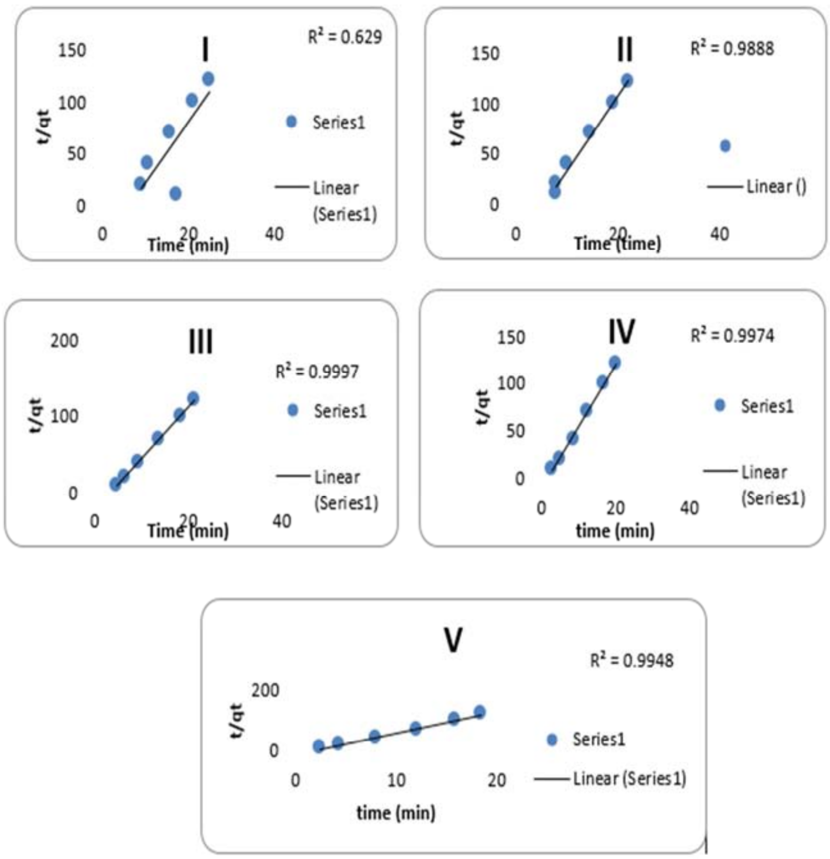


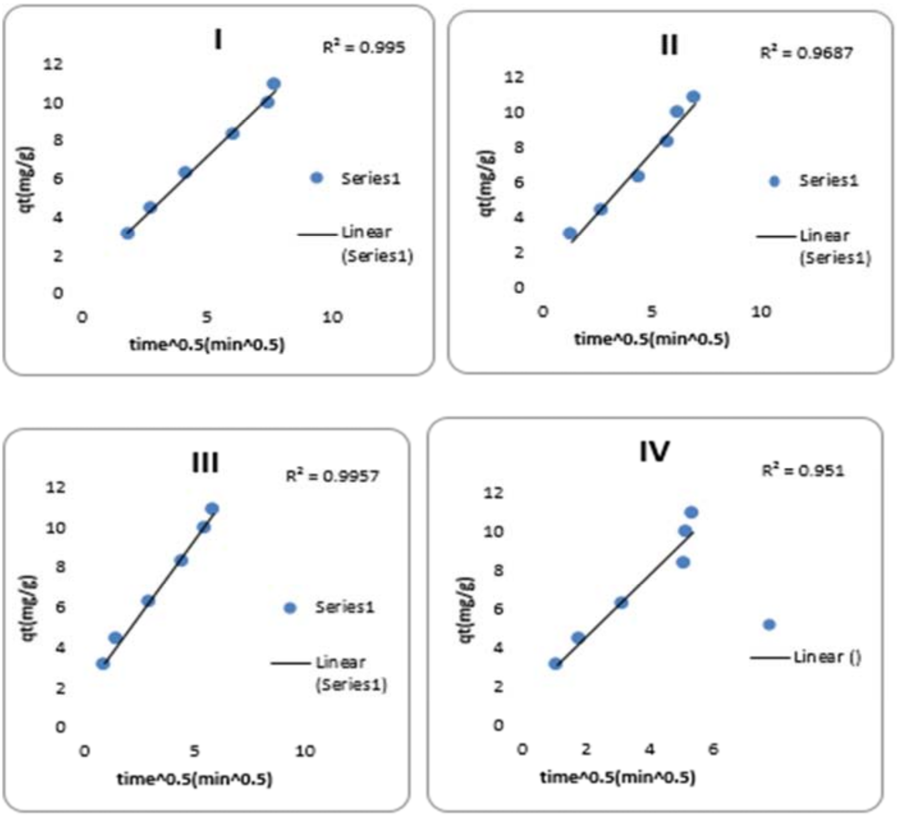

***Figure 5.*** *A plot of % removal of Methylene Blue against Time for different initial dye concentrations at constant adsorbent dosage of 0.3 g at different time intervals.*




Subfigure (A).




Subfigure(B).




Subfigure (C).

***Figure 6.*** *Plots of the kinetic models; SubFigureure (A) Intra- particle diffusion plot ofIn($q_e$-$q_t$) against time for different particle sizes 0.18 mm(I), 0.28 mm(II) and 0.45 mm(III), at constant Dye concentration of 100 ppm and constant adsorbent dosage of 0.3 g at different time intervals.; SubFigureure (B) Pseudo-second order plot ofIn($q_e$-$q_t$) against time for different Dosage 0.1 g(I), 0.2 g(II), 0.3 g(III), 0.4 g(IV), 0.5 g(V)at constant Dye concentration of 100 ppm at different time intervals.; SubFigureure (C) Intra- particle diffusion plot of In(qe-qt) against time for Different initial dye concentrations 100 ppm(I), 200 ppm(II), 300 ppm(III), 400 ppm(IV) at constant adsorbent dosage of 0.3 g at different time intervals.*

# 4. Conclusion

The kinetics of sorption of methylene blue on *Lagenariasphaerica*was studied. The results of this investigation show that *Lagenariasphaerica,* an agricultural waste comprising of the mesocarp and seed has a suitable adsorption capacity for the removal of methylene blue dye from aqueous solutions. The maximum percentage removal of methylene blue dye from the aqueous solution with initial concentration of 100 ppm after 24 hrs stood at 90.1%. The experimental results were fitted in Pseudo-first order, Pseudo-second order and intra-particle diffusion kinetic models and also in Langmuir and freundlich isotherms. The data indicates that the adsorption kinetics follows the Pseudo-second order and intra-particle diffusion models, Langmuir and freundlich isotherm best described the batch process. It can be concluded that *Lagenariasphaerica*unmodified is a potential and active biosorbent for the removal of methylene blue dye from industrial waste water.

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