KINETIC STUDIES OF METHYLENE BLUE ADSORPTION ON TO ACTIVATED CARBON FROM SEA SNAIL SHELL WASTE *(BITHYNIA TENTACULATA)*

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ABSTRACT

Activated carbon has been prepared from sea snail shell waste by chemical activation method using calcium chloride as activating agent. The effects of activating agent concentrations (2.5 M and 3.0 M) of calcium chloride and activation temperature (700°C- 900°C) were studied. The activated samples were characterized on the physical properties which showed increase with increasing temperature except for density and ash content that decreased; moisture content. A kinetic study was performed and experimental data fitted to both pseudo first and second order kinetics equation, which gave values of k_1 = 0.115 min and k_2 = 0.0114 min/mol. L. The pseudo second order described better with r^2 = 0.998 compared to first order model and intra -particle diffusion model. The experimental data was also fitted to Langmuir, Freundlich and Nerst models. The correlation coefficient of Langmuir model was higher r^2= 0.998 compared to Freundlich and Nerst showing monolayer adsorption. The adsorption capacity q_{max} and n values were also determined; 0.752 and 1.656 respectively. Iodine number was also determined which is an equivalent to surface area of the activated carbon available for the adsorption of ions in aqueous solution. There was no marked difference in the result with varying activating agent concentrations.

KEYWORDS:

Pseudo Kinetic Studies, Activated carbon, Sea Snail Shell, Adsorption

1. INTRODUCTION

Activated carbons form a large and important class of porous solids, which have found a wide range of technological applications [1]. The characteristics and structural properties of activated carbon depend on the physical and chemical properties of the precursor, method of activation, oxidizing agent, time and temperature employed. Activated carbons are carbonaceous material that can be distinguished from elemental carbon by the oxidation of the carbon atoms found on the inner and outer layers [2]. These materials are characterized by their large surface area, porous surface containing functional group. For this reason, activated carbons are widely used as adsorbents for the removal of organic chemicals and pollutant from air, gases, portable water and also in wastewater treatment [3]. The surface oxygen functional groups can be easily introduced to the carbon by different activation methods including dry and wet oxidizing agents. Wet oxidation methods involve the reaction between the carbon surface and solutions of oxidizing agents such as phosphoric acid (H_3PO_4), Nitric acid (HNO_3), Zinc chloride ($ZnCl_2$), Potassium hydroxide (KOH), Potassium permanganate ($KMnO_4$), Calcium chloride ($CaCl_2$) etc. One of the fastest growing areas is the environmental application of activated carbon to treat effluent from textile industry. These effluent when discharged, defaces the look of natural water and highly toxic [4], also causes harm to mammalian cells by causing kidney tumors [5, 6]. Many treatment methods have been employed to remove dyes from wastewater; using physical and chemical methods [7] which are considered expensive in terms of energy and reagents consumption [8, 9].

In recent times, the interest in the production of activated carbon from agricultural waste cannot be over emphasized; an added value to waste. Several substances have been screened as alternative to conventional raw materials for the production of activated carbon which include; palm seed coat [10], coconut shells [11], sepiolite pellet [12], and bamboo stem wastes[13] tridax procumbens 14], olive stones [15], cow bone [16], land snail shell [17], orange waste [18], terminalia arjuna nuts [19], euphorbia antiquorum L[20], periwinkle shell [21]. The present study will consider sea snail shell to produce activated carbon for the adsorption of model solution of standard dye (methylene blue) for kinetic study.

2. MATERIALS AND METHOD

2.1 Materials

Sea snail shell precursors were supplied by Amassoma traders, Bayelsa state. Other materials include, calcium chloride dihydrate ($CaCl_2(OH_2)_2$) laboratory standard reagent, distilled water and Equipments include: weighing balance, (Denver Instrument Vecstar Muffle Furnace, Oven (Thermostatic Drying Oven DHG-9202), **d**esiccators, pH meter, UV spectrophotometer and Moisture analyzer.

2.2 Cabonization

Fresh sea snail shells were washed with di-ionized water, sun dried for 24 hours to drain off water and further dried in an oven at 105^{o}C to complete dryness. The dried shells were crushed into smaller sizes and stored in air tight container for the carbonization process. The dried samples were carbonized using the Vecstar Muffle Furnace at high temperatures of 700^{o}C, 800^{o}C, and 900^{o}C for 3 hours 30 minutes due to the hard nature of the precursor and cooled to room temperature, stored in discicator for the activation.

2.3 Chemical activation.

50 g of the carbonized sample was mixed with 0.053L of 2.5 M calcium chloride solution in a beaker. The mixture was left for 5 minutes to allow escape of gases formed in the exothermic reaction before covering with a lid and left for 24 hours. The activated carbon was filtered out, washed with di-ionized water to remove any residual chemical and allowed to be drained completely. The sample was dried in oven at 105^{o}C for 1 hour 25 minutes. The same process was repeated with 3.0 M of calcium chloride. The activation temperatures were 700^{o}C, 800^{o}C, and 900^{o}C. For easy description samples were identified as 1A700, 1B800, 1C900 and 2A700, 2B800, 2C900 for concentrations of 2.5 M and 3.0 M respectively.

2.4 Characterization

Characterization of samples were carried out on pore volume, porosity, moisture Content, Ash Content, pH, bulk density and iodine number described earlier [16].

2.5 Batch adsorption study

The batch adsorption study was conducted on activated carbon using methylene blue by varying the dosage of activated carbon. 5 % solution of methylene blue was prepared and mixed with distilled water up to 0.1 L. The activated carbon 1C900 and 2C900 with high pore volume and porosity were used for the adsorption study. A kinetic study was carried out with 0.5 g of the activated carbon sample on methylene blue solution with an interval of 30 minutes and solution of methylene blue was withdrawn for the kinetic study.

The quantity of sample was varied from 0.5 g to 2.5 g of activated carbon and was added into 0.02 L of the methylene blue solution and mixed thoroughly by shaking it in 8 minutes and left for 1 hour to settle. The solution was filtered using a filter paper and the filtrate was analyzed using UV spectrophotometer with a wave length of 640 nm. The amount adsorbed at equilibrium, q_e (mg g^{-1}), was calculated by

$$q_e = \frac{(C_0 - C_e)V}{1000W} \quad (1)$$

where C_0 and C_e (mg l^{-1}), are the liquid-phase concentration of methylene blue at initial and final equilibrium respectively. V is the volume of the solution (liter), and W is the mass of the dry adsorbent used (g).

2.6 Iodine number

50 mg of activated carbon was placed in a 0.025 L conical flask and 0.02 L of the reagent was transferred quantitatively into the flasks followed by addition of 0.015 L of chloroform. The flask and its content were kept in the dark in a laboratory cupboard for 11 hours for halogenation to take place. 0.01 L of saturated potassium iodide was added to each flask. The burette was then filled to the 0.05 L mark with 0.1M solution of sodium thiosulphate. 0.05 L of distilled water was further added to the flask and titrated against the thiosulphate until the solution turns pale yellow. 3 drops of freshly prepared starch indicator was added which turns blue into black. The titration continued until the solution turns colorless. A blank titration was also conducted with chloroform.

Iodine number was calculated using the formula:

$$\text{Iodine No} = \frac{(B - S) * M * 12.69}{sample\ weight} \quad (2)$$

where B = blank titration, S = sample M = molarity of thiosulphate, 12.69 was used as conversion from meq thiosulphate to g iodine, M.W iodine = 127.9

3. RESULTS AND DISCUSSION

3.1 Characterization of activated carbon

The properties of the characterized sample are presented in Table 1. The pH value of activated carbon is a measure of acidity or basicity which influences adsorption by affecting the surface properties of the adsorbents and ionization or dissociation of the adsorbate molecules. At higher pH value, dye adsorbed on the adsorbent surface can decrease considerably. For colour adsorption, its efficiency decreases in an alkaline medium. As can be seen in Table 1, the activated carbon produced with the impregnation of 2.5 M and 3.0 M $CaCl_2$ fall within an alkaline range (8.49 - 8.60). In sugar decolourization, a distinctive acidic activated carbon may cause inversion of sucrose while alkaline can cause degradation or organic impurities. Hence, an activated carbon with pH value range 6-9, has found to be acceptable for most applications [20, 22]. Density is also important in many industrial applications, especially in sugar decolourization where high viscosity syrup is displaced through a column of activated carbon. The density obtained ranges from 2.5 - 0.7 mg/L comparable to work reported in literature [23]

The ash content is a residue, inorganic, inert and unusable part present in the activated carbon when these are burnt off. The ash content consists mainly of aluminum, iron, silica, magnesium, and calcium. The lower the ash content, the better the activated carbon. Sample C1900 and C2900 have relatively lower ash content (40.50 -37.00). The ash content decreased with the increase in temperature. The moisture content obtained was 0.22 and 0.24 for samples 1C900 and 2C900 comparable to results reported in literature [22] using *Delonix regia* fruit pod (0.22 ± 0.14). The pore volume also increased with increase in temperature. A similar trend was observed with $CaCl_2$, where there was also an increase in pore volume as the temperature was increased from 700^{o}C to 900^{o}C [17].

Table 1: Physical properties of activated carbon impregnated with $CaCl_2$

Temperature (^{0}c)	**pH**	**Bulk density (mg/L)**	**Ash content (%)**	**Moisture content (%)**	**Pore volume (m^3)**	**Porosity**
1A (700) 2.5M	8.49	2.50	43.50	0.80	0.28	0.156
1 B(800)	8.54	1.25	41.50	8.00	0.38	O.19
1C (900)	8.60	0.714	40.50	11.00	0.80	0.314
2A(700) 3.0M	8.58	2.00	43.00	1.00	0.25	0.139
2B(800)	8.56	1.429	38.00	10.00	0.42	0.21
2C(900)	8.58	1.042	37.00	12.00	0.77	0.275

3.2 Effect of dosage of activated carbon

The effectiveness of activated carbon is specified by the amount of substance it can adsorb per unit weight of activated carbon. This is helpful in model prediction for analysis and design of an adsorption process. Figure 1 shows that the percentage removal of methylene blue increased with increase in adsorbent dose. The adsorption of adsorbate increases with an increase in the availability of more adsorption sites resulting from the increased adsorbent dose [24]. The initial uptake of adsorbate was about 80 % at optimum adsorbent value of 0.5 g. The percent uptake remained constant with the increase in amount of dosage. This can be attributed to saturated sites which could no longer accommodate adsorbate molecules. Varying the reagent concentration did not affect the percentage removal of methylene blue.

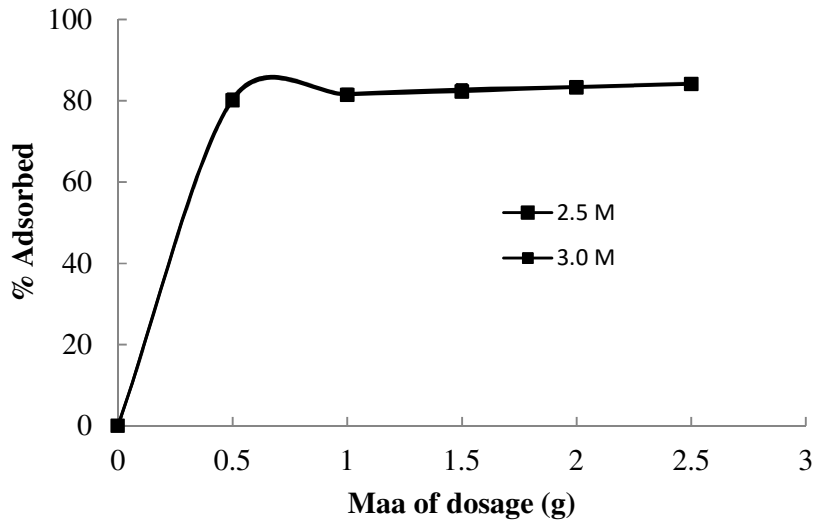

Figure 1. % Adsorbed of Methylene Blue on to 1C900 and 2C900 at various concentration of activation reagent; Adsorbent dosage 0.5 g to 2.5 g

3.2 Kinetics studies

The factors that control the rate and mechanism of adsorption are physical and/or chemical properties of adsorbent, ambient temperature, solution pH and nature of absorbate [20]. The kinetic models proposed by research groups are useful for design and optimization of effluent treatment processes. The models considered to study the mechanism of methylene blue adsorption on to activated carbon prepared from sea snail shell are: pseudo first order, pseudo second order and intra-particle diffusion.

The proposed pseudo first order kinetic model [19] with the linear form is given as

$$\ln\left(q_e - q_t\right) = \ln q_e - \frac{k_1}{2.303}t \tag{3}$$

k_1 is rate constant, q_e and q_t are the amount of methylene blue adsorbed at equilibrium and time (min), respectively. The values of k_1 and q_e were determined from the linear plot of $\ln\left(q_e - q_t\right)$ versus t from the slope and intercept (Figure not shown). The rate constant k_1 was found to be 0.115 min with correlation coefficient of 0.817. The r^2 value, .> 0.8 and 60 % deviation between the experimental and calculated adsorption capacities $q_{e\,exp}$ and $q_{e\,cal}$ suggest that the adsorption data fitted poorly to pseudo first order kinetics.

3.2.1. Pseudo Second order kinetics

The linearized form of the pseudo second-order model based on equilibrium adsorption is expressed as:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^{\,2}} + \left(\frac{1}{q_e}\right)t \tag{4}$$

Where k_2 (g/mg min) is the rate constant of the pseudo second order model, the initial adsorption rate, h (mg/g.min) as t approaches zero can be defined as $h = k_2 q_e^2$. Figure 2 shows the pseudo second order plot for the adsorption of methylene blue by SSSAC and the results are presented in Table 2. The q_e and k_2 values can be determined from the slope and intercept of the plot between $\frac{t}{q_e}$ *and* t . The theoretical values of of the adsorption capacities q_e of the kinetic model were compared to the actual value which is presented in Table 2. It can be observed that the actual value is closer to the theoretical value obtained with pseudo second order; 11.28 mg/g and 12.82 mg/g respectively. A deviation of 5.6% less than that obtained with pseudo first order kinetics. The rate constant k_2 was found to be 1.142 x10^{-2} g/mg.min with the highest correlation coefficient of 0.998. The initial adsorption rate h, was determined to be 1.67 x 10^{-3} mg/g.min. The result showed that pseudo second order kinetics describes the adsorption of methylene blue onto the activated carbon and it is chemical adsorption that is said to be the slowest mechanism [25].

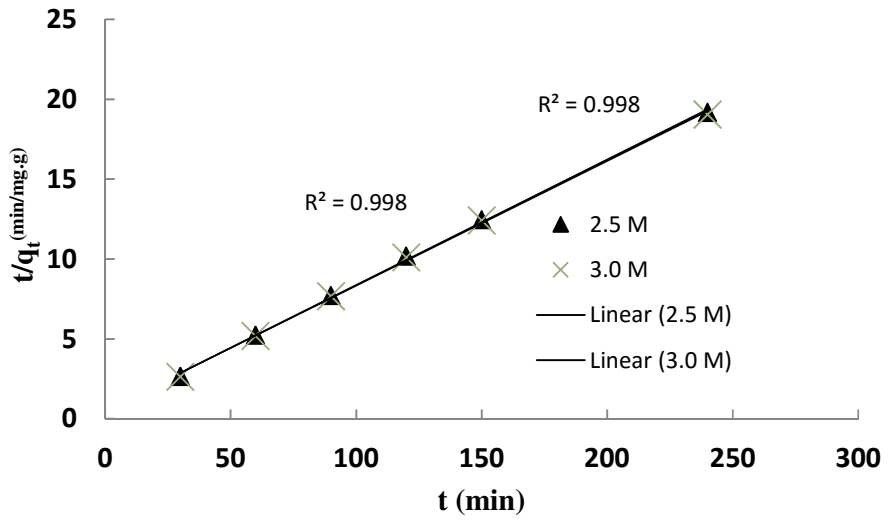

Figure 2. Pseudo second order kinetics for adsorption of methylene blue on to 1C900 and 2C900; Adsorbent dosage, 0.5 g

3.2.2 Intra particle diffusion model

In adsorption process, initial adsorption occurs on the surface of the adsorbent but there is also possibility of the adsorbate to diffuse into interior pores of the adsorbent. A proposed kinetic model [26] suggesting if the adsorption is intra particle diffusion exists is given in equation 6.

$$q_e = k_d t^{1/2} \qquad (6)$$

Where k_d is the intra particle diffusion rate constant, which is determined by the plot of q_e versus $t^{1/2}$ (See Figure 3) and the results are presented in Table 2. The intra particle diffusion rate constant was found to be 0.119, with a correlation coefficient r^2 ranging from 0.963 to 0.966. As can be seen, the linear plot did not pass through the origin which may be due to the variation of mass transfer in the initial and final stages of adsorption [27]. This deviation and the high

correlation coefficient indicates that pore diffusion may be the controlling step and play a major role for the adsorption of methylene blue by the activated carbon prepared [20].

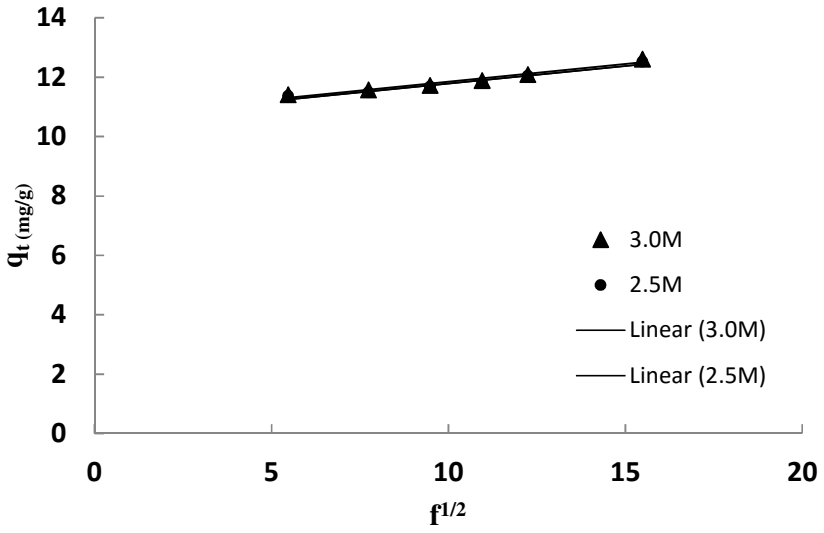

Figure 3 Intra particle diffusion plot for the adsorption of methylene blue on to 1C900 and 2C900 ; adsorbent dosage , 0.5 g

3.3 Isotherm studies

The adsorption isotherm data were fitted to both the Langmuir, Freudlich and Nerst models. The linearized plots are shown in Figures 4, 5 and 6 respectively.

3.3.1 Langmuir Isotherm model

The linear form of Langmuir's isotherm model is given in equation 7.

$$\frac{c_e}{q_e} = \frac{1}{q_{\max}} k_L + \frac{1}{q_{\max}} c_e \qquad (7)$$

Where

C_e is the equilibrium concentration of the adsorbate (Methylene blue) (mg/l), q_e is the amount of adsorbate adsorbed per unit mass of adsorbate (mg/l), $\frac{1}{q_{\max}} k_L$ is the intercept which is related to monolayer adsorption of adsorbent towards adsorbate., $1/q_{max}$ is the slope obtained from the plot of C_e/q_e vs C_e, shown in Figure 4. K_L/q_{max} and $1/q_{max}$ were calculated from the plot. The values are presented in Table 2. The q_{max} is related to adsorption capacity, (mg/g) while K_L is the energy of adsorption (L/mg) and these values were determined from the slope and intercept of the linearized plot shown in Figure 4. The q_{max} was found to range from 0.752 mg/g to 0.805 mg/g, comparable with values in literature (0.2191-0.0558) for adsorption of lead from industrial wastewater onto commercial activated carbon and periwinkle shells [21].

A high correlation coefficient value greater than > 0.9 was obtained, indicating a better fit to the experimental data. Similar results have been found in literature in the adsorption of methylene blue by coconut shell activated carbon [28].

The essential characteristics of Langmuir isotherm which is expressed by a dimensionless separation factor R_L [29] is given as:

$$R_L = \frac{1}{\left(1 + K_L C_0\right)} \tag{8}$$

The value of R_L indicates the nature of the adsorption process, $R_L > 1$ unfavourable, $R_L = 1$ Linear, $0 < R_L < 1$ favorable, and $R_L = 0$ irreversible, Where K_L is the Langmuir constant and C_0 is the initial concentration of adsorbate (mg/L). The R_L value was found to range from 0.014 to 0.013 which indicates a favorable adsorption process. The increase in concentration of activating reagent from 2.5 M to 3.0 M decreases the values of both R_L and K_L. The K_L value was found to vary from 75.5 to 72.3.

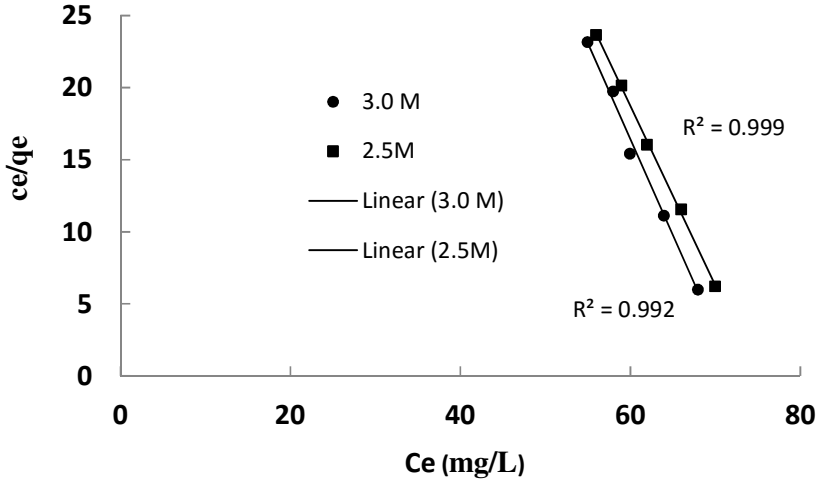

Figure 4 Langmuir isotherm for the adsorption methylene blue on to 1C900 and 2C900 at various concentration of activation reagent, Adsorbent dosage , 0.5 g

3.3.2 Freundlich isotherm

The well known logarithmic form of Freundlich model is given by the following equation

$$Log\, q_e = Log\, K_f + \frac{1}{n} Log\, C_e \tag{9}$$

Where q_e is the amount adsorbed at equilibrium (mg/g), C_e is the equilibrium concentration of the adsorbate. K_f is the adsorption capacity of the adsorbent. $(mg/g^{-1})(mg^{-1})^{1/n}$, n indicates how favourable the adsorption process is, $\frac{1}{n}$ is the slope, ranging between o and 1; a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero. The values can be calculated from the slope and intercept of the plot log q_e versus log C_e. The K_f and n values were found to be 3.48 and 1.656, with the sample activated with concentration of 2.5M. The result is comparable to values obtained by activated carbon prepared from sugarcane and chelex [30]. The n values between 1 and 10 represent beneficial adsorption [20,]. The value of n being greater than 1.0, indicates that the adsorption of methylene blue on to

1C900 and 2C900 the process is beneficial. Freundlich model fits well to data at higher and intermediate concentrations because the equation does not approach Henry's Law of ideal dilute solutions [20].

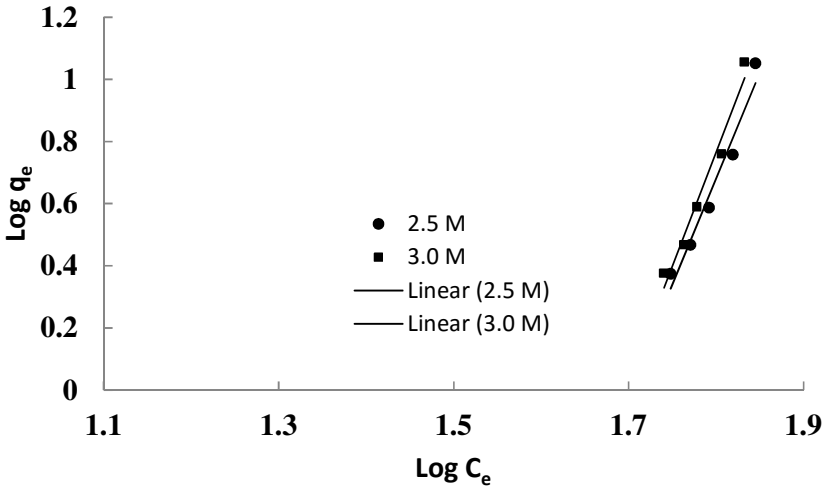

Figure 5 Freundlich isotherm for the adsorption of methylene blue on to 1C900 and 2C900 at various concentration of activation reagent, Adsorbent dosage, 0.5 g

3.3.3 Nerst Model

The Nerst equation is characterized by only one constant, K_p the partition ratio, also called the distribution coefficient which can determine from equation 10.

$$q_e = k_p c_e \tag{10}$$

Nesrt isotherm can be used to describe the distribution of a solute between two immiscible solvent; similar to the situation where the absorbate dissolves in the solid adsorbent [31]. The partition ratio is used to compute the standard affinity of adsorption given in equation 11.

$$\Delta \overset{o}{U} = -RT \ln K_p \tag{11}$$

Where R is the gas constant and T is the absolute temperature.

The partition constant for the adsorption of methylene blue by 1C900 and 2C900 is presented in Table 3. The partition constant ranges from 0.671 to 0.604, with coefficient values of $R^2 > 0.8$. The standard affinity of adsorption was found to be positive; 1.22 and 0.96. Similar results were reported [32], where a partition value of 0.726 was recorded using activated carbon prepared from biopolymer chitin to adsorb xylenol orange whilst 9.571 and 8.784 were obtained by [33] with ACZN and ACSN respectively.

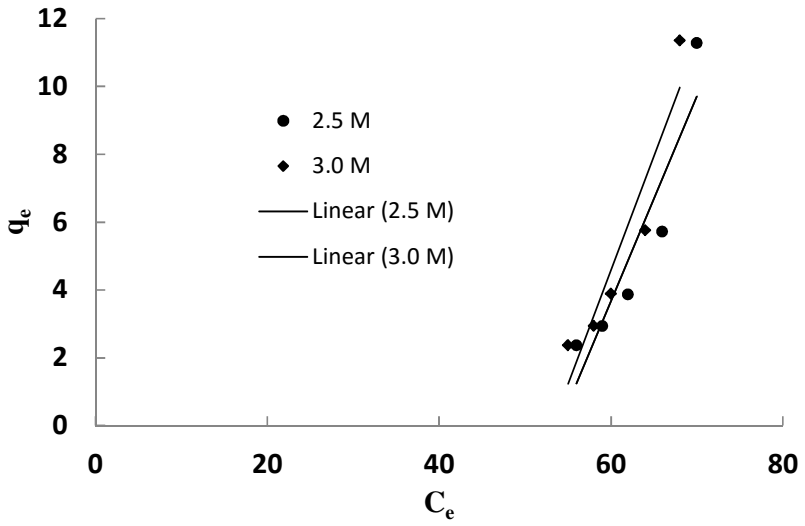

Figure 6 Nerst isotherm for adsorption of methylene blue on to 1C900 and 2C900 at various concentration of activation reagent, Adsorbent dosage, 0.5 g

Table 2 Calculated kinetic parameter for the adsorption of methylene blue on 1C900 and 2C900

Concentration (M)	**2.5**	**3.0**
Pseudo first order kinetics		
k_1 (min^{-1})	0.115	0.115
q_e cal (mg/g)	4.618	4.845
q_e exp (mg/g)	11.28	11.36
r^2	0.817	0.817
Pseudo second order kinetics		
k_2	0.0114	0.0113
q_e cal	12.82	12.82
r^2	0.998	0.998
Intra particle diffusion model		
K_d (mg/g.min)	0.119	0.117
r^2	0.966	0.963

Table 3 Results of various isotherm plots for the adsorption methylene blue on to 1C900 and 2C900

Concentration (M)	**2.5**	**3.0**
Langmuir model		
$q_{max\ (mg/g)}$	0.752	0.805
K_L (L/mg)	72.3	75.0
R_L	0.014	0.013
r^2	0.999	0.992
Freundlich model		
n	1.656	1.490
K_f	3.480	3.551
r^2	0.861	0.884
Nerst model		
k_d	0.604	0.671
r^2	0.861	0.884

4. IODINE NUMBER

Iodine number is a relative indicator of porosity in an activated carbon. Iodine adsorptions are commonly used method to characterize activated carbon performance. The information on pore size and surface area can be obtained from the characteristics of activated carbon. Iodine is a small sized molecule so it indicates the capacity of carbon to adsorb smaller molecules and its adsorption is restricted to micro-pores [33]. The values obtained showed increase in iodine number from 50.76 to 94.53 as temperature increases 700^{o}C to 900^{o}C.

5. CONCLUSION

The results of this study showed that activated carbon can be conveniently obtained from sea snail shell waste for the adsorption of dye in aqueous solution. It was found that adsorption of methylene blue reached maximum uptake of 80.60 % with 0.5 g. The physical properties are comparable to activated carbon obtained from other agricultural wastes. The pseudo second – order kinetic model, fits the adsorption process well with the highest correlation coefficient of 0.998. This shows that chemical adsorption is the rate limiting mechanism. The adsorption of methylene blue is well described as monolayer since the Langmuir model has relatively high correlation coefficient $R^2 > 0.998$. The activated carbon prepared from sea snail shell showed affinity for methylene blue dye adsorption.

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