

Production of Activated Carbon and Characterization from Snail Shell Waste (*Helix pomatia*)

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Abstract

Snail shell waste (*Helix pomatia*) has been evaluated as raw material for the preparation of activated carbon using $ZnCl_2$ and $CaCl_2$ with the temperature ranging from $500^\circ C$ to $800^\circ C$. The activated carbon prepared was characterized, showing effect of temperature on ash content, pore volume and porosity. The adsorption isotherm for methylene blue was carried out on the activated carbon in a batch study. The adsorbent exhibited excellent adsorption for methylene blue. The experimental data were used for both Langmuir and Freundlich models. The adsorption coefficients of Langmuir isotherm were found to be 0.996 and 0.957 for $CaCl_2$ and $ZnCl_2$ while 0.969 and 0.962 were obtained for the Freundlich isotherm respectively. The value of R_L was found to be 0.75 and 0.38 for samples CC and ZZ respectively, which is an indication that activated carbon impregnated with $CaCl_2$ and $ZnCl_2$ is favourable for adsorption of methylene blue under the conditions used in this study.

Keywords

Activated Carbon, Snail Shell Waste, Methylene Blue, Adsorption

1. Introduction

In the last decade, public awareness over the quality of drinking water has attracted interest in the global world [1]. This is reflected in the demand for home water filter, designed to remove objectionable taste, odour and organic contaminants from water, which has experienced substantial growth.

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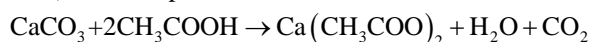
Activated carbon is widely used for the purpose due to the large surface area available for adsorption or chemical reactions [2] as a result of its high degree of micro porosity.

It has a high adsorption capability due to its high internal surface area and porosity formed during carbonization. The presence of activating agents and carbonization conditions influenced the development of pore structures [3]. These materials are characterized by their extraordinary large specific surface areas, well-developed porosity and tunable surface containing functional groups [4]. The surface oxygen functional groups can be easily introduced to the carbon by different activation methods including dry and wet oxidizing agents. Dry oxidation method involves the reaction with hot oxidizing gas such as steam and CO₂ at temperatures above 700°C [5], while that of wet oxidation involves the reaction between the carbon surface and solutions of oxidizing agents such as phosphoric acid (H₃PO₄), nitric acid (HNO₃), hydrogen peroxide H₂O₂, zinc chloride (ZnCl₂), potassium permanganate (KMnO₄), ammonia sulphate (NH₄)₂SO₈, potassium hydroxide (KOH), etc. Its primary source is from organic material with high carbon content (coal, wood, peat, coconut shells, etc). Granular activated carbon is produced by grinding, adding a suitable binder to give it the hardness, re-compacting and crushing to the correct size.

It finds application in areas such as wastewater treatment, water purification, discoloration, and removal of toxic organics, heavy metals ions [6] and recovery of organic and inorganic compounds from gaseous and liquid streams. It is also used in the purification of electroplating solution (bright nickel plating solution) which is a main technique for the removal of organic impurities. Activated carbon is also utilized in both field and industrial processes such as spill cleanup, ground water remediation, gasoline dispersion operation, air purification, drinking water filtration and volatile organic compound capture from painting. Activated carbon is used to treat poisonings and overdoses following oral ingestion. It is thought to bind to poison and prevent its adsorption by the gastrointestinal tract. Activated carbon is 50% w/w combination with celite, chromatographic separation of carbohydrates (mono-, di-trisaccharides) using ethanol solution (5% - 50%) as mobile phase.

There has been an increasing interest in the production of activated carbon from agricultural by-products and industrial waste [7], such as cocoa pod husk [3], periwinkle shell [8], walnut shell, peach stone, physic nut waste, coconut shells, palm kernel shells, and bamboo stem wastes [9] [10]. Others include: olive stone, sugarcane bagasse, pecan shells, palm seed, apple pulp [11]-[14], rubber seeds, and molasses, resins, and dried sewage sludge paper mill sludge, old newspapers [15]-[19], and waste tires [20].

However, the preparation of activated carbon from snail shell waste is scarce in literature. The significant feature of snail shell-based activated carbon is that it is mostly made of calcium carbonate, crystals organized within a matrix of protein in two principal forms: aragonite and calcite. The crystal type in the shell of the land snail (*Helix pomatia*) is normally aragonite, but calcite has also been found in repaired areas of the shell. When calcium carbonate (CaCO₃) dissolves in any common acid, carbon dioxide (CO₂) is generated: the dissolution of CaCO₃ in acetic acid (CH₃COOH) can be represented as follows.



Though the major composition of snail shell is calcium carbonate (97.5%), it is composed of other elements like calcium phosphate, calcium silicate, magnesium carbonate, magnesium oxide, iron oxide, manganese oxide and other organic substances [21]. Snail shell is usually neglected and abundant agricultural waste. As a result of the chemical composition of the shell, it can be used in wastewater treatment either as a coagulant or as an adsorbent. Therefore it is interesting to develop snail shell activated carbon of high porosity. The objective of this work is to prepare study snail shell based activated carbon using ZnCl₂ and CaCl₂. The prepared activated carbon with characterized properties was used for removal of methylene blue for the adsorption isotherm.

2. Experimental and Methods

2.1. Pre-Treatment of Waste Snail Shells

The waste snail shells used in this study was supplied by local market traders (Amassoma). Prior to the carbonization and activation experiments snail shell were washed with warm water, rinsed with distilled water, dried at 105°C for 3 hours and then allowed to cool in desiccators.

2.2. Carbonization/Activation

The dried snail shells were carbonized in a Vecstar Furnace at different temperatures: 500°C, 600°C, 700°C, and

800°C for 3 hours and the charred snail shells were allowed to cool to room temperature. The carbonized snail shells were chemically activated using two different methods.

Method 1

50 grams of the carbonized snail shell in 150 ml of 0.5 M ZnCl₂ solution was thoroughly mixed and heated until it formed slurry. The slurry was transferred to a crucible and heated at 800°C for 3 hours and allowed to cool to room temperature and washed with distilled water, dried in an oven at 110°C for 2 hours. The sample was cooled to room temperature and stored in an air-tight container, ready for use. The same procedure was repeated with 50 grams of the carbonized in 50 ml of 25% concentrated solution of CaCl₂, to soak the carbonized snail shells completely; for 3 minutes to allow absolute escape of gases formed from the exothermic reaction and covered with a lid for 24 hours. The sample was removed and transferred into a drain tray, for 30 minutes, washed repeatedly with distilled water to remove traces of chemical, drained and transferred into an oven at 110°C for 2 two hours, cooled and stored for use. The same was repeated at temperatures of 500°C, 600°C, and 700°C for the both samples.

2.3. Characterization

2.3.1. pH

2.0 grams of the adsorbent (Z₁) was weighed out using a sensitive weighing balance. The weighed activated carbon was washed thoroughly for 5 minutes with 30 ml distilled water and filtered using a filter paper and pH of filtrate was measured using a pH meter. This procedure was repeated for samples Z₂, Z₃, Z₄ and C₁, C₂, C₃, and C₄.

2.3.2. Pore Volume and Porosity

The same quantity sample Z₁ was transferred into a measuring cylinder of (10 ml) and volume of the particles was recorded. This sample was placed into a beaker containing 20 ml of distilled water and boiled for 5 minutes (to displace air in the sample). The content was superficially dried and weighed. The increase in weight of the sample divided by the density of water gave the pore volume for sample Z₁. Porosity was calculated by dividing the pore volume of the particle with the total volume of the particle.

2.3.3. Ash Content

2.0 grams of sample Z₁ was placed into a crucible, and reweighed with its content heated in a furnace at 900°C for 3 hours. The sample was cooled to room temperature and reweighed. Ash content was calculated between the differences in weight.

2.3.4. Determination of Moisture Content

2.0 grams of sample Z₁ was dried using a Moisture Analyzer at 150°C, until weight of sample was constant. The moisture content was determined using the equation:

$$X_0 = \frac{W_1 - W_2}{W_1} \times 100$$

where X₀ = moisture content on wet basis, W₁ = initial weight of sample in grams, W₂ = final weight of sample (in grams) after drying.

2.3.5. Bulk Density

5.0 grams of sample Z₁ was transferred into 25 cm³ of distilled water using measuring cylinder. The volume of the water displaced was recorded. The bulk density was calculated by dividing the mass of the sample Z₁ by the volume of water displaced which was repeated for samples Z₂, Z₃, Z₄, C₁, C₂, C₃, and C₄.

3. Adsorption Isotherm

Varying weights ranging from 1 gram to 5 grams of the activated carbon carbonized activated at 800°C with ZnCl₂ and CaCl₂ were measured separately into 30 ml of the prepared methylene blue solution and mixed thoroughly by a shaker machine for 5 minutes and left for 2 hours. The solution was filtered with a filter paper and

the filtrate was collected for analysis. The equilibrium concentration was determined by using a spectrophotometer. The amount of adsorption at equilibrium, q_e ($\text{mg}\cdot\text{g}^{-1}$), was calculated by

$$q_e = \frac{(C_o - C_e)V}{W} \quad (2)$$

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

4. Results and Discussion

Activated carbon was produced from waste snail shells by impregnating with 0.5 M ZnCl_2 and 2.25 M CaCl_2 . The activated carbon was characterized to determine: pH, moisture content, bulk density, pore volume, porosity, and ash content.

4.1. Physical Properties

Density is particularly important in sugar decolourization where high viscosity syrup is displaced through a column of activated carbon while bulk density is important when carbon is removed by filtration because it determines how many pounds can be contained in a filter of a given cake [22]. Samples Z_2 and C_2 gave the highest bulk density of 2.8 g/ml and 2.0 g/ml at 600°C. Higher density provides greater volume activity and normally indicates better quality activated carbon [23].

When two carbons differing in bulk density are used at the same height per liter, the carbon having higher bulk density will be able to filter more liquor volume before the available cake space is filled. Carbon with an adequate density also helps to improve the filtration rate by forming an even cake. Generally, a carbon with a bulk density of about 0.5 g/ml is adequate for sugar decolourization [24]. The pH of activated carbon may influence colour by changing the sensitive fraction of solution colourants, causing unreliable colour measurements [25]. Acid carbons for example may be a better decolourizer [26] [27], but a sugar refinery would seldom employ a highly acidic carbon because the acid would cause inversion of sucrose to non-crystallizable sugars with low yield [28]. The pH of sample Z_1 is 7.23, while sample C_3 has a pH of 7.36. The result showed that the Activated carbon impregnated with ZnCl_2 and CaCl_2 are alkaline. In sugar decolourization, a distinctively acidic activated carbon may cause inversion of sucrose, and if distinctively alkaline it can cause degradation of organic impurities [28]. Hence, a carbon pH of 6 - 8 is acceptable for most application [29]. Ash content of carbon is the residue that remains when the carbonaceous portion is burnt off. The ash consists of mainly minerals such as silica, aluminum, iron, magnesium and calcium.

Ash in activated carbon is not desirable and is considered an impurity. Ash leached into sugar liquor during the process of decolourization is known to cause uneven distribution of heat in the boiler during sugar crystallization. Ash may also infer with carbon adsorption and catalysis of adverse reactions. Ash in activated carbon is not desirable and is considered an impurity. Also, ash content may affect the pH of the carbon since the pH in most commercial carbon is produced by their inorganic components. Sample Z_4 (800°C) has relatively lower ash content compared to other samples. **Figure 1** shows that increase in carbonization temperature reduces the ash content. It is known that materials with the lowest ash content are most active [30]. The trend is different with ZnCl_2 where the ash content increases with temperature to a peak of 88 and sharply decreased to 25.

The moisture content is the amount of water physically bound to the activated carbon under normal condition. The moisture content of activated carbon dopped with ZnCl_2 and CaCl_2 respectively; samples Z_4 and C_4 have moisture content of less than 1%, while sample Z_3 and C_1 have the highest moisture of 1.75% and 1.70% respectively. Studies have shown that lower moisture content increases the rate of adsorption of contaminants [22]. The practical limit for the level of moisture content allowed in activated carbon varies within 3% - 6% [22]. The moisture content also have the same trend to that of ash content but C_4 has lower moisture content compared to Z_4 at 800°C (see **Figure 2**).

It has been established that the large internal area of activated carbon is attributed to its porosity [31]. Sample Z_4 and C_4 have the highest porosity value in their respective categories. This shows that increase in activation temperature increases the porosity, as indicated in samples Z_1 and Z_2 (0.35, 0.50) and C_1 and C_2 (0.28, 0.38) but

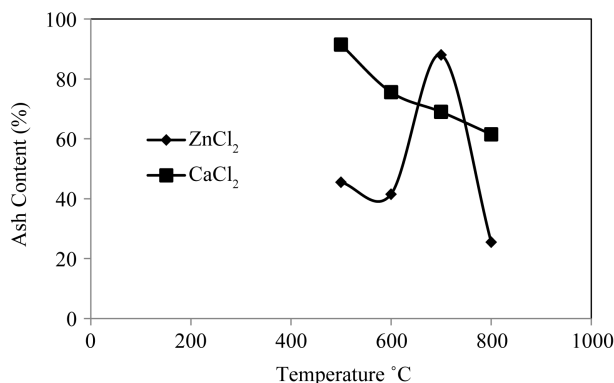


Figure 1. Effect of temperature on ash content doped with ZnCl₂ and CaCl₂ activated carbon.

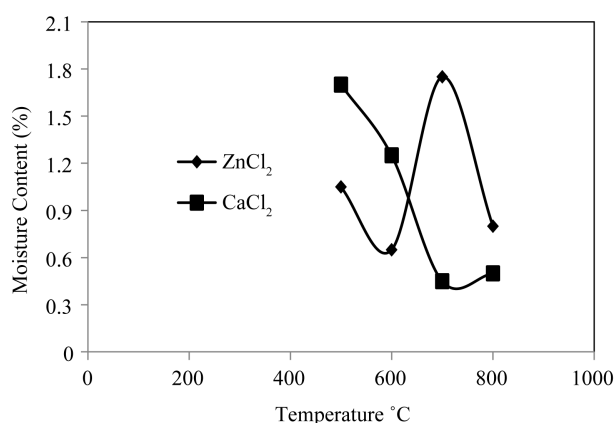


Figure 2. Effect of temperature on moisture content doped with ZnCl₂ and CaCl₂.

samples Z₃ and C₃ seemed to deviate from that pattern as there was a little decrease in the porosity (0.48, 0.29). **Figure 3** shows a steady rise in porosity from 500°C to 600°C, and decreased in porosity at 700°C, then showed an exponential rise to 800°C. The porosity is as a result of the doping agents that helped degradation of organic material to release volatile matter and development of micro porous structure [32]. A similar trend was observed with pore volume impregnated with ZnCl₂, where there was increase as temperature increases and showed a decrease at 700°C. For the C₄ sample, the pore volume increased with an increase in temperature (**Figure 4**).

4.2. Adsorption Isotherm

Adsorption equilibrium is the most significant and helpful data in an adsorption system. It is also helpful in model prediction for analysis and design of an adsorption process. Methylene blue number is correlated with the ability of activated carbon to adsorb colour and high molecular weight substances. Several equilibrium isotherm equations namely, Langmuir, Freundlich, etc. have been evaluated to represent the experimental sorption isotherm data.

Figure 5 shows result of methylene blue adsorption onto snail shell activated carbon conducted in batch studies. The methylene blue adsorption followed the Langmuir isotherm which is consistent with results of others [33].

Langmuir Model

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

$$\frac{C_e}{q_e} = \frac{1}{K_L} + \frac{a_L}{K_L} C_e \quad (3)$$

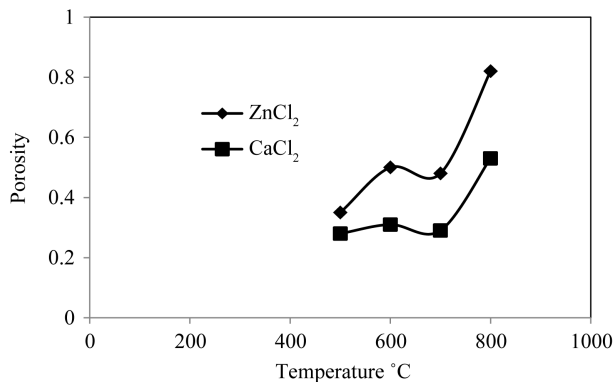


Figure 3. Effect of temperature on porosity doped with ZnCl₂ and CaCl₂.

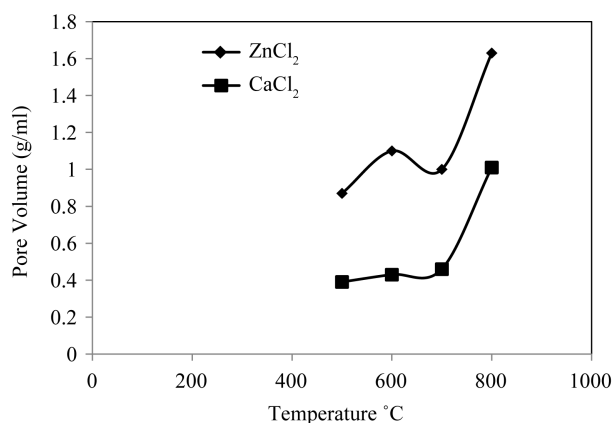


Figure 4. Effect of temperature on pore volume doped with ZnCl₂ and CaCl₂.

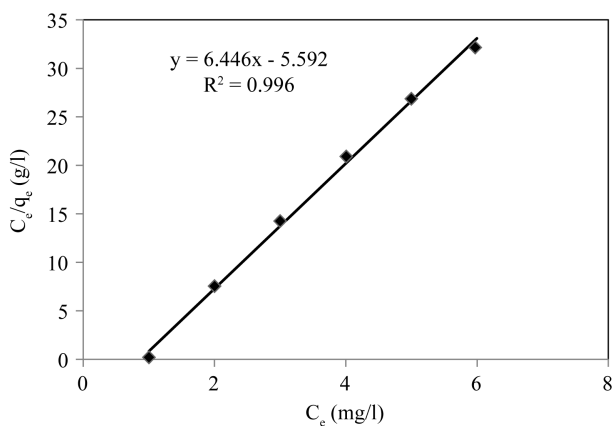


Figure 5. Adsorption isotherm of methylene blue dye on activated carbon doped with calcium chloride.

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), $\frac{1}{K_L}$ is the intercept which is related to monolayer adsorption of adsorbent towards adsorbate, $\frac{a_L}{K_L}$ is the slope obtained when $\frac{C_e}{q_e}$ was plotted against C_e in Figure 5 and

Figure 6 for samples CC and ZZ respectively, k_L and $\frac{a_L}{K_L}$ were calculated from this isotherm (see **Table 1**).

The essential characteristics of the Langmuir isotherm can be expressed in terms of a dimensionless equilibrium parameter (R_L) [34] defined by

$$R_L = \frac{1}{a_L} + K_L \quad (4)$$

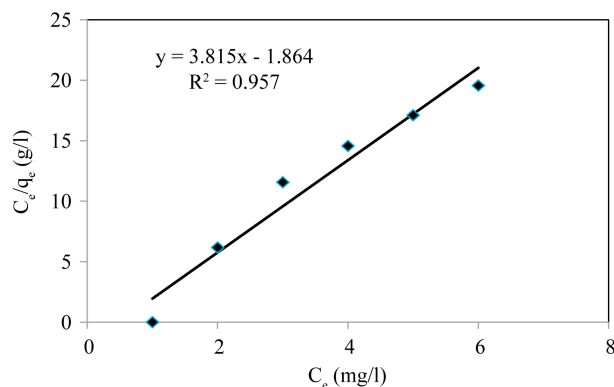


Figure 6. Adsorption isotherm of methylene blue dye on activated carbon doped with $ZnCl_2$.

Table 1. Samples and description.

Sample	Description
A1	Carbonized at 500°C and activated using 0.5 M $ZnCl_2$
A2	Carbonized at 600°C and activated using 0.5 M $ZnCl_2$
A3	Carbonized at 700°C and activated using 0.5 M $ZnCl_2$
A4	Carbonized at 800°C and activated using 0.5 M $ZnCl_2$
C1	Carbonized at 500°C and activated using $CaCl_2$ (25%)
C2	Carbonized at 600°C and activated using $CaCl_2$ (25%)
C3	Carbonized at 700°C and activated using $CaCl_2$ (25%)
C4	Carbonized at 800°C and activated using $CaCl_2$ (25%)
CC1	1 gram of $CaCl_2$ activated carbon
CC2	2 grams of $CaCl_2$ activated carbon
CC3	3 grams of $CaCl_2$ activated carbon
CC4	4 grams of $CaCl_2$ activated carbon
CC5	5 grams of $CaCl_2$ activated carbon
ZZ1	1 gram of $ZnCl_2$ activated carbon
ZZ2	2 grams of $ZnCl_2$ activated carbon
ZZ3	3 grams of $ZnCl_2$ activated carbon
ZZ4	4 grams of $ZnCl_2$ activated carbon
ZZ5	5 grams of $ZnCl_2$ activated carbon

The value of R_L indicates the type of isotherm to be either unfavourable ($R_L > 1$), linear ($R_L = 1$), favourable ($0 < R_L < 1$), or irreversible ($R_L = 0$). The value of R_L was found to be 0.75 and 0.38 for samples CC and ZZ respectively in **Table 2** which is an indication that activated carbon impregnated with $ZnCl_2$ is favourable for adsorption of methylene blue under the conditions used in this study.

4.3. Freundlich Isotherm

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

$$\ln q_e = \ln K_F + \frac{1}{n} C_e \quad (5)$$

where q_e is the amount adsorbed at equilibrium ($mg \cdot g^{-1}$), C_e is the equilibrium concentration of the adsorbate, K_F is the adsorption capacity of the adsorbent ($mg \cdot g^{-1}$) (mg^{-1})^{1/n}, n indicates how favourable the adsorption process is, $\frac{1}{n}$ is the slope, ranging between 0 and 1, a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero [34]. $\ln K_F$ is the intercept. **Table 3** shows the result of n and K_F , which can be defined as the adsorption or distribution coefficient and represents the quantity of methylene blue adsorbed in activated carbon adsorbent of a unit equilibrium concentration.

The plots of $\ln q_e$ versus $\ln C_e$ in **Figure 7** and **Figure 8** give straight lines with slope $1/n$ of 0.364 and 0.320 for samples CC and ZZ respectively. This shows that the methylene blue adsorption conform to the model of Freundlich isotherm. The value of $1/n$ in both samples (CC and ZZ) indicates favourable adsorption [34].

With sample CC, the Langmuir model gives a better fit ($R^2 = 0.996$) than the Freundlich model ($R^2 = 0.969$), while ZZ for Freundlich model fits better ($R^2 = 0.962$) than the Langmuir model ($R^2 = 0.957$).

Table 2. Langmuir isotherm constants for methylene blue on activated carbon.

Sample	$\frac{a_L}{K_L}$	$\frac{1}{K_L}$	a_L	K_L	R_L	R^2
CC	6.446	5.592	1.16	0.18	0.75	0.996
ZZ	0.3815	1.864	2.06	0.54	0.38	0.957

Table 3. Freundlich isotherm constants for methylene blue.

Sample	n	$K_F (mg \cdot g^{-1}) (mg^{-1})^{1/n}$	R^2
CC	2.75	1.48	0.969
ZZ	3.13	1.48	0.962

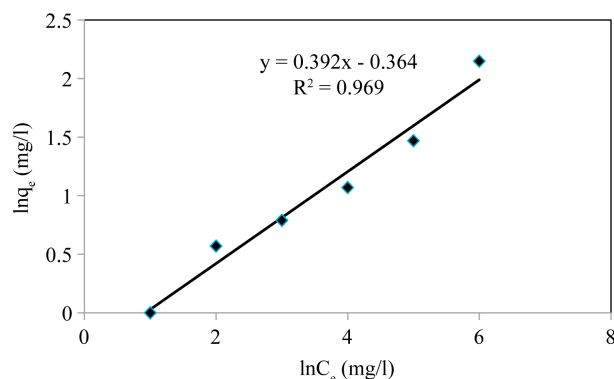


Figure 7. Adsorption isotherm of methylene blue dye on activated carbon impregnated with $CaCl_2$.

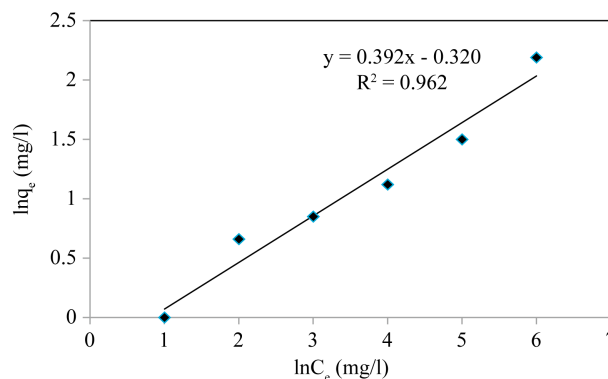


Figure 8. Adsorption isotherm of methylene blue dye on activated carbon impregnated with $ZnCl_2$.

5. Conclusion

The porosity developed in the activated carbon is due to the impregnation of the doping agents ($ZnCl_2$ and $CaCl_2$). This is because the chemical reactions caused by hydrolysis of the doping agents weaken the structure of the carbonized snail shell and release volatile matter which is responsible for the porosity and adsorption capacity of the carbon. Though, analysis indicated better porosity for activated carbon doped with $ZnCl_2$ compared to $CaCl_2$. Both samples CC and ZZ showed favourable adsorption with better fit of $R^2 = 0.996$, 0.957 and 0.969 , 0.962 for Langmuir and Freundlich respectively. Activated carbon from snail shell can be used to build filter systems for treatment of waste water from dye industry.

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Nomenclature

q_e	Amount adsorbe at equilibrium ($\text{mg}\cdot\text{g}^{-1}$)
C_e	Equilibrium concentration of adsorbate (mg/l)
K_F	Quantity of methylene blue adsorbed in activated carbon adsorbent of a unit equilibrium concentration ($\text{mg}\cdot\text{g}^{-1}$) (mg^{-1}) ^{1/n}
n	Adsorption or distribution coefficient