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Research Article

The Adsorption Efficiency of Chemically Prepared Activated Carbon from Cola Nut Shells by ZnCl₂ on Methylene Blue

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The adsorption of methylene blue from aqueous solution onto activated carbon prepared from cola nut shell has been investigated under batch mode. The influence of major parameters governing the efficiency of the process such as, solution pH, sorbent dose, initial concentration, and contact time on the removal process was investigated. The time-dependent experimental studies showed that the adsorption quantity of methylene blue increases with initial concentration and decreasing adsorbent dosage. The equilibrium time of 180 min was observed and maximum adsorption was favoured at pH 3.5. The dye removal using 0.1 g of adsorbent was more than 90%. This dosage (0.1 g) was considered as the optimum dosage to remove methylene blue from aqueous solutions. The equilibrium adsorption data were analyzed by the Freundlich, Langmuir adsorption isotherm models. The kinetics of methylene blue solution was discussed by pseudo-first-order, pseudo-second-order, and Elovich models. The adsorption process follows the Elovich rate kinetic model, having a correlation coefficient in the range between 0.9811 and 1.

1. Introduction

Effluent from textile, dying, cosmetics, food processing, and paper making industries is polluted by dye stuff materials which of course are transported to streams which support life in our mother planet. But, anthropogenic activities have caused a great harm to the quality of our lifeline, that is, water. Because of fast depletion of the freshwater resources, the removal of dye from wastewater, particularly, by activated carbon has been reported widely. However, a survey of literature revealed that methylene blue has been used particularly for adsorption studies, not only because of its environmental concern but also for the fact that it has been recognized as a model adsorbate for the removal of organics as a result of its known strong adsorption to activated carbons [1].

Discharge of these coloured effluents presents a major environmental problem for developing countries because of their toxic and carcinogenic effects on living beings [2]. The presence of methylene blue in water system is unacceptable and even visible at concentrations as low as 1 ppm thereby affecting the aquatic systems. It also inhibits sunlight diffusion into the water, consequently reducing the photosynthetic process of aquatic plants [3]. Hence, the remediation of methylene blue from effluents before discharge into the water environment is essential. There are many methods used for the treatment procedures among which are chemical precipitation, filtration, ion exchange, reverse osmosis, ultrafiltration, electrochemical deposition, and coagulation. These methods differ in their efficiency to remove the dyes and also in the cost. However adsorption remains the outmost process because of its simplicity, high efficiency, easy recovery, and so forth [4]. Many adsorbent have been applied for the removal of methylene blue [5–11].

In 2003, it has been reported that the consumption of activated carbon risen up to about 500300 tones (five hundred thousand three hundred tones) and that 7% increase shall be felt annually [5].

Adsorbent	BET (m ² /g)	Pore volume	Pore size (Å)	Surface aspect
AC	Surface area at p/p° = 0.206156171: 663.6573 m ² /g BET Surface Area: 647.7374 m ² /g	Adsorption total pore volume of pores 0.332061 cm ³ /g	Adsorption average pore width (4V/A by BET): 20.5059 Å	Heterogeneous
	Adsorption cumulative surface area of pores 86.433 m ² /g	Adsorption cumulative volume of pores 0.082139 cm ³ /g	Adsorption average pore diameter (4V/A): 38.013 Å	with micropores and mesopores
	Desorption cumulative surface area of pores 93.3700 m ² /g	Desorption cumulative volume of pores 0.082827 cm ³ /g	Desorption average pore diameter (4V/A): 35.483 Å	

Table 1: Summary of the BET (m^2/g) , pore volume (cm^3/g) , pore size (Å) and surface aspect of the chemically prepared activated carbon by $ZnCl_2$ from cola nut shells.

Since activated carbon is very expensive and efficient, it makes adsorption process more costly. As a result, low-cost and readily available alternative lignocellulosic materials are in need, while methods of production are developed.

Biomass waste including corn cob, coconut shell, palm shell, apple pulp, chickpea husks, grain sorghum, pistachio nut shell, olive stones and walnut shell, cherry stones, wild rose seeds, rice bran, oil palm shell, rubber tree seed coat, cotton stalk, tea waste, sugarcane bagasse, and empty oil palm fruit bunches has been found to be suitable precursors owing to their high-carbon and low-ash contents [12]. Chemical activation of activated carbon has been reported as more advantageous over physical activation due to higher yields, more surface area, and better development of porous structures in carbon [12].

In this research, a local agricultural waste, cola nut shells, was used to produce activated carbon. This is due to the availability and cost-free of the material with high-carbon and low inorganic content. Moreover, little or no work has been reported on the production of activated carbon using cola nut shells.

Several authors have studied the carbonization of lignocellulosic materials [13, 14] and found that the main degradation takes place, between 200–350°C, with evolution of $\rm H_2O$, $\rm CO$, $\rm CO_2$, $\rm CH_4$, aldehydes, and so forth. Distillation of heavier hydrocarbons (tar) takes place in the range 350–500°C, and above 500°C, there is little weight loss, thus indicating that the basic structure of the char has already been formed.

There is a contraction in dimensions of the lignocellulosic precursor during the carbonization process. This change in dimension is important in chemical activations, as the reagent has to be incorporated into the interior of the particles where it inhibits the expected contraction with increasing temperature which implies that the reagent may act as a template for the creation of microporosity [15]. This analysis of the porosity development allows for a comparison of methods of activation of each reagent.

Of the many reagents proposed for chemical activation, (zinc chloride, phosphoric acid, aluminium chloride, magnesium chloride, potassium hydroxide, sodium hydroxide, etc.), the most commonly used, industrially, are ZnCl₂, H₃PO₄, and KOH. However, the effects of ZnCl₂ on activation of carbonaceous materials have been of particular interest. Also the effects of this chemical on a single precursor have not been thoroughly investigated in the literature, and the

information published is limited, but activated carbon with high specific surface area using ZnCl₂ as activating agent has been reported. ZnCl₂ known as Lewis acid is a strong dehydrating agent which could alter the structure of carbon to form the porous structure [16].

The aim of this work was to study the adsorption efficiency of the prepared activated carbon from cola nut shells on the removal of methylene blue dye from aqueous solution.

2. Experimental Methods

2.1. Treatment of Adsorbents

2.1.1. Cola Nut Shells. Cola nut shells were initially washed with deionised water, sun dried, and grained mechanically using a grinding machine (RETSCH) then screened through a sieve (Retsch) to get geometrical size of ≤1.5 mm, which by the use of a beaker was kept into an oven at 110 $^{\circ}$ C for a period of 24 hours, removed, and cooled in a dessicator containing CaCl₂ (drying agent) for 30 min. The lignocellulosic material was removed from the dessicator, and a 1:1 mass reagent ratio was applied, mixed, and dried for 24 hours at 110 $^{\circ}$ C.

2.2. Carbon. Carbonization and activation were accomplished in a single step by carrying out thermal decomposition of the raw material (cola nut shells) impregnated with ${\rm ZnCl_2}$ as activating agent in an inert atmosphere or in the absence of oxygen in a carbolite furnace at 500°C for 1 hour as resident time. After activation, the furnace was allowed to cool to room temperature. The pyrolysed carbons were leached with 1% HCl (v/v) for 2-3 hours and washed several times with distilled water until a neutral pH was achieved. Later the carbon paste was dried in an oven at 110°C for at least 24 hours before application.

2.3. Chemical Reagents. Laboratory grade methylene blue (MB), a cationic dye having molecular formula of $C_{16}H_{18}N_3SCl$ supplied in powder form by Merck, was used without further purification for the preparation of synthetic aqueous solution.

3. Preparation of Solutions

3.1. Methylene Blue Solution. All the experiments were conducted with artificial wastewater stock solution prepared by

$$H_3C$$
 N
 CH_3
 CH_3
 CH_3

FIGURE 1: The structure of methylene blue (MB).

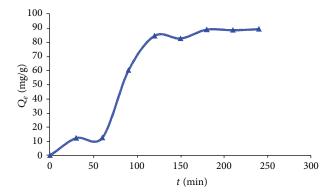


FIGURE 2: The effects of contact time on sorbent-sorbate interaction time.

dissolving analytical grade of methylene blue in distilled water.

The chemical formula of methylene blue is shown in Figure 1 [17].

A stock solution of methylene blue of concentration 500 ppm (500 mg/L) was prepared by dissolving (0.5000 \pm 0.0001)g of methylene blue in a 1000 mL volumetric flask, agitated by using a magnetic stirrer for a period of time and completed with distilled water up to the mark. This solution was again stirred with a magnetic stirrer for two hours to obtain homogeneity. Solutions of various concentrations were obtained by dilution. Solution of hydrochloric acid (0.1 M) and sodium hydroxide (0.1 M) was also prepared for pH adjustments.

3.2. The Batch Adsorption Study. The batch experiments of the adsorption studies were conducted at room temperature (25°C) in a 250 mL screw-cap conical flask. For each run, 0.1–0.5 g of the adsorbent dosage was weighed and placed in the flask containing 30 mL solution of methylene blue of a desired concentration (ranging from 50 to 300 ppm), pH between 2 and 8. The suspension was stirred for interval of time between 10–240 min, using a magnetic agitator and stirrer at a controllable speed. After agitation, the suspensions were filtered using Whatman no. 1 filter paper. The concentration of the filtrate was determined by using a UV-visible spectrophotometer. The absorbance was measured at the maximum absorption wavelength of 668 nm.

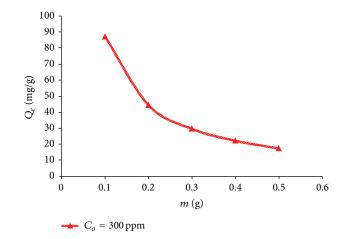


Figure 3: The effect of carbon dose for the uptake of methylene blue.

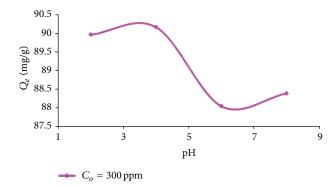


FIGURE 4: The effect of pH on methylene blue adsorption by activated carbon prepared from cola nut shells by chemical activation with ZnCl₂.

The quantity adsorbed by a unit mass of an adsorbent at equilibrium (Q_e) and the adsorption percentage (%R) at an instant were calculated using the relations (1), respectively,

$$Q_e = \frac{C_o - C_t}{m} V,$$

$$\% R = \frac{C_o - C_t}{C_o} 100,$$
(1)

where C_e is the concentration of adsorbate at equilibrium; C_t is the concentration of adsorbate at time t; C_o is the initial concentration; m is the mass of the adsorbent; and V is the volume of the adsorbate.

4. Results and Discussion

4.1. The Characteristics of Activated Carbon Obtained from Cola Nut Shells. Table 1 summarizes the characteristics of activated carbon prepared from cola nut shells.

It can be seen that the nature of the prepared activated carbon consisting mainly of mesopores which are very favourable for adsorption of large molecules like methylene

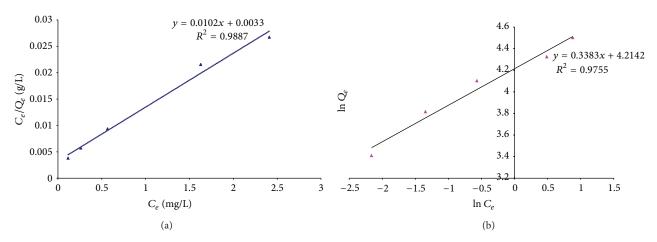


FIGURE 5: The linear plots, (a) from Langmuir adsorption model and (b) from Freundlich adsorption model.

blue with an average recommended, significant surface area between $500-1500 \,\mathrm{m}^2/\mathrm{g}$, reported by Chand and Bansal [18] in his book, that widely used activated carbon adsorbents has specific surface area on the order 500 to $1500 \,\mathrm{m}^2/\mathrm{g}$ and pore volume on the order of 0.20 to $0.60 \,\mathrm{cm}^3/\mathrm{g}$.

4.2. Adsorption Contact Time Test. Methylene blue adsorption on activated carbon prepared from cola nut shells increases with increasing agitation time as shown in Figure 2.

The removal rate was slow within the first 50 min then increases rapidly and gradually diminished to attain equilibrium within 180 min beyond which there was no significant increase in the removal rate. It is thought that the first step leads to surface adsorption and the second step leads to intraparticle transport from bulk fluid to the external surface of the porous adsorbent [19]. This trend agrees with the report of other investigators [20]. The stages of sorption of methylene blue on activated carbon might be controlled by the diffusion process from bulk to the surface or the heterogeneous nature of the adsorbent.

4.3. Effect of Carbon Dose. In order to study the effect of adsorbent dosage on the adsorption of methylene blue, a series of adsorption experiments were carried out with different adsorption dosages varying from 0.1 to 0.5 g at initial concentration of 100 ppm.

The effect of carbon dose for the uptake of methylene blue by activated carbon from cola nut shells was found to decrease by increasing the adsorbent dose. This may be allocated to the fact that as the amount in grams of adsorbent is increased the total surface area available for the adsorption of methylene blue reduces as a result of overlapping or aggregation of adsorption sites [21]. Furthermore, maximum quantity adsorbed, $Q_e=87.37~{\rm mg/g}$, was reached by 0.1 g activated carbon prepared from agricultural waste (cola nut shells, see Figure 3).

4.4. Effect of pH Experiments. To study the effect of pH on methylene blue (MB) adsorption, 0.1 g of activated carbon

was added to solutions containing 300 ppm of methylene blue ions. The initial pH values were adjusted from 2–8 using 0.1 M HCl and 0.1 M NaOH. After the suspensions were shaken for 180 min, equilibrium time, at room temperature, they were filtered using Whattman no. 1 filter paper and analyzed for residual methylene blue concentration.

Figure 4 shows higher adsorption of methylene blue at low pH = 3.5 which is due to increased protonation by neutralization of the negative charges at the surface of adsorbent. But for pH < 3.5, the number of negatively charged adsorbent sites decreased and the number of positively charged surface sites increased, which did not favour the adsorption of positively charged dye cation due to electrostatic repulsion. In addition, lower adsorption of methylene blue at acidic pH might be due to the presence of excess H⁺ ions competing with dye cations for the available adsorption sites. For pH values higher than 7, the amount of dye removal started to increase, and in this case, may be in alkaline solutions, methylene blue might have stepwise, demethylated to other common dyes [7]. This indicated that the adsorption capacity of activated carbon prepared from cola nut shells was pH dependent.

5. Adsorption Isotherms and Kinetic Models

5.1. Adsorption Isotherm Models. An adsorption isotherm is the relationship between the adsorbate in the liquid phase and the adsorbate adsorbed on the surface of the adsorbent at equilibrium at constant temperature. In order to successfully represent the dynamic adsorptive behavior of any substances from the fluid to the solid phase, it is important to have a satisfactory description of the equilibrium state between the two phases composing the adsorption system. The Langmuir and Freundlich isotherms are the two most well-known isotherms which have been used to describe the equilibrium of adsorption systems.

5.2. The Langmuir Adsorption Isotherm. The Langmuir isotherm takes an assumption that adsorption occurs at

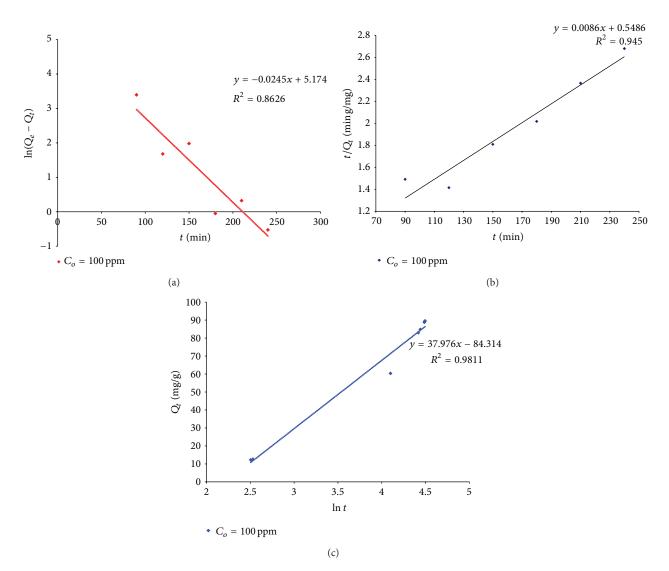


FIGURE 6: The linear plots, (a) pseudo-first-order and (b) pseudo-second-order, (c) Elovich kinetic models for the adsorption of methylene blue onto activated carbon prepared from kola nut shells.

specific homogeneous sites within the adsorbent. The equation is as follows:

$$Q_e = \frac{Q_m K C_e}{1 + K C_e},\tag{2}$$

where Q_e (mg of adsorbate per g of adsorbent) is the adsorption density at the equilibrium solute concentration C_e . C_e is the equilibrium concentration of adsorbate in solution (mg/L). Q_m (mg of solute adsorbed per g of adsorbent) is the maximum adsorption capacity corresponding to complete monolayer coverage.

K is the Langmuir constant related to energy of the adsorption (*L* of adsorbate per mg of adsorbent).

The previous equation can be rearranged to the following linear form:

$$\frac{C_e}{Q_e} = \frac{1}{Q_m K} + \frac{C_e}{Q_m}. (3)$$

The linear form can be used for linearization of experimental data by plotting C_e/Q_e against C_e . The Langmuir constants Q_m and K can be evaluated from the slope and intercept of the linear equation:

$$Q_e = \frac{Q_m K C_e}{1 + K C_e},\tag{4}$$

where Q_e (mg of adsorbate per g of adsorbent) is the adsorption density at the equilibrium solute concentration C_e . C_e is the equilibrium concentration of adsorbate in solution (mg/L).

 Q_m (mg of solute adsorbed per g of adsorbent) is the maximum adsorption capacity corresponding to complete monolayer coverage.

K is the Langmuir constant related to energy of the adsorption (*L* of adsorbate per mg of adsorbent).

	Equation	R^2	Q_e	Q_m	K_L	K_F	1/n
Langmuir	y = 0.0102x + 0.0033	0.9887	89.22	87.12	3.48	/	/
Freundlich	v = 0.3383x + 4.2142	0.9755	/	/	/	67.64	0.358

Table 2: Parameter values of the isotherms adsorption of methylene blue onto activated carbon from cola nut shells.

TABLE 3: Parameter values of the kinetic studies of the adsorption of methylene blue onto activated carbon from cola nut shells.

	Equation	R^2	K_1	K_2	α	β
Pseudo-first-order	y = -0.0245x + 5.174	0.8626	0.0232	/	/	/
Pseudo-second-order	y = 0.0086x + 0.5486	0.945	/	0.0198	/	/
Elovich	y = 37.976x - 84.314	0.9811	/	/	4.44	0.0257

The previous equation can be rearranged to the following linear form:

$$\frac{C_e}{Q_e} = \frac{1}{Q_m K} + \frac{C_e}{Q_m}. (5)$$

The linear form can be used for linearization of experimental data by plotting C_e/Q_e against C_e . The Langmuir constants Q_m and K can be evaluated from the slope and intercept of the linear equation.

5.3. The Freundlich adsorption Isotherm. The Freundlich isotherm is an empirical equation employed to describe the heterogeneous system (16). The equation is given below:

$$Q_e = K_f C_e^{1/n}, (6)$$

where Q_e is the quantity of solute adsorbed at equilibrium (adsorption density: mg of adsorbate per g of adsorbent). C_e is the concentration of adsorbate at equilibrium, where K_f and 1/n are empirical constants dependent on the nature of sorbent and sorbate and the temperature. Both of these values (K_f and 1/n) are important in selecting an adsorbent as a separating medium, in which K_f (mg/g) is the over-all adsorption capacity (Q_e at $C_e = 1$ mg/L or $\log C_e = 0$) and 1/n is the heterogeneity factor. The heterogeneity factor 1/n indicates the strength of bond energy between sorbate and sorbent.

This equation is conveniently used in linear form by taking the logarithmic of both sides as

$$\ln Q_e = \ln K_f + \frac{1}{n} \ln C_e. \tag{7}$$

A plot of $\ln Q_e$ against $\ln C_e$ yielding a straight line indicates the confirmation of the Freundlich isotherm for adsorption. The constants can be determined from the slope and the intercept.

From Figure 5, the adsorption isotherm of methylene blue shows that the data both fitted Langmuir and Freundlich isotherms but fitted better in Langmuir isotherm equation by comparing the R^2 values of Langmuir and Freundlich isotherm as shown in Table 2.

This implies that a monolayer sorption proceeds over a surface containing a finite number of adsorption sites and uniform strategies of adsorption with no transmigration of adsorbate in the plane of surface. In the literature, several authors reported the difficulties of comparing adsorption intensities, due to lack of consistencies in data. 1/n, which is a measure of surface heterogeneity, is also a measure of adsorption intensities. Adsorption on the surface becomes more heterogeneous as 1/n values get closer to zero. From Table 1, 1/n < 1 shows normal adsorption [22].

5.4. Kinetic Adsorption Models. In the present research, the kinetics of adsorption of methylene blue on the activated carbon prepared from cola nut shells by chemical modification by ZnCl₂ was studied. Three types of kinetic models, pseudo-first-order (Lagergren equation), pseudo-second-order model (Ho equation), and Elovich model [6], were employed to fit experimental data (Figure 6).

The straight lines plots showed a good agreement of experimental data with pseudo-second-order model and Elovich model. Table 3 listed the results of rate constant studies for different kinetic models. Comparing the \mathbb{R}^2 values, pseudo-second-order and Elovich models show a significant agreement with adsorption mechanisms being the two, with successfulness of the Elovich model. The general explanations for this form of kinetic equation involve a variation of the energetic of chemisorption with the heterogeneous active sites in activated carbon. This supports that the heterogeneous sorption mechanism is likely to be responsible for the methylene blue uptake. Elovich model basically supports chemisorption [23].

6. Conclusion

This study confirmed that the activated carbon prepared from a low-cost agricultural waste (cola nut shells) could effectively remove methylene blue from an aqueous solution with the optimal pH for favourable adsorption of methylene blue equal to 3.5. The adsorbent dosage had an effect on the adsorption process of methylene blue. The adsorption equilibrium was reached in 180 min with the isothermal data fitted well in Langmuir model and Freundlich model indicating a chemisorption adsorption. Also the adsorption process followed Elovich rate kinetic model.

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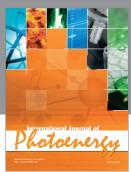
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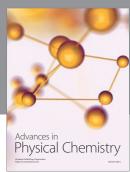
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