

Comparative Analysis of Adsorption of Methylene Blue Dye Using Carbon from Palmkernel Shell Activated by Different Activating Agents

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Abstract— Activated carbon was produced from palm-kernel shell using NaOH and KOH solutions as the activating agents. The same was tested to determine its adsorptive capacity and efficiency using methylene blue solution. Particle sizes of the produced carbon were in the range of 600 – 2000 μm . The adsorption process was carried out at two different temperatures using methylene blue as adsorbate. It was observed that adsorption of methylene blue dye using carbon activated impregnated with KOH gave better result than with NaOH. Based on the adsorption parameters obtained, the process was found to be better described by the Freundlich adsorption isotherm.

Keywords— Activated carbon; palm-kernel shell; adsorptive capacity; efficiency; activating agents

I. INTRODUCTION

Activated carbon is a carbonaceous amorphous material which is synthesized from specific raw materials and active reagents. Its adsorptive property is due to the presence of micro porous structures which are responsible for its good adsorption capacity, high surface reactivity and specific surface area [12], [8]. Various adsorbents exist but the evolvment and preference of activated carbon over other adsorbents is due to its low cost and wide range of applications such as water purification, decaffeination, decolourization of liquids, sewage treatment and gas purification. Activated carbons are of two types. The first is the granular class characterized by an average particle size in the range of 1.0 to 5.0 mm [5]. Due to their particle sizes, they are preferable for gas adsorption. They are also used for water treatment, deodorization and separation of components of flow systems [13].

Powdered activated carbon being the second class, are of smaller particle size relative to the granular activated carbon. The particle size is smaller than 200 mesh (74 μm). They appear as fines and are not commonly used in water treatment plants because as a large amount of waste water is passed through the filter bed, some of the particles pass along thus contaminating the water to be treated. The production of activated carbon is either a two-step process, i.e. carbonization followed by physical activation or a single-step carbonization and chemical activation. Carbonization process helps to

increase the carbon content of the precursor by thermal means which occurs in the absence or little amount of oxygen in order to avoid complete combustion whereas, in chemical activation, the carbonized material is impregnated with a certain ratio of chemicals called activating agents which modifies and improves the pore structure of the charcoal as well as its adsorptive properties.

Materials such as wood, peat, lignite and coal can be used in producing activated carbon because of their high carbon contents and due to their inherent surface functional groups which help to improve the surface chemistry of the carbon thereby enhancing its adsorptive capacity. Materials with very high lignin content have macro pores e.g. grape and jatropa seeds. Nut shells and woods are of highly cellulosic with less amounts of lignin. They are micro porous which makes them suitable for use in the production of activated carbon. The selection of solid wastes as precursors for activated carbon depends on their potentials for obtaining high quality activated carbon, the presence of minimum inorganic substances, raw materials storage life, volume and cost of raw materials [3]. The total amount of inorganic constituents associated with activated carbon is usually in the range of 0.3-0.6 % of the total composition and vary from one carbon source to another depending on the source of raw materials and activating agents added during the production process. As a byproduct of carbon synthesis is ash which if not controlled, reduces the overall activity of activated carbon as well as the efficiency of regeneration.

Recently, exploration of the use of biomass as a source of carbon for the production of activated carbon has become a great feat. These materials are wastes from domestic places, industries and farmlands which include coconut shells, palm kernel shells, sawdust and rice husks. Biomass residue from palm oil industries are also a good source fuels/renewable energy. With an increase in the number of oil palm industries, these biomass resources are also on the increase, as is evident in countries like Malaysia, Indonesia and Thailand. Palm kernel shells are the residues left after extraction of the kernels from the shells of the palm fruits. As an agricultural waste, palm kernel shell has been reported to have same calorific characteristics as coconut shell. Due to its high density, high carbon and low ash content, palm kernel shell is used as a

precursor for the manufacture of activated carbon [11]. Activated carbon can be produced from carbon-rich low-cost agricultural by-products such as wood, shells of fruits and other raw materials [10]. In addition, because of their availability in large quantities, agricultural by-products are used to prepare activated carbon. Furthermore, the disposal of these waste materials constitutes nuisance to the environment and poses serious health risks to the inhabitants. Also, water from breweries and textile industries may contain heavy metals like copper, zinc and dyes which are also harmful/poisonous and if consumed could lead to increased mortality.

Dyes are carcinogenic and highly toxic, thereby posing health hazards and environmental problems to living organisms [1]. Dyes in water give out a bad colour and can cause diseases like haemorrhage and severe skin irritation. They prevent the process of photosynthesis in plants by preventing sunlight from penetrating the surface of dye contaminated water in contact with any green plant [6]. Urine contains compounds such as urea, chloride, sodium, potassium and creatinine which add up to 5 % with the remaining 95 % being water. The water in urine can easily be extracted and purified by using activated carbon which also removes the odour, colour and taste of urine, thus making it fit for drinking. The accumulation of biomass around drainage systems could result to stagnancy of water, a breeding place for mosquitoes which are carriers of plasmodium and precursors for malaria. Various techniques that have been used to produce clean water include sedimentation, reverse osmosis and absorption [15] but due to the high maintenance/equipment costs and the routine involved in their operations, adsorption using activated carbon is widely preferred.

Activated carbon is used as an adsorbent in water systems due to its high porosity and large surface area. The most commonly used activated carbon is that produced from coconut shell using Phosphoric acid as the activating agent. Micro-pores have been reported as being majorly responsible for adsorption by adsorbents because they possess molecular dimensions hence, a solute molecule will readily penetrate into a pore having a particular diameter [14]. According to [8], a carbonation process entails the pyrolysis of the precursor material at a temperature of 600 – 1200 °C in an inert atmosphere consisting of nitrogen or argon. It is usually carried out in a rotary kiln; the basic microstructure of the char with micro-porosity is formed around 5000C. Hence, the reason for the choice of temperatures in this research i.e. 600-800oc. However, they confirmed the discontinued use of ZnCl₂ because of its bad environmental impact. Activation time and temperature effects were studied for the carbonization process and properties of activated carbon. It was found that the activation times normally used were from 1 hour to 3 hours for palm kernel and coconut shells but as time and temperature increased, the percentage yield decreased gradually and the BET surface area also increased [17].

In order to expand the supply of activated carbon, other low cost agricultural waste such as palm kernel shell products have been explored and are found suitable for the production of activated carbon. In addition, not much work is made

available on the production of activated carbon from palm kernel shells using Sodium hydroxide relative to Potassium hydroxide. However, this paper compares activated carbon formed by actions of NaOH and KOH on a desired raw material (palm kernel shell) for the adsorption of a textile dye. The adsorptive capacity of the activated carbon produced was determined by varying the particle size. The effect of particle size on adsorption rate of the activated carbon was also studied. Adsorption is a process that occurs when a gas or liquid solute attaches or cling onto the surface of a solid usually known as the adsorbent, forming a molecular film known as the adsorbate [7]. It is a surface phenomenon leading to the attachment of one substance to another. The efficiency of different activating agents such as NaOH and KOH on different particle sizes of carbon has been evaluated. This is aimed at encouraging waste-to-wealth management in prospective countries, improving their nations' economies by providing job opportunities and avoiding the cost of maintenance of process equipment used in wastewater treatment.

Particle size of activated carbon may or may not have negligible influence on the total surface area available for adsorption depending on the number of micro-pores present, which may not change largely with the particle size. The only difference may then be in the contact time between the larger and smaller particle sizes for adsorption to occur due to effects of diffusion. For smaller activated carbon particles, equilibrium is more easily achieved because of their ability to reduce internal diffusion and mass transfer limitation of the adsorbate on the surface of the adsorbent. Smaller carbon particles have a greater surface area to volume ratio making them more accessible to diffusion or adsorption. Smaller sized particles of a given mass of carbon have a greater surface area to volume ratio than larger particles of same mass. This paper looks into the effect of particle size on the adsorption capacity of the produced adsorbent by keeping the adsorption time constant.

II. METHODOLOGY

The sourced raw material and production processes are discussed here.

A. Materials Used

Chemicals used include hydrochloric acid of 1 M concentration, sodium hydroxide pellets, 40 g, 36 g potassium hydroxide and 10 g of methylene blue powder and distilled water.

B. Apparatus Used

Whatmann's 2V filter paper- 18cm diameter, litmus paper, automatic sieve shaker, furnace (Carbolite), alsin crucibles, 70 g & 270 g, Oven, UV- Spectrophotometer, weighing scale, 250mL beakers, Erlenmeyer flasks, 250ml, Büchner funnel, petri dishes

C Procedure

The palm kernel shells were collected and cleaned to remove impurities. They were then dried in the oven at 105 0C for one hour. The shells were crushed with a mortar and further ground with an electric grinder. The ground particles

were then screened using Matest-Treviolo automatic sieve shaker (Italy) to obtain samples of four different particle sizes in the range of 600 μm – 2 mm. Each sample was then introduced into the furnace and allowed to char for 1 hour at a temperature of 600 $^{\circ}\text{C}$ to enrich the carbon content and to initiate pore formation in the samples. The charred product was allowed to cool down to room temperature. NaOH and KOH were separately introduced to the samples as activating agents i.e. to each sample weighing 20 g, 30 cm³ of 1.0 M solution of the activating agents were mixed in separate beakers. The contents of the beakers were thoroughly mixed and allowed to soak for 24 hours before being transferred into crucibles which were transferred into an oven at varying temperatures of 600 $^{\circ}\text{C}$ to 800 $^{\circ}\text{C}$ at 20 minutes time interval. The product was allowed to cool to room temperature and washed with distilled water using a Buchner funnel. The samples were further dried in the oven at a temperature of 105 $^{\circ}\text{C}$ for 2 hours. After drying, the samples were kept in air-tight containers for characterization.

B. Determination of Physico-Chemical Properties

1) Moisture Content

For analysis of the moisture content of the carbon sample, ASTM D2867-70 was used. 1.0 g of activated carbon sample was put into a crucible and weighed. The crucible was placed in an oven at 150 $^{\circ}\text{C}$ for 3 hours. It was allowed to cool in a desiccator to ambient temperature and then weighed. The percentage moisture content was calculated from (1).

$$M_c = 100 * [(B - F) / B - G] \quad (1)$$

Where:

M_c = moisture content of activated carbon; B = mass of crucible with lid plus original sample in grams (g); F = mass of crucible with lid plus dried sample in grams (g); G = mass of crucible with lid in grams (g)

2) Ash Content

Here, the ASTM D2866-70 was used: 1.0 g of dried carbon was weighed, placed in a crucible and transferred into the furnace at 650 $^{\circ}\text{C}$ for 3 hours. The crucibles were allowed to cool and then put in a desiccator for further cooling to ambient temperature for 1 hour. The dried carbon was weighed and the ash content was calculated from (2).

$$A_c = 100 * [(F - G) / B - G] \quad (2)$$

Where:

A_c = ash content of activated carbon; G = mass of empty crucible plus dried sample in grams (g); F = mass of crucible plus ashed sample in grams (g); A_c = mass of crucible plus dried sample in grams (g); C = Methylene Blue Adsorption

Methylene blue is a basic organic dye with a molecular formula of $\text{C}_{16}\text{H}_{18}\text{N}_3\text{ClS}$ and a molecular weight of 319.85 g/gmol. Powdered Methylene blue was used as the adsorbate. 1 g/L stock solution of methylene blue dye was prepared by dissolving 1.0 g of dye powder in 1.0 L of distilled water. Adsorption tests were conducted by weighing 2.0 g of activated carbon and adding it to 25 mL of 1000 mg/L methylene blue solution. The mixture was shaken for 24 hours, it was then filtered and the residual concentration of methylene blue was measured at a wavelength of 665 nm using an UV/Vis Spectrophotometer (Genesys 10S) hence, the final concentration of methylene blue was determined

3) Calibration Curve

Calibration curve was used to determine the final concentration of dye present in the solution. Different concentrations of methylene blue solution were prepared in mg/l. Absorbance of the methylene blue solution was measured at a wavelength of 665 nm; various masses (0.1g, 0.5g, 1.0g, 1.5g and 2.0g) of activated carbon were mixed with aqueous solution of methylene blue at 800 $^{\circ}\text{C}$ with average particle size of 600 μm . The mixture was thoroughly agitated with a mechanical shaker to improve the efficiency of adsorption. The adsorption capacity for each of the masses was calculated while keeping all other conditions constant.

4) Carbon Dosage

0.1 g, 0.5 g, 1.0 g, 1.5 g and 2.0 g carbon particles of 600 μm size were each mixed with some quantity of aqueous methylene blue solution at 800 $^{\circ}\text{C}$. The mixture was agitated using a mechanical shaker in order to improve their adsorption efficiencies. The adsorption capacity of each of the separate mixtures was then determined with other conditions constant.

III. RESULTS AND DISCUSSION

Table 1 shows the recorded moisture and ash contents of the activated carbon produced.

Table 1: Physico-chemical properties of palm kernel shell activated carbon

Moisture Content (g/g)	Ash Content
10.0	3.3

In order to ensure some degree of accuracy, an average of two runs were performed. Absorbance data was recorded using the UV/Vis Spectrophotometer (Genesys 10) at a wavelength of 665 nm. Using data in Table 2, a calibration graph was obtained by plotting the absorbance of known concentrations of methylene blue solution.

In Table 2, it could be seen that the absorbance of the methylene blue increased with increased concentrations of methylene blue solution i.e. the lowest absorbance, 0.689 was obtained at 0.5 mg/L and the highest absorbance value, 4.893 was recorded at the highest concentration 500 mg/L of methylene blue – which agrees with the Beer-Lambert’s law. Fig.1 below is the absorbance curve for methylene blue solution.

Table 2: Concentration and absorbance of methylene blue

Concentration (mg/L)	Absorbance
0.5	0.689
1	0.735
3	0.897
5	1.034
10	1.859
30	2.456
50	2.968
120	3.488
200	3.877
375	4.500
500	4.893

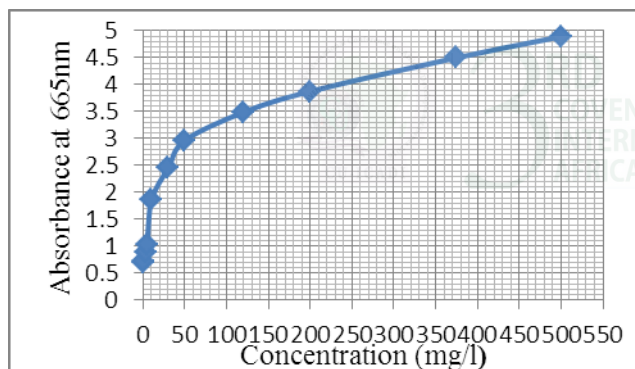


Fig.1: Absorbance of methylene blue at different concentrations

From the absorption curve in Fig.1, as observed from the absorbance spectrum of methylene blue solution, at the specified light wavelength of 665 nm for which the absorbance of methylene blue solution was observed on the spectrophotometer, the absorbance of methylene blue solution increased at high concentrations of the solution.

A. Effect of Temperature and Particle size

From literature, since the activity of KOH begins at about 700°C, temperatures below and above this value were chosen for this investigation in order to determine its effect when used as an impregnating agent between the upper and lower limits i.e. below and above the critical temperature. Temperature

variation with adsorption of methylene blue by different activating agents was investigated. The results are as shown in Tables 3 and 4 for the effect of temperature on the rate of adsorption using NaOH and KOH as activating agents for the produced carbon respectively. The amount of methylene blue adsorbed was calculated using (3).

$$Q_s = \frac{C_o - C_s}{m} \tag{3}$$

Table 3: Effect of Temperature on adsorption with NaOH as activating agent

Table 3, shows the adsorption capacity of activated carbon for different particle sizes in the range of 600 - 2000 µm at two different temperatures (i.e. 600 and 800°C) with recorded final concentrations of the solution in the adsorbent. At 600 °C, for a particle size of 600 µm, the final concentration of the solution was found to be lowest with an absorbance of 1.0. Other absorbance values recorded were 1.154, 1.219 and 1.239 for particle sizes of 850, 1000 and 2000 µm. At a higher temperature, say 800 °C, lower size particles, recorded higher concentrations of adsorbed methylene; this was also in accordance with the decrease in final concentrations of the solution i.e. 4, 6, 6.5 and 8.0 mg/L which correspond to adsorbed methylene concentrations of 498, 497, 496.8 and 495 respectively. However, it can be seen that an increase in temperature (i.e. by about 200 °C improved the actual adsorption power of the activated carbon.

B. Effect of Temperature on Adsorption

Table 4: Effect of temperature on adsorption with KOH as activating agent

Temperature (°C)	Particle size (µm)	Absorbance	Final Concentration (mg/L)	Methylene Blue adsorbed (mg/L)
600	600	1.000	5.000	497.5
	850	1.154	6.500	496.8
	1000	1.219	7.000	496.5
	2000	1.239	9.000	495.5
800	600	0.956	4.000	498.0
	850	1.163	6.000	497.0
	1000	1.217	6.500	496.8
	2000	1.227	8.000	496.0

Temperature (°C)	Particle Size (µm)	Absorbance	Final Concentration (mg/L)	Methylene Blue adsorbed (mg/L)
600	600	0.937	5.000	497.5
	850	1.149	6.000	497.0
	1000	1.197	6.500	496.8
	2000	1.212	7.000	496.5
800	600	0.889	2.000	499.0
	850	1.153	6.100	497.0
	1000	1.195	6.300	496.9
	2000	1.209	6.500	496.8

In Table 3, with NaOH as activating agent and for particle size in the range of 600 – 2000 µm, the absorbance values at 600 °C varied from 1 – 1.239 for particle sizes, and varied between 0.956 – 1.227 for 600 – 2000 at 800 °C while in table 3.4, the adsorbed methylene blue concentrations were between 497.5 – 496.5 mg/L at 600 °C, and 499 – 496.8 mg/L, at 800 °C, for the same particle sizes considered. By comparing the adsorbed methylene blue values in Tables 3 and 4, it is clear that KOH is a better activating agent than NaOH as the concentration of adsorbed methylene blue was lower with NaOH than with KOH as activating agent. This can be drawn from the fact that Potassium is higher up than sodium in the electrochemical series of metals. The results in Tables 3 and 4 are in agreement with the observations of other researchers. However, considering the concentrations of adsorbed methylene blue in Tables 3 and 4, it could be seen that there is difference in corresponding values of adsorbed methylene blue hence, the higher the absorbance of the activated carbon, the lower the concentration of methylene blue adsorbed and vice versa.

The adsorption capacity of the activated carbon was calculated from (4) using data in Table 6.

$$q_e = \left[\frac{(C_o - C_e)}{W} \right] * V \quad (4)$$

In Table 5, with NaOH as activating agent, the adsorption capacity of the activated carbon increased for smaller particles than for larger particles at 600 °C i.e. considering the particle sizes between 600 and 2000 micrometer, it can be deduced that for particles of size 600 µm, the adsorption capacity (i.e. 12.438 mg/g), is higher than 12.419 mg/g, 12.413, and 12.388 mg/g for particles of size 850 µm, 1000 µm and 2000 µm respectively. These values show that the adsorption capacity for particles of size 850 µm is higher than the adsorption capacity for 1000 µm and vis-à-vis. At 800 °C, the adsorption capacities obtained were 12.450 mg/g, 12.425 mg/g, 12.419

mg/g and 12.400 mg/g for particles of size 600 µm, 850 µm, 1000 µm and 2000 µm respectively. This is due to the increased surface area to volume ratio of the smaller particles relative to the larger ones. Comparing the absorption capacities for the particles at 600 and 800 °C, it can be deduced that increase in temperature improved the adsorption capacities of the particles. The variation of adsorption capacity versus particle size at different temperatures with NaOH as activating agent.

Table 5: Effect of particle size on adsorption with NaOH as activating agent

Temperature (°C)	Particle Size (µm)	Final Concentration (mg/L)	Adsorption Capacity, qe(mg/g)
600	600	5.000	12.438
	850	6.500	12.419
	1000	7.000	12.413
	2000	9.000	12.388
800	600	4.000	12.450
	850	6.000	12.425
	1000	6.500	12.419
	2000	8.000	12.400

C. Effect of Particle Size on Adsorption

In Table 6, at a 600 °C, with KOH as activating agent, the adsorption capacities recorded for the carbon particles, showed that for particle sizes of 600, 850, 1000 and 2000 µm, the corresponding adsorption capacity (12.438 mg/g) for a particle size of 600 µm is higher than the adsorption capacity (12.425 mg/g) for 850 µm particles, and higher for particles of size 850 µm than the adsorption capacity (i.e. 12.419 mg/g) for particles of size 1000 µm and higher for 1000 µm than for 2000 µm particles with adsorption capacity value of 12.413 mg/g). The same trend can be seen in the calculated adsorption capacities i.e. 12.475, 12.426, 12.421 and 12.419 mg/g for particle sizes of 600, 850, 1000 and 2000 µm respectively. By comparing adsorption capacities of the particles at different temperatures (i.e. 600 and 800 °C), the value increased from: 12.435 to 12.475 mg/g, 12.425 to 12.426 mg/g, 12.419 to 12.421 mg/g and 12.413 to 12.419 mg/g for particles of size 600, 850, 1000 and 2000 µm respectively. Furthermore, the adsorption capacity values at 600 µm were the highest as compared with other adsorption values for particles of other sizes. Variations in adsorption capacity of carbon activated with KOH versus particle size at different temperatures.

Table 6: Effect of particle size on adsorption with KOH as activating agent

Temperature (°C)	Particle Size (µm)	Final Concentration (mg/L)	Adsorption Capacity, q_e (mg/g)
600	600	5.000	12.438
	850	6.000	12.425
	1000	6.500	12.419
	2000	7.000	12.413
800	600	2.000	12.475
	850	6.100	12.426
	1000	6.300	12.421
	2000	6.500	12.419

CONCLUSION

Palm kernel shell is a good raw material from which an effective adsorbent can be made for the removal of methylene blue dye in aqueous solution. The use of KOH as activating agent generated more pore sites which resulted in increased capacity and hence gave a better performance than NaOH. Also, the final/residual concentration of the methylene blue solution decreased with increased carbon dosage. In preparing activated carbon for adsorption purposes, it is important to take into consideration, the effect of particle size as smaller particles have better performance than larger sized particles which is also a measure of their efficiency of adsorption. Thus, the lower adsorption efficiencies were peculiar to larger particles because of the fewer adsorption sites formed as a result of the surface area available for adsorption which in turn results in a higher residual concentration of methylene blue solution. The maximum adsorption capacity Q_m for monolayer was calculated to be 9.3371 mg/g, at the higher of the two temperatures i.e. at 800 °C with a particle size of 600 µm using KOH as activating agent. Furthermore, based on the calculated values of $1/n$, the Freundlich isotherm gave a better description of the adsorption process than the Langmuir's which gave a value of $1/n > 1$.

NOMENCLATURE

Symbol	Designation
X	mass of solute adsorbed per unit mass of adsorbent (mg/g)
X_m	maximum monolayer adsorption capacity mg/g

D. Efficiency of Activated carbon

E. Adsorption Isotherm

- b adsorption constant L/mg
- C_e equilibrium concentration of solute after adsorption mg/L
- q_e solute adsorbed per unit mass of adsorbent mg/g
- K adsorption constant
- n intensity of adsorption
- M_c moisture content of activated carbon
- B mass of crucible with lid and dry sample (g)
- F mass of crucible + lid + dry sample g
- G mass of crucible with lid and dry sample (g)
- A_c ash content
- Q_s quantity of methylene blue adsorbed per liter (l^{-1})
- C_o initial concentration of methylene blue (mg/l)
- C_s final concentration of methylene blue (mg/l)
- M mass of ash content used (g)

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