ADSORPTION OF METHYLENE BLUE BY USING ACTIVATED CARBON FROM EMPTY FRUIT BUNCH AND PALM PRESS FIBER

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A thesis submitted in fulfilment
of the requirements for the award of the Degree of
Bachelor of Chemical Engineering

Faculty of Chemical & Natural Resources Engineering
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JANUARY 2012

STUDENT'S DECLARATION

I declare that this thesis entitled "Adsorption of Methylene Blue by Using Activated Carbon from Empty Fruit Bunch and Palm Press Fiber" is the result of my own research except as cited in references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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Dedicated to my wonderful supervisor, my lovely parents, my supportive siblings,
my cheerful friends, and all faculty members
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ABSTRACT

Empty Fruit Bunch (EFB) and Palm Press Fiber (PPF) are abundance of agricultural wastes. These fiber are basically incinerated and used in mulching purposes. Adsorption of methylene blue by using both of fiber is one way to increase the uses of them. Methylene blue is one of dyestuff that harmful to human and aquatic life. Effect of contact time, agitation speed and dosage of activated carbon is investigated. Activation the activated carbon using impregnated of KOH solution is used. Same ratio of fiber which is (1:1) (wt %) is prepared. The higher the contact time, the higher the amount of adsorption capacity obtained. At certain time there got the lower number of adsorption capacity because the contact time taken is not sufficient since the process of adsorption taking a long time to achieve the equilibrium. In term of agitation speed, 170 rpm of speed gave the highest adsorption capacity compared to 200 rpm and 700 rpm. According to Langmuir isotherm model, it is showed that 170 rpm gave 5.025 mg/g of adsorption capacity. It is because the Van der Waals forces are weak to bind the molecules. So, the molecules that already attracted to the molecules surface are desorbed back if the agitation speed too high. For dosage impact, the adsorption capacity decreased sharply with the increasing of adsorbent dosage because of the overlapping of adsorption site that cause the overcrowding of adsorbent particles.

ABSTRAK

Buah Tandan Kosong (BTK) dan Serat Sabut Kelapa Sawit (SKS) ialah sejenis sisa daripada pertanian. Kedua-dua serat ni secara umumnya dibakar dan digunakan untuk penjagaan tumbuhan. Penyerapan metilena biru menggunakan kedua-dua serat ini adalah satu cara untuk meningkatkan penggunaan kedua-dua serat ni. Metilena biru ialah salah satu pewarna yang membahayakan manusia dan kehidupan air. Kesan perhubungan masa, kelajuan pengacauan dan jumlah karbon teraktif telah dikaji. Proses pengaktifan karbon teraktif menggunakan larutan KOH digunakan. Nisbah serat yang digunakan adalah sama iaitu (1:1) (wt %). Berdasarkan keputusan, semakin tinggi masa diambil, semakin tinggi jumlah kapasiti penyerapan. Pada sesetengah masa, terdapat penurunan jumlah kapasiti penyerapan kerana masa yang diambil tidak mencukupi sedangkan proses penyerapan memerlukan masa yang panjang untuk mencapai keseimbangan. Dari segi kelajuan pengacauan, kelajuan 170 rpm memberikan kapasiti penyerapan yang tertinggi berbanding 200 rpm dan 700 rpm. Berdasarkan model Langmuir isotherm, ia menunjukkan bahawa 170 rpm memberikan 5.025 mg/g jumlah kapasiti penyerapan. Hal ini kerana daya Van der Waals yang lemah untuk menyatukan molekul-molekul. Maka, molekul-molekul yang sudah tertarik ke molekul-molekul di permukaan akan didesorbsi kembali apabila kelajuan yang digunakan terlalu tinggi. Bagi keputusan jumlah karbon teraktif, kapasiti penyerapan berkurang dengan pertambahan jumlah karbon teraktif kerana pertindihan kawasan penyerapan yang menyebabkan kesesakan zarah-zarah adsorben.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

This study is about production of activated carbon (AC) from empty fruit bunch (EFB) fiber. In order to modify the AC, oil palm press fiber is also added with the EFB fiber. The AC produced then used in adsorption of methylene blue (MB) aqueous solution.

AC is carbon that constitute of many applications, one of which is used as an adsorbent for purification of water, air and other chemical products. This is possible due to highly porous nature of the solid. Furthermore, it consists of extremely large surface area to volume ratio. Surface area of AC is contained in micropores and mesopores. AC also known as high carbon but low inorganic contents material such as wood, lignite and coal (Lua and Guo, 2001).

One of the purposes of AC is works to remove contaminants from water which is called physical adsorption. AC comprised of carbon highly porous nature as said earlier. It provides a large surface area for contaminants or adsorbates to collect.

In other words, physical adsorption occurs because of all molecules utilizes attractive forces particularly the molecules at the surface of a solid or the pore walls of carbon, and these kind of surface molecules seek the other molecules to fasten to. The large internal surface area of carbon provided many attractive forces that work to attract the other molecules (Robert and Anthony). Therefore, contaminants in water are

adsorbed to the carbon's surface by surface attractive forces equivalence to gravitational force (Robert and Anthony).

Adsorption is a process by which AC removes substances from the water. In simple term, adsorption is "the collection of a substance onto the surface of adsorbent solids". It is such one of removal process whereas the particles are bound to an adsorbent particle surface by either chemical or physical attraction. Additionally, adsorption is often confused with absorption.

Many researchers had reported that activated carbon produced by EFB mostly could absorb a lot of pollutants like methylene blue. Therefore, in this research the produced activated carbon is used to examine the potential application of activated carbon from EFB and palm-pressed fiber produced on removal of methylene blue which is known as pollutants.

1.2 Problem Statement

Methylene Blue (MB) is one of dyestuffs which harmful to human beings and hazardous to aquatic organisms. In fact, dye contamination in wastewater can lead to environmental problems. Other than that, AC is the most widely good adsorbent in removal of colour from textiles due to its effectiveness plus high adsorption capacity. However, AC uses is still limited because of high operating cost. EFB and PPF are categorized as the agricultural wastes that mostly incinerated in palm oil mill and used as fertilizer for the farmer used in their farms. Even nowadays a lot of research used EFB and PPF as raw materials, but the production of EFB and PPF are increasing each year. So, the number of unused EFB and PPF are also increasing.

1.3 Research Objectives

The aim of this research is to produce activated carbon from low cost agricultural waste which is EFB and palm-pressed fiber (PPF) in order to treat wastewater.

i) To evaluate the effect of MB initial concentration by varying the speed, contact time and amount of AC

ii) To study about the effectiveness of AC from EFB and oil palm press fiber in adsorption of MB with Langmuir isotherm models.

1.4 Scope of Study

In this research, the ratio of raw material used is equal which is (1:1) (wt %). The production of activated carbon is carried out by using chemical activation method. In chemical activation, thermal decomposition of the raw material impregnated with activating agents like Potassium Hydroxide (KOH) is carried out to accomplish carbonization and activation process. KOH is a chemical that classified as strong base when react in the water solution.

After the impregnation step, the AC sample is carbonized in the furnace by using nitrogen gas (N_2) by fixed the carbonization temperature and time. The carbonization temperature of this activation process is fixed to 240 ^oC and two (2) hours are taken in this research. After carbonization process, the AC produced are washed with HCl and neutralized with distilled water several time until reached the pH 6 – 7.

This research also focuses on the effect of initial concentration of methylene blue in changes of agitation speed with various time and amount of AC used. Then, the adsorption performance is identified by using Langmuir isotherm model.

1.5 Rationale and Significance of Study

In 1990, the world production of AC was estimated to be 375,000 tonnes, excluding at Eastern Europe and China (Mozammel et.al, 2002). Then, in 2002, the activated carbon production reached 200,000 tonnes per year in United States. The AC demands were increased over the year and market growth was estimated at 4.6% per year (Mozammel et.al, 2002).

AC is primarily acted as an adsorbent to remove and decrease the pollutant from wastewater. Nowadays, a lot of industrial companies are built up over the year. Thus, demands of AC become higher over the year too. It is because the environmental issues especially in term of water and air purification are increasing constantly as the development of the countries.

As noted earlier, when the demand of AC increase over the year, then the price of AC production also increase. It is because of the supplier of raw materials become decrease. Thus, new developments of AC by using the lower cost of raw material such as agricultural wastes are developed. EFB and PPF are listed among these low cost wastes.

Applications of AC in order to treat wastewater are expanded especially in pharmaceuticals companies, vegetable oils industries, paper and pulp industries and miscellaneous sector.

CHAPTER 2

LITERATURE REVIEW

2.1 Empty Fruit Bunch (EFB) and Palm Press Fiber (PPF)

EFB is one type of abundance agricultural waste which can be collected in palm oil mill. It got after the fruits are processed to be crude oil. Before the previous researchers found out the uses of EFB, this kind of lignocellulosic waste was only combusted in the incinerator. Other than that, farmers buy EFB from the mill to mulch or use the fiber as fertilizer to protect their plant especially oil palm trees.

Nowadays, the researchers found out the advantages of EFB uses. EFB could be used as raw material in producing paper and pulp. Other than that, with additional of polymer, EFB could be produced the composites. It is because of the suitability of EFB's constituents which shown in Table 2.1. Compound of EFB is mostly consists of lignocellulosic which contains both lignin and cellulose. Other than agricultural residues, sources of lignocellulosics are includes wood, water plants or in other term, it categorized as biomass (Rowell, 1992).

`PPF is one kind of agricultural wastes as similar as EFB. It also can be find at palm oil mills and also categorized as lignocellulosic materials. PPF can be obtained after the fruits are pressed by the machine to get its crude oil. Palm-pressed fiber represents approximately 15 percent of the palm fresh fruit bunch processed (Lau et al, 2006). In 2001, 9.21 million metric tons of palm-pressed fibers were produced by Malaysia palm oil mills.

Figure 2.1: Empty Fruit Bunch

Table 2.1: Chemical Constituents in EFB

Constituent	**% of dry EFB**	**Reference**	
		Khoo & Lee 1991	Law & Jiang 2001
Extractives	3.7 ± 0.3	0.9	2.8
Acid-insoluble lignin	18.8 ± 0.3	17.2	17.6
Ash-free-acid-insoluble lignin	17.8 ± 0.2	-	-
Ash	1.3 ± 0.2	0.7	
Hot-water soluble	7.5 ± 0.8	2.8	
1% NaOH soluble	14.5 ± 2.7	17.2	
Holocellulose	82.4 ± 1.4	70.0	
Cellulose	62.9 ± 2.0	42.7	
Hemicellulose	28.0	32.5 (Leh, 2002)	
Arabinose	2.5 ± 1.1	-	
Xylose	33.1 ± 2.6	-	
Mannose	1.3 ± 0.01	-	
Galactose	1.0 ± 0.0	-	
Glucose	66.4 ± 3.7	-	
		Singh et al.,	1999
Silica (EDAX)	1.8 (atomic)	-	
Copper	0.8 ± 0.7 g/g	23 mg/l	
Calcium	2.8 ± 0.1 g/g	0.25% (CaO)	
Manganese	7.4 ± 0.4 g/g	48 mg/l	
Iron	10.0 g/g	473 mg/l	
Sodium	11.0 ± 0.4 g/g	-	

Source: Law et al. (2007)

2.2 Main Components in Fiber

2.2.1 Lignin

Lignin is formed by removal of water from sugars. It creates aromatic structures. Other than that, these reactions are not reversible. Additionally, there are many possible monomers of lignin, and the types and proportions depend on the source in nature.

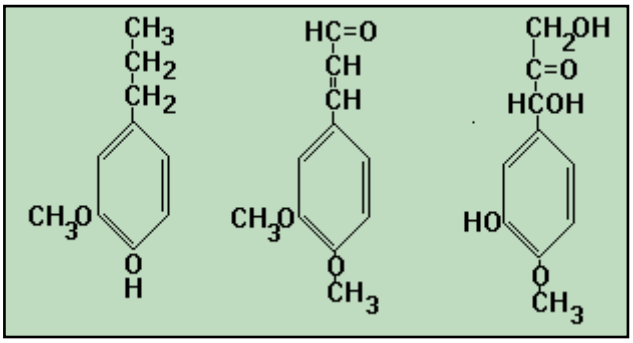

Figure 2.2: Monomers of Lignin

Source: www.rpi.edu , 1996

Lignin is usually found in all vascular plants, mostly between the cells, but also within the cells, and also in the cell walls. It makes vegetables firm and crunchy. Other than that, it functions to regulate the transport of liquid in the living plant or partly by reinforcing cell walls and keeping them from collapsing, partly by regulating the flow of liquid, and it enables trees to grow taller and compete for sunshine (McCardy, 1991).

Because lignin is the strongest component of the plant cell wall, then, the higher the proportion of lignin the lower the bioavailability of the substrate (Richard, 1996). The effect of lignin on the bioavailability of other cell wall components is thought to be largely a physical restriction, with lignin molecules reducing the surface area available to enzymatic penetration and activity (Haug, 1993).

It is resistant to degradation in nature, as being bond together with strong chemical bonds. Plus, it also has a lot of internal H bonds. It is bonded in complex ways to hemicelluloses or carbohydrate in fiber (McCardy, 1991). Even though lignin contains certain carboxylic acids, but it is not categorized as acid. Wood give out acids as it degrades, as similar as paper and board that contains lignin (McCardy, 1991).

2.2.2 Cellulose

Cellulose's empirical formula is $(C_6H_{10}O_5)n$ where n is show as degree of polymerization. It has high molecular weight and linear polymer of repeating beta-D-glucopyranose units (Paperonweb).

Structure of cellulose is categorized as a long chain of linked sugar molecules that gives the remarkable strength of woods. In fact, it is the main component of plant cell walls, and the basic building block for many textiles and for paper (Senese, 2010). For example, cotton is the purest natural form of cellulose.

Additionally, cellulose is classified as a natural polymer, a long chain made by the linking of smaller molecules. The links in the cellulose chain are a type of sugar: ß-D-glucose. In other term, cellulose also called as a polysaccharide that produced by linking additional sugars in the same way exactly. Other than that, the length of the chain varies from a few hundred sugar units in wood pulp to over 6000 for cotton (Senese, 2010).

Cellulose is the major component in the rigid cell walls in plants. It is a linear polysaccharide polymer with a lot of glucose monosaccharide units (Ophart, 2003). Humans are unable to digest cellulose. It is because the appropriate enzymes to breakdown the beta acetal linkages are deficient (Ophart, 2003).

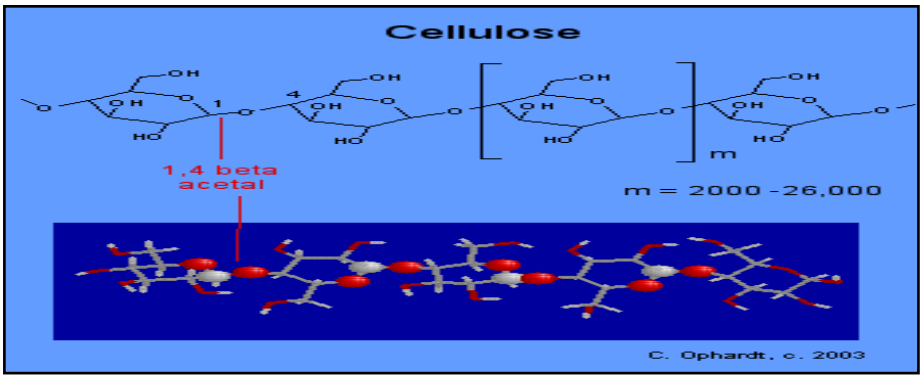

Figure 2.3: Cellulose Structure

Source:C. Ophart, (2003)

2.3 Characteristics of Adsorbent

An adsorbent is a substance, usually porous in nature and comprised with a high surface area that desire to adsorb substances onto its surface by intermolecular forces. Only at very low concentrations is the adsorption isotherm linear, at higher concentrations the adsorption isotherm may be Langmuir or Freundlich in nature (Scott, 2008).

In fact, solutes can be distributed between adsorbent surface and a mobile phase. Thus, the adsorbents are used as a stationary phase in liquid-solid and gas-solid chromatography (Scott, 2008). In fact, adsorbents also could be used in extraction process in order to remove traces of organic materials from large volume of water.

Typical adsorbents that used in gas-solid chromatography are consist of silica gel, alumina, carbon and bonded phases. These are majority used in the separation of the permanent gases and the low molecular weight of hydrocarbon gases (Scott, 2008). While adsorbents used in liquid solid chromatography are mostly silica gel and various types of bonded phases (Scott, 2008). These adsorbents have a very wide of application areas.

2.4 Adsorption Process

Adsorption is a process that is influenced by a few factors which comprised of adsorbent, adsorbate and solvent properties. A solid has the porous structure. Its energetic heterogeneity and surface chemical properties are the main factor that influences adsorption equilibria (Marczweski, 2002).

The activated carbon's surface contains a lot of active chemical groups, part of which is dissociable ones (Marczweski, 2002). In term of adsorbate and solvent, the adsorption process depends on the differences between their chemical properties, structure and interactions in which mutual and with solid surface (Marczweski, 2002).

Basically, adsorption is a process where a solid or activated carbon is used for removing a soluble substance from the water. These types of process usually apply in ground water purification, the de-chlorination of process water, water purification for swimming pools and the polishing of treated effluent.

The process of adsorption described as the molecules from the liquid phase or gas phase will attached in a physical way to a surface. In this case, the surface is from the activated carbon (Lenntech, 2011).

The adsorption uptake is influenced by the concentration of substance in the water, temperature and the polarity of the substance. A polar substance is cannot be removed by activated carbon, while a non-polar substance can be removed totally by the activated carbon (Lenntech, 2011).

Adsorption and absorption is different. Adsorption happens when a substance is attached to the internal surface of activated carbon. Absorption happens when gas is taken in a solution or in a different medium.

2.5 Activated Carbon (AC)

AC is known as a solid, porous, black charcoal and tasteless. AC also defined as a porous carbon material, usually chars, which reacted with gases during or after carbonization in order to increase porosity (Marsh, 1989). Norlia Baharun (1999) defined AC is an organic material that consist of essentially graphitic structure.

The common main features of AC is; graphite like planes which show various degrees of disorientation and the resulting spaces between these planes which constitute porosity, and the built of condensed aromatic rings are referred as Basic Structure Units (BSU) (Benaddi, 2000).

Nowadays, activated carbon is produced using the agricultural wastes which are known as cheaper sources. Earlier, the sources of activated carbon are from wood. Since wood supplies decreasing every year, the researchers found that coconut shell as example is suitable to use as activated carbon.

Production of AC from agricultural wastes is not only because of the prices or the easiness in supplying, but those fibers have a lot of advantages that suitable to use in adsorption process.

Activated carbon's properties are split into two categories which are physical and chemical properties. In term of physical properties, it is more important in the industrial park. AC's physical properties involve its porosity. AC has a very porous surface. Its surface is described as being sponge-like, immersed with microscopic holes and its surface is like sand paper which only viewable with a microscope (theactivatedcarbon).

Ash content and moisture content in AC also can affect the uses of AC. Both of these physical properties are important in order to examine the effectiveness of AC in adsorption. The specific surface area of AC and surface chemistry are classified as chemical properties.

Activated carbon attracted dissimilar bonds within its structure in order to bind to surfaces. It used Van der Waals force to bind to other surfaces. However, it only binds to certain chemicals, such as ammonia, inorganic compounds, alcohols and iodine (theactivatedcarbon).

For many purposes, moisture content does not affect the adsorptive power. However, it obviously dilutes the carbon (Nurul ‘Ain, 2007). Ash content of carbon means the remains residue when the carbonaceous materials are burned off (Nurul ‘Ain, 2007). Ash content leads to increase hydrophilicity and can have catalytic effects, which is causing the restructuring process (Nurul ‘Ain, 2007). Yang (2003) stated that inorganic material contained in AC which is measured as ash content, generally in the range between 2 to 10%.

The specific surface area of the adsorbent affects the adsorption performance. The larger the specific surface area of AC, the better its performance in adsorption process (Guo and Lua, 2003). The optimum specific surface area of AC is between 600 – 1200 m^2/g (Ng et.al, 2002). The adsorption capacity of adsorbent is influenced by its internal surface area and pore volume (Nurul ‘Ain, 2007).

Additionally, the effectiveness of AC is depended on their surface chemistry, as well as their pore size distribution (Radovic, 2001). The surface chemistry is influenced by the chemical composition of the raw material. In the other hand, various surface functional groups contain oxygen, nitrogen and other heteroatoms which have been identified on AC (Nurul ‘Ain, 2007). The heteroatoms bind to the surfaces. Assumed that the character of the functional groups typically found in aromatic compounds. Other than that, it reacts in similar ways with many reagents (Nurul ‘Ain, 2007).

There a various methods to determine surface functional groups and attempts have been study the surface groups by spectroscopic methods, which is by infrared(IR) as the example (Nurul ‘Ain, 2007).

Marsh (1989) found that the word pore came from Greek word, which means a passage. A pore is connected to the external surface of a solid and will allow the passage of fluids through the material (Nurul ‘Ain, 2007). The capacity for molecule in different shapes and sizes are gives different pore sizes. IUPAC is classified porosity into three different group of pore sizes, which are; micropores which have width less than 2nm, mesopores that have width between 2 to 50 nm and macropores in which has width greater than 50 nm (Guo and Lua, 2003).

The pore shapes in AC varied from slit-shaped cracks to spheroidal bubbles (Nurul ‘Ain, 2007). The pore shapes affects on the some properties of carbon and graphite, as example mechanical strength and kinetics reaction.

Micropores are formed in the interlayer which spacing in the range 0.34 to 0.8 nm (Nurul ‘Ain, 2007). Micropores in AC have the greatest influences on the gas adsorption process, while mesopores and macropores are influence in transport of fluid (Nurul ‘Ain, 2007). In adsorption of gas phase, mostly micropores carbon is works while mesopores carbon is applied in liquid phase adsorption processes (Benaddi et.al, 2000).

Mesopores also important as the main transport arteries for this adsorbate (Hu et. al, 2001). The mesopores volume states between 0.1 to 0.5 cm^3 per gram and mesopores surface area are in the range between 20 to 100 m^2 per gram (Hu et.al, 2001).

AC known as amorphous carbon, which show a very disordered microcrystalline structure, whereas graphitic microcrystals are randomly oriented (Gomez-Serrano et.al, 2005). Microstructure also constrictions in the micropores area that control in access much of the micropores spaces (McEnaney, 2002). Other than that, entrances to micropes could be blocked by functional groups that attached to the edges of layer planes, and also by carbon deposits formed by thermal cracking of volatiles released during carbonization (Nurul ‘Ain, 2007).

AC manufactured by the pyrolysis of carbonaceous materials of agricultural materials, such as wood, coal, peat, fruit stones, and shell or synthetic polymer followed by activation of the chars obtained from them (Manocha,2003). The pyrolysis process which is without air involved, finally produced a solid porous carbon. The carbon can be activated by physical or chemical activation.

There are three factors that affecting AC production which are raw material, temperature and activation time. In term of raw material, most organic materials rich in carbon can be used as raw material in manufacturing of AC (Rodriguez-Reinoso, 2002). The factors are taken into consideration are divided into seven. The factors are consist of; high carbon content, low in inorganic content, high density and sufficient volatile content, the stability of supply in the countries, potential extent of activation, the inexpensive material and low degradation upon storage (Nurul ‘Ain, 2007).

In fact, temperature also important in carbonization process. It affects the characteristics of AC produces. The temperature used as low as 200 ^{o}C (Haimour and Emeish, 2005) and high to 1100 ^{o}C (San Miguel et.al, 2003). The optimum temperatures between 400 oC to 500 oC have been reported by the earlier researchers irrespective of the time of activation and impregnation ratio for different raw material (Srinivasakannan and Zailani, 2003). When activation temperature increases, it will reduce the yield of AC respectively. The decreasing in yield paralleled by the increasing activation temperature regarding to the activation reaction (Nurul ‘Ain, 2007). These cases are also manifested in the decreasing volatile content and increases fixed carbon for increasing activation temperature (Nurul ‘Ain, 2007). Percentage of volatile matter decreased when the carbonization temperature increased and the maximum parameter is between 200 ^{o}C to 800 ^{o}C due to rapid carbonization occurring in the region (Haimour and Emeish, 2005).

Activation time also one of factors that important in manufacturing AC. Activation time affects the carbonization process and changes the properties of AC. As the time increased, then, the percentage of yield gradually decreased and the BET

surface area also increased (Nurul ‘Ain, 2007). The extent of decrease in product yield is observed to be reduce when excessive activation occurs (Kim et.al, 2001).

2.6 Langmuir Isotherm Models.

Langmuir isotherm model is to describe the relationship of adsorbate-adsorbent systems. The Langmuir isotherm describes chemisorptions processes. The history of Langmuir isotherm stated that Irving Langmuir was awarded the Nobel Prize in 1932 for his investigation about surface chemistry and Langmuir's isotherm describes the adsorption of adsorbate onto the surface of the adsorbent (Adamson, 1979). There are three assumptions of this isotherm (Adamson, 1979), in which:\

- The surface of the adsorbent is in contact with a solution that contains adsorbate that strongly attracted to the surface.
- The surface has a specific number of sites where the solute molecules could be adsorbed.
- The adsorption involves the attachment of only one molecules' layer to the surface that called as monolayer adsorption.

The equation of Langmuir isotherm model is shown as follows:

$$\frac{C_e}{q_e} = \frac{C_e}{Q} + \frac{1}{bQ} \quad (1)$$

Where Ce is equilibrium concentration (mg/L), qe is the amount of adsorbate adsorbed per mass of adsorbent (mg/g), b is the equilibrium constant related to the sorption energy between the adsorbate and adsorbent (dm3/mg) and Q is limiting amount of adsorbate that can be taken up per mass of adsorbent. Langmuir isotherm model is helpful in predicting the favourability of adsorption system, which is based on the following dimensionless factor

$$R_L = 1/(1 + bC_0) \quad (2)$$

Where C_0 is the highest dye concentration (mg/L). The value of R_L is tabulated in figure 2.2 below.

Table 2.2: Type of isotherm and R_L value

Value of R_L	**Type of Isotherm**
$0<R_L<1$	Favorable
$R_L>1$	Unfavorable
$R_L=1$	Linear
$R_L=0$	Irreversible

Source: J.Thirumal and S.Kaliappan (2011)

When Ce/qe is plotted against Ce got straight line with slope 1/Q is obtained, indicating that the adsorption of methylene blue on activated carbon follows the Langmuir isotherm (J.Thirumal and S.Kaliappan, 2011).

CHAPTER 3

METHODOLOGY

3.1 Apparatus

3.1.1 Furnace

Basically, furnace is used to carbonize the fibers to become char. Furthermore, this equipment is used in activation step. The brand of this furnace is Nabertherm which is manufactured in Malaysia. This kind of furnace can adjust starting time before carbonization process happen.

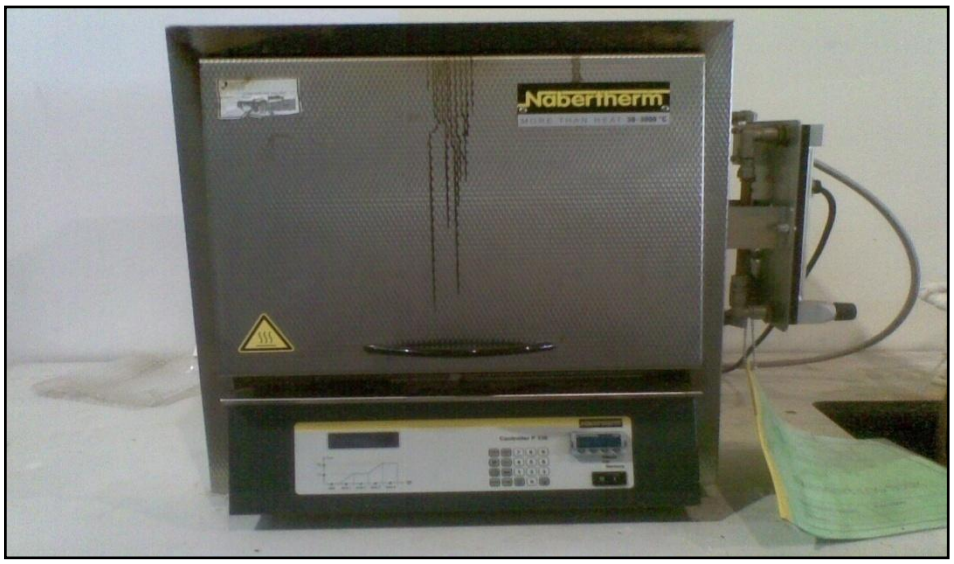

Figure 3.1.1: Nabertherm Furnace

3.1.2 UV – Visible Spectrophotometer

UV – Visible Spectrophotometer or UV – Vis Spectrophotometer is used to measure the adsorption uptake of methylene blue solution by determining absorption capacity from this equipment. Then, the concentration of the solution also can be calculated. The wavelength of methylene blue used is 665 nm.

Figure 3.1.2: UV-Vis Spectrophotometer

3.1.3 Oven

In order to dry the fibers, oven is used. Temperature fixed in this experiment is 105 ^{o}C. The oven that used in this research is Memmert brand.

Figure 3.1.3: Memmert Oven

3.1.4 Hot Plate Stirrer

Hot plate stirrer used to heat and stir the solution of MB when the experiment runs. This type of hot plate stirrer could adjust the speed and temperature needed.

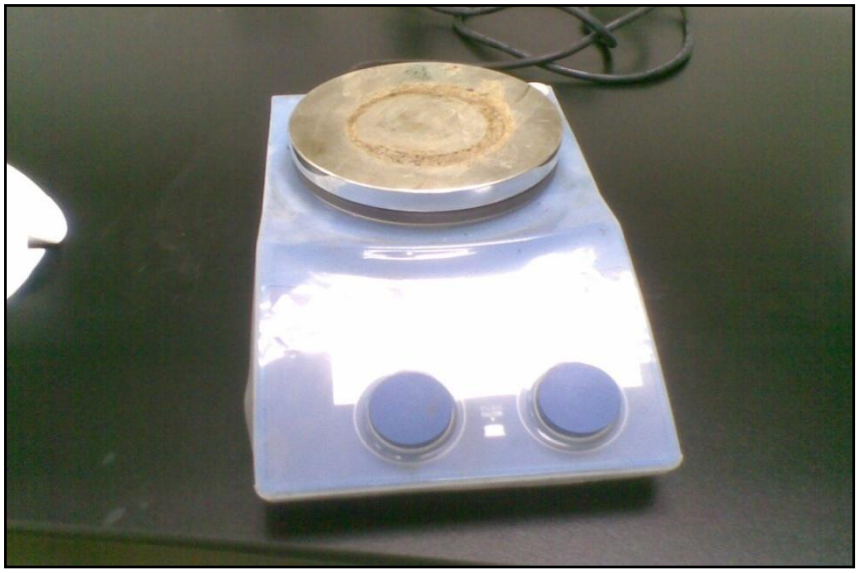

Figure 3.1.4: Hot Plate Stirrer

3.1.5 pH Meter

In this research, one of most important equipment is pH meter. pH meter is used to check the pH of washing solution of AC in order to neutralize it. Moreover, this equipment is important to check the pH of methylene blue solution.

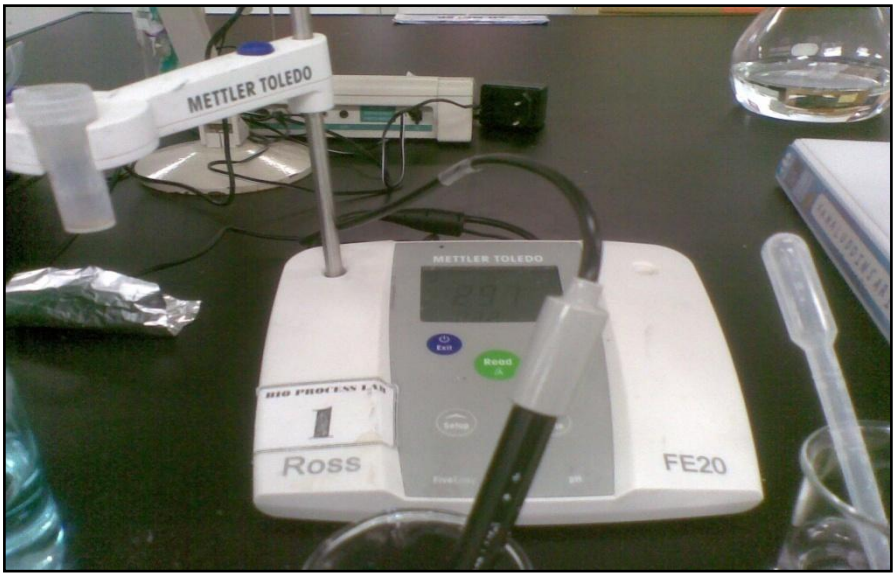

Figure 3.1.5: pH Meter

3.1.6 Measuring Weight

Measuring weight is used to weigh EFB and PPF before carbonization process occur. Then, it used to weigh the amount of activated carbon needed and methylene blue prior to dilute.

Figure 3.1.6: Measuring Weight

3.2 Materials

a) Methylene Blue
b) Hydrochloric Acid
c) Potassium Hydroxide

3.3 Experimental Procedure

3.3.1 Sample Collection

The oil palm EFB and palm-press fiber (PPF) are collected from a palm oil mill in Lepar, which is known as Kilang Sawit LCSB Lepar. It is located in Gambang, Pahang Darul Makmur which is nearby Universiti Malaysia Pahang. Both of these fibers are collected in a box and stored in a store before further uses. Then, the EFB are loose to the fibrous strands before drying into oven.

3.3.2 Activated Carbon Preparation

First, those fibers are dried in an oven at 105 ^{o}C for 24 hours. It is done in order to remove the moisture contents in the fibers since both of fibers are contains a lot of moisture. After that, the dried samples are then carbonized in a furnace at 240 ^{o}C for 2 hours. Char produced are then soaked in Potassium Hydroxide (KOH) solution with impregnation (char:KOH) ratio of (1:1) (wt%). The soaked char then are dried in the oven at 105 ^{o}C for 24 hours. The activation step is conducted in a furnace at 200 ^{o}C to 300 ^{o}C about four hours. The activated product are then washed with hydrochloric acid (HCl) of 0.01 M and distilled water until the pH of washing solution reached 6 to 7.

3.3.3 Effect of Agitation Speed and Contact Time

In order to study the effect of methylene blue initial concentration and contact time on the adsorption uptake, 25 mL of methylene blue solution with initial concentration of 50 ppm is prepared in a 100 mL beaker. 0.25 g of AC is added into each beaker's solution prior to stir by magnetic stirrer on the hotplate stirrer. Then, the solutions are stirred for 5, 10, 15 and 20 minutes for each with rotation speed of 170 rpm. Then, the experiments are repeated by changes the rotation speed to 200 rpm and 700 rpm.

The adsorption capacity (q) = $\frac{[C_o - C_e]V}{W}$ (3)

Where Co is initial concentration (mg/L), Ce is equilibrium concentration after adsorption process (mg/L), V is the volume of the solution (L) and W is the dry mass of activated carbon used (g). The unit of q is mg/g.

3.3.4 Effect of Activated Carbon Dosage

The effect of initial concentration of methylene blue is then studied by varying amount of AC used which started with 0.5 g, 1.0 g, 1.5 g and 2.0 g. 100 ppm of solution is prepared in a 100 mL beaker. The concentration and 50 mL volume of MB is fixed in this experiment. The desired amount of AC added into the solution of MB and then it stirred by using magnetic stirrer with 450 rpm in one hour.

The removal (%) = $\frac{C_o - C_e}{C_o}$ x 100 (4)

3.3.5 Elemental Analysis

After filtered the samples by using filter paper to get the final concentration value, adsorption capacity is measured by using UV – Visible Spectrophotometer by put the samples in the cuvettes. The wavelength of methylene blue set is 665 nm. By using this equipment, the value of absorbance is read.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Calibration Curve

Based on table 4.1, the calibration curve for methylene blue solution is produced according to various concentrations. Then, absorbance result is read by using UV – Visible Spectrophotometer. The wavelength of methylene blue used in this research is 665 nm. This calibration curve is generated to measure the initial concentration of absorbance at certain concentration. Then, this result of absorbance is tended to easier the process to calculate the final concentration in order to calculate adsorption capacity. The result is tabulated in the Table 4.1.1 below.

Table 4.1: Concentration and Absorbance of Stock Solution

Absorbance	Concentration, ppm
0.293	1
0.447	2
0.800	5
1.548	10
2.456	15

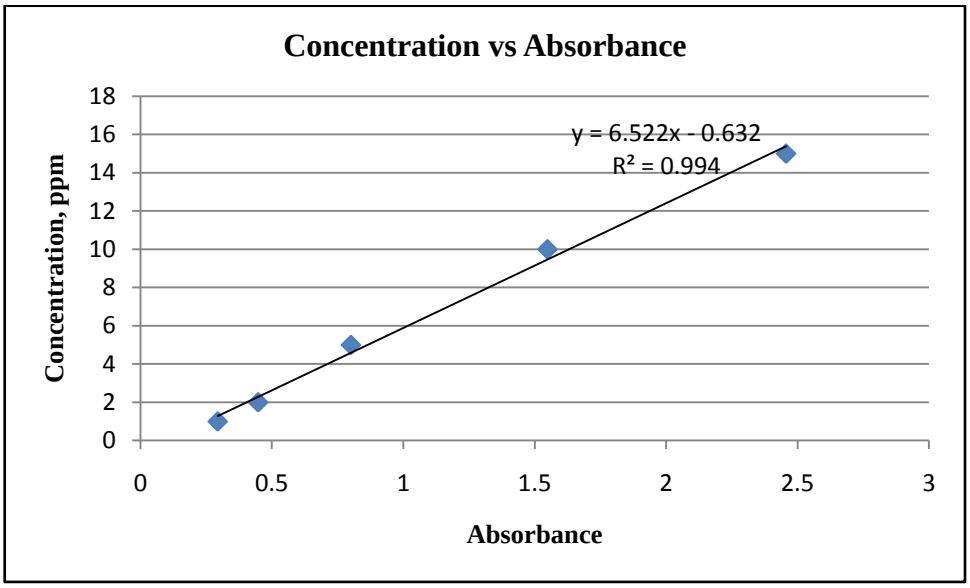

Figure 4.1: Graph Concentration versus Absorbance of Stock Solution

According to the Table 4.1 and Figure 4.1, the concentration of methylene blue is proportional with absorbance. For 1 ppm, the absorbance obtained is 0.293. Then, the absorbance increased for 2 ppm which is 0.447. For 5 ppm, the absorbance got is 0.800. 10 ppm concentration of methylene blue aqueous solution got 1.548 of absorbance which is greater than the 1st sample, 2nd sample and 3rd sample. When the concentration increased to 15 ppm, the absorbance also increased to 2.456. It showed that, concentration is directly proportional to absorbance. The equation obtained from this graph is y = 6.522x – 0.632. The regression (R^2) is 0.994.

As noted earlier, the higher the concentration, the higher amount of absorbance. The higher the concentration of methylene blue absorbs more lights and transmits less light compare to lower concentration. The direct relationship between absorbance and concentration for a solution is known as Beer's Law.

A few samples are prepared in different concentration of each. Then, the absorbances of the solutions are figured out by using UV – Visible Spectrophotometer in a cuvette. The amount of light that get through the solution and strikes the photocell is used to compute the absorbance of each solution. Absorbance is the amount of light that can be measured.

4.2 Effect of Agitation Speed and Contact Time

Table 4.2: Result of Adsorption Capacity for 170 rpm, 200 rpm and 700 rpm.

Time, minute	Adsorption Capacity, mg/g for 170 rpm	Adsorption Capacity, mg/g for 200 rpm	Adsorption Capacity, mg/g for 700 rpm
0	0	0	0
5	4.88	4.81	4.58
10	4.82	4.65	4.64
15	4.9	4.72	4.85
20	4.87	4.92	4.91

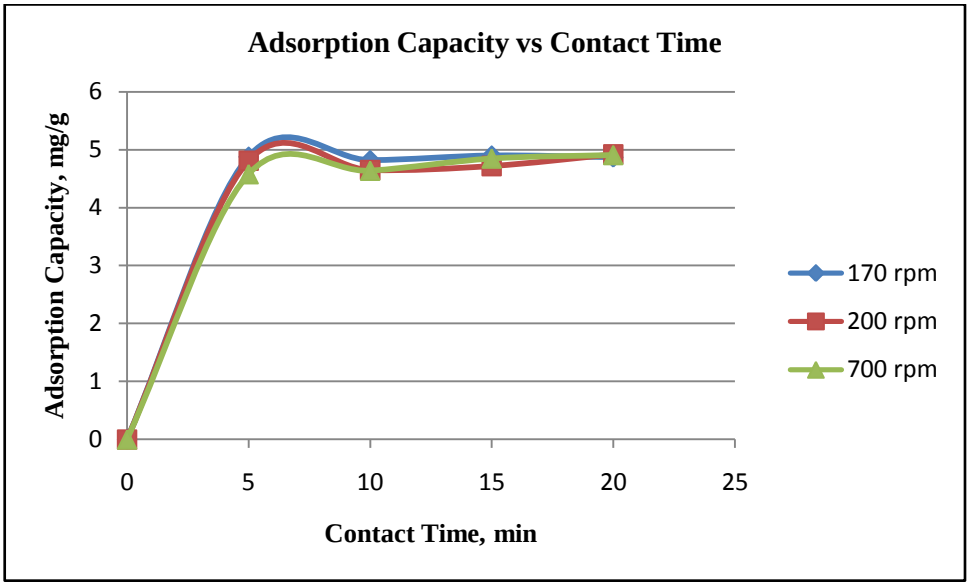

Figure 4.2: Graph of Adsorption Capacity versus Time for 170 rpm, 200 rpm and 700 rpm

Table 4.2 and Figure 4.2 show the result of concentration and adsorption capacity of Methylene Blue after a few minutes with stirring at 170 rpm, 200 rpm and 700 rpm. 0.25 g dry activated carbon/25 mL of 50 ppm of Methylene Blue solution is used for this parameter. The result shows the adsorption process occurred when the adsorption capacity increased with time.

For 170 rpm agitation speed, the adsorption capacity which started with zero is increased with time. The first sample gave zero adsorption capacity because the concentration of Methylene Blue solution is not changed. It is because no contact time on this part. At 5^{th} minutes, the adsorption capacity is increased to 4.88 mg/g. It means the adsorption process started to react. However, for sample of 10^{th} minutes, the adsorption capacity is lower than the sample for 5^{th} minutes which is 4.82 mg/g. For 15^{th} minutes, the adsorption capacity increased back in which 4.9 mg/g but 20^{th} minute sample is showed that the adsorption capacity decreased again to 4.87 mg/g.

For 200 rpm of speed, the adsorption capacity at the 0^{th} minutes is zero because no reaction happened at this time. The adsorption capacity increased at 5^{th} minutes where it got 4.81 mg/g. But, at 10^{th} minutes of contact time, the adsorption capacity is 4.65 mg/g and increased back to 4.72 mg/g and 4.92 mg/g at 15^{th} and 20^{th} minutes of contact time.

There are a few read that got decreasing in term of the adsorption capacity value. As example is at 10^{th} minutes of contact time for 170 rpm and 200 rpm of speed and the other one is at 20^{th} minutes for 170 rpm of speed. This is happened because the contact time taken for the activated carbon adsorbs the molecules of methylene blue is not sufficient.

For speed 700 rpm, the adsorption capacity increased gradually with time. At zeroth minute, no adsorption capacity obtained because the molecules of Methylene Blue is not adsorbed yet by activated carbon. The adsorption capacity increased for 5^{th} minutes and 20^{th} minutes of contact time which is 4.58 mg/g to 4.64 mg/g to 4.85 mg/g and 4.91 mg/g. It means no error occur while doing this experiment.

Based on the observation, for all speeds, the adsorption capacity got the constant value approximately. The adsorption capacity increased and almost achieved the equilibrium. The longest contact time gave the highest adsorption uptake. This is shown by the value of the concentration in the tables. However, according to speed, 170 rpm of speed got the best result compared to 200 rpm and 700 rpm.

The longest contact time gave the highest adsorption capacity. In adsorption process, initially the methylene blue molecules have to face the boundary layer effect and then it diffused from the layer into the activated carbon surface. Finally, the molecules diffused into the porous structure of activated carbon (D.S. Faust, 1983). This phenomenon takes relatively long contact time. The higher initial concentration of methylene blue caused the longer contact time needed to adsorb.

The adsorption capacity also affected by agitation speed. The speed used in this experiment is 170 rpm, 200 rpm and 700 rpm. From the experiment, the lowest speed which is 170 rpm gave the most efficient amount of adsorption capacity compared to others.

Based on theory, the highest adsorption of capacity is affected by agitation speed. When the speed increases, the adsorption capacity also increases. It is due to the fact that the agitation speed improves the diffuses of methylene blue ions towards the surface of activated carbon and decreases the mass transfer resistances and causes faster external mass transfer rate of methylene blue ions and gives higher adsorption capacity (Muataz et.al, 2010). However, when the speed is too high, molecules that already attracted to the surface molecules of activated carbon will be desorbed back in the solution. It is because the Van der Waals forces are too weak to bind the molecules. Therefore, in this case, the 170 rpm of speed is the optimum speed to increase the adsorption capacity.

4.3 Effect of Activated Carbon Dosage

Table 4.3: Data Collection for Dosage of Activated Carbon

Mass of AC, g	Absorbance	Concentration, ppm	Adsorption Capacity, mg/g
0.5	0.359	1.71	9.83
1.0	0.311	1.40	4.93
1.5	0.415	2.07	3.26
2.0	0.437	2.22	2.44

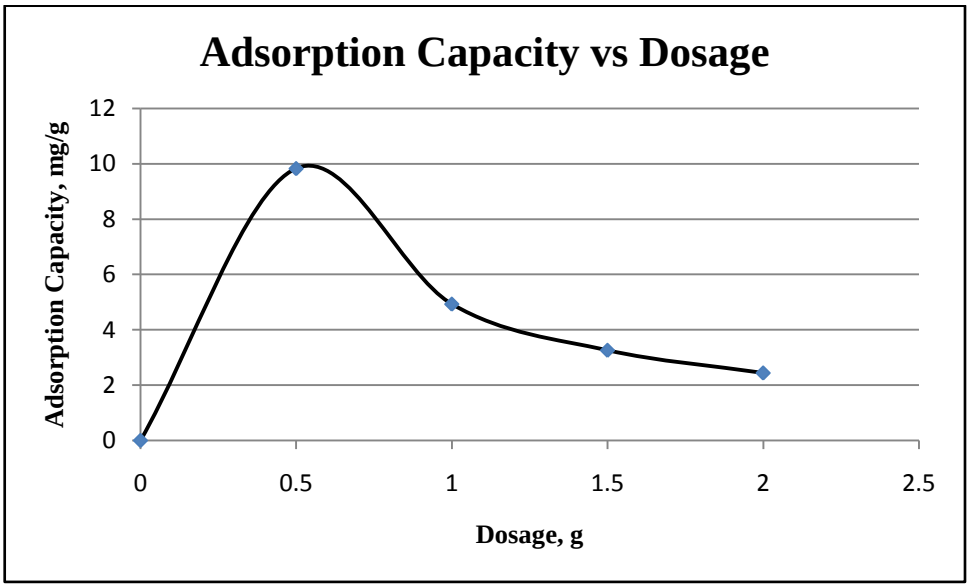

Figure 4.3: Graph Adsorption Capacity versus Dosage of AC

Another parameter run in order to know the effectiveness of activated carbon produced in adsorption process. Dosage of activated carbon is also important in adsorption process. This part, the concentration of methylene blue is constant to 100 ppm. The volume is 50 mL. The dosages prepared are consisted of 0.5 g, 1.0 g, 1.5 g and 2.0 g of activated carbon. The contact time also fixed to one hour operation.

The theory of this experiment is stated that the higher dosage of activated carbon will increase the amount of adsorption capacity. It is because the attractive forces of the molecules of activated carbon increased. So, the attractive forces attracted the hydrophobic substances included in the methylene blue aqueous solution also increased. On the other hand, the increase in adsorption with the activated carbon dosage attributed to greater surface area and increased the adsorption sites.

Based on the result obtained, a graph showed in Figure 4.3. According to the graph, the adsorption capacity at zero gram of activated carbon gave zero adsorption capacity. It is because no physical adsorption occurs at that moment. The adsorption capacity for 0.5 g of activated carbon is 9.83 mg/g. However, for 1.0 g, 1.5 g and 2.0 g, the adsorption capacity obtained after one hour is declined gradually in which 4.93 mg/g, 3.26 mg/g and 2.44 mg/g.

The adsorption capacity decreased sharply with the increasing of adsorbent dosage at 1.0 g. The methylene blue adsorption capacity decreases gradually at adsorbent dosage 1.5 g with 3.26 mg/g adsorption capacity and at 2.0 g with 2.44 mg/g. These results because of the adsorption sites are overlapping as a result of overcrowding of adsorbent particles (Garg et. al, 2003). Furthermore, the high adsorbent dosage could impose a screening effect of the dense outer layer of the cells, thereby shielding the binding sites from adsorbate (Pons, M.P et. al, 1993).

4.4 Adsorption Isotherms

Generally, adsorption isotherm is a model to describe the interaction of adsorbates with adsorbents. Langmuir isotherm could be used as a model to describe the adsorption. The result of Langmuir model is tabulated in Table 4.4 below. The Langmuir isotherm is characterized by the following assumptions. The assumptions are consisted of (1) adsorbated molecules behave ideally, (2) adsorption is monolayer, (3) all the sites on the surface are equivalent, (4) There are no adsorbate-adsorbate interactions, (5) Adsorbed molecules is immobile (J. Thirumal and S Kaliappan, 2011).

Table 4.4: Langmuir isotherm for Various Agitation Speed

Speed, rpm	**b (dm^3/mg)**	**Q (mg/g)**	**R^2**	**R_L**
170	15.308	5.025	0.992	0.00130
200	12.706	4.854	0.996	0.00157
700	11.105	4.739	0.996	0.00180

As shown in the table, the resulting Q value for 170 rpm is 5.025 mg/g which is the highest compared to the others. While the value of Q for 200 rpm is 4.854 mg/g. The agitation speed of 700 rpm only got the value of Q as much as 4.739 mg/g. It is the lowest value of Q. The result indicated that 170 rpm of agitation speed slightly higher adsorption capacity of methylene blue compared to others. The graph plotted of Ce/qe against Ce in Figure C4 Figure C5 and Figure C6 gave straight lines, implies that the adsorbents well fitted to Langmuir isotherm. Value of R^2 is the correlation coefficient which is 0.992 and the value of R_L is 0.00130 for 170 rpm which means the isotherm is in favourable type.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The activated carbons from EFB and PPF with ratio (1:1) (wt%) are derived from chemical activation by KOH activation. There are three parameters that done in this research. The calibration curve which is graph of concentration versus absorbance showed that the Methylene Blue solution got the equation, $y = 6.522x - 0.632$. The concentration is proportional to absorbance.

The results revealed that the activated carbon at 20 minutes showed good adsorption capacity with low content of methylene blue released in aqueous solution compared to others. However, the speed at 170 rpm is more effective than 700 rpm in case that it gave the highest adsorption capacity. It is because 170 rpm is the optimum speed to get higher amount of adsorption capacity. It is due to the molecules of methylene blue that already binds to the molecules surface of the solid are desorbed back related to the weak Van der Waals forces that attract the molecules.

It is observed that the amount of activated carbon is important in order to increase the adsorption capacity. From the result obtained, the adsorption capacity for 0.5 g is the most effective. The adsorption capacity is decreased sharply with the increasing of adsorbent dosage. It is due to the overcrowding of adsorbent particles. Moreover, the higher of activated carbon dosage could impose a screening effect of the dense outer layer of the cells, thereby shielding the binding sites from the adsorbate. From the Langmuir isotherm model, the result got straight line. It showed that the

adsorbents well fitted with Langmuir isotherm model. 170 rpm of speed obtained the highest adsorption capacity which is 5.025 mg/g compared to others. As a conclusion, the adsorption capacity affected by the dosage of activated carbon, contact time and agitation speed.

5.2 Recommendations

In order to get the most efficient activated carbon produced, the thermal activation must be run. It is because activation temperature is one of the most important parameter in producing good activated carbon. It is to enlarge the BET surface area of the solid.

Other than that, the activated carbon produced must be grinded to the smaller size fraction. It because if the size reduces, then, the surface area will increases. Thus, it will affect the adsorption capacity. In doing the experiment, error must be avoided. Activation time also important in producing a good activated carbon. As the time increased, then, the percentage of yield gradually decreased and the BET surface area also increased.

While running the experiment, amount of activated carbon needed must be accurate in order to avoid bias in adsorption process. If the dosage used is not accurate, thus the obtained result is not precise. The pH when neutralized the activated carbon also need to be similar to all the parameters. Furthermore, the methylene blue solution must be diluted precisely. Besides, stirring is one of the important method that must be done while running this experiment. In fact, the activated carbon produced must be safely store from the dirt and other contaminants that could be affect the adsorptivity of adsorbents.

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APPENDIX A

CALCULATION OF STANDARD AND STOCK SOLUTION

Standard Solution of Methylene Blue

Molecular Weight: 355.89 g/mol

For 200 ppm:

$$\frac{200 \times 10^{-3} g/L}{355.89\ g/mol} = 5.62 \times 10^{-4} M$$

In 1L of water:

n = (5.62 x 10^{-4}) (1L) = 5.62 x 10^{-4} mol

m = (5.62 x 10^{-4} mol) (355.89 g/mol) = 0.2 g

For 50 ppm:

$$\frac{50 \times 10^{-3} \frac{g}{L}}{355.89 \frac{g}{mol}} = 1.405 \times 10^{-4} M$$

In 25 mL of water:

M1V1 = M2V2

(5.62 x 10^{-4} mol) (V1 L) = (1.405 x 10^{-4} mol) (0.025 L)

V1 = 6.25 mL

In 50 mL of water:

M1V1 = M2V2

(5.62 x 10^{-4} mol) (V1 L) = (1.405 x 10^{-4} mol) (0.05 L)

V1 = 0.0125 L

V1 = 12.5 mL

For 100 ppm

$$\frac{100\ \times 10^{-3}}{355.89} = 2.81\ \times 10^{-4}\ M$$

M1V1 = M2V2

In 50 mL of water:

$(5.62 \times 10^{-4})\ (V1) = (2.81 \times 10^{-4})\ (0.05)$

V1 = 0.025 L = 25 mL

APPENDIX B

LIST OF DATA COLLECTIONS

Table B1: Data Collection for 170 rpm

Time, minute	Absorbance	Concentration, ppm	Adsorption Capacity, mg/g	Ce/qe
0	7.763	50	0	0
5	0.281	1.20	4.88	0.25
10	0.375	1.81	4.82	0.38
15	0.255	1.03	4.90	0.21
20	0.296	1.30	4.87	0.27

Table B2: Data Collection for 200 rpm

Time, minute	Absorbance	Concentration, ppm	Adsorption Capacity, mg/g	Ce/qe
0	7.763	50	0	0
5	0.392	1.92	4.81	0.40
10	0.635	3.51	4.65	0.75
15	0.528	2.81	4.72	0.60
20	0.213	0.76	4.92	0.15

Table B3: Data Collection for 700 rpm

Time, minute	Absorbance	Concentration, ppm	Adsorption Capacity, mg/g	Ce/qe
0	7.763	50	0	0
5	0.747	4.24	4.58	0.93
10	0.652	3.62	4.64	0.78
15	0.328	1.51	4.85	0.31
20	0.236	0.91	4.91	0.19

APPENDIX C

LIST OF FIGURE

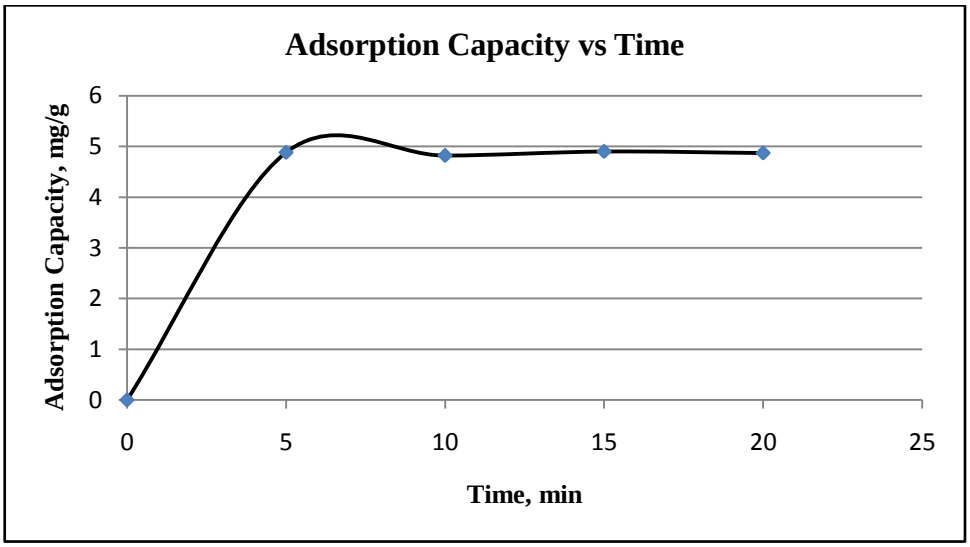

Figure C1: Graph of Adsorption Capacity versus Time at 170 rpm

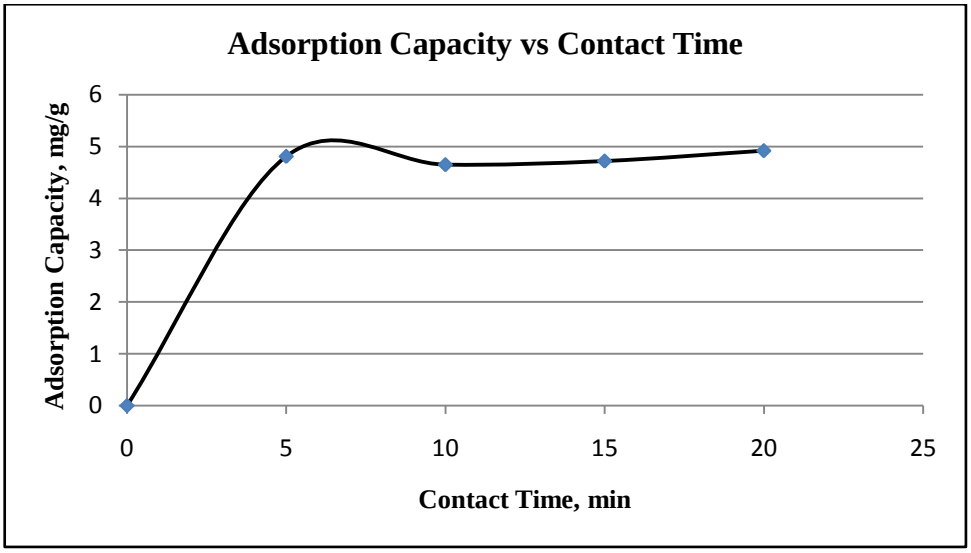

Figure C2: Graph of Adsorption Capacity versus Time at 200 rpm

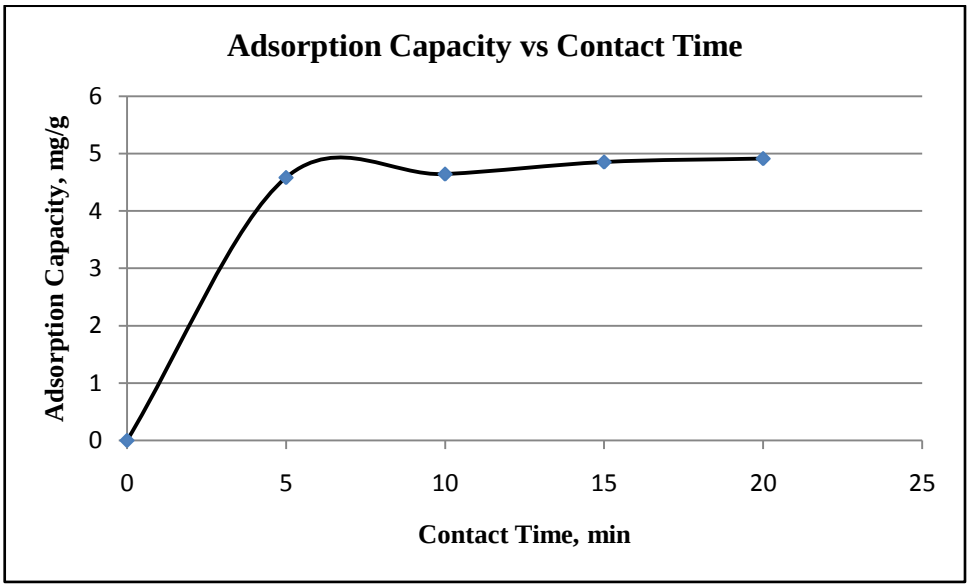

Figure C3: Graph of Adsorption Capacity versus Time at 700 rpm

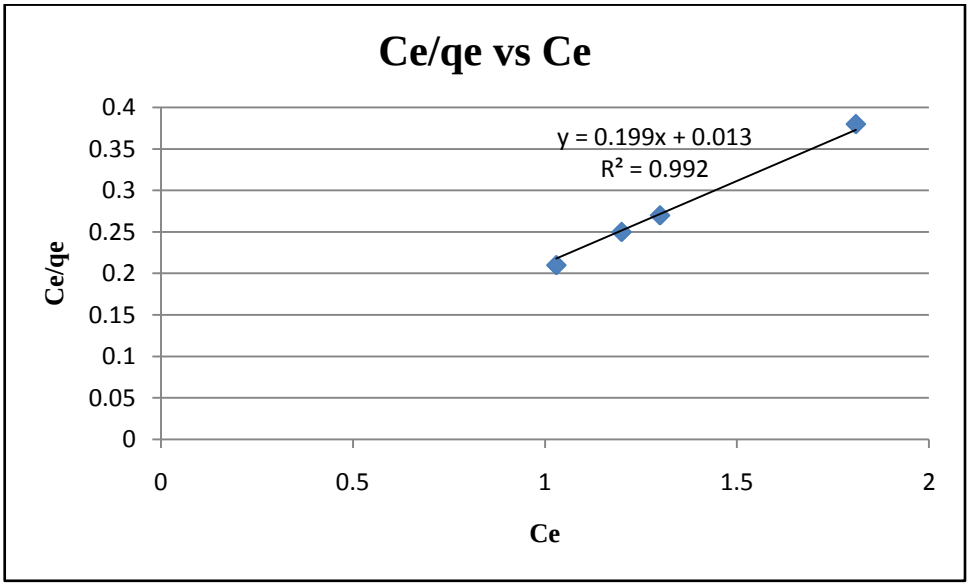

Figure C4: Langmuir Isotherm for 170 rpm

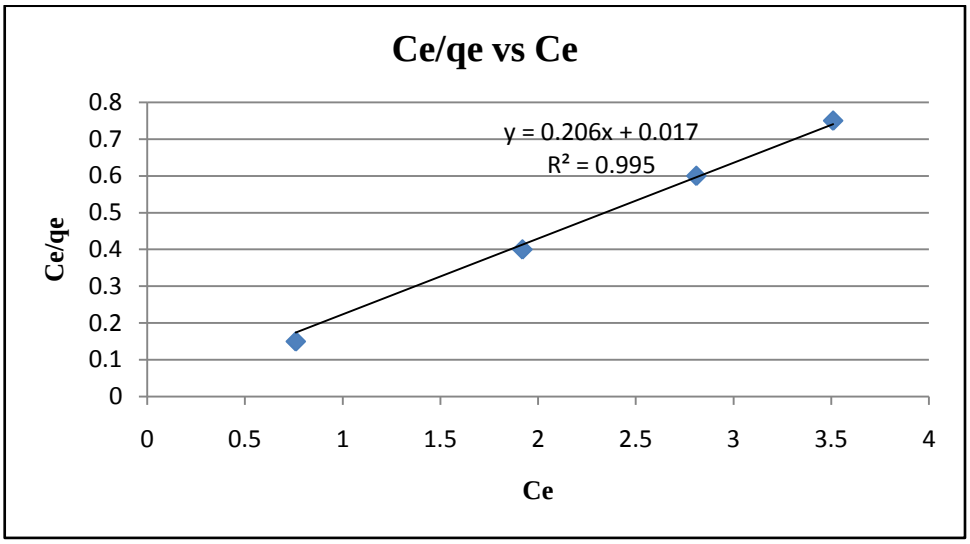

Figure C5: Langmuir Isotherm for 200 rpm

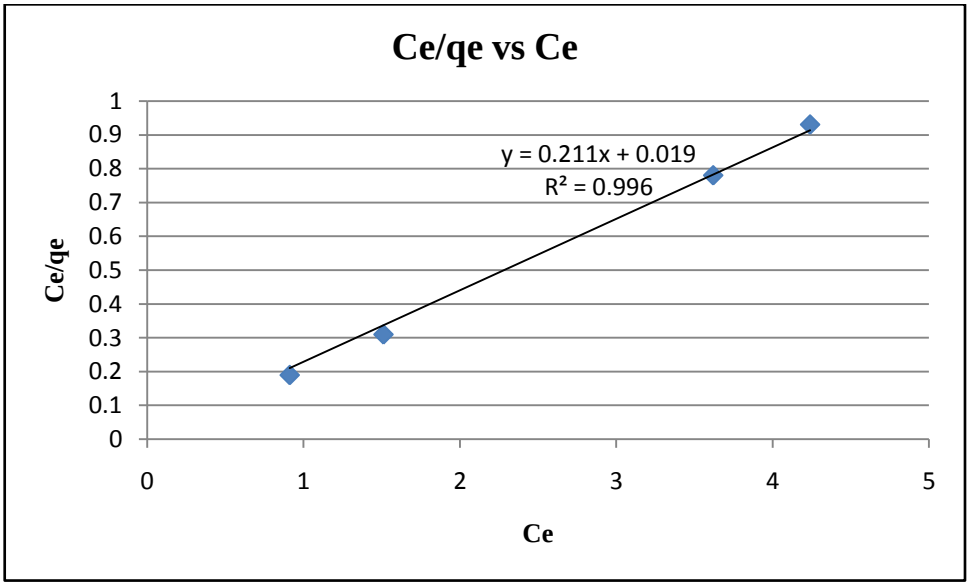

Figure C6: Langmuir isotherm for 700 rpm

Figure C7: Changes of Colour of Methylene Blue

Figure C8: Empty Fruit Bunch Fiber