### ADSORPTION STUDIES OF SYNTHETIC BATIK DYE USING GRANULAR ACTIVATED CARBON IN CONTINUOUS SYSTEM

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A thesis submitted in fulfilment

of the requirement of the award of the

Bachelor Degree of Chemical Engineering

Faculty of Chemical Engineering and Natural Resources Engineering

Universiti Malaysia Pahang

June 2012

#### ABSTRACT

From this experiment, activated carbon as the adsorbent which from coconut shell is taken from industry. As well as methylene blue as the adsorbate. Dye will be flowed to the packed bed continuously. Two parameters will be concerned in this experiment which is initial concentration and time. In order to determine how this parameters will affect the adsorption process, so the percentage of loss concentration of dye will be determined. What will be expected is the higher initial concentration, the higher the adsorption capacity. Similar to time parameter, the longer time it takes, the percentage of concentration loss will be increase. So, from the data,the maximum percent removal of methylene blue is 87.7% for 40 ppm MB concentration, and adsorption start to constant after 3 hour contact time

#### ABSTRAK

Daripada eksperimen ini, arang teraktif yang disediakan daripada kelapa telah diambil dari industri sebagai bahan penjerap. Metilena biru akan dijadikan sebagai bahan terjerap. Pewarna akan mengalir ke katil yang dipenuhi secara berterusan. Dua parameter akan ditekankan dalam eksperimen ini iaitu kepekatan awal dan masa. Untuk menentukan bagaimana parameter ini akan menjejaskan proses penjerapan, jadi peratusan kepekatan kehilangan pewarna akan ditentukan. Apa yang akan dijangka kepekatan permulaan yang lebih tinggi, lebih tinggi keupayaan penjerapan. Serupa dengan parameter masa, masa yang lebih lama ia mengambil masa, kerugian peratusan kepekatan akan meningkat. Jadi, daripada data, peratus penyingkiran maksimum metilena biru adalah 87.7% selama 45 MB kepekatan ppm, dan penjerapan mula malar selepas masa sentuhan 3 jam.

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### LIST OF ABBREVIATION

- BOD: Biochemical oxygen demands
- COD: Chemical oxygen demands
- SD : Suspended Solids

### LIST OF SYMBOLS

- $^{\circ}$  : degree celcius
- g : grammes
- kg : kilogrammes
- L : litre
- µm: micrometer
- mL :millilitre
- ppm: part per million

#### **CHAPTER 1**

#### **INTRODUCTION**

#### 1.0 RESEARCH BACKGROUND

The industries nowadays gives severe impact to our nature by producing a lot of pollution that have the tendency to brings more harmful than good such as air pollution, sound pollution and water pollution. Water pollution is one of the serious environmental issues because it can cause problem to the clean water production and also can contributes to the shortage of clean water supplies. Water pollution occurs when the physicals and chemicals properties of the water are change or in the other word is, there is a contamination in the water such as lakes, rivers, oceans and also ground water.

Wastewater pollution is always consider as dangerous because it has been affected by the hazardous or dangerous chemicals and release to the water source such as rivers, lakes and oceans. The lack of clean water has always been an issue of environmental concern all over the world. This environmental issue is mainly stressed in developing countries today. Just imagine what would happen for the future life if no action will take to control the wastewater pollution. It can affect our sustainable nature and at the same time our source of clean water is not enough to meets the needs of the human from days to days. The wastewater pollution is commonly produced by domestic residences, commercial properties, industry, and agriculture. The unwanted contaminant in the wastewater can be dangerous to the aquatic and human health. It might contain the heavy metal in different concentrations and pH values that are dangerous. There are many industries that contribute to the wastewater pollution and textile industry is a part of it. The textile industry has been condemned as being one of the world's worst offenders in terms of pollution because it requires a huge amount of two components which is chemicals. Water is used transmit the chemicals used during that step and to wash them out before beginning the next step. The water is then expelled as wastewater. It will pollute the environment. When the colouring textile's operation was held, the process involved a lot of chemicals substance. After the process was finished, all the waste dyes will be removed by make it flow down to the rivers or lakes. The wastes coming from these industries can effect on our environment and causing pollution.

Since then, there are so many efforts have been made to solve the problems including increase more stringent water regulation which have lent added urgency to the need to decontaminate wastewater and polluted trade effluent due to many communities rely on groundwater as a primary source of drinking water (Okolo et al., 2000; Edil, 2003). This has forced many companies to find alternative methods to reduce the amount and environmental sensitivity of the liquid waste they produce.

There are several ways to treat the waste water. It consists of physical, chemical and biological method. Microbial degradation, activated sludge and bio film process are the examples of biological method, which accepted for its potentialities but applicable only to lower concentration range. Meanwhile, the chemical and physical methods such as solvent extraction, precipitation, filtration, adsorption, and chemical oxidation are normally used to treat pollutants especially organic compounds and heavy metals. Every particular method is based on the application to the solute concentration range, capacity, cost, reusability and the reproducibility (Kentish & Stevens, 2001; Sarkar et al., 2003; Abburi, 2003).

Among of them, adsorption is widely used in the dyes treatment process because it is very versatile can be used in the wide range (Lawrence et al., 2007). Adsorption is a fundamental process physicochemical treatment of municipal wastewaters, a treatment which can economically meet today's higher effluent standards and water reuse requirements. At the same time, if large volumes of wastewater are involved, treatment processes are carried out on continuously flowing wastewaters. Even though the costing is quite high, it can improve the quality of the treatment.

#### **1.1 PROBLEM STATEMENT**

High level of dyes production and their widespread use in many applications generate colours wastewaters which cause severe water pollution. The pollutant contributes to high suspended solids (SS), chemical oxygen demands (COD), biochemical oxygen demands (BOD), heat, colour, acidity basicity and other soluble substances (Ahn et. al 1999. Since the textile is one the world's worst offenders in terms of pollution in industry, it is best to use continuous system of waste water treatment as the solution.

#### **1.2 RESEARCH OBJECTIVE**

The objective of this research was as follow to study the effect of the initial concentration and times of adsorbate and dosage of adsorbent for removal of methylene blue in continuous system by using different type of biomass activated carbons.

#### **1.3 RESEARCH SCOPE**

In this research, it is focused on the continuous adsorption process of methylene blue from aqueous solution. This research focuses on designing and fabricating the continuous adsorption system.

It is also focusing on the effect of initial concentration of methylene blue in changes and contact time of adsorption. Then, the adsorption performance is identified by using Langmuir Isotherm Model.

#### **CHAPTER 2**

#### LITERATURE REVIEW

#### 2.1DYES

Dyes and pigments are widely used, mostly in the textiles, paper, plastics, leather, food and cosmetic industry to color products. Dyes present in waste water may cause serious environmental pollution problems, in the form of reduced light penetration and reduced photosynthesis. Besides that, dyes are one of the sources of esthetic pollution and eutrophication (Tsai et al., 2004).

Organic compounds such as dyes are problematic pollutants that are often discharged into wastewaters from industrial. Textile industry is one of the most important and rapidly developing industrial sectors. It has a high importance in terms of its environmental impact, since it consumes considerably high amounts of processed water and produces highly polluted discharge water in large amounts.

Dyes are chemicals that binding with material which give colour to the material. Dyes are ionic, aromatic organic compounds with structures including aryl rings which delocalised electron system. The colour of dye is provided by the presence chromophore group. They are widely used in many industries such as textile, rubber, paper, plastic, cosmetic etc. Textile industry gives highest rank of dye usage. (Saiful Azhar et al., 2005).

#### 2.2 CLASSIFICATION OF DYES

Dyes are classified usefully in two separate ways, either accordance to their chemical structure or according to the method of application (dyeing method). In term of chemical structure, dyes may either be inorganic or organic compounds. Both groups can be further subdivided into natural and synthetic representatives.

When classified according to dyeing method, one can be distinguish between anionic, direct, or disperse dyes, depending on whether these substances are use for dyeing protein, cellulose, or polyamide fibers. Furthermore, certain chemically reactive dyes can be used for different substrate, while for the same type of chemical structure are suitable for a single substrate only.

For the two classifications based on chemical structure and on method of application overlap, there is hardly a chemical class of dye which occurs solely in one coloristic group, and vice versa. Furthermore, some coloristic groups can be applied to two or more substrates, whilst others are specified to a single substrate. Both classifications are used by the Colour Index (1971), which lists all dyes and pigments used commercially for large-scale coloration purpose, such as dyeing of textile fibers, for pigment coloration of plastics, paints, printing inks and for the coloration of liquids (solvents).

#### 2.2.1 REACTIVE DYES

Reactive dyes are coloured compounds which contain one or two groups capable of forming covalent bonds between a carbon or phosporus atom of the dye ion or molecule and an oxygen, nitrogen or sulfur atom of a hydroxy, an amino or a mercepto group, respectively, of the substrate. Reactive dyes are used extensively in textiles industries regarding favourable characteristics of bright colour, water-fast, simple application techniques with low energy consumption (Aksu, 2005). According to Aksu et al., (2006), reactive dyes are the largest class of dyes used in textiles industry and it is the azo dyes based on the azo chromogen (-N=N-) and the presence of bright colour due to these azo bonds and associated chromophore.

#### 2.2.2 METHYLENE BLUE, MB

Although the dye is not regarded as a very toxic dye, MB can have various harmful effects on human being and animals. Once inhaled, it can cause heart rate increasing, nausea and vomiting (Hui Deng et. al, 2011). It is generally used for dyeing cotton, wool, and silk. MB can cause eye burns in humans and animals, methemoglobinemia, cyanosis, convulsions, tachycardia, dyspnea, irritation to the skin, and if ingested, irritation to the gastrointestinal tract, nausea, vomiting, and diarrhea (S. Senthilkumaar et.al 2005).

Although the dye is not regarded as a very toxic dye, it can have various harmful effects on living things (S. Cengiz and L. Cavas 2008). Once inhaled, it can cause heart rate increasing, nausea and vomiting. If the amount of intake is large, severe symptoms such as difficulties in breathing, mental confusion, and methemoglobinemia-like syndromes may take place (K.G. Bhattacharyya and A. Sharma 2005). In addition, effluents of the manufacturing and textile industries are discarded into rivers and lakes, threatening aquatic life through reducing light diffusion and consequently inhibiting the photosynthesis processes (P. Waranusantigul et. al 2003).

This dye has been studied because of its known strong adsorption onto solids, and it often serves as a model compound for removing organic contaminants and coloured bodies from aqueous solutions (B.H. Hameed, A.T.M. Din, and A.L. Ahmad 2007). The chemical formula of methylene blue is (Wang et al., 2005).

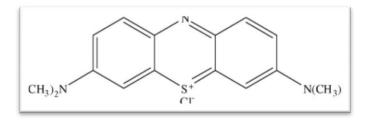


Figure 1.0 Chemical Formula for Methylene Blue

Wavelength of methylene blue is 665 nanometer (Murat Oza, E. Lorkec, and George A. Petroianub 2009).

#### 2.3 ADSORPTION

Adsorption is a process that occurs when a gas or liquid solute accumulates on the surface of a solid or a liquid (adsorbent), forming a molecular or atomic film (the adsorbate). It is different from absorption, in which a substance diffuses into a liquid or solid to form a solution. The term sorption encompasses both processes, while desorption is the reverse process.

Adsorption is more approachable and easy to run due to the price of adsorbent that usually is cheap and does not require a pre-treatment step before its application (Wang et al., 2005). A lot of researchers believes this treatment is to be far more superior than others due to variety reasons ((Sanghi and Bhattacharya, 2002; Meshko et al., 2001;Bulut and Aydin, 2006); including the simple design, low cost, and of course easy to handle.

The continuous adsorption in fixed-bed column is often desired from industrial point of view. It is simple to operate and can be scaled-up from laboratory process. The flow behaviour and mass transfer aspects become peculiar beyond a particular length to diameter ratio of the column. Adsorption in fixed bed columns using activated carbon has been widely used in industrial effluent, since it does not require the addition of chemical compounds in the separation process (J.M Chern and Y.W Chiem, 2003).

#### 2.4 ADSORBENTS

Most of the solid adsorbents of great industrial applications possess a complex porous structure that consists of pores of different sizes and shapes However; the importance of porous structure on adsorption of many contaminants in aqueous solution is not well understood.

Further, a powder is easily recognized as a mass of small dry particles, but the precise definition is inevitably somewhat arbitrary. The term fine powder is also used in an imprecise manner, but it seems reasonable to apply it to a material consisting of particles less than about 1µm. The unit mass of a fine powder contains a large number of small particles and exhibits an appreciable surface area. In addition to the performance, a number of other factors greatly influence the choice and viability of waste materials as adsorbents, for example the cost of processing materials, wastewater selectivity and regeneration of materials. Cost is a very important factor when considering materials for use as adsorbents. It is generally recognized that a material can be deemed 'low-cost' if it requires little processing, is abundant in nature, or is a by-product or waste material from another industry (Bailey et al., 1999). Nonconventional activated carbons exhibited high sorption properties as shown in Table 2.3. The adsorption capacities of a carbon depend on the different sources of raw materials, the history of its preparation and treatment conditions such as pyrolisis temperature and activation time. Many other factors can also affect the adsorption capacity in the same sorption conditions such as surface chemistry, surface charge and pore structure.

Table 1.1Adsorption capacities  $Q_0$  (mg/g) for carbon materials made from solidwastes andcoal-based sorbents

Raw material	Dye	Adsorption	References
		Capacity	
Rattan husk	Methylene blue	294.1	Hameed et al.,
			2007b
Bottom ash coal	Reactive blue 222	3.82	Dincer et al., 2007
Rice husk	Basic green 4	511	Guo et al., 2003
Bagasse	Basic red 22	942	Juang et al., 2002a
Cocunut tree flower	Reactive red 120	181.9	Juang et al., 2002a
Oil palm fibre	Methylene blue	277.7	Tan et al., 2007

Searching for low cost activated carbons has been started a few decades ago as an alternative for expensive coal-based activated carbon. Biomass mainly derived from agricultural solid waste is a preferable option for activated carbon precursors. Biomass materials are cheaper, renewable and abundantly available. Numerous successful attempts have been made to develop activated carbons from various range of agricultural solid waste such as bamboo, rice husk, rubber-wood sawdust, oil palm shell and coir pith. Coconut (Cocos nucifera) is one of the major crops in Malaysia with 142,000 ha of planted land. Therefore, huge amount of solid waste are generated annually, mostly in form of fiber and shell. Naturally, the waste is either left to be rotten or used as burning fuel in which has contributed to environmental disturbances. Hence, the conversion of coconutshell solid waste into activatedcarbon, particularly the shell is most desirable. Coconutshell is very suitable to be converted into activatedcarbon due to its hardness and abrasion resistance. Further, coconutshell chemical composition is akin to hard wood which mainly composed by lignin and cellulose (S. Rodrigues, and G.A.S. Pinto 2007).

#### **ACTIVATED CARBON**

2.4.1

Activated carbons (AC) may deal with almost any organic compound present in the waste. Some advantages such as low-energy demand, possibility of adsorbent regeneration, broad availability of adsorbents and possibility of adsorption from the liquid phase on activated carbons make this an interesting field of research (Ania et al. 2007).

shell, lignite or coal, but almost any carbonaceous material may be used as precursor for the preparation of carbon adsorbents (Rozada et al., 2003). Although various materials are used in the production of commercial activated carbons (wood, coal, peat, lignite, coconut shells), abundant availability and low cost of agricultural by products make them good candidates and sources of raw materials for activated carbons. (R.R Bansode et. al 2003)

Commercially available activated carbons (AC) are usually derived from natural materials such as wood, coconut shell, lignite or coal, but almost any carbonaceous material may be used as precursor for the preparation of carbon adsorbents (Rozada et al., 2003).

Activated carbons are usually derived from natural materials such as wood, coconut

#### 2.4.2 PREPARATION OF ACTIVATED CARBON

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.

For many purposes, moisture content does not affect the adsorptive power. However, it obviously dilutes the carbon (Nurul Ain, 2007). Ash content leads to increase hydrophilicity and can have catalytic effects, which is causing the restricting process (Nurul Ain, 2007). Yang (2003) inorganic material contained in AC which is measured as ash content, generally in the range in between 2 to 10%.

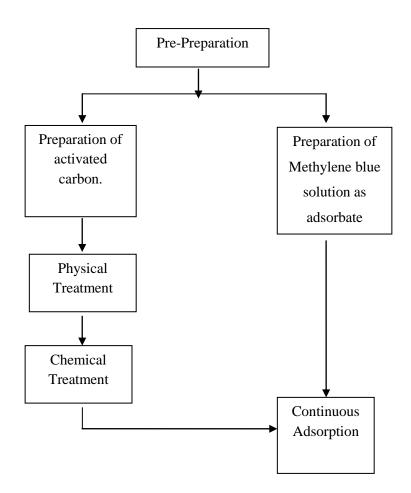
The specific surface area of adsorbent affects the adsorption performance. The larger the specific surface area of AC, the better its performance in adsorption process (Gua and Lua, 2003). The optimum specific surface area of AC is between 600-1200 m<sup>2</sup>/g (Ng et. al, 2002). The adsorption capacity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

**CHAPTER 3** 

METHODOLOGY

## 3.1 OVERVIEW OF METHODS



# 3.2 APPARATUS EQUIPMENT

Table 3.1List of instrument and their purpose in this study

No	Instrument	Purpose
1	UV-Visible Spectrometer	To determine the concentration of Methylene Blue remaining in solutions