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**REMOVAL OF METHYL RED FROM
WASTEWATER USING BANANA PSEUDOSTEM
FIBERS AS AN ADSORBENT**

By

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DECLARATION

I declare that this thesis entitled 'Removal of Methyl Red from Wastewater Using Banana Pseudostem Fibers as Adsorbent' is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted n candidature of any other degree.

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Methyl Red Dye Removal using banana pseudostem fibers adsorbent (raw)

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LIST OF SYMBOLS

λ	Lambda
μm	Micrometer
nm	Nanometer
ml	Milliliter
mg	Milligram
g	Gram
L	Liter
rpm	Revolutions Per Minutes
MR	Methyl Red
$^{\circ}\text{C}$	Degree Celsius
%	Percentage

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Removal of Methyl Red from Wastewater Using Banana Pseudostem Fibers as an Adsorbent

ABSTRACT

Discharging of huge amount of wastewater from textiles industry has caused harm to the environmental. The wastewater treatment has become one of the biggest difficulties confronted by the textiles industry. Nowadays, the capability of agricultural waste as adsorbent to remove dye from the industrial wastewater has been proved by many researcher. Agricultural waste can help to treat the wastewater as well as to reduce environmental problem cause by waste at the plantation. This study represents the potential of banana pseudostem fibers waste as adsorbent for dye removal. The methyl red dye solution was used in this study because methyl red dye that often used in textiles industry can give negative impact to environment and human health. Two type of adsorbent were used in this study which is raw materials and activated carbon from the banana pseudostem fibers. Four parameters of optimization were studied which is contact time, adsorbent dosage, concentration and pH. The experimental result showed, for raw materials from banana pseudostem fibers, the highest percentage of methyl red dye removal was 98.20% and the optimum condition 120 minutes contact time, 0.2g adsorbent dosage, 20 mg/L concentration and pH 3. For the activated carbon, the highest percentage of methyl red dye removal was 99.01% and the optimum condition 30 minutes of contact time, 0.1g of adsorbent dosage, 20 mg/L of concentration and pH 3. This study can conclude that the activated carbon from banana pseudostem fibers have the highest potential to be used as adsorbent to remove dye from the wastewater compared to the raw material from banana pseudostem fibers.

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Penyingkiran Metil Merah Daripada Air Sisa Menggunakan Fiber Batang Pisang Sebagai Penjerap.

ABSTRAK

Pembuangan air sisa dari industri tekstil dalam jumlah yang besar telah menyebabkan kesan buruk kepada alam sekitar. Rawatan air sisa adalah masalah terbesar yang dihadapi oleh industri tekstil. Pada masa kini, kemampuan sisa pertanian sebagai penjerap untuk merawat air sisa daripada industri telah dapat dibuktikan oleh ramai penyelidik. Sisa pertanian dapat membantu untuk merawat air sisa dan juga dapat mengurangkan masalah alam sekitar yang disebabkan oleh sisa pertanian di ladang. Kajian ini mewakili potensi fiber batang pisang sebagai penjerap untuk rawatan menyingkirkan pewarna. Larutan metil merah telah digunakan dalam kajian ini kerana pewarna metil merah yang digunakan dalam tekstil industri boleh memberi impak negatif kepada alam sekitar dan juga kesihatan manusia. Dua jenis penjerap telah digunakan dalam kajian ini iaitu bahan mentah dan karbon diaktifkan daripada fiber batang pisang. Empat parameter pengoptimuman telah dikaji iaitu masa, dos penjerap, kepekatan larutan dan pH. Hasil daripada eksperimen ini menunjukkan, untuk bahan mentah daripada fiber batang pisang, peratusan tertinggi penyingkiran metil merah ialah 98.20% dan keadaan optimum untuk masa ialah 120 minit, 0.2g dos penjerap, 20mg/L kepekatan dan pH 3. Untuk karbon diaktifkan, peratusan tertinggi penyingkiran metil merah ialah 99.01% dan keadaan optimum untuk masa ialah 30minit, 0.1g dos penjerap, 20mg/L kepekatan dan pH 3. Kesimpulan daripada kajian ini ialah karbon diaktifkan daripada fiber batang pisang berpotensi tinggi untuk digunakan sebagai penjerap dalam rawatan penyingkiran pewarna daripada air sisa berbanding dengan bahan mentah daripada fiber batang pisang.

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CHAPTER 1

INTRODUCTION

1.1 Background study

Water pollution is a major global problem caused by human activities to the water bodies (lakes, rivers, ocean and groundwater). There are many pollutants that have been discharged from industries to the water bodies. Due to the pollution, freshwater sources become decrease and unavailable for human use. This statement increase people's fear that whole water supply will be not suitable for drinking and other needs. In the coming decades, water scarcity may leads to social and politically instability, water wars and diseases, unless new ways to supply clean water are found (Kumar Reddy & Lee 2012). The water pollution can cause by discharging of wastewater from textile industry to the environment.

Dye is one of the pollutants that contain in the wastewater. Dye is widely used in textile industry because it is cheap to use as coloring substances as well as easy to apply to any product such as textiles. However, synthetic dyes are very harmful to the human being as well as to the biodiversity (Mas Haris & Sathasivam 2009). There are several types of synthetic dyes that have been used. Methyl red (MR) is one of the examples of synthetic dye. Methyl red dye is a commonly used mono-azo dye in laboratory assays, textiles and other commercial products (Abdullah, 2010). However, eye and skin sensitization and pharyngeal or digestive tract irritation will be occur when inhaled or swallowed the methyl red (Ahmad *et al.*, 2015).

The toxicity of dye in the wastewater can be treated with a necessary treatment. There are several types of treatments which are physical, chemical and biological

treatment that being used to treat the dye. Due to the non-biodegradable dye, the biological treatment was not very effective to remove the dye (Abdullah, 2010). Chemical method is too expensive to apply because of high dosage of chemical need to treat the wastewater (Grag *et al.*, 2004).

One of the most prominent of these treatment methods is adsorption due to its simplicity, efficiency, wide and social acceptability, economic and technical feasibilities (Ogunsile *et al.*, 2014). Activated carbons have been used as adsorbent because of the effectiveness to adsorb. Adsorption limit of activated carbon relies on upon the size of the inner surface, the appropriation of pore size and shape (Rahman *et al.*, 2006). In this century, many research enthusiasm for the utilization of agricultural waste as beginning materials as a result of their ease and across the board accessibility (Mas Haris & Sathasivam 2009). There are many agricultural wastes that can be use as low-cost activated carbon such as, silk cotton hull, coconut tree sawdust, sago industry waste, banana pith, maize cob, rattan sawdust, palm kernel shell, date pits, rice bran, coir pith, rice husk, mango seed kernel powder, straw coconut coir dust, palm fibre, pine cone, coconut shells, groundnut shell, bamboo dust, and wheat husk (Geçgel *et al.*, 2013).

1.2 Problem statement

Nowadays, water has been slightly polluted because of the textile industries. There are many dye use in the industries and one of them is methyl red. Textile industries discharge dye solution to the water bodies and cause water pollution. The water pollution affects many diseases to human and also gives negative impact to aquatic life. The removal of dye from wastewater using chemical and mechanical are expensive and not effective to some dyes because each dyes have its own characteristics.

Due to high cost and low effectiveness of the wastewater treatment method, adsorbent from agricultural waste have been chosen as one of the initiative to treat the wastewater as well as to reduce cost.

The increasing number of agricultural waste nowadays leads to the environmental problem. This is because agricultural industry produces many of waste and does not have proper initiative to figure out how to manage the agricultural waste properly without causing any environmental problem (Isoda *et al.*, 2014). Banana tree is one the most contributed to agricultural waste because all parts of banana tree will become agricultural waste at the plantation field after it being harvested (Mas Haris & Sathasivam 2009). In fact, the banana tree waste will cause pollution from plant degradation which leads to serious concern (Monacha *et al.*, 2001). The abundance of banana tree wastes is suitable to use as agricultural adsorbent to treat the wastewater. In addition, it will reduce the agricultural waste problems that happened nowadays.

1.3 Objectives

The objectives of this study are:

1. To produce activated carbon from the agricultural waste (banana pseudostem fibers).
2. To investigate the potential of banana pseudostem fibers (raw material and activated carbon) as the adsorbent.
3. To analyze the effect of contact time, adsorbent dosage, concentration of the solution and the pH of the solution to remove the methyl red from the wastewater using banana pseudostem fibers (raw material and activated carbon).

1.4 Significance of Study

The results of this study were expected to help to minimize the environmental impact caused by the discharging of the effluent from the industries using the adsorbent from the agricultural waste and also assist to minimize the cost of water treatment. This study helps to decrease the agricultural waste at the plantation. This study can also be used as references to produce new agricultural waste adsorbent for treat the wastewater that discharge from textiles industry. In addition, this study gives the value added to the banana pseudostem fibers by producing the activated carbon from banana pseudostem fibers as adsorbent to remove the methyl red dye from the wastewater.

CHAPTER 2

LITERATURE REVIEW

2.1 Dye

Dye is a coloring substance that adds color or changing color of something such as textiles. The dye is generally applied in an aqueous solution, and may require a mordant to improve the fastness of the dye. Dyes can be classified as follows, anionic: direct, acid and reactive dyes; cationic: basic dyes; non-ionic: disperse dyes (Egwuonwu, 2013). Synthetic dyes are widely utilized for coloring and printing as a part of assortment of commercial enterprises (Sandeep, 2010). Synthetic dyes are man-made which is made from synthetic resources such as petroleum by-products and earth minerals.

Dyes are classified based on their solubility and chemical properties. Dyes are widely used in various industries, such as textiles, leather, plastic, paper and cosmetics, for coloring their final products. Presently, it was estimated about 10,000 of different synthetics dyes and pigments exist and over 7×10^5 are produced annually worldwide (Grag *et al.*, 2004). The release of wastewater may eventually affect humans through the food chain because the wastewater that contained dyes from industry may produce an eco-toxic hazard and introduce potential danger of bioaccumulation (Muthuraman & Teng 2009). Many types of diseases can affect to human that cause by the dyes such as allergic dermatitis and skin irritation (Mohammed *et al.*, 2014). Some of them have been reported to be carcinogenic and mutagenic for aquatic organism and can harm the aquatic life (Lorenc-Grabowska & Gryglewicz 2007).

2.1.1 Methyl red

Methyl red is one of the azo dye which is often used in paper printing and textile dyeing (Lachheb *et al.*, 2002). Azo dye, a large class of synthetic organic dyes that contain nitrogen as the azo group $-N=N-$ as part of their molecular structures (Figure2.1).

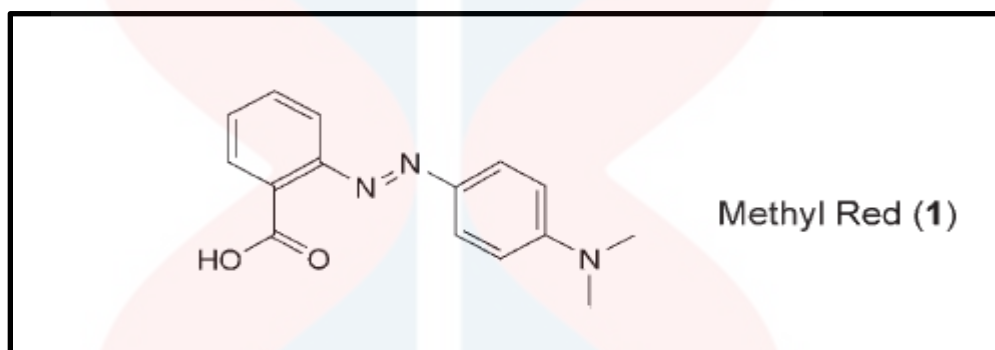


Figure 2.1: Methyl Red (MR) dye structure (Tarawou *et al.*, 2007).

The most easily applied azo dyes are those designated as direct as they contain chemical substituents that make them soluble in water, and they are absorbed from solution by cotton. Tenacity of azo dyes can cause pollution to the environment especially water. Methyl red can cause irritation of the eyes, skin and digestive tract if inhaled or swallowed. In fact, methyl red is mutagenic under aerobic conditions: it undergoes biotransformation into 2-aminobenzoic acid and N, N-dimethyl-4-aminophenyl (Jadhav *et al.*, 2008). Dyes in wastewater are difficult to remove because they are stable to light, heat and oxidizing agents. In short, they are not easily degradable (Mas Haris & Sathasivam 2009).

2.2 Wastewater

Wastewater is any water that has been affected in quality by anthropogenic influence. There are many characteristics of wastewater which can be seen by naked eye. One of the characteristic of wastewater is color where it is easy to detect because the pure water is colorless (Abood Hassan & Abdulhussien 2015). Nowadays, the existence of hazardous and toxic pollutants in industrial wastewater is one of the most serious environmental problems (Tanzim & Zainal Abedin 2015). In fact, the environmental problems can cause by discharge of dye from the textile industries. It is because the wastewater is difficult to treat by conventional method as most of the dyes are withstand to heat and oxidizing agent (Velmurugan *et al.*, 2011).

Color pollution may cause potential toxicity and turbidity problems, thus contributing significantly to the pollution of aquatic ecosystems (Abood Hassan & Abdulhussien 2015). Wastewater discharged from textile industry characterized by high chemical oxygen demand (COD), low biodegradability, high salt content and is the source aesthetic pollution related to color (Abdullah, 2010). Nonetheless, in term of coloring effluent textile industry is the biggest industry releasing coloring effluent, so it is imperative to studies treatment handle that is effective to decrease the color in the pro fluent keeping in mind the end goal to guarantee our water are safe for future generation (Abdullah, 2010).

2.2.1 Wastewater treatment

There are many types of wastewater treatment that can be implemented to treat the wastewater such as biological, physical and chemical treatment. The conventional biological treatment process is incapable to remove the dyes effectively. The wastewater usually treated by either physical or chemical processes. The methods of chemical wastewater treatment are coagulants/ flocculants (Shi *et al.*, 2007) which involves addition of substances such as calcium, aluminum, or ferric ions in to the effluent, as such flocculation is induced (Zhou *et al.*, 2008). The physical wastewater treatment includes membrane-filtration process, reverse osmosis, electrolysis and adsorption techniques (Mohammed *et al.*, 2014). There are advantages and disadvantages of physical and chemical method that have been studied from previous research (Table 2.1).

Table 2.1: Existing and emerging processes for dye removal (Foo & Hameed, 2012)

Physical/chemical methods	Method description	Advantages	Disadvantages
Fenton reagents	Oxidation reaction using mainly H_2O_2 Fe (II).	Effective decolourization of both soluble and insoluble dyes.	Sludge generation.
Ozonation	Oxidation reaction using ozone gas.	Application in gaseous state: no alteration of volume.	Short half-life (20minutes).
Photochemical	Oxidation reaction using mainly	No sludge production.	Formation of by-products.

	H ₂ O – UV		
NaCl	Oxidation reaction using Cl ⁺ to attack amino acid.	Initial and acceleration of azo bond cleavage.	Release of aromatic amines.
Electrochemical destruction	Oxidation reaction using electricity.	Breakdown compounds are non-hazardous.	High cost of electricity.
Activated carbon	Dye removal by adsorption.	Good removal of a wide variety of dyes.	Regeneration difficulties.
Membrane filtration	Physical separation.	Removal of all type of dyes.	Concentrated sludge production.
Ion exchange	Ion exchange resin.	Regeneration: no adsorbent loss.	Not effective for all dyes.
Electrokinetic coagulation	Addition of ferrous sulphate and ferric chloride.	Economically feasible.	High sludge production.

However, these processes were highly cost and could not be effectively used to treat the wide range of wastewater (Grag *et al.*, 2004). For this situation, the adsorption procedure is picked in light of the fact that it is one of the viable techniques for removal dyes from the waste effluent (Velmurugan *et al.*, 2011).

2.3 Adsorption

Adsorption is a surface-based process where an ultrathin layer of gases or liquid forms on the surface of another substances. Adsorption process is widely used to treat the wastewater such as the removal of dye in the wastewater due to their efficiency in the removal of organic and mineral pollutant and for economic consideration (Prasanna *et al.*, 2014). Activated carbon had been widely used as efficient adsorbent in adsorption process to remove the dye effluent in textiles industries (Karthik *et al.*, 2014). The toxicity of the wastewater or removes non-safe organic material from industrial effluents had been decrease through adsorption by adsorbents (Malik, 2004). The process of adsorption has an edge over the other methods due to its sludge free clean operation and completely removed dyes, even from the diluted solution (Velmurugan, *et al.*, 2011).

2.4 Activated carbon

Activated carbons are chemically stable materials and are widely considered as suitable adsorbent for on-site or off-site treatment of polluted groundwater (Thiruvengkatachari *et al.*, 2008). Activated carbon is the most widely used adsorbent as a way to treat and purify industrial wastewater.

Activated carbon, known as activated charcoal, has a large surface area per unit volume and low-volume pores where adsorptions take place (Isoda *et al.*, 2014). Although the activated carbon which is charcoals is available for various adsorption processes, each activated carbon only suitable for sort of specificity which make it useful for a definite purpose only. Several ways can be used to modify the surface chemistry of the activated carbon, like zeolite, activated carbon exhibit shape selectivity (Rahman *et al.*, 2006). Surface chemistry of activated carbon refers to chemically bonded element of

activated carbon which can originate in the starting materials or incorporate during activation or subsequent chemical treatment (Rahman *et al.*, 2006).

The scarcity of activated carbon may cause the activated carbon to become expensive, which leads to the urgent need to explore carbon materials from various types of agricultural waste, which cost significantly less than the commercially available activated carbon, to replace the commercial activated carbon (Madhu *et al.*, 2014). The preparation process of activated carbon from agricultural waste is simple and eco-friendly compared to commercial activated carbon.

2.5 Banana pseudostem fibers

The second most widely cultivated fruit in Malaysia is banana (Rahayu Yet & Abdul Rahim 2014). It is covering about 26,000 ha, with a total production of 530,000 metric tons which means there are many waste produce from whole banana tree such as banana trunk, leaf and stem (Mak Chai *et al.*, 2004). Banana trees are also easy to plant because they do not need water to make it grow. Most of cultivated banana trees come from *Musa* family which is called as *Musa Accuminata* scientifically. The matured banana pseudostem becomes agricultural waste or left to decompose slowly at the plantation field because people only harvest the banana fruit as the food sources (Figure 2.2).

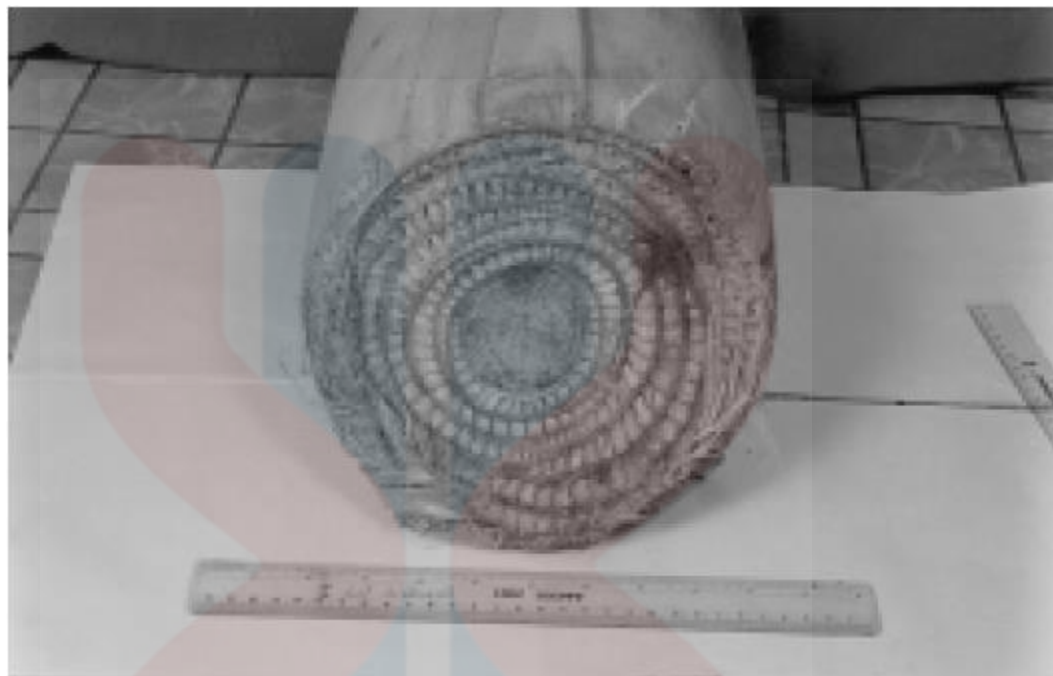


Figure 2.2: Photograph of banana pseudostem fibers (Manocha *et al.*, 2001).

In order to reduce the agricultural waste from banana pseudostem, it can be used as an ideal alternative to the current expensive methods of removing methyl red from the wastewater (Arunachalam & Annadurai 2011). The banana pseudostem fibers are following the characteristic of the most appropriate natural adsorbent which is lowest impact it has on environmental balance, environmentally-friendly and low-cost material originated from agricultural waste and residues (Salit, 2014). In fact, it is biodegradable and non-toxic which is good for reduced environmental pollution. It was reported by Rahim, (2009) that the banana pseudostem fibers can be used as an enzyme immobilization matrix. Therefore, it could be possible to utilize it as well in a different way of application such as for removal of azo dyes (Rahayu Yet & Abdul Rahim 2014).

CHAPTER 3
MATERIAL AND METHOD

3.1 Materials

Chemical	Purity
Methyl red dye	
Sodium Hydroxide (NaOH), 0.1 M	43 %
Hydrochloric Acid (HCl), 0.1 M	38 %

3.2 Apparatus

Instrument	Brand
250 ml conical flask	Schott Duran
150 ml volumetric flask	
Beaker 1000 ml	Schott Duran
Pipette micrometer 100 ml	Watson Nexty
pH meter	Hanna
Measuring cylinder 10 ml	Schott Duran
Grinder	Elba
Sieves	Impact
Weight balance	
Aluminium foils	
Glove	

Filter paper	
Crucible	
Furnace	Hanna
Quartz cuvettes	UV transparent
UV-VIS Spectrophotometer	UV-2600

3.3 Preparation of raw materials

Banana pseudostem fibers were obtained from local farm around the area of Jeli, Kelantan. The banana pseudostem fibers were cut into small pieces and then washed using distilled water to clean the dirt. After that, it will be dried in oven at 65 °C for 24 hours (Rahayu Yet & Abdul Rahim 2014). Then, it will be ground using grinder and sieved through 300 micron sieves to get fine powder (Mas Haris & Sathasivam 2009). The powder and the remaining of banana pseudostem fibers will be kept in the different air-tight container for further use.

3.4 Preparation of activated carbon of banana pseudostem fibers

Banana pseudostem fibers raw materials powder sample were stirred in 100ml of 0.1 M sodium hydroxide, (NaOH) for one hour at room temperature then filtered. The samples will be put in the furnace at 55°C for 24 hours (Rahayu Yet & Abdul Rahim 2014). The sample then were washed using distilled water to neutral pH. Filter paper and vacuum pump was used when washing the sample. The pH was measured using pH meter until the washing solution reached at pH 7. The activated carbon produced from

banana pseudostem fibers was stored in air-tight containers for further adsorption studies.

3.5 Preparation of methyl red solution

For this study, methyl red was used as adsorbate. A stock solution of methyl red was prepared by dissolving 0.100g of dye in 1 L of distilled water to get the concentration of 100mg/L. The prepared stock solution was then wrapped with aluminium foil and stored in the dark to prevent exposure to direct light (Mas Haris & Sathasivam 2009). The dilutions were prepared by diluting the methyl red dye stock solution with distilled water. 10 ml of methyl red dye solution were taken from the stock solution using micropipette into the volumetric flask. Then, the volumetric flask was filled up to the 100 ml mark with distilled water. After that, the solution in the volumetric flask was poured into beaker and the solution was diluted to get the required concentration which is 2, 4, 6, 7, 8 and 10 mg/L. For the concentration, 2 mg/L were pipette using micropipette and put into volumetric flask. The distilled water was poured into the volumetric flask until it reaches to 100 ml marks. The solution was shaken and the solution was put into cuvette using dropper. The reading was analyzed using UV-VIS Spectrophotometer at 463 nm wavelength, λ . The steps were repeated to get the required concentration and the results from the reading were used to perform calibration curve. However, the calibration curve from figure 4.1 did not reflect on the result due to experimental error in the beginning of the experiment.

3.6 Preparation of blank

The blank preparation was set up as a control for this experiment. This preparation was carried out by setting up a set of 250 ml conical flask with 100mg/L

concentration of 50ml methyl red solution. The pH was adjusted at pH 3 with 0.1M HCl or 0.1M NaOH and the pH solution was measured by pH meter. The solution was analyzed using UV-VIS Spectrophotometer. The data were recorded for further use.

3.7 Batch of optimization study

The effects of contact times, adsorbent dosage, concentration and pH were studied. The initial concentration was 100mg/L for all experiment. For the purpose of optimizing, the amount of the adsorbent was performed by adding different weight of adsorbent dosage in the range of 0.1 - 0.3g into 250ml conical flask with 100mg/L concentration of 50ml methyl red solution. The different range of concentrations of the dye solutions between 20mg/L – 100mg/L were used in this study. The contact times to treat the serial dilution were studied by varying the time between 30 – 120 minutes with constant speed of the stirrer. The pH values were in the range of 3 – 9 to conduct the study. All the optimization experiment was conducted at ambient temperature. The speed of stirrer was constant at 150 rpm for 120 minutes using mechanical shaker which was sufficient to reach equilibrium (Mas Haris & Sathasivam 2009).

3.7.1 Effect of contact time

The experiment was carried out to study about the effect of time on efficient removal of dye from methyl red solution by setting up 5 set of 250 ml conical flask with 100mg/L concentration of 50ml methyl red solution. The pH of the solution was adjusted at pH 3 by adding appropriate amount of either 0.1M HCl or 0.1M NaOH and the pH will be measured by using pH meter (Mas Haris & Sathasivam 2009). 0.2g of banana pseudostem fibers adsorbent with 300 µm of the size particles were added into

the solution. The effects of contact time were studied by varying the contact time (0, 30, 60, 90 and 120 minutes). The solutions were stirred at constant speed of 150 rpm at ambient temperature for 120 minutes (Mas Haris & Sathasivam 2009). The solutions then were filtered using filter paper and were analyzed using UV-VIS Spectrophotometer at 463 nm wavelength, λ .

3.7.2 Effect of adsorbent dosage

The effect of adsorbent dosage was investigated on the adsorption rate of dye by setting up 4 set of 250ml conical flask with 100mg/L concentration of 50ml methyl red solution. The pH of the solution was adjusted at pH 3 by adding appropriate amount of either 0.1M HCl or 0.1M NaOH and the pH was measured by using pH meter (Mas Haris & Sathasivam 2009). Different weights of adsorbent (0g, 0.1g, 0.2g and 0.3g) with 300 μm of particles size were added into 100mg/L concentration of 50ml methyl red solution. The solutions were stirred at constant speed of 150 rpm at ambient temperature for 120 minutes. The solutions then were filtered using filter paper and were analyzed using UV-VIS Spectrophotometer at 463 nm wavelength, λ .

3.7.3 Effect of concentration of the solution

The effect of concentration of banana pseudostem fibers sample was studied by setting up 5 set of 250ml conical flask with 100mg/L concentration of 50ml methyl red solution. The pH of the solution was adjusted at pH 3 by adding appropriate amount of either 0.1M HCl or 0.1M NaOH and the pH was measured by using pH meter. 0.2g of banana pseudostem fibers adsorbent with 300 μm of the size particles were added into the different concentration (20, 40, 60, 80 and 100 mg/L) of 50ml methyl red solution.

The solution was stirred at constant speed of 150 rpm at ambient temperature for 120 minutes. The solution then was filtered using filter paper and was analyzed using UV-VIS Spectrophotometer at 463 nm wavelength, λ .

3.7.4 Effect of pH

The effect of pH on the amount of colour removal was analyzed by setting up 4 set of 250ml conical flask with 100mg/L concentration of 50ml methyl red solution. The pH of the solution was adjusted at pH 3, 6, 7 and 9 by adding appropriate amount of either 0.1M HCl or 0.1M NaOH and the pH was measured by using pH meter. 0.2g of the adsorbent weight with 300 μm particles size was added into the 100mg/L concentration of 50ml methyl red solution. The solution was stirred at constant speed of 150 rpm at ambient temperature for 120 minutes. The solution then was filtered using filter paper and was analyzed using UV-VIS Spectrophotometer at 463 nm wavelength, λ .

3.8 Analysis section

All samples were analyzed using UV-VIS Spectrophotometer at 463 nm wavelength, λ . Then the serial dilutions were calculated from calibration curve as well as the samples. The percentages, %, of dye removal were calculated as the following equation:

$$E = \frac{C_0 - C}{C_0} \times 100$$

Where, C_0 is the initial concentration, C , is the final concentration (Rahayu Yet & Abdul Rahim 2014).

CHAPTER 4

RESULT AND DISCUSSION

4.1 Calibration Curve

The calibration plot for Methyl Red Dye solution analyzes using UV-VIS Spectrophotometer at 463nm wavelength, λ was obtained as shown in Figure 4.1.

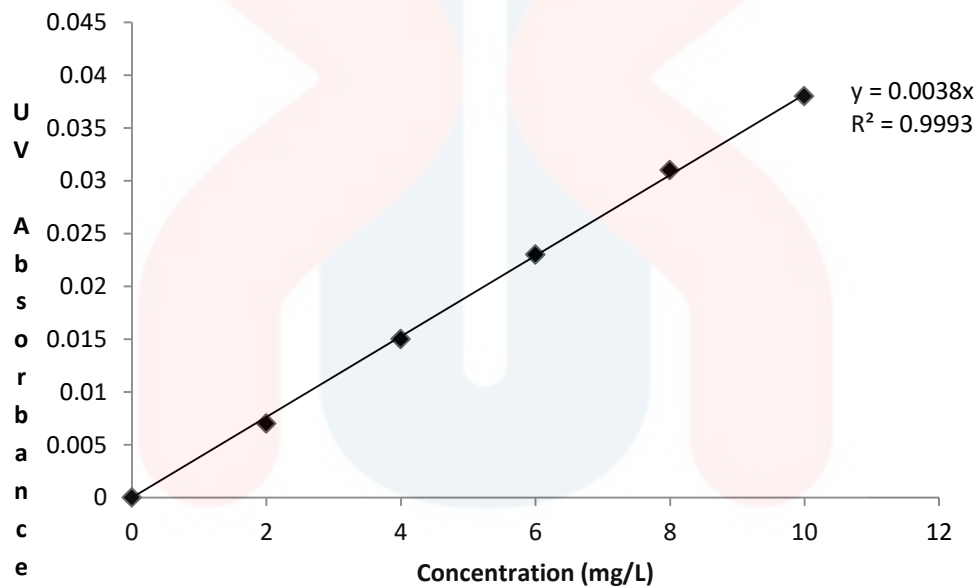


Figure 4.1: Calibration Curve for Methyl Red Solution

Calibration curve is a common and important step in analytical methods that helps to understand of how to set up calibration experiments and how to analyze the result obtained from the UV-VIS Spectrophotometer (Alexander *et al.*, 2011). The calibration curve showed that how meticulous the person when used the apparatus and instruments. The correlation coefficient, R^2 is one of the statistics commonly used in analytical measurement. The correlation coefficient, R^2 also shows the accuracy of the

result and confirmed the functionality of the instrument (Adeeyinwo *et al.*, 2013). The value of R^2 is 0.9993 as shown in figure 4.1 showed that the dilution for the 100mg/L concentration of methyl red solution are accurate. The value of $y = 0.0038x$ were used to make calculation for the result that obtained from the experiment that have been conducted.

4.2 Effect of contact time on the percentage of methyl red dye removal using banana pseudostem fibers adsorbent (raw)

The relationship between the effects of contact time on the percentage of methyl red dye removal by using raw banana pseudostem fibers adsorbent was shown in Figure 4.2.

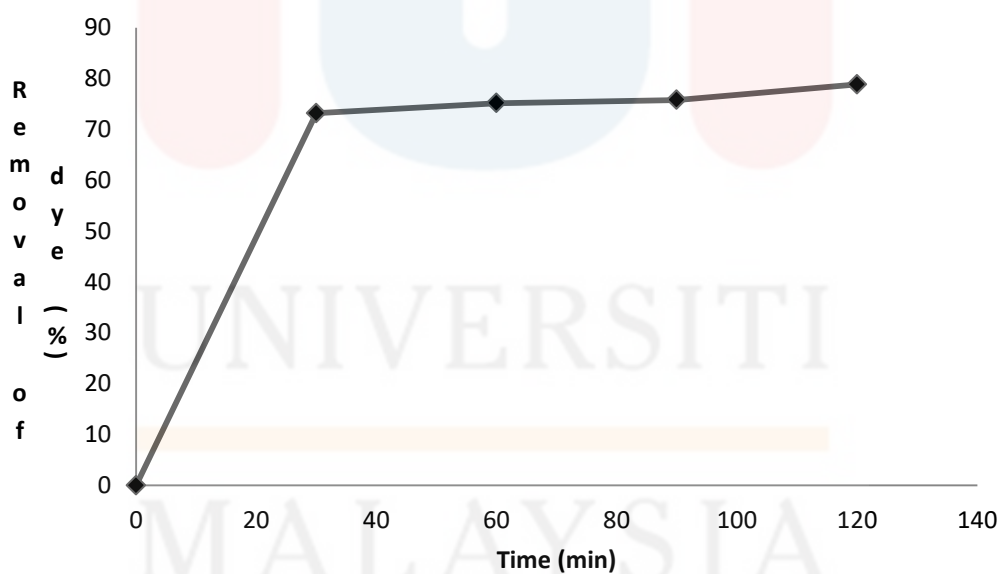


Figure 4.2: The Effect of Contact Time on The Percentage of Methyl Red Dye Removal using banana pseudostem fibers adsorbent (raw)

This study was performed at the range of 30 minutes – 120 minutes of contact time. The graph shows that the percentage of methyl red dye removal slightly increases along the contact time in the range of 30 minutes to 120 minutes.

The highest percentage of methyl red dye removal was take place at 120 minutes contact time which is 78.83% removal. The lowest percentage of methyl red dye removal was take place at 30 minutes contact time which is 73.15% removal. It was showed that the adsorption process is highly depend on the contact time and was observed that the percent removal of dye increase gradually with the contact time same as the graph shown at figure 4.2 (Sivakumar *et al.*, 2012). Based on this study, it shown that the most optimum time to remove the methyl red dye was at 120 minutes of contact time.

4.3 Effect of contact time on the percentage of methyl red dye removal using banana pseudostem fibers adsorbent (activated carbon)

The relationship between the effects of contact time on the percentage of methyl red dye removal by using activated carbon banana pseudostem fibers adsorbent was shown in Figure 4.2.

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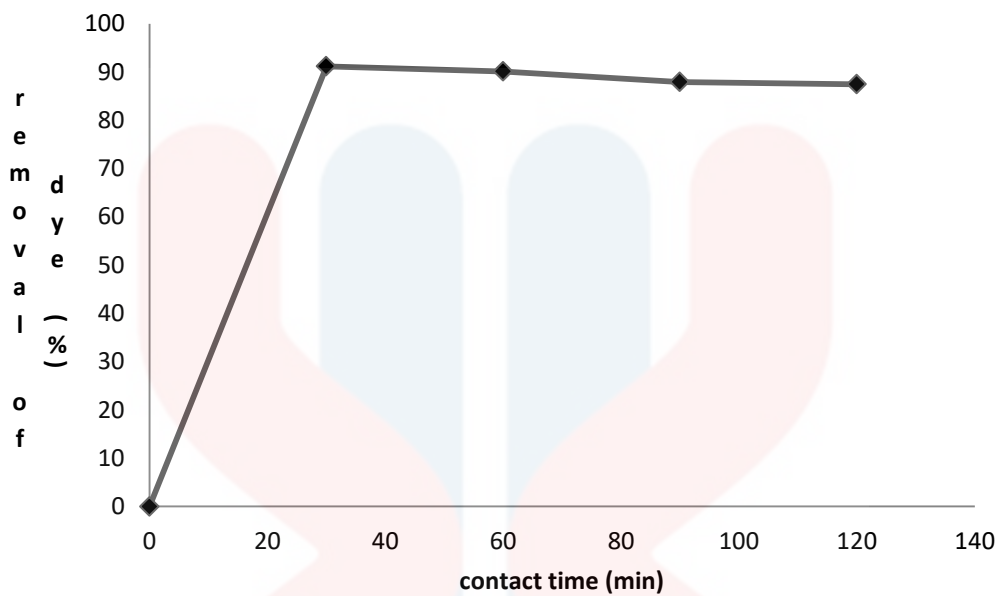


Figure 4.3: The Effect of Contact Time on The Percentage of Methyl Red Dye Removal using banana pseudostem fibers adsorbent (activated carbon)

This study was conducted at the range of 30 minutes – 120 minutes of contact time. The result shows that the graph increase for the first 30 minutes but then the graph slightly decrease after 30 minutes of contact time. Based on the result, the adsorption process highly occurs at first 30 minutes and the adsorption process start to slow after 30 minutes of contact time.

In this study, the highest percentage of methyl red dye removal was 91.17% at 30 minutes of contact time which is the optimum condition for removal of methyl red dye due to the highly negativity charged surface of the activated carbon for adsorption (Shanti *et al.*, 2012). The percentage removal of methyl red dye at 120 minutes of contact time was the lowest compared to others which is 87.47% removal. So 120 minutes of contact time was not suitable to remove the methyl red dye.

At 120 minutes, the percentage removal are lower due to accumulation of dye molecules with the increase of contact time make it almost impossible to diffuse deeper into the adsorbent structure at highest energy site. The accumulation deny the influence of contact time as the mesopores get filled up and start offering resistance to diffusion of accumulated dye molecule in the adsorbent (Tripathi *et al.*, 2009). Based on this study, the optimum condition to remove the methyl red dye was at 30 minutes of contact time. This was proved by the fact that a large number of vacant surface sites are available for the adsorption during the initial stages (Gullipalli *et al.*, 2011).

4.4 Effect of adsorbent dosage on the percentage of methyl red dye removal using banana pseudostem fibers adsorbent (raw)

The result for the effect of adsorbent dosage on the percentage of methyl red dye removal was shown at Figure 4.4 below.

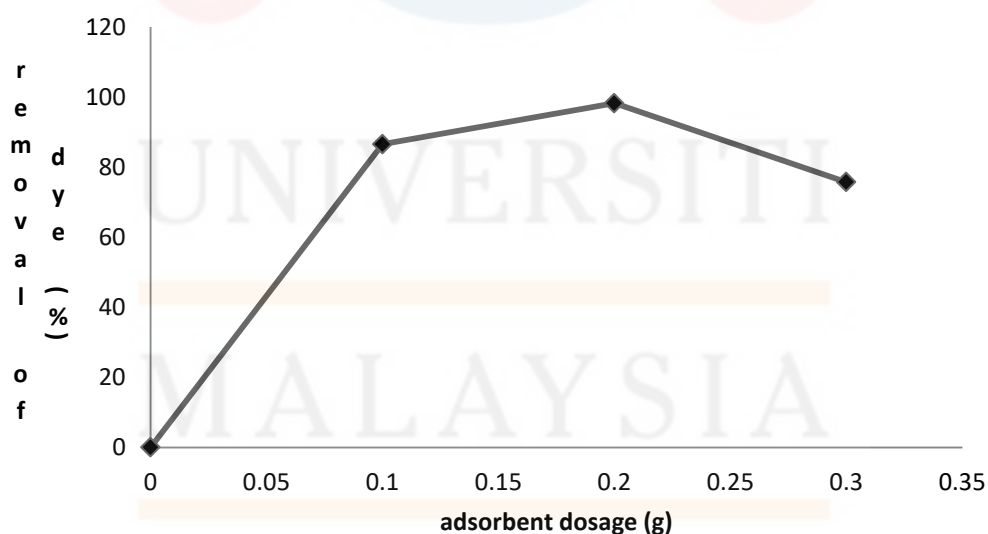


Figure 4.4: The Effect of Adsorbent Dosage on The Percentage of Methyl Red Dye Removal using banana pseudostem fibers adsorbent (raw)

This study was conducted by using adsorbent dosage range 0.1g -0.3g. The result shows that the graph of the percentage of the methyl red dye removal increase at 0.1g and 0.2g of adsorbent dosage while the graph of percentage of methyl red dye removal start to decrease at 0.3g of adsorbent dosage. It is because the presence of available sites on the adsorbent caused the rapid adsorption for adsorbent dosage 0.1g and 0.2g.

In this study shows that the optimum condition for removal of methyl red dye using 0.2g of raw banana pseudostem fibers as adsorbent. This is because the percentage of methyl red dye removal for 0.2 g of adsorbent dosage is the highest which is 98.20% removal. The percentage of methyl red dye removal start to decrease when used 0.3g of raw pseudostem fibers as adsorbent which is 75.67% removal due to the binding site were used up before all the dye were adsorb. It is because the active site of the adsorbent has the lower capacity to adsorb the methyl red dye (Ogunsile *et al.*, 2014). So 0.2g of adsorbent dosage was the most suitable condition to remove the methyl red dye using raw banana pseudostem fibers.

4.5 Effect of adsorbent dosage on the percentage of methyl red dye removal using banana pseudostem fibers adsorbent (activated carbon)

Figure 4.5 shows that the effect of adsorbent dosage on the percentage of methyl red dye removal using activated carbon of banana pseudostem fibers as adsorbent.

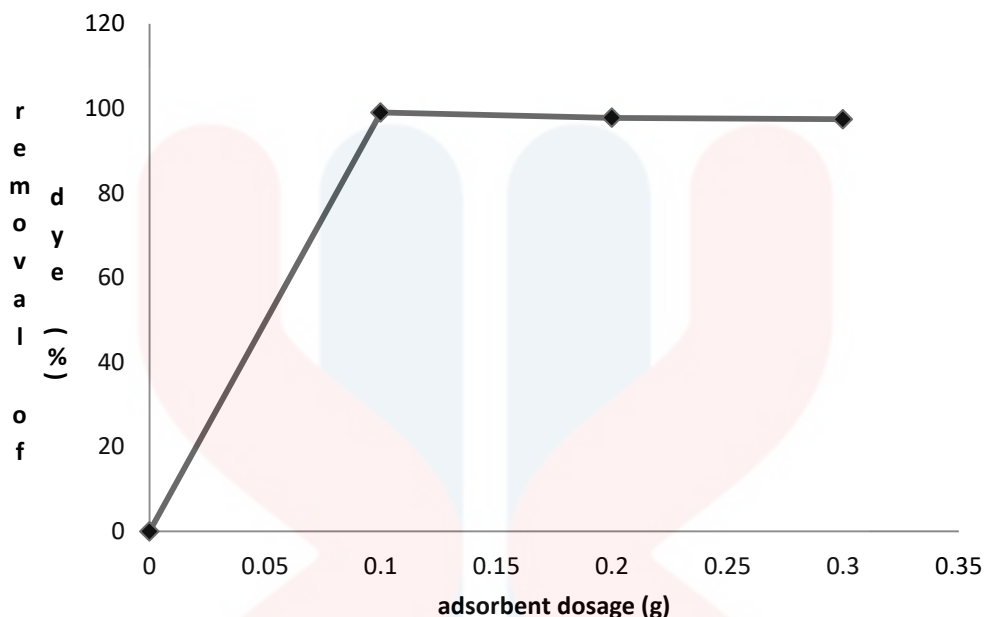


Figure 4.5: The Effect of Adsorbent Dosage on The Percentage of Methyl Red Dye Removal using banana pseudostem fibers adsorbent (activated carbon)

The study was conducted by using adsorbent dosage at the range of 0.1g – 0.3g. Figure 4.5 shows that the graph was increases to 0.1 g of adsorbent dosage and then slightly decline from 0.2 g to 0.3 g of adsorbent dosage. The decline of the graph may be attributed by the decreasing of sorbents surface area and the unavailability of more adsorption site resulting from more dose of adsorbent (Egwounwu, 2013). The decline also shows that the adsorption process had reached the equilibrium state.

The result of the effect of adsorbent dosage on the percentage of the methyl red removal using activated carbon of banana pseudostem fibers shows that the highest percentage removal of methyl red dye was at 0.1g of adsorbent dosage. Based on this study, the percentage of methyl red removal at 0.1g of adsorbent was 99.01% removal which is the optimum condition for removal of methyl red dye. This implied that using

lower adsorbent dosage will be more advantages than the higher one due to the presence of available sites of the adsorbent (Oqunsile *et al.*, 2014).

The percentage of methyl red dye removal was the lowest at 0.3g of adsorbent dosage which is 97.39%. This is because, on reaching the equilibrium the percentage of removal will decrease with increasing the amount of adsorbent dosage. In fact, increase of adsorbent dosage leads to increase iron content in active site so the iron may block the adsorption site of the activated carbon (Das & Mondal, 2011). The percentage of methyl red dye removal also decreases due to the limited availability of the active site.

4.6 Effect of concentration on the percentage of methyl red dye removal using banana pseudostem fibers adsorbent (raw)

Figure 4.6 shows the effect of the concentration on the percentage of methyl red dye removal using raw banana pseudostem fibers adsorbent.

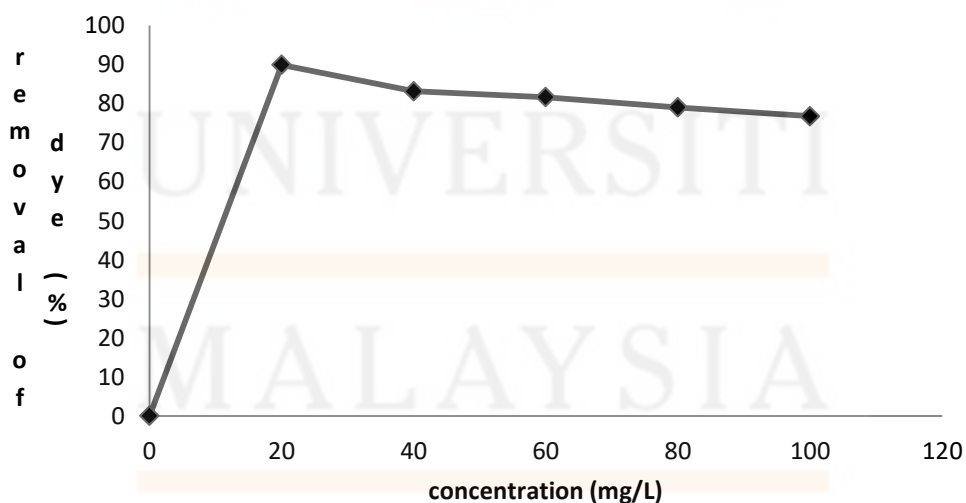


Figure 4.6: The Effect of Concentration on The Percentage of Methyl Red Dye Removal using banana pseudostem fibers adsorbent (raw)

The study was performed by using the range of 20 mg/L – 100 mg/L concentration of methyl red dye solution. The graph substantially increases at the 20 mg/L concentration of methyl red dye solution and then moderately decreases until 100 mg/L concentration of methyl red dye solution. Figure 4.6 shows that the highest percentage of removal take place at 20 mg/L concentration of methyl red dye solution which is 89.91% removal. The lowest percentage of the removal was 76.76% removal which is occurs at 100 mg/L. It was found that an increasing in the dye concentration had caused the decreasing in the percentage of removal (Abood Hassan & Abdulhussein, 2015). So at 100 mg/L concentration was not suitable for the removal of methyl red dye.

Based on the study, 20 mg/L concentration was the most optimum condition to remove methyl red dye using raw banana pseudostem fibers as adsorbent. This is because, the higher the concentration of methyl red dye solution, the higher the interaction between methyl red dye solution and the raw banana pseudostem fibers adsorbent. So that, the mass transfers resistant between the methyl red dye solution and the raw banana pseudostem fibers adsorbent were lower (Maarof *et al.*, 2003).

4.7 Effect of concentration on the percentage of methyl red dye removal using banana pseudostem fibers adsorbent (activated carbon)

Figure 4.7 shows the relationship between the effect concentration and the percentage of methyl red dye removal by using activated carbon of banana pseudostem fibers as adsorbent.

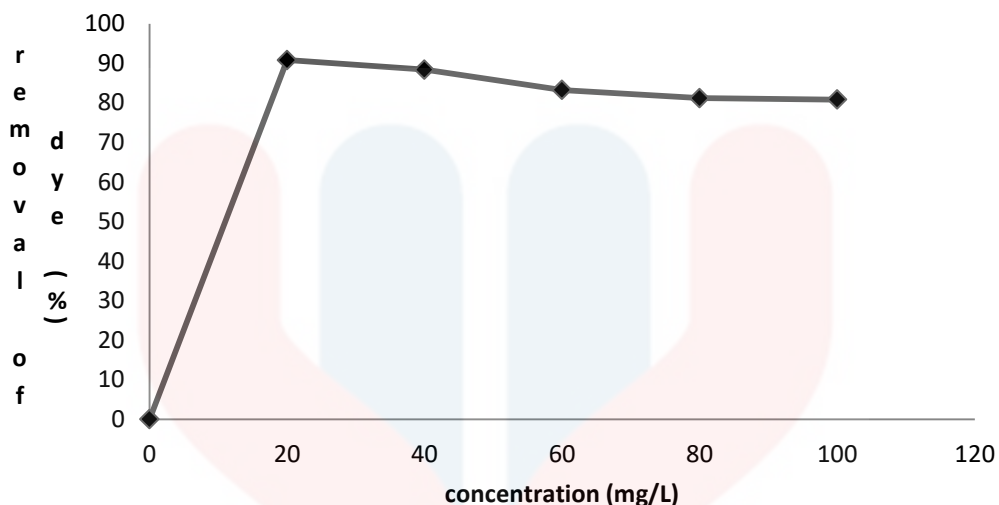


Figure 4.7: The Effect of Concentration on The Percentage of Methyl Red Dye Removal using banana pseudostem fibers adsorbent (activated carbon)

The study was conducted using the range of 20 mg/L – 100 mg/L concentration of methyl red dye solution. The result shows the graph ascending to 20 mg/L concentration of the methyl red dye solution and then slightly decreases in the range of 20 mg/L – 100 mg/L concentration of methyl red dye solution. It shows that the percentage of removal was decreases with increasing of initial concentration. Figure 4.7 also shows that the graph start to be constant at the range of 80 mg/L to 100mg/L due to reach the equilibrium (Raxena & Sharma, 2016).

The highest percentage of removal of methyl red dye using activated carbon of banana pseudostem fibers as adsorbent was at 20mg/L of concentration which is 90.81% removal. The lowest percentage of removal of methyl red dye using activated carbon of banana pseudostem fibers as adsorbent was at 100 mg/L which is 80.81%. Based on this study, the optimum condition to remove the methyl red dye was at 20 mg/L

concentration of methyl red dye solution. This is because, at low concentration most of methyl red dye in the sample solution might be contact with active sites of the adsorbent and when the concentration increase all the methyl red will not available to contact with the active surface due to active site already filled up (Rahman *et al.*, 2012).

4.8 Effect of pH on the percentage of methyl red dye removal using banana pseudostem fibers adsorbent (raw)

Figure 4.8 shows the effect of pH on the percentage of methyl red dye removal using raw banana pseudostem fibers adsorbent.

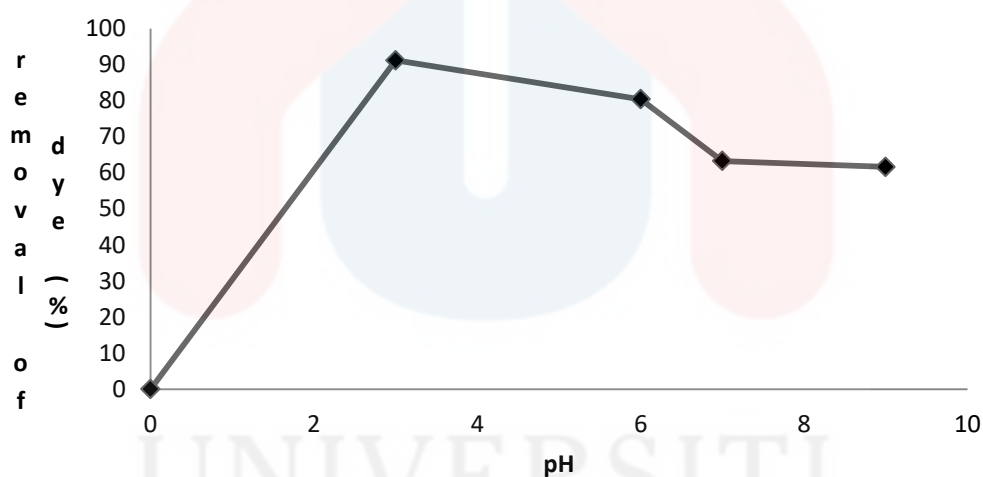


Figure 4.8: The Effect of pH on The Percentage of Methyl Red Dye Removal using banana pseudostem fibers adsorbent (raw)

The study was performed over a pH range 3 – 9 to obtain the optimum pH for the percentage of methyl red dye removal. The result shows that the graph dramatically increases to the initial pH 3 and then decrease over the pH range 6 – 7. The graph shown that the highest percentage of the removal of methyl red dye using the raw banana

pseudostem fibers as adsorbent occurs at pH 3 which is 91.17% removal. The lowest percentage of the removal of methyl red dye was occurs at pH 9 which is 61.62% removal.

The graph shows that it becomes constant at the pH range 7 – 9. It is because the adsorption process at the pH range 7 – 9 already achieved equilibrium state (Gecgel *et al.*, 2013). The graph showed from figure 4.8 shows that the alkaline have low adsorption process. The efficiency of sorption is dependent on the pH of the solution because variation of pH leads to the variation in the degree of ionization and the surface properties of the sorbent (Gupta *et al.*, 2007). Based on this study, pH 3 was the optimum condition to remove the methyl red dye. It proves that the uptakes were much higher in acidic solution than those in neutral and alkaline conditions.

4.9 Effect of pH on the percentage of methyl red dye removal using banana pseudostem fibers adsorbent (activated carbon)

Figure 4.9 shows the relationship between the effect of pH on the percentage of methyl red dye removal by using activated carbon of banana pseudostem fibers as adsorbent.

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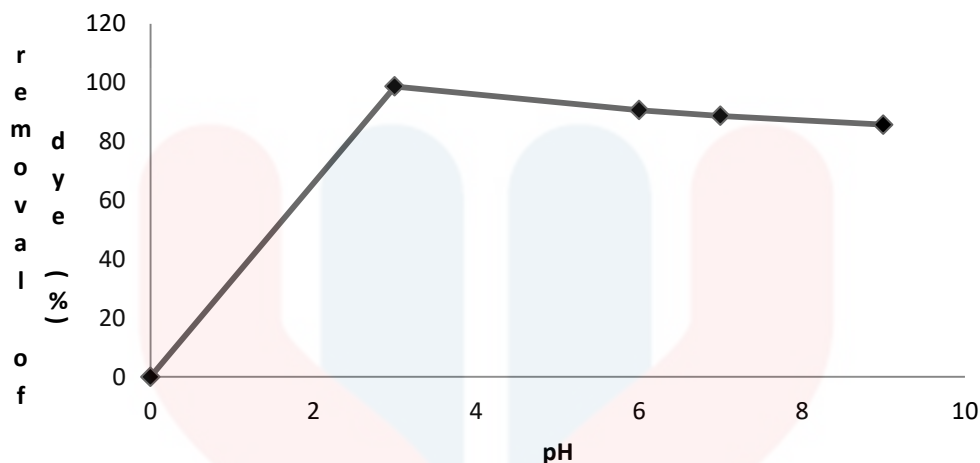


Figure 4.9: The Effect of pH on The Percentage of Methyl Red Dye Removal using banana pseudostem fibers adsorbent (activated carbon)

The study was performed using different range of pH which is pH 3 – 9. Initial value of pH may enhance or depress the uptake. The graph from the figure 4.9, it proved that the percentage of adsorption decrease when the pH value increase. The graph increases at the initial value of pH 3 but then slightly decreases at the range of pH 6 – 9. The graph slightly decreases start from pH 6 to pH 9 because the adsorption process had become low due to electrostatic repulsion (Khattri & Singh, 2009). Furthermore, the activated carbon takes on a positive charge by adsorbing hydrogen (H^+) ions when immersed in aqueous solution so that, as the pH value increases, the number of positive charge decrease (Malik, 2004).

The highest percentage of removal of methyl red dye was at pH 3 which is 98.65%. The lowest percentage of removal of methyl red dye was at pH 9 which is 85.68% removal. The lower adsorption process in alkaline medium can be attributed to the competition from excess hydroxide ions (OH^-) with the anionic dye molecules for

the adsorption site. Based on this study, the adsorption process by the activated carbon of banana pseudostem fiber was occurs in acidic condition which is pH 3 and it is optimum condition to use for methyl red dye removal.



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CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

As a conclusion, through this study, the potential of the raw materials and activated carbon were identified which is can be effectively used as adsorbent for the removal of Methyl Red dye from wastewater. Based on the percentage removal of methyl red dye from the wastewater between raw material and the activated carbon of banana pseudostem fibers as adsorbent, this study identified that the activated carbon was the most suitable to be use as adsorbent compared to raw materials. The porosity of the activated carbon resulting in increased uptake in the adsorption of methyl red dye.

The effect of various parameters such as contact time, adsorbent dosage, concentration and pH were investigated in this study. The optimum conditions for removal of Methyl Red dye from wastewater by using banana pseudostem fibers were successfully identified that obtained from the highest percentage of the Methyl Red dye removal. From this study, the optimum condition that show the highest percentage of the methyl red dye removal which is 99.01% were at 30 minutes of contact time, 0.1g of the adsorbent dosage, 20 mg/L concentration of methyl red dye solution with pH 3 by using activated carbon produced from banana pseudostem fibers. It concludes that activated carbon produced from banana psuedostem fibers have the highest potential to be used as adsorbent to remove methyl red dye compared to raw material from banana psuedostem fibers.

5.2 Recommendation

For the recommendation, this study can be further improved by using the other types of chemical to enhance the porosity of the activated carbon. The better the porosity of the activated carbon, the higher the adsorption process can take place. The porosity of the activated carbon plays the important role in the adsorption process. Hence, the activated carbon with better porosity can help to treat the wastewater from the textiles industry effectively.

The second recommendation is this study can be further improved by different adsorption isotherms models. There are several of adsorption isotherms models such as Langmuir Isotherm Model and Freundlich Isotherm Model. These adsorption isotherms can help to give better understanding on adsorption process as well as can help to get more accurate result from the adsorption process.

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APPENDIX A

Table A.1: Calibration Curve

Concentration (mg/L)	Absorbance UV
2	0.007
4	0.015
6	0.023
8	0.031
10	0.038

Table A.2: Effect of contact time on the percentage of methyl red dye removal using raw material from banana pseudostem fibers as adsorbent.

Contact time (min)	UV-VIS Spectrophotometer Reading	Removal of Dye (%)
0	0	0
30	0.298	73.15
60	0.276	75.14
90	0.269	75.76
120	0.235	78.83

Table A.4: Effect of adsorbent dosage on the percentage of methyl red dye removal using raw material from banana pseudostem fibers as adsorbent.

Adsorbent dosage (g)	UV-VIS Spectrophotometer Reading	Removal of Dye (%)
0	0	0
0.1	0.15	86.49
0.2	0.02	98.20
0.3	0.27	75.67

Table A.6: Effect of concentration on the percentage of methyl red dye removal using raw material from banana pseudostem fibers as adsorbent.

Concentration (mg/L)	UV-VIS Spectrophotometer Reading	Removal of Dye (%)
0	0	0
20	0.258	76.76
40	0.233	79.00
60	0.204	81.62
80	0.187	83.15
100	0.112	89.91

Table A.8: Effect of pH on the percentage of methyl red dye removal using raw material from banana pseudostem fibers as adsorbent.

pH	UV-VIS Spectrophotometer Reading	Removal of Dye (%)
3	0.098	91.17
6	0.218	80.36
7	0.408	63.24
9	0.426	61.62

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APPENDIX B

Table B.1: Effect of contact time on the percentage of methyl red dye removal using activated carbon from banana pseudostem fibers as adsorbent.

Contact time (min)	UV-VIS Spectrophotometer Reading	Removal of Dye (%)
0	0	0
30	0.098	91.17
60	0.110	90.10
90	0.134	87.93
120	0.139	87.47

Table A.5: Effect of adsorbent dosage on the percentage of methyl red dye removal using activated carbon from banana pseudostem fibers as adsorbent.

Adsorbent dosage (g)	UV-VIS Spectrophotometer Reading	Removal of Dye (%)
0	0	0
0.1	0.011	99.01
0.2	0.025	97.74
0.3	0.029	97.39

Table A.7: Effect of concentration on the percentage of methyl red dye removal using activated carbon from banana pseudostem fibers as adsorbent.

Concentration	UV-VIS Spectrophotometer Reading	Removal of Dye (%)
0	0	0
20	0.213	80.81
40	0.209	81.17
60	0.186	83.24
80	0.129	88.38
100	0.102	90.81

Table A.9: Effect of pH on the percentage of methyl red dye removal using activated carbon from banana pseudostem fibers as adsorbent.

pH	UV-VIS Spectrophotometer Reading	Removal of Dye (%)
3	0.015	98.65
6	0.104	90.63
7	0.126	88.65
9	0.159	85.68

APPENDIX C



Figure C.1: Concentration for calibration curve.



Figure C.2: Activated carbon produced from banana pseudostem fibers.



Figure C.2: The solutions were stirred at constant speed of 150 rpm at ambient temperature for 120 minutes.



Figure C.3: The solutions were filtered using filter paper.