

**Dye Waste Removal By Using Untreated Sugarcane Bagasse and Untreated
Coconut Husk : A Comparative Study**

by

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Dissertation submitted in partial fulfilment of

the requirements for the

Bachelor of Engineering (Hons)

(Chemical Engineering)

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CERTIFICATE OF APPROVAL

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Approved by,

(Dr Lemma Dendena Tuffa)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
September 2012

CERTIFICATE OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgement and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

CHE MUHAMMAD BUKHARI BIN CHE MOHD RAZALI

ABSTRACT

Dyes are usually present in trace quantities in the treated effluents of many industries. The effectiveness of adsorption for dye removal from wastewaters has made it an ideal alternative to other expensive treatment methods. This study investigates the potential use of untreated sugarcane bagasse and of untreated coconut husk for the removal of 10ppm methylene blue solution with time. At the end of the experiment, there will be selection on the best waste adsorbents based on the result of dye removal percent. Sugarcane bagasse and coconut husks were prepared at six periods which were 30min, 1 hour, 2 hour, 4 hour, 6 hour and 24 hour. Characterizations of the sugarcane bagasse and coconut husks have been conducted in order to understand the mechanisms involve to absorb the methylene blue. From FTIR spectra analysis, it suggests that adsorption happen through chemical complexation. SEM images proves that adsorption process occur, where it is proved by the obtained before and after the process. Based on the results obtained, it showed that untreated sugarcane bagasse has better adsorption ability, compare to coconut husk, and proves to be the most effective for biomass adsorbents in wastewater treatment industry.

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CHAPTER ONE

1. INTRODUCTION

1.1 BACKGROUND STUDY

Almost every industrial dye process involves a solution of a dye in water, in which the fabrics are dipped or washed. After dying a batch of fabric, it's cheaper to dump the used water – dye effluent – than to clean and re-use the water in the factory. So dye factories across the world are dumping millions of tons of dye effluent into rivers.

Most countries require factories to treat dye effluent before it is dumped. Separating the dye chemicals from the water will result in a dye sludge, and cleaner water. The water, which still contains traces of dye, is dumped into the river, and leaves the problem of what to do with the sludge? Hence, the treatment of dyes in industrial wastewaters poses several problems as the dyes are generally stable to light and oxidation and hence they cannot be treated by conventional methods of aerobic digestion.

Adsorption is one of the methods commonly used to remove dye waste from industrial effluent. It has been studied that adsorption process is a prominent method for removal of organic pollutants practically used by industries. Among the other treatment processes (e.g., chemical precipitation, electrolysis, ion exchange, and etc.), adsorption is found to be highly effective, cheap, and easy to adapt. However, adsorption by using biomass or also referred to adsorption nowadays has become more favorable compared to the conventional adsorption. This is because it is abundant and low cost, and also environmental friendly.

1.2 PROBLEM STATEMENT

Problem Identification

Dye is hazardous from industrial wastewater and can affect the health if they are exceeding the allowable limit. For methylene blue, it causes hemolytic anemia, hyperbilirubinemia, and acute renal failure. This resulted from enteric administration and peritoneal absorption of methylene blue used to check the integrity of a gastrostomy tube site.

Significant of the Project

Techniques presently in existence for removal dye from wastewater are relatively expensive involving either elaborate and costly equipment or high costs of operation with ultimate disposal problems (Cheng-Shlun and Shang- Da, 1994). In view of these reasons, development of a more cost effective remediation process using biological system for removal of dye from wastewater is necessary. The implementation is suitable because it is both environmentally acceptable and cost-effective. Studies on the treatment of effluent containing dye have revealed adsorption to be a highly effective technique for the removal of heavy metal from waste stream, and activated carbon has been widely used as an adsorbent. However, superior activated carbon is expensive, so this project is initiated to produce low-cost absorbent. Biomass such as sugarcane bagasse and coconut husk are commonly available agriculture waste in Malaysia was used in this study because of its abundant availability and low-cost.

1.3 OBJECTIVE AND SCOPE OF STUDY

The objectives of this project are listed as follows:

1. To characterize the physical and chemical properties of sugarcane bagasse and coconut husk
2. To prepare adsorbent at various preparation parameters.
3. To determine the best adsorbent based on dye removal.

The scope for this project will cover from preparation of sugarcane bagasse and coconut husk, characteristic study of sugarcane bagasse and coconut husk samples throughout the preparation stages and determination of the adsorption capacity. The results obtain then will be properly showed and discussed in a report documented throughout the project.

1.4 RELEVANCE OF PROJECT

Sugarcane bagasse and coconut husk, an agricultural waste, have the potential to be used as adsorbent for the removal of dye waste solution from aqueous solutions. Removal of dye waste solution from wastewater to certain levels is important as per to meet discharge requirements that intended to avoid any harmful effects towards the environment. However, existing methods of treating wastewater have several disadvantages and the implementation cost is high.

In this project, sugarcane bagasse and coconut husk are tested due to its easy availability, low cost and laboratory process suitability. These wastes could be used as cheaper alternative for dye waste removal through adsorption process. Thus, these two waste are selected to be discovered based on the ability of dye removal therefore proposed for future ways of waste water treatment.

1.5 FEASIBILITY OF PROJECT

Sugarcane bagasse and coconut husk that are going to be used in this project is easily available in Malaysia. Several adsorption studies will be conducted in a period of one year which should be sufficient to finish the project. All equipments needed in this project are available in the Chemical Engineering Laboratory as well as the chemicals involved. With all the resources provided, this project can be considered as a feasible project within the time frame given.

CHAPTER TWO

2 LITERATURE REVIEW

2.1 Dye Pollution

Colour is the first contaminant to be recognized in water and has to be removed from wastewater before discharging it into water bodies. Most of the industries in Malaysia, textile, paper, printing, leather, food, cosmetics, etc. use dyes to colour their final product. Due to their good solubility, synthetic dyes are common water pollutants and they may frequently be found in trace quantities in industrial wastewater. However, the effluents of dye-bearing wastewater into natural streams and rivers take a severe problem, as dyes produce toxicity to aquatic life and are damaging the nature of the environment. However, wastewater containing dyes is very difficult to treat, since the dyes are unmanageable organic molecules, resistant to aerobic digestion, and are stable to light, heat and oxidizing agents due to their structure and molecular size [3,4].

Most of the used dyes are stable to photodegradation, bio-degradation and oxidizing agents [5]. Currently, several physical or chemical processes are used to treat dye overloaded wastewaters. However, these processes are costly and cannot effectively be used to treat the wide range of dye wastewater. The advantages and disadvantages of some methods of dye removal from wastewaters are given in Table 1.

Physical/chemical methods	Advantages	Disadvantages
Fentons reagent	Effective decolourisation	Sludge generation
Ozonation	No change in effluent volume	Shorthalf life (20 min)
Photochemical	No sludge generation	Formation of byproducts
NaOCl	Initiate azo-bond cleavage	Release of aromatic amines
Cucurbituril	Good sorption capacity for dyes	High cost
Electrochemical	Non-hazardous end products	High cost of electricity
Activated carbon	Highly effective for various dyes	Very expensive
Peat	Good adsorbent	Surface area is low
Silica gel	Effective for basic dyes	Side reactions in effluent
Membrane filtration	Removes all dyes	Concentrated sludge production
Ion exchange	No adsorbent loss	Not effective for all dyes

Table 2.1 Advantages and disadvantages of the methods used for dye removal from industrial effluent[6]

2.2 ADSORPTION

Among the various techniques of dye removal, adsorption is the procedure of choice and gives the best results as it can be used to remove different types of coloring materials [7,8]. The adsorption process provides an attractive alternative especially if the adsorbent is inexpensive and readily available. The use of activated carbon for treatment of colour effluents has been investigated extensively (Molvar 1970; McKay, 1982). Fullers earth and bauxite have also been reported to be successful in removing colour on a laboratory scale (Pearson 1913; Thorton and Moore, 1953). If the adsorption system is designed correctly it will produce a high-quality treated effluent. Most commercial systems currently use activated carbon as adsorbent to remove dyes in wastewater because of its excellent adsorption ability. Adsorption techniques have gained favor in recent years because of their proven efficiency in the removal of pollutants from effluents too stable for chemical process.

Activated carbon (powdered or granular) is the most widely used adsorbent because it has excellent adsorption efficiency for organic compounds, but its use is usually limited due to its high cost. Therefore, a number of low cost and easily available materials, such as waste biomass, are being studied for the removal of different dyes from aqueous solutions at different operating conditions. Forest and agricultural production byproducts have been long considered as potential dye adsorbents. Table 2 shows comparison for adsorption of some dyes on various adsorbents

Adsorbent(s)	Dye(s)	References
Duckweed	Methylene blue	[9]
Sewage Sludge	Basic red 46	[10]
Waste newspaper	Basic blue 9	[11]
Rice husk	Malachite green	[12]
	Acid yellow 36	[13]
	Acid blue	[14]
	Acid orange 10	[15]
Sugarcane bagasse	Congo red	[16]
Coir pith	Basic blue 10	[17]
Straw	Methylene blue	[18]

Table 2.2 : Comparison for adsorption of some dyes on various adsorbents.

2.3 SUGARCANE BAGASSE AND COCONUT HUSK AS ADSORBENT

Sugar cane bagasse is a byproduct of sugarcane industries obtained after the extraction of juice for production of sugar. It is presently used as fuel for boilers or supplied as raw material for the manufacturing of pulp, paper and building boards. Bagasse must be modified physically and chemically to enhance its adsorptive properties towards organic molecules or metal ions, routinely found in water and wastewater. This is effectively accomplished by converting bagasse to an activated carbon. Bagasse is reported as a suitable resource for preparation of activated carbon [19]. Another adsorbent, coconut husk, a byproduct of coconut, is being used for the production of charcoal, fuel and brooms. The investigation on discovering the potential of such a material in removing dyes from solution. According to the Department of Statistical Malaysia (1985), Malaysia produced a total of 120,195,000 coconuts in 1984. The bulk of coconut husk is made up of cellulose and lignin (60%) [1]. the hydroxyl groups in these two polymeric substance provide sites for adsorption of dyes. Preliminary investigation showed that cationic dyes like Astrazone Red GTLN, Astrazone Pink FG, Astrazone Blue BG and methylene blue could be readily adsorbed on the husk. However, neutral dyes like methyl blue orange and phenol red showed no such activity.

2.4 FACTORS AFFECTING ADSORPTION

There are some factors that affect the adsorption process. It involves the contact time, initial concentration, adsorbent mass, pH, and adsorbent particle size.

Particle Size

The percentage adsorption increased with decrease in particle size of sugarcane biomass (BO Opeolu et al., 2010). Horsfall and Spiff (2004), also reported that an increase in surface charge density is an indication of enhanced adsorption sites as a result of greater surface charges, indicating an ion exchange process between the adsorbent and the metal ion.

pH

At high pH, metal ion may be forced to bind to low affinity ligands such as hydroxyl and carboxyl group but at low pH, the binding may occur through high affinity ligands only (Michael Horsfall Jr. et al., 2004). This is an indication that the degree of ionization on the biomass surface is pH dependent (Michael Horsfall Jr. et al., 2004) which later affect the metal adsorption capacity.

Contact Time

BO Opeolu et al., (2010) concluded that optimal adsorption was achieved in 2 h, though adsorption increased with increased contact time. This is inconsistent with the findings of Veli and Alyuz (2007), who reported an optimal time of 5 min when natural clay was used as adsorbent.

Adsorbent Mass

Increases in the amount of biomass resulted in increased percentage adsorption (Mahvi et al. 2005). These results are similar to those reported by Kok et al. (2001), and Nomanbhay and Palanisamy (2005).

Initial Concentration

Percentage adsorption for both adsorbents increased with increase in metal concentration (BO Opeolu et al., 2010). This is consistent with other studies that tested the effect of metal concentration on metal adsorption by Vijayaraghavan et al., (2004) and Ilhan et al., (2004). Adsorption increased to a certain level and continued until the adsorbent surface became saturated.

As the husk and bagasse are readily available, the potential of these two adsorbents are discovered in removing dyes from simulated water as following the aim of the experiment which is to determine the comparison of ability in removing of a dye, methylene blue, from simulated wastewater by sulphuric acid treated sugar cane bagasse and sulphuric acid coconut husk.

CHAPTER THREE

3 METHODOLOGY

3.1 RESEARCH METHODOLOGY

Research is a method taken in order to gain information regarding the major scope of the project. The sources of the research cover the handbook of condensate stabilization unit, e-journal, e-thesis and several trusted link.

The steps of research:

1. Preparation of biomass.
2. Preparation of heavy metal solutions.
3. Characterization of biomass
4. Adsorption study.
5. Analysis

3.2.1 Preparation of biomass.

The steps involved here is from obtaining the sugarcane bagasse until the biomass is ready to be used as adsorbent:

1. Raw sugarcane was obtained from Seri Iskandar, Perak.
2. The sugarcane was dried at 130°C for 48 hours period.
3. The dried sugarcane was ground using the laboratory blender.
4. Grinded sugarcane bagasse was then sieved to separate the sizes. The selected size is 500 µm.
5. The prepared biomass then was kept in airtight containers.

6. Steps 1 to 5 are repeated by using raw coconut husk

3.2.2 Preparation of dye solutions.

Dye solution was prepared by providing 10ppm methylene blue solution.

3.2.3 Characterization of biomass

The prepared biomass was characterized by using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscope (SEM), and UV – visible spectroscopy, before and after adsorption study.

- (1) For FTIR analyses, 1-3 mg of the prepared sugarcane bagasse and coconut husk about 250 to 300 mg or a small scoop of KBr was grounded together in the agate mortar. Then the mixture of sample and KBr was pressurized gradually by hydraulic press to 10 ton. After that the KBr disc is removed and transferred to sample holder of the spectrometer. The spectrum of sample was scanned, and the spectrum was analyzed for its functional groups.
- (2) SEM was used in order to obtain the morphological image of the sugarcane bagasse sample. The magnification applied during the process is 300X, 1500X, 2000X, and 5000X.
- (3) UV-visible was used in order to obtain percentage of removal methylene blue in the simulated solution based on respective period.

3.2.4 Adsorption study.

The purpose of adsorption study is to observe the adsorption activities. Adsorption study was carried out by mixing the sugarcane bagasse dye solution, followed by coconut husk. Contact period was varied. Samples were then filtered to get the filtrate before using UV-visible Spectrophotometer.

Below are the procedures conducted to study the adsorption capacity of sugarcane bagasse and coconut husk using UV-visible Spectrophotometer.

1. 1 g of 500 μm sugarcane bagasse was added into 100mL of 10ppm in 250-mL Erlenmeyer flask.
2. The solution was left to mix on a water bath shaker at 60°C for 30min, 1 hour, 2 hour, 4 hour, 6 hour and 24 hours
3. The solutions were then filtered and the filtrates were analyzed UV-visible Spectrophotometer to determine the final equilibrium dye concentration.
4. Steps 1 to 3 were repeated by using coconut husk as adsorbent
5. Graphs were plotted to observe the effect of time on the adsorption activities.

3.2.5 Analysis

Samples before and after adsorption study are analyzed using several equipments including Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscope (SEM), Energy and UV – visible spectroscopy

Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) is a technique which is used to obtain an infrared spectrum of absorption, emission, photoconductivity or Raman scattering of a solid, liquid or gas. The goal is to measure how well a sample absorbs light at each wavelength. The raw data (light absorption for each mirror position will be processed into the desired result (light absorption for each wavelength) by using common algorithms called the Fourier transform. The main purpose of performing FTIR analysis in this study is to identify the functional groups that exist in the biomass sample.



Figure 3.1: Fourier Transform Infrared Spectroscopy (FTIR)

Scanning Electron Microscope (SEM)

A scanning electron microscope (SEM) is a type of electron microscope that images a sample by scanning it with a beam of electrons in a raster scan pattern. The electrons interact with the atoms that make up the sample producing signals that contain information about the sample's surface topography, composition, and other properties such as electrical conductivity. The existence of pores can be easily observed due to the combination of higher magnification, larger depth of focus and great resolution.



Figure 3.2: Scanning Electron Microscop 1

UV-Vis spectrometers

UV/Vis spectrometers are used to measure the concentration of chemical (analyte) in solution. In the visible region, reagents are often added that form a coloured compound with the analyte. The absorption (or reduction in light transmission) of light at a particular wavelength is proportional to the concentration. A UV spectrum is a graph of Absorbance vs. wavelength and is unique for certain compounds that absorb light in the UV region. So this can be used to identify unknown compounds.



Figure 3.3: UV-Vis spectrometers

3.2 WORKFLOW SUMMARY

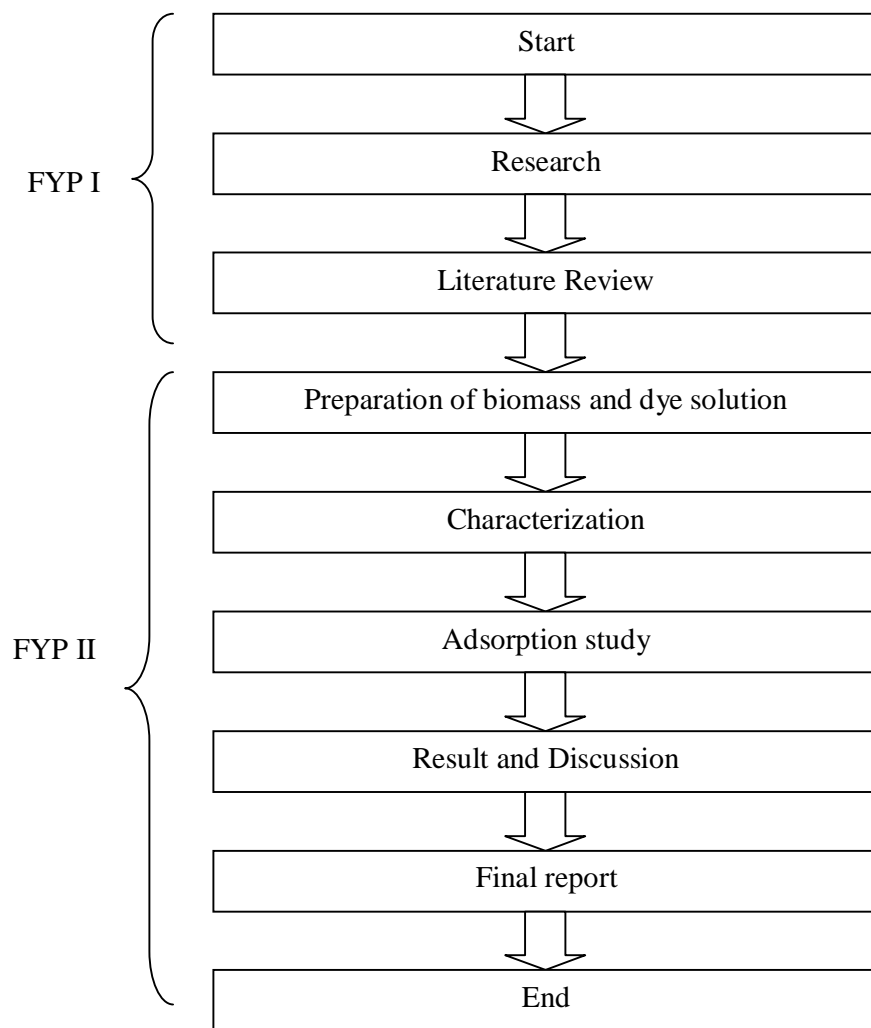


Figure 3.4 Workflow Summary

3.3 GANTT CHART, KEY MILESTONE

No.	Detail/ Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14
1	Selection of Project Topic								Mid-semester break							
2	Preliminary Research Work															
3	Submission of Extended Proposal Defence															
4	Proposal Defence															
5	Project work continues															
6	Submission of Interim Draft Report															
10	Submission of Interim Report															



 Suggested milestone
 Process

Table 3.1 : FYP 1 Gantt Chart

No	Detail/Week	1	2	3	4	5	6	7		8	9	10	11	12	13	14	15
1	Preparation of sugarcane baggase (SB)								Mid-semester Break								
2	Preparation of dye wastewater solution																
3	Adsorption study (SB)																
4	Preparation of coconut husk (CH)																
5	Preparation of dye wastewater solution																
6	Adsorption study (CH)																
7	Submission of Progress Report																
8	Pre-EDX																
9	Submission of Draft Report																
10	Submission of Dissertation (soft bounded)																
11	Submission of Technical Paper																
12	Oral Presentation																
13	Submission of Project Dissertation (Hard bound)																

Table 3.2 : FYP 2 Gantt Chart

CHAPTER FOUR

4 RESULTS AND DISCUSSIONS

4.1 Results

For this results and discussion section, the samples of the sugarcane bagasse will be called by SB and coconut hus will be called coconut husk

4.1.1 SEM results (300X)

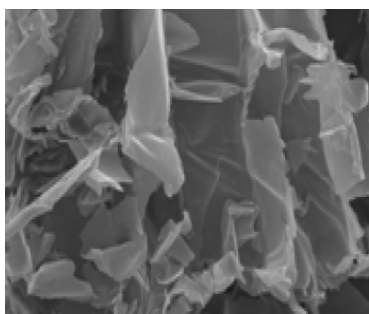


Figure 4.1 Morphology of SB before adsorption

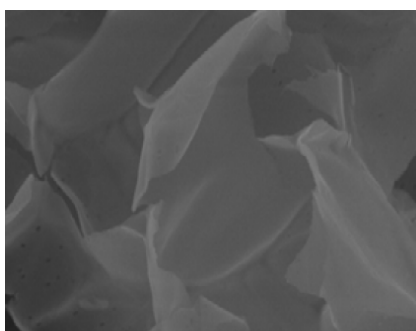


Figure 4.2 Morphology of SB after adsorption

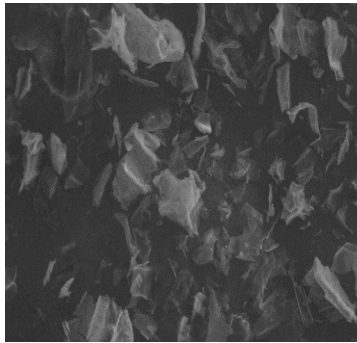
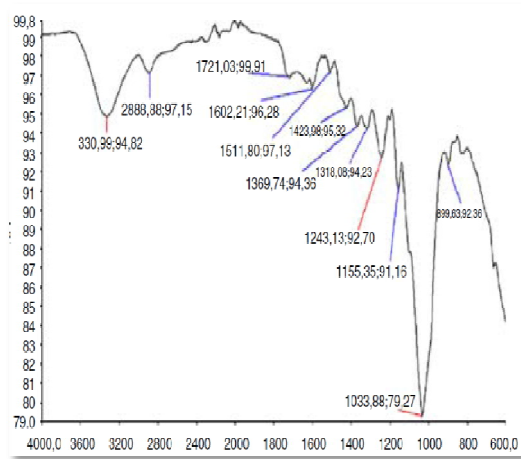


Figure 4.1 Morphology of CH before adsorption

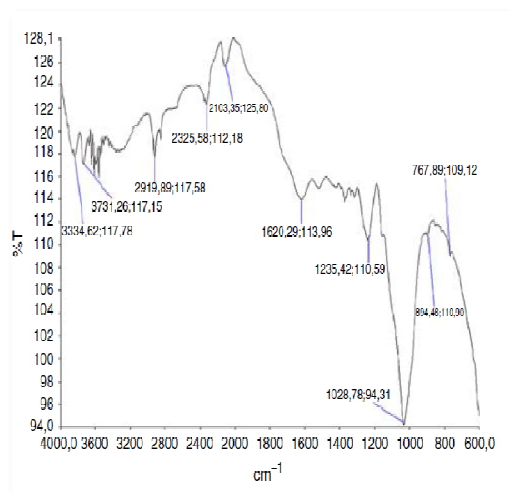


Figure 4.1 Morphology of SB after adsorption

4.1.2 FTIR result

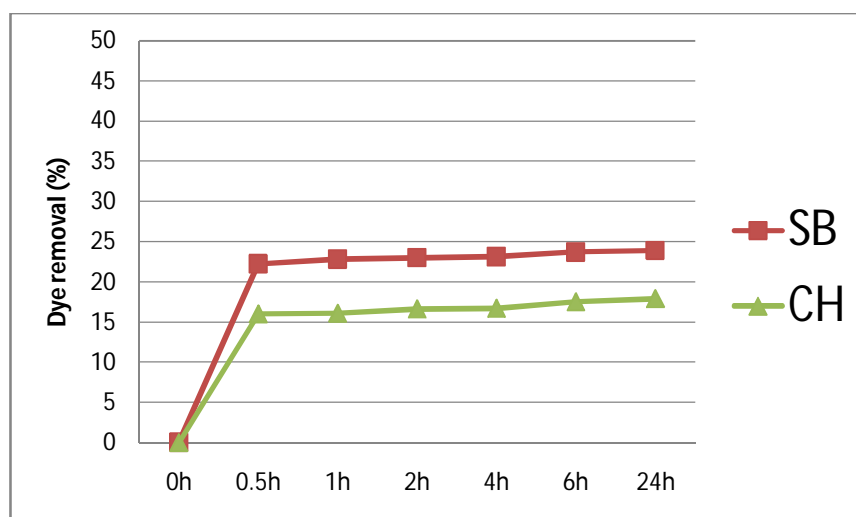


Graph 4.1 FTIR for SB



Graph 4.2 FTIR for CH

4.1.3 Adsorption Study



Graph 4.3 Dye percentage removal vs contact time

4.2 Discussion

Based on the experiment, the discussion were justified based the result obtained: the filtrate and the biomass should were tested by SEM, FTIR and also UV-visible spectrophotometer.

SEM images for SB and CH were obtained for both before and after adsorption process. The magnification of 300X was used for the images in order to get a clearer picture of the biomass wall. The objective is to compare the differences of the morphology of the samples before and after the experiment.

From images for the biomass before adsorption, it shows that SB samples do have bigger size pores at it surface wall and it is also rough, compare with CH. This suggests that SB has more potential to bind methylene blue ion through adsorption process. These ions will simply fill the bigger pores and attached there, thus reducing the amount of dye from its solution. In fact, the surface walls of the SB and CH become smoother than the previous images after the adsorption process. It is proven that adsorption of dye did occur on the surface walls When dye ions filled up the pores, the surface become smoother based SEM images.

This explanation proves that adsorption can happen through adsorption mechanism in these two biomasses.

FTIR analyses of the SB samples and CH samples were also carried out before and after the adsorption process in order to verify the chemical functional groups that exist in the samples. Possible mechanisms of adsorption process that occurred during the experiment can be determined based on the functional groups.

The existence of several chemical functional groups on the biomass would assist the uptake of methylene blue ions during the wastewater treatment. The binding capacity changes according to the affinity between the two elements as it depend on the strength of the electron clouds between the chemical constituents and the ions. Cation in an aqueous solution often forms chemical bonds to anions or neutral molecules that have lone pairs of electrons. Complexation could also be

created between the cations and the oxygen atoms due to the two lone pairs of the electrons.

From the percentage removal of dye versus contact time, it shows that dye ions are removed from the solution. Along the time frame, the dye concentration gradually increase as there should be adsorption process occur as dye percentage removed in SB is lower (20-25%) compared by dye percentage in CH (15-20%). This causes similar with the justification on the SEM finding, based on the characteristics of adsorbents itself.

. Therefore, SB samples adsorb more dye compound than CH. Based to result of SEM, the dye ion ions will simply fill the bigger pores and attached there, thus reducing the amount of dye from its solution. These results prove the justification on the morphology analysis.

CHAPTER FIVE

5 CONCLUSIONS AND RECOMMENDATIONS

It is proved that sugarcane bagasse has higher of adsorption ability compared with coconut husk as the presence of chemical groups and pores characteristic of sugarcane bagasse provide possible mechanism towards better adsorption capacity , compared with coconut husk. The percentage remain of the dye solution in sugarcane bagasse samples is lower than coconut husk sample which were attained by UV-vis spectrometer, which at the same time report the activity of adsorption process at certain contact time for process. From FTIR spectroscopy, it suggests that adsorption can. Adsorption mechanism also happened at the surface wall of the SB and CH, which has been proved by the smoothening of the wall after the adsorption, obtained by SEM images.

For the expansion and continuation of the project, several recommendations have been identified. The recommendations are:

1. Dye waste removal by using treated sugarcane bagasse and treated coconut husk : a comparative study
2. Heavy metal removal by using treated sugarcane bagasse and treated coconut husk : a comparative study .
3. Five difference samples which the highest most abundant in Malaysia based on the current time
4. The dye solution is taken from the actual industrial effluent.

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