ADSORPTION OF METYHLENE BLUE AND FERROUS ION FROM AQUEOUS SOLUTION USING COCONUT HUSK

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A thesis submitted in partial fulfillment of the requirement for the award of degree the of Bachelor of Chemical Engineering

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ABSTRACT

The adsorption process provides an attractive alternative treatment, especially if the adsorbent is inexpensive and readily available. The biomaterials such as coconut husk are low cost agricultural waste residues and are easily available in large quantity in Malaysia. The ability of coconut husk to remove ferum (ii) and Methyene Blue (MB) from aqueous solution as low cost adsorbent was investigated. The adsorption was investigated in three parameters, the effects of the adsorbent dosage, initial concentration and contact time were studied in a batch experiments at room temperature (± 27^{o}C). The coconut husk was dried up at 80^{o}C for 24 hours, and then sieved using sieve shaker. The aqueous solution was mixed with 0.5g adsorbent starting at 100 until 200 minutes with different size of adsorbent in range 500µm to 2mm, while keeping the speed shaker constant at 200RPM. The solution and the adsorbent was separate using filter paper. The same procedure was repeated for initial concentration effect in range 0.25 to 0.05 g/L for FeSO4 and 0.005 to 0.15 g/L for MB, and adsorbent dosage effect in range 2.5 to 5g. Samples of MB and ferum (ii) after the uptake are analyzed with UV-Vis spectrophotometer. Results show that 95.6 - 99.4% of MB removal efficiency was attained in the coconut husk with 500µm of size at 200 minutes of contact time in 0.25g/L of initial concentration. Meanwhile, the removals of ferum (ii) from $FeSO_4$ are 31.2 – 80.8 % respectively. The study is economically feasible and it is proven to be favorable. The results show that coconut husk could be employed effective and low cost material for removal of ferum (ii) and dyes from aqueous solution.

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ABSTRAK

Proses jerapan memberikan rawatan alternatif yang menarik, terutama jika penjerap murah dan tersedia. Bahan semulajadi seperti sabut kelapa kos rendah dari sisa pertanian dan mudah terdapat dalam jumlah yang besar di Malaysia. Kemampuan sabut kelapa untuk menjerap ion besi (ii) dan Methyene Blue (MB) dari larutan air sebagai penjerap murah diselidiki. Jerapan ini diselidiki dalam tiga parameter, kesan daripada dos penjerap, kepekatan awal dan masa dipelajari dalam percubaan secara kumpulan pada suhu ruangan (± 27^{o}C). Sabut kelapa dikeringkan pada 80^{o}C selama 24 jam, selepas itu di tapis menggunakan tapis penggoyang. Larutan dicampur dengan penjerap 0.5g bermula dari 100 hingga 200 minit dengan saiz yang berbeza dari penjerap dalam kadaran 500µm sehingga 2mm, sementara menjaga kelajuan malar penggoncang pada 200RPM. Larutan dan penjerap diasingkan dengan menggunakan kertas penapis. Prosedur yang sama diulang untuk kesan kepekatan awal dalam rentang 0.25-0.05 g / L untuk FeSO4 dan 0.005-0.15 g / L untuk MB, dan kesan dos penjerap dari 2.5 sampai 5g. Larutan MB dan ion besi (ii) selepas serapan tersebut dianalisis dengan spektrofotometer UV-Vis. Keputusan kajian menunjukkan bahawa 95.6-994% dari peratusan serapan MB yang didapati di sabut kelapa dengan 500µm saiz pada 200 minit masa dalam 0.25g / L kepekatan awal. Sementara itu, penghilangan ion besi (ii) dari FeSO4 adalah 31.2-80.8% masing-masing.. Penyelidikan ini secara ekonomi layak dan itu terbukti menguntungkan. Keputusan kajian menunjukkan bahawa sabut kelapa boleh digunakan sebagai bahan yang berkesan dan murah untuk menjerap ion besi (ii) dan pewarna dari larutan air.

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TABLE OF CONTENTS

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LIST OF TABLE

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LIST OF FIGURE

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LIST OF ABBREVIATIONS

WHO	-	World Health Organization
MB	-	Methylene Blue
SSRI	-	selective serotonin reuptake inhibitors
CO_2	-	Carbon dioxide
$FeCl_2$	-	Ferum chloride
$FeSO_4$	-	Ferum sulphate
C_o	-	Initial concentration
C_f	-	Final concentration
RPM	-	Rotate per minute
PPM	-	Part per million

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LIST OF APPENDICES

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LIST OF EQUATIONS

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ADSORPTION OF METHYLENE BLUE AND FERUM (II) FROM AQUEOUS SOLUTION USING COCONUT HUSK

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Abstract: The adsorption process provides an attractive alternative treatment, especially if the adsorbent is inexpensive and readily available. The biomaterials such as coconut husk are low cost agricultural waste residues and are easily available in large quantity in Malaysia. The ability of coconut husk to remove ferum (ii) and Methyene Blue (MB) from aqueous solution as low cost adsorbent was investigated. The adsorption was investigated in three parameters, the effects of the adsorbent dosage, initial concentration and contact time were studied in a batch experiments at room temperature (± 27°C). The coconut husk was dried up at 80°C for 24 hours, and then sieved using sieve shaker. The aqueous solution was mixed with 0.5g adsorbent starting at 100 until 200 minutes with different size of adsorbent in range 500µm to 2mm, while keeping the speed shaker constant at 200 RPM. The solution and the adsorbent was separate using filter paper. The same procedure was repeated for initial concentration effect in range 0.25 to 0.05 g/L for $FeSO_4$ and 0.005 to 0.15 g/L for MB, and adsorbent dosage effect in range 2.5 to 5g. . Samples of MB and ferum (ii) after the uptake are analyzed with UV-Vis spectrophotometer. Results show that 95.6 - 99.4% of MB removal efficiency was attained in the coconut husk with 500µm of size at 200 minutes of contact time in 0.25g/L of initial concentration. Meanwhile, the removals of ferum (ii) from $FeSO_4$ are 31.2 – 80.8 % respectively.. The study is economically feasible and it is proven to be favorable. The results show that coconut husk could be employed effective and low cost material for removal of ferum (ii) and dyes from aqueous solution.

Keywords:coconut husk, Methylene Blue, Ferum sulphate, biosorbent dosage, initial concentration, contact time, UV-Vis spectrophotometer.

1.0 Introduction

Water pollution is now emerging as a major problem in the developing world and its abatement has always been issued in our environmental concern today. The problem of removing pollutants from water and wastewater has grown with rapid industrialization. In Malaysia, 97 % of the effluent discharged is mainly from three industrial categories, which are food industry, chemical industry and textiles industry (Azira et al., 2004; Habib et al., 2006).Among various industrial processes, dyes from textile industries produce a large amount of color effluent which is unacceptable under Malaysia environmental regulations besides other parameters such as COD, BOD, dissolved oxygen, total iron, and others (Tan et al., 2000). Industries such as mining, steel and electroplating, discharge aqueous effluents containing relatively high levels of heavy metals such as silver, cadmium, copper, cobalt, chromium, zinc, iron and lead. Untreated effluents from these manufacturing processes have an adverse impact on the environment and remedial action is needed.

The discharging of dyes and metal ion effluents into natural streams and rivers are causing severe problems, as dyes impart toxicity to aquatic life, thus damaging the aesthetic nature of the environment because dyes used in industry are mostly stable to light and oxidation, as well as resistant to aerobic digestion. Dyes and metal ion are not commonly used in textile industries but also many other industries such as plastics, paper, food, cosmetics, paints, leather, art and craft, printing inks and rubber. Dyes and iron discharged into rivers that meant for public water supply may not meet the drinking water quality standards. Dyes and their degradation products may be carcinogen and toxic if these effluents are treated inefficiently before discharging to the rivers or streams, they could bring negative impact to human health (Kadirvelu et al., 2003)

Ferum does not clearly alter in pure water or in dry air, but when both water and oxygen are present (moist air), iron corrodes. Its silvery color changes to a reddish-brown, because hydrated oxides are formed. The various treatment methods for the removal of color and heavy metals are coagulation using alum, lime, ferric sulfate, ferric chloride, chemical oxidation using chlorine and ozone, membrane separation processes, adsorption and so on. Adsorption is one the physico-chemical treatment processes found to be effective in removing heavy metals and color from aqueous wastewater.

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The WHO allowable concentration in drinking water for Fe is 0.3 mg/l. The Malaysian limit for discharge of Fe into inland water is 1 mg/l, Environmental Quality Act and Regulations (Anonymous, 2002). Hence it is necessary to remove Fe from water/ wastewater to acceptable levels. There are several techniques of removal of dyes from waste water. Some of them are, by flotation, precipitation, oxidation, filtration, coagulation, ozonation, supported liquid membrane, and also biological process (Mahmoud *et al*., 2007). Meanwhile, a new and more environmental friendly method, the biosorption process is proven to be a promising process to remove dyes from effluent. It is stated by Ju (2006) in his research on 2006, biosorption is also known as the uptake or accumulation of chemicals by biomass. This process is similar to adsorbent process which it is cost-effective, easy to operate, simply designed and insensitivity to toxic substances.

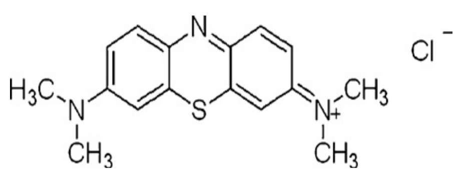
(William et al., 2007; Chern et al., 2001; Larous et al., 2005) since commercially available activated carbon is very expensive, now the research is focused on the use of low cost adsorbents derive from agricultural and wood waste. Some of the proven efficiently adsorbent materials are *Posidonia Oceanica* (L.) fibre (Ncibi *et al*., 2007), orange and banana peels (Annadurai *et al.*, 2002), pumpkin seed hull (Hameed *et al*., 2007) , bagasse, coir pith, rice husk, coconut husk and so on have been used for dye and ferrous ion removal from waste water.

2.0 Materials and Methodology

2.1 Adsorbate

i) Methylene Blue

Methylene Blue, $C_{16}H_{18}ClN_3S.2H_2O$, is a cationic dye purchased from Fisher Scientific. The MB was chosen in this study because of its known strong adsorption onto solids. The maximum adsorption wavelength of this dye is 664 nm. The structure of MB is shown in scheme 1. In this study, MB was used without further purification. All of the MB solution was prepared with distilled water. The stock solution of 1000 mg/L was prepared by dissolving MB in 1000 mL distilled water. The experimental solution was gained by diluting the stock solution with distilled water. The scheme 1 shows the structure of MB:



Scheme 1. The structure of MB

ii) Ferum sulphate, $FeSO_4$

The maximum adsorption wavelength of this dye is 510 nm. In this study, ferrous sulphate $FeSO_4$ was used without further purification. All of the ferrous sulphate solution was prepared with distilled water. The stock solution of 1000 mg/L was prepared by dissolving MB in 1000 mL distilled water. The experimental solution was gained by diluting the stock solution with distilled water.

2.2 Adsorbent

The coconut husk was obtained from local fruit field in Gambang. Then remove the outer layer before proceed to another process and remove the adhering dirt using distilled water then were dried, crushed and sieve. It was then oven dried at 80^{o}C for 24 hours to remove moisture. After drying, the dried samples were crushed and blended using dry blender they were sieve through a size 500µm- 2mm.. No further chemical or physical treatments prior to adsorption experiments.

2.3 Adsorption studies

Adsorption experiments were carried out in a batch process using aqueous solution of Methylene Blue (MB) and Ferum Sulphate, $FeSO_4$. Parameters studied were adsorbent dosage, initial concentration and contact time. Stock solution of MB and Ferum Sulphate was prepared and standard solutions for the experiments were diluted from stock.

The experiments were conducted in a rotary shaker at 200RPM and at room temperature, 27^{o}C using 250mL conical flasks containing 100mL dye solution at different size of adsorbent (coconut husk) in different of time from 100-200 minutes. After the optimum time will achieve, it used for adsorbent in several of initial concentration. A number of doses of coconut husk were added to each flask, and then the flasks were sealed up with parafilm or

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aluminium foil to prevent any spilling that could change the volume of solution during the experiments. After shaking the flasks for a period of time intervals, the aqueous samples were taken and the MB concentrations were measured by UV-Vis Spectrophotometer (Hitachi, Model U-1800) at its maximum wavelength, 664 nm.The percentage of MB removal from solution was calculated by:

$$R(\%) = \frac{C_o - C_f}{C_o} \; x \; 100 \qquad (2.1)$$

Where C_o is the initial MB concentration and C_f is the MB concentration (mg/L) at any time. Then the overall method was repeated for Ferum Sulphate, $FeSO_4$ solution which 510nm for its maximum wavelength.

3.0 Results and Discussion

3.1 Effect of contact time by different sizes of adsorbent

Adsorption rate can also be affected by varying the contact time during shaking the samples of MB and $FeSO_4$ solution. The study of contact time was conducted with various sizes of adsorbent (coconut husk) in range 2mm, 1mm, 800 µm, 630 µm and 500 µm. while keeping the other parameters constant (C_O = 0.25 g/L, adsorbent dosage = 0.5 gram, shaker speed= 200rpm). Figure 3.1 shows the result the percentage of MB removal and figure 3.2 is the percentage of ferrous ion removal. It can be explained clearly that the MB and ferrous ion removal was increase with the increasing of time contact between the dye and adsorbent. From this result, it also can determined the optimum size of adsorbent for both solution which the percentage removal increase on 500 µm than 630 µm to 2 mm, which it reached at 97.2% for MB removal and 83.2% for ferrous ion removal at 200 minutes. The dyes ferrous ion adsorbed increased as the adsorbent particle size decreased. It was suggested that the increase in sorption depended on the large external surface area for small particles removes more dye in the initial stages of the sorption process than the large particles. For this reason, for all batch experiments, particle sizes of 500 µm and 200 minutes have been selected for further parameter.

a)

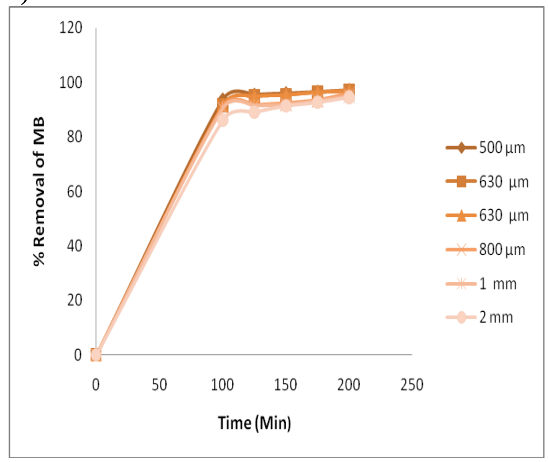

Figure 3.1: The effect of contact time for the percentage removal of MB. Speed=200 rpm, dosage=0.50g, Co=0.25g/L:

b)

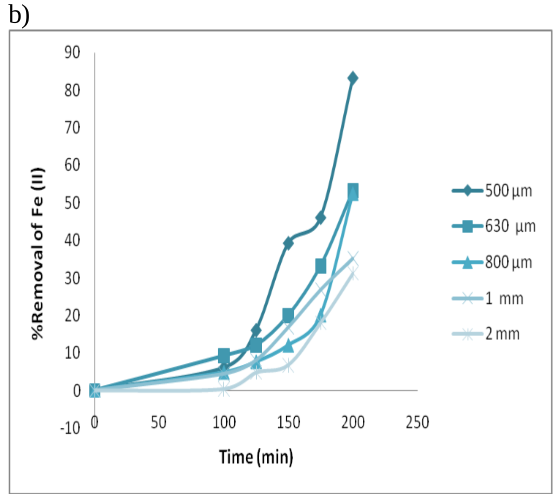

Figure 3.2: The effect of contact time for the percentage removal of ferrous ion. Speed=200 rpm, dosage=0.50g, Co=0.25g/L:

3.2 Effect of initial concentration

The effects of initial concentration for the removal of the dye were carried out by varying the value of initial concentration of MB solution from 0.05 to 0.25 g/L and $FeSO_4$ solution from 0.005 to 0.15 g/L with fixed value of other parameters (Dosage = 0.5 g, shaker speed = 200 rpm, time = 200 minutes). The result in Figure (a) indicates that as the initial

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concentration, when the dye concentration was increased from 0.005 to 0.15 g/L the percentages of dyes sorbed decreased from 95.6 to 99.4%. It is because when in the low concentration, the dyes was decreased and it rapidly decreased by increasing of initial concentration. The dyes it forms of color for the solution, it different with $FeSO_4$ solutions which have particles of ferrous ion in solution. And b) Show that when $FeSO_4$ solution was increased, the percentage removal of ferrous ion from 31.2 to 80.8%. It means that the adsorption is highly dependent on initial concentration of metal ion. It is because of at lower concentration, the ratio of initial number of metal ions to the available surface area is low subsequently the fractional adsorption become independent of initial concentration. However, at high concentration the available sites of adsorption becomes fewer and hence the adsorption of metal ions is dependent upon initial concentration. The relation between the amount of metal ion adsorbed on the initial concentration of ferrous and MB are shown in Figure 2:

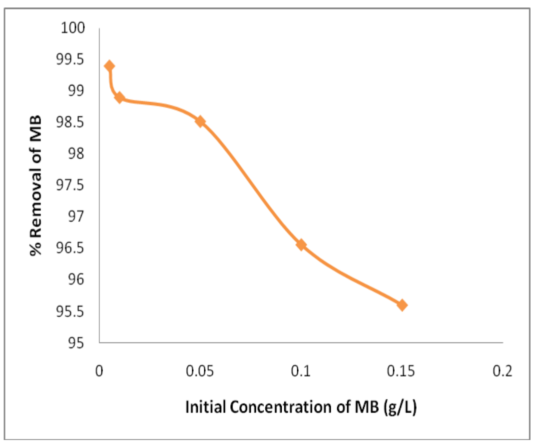

Figure 3.3: The effect of initial concentration for the percentage removal of MB Speed=200 rpm, time=200minutes, dosage=0.5 g

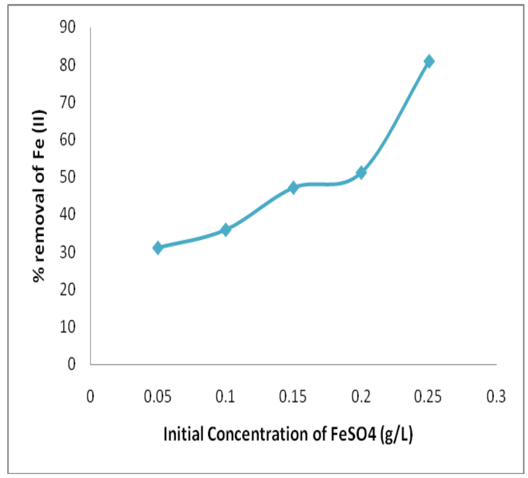

Figure 3.4: The effect of initial concentration for the percentage removal of ferrous ion. Speed=200 rpm, time=200minutes, dosage=0.5 g.

3.3 Effect of adsorbent dosage

The study of adsorbent dosage effect on MB dye and $FeSO_4$ solution uptake can be observed in figure 3.5 and 3.6. Experiment is conducted by using coconut husk doses as adsorbent range from 0.5 g to 2.5 g while keeping the other parameters constant (C_o = 0.25 g/L, shaker speed = 120 rpm, time = 120 minutes). Results showed that the adsorption of the metal ions increased with increasing adsorbent dosage. This result can be explained as a consequence of a partial aggregation, which occurs at higher adsorbent dosage giving rise in a decrease of active sites on the adsorbent. Therefore, the amount of adsorbent was selected as 2.5 g.

The increasing value of percentage removal of dye and ferrous ion uptake might be due to increase of surface area which means increasing of availability of sorption sites for removal with the increase in adsorbent dosage. Whereas, the decreasing in dye sorbed per unit mass with increasing of coconut husk dosage can be because of reduction in effective surface area, or in other word, the adsorbate is saturated.

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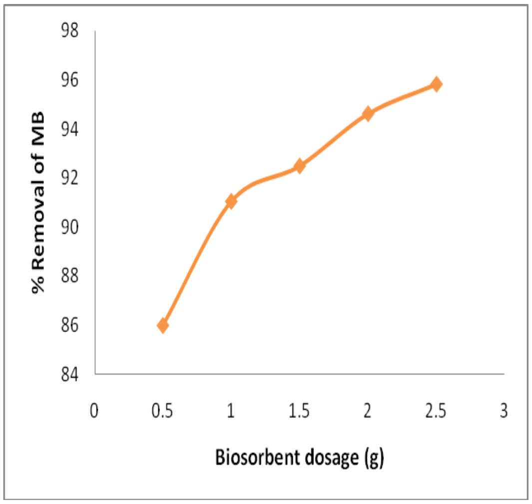

Figure 3.5: The effect of adsorbent dosage for the percentage removal of MB. Speed=200 rpm, time=200minutes, dosage=0.5-2.5g, C_o=0.25 g/L

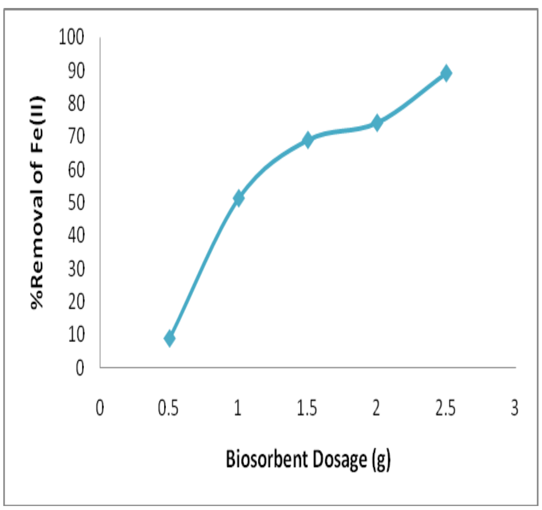

Figure 3.6: The effect of adsorbent dosage for the percentage removal of ferrous ion. Speed=200 rpm, time=200minutes, dosage=0.5-2.5g, C_o=0.25 g/L

4.0 Conclusion

Results and analyses obtained from this study proven that the adsorption of methylene blue and ferum(ii) from aqueous solution using coconut husk is a success. The percentage removal of MB and ferum(ii) can be enhanced by the adjustments on the contacts time, initial concentration and adsorbent dosage, where the results shows that the optimum conditions for the uptake of methylene blue are at 0.25g/L, 200 minutes, and 2.5 g of adsorbent. Meanwhile, for the ferum(ii) are at 0.005g/L, 200 minutes, and 2.5 g of adsorbent respectively, which for the both aqueous solution was conducted on 500µm size of coconut husk.

Increasing of adsorbent dosage increased the dye and ferum(ii) removal because of the enhancement of the availability of sorption sites as the coconut husk is added to the solution. Effects of initial concentration of ferrous ion is studied as well and the results shows that the uptake of ferrous ion rising when the concentration of $FeSO_4$ is increased. This is because of due to the stronger interaction between molecule of ferrous ion and the particle of coconut husk. Meanwhile, it's different with initial concentration of MB, which the percentages of dyes sorbed decreased with increase of initial concentration. It is because when in the low concentration, the dyes was decreased and it rapidly decreased by increasing of initial concentration. The dyes it forms of color for the solution, it different with $FeSO_4$ solutions which have particles of ferrous ion in solution. Meanwhile, the study of contact time between MB and $FeSO_4$ solution and sorbent shows increasing of contact time was increase the percentage removal of dye and ferrous ion.

It can be concluded that the coconut husk is an effective and alternative low cost adsorbent for the removal of Ferrous and MB ions from wastewaters in terms of high adsorption capacity, natural and abundant availability. In spite of the scarcity of consistent cost information, the widespread uses of low-cost adsorbents in industries for wastewater treatment applications today are strongly recommended due to their local availability, technical feasibility, engineering applicability, and cost effectiveness. If low cost adsorbents perform well in removing Methylene blue and iron at low cost, they can be adopted and widely used in industries not only to minimize cost inefficiency, but also improve profitability. Undoubtedly low cost adsorbents offer a lot of promising benefits for commercial purposes in the future.

4.1. Recommendations

It is recommended to do a research in other parameter such as effect of temperature;

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pH or speed agitation. It also can be developed and applied in real wastewater.

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Water and wastewater pollution is now emerging as a major problem in the developing world and its abatement has always been issued in our environmental concern today. The problem of removing pollutants from water and wastewater has grown with rapid industrialization. In Malaysia, 97 % of the effluent discharged is mainly from three industrial categories, which are food industry, chemical industry and textiles industry (Azira et al., 2004; Habib et al., 2006).Among various industrial processes, dyes from textile industries produce a large amount of color effluent which is unacceptable under Malaysia environmental regulations besides other parameters such as COD, BOD, dissolved oxygen, total iron, and others (Tan et al., 2000). Industries such as mining, steel and electroplating, discharge aqueous effluents containing relatively high levels of heavy metals such as silver, cadmium, copper, cobalt, chromium, zinc, iron and lead. Untreated effluents from these manufacturing processes have an adverse impact on the environment and remedial action is needed.

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The discharging of dyes and metal ion effluents into natural streams and rivers are causing severe problems, as dyes impart toxicity to aquatic life, thus damaging the aesthetic nature of the environment because dyes used in industry are mostly stable to light and oxidation, as well as resistant to aerobic digestion. Dyes and metal ion are not commonly used in textile industries but also many other industries such as plastics, paper, food, cosmetics, paints, leather, art and craft, printing inks and rubber. Dyes and iron discharged into rivers that meant for public water supply may not meet the drinking water quality standards. Dyes and their degradation products may be carcinogen and toxic if these effluents are treated inefficiently before discharging to the rivers or streams, they could bring negative impact to human health (Kadirvelu et al., 2003; Janos et al., 2003).

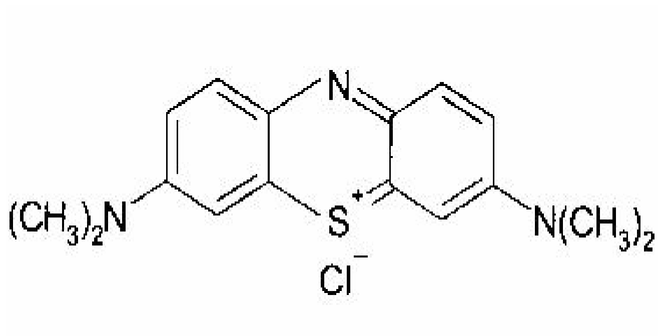

Figure 1.1: Structure of Methylene Blue

Ferrous ion does not clearly alter in pure water or in dry air, but when both water and oxygen are present (moist air), iron corrodes. Its silvery color changes to a reddish-brown, because hydrated oxides are formed. The various treatment methods for the removal of color and heavy metals are coagulation using alum, lime, ferric sulfate, ferric chloride, chemical oxidation using chlorine and ozone, membrane separation processes, adsorption and so on. Adsorption is one the physico-chemical treatment processes found to be effective in removing heavy metals and color from aqueous wastewater.

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The WHO allowable concentration in drinking water for Fe is 0.3 mg/l. The Malaysian limit for discharge of Fe into inland water is 1 mg/l, Environmental Quality Act and Regulations (Anonymous, 2002). Hence it is necessary to remove Fe from water/ wastewater to acceptable levels. There are several techniques of removal of dyes from waste water. Some of them are, by flotation, precipitation, oxidation, filtration, coagulation, ozonation, supported liquid membrane, and also biological process (Najim *et al.*, 2008). Meanwhile, a new and more environmental friendly method, the biosorption process is proven to be a promising process to remove dyes from effluent. It is stated by Ju (2006) in his research on 2006, adsorption is also known as the uptake or accumulation of chemicals by biomass or agricultural solid waste. This process is similar to adsorbent process which it is cost-effective, easy to operate, simply designed and insensitivity to toxic substances.

Activated carbon is well known as the most widely used adsorbent and proven to be effective for removal of dye due to its large surface area, micro-porous structure, high adsorption capacity, etc, (Wang *et al.*, 2008) but since it is high cost, this limits its usage in large scale production. Therefore, researches have investigated other alternatives adsorbent which is cost-effective, efficient and easily available materials for the adsorption process.

(William et al., 2007; Chern et al., 2001; Larous et al., 2005) since commercially available activated carbon is very expensive, now the research is focused on the use of low cost adsorbents derive from agricultural and wood waste. Some of the proven efficiently adsorbent materials are *Posidonia Oceanica* (L.) fibre (Ncibi *et al.*, 2007), orange and banana peels (Annadurai *et al.*, 2002), pumpkin seed hull (Hameed *et al.*, 2007), bagasse, coir pith, rice husk, coconut husk and so on have been used for dye and ferrous ion removal from waste water.

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Figure 1.2: The Coconut Husk

In this study coconut husk was designed to investigate. The parameters studied include contact time, dosage of coconut husk and initial concentration on the sorption capacity by using Methylene Blue dye and ferum (ii) sulphate as the adsorbate.

1.2 Problem Statement

Environmental pollution resulted from wastewater pollution is increasing throughout the world with the growth of industrial activities. Trace metals and dyes are widely spread in environment and may enter the food chain from the environment. It is well recognized that heavy metals and dyes in the environment are hazardous to human and the other living organisms. Unlike organic pollutants, heavy metals are non-biodegradable and therefore, the removal of them is extremely important in terms of healthy of livings specimens. Owing to its color, it is the most easily recognizable pollutant.

Although it may be present in minute quantities, its presence is still clearly undesirable from the aesthetic point of view. 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

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