

BIOSORPTION OF METHYLENE BLUE AND ACID ORANGE 7 BY
MODIFIED HEXADECYLTRIMETHYLAMMONIUM BROMIDE RICE HUSK

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To my beloved family:

Ahmad Jani bin Hussin

Noor Aishah binti Abdul Karim

Mohd Aswad bin Ahmad Jani

Nordiana binti Salim

Mohd Azrul Fahmi bin Ahmad Jani

and all Hussin's and Abdul Karim's family members

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ABSTRACT

In this study, rice husk (RRH) was modified with surfactant (SMRH) to remove dyes in batch mode and was compared with unmodified rice husk (RRH). SMRH was prepared by mixing the RRH with different concentrations of cationic surfactant Hexadecyltrimethylammonium Bromide (HDTMABr). All samples were then characterized with Fourier Transform-Infrared Spectroscopy (FTIR). Screening experiment was done by comparing adsorption capacity of SMRH and RRH toward several anionic and cationic dyes with concentration of dyes at 50 mg/L. From the experiment, RRH and SMRH show affinity towards cationic and anionic dyes, respectively. The adsorptive potential of the adsorbents was further investigated towards methylene blue (MB) and acid orange 7 (AO7) with different concentration of dyes solution (5.0 - 1000.0) mg/L and different concentration of HDTMABr modified rice husk (1.0, 2.5 and 4.0) mM. Langmuir and Freundlich isotherms were used to describe adsorption isotherms. The results shows that the removal of AO7 was higher for SMRH compared to RRH meanwhile MB removal was higher for RRH compared to SMRH. Modification of rice husk by surfactant was proven to decrease the adsorption site for MB. On the other hand, the presence of surfactant on the SMRH increases the adsorption site and allows more adsorption to occur for AO7. The equilibrium data were better described by Langmuir isotherm with maximum adsorption capacity of AO7 was 200.0 mg/g and 100.0 mg/g for MB. In conclusion, the modification of rice husk by HDTMABr decreased the adsorption towards cationic dyes but increased the adsorption towards anionic dyes.

ABSTRAK

Dalam kajian ini, sekam padi (RRH) telah diubahsuai dengan surfaktan (SMRH) untuk menyingkirkan pewarna dalam mod kelompok dan dibandingkan dengan sekam padi yang tidak diubahsuai (RRH). SMRH telah disediakan dengan mencampurkan RRH dengan kepekatan surfaktan kationik Heksadesiltrimetilammonium Bromida (HDTMABr) yang berbeza. Semua sampel kemudiannya dicirikan dengan kaedah spektroskopi inframerah (FTIR). Eksperimen saringan telah dilakukan dengan membandingkan kapasiti penjerapan SMRH dan RRH keatas beberapa pewarna anionik dan kationik dengan kepekatan pewarna pada 50 mg/L. Daripada ujikaji tersebut, RRH menunjukkan kapasiti penjerapan keatas pewarna kationik dan SMRH keatas pewarna anion. Potensi serapan penjerap terus dikaji terhadap metilina biru (MB) dan asid oren 7 (AO7) dengan kepekatan larutan pewarna yang berbeza (5.0-1000.0) mg/L dan kepekatan HDTMABr (1.0, 2.5 dan 4.0) mM yang berbeza. Isoterma Langmuir dan Freundlich telah digunakan untuk menggambarkan jenis isoterma penjerapan. Keputusan menunjukkan bahawa penyingkiran AO7 adalah lebih tinggi untuk SMRH berbanding RRH sementara penyingkiran MB adalah lebih tinggi untuk RRH berbanding SMRH. Pengubahsuaian sekam padi oleh surfaktan telah terbukti mengurangkan kawasan penjerapan MB dalam larutan. Sebaliknya, kehadiran surfaktan pada SMRH meningkatkan kawasan penjerapan dan membolehkan lebih penjerapan AO7 berlaku. Data keseimbangan telah digambarkan dengan lebih baik oleh isoterma Langmuir dengan kapasiti penjerapan maksimum AO7 adalah 200.0 mg/g dan 100.0 mg/g untuk MB. Kesimpulannya, pengubahsuaian sekam padi oleh HDTMABr mengurangkan penjerapan terhadap pewarna kationik tetapi meningkatkan penjerapan terhadap pewarna anionik.

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LIST OF SYMBOLS

$^{\circ}\text{C}$	-	Degree Celsius
C_i	-	Initial concentration
C_e	-	Equilibrium concentration
cm	-	Centimeter
g	-	Gram
kg	-	Kilogram
kV	-	Kilo Volt
L	-	Liter
m	-	Meter
M	-	Molar
mg	-	Milligram
min	-	Minute
mL	-	Milliliter
mm	-	Millimeter
mmol	-	Millimole
nm	-	Nanometer
ppm	-	Parts per million
λ	-	Lambda

LIST OF ABBREVIATIONS

HDTMABr	-	Hexadecyltrimethylammonium Bromide
FTIR	-	Fourier Transform Infrared
IR	-	Infrared
[MB]	-	Concentration of Methylene blue
[AO7]	-	Concentration of Methyl orange
RRH	-	Raw rice husk
SMRH	-	Surfactant modified rice husk
1 SMRH	-	Rice husk treated with 1.0 mM concentration of (HDTMABr)
2.5 SMRH	-	Rice husk treated with 2.5 mM concentration of (HDTMABr)
4 SMRH	-	Rice husk treated with 4.0 mM concentration of (HDTMABr)
CMC	-	Critical Micelle Concentration
UV - Vis	-	Ultra Violet-Visible

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CHAPTER 1

INTRODUCTION

1.1 Research Background

The rise of the textile industry in recent centuries has a significant effect on the economic resources of the world. Nevertheless, the increase of textile industry also raised environmental concerns against pollution resulting from waste disposal of this industry as it is among the largest users of chemicals and water. During the textile production, dyes are widely used for coloring purpose such as in staining and printing process. However, not all dyestuff binds to the textile during the process. In fact, it is estimated 10 to 25 % of the dyes are lost throughout the process while 2 to 20% are directly discharged as effluent which may results in serious water pollution (Carmen and Daniela,2012).

In this study, methylene blue (MB) and acid orange 7 (AO7) were selected to be studied as model dyes mainly because these dyes are widely used in several industries such as paper and textile industries. In addition, their presence in effluent was harmful to environment as their breakdown products are toxic and carcinogenic due to the aromatic compound such as naphthalene and benzidine (Carmen and Daniela, 2012). Moreover, waste water containing dyes is reported to be among the most difficult waste water to be treated because of the complex molecular structure and high water solubility properties of dyes (Forgacs *et al.*, 2004; Crini, 2006; Gupta, 2009)

In relation to the problems, many techniques have been implemented to remove dyes in waste water such as by flocculation, sedimentation and filtration (Tarley and Aruda, 2003; Yun-nen *et al.*, 2010). Unfortunately, according to Srinivasan and Viraraghavan (2010) these methods are expensive, have low removal efficiency and effective for only certain type of dyes hence, adsorption method is more preferable as researchers believed this process is more efficient and lower-price compared to other methods (Nghah *et al.*, 2011; Zhang *et al.*, 2012).

Adsorption is a surface phenomenon in which it allows the adsorbent to passively concentrate and bind the adsorbate on its cellular surface due to the binding force of atom, molecules and ions of adsorbate toward adsorbent surface (Park *et al.*, 2010). In the adsorption process activated carbon is one of the best candidate due to its large surface area properties, high reactivity on the surface and also high sorption capacity (Yun-nen *et al.*, 2010). However, commercialized activated carbons are expensive and their manufacturing process possesses many problems (Ola Abdelwahab *et al.*, 2005). Therefore, researchers tried to find better substitutes which offer lower cost yet efficient to replace activated carbon which has led to the use of agricultural waste since they are low cost, high availability and more importantly the lignocellulosic surface of the adsorbent that is favorable for adsorption process (Gupta, 2009; Tsai and Chen, 2010). Furthermore, the decision of using agricultural wastes as adsorbent will alleviate pollution problems because instead of thrown away the waste, there can be re-used for something beneficial. Example of agricultural wastes for dye removal were wheat straw (Zhang *et al.*, 2012), papaya stem (Krishni *et al.*, 2013), maize husk (Jalil *et al.*, 2012) and citrus bagasse (Bhatti *et al.*, 2012).

Another advantage of using biological origin such as agricultural waste is they are able to remove dyes either in natural form or undergo physical or chemical modification. Thus, to improve their adsorption capacity and stability, several modifications were done. It was supported by the studies done by previous researchers who claimed that modified agricultural waste shows more adsorption capacity compared to the unmodified one (Su *et al.*, 2013; Senthil Kumar *et al.*, 2013; Ong *et al.*, 2013; Sureshkumar and Namasivayam, 2008). The modifications can be done by using base, acid, organic or other solutions.

The choice of rice husk for this study was due to its properties that meet the criteria as a good adsorbent. There are a lot of studies done using rice husk for dye removal (Ola Abdelwahab *et al.*, 2005; Chakraborty *et al.*, 2011) but only a few focusing on using cationic surfactant on rice husk (Safa and Bhatti, 2011). Cationic surfactant is believed to increase the adsorption capacity on rice husk surface (Sadonet *et al.*, 2012). The cationic surfactant used in this study was Hexadecyltrimethylammonium Bromide (HDTMABr). It is composed of “permanent charged pentavalent nitrogen and a long straight alkyl (C16) chain” (Namasivayam and Sureshkumar, 2006). Sorption of HDTMABr on rice husk can change the chemistry of the external surface of rice husk where bilayer-like structure of HDTMABr molecules is formed and thus the charge on the surface of rice husk change from negative to positive charge. It will lead to production of electrostatic attraction with a negative charge dye molecules (Baskaralingam *et al.*, 2006).

To date, there is no study reported on removal of methylene blue (MB) and acid orange 7(AO7) by surfactant modified rice husk. Thus, it is very interesting to investigate the adsorption behavior of surfactant modified rice husk toward anionic (AO7) and cationic (MB) dyes in aqueous solution as it might lead the adsorption knowledge into a new level as well as for environment preservation.

1.2 Research Problems

According to Zhang *et al.* (2012) it is estimated that almost 70,000 tonnes of dyes are released into the water every year. Unlike organic pollutant, dyes are not easily removed from the waste water due to its high solubility towards the water (Crini, 2006). Other than that, dyes are also harmful (Mohamed, 2004) and can be fatal. This situation causes various ecological problems worldwide and if occur in fresh water, the water might be toxic and unsuitable to be used as potable water sources anymore. Thus, it is very crucial for researchers to find an efficient method to tackle these pollution problems.

Many researchers find that adsorption methods are able to remove dyes in waste water efficiently (Tarley and Aruda, 2003; Yun-nen *et al.*, 2010; Bhatnagar and Silanpaa, 2010). This is due to its simple design and requires low initial installment as well as small space. Also, this process produces a sludge free environment (Sadon *et al.*, 2012). The most common used adsorbent is activated carbon however, it is costly. Thus, this research aims to tackle this problem by using low cost adsorbent from agricultural waste which offers similar functions in terms of adsorption activity.

In this study, rice husk was selected as adsorbent due to the presence of lignocellulose structure. Most plant waste have negative charged surface in water which cause them unable to adsorb anionic (negative charged) dyes efficiently (Chen *et al.*, 2011). In addition, direct use of rice husk might not be efficient enough for adsorption due to low sorption capacity because of low specific surface area for solute – surface interaction (Laszlo and Dintzis, 1994). According to Wan Ngah and Hanafiah (2008), direct use of agricultural adsorbent might caused secondary pollution results from the release of some soluble compounds of the materials. Therefore, several modifications were made to the adsorbent surface to enhance their effectiveness on adsorbing anionic dyes. The modifications included mechanical modifications whereby the rice husk was ground into smaller size to increase solute-surface interaction, physical modification where the rice husk was sun dried and oven dried to remove moisture as well as chemical modification with cationic surfactant to allow more specific area for dye adsorption.

1.3 Research Objectives

- a) To prepare and characterize rice husk in powder form.
- b) To prepare surfactant modified rice husk (SMRH) with different surfactant loading.
- c) To screen the adsorption capacity of unmodified and modified rice husk (SMRH) with several anionic and cationic dyes at fixed dyes concentration.

- d) To study the biosorption behavior of methylene blue (MB) and acid orange 7 (AO7) by unmodified and modified rice husk (SMRH).

1.4 Research Scope

The scope of this study was to analyse the potential of surfactant modified rice husk (SMRH) as low cost natural adsorbent to remove cationic (MB) and anionic (AO7) dyes from solution. It was done by observation on several adsorption parameters such as concentration of dyes and concentration of surfactant onto rice husk during modification. In addition, characterization of modified and unmodified rice husk by FTIR spectroscopy was done to observe if modification changes the chemical linkages of the samples.

The maximum adsorption of dyes by studied samples was calculated by adsorption isotherm and all the data in this study were obtained from batch experiments in laboratory scale.

1.5 Research Significance

In this experiment, methylene blue (MB) and acid orange 7(AO7)acted as the adsorbate which removal of these dyes become the major aim of this study. The release of these dyes into the effluent may cause severe water pollution as had happened in the Citarum River located in West Java where the people there suffered serious skin disease due to the pollution of the main source of water (Brigden *et al.*, 2013).Therefore this adsorption study might help in tackling the dye pollution problems so that number of water pollution cases will be reduced.

Activated carbon was proven to be a good adsorbent as it has high adsorption capacity and high contact surface area due to its micro porous structure however, its

production cost is expensive. Therefore, modified rice husk is a potential low-cost replacement of adsorbent since it was selective towards dye adsorption. In addition, being an agricultural waste make it easily to be found at almost no cost especially in Malaysia which is among the largest rice-producing countries (Akinbile *et al.*, 2011).

As this project manage to remove dyes in water, it should benefit the society by improving the wastewater management especially in dye polluted area and consequently providing a cleaner environment. Thus this project is relevant to human health as well as environment.

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