SYNTHESIS AND CHARACTERIZATION OF ADSORBENT DERIVED FROM PANDAN LEAVES FOR METHYLENE BLUE REMOVAL

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Specially dedicated to

my beloved parents,

my siblings,

my best friends,

and

to all that involve in writing this thesis….

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ABSTRACT

Dyes are an important type of pollutants that come in a large amounts which originated from industries such as textile, paper and pulp making, and petroleum. The elimination of dyes from wastewater is challenging as they have a synthetic origin and possess complex aromatic molecular structures which caused them to structurally inert and difficult to biodegrade, and unfortunately, the industries have overlooked this aspect in the treatment of wastes. Adsorption is known to be the superior technique compared to all other chemical for dye treatment. In this study, adsorbent was synthesized through chemical activation process by NaOH. The pandan leaves adsorbent was characterized by Fourier Transform Infrared (FTIR) and Brunauer-Emmett-Teller (BET). Results revealed that the pandan leaves adsorbent was successfully synthesized. The BET surface area for the adsorbent is 6.79 m^2/g. The adsorption capacity was found to be dependent on reaction time, pH, adsorbent dosage and initial dye concentration. The highest amount of adsorption capacity is 24.113 mg/g that was found at the adsorbent dosage of 0.02 g. The adsorption data for MB dye were well fitted Langmuir isotherm model and obeyed the pseudo-second order kinetics model. The value of standard Gibbs free energy for temperature 303.15 K is -1.895, temperature 323.15 K is -5.228 and temperature 333.15 K is -5.581. The negative value of standard Gibbs free energy indicated the feasibility and spontaneity of the adsorption processes and the positive value of enthalpy change confirmed the endothermic nature of overall process.

ABSTRAK

Pewarna, adalah jenis bahan pencemar dalam jumlah yang besar yang berasal dari industri seperti tekstil, kertas dan pembuatan pulpa, dan petroleum. Untuk merawat sisa pewarna ini adalah sangat mencabar kerana ia mempunyai struktur aromatic yang kompleks yang meyukarkan ia untuk terurai dan banyak industri telah mengabaikan untuk merawat sisa pewarna ini. Penjerapan dikenali sebagai teknik yang unggul berbanding dengan semua teknik kimia yang lain untuk rawatan pewarna. Dalam kajian ini, bahan penjerap yang dihasilkan daripada daun pandan telah digunakan untuk penyingkiran pewarna Methylene Blue. Penjerap mereka disintesis melalui proses pengaktifan kimia dengan menggunakan NaOH. Penjerap daun pandan dicirikan oleh Fourier Transform Infrared (FTIR) dan Brunauer-Emmett-Teller (BET). Hasil kajian menunjukkan bahawa penjerap daun pandan telah berjaya dihasilkan. Luas permukaan BET untuk penjerap adalah 6.79 m^2/g. Kapasiti penjerapan didapati bergantung kepada masa tindak balas, pH, dos penjerap dan kepekatan awal pewarna. Kapasiti penjerapan yang paling tinggi ialah 24.113 mg/g yang diperolehi pada penggunaan adsorben sebanyak 0.02 g. Data penjerapan telah dipadankan dengan model isotherma Langmuir dan didapati mematuhi model kinetic pseudo-tertib-kedua. Nilai piawai tenaga bebas Gibbs untuk suhu 303.15 K ialah -1.895, suhu 323.15 K ialah -5.228 and suhu 333.15 K ialah -5.581. Nilai negatif piawai tenaga bebas Gibbs menunjukkan kebolehlaksanaan dan proses penjerapan spontan. Nilai negatif entalpi mengesahkan sifat endotermik dalam keseluruhan proses penjerapan.

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LIST OF ABBREVIATIONS

AOPs	-	Advanced Oxidation Processes
BET	-	Brunauer-Emmett-Teller
CO_2	-	Carbon Dioxide
COD	-	Chemical Oxygen Demand
FTIR	-	Fourier Transform Infra-Red
H_2O	-	Water
H_3PO_4	-	Phosphoric Acid
HCl	-	Hydrochloric Acid
H_2SO_4	-	Sulfuric Acid
KOH	-	Potassium Hydroxide
LCAs	-	Low Cost Adsorbent
MB	-	Methylene Blue
MO	-	Methyl Orange
NaOH	-	Sodium Hydroxide
NaCl	-	Sodium Chloride
Na_2SO_4	-	Sodium Sulfate
UV	-	Ultra Violet
$ZnCl_2$	-	Zinc Chloride

LIST OF SYMBOLS

Ce	-	Equilibrium concentration
Co	-	Initial concentration
g	-	Gram
g/mol	-	Gram per mol
hr (s)	-	Hour (s)
µm	-	Micro Meter
J	-	Joule
K	-	Kelvin
kJ	-	Kilo Joule
k1	-	Adsorption rate constant of first order adsorption
k2	-	Adsorption rate constant of second order adsorption
KF	-	Freundlich constant
kg	-	Kilogram
kJ/mol	-	Kilo Joule per mol
KL	-	Langmuir constants related to the rate of adsorption
kp	-	Intraparticle diffusion rate constant
L	-	Liter
M	-	Molar
m^2/g	-	Meter square per gram
mg	-	Milligram
mg/g	-	Milligram per gram
mg/L	-	Milligram per liter
min	-	Minute
mg/g	-	Miligram per gram
mg/L	-	Miligram per Liter

mm	-	Millimeter
n	-	Freundlich constant
nm	-	Nanometer
°C	-	Degree celcius
q_e	-	Amount of adsorbent at equilibrium
Q_o	-	Langmuir constants related to adsorption capacity
q_t	-	Equilibrium rate constant
R^3	-	Correlation coefficient
R_L	-	Separation factor
t	-	Time
$t^{1/2}$	-	Half-life time
V	-	Volume
W	-	Weight of adsorbent
wt%	-	Weight percent
ΔG	-	Gibbs Free Energy
ΔH	-	Entropy
ΔS	-	Entropy

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CHAPTER 1

INTRODUCTION

1.1 Background Study

The broad use of pigments and dyes began as in response to the development of paper, printing, pharmaceutical, cosmetic and the calfskin and textile dyeing industries. Each year, around 10,000 unique colours, which measures to nearly 0.7 million tons, are delivered every year for different industrial purposes. However, as a result, a significant number of these colours are eliminated along with waste, particularly during the colouring process. As documented by Nethaji *et al.,* (2010), many of these eliminated dyes can be distinguished as harmful or even carcinogenic, in which they can cause cancers among those who are exposed to them. Hence, the elimination of these colour substances bodies of water can cause contamination which make them unfit for the live of animals such as amphibians and fishes.

As a result, the large scale use of synthetic dyes in industries such as pharmaceutical, textile, food cosmetic, as well as other different commercial enterprises have created worrying environmental issues. According to Gao *et al.*, (2015), an estimated 35,000 tonnes of dyes are eliminated annually into the natural habitat of amphibians. Furthermore, the presence of these colours can also bring potential harms to the wellbeing of humans, since they can affect the health of vital organs such as central nervous system, kidney, brain and liver. In the meantime, a vast majority of dyes are made out of complex fragrant structures, which cause inherent difficulties in biodegrading these contaminants.

Meanwhile, water contamination is an imminent crisis in today's society. This happens as the increase of both industrial and agricultural activities have generated different types of harmful toxins to be released into the environment, which caused a global water contamination crisis. Thus, as claimed by Anastopoulos *et al.,* (2014), polluted wastewater should treated and purified before it is released into the environment, especially rivers and beaches.

The contamination of water by both metals and dyes should be considered as an environmental crisis. This is because the toxins in these contaminants interfere with the natural properties of water and are poisonous to flora, fauna and individuals. For instance, some dyes may be carcinogenic, the exposure towards metals are also dangerous, even at a generally low concentration. Hence, the combination of new activated carbons for water treatment is a continuous research subject in ecological contamination which takes into account these realities (Cordero *et al*., 2014).

Dyes are an important type of pollutants large amounts which originated from industries such as textile, paper and pulp making, tanning and petroleum. They are mainly used to modify the colour characteristics of different substrates such as paper, fabric and leather. As demonstrated by Anastopoulos *et al.,* (2014), the presence of dyes in the environment can largely affect the photosynthetic activity. Besides that, many dyes are toxic and carcinogenic, which affects the aquatic biota and human health.

In order to eliminate dyes from wastewater, various methods, including adsorption, coagulation, advanced oxidation, and membrane separation have been used. Consequently, adsorption is considered as one of the most effective processes in advanced treatment of wastewater Thus, many industries mainly used the adsorption techniques in the tertiary stage of biological treatment, in order to eliminate dangerous inorganic or organic pollutants in wastewaters. Anastopoulous *et al.,* (2014) mentioned that a lot of researches have been recently published and primary goal of these researches is the elimination of different pollutant, either in gas or liquid, through the use of adsorbent materials.

Consequently, various researchers have garnered interests in researching the utilisation of adsorption processes, in particularly those employing alternative adsorbents materials. This is because, adsorption, from the operational perspective, is advantageous over other treatment processes in eliminating soluble substances such as dyes, heavy metals and phenolic compounds from wastewaters. According to Piccin *et al.,* (2016), among these advantages are the low initial investment required, the simplicity of its design and its operation, the lower energy use and its higher efficiency when compared to other conventional and other non-conventional processes in reducing contamination and toxicity of the treated wastewater.

In the meantime, a majority of researches focusing on adsorption has utilised aqueous solutions which contains “synthetic effluent” as the adsorbate of interest. Hence, most of these researches do not considered the complexity of the real effluents in optimising the process. Therefore, researches using a single component, as well as multi component researches which use a limited number of components, are interesting as they considered the multiple perspectives in the process of obtaining information, in regards to the mechanisms and nature of the process. However, as mentioned by Piccin *et al.,* (2016), this type of study can lead to imprecise results as the technology transfer implemented do not consider the optimization of the processes for their application in treating industrial wastes.

In the meantime, because of its efficiency, commercial-grade activated carbon is the most commonly used adsorbent for the elimination of colour in wastewater. On the other, its vast use is hindered because of its high cost. Moreover, powdered activated carbons are very difficult to dispose of as it consist of very fine powder that can remain suspended in treated water for a long period of time. Hence, as a measure to reduce the economical and environmental cost of such treatment, several alternative adsorbents have been studied. These alternative adsorbents are considered as low-cost, as they are either abundant in nature, or derived from an industrial by-product. As mentioned by Sonai *et al.,* (2016), these adsorbents can be made from seaweed, peat, chitosan leaves, and waste from the agricultural industry, sawdust, and sludge.

1.2 Problem Statement

According to Crini (2006), decolorization and/or bioadsorption of dye wastewater by (dead or living) biomass, white-rot fungi and other microbial cultures was the subject of many studies reviewed in several recent papers. In particular, these studies demonstrated that biosorbents derived from suitable microbial biomass can be used for the effective removal of dyes from solutions since certain dyes have a particular affinity for binding with microbial species. In spite of good sorption properties and high selectivity some problems can occur. The sorption process is slow: in the case of biomass of *Aspergillus niger* equilibrium was reached in 42 h. Another problem is that the initial pH of the dye solution strongly influenced the biosorption. Biosorption was also influenced by the functional groups in the fungal biomass and its specific surface properties. Biosorption performance depends on some external factors such as salts and ions in solution which may be in competition. Other limitations of the technology include the fact that the method has only been tested for limited practical applications since biomass is not appropriate for the treatment of effluents using column systems, due to the clogging effect. Because of major limitations regarding its efficient utilization in a column reactor, there is a need for it to be immobilized. This step forms a major cost factor of the process.

In the meantime, the pandan plant can easily found throughout the year as it thrives in the hot and humid weather of Malaysia. The plant normally grows in damp, swampy areas and commonly seen in villages where it grows wild and in abundant. Meanwhile, the urban area residents normally cultivated the plant in pots. The abundance of pandan in the local area has generated the interest to study the use of adsorbent, which is derived from pandan leaves, in the adsorption of the Methylene Blue (MB) dye. Consequently, different types of methods are employed to produce the adsorbent and to enhance the capacity of the dye's adsorption.

1.3 Objectives

The research objectives are:

1. To synthesize and characterize adsorbent from pandan leaves.

2. To test the performance of the synthesized adsorbent towards removal of methylene blue dye.

3. To analyze kinetic, isotherm and thermodynamic of the adsorption.

1.4 Research Scope

The synthesis of pandan leaves adsorbent was formed by chemical activation method. Initially, screening was done with two different activating agents which are $ZnCl_2$ and NaOH with the same concentration of 0.05 M. The best chemical activating agent will selected to be utilized in the synthesis of pandan leaves adsorbent for the methylene blue dye adsorption study.

This research is about synthesis adsorbent from pandan leaves. Adsorbent that been develop is used in dye removal process by using adsorption method. The chemical and physical properties of pandan leaves activated carbon were studied through the characterization of the adsorbent. The characterization involves are Fourier Transform Infrared (FTIR) spectroscopy and Brunauer–Emmett–Teller (BET).

The performance of pandan leaves as the adsorbent in the removal of methylene blue dye from aqueous solution were performed according to a batch adsorption method. Various parameters involved in the investigation of optimum condition for adsorption such as, effect of reaction time (10 – 180 min), pH (2 – 11) adsorbent dosage (0.02 – 0.1 g), initial dyes concentration (2 – 10 mg/L), and temperature (30, 50, and 60 ºC).

The equilibrium isotherms studied by the Langmuir and Freundlich isotherms models. The Pseudo-first order, Pseudo-second order and intraparticle diffusion were applied to experimental data in order to clarify the adsorption kinetic dyes onto adsorbent. The thermodynamic parameters of the adsorption of methylene blue dye including the Gibbs free energy (ΔG), enthalpy (ΔH), and entropy (ΔS) were calculated using Van’t Hoff equation.

1.5 Research Significance

The leather, textile, paper and pulp industries eliminate a large quantities of highly coloured wastewater. These wastewater contain dyes which can pollute into the nearby rivers or land area. Most of these wastes are untreated since conventional treatment methods can incur a high operating cost. As a result, traces of dyes are still present in the environment. This can be dangerous as even when a small amount of dyes present in the water (<1 mg/L), they are highly visible. Furthermore, based on their chromophore group, an estimated 20–30 different groups of dyes are considered as pollutants; and hence, mentioned in Ahmad *et al.,* (2015), the elimination colour from wastewater has become one of the major environmental concerns.

Industries wastewater from the textile, leather, paper, ink and cosmetic industries are well are severely contaminated with dyes, surfactants pigments, and many other toxic chemicals. Ultimately, these contaminated wastewater ultimately will flow to the surface water reservoir and can lead to damaging consequences. This is because, dye-contaminated water, even those that contain very low concentration of dyes, are highly visible and aesthetically unpleasing. Furthermore, most dyes are toxic and can primarily contaminate surface water. Consequently, water biota is the primary victim of dye contamination, where long exposures to dyes in the water can lead to the contamination of the food chain and adverse health effects. Hence, as proposed in Asadullah *et al.,* (2013), there is a mandatory need to reduce the concentration of contaminant in wastewater to below the acceptable range before the water are released into the environment through the use of appropriate treatment process.

In the meantime, Methylene blue (MB), is a basic dye, which is initially used to dye materials such as paper, silk, plastics, leather, and cotton mordant with tannin. Moreover, it is also used by the office supplies industry in the production of inks and copying paper. The elimination of MB dyes into the environment has dire toxicological and aesthetical reasons. For instance, MB dyes can hinder the penetration of light, damage the quality of the receiving streams as well as being toxic to the organisms in the food chain organisms. On the other hand, the elimination of dyes from wastewater is challenging as they have a synthetic origin and possess complex aromatic molecular structures which caused them to structurally inert and difficult to biodegrade, and unfortunately, the industries have overlooked this aspect in the treatment of wastes. Consequently, the removal of synthetic dyes garnered great concern from researchers, industry champions and the public as dyes and the by-product of their degradation process may be carcinogenic and toxic to humans and animals. As a result, Kumar *et al.*, (2011) advocated that the treatment of wastewater with dyes cannot just solely depend on biodegradation.

1.6 Thesis Outline

This study consist of five chapters, which present the research in sequential order. Chapter 1 introduces the background study, problem statement, objectives, scopes, and research significance of this study. Chapter 2 reviews the literature those related to anionic and cationic dyes, adsorbent, and current issues about the adsorption process. Chapter 3 describes the experimental procedures and characterization of the raw materials and adsorbent. Chapter 4 analyzes and discusses the characterization and experimental results. Finally, Chapter 5 is the conclusion and recommendation for further study about this research.

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