

**EQUILIBRIUM, KINETICS AND THERMODYNAMICS OF ACID DYE
ADSORPTION ON PALM OIL EMPTY FRUIT BUNCH**

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UNIVERSITI TEKNOLOGI MALAYSIA

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**A project report submitted in partial fulfilment of the
requirements for the award of the degree of
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*Specially dedicated to
my dearest family members for the encouragement, support and inspiration
throughout my journey of education.....*

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In the name of Allah, the Most Gracious and the Most Merciful.

All praised is to Allah S.W.T the sustainer of the world and may there be His blessing to all messengers and His last messenger, the prophet Muhammad S.A.W and his family, companies, followers and the entire believer till the end of time.

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ABSTRACT

A low-cost, easily obtained and eco-friendly adsorbents has been investigated as an ideal alternative to the current expensive methods such as using activated carbon for removing dyes from wastewater. This study investigated the use of palm oil empty fruit bunch (POEFB) fibre, an agricultural waste, for the removal of Acid Orange 51 (AO51) dye from aqueous solutions. Batch mode experiments were conducted at room temperature (28 ± 2)°C to study the effect of particle size (75 μm , 250 μm and 2 mm), initial dye concentrations (10-200 mg/L) and temperature (30-50 °C) of adsorption characteristics of AO51 dye onto treated and non-treated POEFB fibre. It was found that the adsorption removal of AO51 dye by treated POEFB fibre was ~5%, which was far lower than that of ~60% on the untreated POEFB fibre. The equilibrium adsorption isotherms and kinetics were further investigated for untreated POEFB fibre. The adsorption equilibrium data were analyzed by Langmuir, Freundlich and Temkin isotherm models. The results indicated that the equilibrium sorption fitted well with the Freundlich isotherm, displaying higher regression coefficient, R^2 value. Adsorption kinetic data were modeled using the pseudo-first-order, pseudo-second-order and intraparticle diffusion models. It was shown that pseudo-second-order kinetic model could best describe the adsorption kinetics. Isotherms have also been used to obtain the thermodynamic parameters, namely enthalpy and entropy of adsorption. The result showed negative value for both enthalpy (-13.91 kJ/mol) and entropy (-38.43 J/mol K) changes. Based on the isotherm studies, it can be concluded that the adsorption of AO51 dye onto untreated POEFB is favorable via physical adsorption. Thermodynamically, the final adsorbed state of the dye molecule is lower in energy compared to initial state. These results indicate the potential of using POEFB fibre as a low-cost adsorbent material for adsorption of the AO51 dye from aqueous solutions.

ABSTRAK

Bahan penyerap kos rendah yang mudah di perolehi dan mesra alam telah dikaji sebagai alternatif yang ideal kepada kaedah semasa yang mahal seperti menggunakan karbon teraktif untuk menyingkirkan pencelup daripada air sisa. Kajian ini mengkaji penggunaan serat buah tandan kosong kelapa sawit, yang merupakan sisa pertanian, untuk menyingkirkan pencelup 'Acid Orange 51' (AO51) daripada larutan air. Ujikaji secara kelompok telah dilakukan pada suhu bilik (28 ± 2 °C) untuk mengkaji kesan saiz zarah ($75\ \mu\text{m}$, $250\ \mu\text{m}$ dan $2\ \text{mm}$), kepekatan awal pencelup ($10\text{-}200\ \text{mg/L}$), dan suhu ($30\text{-}50$ °C) bagi ciri-ciri penyerapan pencelup AO51 ke atas serat buah tandan kosong kelapa sawit yang dirawat dan tidak dirawat. Didapati bahawa penyerapan penyingkiran pencelup AO51 pada serat buah tandan kosong kelapa sawit yang dirawat adalah $\sim 5\%$, iaitu sangat rendah berbanding dengan $\sim 60\%$ bagi serat buah tandan kosong kelapa sawit yang tidak dirawat. Keseimbangan penyerapan isoterma dan kinetik dikaji dengan lebih lanjut bagi serat buah tandan kosong kelapa sawit yang tidak dirawat. Data keseimbangan penyerapan dianalisis menggunakan model isoterma 'Langmuir', 'Freundlich' dan 'Temkin'. Hasil menunjukkan bahawa keseimbangan penyerapan berpadanan baik dengan model isoterma 'Freundlich', memaparkan nilai pekali regresi, R^2 , yang paling tinggi. Data kinetik penyerapan dimodelkan menggunakan model pseudo tertib pertama, pseudo tertib kedua dan resapan intrazarah. Keputusan menunjukkan bahawa model kinetik pseudo tertib kedua adalah terbaik yang boleh menggambarkan kinetik penyerapan. Isoterma juga digunakan untuk memperoleh parameter termodinamik, yang dinamakan penyerapan entalpi dan entropi. Keputusan menunjukkan nilai negatif untuk kedua-dua perubahan entalpi ($-13.91\ \text{kJ/mol}$) dan entropi ($-38.43\ \text{J/mol K}$). Berdasarkan kepada kajian isoterma, dapat disimpulkan bahawa penyerapan pencelup AO51 ke atas buah tandan kosong kelapa sawit yang tidak dirawat adalah baik melalui penyerapan fizikal. Secara termodinamikanya, keadaan akhir terjerap bagi molekul pencelup adalah lebih rendah dalam aras tenaga

dibandingkan dengan keadaan awal. Keputusan ini menunjukkan potensi menggunakan serat buah tandan kosong kelapa sawit sebagai bahan penyerap kos rendah untuk penyerapan pencelup AO51 daripada larutan air.

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LIST OF ABBREVIATIONS

AO51	- Acid Orange 51
BDDT	- Brunauer, Deming, Deming and Teller
BET	- Brunauer, Emmett and Teller
EFBU-75	- Untreated palm oil empty fruit bunch with particle size 75 μm
EFBU-250	- Untreated palm oil empty fruit bunch with particle size 250 μm
EFBU-2000	- Untreated palm oil empty fruit bunch with particle size 2000 μm
EFBT-75	- Treated palm oil empty fruit bunch with particle size 75 μm
EFBT-250	- Treated palm oil empty fruit bunch with particle size 250 μm
EFBT-2000	- Treated palm oil empty fruit bunch with particle size 2000 μm
FTIR	- Fourier Transform Infrared Spectroscopy
IUPAC	- International Union of Pure and Applied Chemistry
KBr	- Potassium bromide
POEFB	- Palm oil empty fruit bunch
SEM	- Scanning Electron Microscopy
UV-Vis	- Ultraviolet-Visible Spectroscopy

LIST OF SYMBOLS

a	-	Langmuir constant related to the affinity of the binding site
A	-	Equilibrium binding constant corresponding to the max binding energy
A	-	Absorbance
A_m	-	Average area of one adsorbate molecule in the complete monolayer
b	-	Temkin isotherm constant
b_B	-	Path length/sample cell length
C	-	Concentration
C_0	-	Initial concentration
C_e	-	Equilibrium concentration
C_{Ae}	-	Amount adsorbed on the solid at equilibrium
Hz	-	Hertz
i	-	Intercept of a linear graph
k_1	-	Rate constant of pseudo-first-order adsorption
k_2	-	Rate constant of pseudo-second-order adsorption
k_{diff}	-	Intraparticle diffusion rate constant
k_d	-	Distribution coefficient
K_L	-	Langmuir equilibrium constant
K_F	-	Empirical constant in Freundlich equation
m	-	Weight of adsorbent
M	-	Molar
N_0	-	Avogadro number
P	-	Gas pressure
P	-	Equilibrium pressure
P^0	-	Saturation pressure

q_e	-	Ratio of the weight of adsorbed adsorbate to the weight of adsorbent
q_{max}	-	Maximum adsorption at monolayer coverage
q_t	-	Amount of adsorbate adsorbed at time
R	-	Gas constant (8.314 J/mol K)
R_L	-	Separation factor
R^2	-	Correlation coefficient
s	-	Slope of a linear graph
S	-	Specific surface area
S_{BET}	-	BET surface area
T	-	Temperature
V	-	Volume
V_m	-	Volume of gas adsorbed when monolayer coverage is formed
x	-	Mass of adsorbate adsorbed
ε	-	Molar adsorptivity
ΔH°	-	Enthalpy change
ΔS°	-	Entropy change
λ	-	Wavelength
λ_{max}	-	Wavelength at which the absorbance is maximum

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CHAPTER I

INTRODUCTION

1.1 Research Background

Environmental pollution problems in Malaysia have a long history. One of the major problems concerning environmental pollutants is wastewater problem. Wastewater comes from domestic and industry. In industry, the biggest sources of industrial wastewater come from textiles, leather, paper, plastic and other dyeing industries.

The presence of dyes in effluents is a major concern due to their adverse effects to many forms of life. The discharge of dyes in the environment is a matter of concern for both toxicological and esthetical reasons. Industries such as textile, leather, paper and plastics use dyes in order to colour their products and also consume substantial volumes of water. As a result, they generate a considerable amount of coloured wastewater. It is estimated that more than 100,000 commercially available dyes with over 7×10^5 tonnes of dyestuff produced annually and from 40,000 to 50,000 tonnes of dye are discharged to surface water every year (Parvathi and Maruthavanan, 2010). It is recognized that the public perception of water quality is greatly influenced by the colour. The colour is the first contaminant to be recognized in wastewater. The presence of even very small amounts of dyes in water (less than 1 mg/L for some dyes) is highly visible and undesirable (Pavan *et al.*, 2007; Rafatullah *et al.*, 2009). Due to large-scale production and extensive application, synthetic dyes can cause considerable environmental pollution and are serious health-risk factors (Forgacs *et al.*, 2004).

Most dyes are non-biodegradable in nature, which are stable to light and oxidation. Therefore, the degradation of dyes in wastewater either traditional chemical or biological process has not been very effective to treat these effluents. Lots of techniques had been developed in wastewater treatment such as electrolyte extraction, chemical precipitation, evaporation, cementation, adsorption and filtration. Adsorption is the most practised technique among several chemical and physical methods (Alam *et al.*, 2008).

Activated carbon was once a popular choice for removing heavy metals and dyes from wastewater (Babel and Kurniawan, 2003). However, commercially available activated carbon is still considered expensive. This is due to the use of non-renewable and relatively expensive starting material such as coal, which is unjustified in pollution control applications (Martin *et al.*, 2003). This high cost for producing activated carbon had made an essential for researchers to find alternative adsorbents that are lower in cost and as comparable effective as activated carbon.

1.2 Problem Statement

Adsorption on activated carbon is a well-recognized method for the treatment of water and wastewater. The high material cost, however, limits its use in wastewater treatment. Therefore, in recent years, innovative adsorption using locally available low cost adsorbent for dyes removal has been extensively investigated. New economical, easily available and highly effective adsorbents are still needed.

Currently, researchers have worked on palm oil empty fruit bunch as an effective adsorbent for dyes removal. Hameed and El-Khaiary (2008), successfully utilized palm oil empty fruit bunch as a low-cost adsorbent for the removal of hazardous dye like malachite green. The equilibrium adsorption data were analyzed by the Freundlich isotherm, the Langmuir isotherm and the multilayer adsorption isotherm. Ofomaja and Ho, (2007), also revealed the potential of palm oil empty fruit bunch as a good sorbent for the anionic dyes in wastewater.

Several techniques have been used for the treatment of palm oil empty fruit bunch as an effective adsorbent for dyes removal from wastewater such as modifying the palm oil empty fruit bunch with silylation by trimethylchlorosilane (TMCS) and diethyldichlorosilane (DEDCS) (Rattanawong *et al.*, 2007). Abia and Asuquo (2007), carried out research using mercaptoacetic acid modified palm oil empty fruit bunch adsorbent. Guo and Lua (2003), carried out research by investigating the effects of activation temperature and phosphoric acid impregnation on the textural and chemical properties of the prepared adsorbent.

In other research, the potential use of palm oil empty fruit bunch pretreated with formaldehyde and sulphuric acid for the removal of methylene blue (Saad *et al.*, 2007a) and methyl red (Saad *et al.*, 2007b) dyes from simulated wastewater were also carried out. The data may be useful for designing and fabrication of an economically cheap treatment process.

However, no studies have been reported on the use of palm oil empty fruit bunch for the removal of acid dyes from wastewater. Thus, this study is undertaken to investigate the sorption equilibrium, kinetics and thermodynamics of Acid Orange 51 dye onto palm oil empty fruit bunch.

1.3 Objective of the Study

The objective of this study is to investigate the equilibrium, kinetics and thermodynamics of Acid Orange 51 adsorption onto untreated and acid treated palm oil empty fruit bunch.

1.4 Scope of the Study

In the preparation of treated palm oil empty fruit bunch adsorbent, many factors may affect the percent yield and the quality of product. These factors include the type of chemical activating agent, amount of activating agent, activation temperature and time, as well as washing method. Sulphuric acid (H_2SO_4) was used as the activating agent with the palm oil empty fruit bunch with the ratio 1:1. The activation process was carried out in a muffle furnace for 24 hours at 150 °C.

The properties of the prepared adsorbent were characterized by Fourier Transformed Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM) and Nitrogen Adsorption analysis at 77K. The concentration of adsorbate was determined using the Ultraviolet-Visible (UV-Vis) Spectroscopy.

The prepared empty fruit bunch adsorbent was used to adsorb acid dye for the adsorption capacity study. In this study, Acid Orange 51 dye was used as adsorbate. The adsorption capacity of the acid dye onto different particles size of both prepared adsorbents, untreated empty fruit bunch (EFBU) and treated empty fruit bunch (EFBT), were determined and compared. The equilibrium data of the adsorption process was studied using Langmuir, Freundlich and Temkin isotherm while the kinetic of adsorption process was evaluated using Pseudo-first-order, Pseudo-second-order and intraparticle diffusion model. The effects of temperature were also investigated in this study in order to evaluate the adsorption thermodynamic. From this study, the effectiveness of the palm oil empty fruit bunch as adsorbent of removal acid dye was determined.

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