PREPARATION, MODIFICATION AND CHARACTERIZATION OF ADSORBENTS FROM PALM FATTY ACID DISTILLATES (PFAD) FOR DYE REMOVAL FROM AQUEOUS SOLUTIONS

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DEDICATION

To my beloved wife, daughters, and sons,

for understanding the many nights, I was away thanks!

To God be the glory.

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ABSTRACT

Adsorption is an effective approach in the treatment of wastewater as it utilizes low cost adsorbent, no sludge, and is simple to operate. Palm fatty acid distillates is a suitable precursor of adsorbents due to its high carbon content, less commercial value and abundant availability. This work is aimed to evaluate the adsorption properties of adsorbents prepared from palm fatty acid distillates for methylene blue and congo red removal. Adsorbents with more yield were selected with the aid of response surface methodology (RSM) using Box Behnken design (BBD) approach. The resultant adsorbents were characterized for ultimate analysis, thermogravimetric analysis, surface area analysis, Fourier transform infrared spectroscopy analysis, surface morphology analysis, X-ray spectroscopic property, Boehm titration and point of zero charge of adsorbents. The surface area was between 212-222 m^2/g, pore volume was 7.0 $x10^{-7}$-2.8 $x10^{-7}$ m^3/g, pore size 51-122 A and the pore structure was majorly micropore, Boehm titration showed the presence of carboxylic, lactonic, phenolic and basic groups on the surface. The pH of adsorbent was 5 while the point of zero charge was 5.5. The adsorption of methylene blue and congo red were studied at varying concentrations (5 – 300 mg/L), contact time (10min – 72 h), temperature (30 -70 °C) and pH (2 -10). RSM software using BBD approach was utilized to determine the optimum adsorption capacity of methylene blue and congo red by adsorbents and to know interrelationships and interactions among the factors that enhance maximum adsorption. The optimum adsorption of methylene blue was 47.8 mg/g at maximum initial concentration of 100 mg/L, temperature of 70 °C, and contact time of 18 h and pH of 6 compared with experimental value of maximum methylene blue adsorption of 56 mg/g at concentration of 100 mg/g, temperature of 70 °C, time of 36 h and pH of 5 was found to be suitable for methylene blue adsorption. Congo red adsorption was 189 mg/g at concentration of 300 mg/L, temperature of 70 °C contact time of 15 h and pH of 6. The experimental value of congo red optimum is 237 mg/g at concentration of 300 mg/L, temperature of 70 °C, time of 30 h and pH of 5. The model is suitable for congo red. The adsorption of methylene blue and congo red dyes by adsorbents was well described by Langmuir and Temkin models. The adsorption of dyes fitted into both pseudo-first order and pseudo-second order kinetic models, suggesting physiscochemical adsorption. Weber-Morris or intraparticle diffusion model reveals that the intraparticle diffusion is involved, but it is not the only rate-limiting step. Boyd model shows that film diffusion is the controlling mechanism for both methylene blue and congo red adsorption. The dyes adsorption is thermodynamically endothermic and spontaneous in nature.

ABSTRAK

Penjerapan merupakan pendekatan yang berkesan bagi rawatan air sisa kerana ia menggunakan penjerap kos rendah, tidak menjana enapcemar, dan mudah untuk digunakan. Asid lemak sawit tersuling adalah bahan penjerap yang sesuai kerana mempunyai kandungan karbon yang tinggi, kurang nilai komersial dan banyak tersedia. Kajian ini bertujuan untuk menilai sifat penjerapan penjerap yang dirumus daripada rawatan asid lemak sawit tersuling untuk penyingkiran metilena biru dan kongo merah. Penjerap dengan hasil jerapan yang tinggi telah dipilih dengan bantuan kaedah sambutan permukaan (RSM) menggunakan pendekatan Box-Behnken (BBD). Penjerap terhasil dicirikan untuk analisis muktamad, analisis termogravimetrik, analisis kawasan permukaan , spektroskopi inframerah jelmaan Fourier, analisis kumpulan berfungsi, analisis morfologi permukaan, sifat spektroskopik sinar-X, titrasi Boehm dan titik sifar caj penjerap. Luas permukaan penjerap adalah antara 212-222 m^2/g, isipadu liang adalah 7.0 $x10^{-7}$-2.8 $x10^{-7}$ m^3/g, saiz liang adalah 51 -122 A dan struktur utama liang adalah liang-liang mikro, titrasi Boehm menunjukkan kewujudan kumpulan karbosilik, laktonik, fenolik dan kumpulan-kumpulan asas atas permukaan. pH penjerap adalah 5 manakala titik sifar caj adalah 5.5. Penjerapan metilena biru dan kongo merah dikaji pada pelbagai kepekatan (5 - 300 mg/L), masa sentuhan (10 min - 72 jam), suhu (30 - 70 °C) dan pH (2 - 10) yang berbeza-beza. Perisian RSM dengan pendekatan BBD telah digunakan untuk menentukan kapasiti penjerapan yang optimum untuk metilena biru dan kongo merah dan untuk mengetahui hubungan dan interaksi antara faktor-faktor yang meningkatkan penjerapan maksimum. Penjerapan optimum metilena biru adalah 47 mg/g pada kepekatan awal maksimum 100 mg/L, suhu 70 °C, 18 jam masa sentuhan dan nilai pH 6 berbanding dengan nilai kajian dimana penjerapan maksimum metilena biru adalah 56 mg/g pada kepekatan 100 mg/g, suhu 70 °C, 36 jam dan pH 5 adalah sesuai untuk penjerapan metilena biru. Penjerapan kongo merah adalah 189 mg/g pada kepekatan 300 mg/L, suhu 70 °C, masa sentuhan 15 jam, dan pH 6. Nilai kajian menunjukkan penjerapan optimum kongo merah adalah 237 mg/g pada kepekatan 300 mg/L, suhu 70 °C, masa sentuhan 30 jam dan pH 5. Model ini sesuai untuk penjerapan kongo merah. Penjerapan metilena biru dan kongo merah oleh penjerap juga telah dijelaskan dengan baik oleh model Langmuir dan Temkin. Penjerapan pencelup ini sesuai untuk kedua-dua model kinetik pseudo tertib pertama dan tertib kedua, mencadangkan penjerapan fizikokimia. Weber-Morris atau model resapan intrazarah mendedahkan bahawa penjerapan intrazarah juga terlibat, tetapi ia bukan hanya langkah yang menghad kadar. Model Boyd menunjukkan bahawa penyerapan lapisan merupakan mekanisma kawalan bagi penjerapan metilena biru dan kongo merah. Penjerapan pencelup adalah endotermik secara termodinamik dan bersifat spontan.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Industrial growth, urbanization expansion and general increase in world population have led to increase in waste generation from industry as a result of the increase in industrial production activities to meet the ever-increasing demands for industrial products for man's use and consumption. These have contributed greatly to surface water and underground water becoming contaminated, harmful and polluted in most countries of the world with Malaysia not being an exception (Afroz *et al*, 2014; Afroz & Rahman, 2017; Alsaffar *et al*, 2016; Nurul-Ruhayu *et al* 2015).

In Malaysia, industries that produce appreciable volumes of wastewater include refining and petrochemical industries, palm oil and palm oil refinery industries, poultry and meat processing industries, chemical and fertilizer companies, paper and fiber industries, textile, steel mills, mineral processing industries and a host of others. Malaysia has undergone a period of rapid growth in the manufacturing sector since the 1960s and is continuing to attract new industries to the country. This growth has been accompanied by a corresponding increase in the number of industries generating hazardous waste. The contribution of the manufacturing industry to the Gross Domestic Product increased from 11% in 1966 to 24% in 1988. In 1987 Malaysian industry produced nearly 400,000 m^3 of hazardous waste, more than half coming from the states of Selangor and Penang (Yang *et al*., 2016; Aja, *et al* 2016; Ong *et al* 2018).

Water pollution has therefore become a serious problem to mankind and the ecosystem (Chen *et al*, 2016; Dai *et al*., 2018). Different techniques of wastewater treatment have been suggested and applied in the past decades. Adsorption has been a preferred method for obvious reasons of low cost, simplicity of equipment and process, versatility and easy way of operation (Anastopoulos *et al,* 2017; Bhatnagar & Anastopoulos, 2017; Nasrullah *et al*., 2019). Low cost adsorbent materials can be obtained from different sources namely agricultural waste source, natural source, industrial waste/by-products source and agro-industrial waste/by-products source.

Agricultural waste is obtained from production of residues in agricultural and forestry processing. Examples are husks, straw, grouts and crumbs. The large quantity of agricultural wastes has many advantages in wastewater treatment such as low cost, environmental friendliness, chemical stability, abundant source, short regeneration cycle and green energy. Additionally, their surface has large specific area and high porosity. has made adsorption easier. Also their cell structures contain some active chemicals like pectin, hemicellulose, cellulose and lignin which could react with some chemical ions in waste water for an easy removal (Abdelhafez & Li, 2016; Basu *et al*, 2017; Guiza, 2017; Qu *et al*, 2018; Saxena *et al*, 2017).

Natural clay minerals are considered as adsorbents because they are abundant natural resources, low cost and great capacity for adsorption and ion exchange. Clay materials with layer structures are the preferred materials for adsorbent production. The adsorption capacity of clay minerals emanates from the negative charge of their surface structure and can adsorb positively charged ions in the sewage. Moreover, adsorption properties emanated from high surface area and good porosity. Over many years, the preparation and utilization of clay has attracted researchers' interest and the interactions between pollutants and clay particles have been studied. Clay minerals can effectively adsorb both organic and inorganic pollutants (Devi & Saroha, 2017; Uddin, 2017).

Industrial waste has been used to prepare adsorbents due to its low cost, rich sources and wide distribution. Sludge, red mud fly ash etc. are common samples of industrial waste. Chemical treatment or modification on these wastes can enhance their adsorption capacity, can equally be used to adsorb hazardous chemicals from waste water (Gwenzi *et al.,* 2017; Giraldo *et al.*, 2014; Simate *et al., 2016).* Industrial waste can also be used to produce adsorbents for dyes removal from waste industrial water (Silva *et al*., 2018).

Activated carbon has been used for waste water treatment for a long period of time, research is gradually changing towards developing low-cost agro-industrial wastes/by-products based adsorbents such as palm kernel shell, palm oil mill effluent, EFB, coconut waste, fly ash, slag sludge, palm fatty acid distillates (PFAD), etc.(Devi & Saroha, 2017; Hafshejani *et al.*, 2016; Faridah *et al*., 2018; Hidayu & Muda, 2016; Runtti, 2016; Tay, 2009; Wirasnita *et al*, 2014).

There are large amount of agricultural wastes and industrial by-products that create environmental challenges in various form (soil, air and water) (Deng *et al.*, 2015). Agro industrial wastes/by-products are produced from industries that use agricultural produce as raw material for the production activities. The waste/by-products from such companies are called agro industrial wastes. Ineffective treatment of such waste and availability may cause serious environmental challenges (Anastopoulos et al., 2017). The treatment methods available are expensive, complex and can easily cause secondary pollution. Most of these by-products can be transformed into reuse materials. This will reduce pollution challenge to a large extent (Li *et al.*, 2017; Omo-Okoro *et al.*, 2018; Silva *et a.*, 2018a) . The focus of this study is the conversion of palm fatty acid distillates (PFAD), a semi-solid by-product obtained in the course of converting crude palm oil to refined vegetable oil by palm oil refinery companies, to adsorbents for the removal of dyes from wastewater.

Thus, the use of low-cost adsorbents derived from industrial by-products for waste removal from waters is desirable. The industrial by-products from palm oil refinery industries are abundantly available and cheap and are underutilized as a result of growth and expansion in palm oil processing and refining industries. This explains the choice of the by- product from palm oil refinery industry, i.e., palm fatty acid distillates (PFAD), for the production of low-cost adsorbents for the treatment of waste water as the main focus of this research work

1.2 Problem Statement

Increase in the operations and activities of industries in order to meet the demand of ever growing population and industrial consumptions of raw materials have led to the increase in waste or by-products generation of palm oil and palm oil refinery industries. Industrial wastes from this sector of production are on the increase and currently under-utilized and in recent years have become a serious environmental challenge due to introduction of more federal and state legislation regulating their disposal

In the same vein, industrial wastewater containing dyes are a serious health and environmental risk. Most dyes used in textile industries are stable to light and are not biologically degradable. In order to minimize the risk of pollution problems from such effluents, it is important to adequately treat them before discharging them to the environment. Research activities are ongoing for some years now to develop cheaper and more effective adsorbents. As agricultural waste and industrial by-products are cheaper and available, activities towards different alternative materials for adsorbents have increased (Bhatnagar *et al.*, 2015). Clay materials like, zeolites kaolinite bentonite, perlite (Ruiz et al., 2014). Agricultural waste materials like pulp, corn cob, rice husk, coconut shell; industrial wastes/by-products like palm fatty acid distillates, palm kernel cake, waste carbon cyclodextrin, cotton are used (Acquah *et al*, 2016; Hadi, *et al*., 2015; Simate *et al.*, 2016). Biological processes are not considered to be the best method for the removal of waste from the industrial wastewater. Lately, adsorption processes have begun to gain considerable importance in the treatment of industrial wastewater. Adsorbents are very effective and widely used due to their excellent adsorption ability, cost and availability (Sze, et al., 2015). Currently, by-products from industries have not been fully exploited for adsorbents preparation for pollution control, this explains why this study focusses on palm fatty acid distillates (PFAD), a by-product from palm oil refinery for pollutants removal.

Adsorption has been discovered to be the cheap and widely used method in wastewater treatment and superior to chemical and physical treatment methods in terms of ability to remove non-biodegradable contaminants from wastewaters (Bhatnagar et al., 2015). The reasons being that the raw materials for production are abundant, cheap and simple at the same time the adsorption capacity is very high. Some adsorbents have low yield and low adsorption capacity, this explains why adsorbents are exposed to physical, chemical or combination of both methods in order to increase the yield and adsorption capacities. Palm fatty acid distillate is treated chemically in order to increase the yield and adsorption capacities of adsorbents prepared from it.

In an attempt to utilize the benefit associated with adsorbents, the use of abundantly, locally available, low cost adsorbent derived from palm oil refinery by-products are proposed in this study. In addition, the low cost adsorbent proposed as single use material to avoid regeneration problems. After use, the loaded adsorbent could be disposed of by land filling or incineration. The precursor, being a new source of preparing adsorbent, the performance evaluation and the effect of parametric variables on the performance and the characteristic properties of the adsorbent will be a subject of study in this research work.

1.3 Aim and Objectives

This research work is aimed at evaluating the adsorption capacity and performance of adsorbents produced from palm fatty acid distillates (PFAD) in the removal of dyes from aqueous solutions.

The specific objectives are;

I. To prepare and characterized adsorbents from palm fatty acid distillates (PFAD) by carbonization and chemical modification with zinc chloride at temperature range of 500 ^{o}C-600 °C and compare the characteristic properties.

II. To evaluate the variables leading to the optimum production of adsorbents by using response surface methodology (RSM) and design of experiment (DOE).

III. To assess the efficiency of adsorbents for methylene blue and congo red removal at different initial concentrations, contact times, solution pH and temperatures by using design of experiment approach using Box Behnken Design method (BBD).

IV. To evaluate the adsorption equilibria, kinetics and thermodynamics of dyes adsorption.

1.4 Scope of the Study

Adsorbents were prepared from PFAD by carbonizing the precursor to temperature range of 500-600 ° C for the period of 0.5-1h. After, another set of adsorbents were prepared from the precursor by mixing the precursor with zinc chloride as a modifier at different mixing ratio of PFAD and zinc chloride. These various mixture were then carbonized at the same temperature range of 500-600 °C and varying the time of carbonization between 0.5h and 2h. All the adsorbents prepared from the two processes were characterized and the results of characterization were compared.

Response surface methodology (RSM) feature of Design Expert software (version 7.1.6, Stat-Ease, Inc., Minneapolis, USA) was used to design the experimental runs in order to reduce the number of experiments. The software was used to determine the effect of variables leading to optimum adsorption performance of adsorbents prepared. The effect of temperature, the effect of time and the effect of mixing ratio of precursor and zinc chloride were studied.

The effect of adsorbent dosage contact time between the adsorbents and adsorbates, temperature and initial concentrations of methylene blue and congo red on efficiency of adsorbents in removing dyes from solution were also studied.

The adsorption equilibria, kinetics and thermodynamics of dyes adsorption were evaluated. Isotherm models of Langmuir, Freundlich, and Temkin were used to check the nature of the adsorption. Pseudo-first order, pseudo-second order, intraparticle diffusion and Boyd models were used to predict the kinetics and the rate order of reaction. The thermodynamic behaviour of the process were equally studied and the entropy, enthalpy and the Gibbs energy of the processes were evaluated.

1.5 Significance of the Study

Malaysia, a big producer of palm oil and refined oil products in the world has a large number of palm oil refinery industries that convert the oil produced into domestic and industrial raw materials for other associated companies. A large quantity of palm oil and refined oil are also exported to other countries. This gives rise to production of PFAD and continuous increase in production of waste and by-products as the production increases yearly. The use of available, cheap and abundant wastes for production of adsorbents- for subsequent use in the treatment of waste water generated by the industries in Malaysia will go a long way in in combating environmental pollution challenges and waste water management which helps in environmental sustainability and to overcome in combating the potential health challenge associated with waste water from industries. It will also serve as source of revenue generation and job creation in the country. It will equally help the ever growing palm oil industries in their waste management as well as enhancing and improving on their production process and technology. More so, carbon adsorption technology is a better and more effective way for water treatment application as it is known to be cheapest treatment method and especially in treating non-biodegradable metals in waste water. The prominent advantage of adsorbents is its characteristic properties in removing wide range of toxic elements to a non-detectable level and also its ability for specific organic and inorganic substance removal.

1.6 Organization of the Study

This thesis comprises of five chapters. In each chapter, the relevant subjects were discussed as follows: The background of the study discussed in chapter one. The problems prompted this study was highlighted. The objective, scope, and significance of this research were also stated. Chapter two focuses on the reviews of relevant and related literature to the study area. The preparation processes of adsorbents and their modifications for dye removal from wastewater. The use of high temperature environment for adsorbents preparation, modification strategies and the use of relevant software for optimization of the production of adsorbents were explored. Chapter three consists of selection of equipment and materials, the methodology employed and steps followed in accomplishing the objectives of the research work. The procedures involved the production of bio-char, characterization of the raw materials, biochar and the produced modified adsorbents, activation process with zinc chloride and adsorption studies. Adsorption isotherms and kinetics equations were introduced to describe the experimental data. The thermodynamics properties and the use of design expert software for the optimization of process parameters were highlighted. In the same vein, methylene blue and congo red dyes adsorption were elaborated. In this chapter various characterization techniques were also explained. Chapter four presents the results of using palm fatty acid distillates (PFAD) for the preparation, modification and characterization of adsorbents at different environmental conditions. It also focused on the physicochemical properties of the modified and unmodified adsorbents. Experimental results in relation to the adsorption of both basic and acidic dyes by the adsorbents, and their correlation with the empirical models and optimization were verified accordingly. In chapter five, the findings from this research work were presented and recommendations were made for future work

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LIST OF PUBLICATION

Indexed Journal

1. **Hamzat Bashir Aderemi**, Muhammad Abbas Ahmad Zaini and Noor Shawal Nasir (2018) Adsorbents from the by-products of palm oil refinery for methylene blue removal Malaysian Journal of Analytical Sciences vol 22 No 4 642 -647. https://doi.org/10.17576mjas-2018-2204-10. **(Indexed by ISI and Scopus)**

2. **Hamzat Bashir Aderemi,** Muhammad Abbas Ahmad Zaini and Noor Shawal Nasir (2018) Physicochemical properties of char derived from palm fatty acid distillate Malaysian Journal of Fundamental and Applied Sciences vol 14 No 13 (403 – 406) DOI: 10.11113/mjfas.v14n3.1084 **(Indexed by ISI and Scopus)**

3. **Hamzat Bashir Aderemi,** Muhammad Abbas Ahmad Zaini and Noor Shawal Nasir (2017) Adsorbents from the by-products of palm oil refinery for methylene blue removal. Presented at the 2nd International Conference on Separation Technology (ICoST 2017) Johor bahru, Malaysia. **(Indexed by ISI and Scopus)**

APPENDIX A

Table of pH(pzc) For Selected Adsorbents

initial pH	Final pH	AD5Z3T0.5	AD5Z3T2	AD6Z3T0.5	AD6Z3T2
0	0	0	0	0	0
2	2	2.3	2.3	2.9	3.7
4	4	5	5.1	5.5	5.6
6	6	5.4	5.2	5.5	5.6
8	8	6.1	6.3	6.6	6.6
10	10	6.5	6.6	6.8	7
12	12	11.8	11.9	12	12

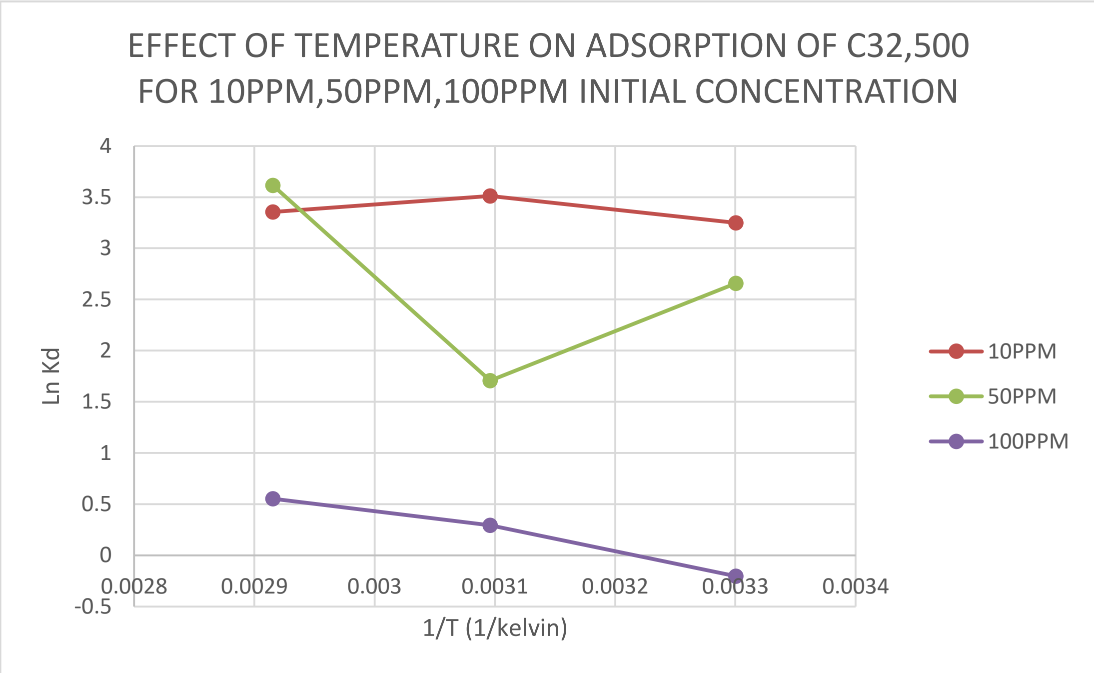
APPENDIX B

Selected Adsorbents for Rsm, Box Behnken Design Approach

serial	material	yield%
1	C23	26
2	C33	30
3	C11	20
4	C21	27.4
5	C31	15
6	C12	26.34
7	C22	25.26
8	C32	18.01
9	C13	27.33
10	C23	36.33
11	C33	35.28
12	C11	29.65
13	C21	21.71
14	C31	33.08
15	C12	12.17
16	C22	8.73
17	C32	11.6

APPENDIX C

Effect of Temperature on Methylene Blue Adsorption At Different Initial Concentration



APPENDIX D

Summary of R^2 Of Selected Adsorbents Prepared At 500 And 600 °C On Congo Red Adsorption

Sample	**500 °C**	**600 °C**
C13	0.8168	0.8573
C23	0.6367	0.8849
C33	0.5213	0.6799
C11	0.4278	0.0416
C21	0.1275	0.4924
C31	0.4269	0.5302
C12	0.4558	0.2945
C22	0.5843	0.7532
C32	0.6011	0.7236