REMOVAL OF BISPHENOL A FROM AQUEOUS SOLUTION BY ADSORBING ONTO AN ACTIVATED CARBON OF THE AGRICULTURAL WASTE

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This thesis specially dedicated to my beloved mother, father and brothers

Thank you very much for the endless love, support and prayer.

I love you all.

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ABSTRACT

Adsorption of bisphenol A (BPA), an endocrine disrupting compound, from aqueous media was studied using activated carbon derived from oil palm empty fruit bunch (EFB) waste. Oil palm EFB waste is a readily available biomass in Malaysia. Annually, Malaysia palm oil mill generates millions tonnes of oil palm EFB waste which is not effectively utilized. In this work, activated carbon was prepared by impregnating EFB for 24 hours in 10% of zinc chloride solution. The impregnated EFB was heated in a horizontal tube furnace under nitrogen flow at 500^{o}C for 1 hour. The samples were characterized by means of Fourier Transform Infrared Spectrometry, Brunauer-Emmett-Teller, and Field Emission Scanning Electron Microscopy. The proximate analysis including moisture content, ash content, bulk density, pH, conductivity and pH at zero charge was conducted to identify the psychochemical properties of the adsorbent. Batch adsorption test was carried out by varying contact time, activated carbon dose, agitation speed, initial BPA concentration, temperature and pH of the solution. The analyses showed that the oval-shaped micro pores were developed in carbon surface causing increase in surface area from 4.29 m^2/g to 86.62 m^2/g. The highest adsorption removal of BPA achieved up to 96.1% for 48 hours. The equilibrium data were perfectly represented by Langmuir isotherm with maximum monolayer adsorption capacity of 41.98 mg/g. Kinetic studies indicated that the adsorption process followed the pseudo-second-order kinetic with a rate constant of 0.3 $x10^{-3}$ mg/g min. The thermodynamic studies showed that the adsorption capacity increased by the increase in temperature. The results indicate that the activated carbon prepared from EFB has potential as a low cost bio-adsorbent for the removal of BPA from aqueous solution.

ABSTRAK

Penjerapan Bisphenol A (BPA), salah satu daripada bahan penganggu endokrin, dari media akueus telah dikaji menggunakan karbon teraktif yang diperolehi daripada sisa tandan buah kosong kelapa sawit (EFB). Sisa EFB kelapa sawit merupakan biojisim sedia ada di Malaysia. Setiap tahun, kilang minyak sawit di Malaysia menjana jutaan tan sisa EFB kelapa sawit yang belum dimanfaatkan secara berkesan. Dalam kajian ini, karbon teraktif telah dihasilkan dengan mengimpregnasi EFB selama 24 jam dalam 10% larutan zink klorida. Sampel terimpregnasi telah dipanaskan di bawah aliran gas nitrogen dalam relau tiub mendatar pada 500℃ selama 1 jam. Pencirian sampel dilakukan dengan kaedah Spektroskopi Inframerah Transformasi Fourier, Brunauer-Emmett-Teller dan Mikroskopi Imbasan Elektron Pancaran Medan. Analisis yang lain termasuk kandungan lembapan, kandungan abu, ketumpatan pukal, pH, konduktiviti dan pH pada cas kosong telah dijalankan untuk mengenal pasti ciri-ciri kimia fizik bahan penjerap. Ujian penjerapan berkelompok telah dijalankan dengan mengubah masa sentuhan, jumlah karbon teraktif, kelajuan pengacauan, kepekatan awal BPA, suhu dan pH larutan. Analisis menunjukkan bahawa liang mikro berbentuk bujur terbentuk pada permukaan karbon menyebabkan luas permukaan meningkat dari 4.29 m^2/g ke 86,62 m^2/g. Penyingkiran penjerapan BPA tertinggi telah dicapai sehingga 96.1% selama 48 jam. Data keseimbangan mengikut isoterma Langmuir dengan kapasiti penjerapan lapisan tunggal maksimum sebanyak 41.98 mg/g. Kajian kinetik menunjukkan proses penjerapan mengikut kinetik tertib-pseudo-kedua dengan pemalar kadar 0.3 $x10^{-3}$ mg/g min. Kajian termodinamik menunjukkan kapasiti penjerapan bertambah dengan peningkatan suhu. Keputusan kajian ini menunjukkan bahawa karbon teraktif yang dihasilkan daripada EFB berpotensi sebagai penjerap-bio kos rendah untuk penyingkiran BPA daripada larutan akueus.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS

%	Percent
˚C	Celcius
∆G˚	Gibbs free energy change
∆H˚	Enthalpy change
∆S˚	Entropy change
λ	Lambda
µ	Micro
A	Temkin equilibrium binding constant
B	Temkin constant related to heat of sorption
C	Carbon
C_0	Concentration at initial
C_e	Concentration at equilibrium
C_t	Concentration at time
cm	Centimetre
g	Gram
h	Hour
H_2SO_4	Sulphuric acid
H_3PO_4	Phosphoric acid
HCl	Hydrochloric acid
K	Kelvin
k_1	Pseudo-first-order rate constant
k_2	Pseudo-second order rate constant
k_d	Thermodynamic equilibrium constant
k_{dif}	Intraparticle diffusion rate constant
kJ/mol	Kilo joule per mole
K_F	Freundlich constant

K_L	Langmuir constant
K_2CO_3	Potassium carbonate
KBr	Potassium bromide
kPa	Kilopascal
L	Litre
M	Molar
mg/g	Milligram per gram
mg/L	Milligram per Litre
min	Minute
g/mol	Gram per Mole
N_2	Nitrogen
NaCl	Sodium chloride
NaOH	Sodium hydroxide
nm	Nanometer
pH_{PZC}	pH at zero point charge
q_e	Adsorption capacity at equilibrium
q_m	Maximum adsorption capacity
q_t	Adsorption capacity at time
ppm	Part per millions
R	Universal gas constant (8.314 J/mol K)
R^2	Coefficient correlation
R_L	Dimensionless constant
rpm	Rotor per Minute
Si	Silicon
t	Time
T	Temperature
V	Volume
W	Weight
wt%	Percent weight
$ZnCl_2$	Zinc chloride

LIST OF ABBREVIATIONS

ABTS:	2,2′-azino-bis-(3-ethylbenzthiazoline-6-sulfonic acid)
AC	Activated carbon
AC0	Activated carbon without chemical activation
ACK	Activated carbon treated with K_2CO_3
ACN	Activated carbon treated with NaOH
ACP	Activated carbon treated with H_3PO_4
ACS	Activated carbon treated with H_2SO_4
ACZ	Activated carbon treated with $ZnCl_2$
ASTM	American Society for Testing and Materials
BPA	Bisphenol A
BPL	Bituminous coal-based
BET	Brunauer-Emmett-Teller
EDX	Energy Dispersive X-Ray
EFB	Empty fruit bunch
FESEM	Field emission scanning electron microscope
FTIR	Fourier transform infrared spectroscopy
GAC	Granular activated carbon
PAC	Powder activated carbon
PCB	Coconut shell-based
UV-Vis	Ultraviolet-visible

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Over last decades, the trace level of endocrine disrupting compounds (EDCs) has been found in the wastewater, ground water, surface water, drinking water by researchers (Auriol *et al.*, 2006; Chang *et al.*, 2009; Daughton, 2001; Deblonde *et al.*, 2011; Duong *et al.*, 2010; Rahman *et al.*, 2009; Snyder and Benotti, 2010; Snyder, 2008). An environmental EDC is define by United States of Environmental Protection Agency (US-EPA) as an exogenous agent that interferes with the synthesis, binding, secretion, action, transport or elimination of natural hormones in the body which are responsible for the maintenance of reproduction, homeostasis, behavior, and/or development (Rahman *et al.*, 2009). Some reports describe that in many cases, these pollutants are not completely removed in wastewater treatment and discharged into receiving waters in the end it can affect drinking water supplies and ecosystems.

Endocrine disrupting compounds consist of naturally occurring compounds and man-made chemicals including plasticizer, pesticides, pharmaceuticals, personal care products, industrial additives, hormones excreted by animals and humans and other chemicals pose as endocrine disrupter. Among the endocrine disruptors discovered, 17a-ethynylestradiol (EE2), estrone (E1), 17b-estradiol (E2), octylphenol (OP), nonylphenol (NP), bisphenol A (BPA), and phytoestrogen group are frequently found EDCs in the environment (Duong *et al.*, 2010; Shareef *et al.*, 2008).

Bisphenol A [2,2-bis(4-hydroxyphenyl)propane] is one of the highly produced industrial chemicals in the world. In 2012, the world-wide production of bisphenol A was over the 4,600,000 tonnes per year. Asia is the dominant BPA producer; almost 53% of global production volume of BPA is manufactured in Asia, which is equivalent to around 2.4 million tonnes, followed by Europe and North America, controlling 25% and 18%, respectively. United stated, Taiwan, China, South Korea and Japan are in the top five of BPA manufacturers. In 2011, BPA production growth recorded at 5.25% per year. The growth of BPA market is driven by the increasing demand for the BPA-based products. With this high growth rate, the global BPA production is expected to reach 5.4 million tonnes in 2015. Further growth of BPA consumption is expected mostly from Asia Pacific, especially from China due to BPA ban in baby bottles and children food packaging in North America (Mcgroup, 2013).

BPA is used as a monomer to synthesize polycarbonate and epoxy resins in the manufacture of various consumer products such as baby bottles, storage containers, microwaveware, tableware, water pipes, CD, DVD, internal lining in food and beverage cans, and surface-coating on water storage tanks. Around 70% used in polycarbonates, 26% in epoxy resins and 4% used in some applications such as flame-retardants, brake fluids, and thermal papers (Fernandez *et al.*, 2007; Staples *et al.*, 1998).

Some studies have been reported that BPA was detected in a large number of aquatic environment, such as surface, ground and waste water effluent, in many countries include Malaysia (Klecka *et al.*, 2009; Vethaak *et al.*, 2005; Fu et al., 2007; Duong *et al.*, 2010; Santhi *et al.* 2012). In the sampling conducted at 2007-2008, Duong *et al.*, (2010) found that Salut river water, Malaysia containing 7.4-10.8 ng/L of BPA. Santhi *et al.* (2012) have also been reported that BPA was present in 93% of surface water samples. The level of BPA was found up to 215 ng/L in the Langat river basin, Malaysia. These studies showed that BPA was an ubiquitous contaminant present in water sources due to its widespread uses.

Concern for the decreased water quality and the safe drinking water has led researchers developing wastewater treatment process to remove environmentally interfering substances in wastewater. Some methods have been developed for the removal of pollutants from water (water treatment) based on physical, chemical, electrical, thermal and biological principles. These are consist of screening, filtration and centrifugation, micro- and ultra-filtration, crystallization, sedimentation and gravity separation, flotation, precipitation, coagulation, oxidation, solvent extraction, evaporation, distillation, reverse osmosis, ion exchange, ozonation, electrolysis, and adsorption. Among wastewater treatment technologies that have been adopted to remove pollutants, adsorption by activated carbon is considered as the most efficient methods due to its wide range of applications and ease of operation. It can be applied for the elimination of organic and inorganic pollutants in water with removal up to 99.9% (Ali, 2013; Cecen and Aktas, 2011; Gupta *et al.*, 2012; Pawlowski, 1982).

Adsorption is the selective removal of a contaminant or impurity in a fluid by contacting the fluid with a solid adsorbent. It has been widely accepted as an effective purification method both for drinking and wastewater However, the overlying cost activated carbon made more research for looking the alternative low cost adsorbents. Several studies have reported the activated carbon could be prepared from various agricultural by-products such as activated carbon derived from hazelnut shell (Doğan *et al.*, 2008), durian shell (Chandra *et al.*, 2007), almond shell (Bautista-Toledo *et al.*, 2005), olive bagasse (Demiral *et al.*, 2008), palm shell (Adinata *et al.*, 2007), rattan sawdust (Hameed and Rahman, 2008), coconut husk (Tan *et al.*, 2008), and coconut shell (Cazetta *et al.*, 2011).

Malaysia is one of largest producer and exporter of palm oil which is the main source of edible oil. As a major agricultural industry in Malaysia, palm oil mill leaves a huge amount of biomass residues, such as trunks, fronds, shell and empty fruit bunch. Along with the growing demand for palm oil, the expansion of oil palm plantation area in Malaysia is increased. In 1996, the hectarage of oil palm plantation stood at 2.6 million hectares. In 2011, it was reached 5 million hectares then at December 2013 was increased to 5.2 million hectares plantation area (MPOB, 2014).

The rapid growth of palm oil production cause the amount of oil palm residues increased.

Empty fruit bunches, fibers and shells of oil palm are the primary solid wastes from palm oil mill. Palm oil mill generates 7% shells, 14% fibers, and 23% empty fruit bunches (EFBs) per ton of fresh fruit bunches (FFBs). EFB alone accounts for 19.5 million tons in 2008 (Omar *et al.*, 2011). Traditionally empty fruit bunches have been burnt in incinerator by palm oil mills. Their ash was then recycled to the plantation as a fertilizer. However, the incineration of EFB has been discouraged since it may cause pollution problem. Instead most of EFB is returned to the field being mulch (Yusoff, 2006). To make better use of this waste, in this study EFB was proposed to be made activated carbon as low-cost adsorbent. EFB contains lignocellulosic materials that can be readily turned into activated carbon.

Recently, the potential of EFB-activated carbon as low-cost adsorbent has been intensively studied by Alam *et al.* and Hameed *et al.* In their studies, Alam *et al.* (2007a,b, 2008, 2009) prepared activated carbon from EFB using physical activation in this case air and carbon dioxide to remove contaminants such as phenol, 2,4-dichlorophenol and zinc (II). Although the preparation of EFB-activated carbon with chemical activation have been reported by Hameed *et al.* (2009), there are no studies reported on physical and chemical properties of the EFB-activated carbon and the sole chemical reagent used is potassium hydroxide. This preparation of activated carbon with various chemical reagents will give a new knowledge on the physical and chemical properties of activated carbon thus contributing as a consideration factors for the adsorption study of BPA with a suitable activated carbon.

1.2 Problem Statement

The low-level of estrogenic compounds found in surface water has caused increasing concern in the contamination of water resources regarding the possible exposure impacts to animal, human and ecosystems. This is because surface water is used as recipients for wastewater. Most effluents generated from municipal and industrial plants, carrying various types of pollutants, end up in rivers, streams or lakes. Even after it was treated, some pollutants especially EDC still can be detected. Concern on the presence of EDC in environments was strengthened by reports in the adverse changes of reproductive and developmental health of wildlife. Some studies indicated that endocrine disrupting compounds, both naturally occurring hormones and synthetic compounds such as BPA, have the potential to disrupt the endocrine system or at least could be partly responsible for the observed changes (Fürhacker *et al.*, 2000; Rodriguez-Mozaz *et al.*, 2004).

In 1930, BPA has been discovered as an artificial estrogen. Its estrogenic effect was used in cattle and poultry industry to enhance their animal growth. In the mid-1930s, BPA was ever used for women as an estrogen replacement before being replaced by diethylstilbestrol (DES). Even though BPA is a less potent estrogen than DES, they have many similarities. Many studies has reported that BPA shown endocrine-disrupting effects. Bisphenol A is expected to interact with the estrogen receptor and possibly cause hormonal effects. BPA may block or mimic hormones and disrupt normal bodily functions if absorbed into the body resulting in behavioral changes, alter growth and reproduction system and early secondary sexual maturation. Through in vivo screenings, BPA has been shown to be estrogenic (Erler and Novak, 2010; Kang *et al.*, 2006).

As stated by United States EPA in 1993, reference dose (RfD) for chronic oral exposure of BPA is 0.05 mg/kg body weight per day (EPA, 1993). Although the safety of BPA uses in consumer products has been claimed by related industry, over the past ten year studies, many studies in animals have reported that BPA can produce adverse endocrine effects at doses significantly lower than reference dose values. Some scientists claimed that a low-dose BPA could be potential contributor to diabetes, obesity, reproductive disorders, sexually dimorphic behaviors, asthma, cardiovascular diseases, as well as breast, prostate and uterine cancer (Erler and Novak, 2010; Hengstler *et al.*, 2011; Kang et al., 2006). These created debating between the experts whether a low-dose BPA may cause adverse effects. In addition to its increased availability in the environment, and its estrogenic activity in specific responses *in vivo* and *in vitro*, the adverse effects of BPA exposure toward human health at low doses become possible (Vandenberg *et al.*, 2007; Vom Saal and Welshons, 2006).

1.3 Research Objectives

The objectives of study are:

1. To characterize the physical and chemical properties of activated carbon derived from oil palm empty fruit bunch waste.
2. To investigate the effects of contact time, activated carbon concentration, BPA concentration, pH, agitation and temperature in the removal of BPA.
3. To determine the adsorption behaviour of BPA into the prepared activated carbon by kinetic, isotherm and thermodynamic calculation.

1.4 Scope of study

The present study utilized oil palm empty fruit bunch to produce activated carbon using chemical agents such as zinc chloride, sulphuric acid, phosphoric acid, potassium carbonate, and sodium hydroxide. Carbonization was performed in one-step process using horizontal furnace and activation temperature was fixed at 500^{o}C. The raw oil palm empty fruit bunch and the prepared-activated carbons were characterized using Micromeritics ASAP 2000, field emission scanning electron microscope (FESEM), Fourier transform infrared spectrometer (FTIR), muffle furnace and pH meter to determine the physical characteristics and surface chemistry of the adsorbents.

The efficiency of activated carbon in removing BPA from aqueous solution was studied. Residual BPA content was analyzed using UV-visible spectrophotometer at wavelength 277 nm. BPA adsorption capacity was determined and factors influencing BPA adsorption were investigated through several parameters including contact time, initial BPA concentrations, initial pH, activated carbon dosage, agitation, humic acid, and temperature.

1.5 Significance of Research

The significance of the current research is to provide an alternative bioadsorbent from oil palm empty fruit bunch waste for the removal of BPA. Oil palm empty fruit bunch is an abundant local waste in Malaysia. As a renewable biomass, it is not widely used as an activated carbon. Recently, activated carbon from agricultural wastes have been successfully applied to biosorption process for removal of heavy metal ions, phenol and dyes (Alam *et al.*, 2007a; Demirbas, 2009; Namasivayam *et al.*, 2007).

Although several studies have reported the removal of bisphenol A by adsorption onto several adsorbents (Bautista-Toledo et al., 2005; Gong *et al.*, 2009; Nakanishi *et al.*, 2002; Tsai *et al.*, 2006; Zhou *et al.*, 2011). However, mostly they are commercial activated carbons and mineral sources (non-renewable) as adsorbents. To our knowledge, there is no report investigating the removal of bisphenol A using EFB, a promising precursor and the preparation of activated carbons from oil palm EFB waste employing $ZnCl_2$ as chemical agent.

The conversion of this waste into activated carbon can help oil palm mills in reducing the disposal problem of oil palm EFB waste. By produce activated carbon from oil palm EFB with a suitable method, value-added product will be produced from an unwanted agricultural waste thus encourage the economy of agricultural industry and contribute to solve part of the wastewater and environmental problems.

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