

A COMBINED ADSORPTION AND OZONATION PROCESS FOR REMOVAL
OF LINDANE IN AQUEOUS MEDIUM

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ABSTRACT

Gamma-hexachlorocyclohexane or lindane (γ -HCH) removal using a combination method of adsorption and ozonation was investigated. Lindane is one of the persistent organochlorine pollutants (POPs), used as a pesticide commonly in agricultural industry. The combination of both methods was studied based on adsorptive capability of banana fronds activated carbon which act as an adsorbent and powerful ozonation agent. The raw banana frond was impregnated with ZnCl_2 prior to the process of carbon activation. Gas Chromatography-Flame Ionization Detector (GC-FID) equipment was used to analyze the concentration of lindane solution before and after treatment. The formation of ozone in aqueous solution was studied by using Ultraviolet-Visible Spectrophotometry (UV-Vis). The study showed that the highest removal of lindane was achieved at 91% by using the adsorption method, 92% by using ozonation method and 99% by using a combination of both methods. The equilibrium data of adsorption was analyzed using two isotherm models Langmuir and Freundlich, and Langmuir isotherm ($R^2 = 0.99$) was found to fit the data better than Freundlich isotherm. The adsorption kinetics was studied, and the study indicated that the kinetic model followed pseudo-second-order ($R^2 = 0.99$). The functional groups of adsorbents were investigated using Fourier Transform Infrared Spectroscopy (FTIR), and it was found that a large number of carbonyl groups that represent hydrogen bond and hydroxyl groups had influenced the capability of adsorption by the banana fronds activated carbon towards lindane. In addition, the surface morphology of adsorbent was investigated using Field Emission Scanning Electron Microscope (FESEM). The study showed an increment of available pore sites after carbon activation of raw banana fronds soaked with ZnCl_2 . Brunauer-Emmett-Teller (BET) supported by the FESEM results shows 4.86 m^2/g and 4.77 m^2/g of available pore sites for banana fronds activated carbon with and without impregnation of ZnCl_2 activated carbon, respectively. The results proved that the combination method of adsorption and ozonation is a highly potential method for the removal of lindane in aqueous solution.

ABSTRAK

Penyingkiran gamma-heksaklorosikloheksane atau lindana (γ -HCH) menggunakan kaedah gabungan penjerapan dan pengozonan telah dikaji. Lindana adalah salah satu daripada bahan pencemar organoklorin kekal (POPs), yang biasa digunakan sebagai racun makhluk perosak dalam industri pertanian. Gabungan kedua-dua kaedah telah dikaji kerana ia menggunakan keupayaan penjerapan pelepah pisang karbon teraktif sebagai bahan penjerap dan agen pengozonan yang berkesan. Pelepah pisang perlu direndam dengan $ZnCl_2$ terlebih dahulu sebelum proses pengaktifan karbon. Peralatan pengesan kromatografi gas pengesan ion nyala (GC-FID) digunakan untuk menganalisis kepekatan lindana dalam larutan sebelum dan selepas dirawat. Pembentukan ozon dalam larutan akueus dikaji dengan menggunakan spektrofotometer ultra lembayung-nampak (UV-Vis). Kajian ini menunjukkan bahawa penyingkiran tertinggi adalah pada 91% dengan menggunakan kaedah penjerapan, 92% menggunakan kaedah pengozonan dan 99% menggunakan penggabungan kedua-dua kaedah. Data keseimbangan penjerapan dianalisis dengan menggunakan dua model isotem Langmuir dan Freundlich dan isotem Langmuir ($R^2 = 0.99$) lebih bersesuaian dengan data berbanding isotem Freundlich. Kinetik penjerapan telah dikaji dan menunjukkan model kinetik mengikuti pseudo-turunan-kedua ($R^2 = 0.99$). Kumpulan berfungsi bagi penjerap telah disiasat menggunakan Spektroskopi Inframerah Transformasi Fourier (FTIR) dan mendapati sebilangan besar kumpulan karbonil yang terdiri daripada hidrogen dan hidroksil telah mempengaruhi keupayaan penjerapan pelepah pisang karbon teraktif terhadap lindana. Morfologi permukaan bahan penjerap telah dianalisis menerusi Mikroskop Elektron Imbasan Pelepasan Medan (FESEM). Kajian ini menunjukkan peningkatan liang sedia ada selepas pengaktifan karbon daripada pelepah pisang yang direndam dengan $ZnCl_2$. Keputusan Brunauer-Emmett-Teller (BET) yang telah disokong oleh FESEM mendapati 4.86 m^2/g dan 4.77 m^2/g liang yang ada bagi karbon aktif pelepah pisang dengan dan tanpa direndam $ZnCl_2$. Keputusan membuktikan bahawa kaedah gabungan antara penjerapan dan pengozonan adalah kaedah yang berpotensi tinggi untuk penyingkiran lindana dalam larutan akues.

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LIST OF ABBREVIATIONS

AC	- Activated Carbon
AOPs	- Advanced Oxidation Processes
BET	- Brunauer-Emmett-Teller
BFAC	- Banana Fronds Activated Carbon
BFC	- Biomimetic Fat Cell
BHC	- Benzene Hexachloride
BOD	- Biological Oxygen Demand
BPA	- Bisphenol A
COD	- Chemical Oxygen Demand
DO	- Dissolved Oxygen
EDCs	- Endocrine Disrupting Compounds
EPA	- Environmental Protection Agency
FDA	- Food and Drug Administration
FESEM	- Field Emission Scanning Electron Microscopy
FTIR	- Fourier Transform Infrared Spectroscopy
GAC	- Granular Activated Carbon
GC-FID	- Gas Chromatography-Flame Ionization Detector
HCH	- Hexachlorocyclohexane
INQUINOSA	- Industrias Químicas del Noroeste Sociedad Anónima
IUPAC	- International Union of Pure and Applied Chemistry
NOM	- Natural Organic Matter
PAC	- Powder Activated Carbon
POPs	- Persistent Organochlorine Pesticides
PPCPs	- Pharmaceuticals and Personal Care Products
SS	- Suspended Solids
TOC	- Total Organic Carbon
UNEP	- United Nations Environment Programme
UV	- Ultra Violet
UV-Vis	- Ultraviolet-Visible Spectrophotometry
WHO	- World Health Organization

LIST OF SYMBOLS

g	-	Gram
m	-	Moles
°C	-	Celsius
%	-	Percent
ng	-	Nanogram
L	-	Litre
mg	-	Milligram
α	-	Lpha
β	-	Beta
γ	-	Gamma
δ	-	Delta
ϵ	-	Epsilon
η	-	Eta
Θ	-	Theta
Pa	-	Pascal
K_{oc}	-	Soil sorption coefficient
mm	-	Millimeter
Hg	-	Mercury
kg	-	Kilogram
ppm	-	Parts per million
K_{ow}	-	Partition coefficient
cm^3	-	Centimeter cube
μ	-	Micro
O_3	-	Ozone
$\cdot OH$	-	Hydroxyl radicals
min	-	Minute
mM	-	Millimolar
V	-	Volt

H_2O_2	-	Hydrogen Peroxide
OH^-	-	Hydroxide ions
nm	-	Nanometer
MW	-	Molar weight
mL	-	Milliliter
cm	-	Centimeter
d	-	Diameter
KOH	-	Potassium hydroxide
K_2CO_3	-	Potassium carbonate
$ZnCl_2$	-	Zinc chloride
NaOH	-	Sodium hydroxide
KI	-	Potassium iodide
HCl	-	Hydrochloric acid
H	-	Hour
q_e	-	Equilibrium adsorption capacity
Q_m	-	Maximum adsorption capacity
C_o	-	Initial concentration
C_e	-	Equilibrium concentration
KBr	-	Potassium bromide
K	-	Kelvin
N_2	-	Nitrogen

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CHAPTER 1

INTRODUCTION

1.1 Background Study

Pesticides are chemicals commonly used in the agriculture industry as plant protection to control the quality of products by destroying pests. The pesticide comprised of insecticide, herbicide, molluscicide, nematicide, avicide, piscicide, rodenticide, bactericide, animal and insect repellent, fungicide, antimicrobial, sanitizer, and disinfectant. Pesticides are not only used in the agriculture sector but also as non-agricultural purposes since it works to attract, seduce, and destroy any pests.

Besides that, some pesticides used for medical applications that act as prevention of vector-borne diseases such as malaria and dengue. The vector-borne disease, which represents one of the major global health issues, also become one of the reasons for the high demand of pesticides. Not just that, the ever-increasing demand for food has cause the increase in agriculture activity which led to highly pesticide application to feed a growing world population (Pavlíková *et al.*, 2012).

Even though it has many benefits since it can destroy any pests, but it also produce side effects in terms of health issues. Over the last decades, the public and government has been concerning the potential hazards of the uncontrolled use of pesticides. In Malaysia there are several legislations in regards to these issues such as Pesticides Act 1974, Poison Act 1952, the Environmental Quality Act 1974, Fumigation of Hydrogen Cyanide Act 1953, Occupational Safety and Health Act 1994 and many regulations and guidelines (Ali and Shaari, 2015).

Thus, a large number of researchers have studied the effects of pesticides that pose serious human health and ecosystem issues. Some of these pesticides are grouped as persistent organochlorine pollutants (POPs) and are considered as most toxic pesticides due to their persistence behavior in the environment and toxicity towards the human. According to the United Nations Environment Program (UNEP), POPs defined as a chemical that persists or non-degradable in the environment even through chemical or biological degradation. Therefore, the pesticide is bio-accumulated through the food web, long-term toxicity in soils, water, sediments, and biota, and can cause health effects towards human and the environment (Laver, 2015).

Since POPs are non-polar pesticides, their molecules can accumulate in fatty tissues and promote their biomagnification in the food chain (Jacob, 2013). As a result, high concentration of these molecules often found in tissues, adipose tissues or fat, milk and serum of human beings because POPs are top in the food web (Lee *et al.*, 2017). Due to their high persistence in the environment even though through chemical and biological degradation, the Stockholm Convention has banned the use of pesticides under the POPs group since 2009.

Gamma-hexachlorocyclohexane (γ -HCH), which is known as lindane, a member of the POPs group is commonly used as insect pests in the agriculture industry due to its insecticide behavior. Besides that, it was also being used for medicinal and public health applications. In medical applications basically, lindane is formulated as lotion or shampoo to treat lice and scabies (Goldust *et al.*, 2013).

Lindane is a colorless to brown crystalline which exist in the form of powder or solid with a molecular weight of 290.83 g/mol and a melting point of 112°C (Laver, 2015). The solubility of lindane practically can be performed with a presence of ethanol, benzene, chloroform, acetone, or ether since it is insoluble with water (Report on Carcinogens, 2011). Based on the previous study has been reported that lindane had been detectable in biological samples such as urine, serum, semen, adipose tissue, milk, and brain, in fact, it also can be found in environmental samples

such as surface water, wastewater, groundwater, soil, fish, and vegetables (Wang and Liang, 2018; Lee *et al.*, 2017).

Due to the concern of decreased in water quality, a few conventional methods have been developed to apply for pesticides removal from water. Therefore, the treatment process of pesticides has becomes a real challenge due to variable concentration and volume to meet the quality standards for a direct discharge of pesticides into the surface water. Several methods introduced to overcome the critical issue of these pesticides including adsorption (Tor *et al.*, 2013), the simultaneous presence of coagulants and adsorption (Shabeer *et al.*, 2014), treatments with nanoparticles (Román *et al.*, 2016), ozonation (Begum and Gautam, 2012), photo-Fenton process (Nitoi *et al.*, 2013) and biological degradation (Pannu and Kumar, 2017; Satish *et al.*, 2017).

Among pesticides treatment technologies that have implemented before, adsorption and ozonation is a commonly used method for the pesticide treatment process. In recent years, treatment process of contaminated water and wastewater through a combination of adsorption by activated carbon (AC) and ozonation has emerged as one of the best options by researchers.

Adsorption is a process by which molecules, atoms, or ions with particular characteristics of size and polarity from the liquid phase are attracted and held to the adsorbent surfaces (El-Essawy *et al.*, 2017). The activated carbon has been proposed to be the most efficient adsorbent due to its high surface area and adsorptive capability to remove contaminants from solution rapidly and effectively (Lazim, 2016). Besides, in the combination of adsorption and ozonation methods, the AC will accelerate the decomposition rate of the ozone (O_3) to form hydroxide ions (OH^-) in the solution (Abdayem *et al.*, 2017). However, the overlying cost of commercial AC, cause the past researchers looking for another alternative that could save the cost of AC preparation (Wirasnita, 2015).

Generally, activated carbon from agricultural waste is an inexpensive material suitable to replace commercial AC. Moreover, the effectiveness of agricultural waste AC to remove pesticides from contaminated water or wastewater has proved through the study of chlorothalonil removal by casuarina charcoal (Gar Alalm and Nasr, 2018), metribuzin and furadan removal by bamboo (Santana *et al.*, 2017), 2,4-dichlorophenoxyacetic acid removal by langsung empty fruit bunch (Njoku *et al.*, 2015), diuron removal by wood composites (Cansado *et al.*, 2017), and lindane removal by date stones (El-Kady *et al.*, 2013).

On the other hand, the ozonation method is considered as the most efficient methods due to their availability of most powerful oxidant agents (Sousa *et al.*, 2018). It is one of the promising techniques for water and wastewater disinfection and capable of degrading many target compounds (Umar *et al.*, 2013). Recent years, some researchers reported that ozonation method are also be used to remove various type of pesticides such as beta-hexachlorocyclohexane (Cruz-González *et al.*, 2018), atrazine (Zhou *et al.*, 2016), permethrin (Chen *et al.*, 2013), endosulfan (Landeros *et al.*, 2017), chlorpyrifos ethyl (Kusvuran *et al.*, 2012) among many others. Different published works have reported that oxidation of wastewater by O₃ to remove lindane which has resulted in 82% removal (Begum and Gautam, 2012), 58.8% removal (Derco *et al.*, 2013), and 55% removal (Ormad *et al.*, 2008).

1.2 Problem Statement

Excessive use of lindane has been one of the major source of environmental pollution because it can affect human health, animal, and ecosystem due to it's toxicity. The toxicity of lindane may interrupt the ecosystem of the environment, especially to the surface water. The presence of lindane in the human body system as a result of lindane use and exposure, which caused health issue has raised concern to control the usage of lindane. It may affect the liver, kidneys, nervous system, and can be a carcinogen. The Stockholm Convention on Persistent Organic Pollutants has proclaimed the use of lindane in the agriculture sector was banned.

There are a lot of conventional methods that have introduced to treat pesticides. However, the results obtained from the past study showed that the lindane not totally can be removed even after the treatment due to their persistence behavior and design with strong bonding. Thus, this study investigated the potential of a combination of two conventional methods which are adsorption by using AC and ozonation as to maximize the possibility to degrade lindane from aqueous solution.

The previous study reported there are beneficial effects of removing pollutants or organic matter from water through a combination of adsorption by using AC and ozonation. The presence of AC in O₃ reaction acts as a promoting agent of hydroxide radicals in the ozonated sample and can increase the reaction rate of removal (Guiza *et al.*, 2004). Besides, AC is often used as catalyst support because it can satisfy most of the desirable properties required for suitable support.

The others problem in the study of lindane removal is also related to the cost of treatment preparation. Due to the high cost of commercial AC preparation, there is an alternative way to provide AC inexpensively by identified the potential agricultural waste as an adsorbent to promote lindane removal. In this study, banana fronds from the banana tree, which belongs to the family *Musaceae* were chosen as a potential agricultural waste to replace commercial AC.

The banana fronds have been extensively used as an agriculture waste AC due to its abundantly available since it provides one of the highest nutritional fruit crops cultivated around the world and considered as economical adsorbents while against cationic, anionic, and neutral pollutants (Ahmad and Danish, 2018). Furthermore, raw banana fronds can be chemically modified to prepare a better AC and may present as a good adsorbent against various pollutants compared to commercial AC.

The banana frond is potential to replace the commercial AC because it is readily available, and in term of the cost, it is more affordable compared to commercial AC. Besides, the composition of banana fronds, which consists of

lignocellulosic material, can provide a better porous of activated carbon (Suhas *et al.*, 2016). Not just that, the lignocellulosic waste had a high carbon content which performs a better behavior of activated carbon (Santos-Clotas *et al.*, 2019).

1.3 Research Objectives

The present study aims to investigate the potential of the combination of adsorption by using AC produce from banana fronds and ozonation for lindane removal in aqueous solution. The specific objectives of the study are as follows:

- i. To study the effect of impregnation methods using different chemical of producing banana fronds activated carbon (BFAC).
- ii. To analyze the effects of physico-chemical parameters for the lindane removal by using activated carbon and ozonation treatment.
- iii. To evaluate the adsorption kinetics and isotherms of lindane on activated carbon.
- iv. To determine the maximum percentage of lindane removal by combining activated carbon and ozonation treatment.

1.4 Scopes of Study

This study focuses on the treatment of lindane in aqueous solution by using a method of combination of adsorption and ozonation. The banana fronds were used as an agricultural waste AC to investigate their potential as an adsorbent for lindane removal. In preparation of BFAC, a few chemical reagents which are zinc chloride ($ZnCl_2$), sodium hydroxide (NaOH), potassium carbonate (K_2CO_3) and potassium hydroxide (KOH) used to impregnate with raw banana fronds before carbon activation as to increase their adsorption capability.

Batch studies of adsorption treatment were performed to study the effects of mass adsorbent, initial concentration of lindane, and contact time towards lindane removal. The characteristics of the adsorbent before and after treatment investigated through FTIR, FESEM, and BET. Adsorption mechanisms were investigated through adsorption isotherms and adsorption kinetics, which are Langmuir and Freundlich isotherm model and pseudo-first-order, pseudo-second-order, and intraparticle diffusion model respectively.

Batch studies of ozonation treatment were performed to study the effects of contact time, pH level, initial concentration of lindane, and ozone dosage. The formation of O₃ in aqueous solution studied by using Ultraviolet-Visible Spectrophotometry (UV-Vis). The study of the combination of both methods was performed based on the optimum results obtained from the operating parameters studies in individual experiments. In individual and combination studies, the Gas Chromatography-Flame Ionization Detector (GC-FID) equipment was performed to analyze lindane pollutants in solution.

1.5 Limitation of Study

In this study, there are a few limitation needed to be considered during the experiments. Firstly, during the process of carbon activation of banana fronds, the temperature of the pyrolysis process needs to be set at 400°C as the fiber composition of banana fronds will affect the quality of the activated carbon. If the temperature is set too high, the banana fronds would transform into ash. While, if the temperature were too low, the banana fronds would not be completely transform into activated carbon.

Besides that, the pollutants solution are being prepared in milligram per liter volume as the equipment of GC-FID cannot detect the low volume of pollutants in the solution. The studied sample solution are prepared in duplicate samples due to

the fast reactions of O_3 that might interrupt the study of contact time due to sample withdrawn process.

1.6 Significance of Research

The significance of this study is to provide a potential method through a combination of adsorption by BFAC and ozonation for lindane removal in aqueous solution. Even though several studies have reported the removal of lindane through adsorption and ozonation method, but there is still no result which completely removes lindane from aqueous solution. In the current knowledge, there is no report investigating the removal of lindane through the combination of both adsorption and ozonation methods, as a promising way to increase the efficiency of lindane removal.

Besides, the utilization of BFAC to replace commercial AC is environmentally friendly, inexpensive, sustainable resources, and easy to get. Furthermore, agricultural waste AC is expected to be more effective adsorbent to remove lindane particles due to their special microporous structure, and high surface area and volume. It also can act as promoting agents to accelerate the transformation of O_3 to form hydroxyl free radicals. By optimizing of both methods, it might solve the crisis of water contamination by lindane and resulting in reduces of exposure towards human and wildlife.

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