SYNTHESIS AND CHARACTERIZATION OF HYDROCHAR DERIVED FROM WASTE MATERIAL

LOH KAI PIN

FACULTY OF ENGINEERING

UNIVERSITI MALAYSIA SABAH

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SYNTHESIS AND CHARACTERIZATION OF HYDROCHAR DERIVED FROM WASTE MATERIAL

LOH KAI PIN

THESIS SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE DEGREE OF BACHELOR OF MECHANICAL ENGINEERING

FACULTY OF ENGINEERING
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DECLARATION

I hereby declare that this thesis, submitted to University Malaysia Sabah as a partial fulfilment of the requirement for the degree of Bachelor of Mechanical Engineering, has not been submitted to any other university for a degree. I also certify that the work described herein is entirely my own, except for quotations and summaries of sources which have been duly acknowledged.

22 May 2022

LOH KAI PIN

(BK18110094)

CERTIFIED BY

IR. TS. DR. MELVIN GAN JET HONG

SUPERVISOR

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Sincerely,

Loh Kai Pin

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ABSTRACT

Agricultural waste management is one of the popular issues of waste management. This research focuses on the synthesis of hydrochar using waste material with relatively simpler, cheaper and more environmentally friendly technique which is hydrothermal carbonization treatment. There is no pre-treatment of the precursors involved in this treatment. The precursor used in this research is sawdust which is low cost, discarded source and can be obtained from UMS Faculty of International Tropical Forestry. In this research, sawdusts were heated through hydrothermal carbonization treatment at the temperature of 160°C, 180°C and 200°C by 2 hours for set 1. Besides that, there is another one sample added in set 1 which is heated through heat treatment at the temperature of 200°C by 2 hours. For set 2, sawdusts were heated through hydrothermal carbonization treatment at the same temperature of 200°C by difference residence time of 2 hours, 3 hours and 4 hours. Then, the product after heated is hydrochar. The hydrochar's structure and morphology, elemental composition, UV absorbance and efficiency were characterized using Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDX), Ultraviolet-Visible Spectroscopy (UV-Vis) and Solar Vapor Generation. The hydrochars are observed to show wood chip like structure under SEM. For the elemental composition, the carbon content of the hydrochars are between 74.97% and 79.85%. The hydrochars heated at 200°C has a higher UV absorbance than the hydrochars heated at 160°C and 180°C and carbonized sawdust heated at 200°C. For the solar vapor generation, the average efficiency of the hydrochars and carbonized sawdust are between 42.59% and 66.59%. The hydrochar heated at 200°C by 4 hours (HC200-4) was chosen as the best hydrochar within the hydrochars, because of its black color properties and the highest carbon content. Besides that, HC200-4 has the stable UV absorbance between the range of wavelength. Lastly, HC200-4 has an average efficiency of 63.67% which considered as high efficiency compared to the other hydrochar.

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ABSTRAK

Pengurusan sisa pertanian adalah salah satu isu popular untuk pengurusan sisa. Kajian ini memberi tumpuan kepada sintesis hidrokar menggunakan bahan sisa dengan teknik yang agak mudah, lebih murah dan lebih mesra alam yang merupakan rawatan karbonisasi hidroterma. Jugalah tiada pra-rawatan prekursor yang terlibat dalam rawatan ini. Prekursor yang digunakan dalam kajian ini adalah serbuk gerhaji yang kos rendah, sumber yang dibiar dan boleh diperolehi dari UMS Fakulti Perhutanan Tropika Antarabangsa. Dalam kajian ini, serbuk gergaji dipanaskan melalui rawatan karbonisasi hidrotermal pada suhu 160°C, 180°C dan 200°C dengan 2 jam untuk set 1. Selain itu, terdapat satu lagi sampel yang ditambah dalam set 1 yang dipanaskan melalui rawatan haba pada suhu 200°C dengan 2 jam. Untuk set 2, serbuk gergaji dipanaskan melalui rawatan karbonisasi hidrotermal pada suhu yang sama 200°C oleh masa pemanasan berbeza daripada 2 jam, 3 jam dan 4 jam. Kemudian, produk selepas pemanasan adalah hidrokar. Struktur dan morfologi, komposisi unsur, penyerapan UV dan kecekapan hidrokar dicirikan dengan menggunakan Mikroskopi Pengimbasan elektron (SEM), Spektroskopi Sinar-X Tenaga (EDX), Spektroskopi Ultraviolet-Kelihatan (UV-VIS) dan Generasi Wap dengan Solar. Hidrokar diperhatikan untuk menunjukkan struktur seperti cip kayu di bawah SEM. Bagi komposisi unsur, kandungan karbon hidrokar adalah antara 74.97% dan 79.85%. Hidrokar yang dipanaskan pada 200°C mempunyai penyerapan UV yang lebih tinggi daripada hidrokar yang dipanaskan pada 160°C dan 180°C dan serbuk gergaji berkarbonat yang dipanaskan pada 200°C. Bagi generasi wap dengan solar, kecekapan purata hidrokar dan serbuk gergaji berkarbonat adalah antara 42.59% dan 66.59%. Hidrokar yang dipanaskan pada 200°C sebanyak 4 jam (HC200-4) dipilih sebagai hidrokar terbaik dalam hidrokar, kerana sifat warna hitamnya dan kandungan karbon tertinggi. Selain itu, HC200-4 mempunyai penyerapan UV yang stabil di sepanjang julat panjang gelombang. Akhir sekali, HC200-4 mempunyai kecekapan purata 63.67% yang dianggap sebagai kecekapan yang tinggi berbanding dengan hidrokar yang lain.

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LIST OF ABBREVIATIONS

HTC	Hydrothermal Carbonization Treatment
SD Raw	Raw Sawdust
HC160	Hydrochar Heated at 160°C by 2 hours
HC180	Hydrochar Heated at 180°C by 2 hours
HC200-2	Hydrochar Heated at 200°C by 2 hours
HC200-3	Hydrochar Heated at 200°C by 3 hours
HC200-4	Hydrochar Heated at 200°C by 4 hours
CSD200-2	Carbonized Sawdust Heated at 200°C by 2 hours
SEM	Scanning Electron Microscope
EDX	Energy Dispersive X-ray Spectroscopy
UV-Vis	Ultraviolet-Visible Spectroscopy

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LIST OF SYMBOLS

η	Photothermal efficiency
m	Mass flux of seawater/Mass loss of seawater
m_{solar}	Mass flux of seawater in the solar environment
m_{dark}	Mass flux of seawater in the dark environment
ΔH_{LV}	Enthalpy change due to the phase change from liquid to Vapor
I	Solar intensity during the vaporization process
C	Specific heat capacity of water
T	Surface temperature
T_o	Initial surface temperature
ΔH_{vapor}	Enthalpy of vaporization at temperature
A	Cross-sectional area
t	Time taken of the sample to evaporate

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Chapter 1

INTRODUCTION

1.1 Background / Purpose of The Project

Waste materials defined as the substances which are useless, worthless, and excessive. There are almost 90% of waste disposal to sanitary landfills had been reported in Malaysia while the recycle part only 10.5% was done (Ng & Iacovidou, 2020). Unfortunately, there are a lot of misunderstanding and unfamiliar with the usage of waste material. Some waste materials are advantageous, they can be classified with the concept of Three R's that represent the phrase of "reduce, reuse, recycle". The concept of the Three R's was developed by William Russell and Rex Burch, they detailed Three R's concept in their book which called The Principles of Humane Experimental Technique in 1959. Utilization of waste materials such as rice husk ash, red mud and sludge have been done in ceramic production as they contain the chemical compositions which are identical to raw materials used in ceramic production. The raw materials used in ceramic production are non-renewable and pollutant to air due to the releasing of carbon dioxide (Salleh *et al.*, 2021). Therefore, there are waste materials still available to reuse and recycle to generate a new product.

Sawdust known as agriculture waste material. Sawdust is a waste product from the woodworking operations like grinding, sawing or cutting with tools (Ahmed *et al.*, 2018). Due to the tremendous amount of wood wastes were discarded in landfills that made up of timber formwork, the environmental sustainability, pollution and biodiversity were influenced detrimentally (Ince *et al.*, 2021). Hence, the further utilization of the sawdust is required to decrease the amount of wood wastes.

Carbon material can be derived from the sawdust in different type of methods of extraction by physical and chemical reactions. For example, the sawdust act as a

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source to be carbonized, they were carbonized at furnace for 2 hours with the presence of argon gas. The temperature of 500°C and 800°C were used for carbonization process (Melvin *et al.*, 2019). In this study, hydrochars will be derived from sawdust by using hydrothermal carbonization treatment. Hydrothermal carbonization is a thermal conversion process that can convert biomass (sawdust) to a carbonaceous residue (hydrochar) at a relatively low temperature and autogenous pressure (Lu *et al.*, 2012). Hydrochar is a highly dense solid carbonaceous materials that used in variety range of applications between energy, environment, soil improvement and nutrient recovery. Through a further activation process, it will be a pollutants absorber and energy storage. In addition, it acts as soil conditioner through the enhancement of nutrient recovery which enriching nitrogen and phosphorus (Maniscalco *et al.*, 2020).

This research aims to synthesize the hydrochars from sawdust and then characterization of hydrochars by using the equipment like scanning electron microscopy (SEM) with energy dispersive X-ray analysis (EDX) and ultraviolet-visible spectroscopy (UV-Vis). The purpose of this project is to find an alternative solution for disposing of typical agriculture waste such as sawdust, and then determine possible uses of hydrochar by analyzing their surface structure, properties and morphology. In addition, this research aims to find a cheaper, more direct and less processes and methods at low temperatures to treat the waste materials and generate applicable product by using hydrothermal carbonization. This is because most of the previous research on pyrolysis or carbonization of waste materials are conducted at high temperatures (above 500°C), chemically treated or utilize the oxidizing agents such as carbon dioxide before, during or after the carbonization process. Besides that, hydrochar has a potential to form a photothermal conversion material, such as solar absorber. Hydrochar based solar absorber will be used in the setup of solar vapor generation to determine the efficiency of the hydrochar.

The manipulating variables in this research are temperature of hydrothermal carbonization and the residence time. The temperatures used are 160°C, 180°C and 200°C. This is because the safe temperature of hydrothermal autoclave reactor is 200°C. Besides that, the residence time is increasing to 3 hours and 4 hours respectively and they only done at fixed temperature of 200°C. Therefore, there are two sets of specimens, which are heated at 160°C, 180°C and 200°C by constant

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duration of 2 hours (set 1) and heated at fixed temperature of 200°C by 2 hours, 3 hours and 4 hours (set 2). There is another sample which is carbonized sawdust heat treated at the temperature of 200°C using furnace by 2 hours and this sample is added in set 1. The agriculture waste material used is only sawdust from UMS Faculty of International Forestry.

1.2 Problem Statement

The potential for agricultural waste treatment reveals the purpose of this study for various environmental applications. This provides eco-friendly technique and a cheap alternative for the production of hydrochar.

The large amounts of wood wastes produced from wood base industries must be utilized and disposed properly. In Malaysia, there are 3.4 million m^3 of wood wastes like sawdust, bark, wood chips and the other raw materials have been generated annually. For sawdust production, Malaysia has produced almost 0.266 million of dry tones in 1995 (Shafie *et al.*, 2017). Unfortunately, improper disposal of wood wastes always happened in construction sites. Due to the disposal of contaminated wood formwork to landfill, a significant environmental burdens and economic wastage occurred (Lin *et al.*, 2021). The aquatic and terrestrial ecosystem have been affected by the improper waste disposal to landfill. Besides that, open burning of wood wastes also occurred in illegal disposal area. The greenhouse gases like carbon dioxide produced from open burning will cause the variety of health and environmental pollution issues. Therefore, utilization of sawdust and wood wastes has been suggested in Malaysia, such as production of charcoal briquettes, conversion of biomass into electricity (electrical generation) and recycling of wood residues in paper companies, composite manufacturers, and bulk companies (Shafie *et al.*, 2017).

For this research, the utilization of sawdust on the synthesis of hydrochar by hydrothermal carbonization treatment will be conducted since there are excessive wood wastes had been disposed improperly and caused the environmental and health issues. The benefits of hydrothermal carbonization are the sawdust can convert to hydrochar with low energy drying method and the hydrochar has a high energy-to-

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weight ratio (Yoganandham *et al.*, 2020). In addition, this research contributes to reduce the improper disposal of wood wastes.

However, using this method that without chemically treated or direct, it cannot generate high quality carbon materials. There are benefits of synthesis of hydrochar derived from waste material, which are cheap fabrication and comparative eco-friendly technique. Furthermore, the waste material used in this research can be easily obtained in large quantity in UMS Faculty of International Forestry and even whole Sabah.

1.3 Research Objective

The objectives of this project are:

i) To synthesize the hydrochar derived from sawdust using hydrothermal carbonization treatment

ii) To characterize the synthesized hydrochar (structure and morphology, elemental composition, light absorbency)

iii) To determine the photothermal efficiency of the hydrochar based solar absorber

1.4 Scope of Works

Scope helps to visualize the guideline for procedures, analysis and works, in order to have a systematic flow for any adjustment and modification.

1) Literature review of thesis, books and journals.

2) Prepare sufficient quantity of precursor for this project.

3) Hydrothermal carbonization of agricultural waste.

4) Data collection and analysis for the surface morphologies, elemental composition, light absorbency and photothermal efficiency of hydrochar

5) Documentation of project progress

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1.5 General Methodology

In order to synthesis and characterize the hydrochar, systematic and proper standard procedure is required in accomplishing this project. The following flow chart illustrate the subsequence procedure in completing this project.

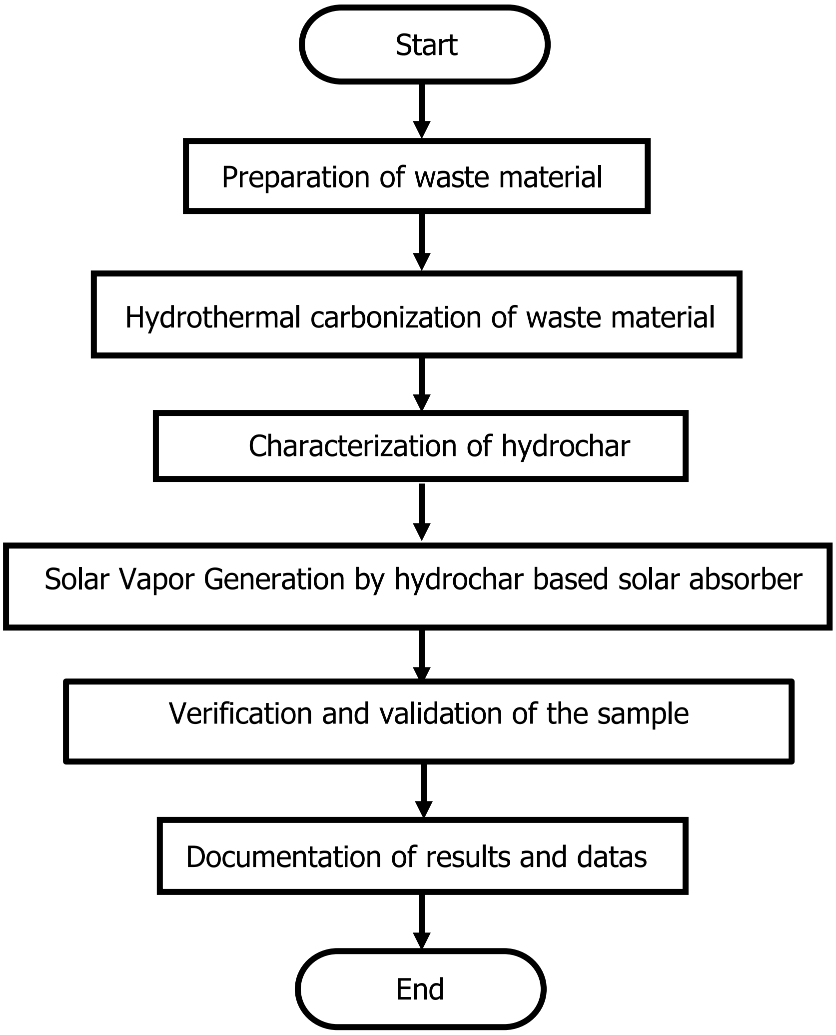

Figure 1.1: Flow chart of the project

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1.6 Thesis Content

This thesis includes the reviews and references from various sources about the background of hydrochar, waste material that used in this research and several applications of hydrochar derived from waste material was mentioned in Chapter 2. Chapter 3 described the research methods that used in the synthesis of hydrochar from waste material, as well as the types of analysis carried out after the hydrochar were prepared. The results obtained are analyzed and discussed in Chapter 4. Finally, the overall conclusion is completed in Chapter 5.

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Chapter 2

LITERATURE REVIEW

2.1 Overview

This chapter will discuss the hydrothermal carbonization, hydrochar, waste materials, sawdust and applications of hydrochar.

2.2 Hydrothermal Carbonization

Hydrothermal carbonization is a thermochemical conversion process that transforms wet biomass waste to hydrochar using heat. There are three reactions occur during the hydrothermal carbonization process, namely dehydration, decarboxylation, and decarbonylation, none of which require pretreatment or drying of the biomass waste (Kumar & Ankaram, 2019). Hydrothermal carbonization is processed in a reactor at the temperatures between 150°C to 250°C, under autogenous pressure, with the residence time ranging from 0.5 to 24 hours. There are operating parameters affect the results obtained like temperature and residence time. The carbon content of the hydrochar increased, but the hydrogen content decreased slightly, and the oxygen content gradually decreased with increasing temperature and residence time. The increase in temperature and residence time starts to remove the oxygen and hydrogen substances. Thus, the hydrochar with low hydrogen to carbon and oxygen to carbon ratios towards coal-like properties. However, the increase in the temperature and residence time leads to a higher degree of material decomposition, fragmentation and solubilisation of organic waste which causes the lower mass yield. Therefore, the optimal condition of the process is determined by the properties of hydrochar. (Wilk *et al.*, 2021)

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2.3 Hydrochar

Hydrochar can be referred as the carbon-rich material synthesized by the hydrothermal carbonization of biomass in the presence of water. It can be synthesized at relatively low temperatures. Besides that, the dry feedstock like rice husk and sawdust or the wet feedstock like animal manures and sewage sludge can be directly used. The hydrochar process is environmentally friendly as it does not produce harmful chemical waste or by-products such as dry pyrolysis. (Niazi *et al.*, 2016)

It begins unevolved from the synthesis of hydrochar with the operating parameters that have the effect on feedstock, like reaction temperature, residence time, biomass-water ratio and presence of catalyst. The reaction temperature affects the characteristics of hydrochar and its thermal behavior. Besides that, the reaction temperature and residence time influence the elemental composition and mass yield of hydrochar. (Wilk *et al.,* 2021) Figure 2.1 has shown scanning electron micrographs (SEM) of hydrochar through hydrothermal carbonization treatment (HTC) by using different residence time but same weight ratio. Longer residence time causes the larger separate spheres formed around every diameter of 100µm. This can be believed that secondary reactions that can occur with higher intensity of HTC treatment. (Sermyagina et al., 2020)

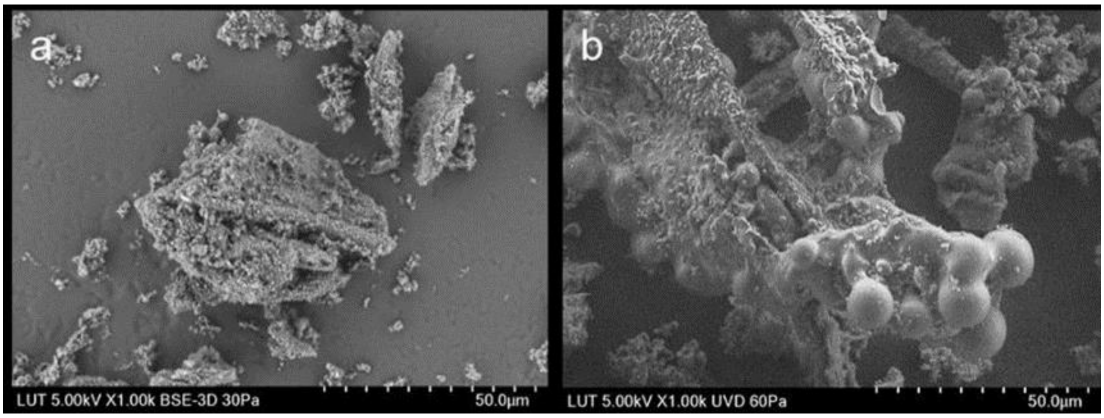

Figure 2.1: SEM of the hydrochar: (a) HTC-2-3/1 (weight ratio of 3:1 ; 2 hours) and (b) HTC-4-3/1 (weight ratio of 3:1 ; 4 hours)

Source: Sermyagina et al. (2020)

The biomass-water ratio also affects the yield of hydrochar. (Oktaviananda *et al.*, 2017) The catalyst is used to increase the degree of carbonization, surface modification, and

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the introduction of key heteroatoms, thereby significantly improving the performance of activated carbon. The catalyst for increasing the carbonization and porosity mostly acidic catalysts, like hydrochloric acid and sulfuric acid. The acidic-catalytic reaction increases the surface area of hydrochar, in addition increases hydrolysis and dehydration reactions and the carbonization level as well. For potassium hydroxide (KOH) activation, hydrochar with rich nitrogen content is used as precursor to synthesize the activated carbon. (MacDermid-Watts *et al.*, 2021) The hydrochar properties like surface structure and morphology, elemental composition, microcrystalline structure, functional group and light absorbency properties can be determined by various characterization apparatus, such as scanning electron microscope (SEM), energy dispersive X-ray analysis (EDX), X-ray diffractometer (XRD), Fournier transform infrared spectroscopy (FTIR) and ultraviolet-visible spectroscopy (UV-Vis).

The uses of hydrochar should be further discussed, because of its advantages in various fields. The feasibility of the biomass waste that can synthesize the hydrochar easily, besides the alternative fossil fuel, the synthesis of hydrochar helps to reduce the waste disposal problems. The low content of nitrogen and sulfur in hydrochar make it more attractive, since the nitrogen and sulfur oxides (NO_x and SO_x) are the airborne pollutants that causing acid rain, thus hydrochar evaluates its competitive advantage in reducing the emission of air pollutants to environment. (Lee *et al.*, 2019)

2.4 Waste Materials

Waste materials fall into categories such as domestic wastes, agricultural wastes, synthetic wastes, commercial wastes, industrial wastes, organic wastes, inorganic wastes and the others. Compared to conventional sources like hydrocarbon, waste materials have the advantages of feasibility and cheap costs in the synthesis of carbon materials. Agriculture industry plays a significant role in contributing the overall economic growth in Malaysia. Large tons of agricultural waste are generated every year, increasing by about 7.5% annually. (Adejumo & Adebiyi, 2020) In Malaysia, there are at least 168 million tons of agriculture waste generated annually, such as wood and oil palm waste, rice husks, coconut fibers, urban wastes and sugar cane wastes.

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