

**MODIFIED BIOCHAR FROM WASTE  
BIOMASS (PALM OIL SLUDGE) TO TREAT  
DYE WASTEWATER.**

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MODIFIED BIOCHAR FROM WASTE BIOMASS (PALM OIL SLUDGE) TO  
TREAT DYE WASTEWATER.

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Thesis submitted in fulfillment of the requirements  
for the award of the degree of  
B. Eng. Tech (Hons) Energy and Environmental

Faculty of Civil Engineering Technology  
UNIVERSITI MALAYSIA PAHANG

FEBRUARY 2023

## ACKNOWLEDGEMENTS

First, I'm thankful to Allah for giving me strength to do this thesis and I'm thanks to myself because worked so hard to do this thesis and never give up. Preparing this thesis, I was in contact with many people, researchers, academicians, and practitioners. They have contributed towards my understanding and thoughts. I wish to express my sincere appreciation to my thesis supervisor, Ts. Dr. Mohd Nasrullah Bin Zulkifli for the encouragement, guidance, critics and understanding. I am also very grateful to my fellow technical advisor, Kak Farah, for their guidance. Without their continued support and interest, this thesis would not have been the same as presented here.

I am also indebted to University Malaysia Pahang (UMP) for the accommodation throughout my bachelor's degree study. I am grateful for the chance to study there and obtain so much knowledge as well as gaining experiences that may very well help me in the future.

My fellow undergraduate students should also be recognized for their support and effort in enduring the last semester. My sincere appreciation to all my colleagues, especially Mohammad Aminuddin bin Zaidon and Muhammad Sufi Shahiran bin Ahmad Azmi, and others who have helped at various occasions throughout the study. Their tips and support are useful indeed. Unfortunately, it is not possible to list everyone in this limited space.

I am grateful for all the unconditional love and support from all my family members. Their unwavering words of encouragement and motivation have helped me so much in the process of completing this study. They are one of the reasons that I can accomplish everything I have and for being who I am today.

## ABSTRACT

Currently, one of the biggest issues affecting the entire world is water pollution brought on by textile manufacturers' inability to properly dispose of their wastewater. Due to the textile industry's large-scale production of brightly coloured wastewater carrying a wide variety of persistent contaminants, wastewater including dyes is a serious polluter of the environment that also negatively impacts human health. Palm oil sludge (POS) is a liquid waste product that can be recovered as low-quality oil. The wastes by the palm oil industry are divided into two categories: solid waste and liquid waste. The palm oil sludge has a bad odour and leads to land and water pollution which indirectly creates hygienic and environmental issues. The expensive and complex types of filtration system in the market make it hard for people to have the good quality of water filter. The idea is to make a low-cost filter by using biomass as a raw material for biochar filter. The biochar undergoes pyrolysis at 3 difference temperature, that is 250C, 300C, 400C and then impregnated in phosphoric acid with 2:1 for chemical. The result is taken and collected. The parameter collected from the data are Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Total Suspended Solid and Colour Admi. Result shown that the temperature effect the result. The best result for all parameter is COD is 61.3%, BOD is 5.13%, TSS is 63.3% and lastly for Colour Admi is 56.75%. Lastly, the temperature at which biochar is produced through pyrolysis can have an impact on its efficiency for treatment of wastewater. This is because the temperature can affect the physical and chemical properties of the biochar, such as its surface area, pore structure, and chemical composition, which can impact its ability to adsorb pollutants from wastewater.



## ABSTRAK

Pada masa ini, salah satu isu terbesar yang menjejaskan seluruh dunia ialah pencemaran air yang disebabkan oleh ketidakupayaan pengeluar tekstil untuk membuang air sisa mereka dengan betul. Disebabkan oleh pengeluaran besar-besaran industri tekstil air sisa berwarna cerah yang membawa pelbagai jenis bahan cemar berterusan, air sisa termasuk pewarna adalah pencemar alam sekitar yang serius yang juga memberi kesan negatif kepada kesihatan manusia. Enapcemar minyak sawit (POS) ialah produk sisa cecair yang boleh diperoleh semula sebagai minyak berkualiti rendah. Sisa oleh industri sawit dibahagikan kepada dua kategori: sisa pepejal dan sisa cecair. Enap cemar kelapa sawit mempunyai bau busuk dan membawa kepada pencemaran tanah dan air yang secara tidak langsung menimbulkan isu kebersihan dan alam sekitar. Jenis sistem penapisan yang mahal dan kompleks di pasaran menyukarkan orang ramai untuk memiliki kualiti penapis air yang baik. Ideanya adalah untuk membuat penapis kos rendah dengan menggunakan biojisim sebagai bahan mentah untuk penapis biochar. Biochar mengalami pirolisis pada 3 suhu perbezaan, iaitu 250C, 300C, 400C dan kemudian diresapi dalam asid fosforik dengan 2:1 untuk bahan kimia. Hasilnya diambil dan dikumpul. Parameter yang dikumpul daripada data ialah Permintaan Oksigen Biokimia (BOD), Permintaan Oksigen Kimia (COD), Jumlah Pepejal Terampai dan Admi Warna. Keputusan menunjukkan bahawa suhu mempengaruhi keputusan. Keputusan terbaik untuk semua parameter ialah COD ialah 61.3%, BOD ialah 5.13%, TSS ialah 63.3% dan terakhir untuk Color Admi ialah 56.75%. Akhir sekali, suhu di mana biochar dihasilkan melalui pirolisis boleh memberi kesan kepada kecekapannya untuk rawatan air sisa. Ini kerana suhu boleh menjejaskan sifat fizikal dan kimia biochar, seperti luas permukaan, struktur liang dan komposisi kimia, yang boleh memberi kesan kepada keupayaannya untuk menyerap bahan pencemar daripada air sisa.

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## LIST OF SYMBOLS

$m^2$	Square Metre
$^{\circ}C$	Degree Celsius
$H_3PO_4$	Phosphoric Acids

## **LIST OF ABBREVIATIONS**

POS	Palm Oil Sludge
WH	Water Hyacinth
TSS	Total Suspended Solid
COD	Chemical Oxygen Demand
BOD	Biochemical Oxygen Demand



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## CHAPTER 1

### INTRODUCTION

#### 1.1 Background of Study

##### 1.1.1 Dye Wastewater

Currently, one of the biggest issues affecting the entire world is water pollution brought on by textile manufacturers' incapacity to properly dispose of their wastewater. (Al-Tohamy et al., 2022). The worldwide economy and environmental degradation are both significantly impacted by the textile industry, particularly China and the estuaries of South Africa. Due to the textile industry's large-scale production of brightly colored wastewater carrying a wide variety of persistent contaminants, wastewater including dyes is a serious polluter of the environment that also negatively impacts human health. Around 7 107 tons of synthetic dyes are generated annually; the textile industry uses over 10,000 tons of these colors. According to their origin, structure, and intended use, dyes are frequently categorized into several groups. (Al-Tohamy et al., 2022)

The textile industry frequently uses azo, direct, reactive, mordant, acid, basic, dispersion, and sulfide dyes among these synthetic colors. Wool, cotton, silk, polyester, polyamide, and acrylic are among the natural and synthetic fibers used in the textile business. In addition, the textile industry employs several very hazardous chemicals at different phases of the production process, including agents for sizing, softening, desiring, brightening, and finishing. However, textile dyes do not adhere to fabric securely and are released untreated as effluent into aquatic habitats including lakes, rivers, streams, and ponds. These dyes have hazardous effects on living things and pose major ecotoxicological risks. (Al-Tohamy et al., 2022)

Heavy elements including mercury, chromium, cadmium, lead, and arsenic, which are necessary in the creation of textile dye color pigments, as well as aromatic compounds have been detected in textile effluent. The creation of textile dye color pigments requires the presence of heavy metals such mercury, chromium, cadmium, lead, and arsenic. Along with the wastewater, these hazardous substances are carried across great distances. They subsequently persist in the water and soil for an extended length of time, causing major health concerns to be living things, decreasing soil fertility and aquatic plants' ability to photosynthesize, and ultimately creating anoxic conditions for aquatic life. Textile dyes affect photosynthesis, plant development, the food chain, recalcitrance, bioaccumulation, and might possibly promote toxicity, mutagenicity, and carcinogenicity. These effects result in a decrease in the aesthetic quality of water bodies. Vast volumes of wastewater with high levels of dissolved solids, organics, metals, salts, and refractory colors are produced because of the large amounts of water utilized in the fabric production process. (Al-Tohamy et al., 2022)

### **1.1.2 Palm oil sludge**

Malaysia, being the world's biggest producer of crude palm oil, has around three hundred sixty-two palm oil mills in operation, producing over Sixty million tons of crop and liquid waste each year. Malaysia was responsible for 25.8% of global palm oil output and 34.3 percent of global palm oil exports in 2020. Malaysia accounted for 9.1% and 19.7% of the world's total production and exports of oils and fats in the same year when other oils and fats produced in the country were included. Malaysia is currently the world's largest exporter. Crude palm oil (CPO) and refined palm oil are the most traded commodities among all palm oil products. Extraction of CPO from fresh fruit bunches necessitates the use of steam for sterilizing and water for dilution, resulting in a significant amount of water being discharged as Palm Oil Mill Effluent (POME). In 2010 Malaysia produce. (Abdullah, 2013)

Due to oil loss during the milling process, Palm oil sludge (POS) is a liquid waste product that can be recovered as low-quality oil. The wastes and by-products generated by the palm oil industry are divided into two categories: solid waste and liquid waste.

POS has a strong foul odor and is a yellowish brown semi-solid at room temperature. The immense waste generated by the palm oil processing poses a hazard to the environment since the waste might be hazardous and requires a large amount of land to be disposed of. It is possible to decrease this waste. Through a chemical or physical process and transformed into a useful product Using waste to generate energy. (Abdullah, 2013)

Adsorbents made from POS have proved to be effective in removing methylene blue, lead, and metal from water in a variety of applications. Many researchers have employed activated biochar for a variety of applications, including heavy metal removal, polycyclic aromatic removal, drug removal, electro sorption, and more. (Iberahim et al., 2019)

### **1.1.3 Water Hyacinth**

These alien species established themselves quickly in this new environment, while native species were occasionally suppressed. When there are no natural predators, this shows that these plants have a high degree of adaptation. These plants are also known for their rapid reproduction in both vegetative and generative states (Pancho J. V.). Water hyacinth takes the place of existing aquatic plants and forms floating mats of interlinked water hyacinth plants that are colonized by a variety of semi-aquatic plant species. Floating mats dominated by big grasses may drift away or be grounded as succession continues. This process can result in rapid and significant changes in wetland ecology, such as the conversion of shallow water areas to swamps. Water hyacinth mats physically slow the flow of water in slow-moving water bodies, allowing suspended particles to precipitate and cause silting. Reduced water flow can lead to flooding and have a negative impact on irrigation systems. In paddy rice, water hyacinth serves as a weed by interfering with germination and establishment. Although this has lately been questioned, water hyacinth is said to produce far more water loss by evapo-transpiration than open water. Water hyacinth displacement can diminish the effective capacity of water reservoirs by up to 400 m<sup>3</sup> of water per hectare, leading reservoir water levels to fall more quickly during dry periods (Wittenberg R, 2001). WH is one of the world's top ten worst weeds, according to the International Union for Conservation of Nature (IUCN) (Tellez T. R,

2008). Most tropical countries' water bodies are infested with WH, which reproduces at a rapid rate (17.5 metric tons/hectare/day), costing millions of dollars to control and eradicate (Simberloff D).

These invasive species prevent sunlight from accessing water systems and reduce oxygen levels, causing water quality to deteriorate and ecosystem lifeforms to suffer. Non-native species that cause economic and environmental harm in places where they are growing are classified as invasive species. Freshwater aquatic plants are the most destructive invasive species on the planet. Most invasive aquatic plants (146 species, or 96.05%) thrive in freshwater. Eichhorniacrasspies, hydrilla (*Hydrilla verticillate*), alligatorweed (*Alternanthera philoxeroides*), Eurasian watermilfoil (*Microphyll spicatum*), water pennywort (*Hydrocotyleranunculoides*), water lettuce (*Pistia stratiotes*), and giant water fern (*Salvinia molesta*) are some of the invasive species (Wang X, 2016). Invasive aquatic plants had significant consequences for ecosystems and economies. Floating invasive aquatic plants can have several negative implications, including limiting access to freshwater for extraction, altering water cycling, and reducing fish and other resource harvesting. Furthermore, the overabundance of floating invasive aquatic plants can disrupt food webs and shade out other aquatic plants. The overabundance of water hyacinth will have an impact on aquatic organism ecology and biodiversity loss.

#### **1.1.4 Biochar**

Biochar may be created from a wide range of organic materials, such as biomass energy crops, agricultural waste, manure, sewage sludge, organic kitchen waste, and so on. Biochar is extremely advantageous due to its numerous uses in industries such as heavy metal and dye removal, pollution cleanup, electricity generation, and so on. Its economic benefit and long-term viability make large-scale implementation possible. Industrial manufacturing processes Pyrolysis technology has emerged as a new area in pollution reduction research, converting lignocellulosic biomass into biochar. During the pyrolysis process, biochar and vapors are formed. During the manufacturing of biochar, gases are emitted. The pyrolysis that follows. The following parameters must be

considered: biomass-based feedstock selection Pre-treatment, particle size, and reaction conditions are all factors. (Spears, 2018)

Biochar is a porous carbon substance created when biomass feedstock is thermochemically decomposed in the absence of oxygen. Biomass derived from POS (palm oil sludge). Pyrolysis, hydrothermal carbonation, gasification, torrefaction, and microwave heating are examples of thermochemical breakdown methods that range in temperature and thermochemical time. Fast pyrolysis and slow pyrolysis are the two types of pyrolysis. We will use a slow pyrolysis method in this research. Fast pyrolysis produces ten percent biochar, seventy percent bio-oil, and twenty percent syngas, whereas slow pyrolysis can produce thirty-five percent biochar, thirty percent bio-oil, and thirty-five percent syngas. The method is chosen depending on how much biochar can be produced. That why choose slow pyrolysis it because slow pyrolysis produces more biochar than fast pyrolysis. Fast pyrolysis only produces ten percent compared to slow pyrolysis thirty-five percent. (Gupta et al., 2022)

Biochar has sparked a lot of interest because of its two different advantages. First, biochar production can offset greenhouse gas emissions because it stores carbon in a stable form, limiting greenhouse gas emissions from biomass degradation into the environment. Second, due to its relatively high surface area and abundance of surface functional groups, biochar is an excellent, low-cost, and environmentally acceptable adsorbent (SFG). Biochar may be used to adsorb metals/metalloids and cleanse water, applied to soils to improve soil fertility and agricultural productivity, used to produce clean energy to partially replace fossil fuels, and used as an adsorbent and catalyst for different pollutants to minimize greenhouse gas emissions. As a result, biochar is becoming more significant as a solution to several global issues like climate change, pollution, and soil degradation.

Biochar's inherent properties make it ideal for recovering nutrients from wastewater, improving activated sludge treatment to reduce energy use for aeration and improve sludge settling ability, increasing energy recovery from sludge through anaerobic digestion, and improving biosolids quality for soil application. Biochar application has been reported to provide agronomic benefits in fertilizer management,

yield, and soil biota. As a potential adsorbent, biochar has potential as a low-cost alternative to activated carbon for wastewater treatment. The integrated use of biochar in wastewater treatment solves today's wastewater management problems. The benefit of using biochar, on the other hand, varies depending on its kind and features, which are dependent on the biomass and pyrolysis circumstances.

Because of its unique properties, including as adsorption capacity, specific surface area, microporosity, and ion exchange capacity, biochar offers a wide range of uses in water and wastewater treatment. The interactions of various pollutants with various attributes of biochar, which are dependent on pyrolysis temperature and feedstock type, govern their removal mechanisms. The temperature at which biochar is pyrolyzed has a significant impact on its characteristics. Biochar has a larger carbon content, hydrophobicity, aromaticity, surface area, and microporosity as the pyrolysis temperature rises. Similarly, when the pyrolysis temperature rises, the pH of the biochar rises due to an increase in ash content in the biochar. Due to the loss of O- and H-containing functional groups, high-temperature (>500°C) biochar exhibits low polarity and acidity. Biochar might be utilized to increase the efficiency of wastewater treatment at various stages. Biochar's use in wastewater treatment might be influenced by microbial cell adsorption, buffering, and immobilization mechanisms. Biochar might help improve the treatment and settling ability of activated sludge by adsorbing inhibitors and hazardous chemicals or providing a surface for microbial immobilization when utilized in the treatment process. Heavy metals (Cr, Cu, Pb, Cd, Hg, Fe, Zn, and as ions) and compounds like nitrate (NO<sub>3</sub>), nitrite (NO<sub>2</sub>), ammonium (NH<sub>4</sub>), phosphorus (P), and hydrogen sulphide (H<sub>2</sub>S) are inorganic pollutants in wastewater that pose a substantial danger to public health and the environment. Biochar made at a lower pyrolysis temperature (about 500°C) has features that make it superior for removing inorganic contaminants. (Pokharel et al., 2020).

## 1.2 Problem statement

Palm oil sludge (POS) may cause serious environmental problems if it is released into the open waterways (Alhaji et al., 2016). At present, most of the POS waste is disposed to the palm plantation itself due to the insufficient utilization of the large waste generated. The sludge has a bad odor and leads to land pollution which indirectly creates hygienic and environmental issues. Therefore, conversion of POS into a value-added material could be a promising way to reduce the impact on the environment.

The environmental effect of dye wastewater leads to the contamination and destruction of natural ecosystems and species living in such areas by exposing them to dangerous substances that may not exist in the natural flow of things. Dye wastewater has a high pH value, high concentration of suspended solids, chlorides, nitrates, metals like manganese, sodium, lead, copper, chromium, iron, and high COD value (Yaseen & Scholz, 2018). Dye wastewater contaminants can affect aquatic life, human health that being exposed to hazardous microbes in wastewater, which can cause diseases including gastroenteritis (diarrhea or vomiting), giardiasis, and cryptosporidiosis (severe stomach cramps, diarrhea, or vomiting). This wastewater is made up of water that has been utilized at home, in industry, and in factories, among other places.

With the existing filter out there, there are so many types of filters used in treating wastewater. Most of the filters are usually very expensive because of their complexity and usually have more downtime to fix clogged filter. This is because there are many layers in the filter. Gravel, coarse sand, charcoal, fine sand, cotton or cloth, and other ingredients are needed to make a water filter.

This is how we come up to control the waste of POS and to make alternative treatment for treating wastewater by using waste palm oil sludge and low-cost material. The process of biochar from Palm oil sludge will go through fast pyrolysis process and apply its as filter then use parameter to see the effectiveness of biochar.



### **1.3 Objective**

The objective of this research study is:

- To synthesis activated carbon from Palm Oil Sludge (POS)
- To design a prototype, recycle bottle of filtration system using activated carbon from palm oil sludge to treat dye wastewater.
- Expensive and complex type of filtration system in the market

### **1.4 Scope Research**

The study's focus was on the synthesis of biochar from palm oil sludge (POS) and water hyacinth. The POS and water hyacinth will be collected in Gambang. At University Malaysia Pahang, the procedure will be carried out in a laboratory environment. The POS and water hyacinth will be collected, crushed, and heated from 120C for about five hours to dry the POS and water hyacinth. Next, TGA analysis is conducted to obtain its suitable temperature range for pyrolysis. After that, POS and water hyacinth undergo a process called fast pyrolysis by taking three temperatures within the temperature acquired from TGA analysis for one hour. Then, the biochar produced is then activated through a process called activation, in which the biochar is impregnated with phosphoric acid. Next, the filter is designed prototype to place the modified biochar as the medium for wastewater filter. The collected dye wastewater will be filtered with the modified biochar. The water filtered is then collected and identifies the parameter, that is COD level, BOD, Total Suspended Solids. The results were then compared with the result from biochar using water hyacinth.

## **1.5 Significant Study**

Biochar is being used to treat dye wastewater because it has a larger surface area, a higher adsorption capacity, and more abundant surface functional groups (SFG). Biochar is a new type of carbon material with a wide range of applications in wastewater treatment. Second, due to its relatively large surface area and abundant surface functional groups, biochar is an effective, low-cost, and environmentally friendly adsorbent (Cha et al., 2016; Inyang et al., 2016). (SFG). Why is it so inexpensive? It does so because it makes use of a low-cost substance. Wastewater treatment is as important for protecting the community. Untreated dye wastewater has the potential to harm aquatic life. This research will show that biochar is a highly effective adsorbent for a variety of contaminants.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Introduction

A literature review is a summary of a topic that contains relevant hypotheses, data, and methods based on prior study. This chapter will go over recent biochar research.

#### 2.2 Raw Material

It is important to classify the biochar feedstock and the production method used, as well as its feasibility, from a production standpoint, because it is largely dependent on the type of feedstock selected (especially, dry, or wet). The classification of biomass into dry or wet feedstocks is determined by its initial moisture content. After harvesting, dry biomass like some wood species and agricultural leftovers has a low moisture content (less than 30%). Wet biomass, on the other hand, is often sewage sludge, algae, vegetable wastes, animal wastes, and other freshly collected biomass with a high moisture content (more than 30%). Prior to processing, wet feedstocks must be dried out. (Knezević, 2009)

#### 2.3 Palm oil sludge

The waste product of the palm oil industry, known as POME, is a liquid waste that can be produced through the processing of crude palm oil. The main oil component of POME is palm oil sludge. (Muanruksa et al., 2019). POME has been extensively treated with anaerobic treatment, resulting in a large volume of sludge. Palm oil sludge (POS) is the solid residue left after treatment in large amounts. As a result, converting POS into a value-added material might be a potential strategy to lessen environmental

effects. Several research on the use of POS as an adsorbent have been published in the literature. Adsorbents manufactured from POS have been demonstrated to be effective in removing methylene blue, lead (Pb<sup>2+</sup>), and metal (Cd<sup>2+</sup> ions) from water in a variety of applications. (Iberahim et al., 2019).

## **2.4 Water Hyacinth**

Water hyacinth or *Eichhornia crassipes* is an invasive floating plant that can be found in bodies of water all over the world. These alien species established themselves quickly in this new environment, while native species were occasionally suppressed. When there are no natural predators, this shows that these plants have a high degree of adaptation. These plants are also known for their rapid reproduction in both vegetative and generative states (Pancho J. V.). These invasive species prevent sunlight from accessing water systems and reduce oxygen levels, causing water quality to deteriorate and ecosystem lifeforms to suffer. Non-native species that cause economic and environmental harm in places where they are growing are classified as invasive species (Wang. X, 2016).

## **2.5 Biochar**

Biochar is one of the three primary products produced when biomass is pyrolyzed under low or no oxygen (O) conditions. Biomass pyrolysis might well be thought of as a system with four interconnected core components or objectives: energy generation, soil improvement, waste management, and climate change and water pollution mitigation. While creating biochar, which may be utilized as an adsorbent for the treatment of contaminated waters, the procedure also minimizes the quantity of biomass (waste) that would otherwise end up in landfills. Biochar is emerging as an ecologically acceptable and cost-effective alternative to other adsorbents such as biomass, industrial waste, and activated carbon for the removal of organic and inorganic pollutants in liquid, solid, and gaseous phases. (Medeiros et al., 2022)

## **2.6 Type of Biochar**

Different feedstock sources and pyrolysis process can change the surface area and porous structure of biochar. Agricultural wastes, rice husks, bagasse, paper products, animal manures, sludge, and even urban green waste are among the various forms of feedstock that may be used to make biochar. The feedstock has an impact on the biochar's adsorption capability, since these minerals can contribute to the development of extra active sorption sites, increasing the adsorption of potentially dangerous metals from wastewater. (Medeiros et al., 2022).

## **2.7 Properties of biochar**

Biochar is a carbon-rich solid substance made from biomass by pyrolysis, a thermochemical process. During pyrolysis, the feedstock's lignin, cellulose, hemicellulose, fat, and starch are thermally broken down, yielding biochar (a solid), bio-oil (partly condensed volatile matter), and non-condensable gases (CO<sub>2</sub>, CO, CH<sub>4</sub>, and H<sub>2</sub>). However, the production of biochar and its characteristics are determined by the pyrolysis setting. Slow pyrolysis at a moderate temperature (350–500°C) and slow heating rate produces a larger yield (30%) of biochar than rapid pyrolysis (600–700°C and fast heating rate) or gasification (temperature 700°C or above), which yields about 10% or less. Surface area, polarity, atomic ratio, pH, and elemental composition all alter significantly depending on the feedstock type and pyrolysis conditions used to make biochar. Biochar's efficacy in wastewater treatment is determined by these qualities. Because of its unique properties, including as adsorption capacity, specific surface area, microporosity, and ion exchange capacity, biochar offers a wide range of uses in water and wastewater treatment. The interactions of various contaminants with various properties of biochar, which are dependent on pyrolysis temperature and feedstock type, regulate the removal methods of different pollutants. The temperature at which biochar is pyrolyzed has a significant impact on its characteristics. Biochar has greater carbon content, hydrophobicity, aromaticity, surface area, and microporosity when the pyrolysis temperature is raised. Similarly, when the pyrolysis temperature rises, the pH of the biochar rises due to an increase in ash content in the biochar. (Pokharel et al., 2020).

## 2.8 Processes for biochar production

Biochar is made by thermochemically converting biomass. Pyrolysis, torrefaction (dry or wet), gasification, and hydrothermal processing are examples of thermochemical processes. For biochar formation, it is important to select the appropriate process and operating conditions (e.g., temperature, vapor and solid residence duration, heating rate, reaction environment). lists the characteristics of various thermochemical processes, as well as typical solid product yields for each process. (Kambo and Dutta, 2015; Liu et al., 2015)

Table 1: Classification of Different Thermochemical Pretreatments in Terms of Operating Conditions and

Process		Operation Temp. (°C)	Heating Rate (°C min <sup>-1</sup> )	Residence Time	Biochar Yield (wt.%)
Pyrolysis	Slow	300–800	5–7	>1 h	35–50
	Fast	400–600	300–800	0.5–10 s	15–35
	Flash	400–1000	~1000	<2 s	10–20
Dry torrefaction		200–300	10–15	30 min–4 h	60–80
Gasification		600–1200	50–100 (°C s <sup>-1</sup> )	10–20 s	<10
Hydrothermal carbonization		180–260	5–10	5 min–12 h	45–70

## 2.9 Pyrolysis

Biochar is made primarily by the pyrolysis of biomass. Pyrolysis is a thermal decomposition process that involves heating biomass at high temperatures under regulated inert circumstances (i.e., very little oxygen or an inert gas environment such as Nitrogen gas). The organic components in biomass are thermally decomposed during pyrolysis, releasing a vapor phase while the solid phase (biochar) remains. Low-molecular-weight compounds (i.e., non - condensable gases such as Hydrogen, Methane, Acetylene, Carbon monoxide, and Carbon dioxide) stay in the gaseous phase while polar and high-molecular-weight compounds (i.e., biooil) are cooled to form liquid phase (i.e., biooil). (Mohan et al., 2006)

Pyrolysis is classified as fast or slow pyrolysis based on its operational parameters, particularly the heating rate. For a brief vapor residence time, fast pyrolysis occurs at 400-600°C with heating rates more than 300°C  $min^{-1}$  (0.5 to 10 seconds). Fast pyrolysis mostly creates bio-oil, but it also produces biochar with a yield of 15-30 wt%. Slow pyrolysis is the method of choice for producing biochar because it yields the highest solid product yield (35 to 50 wt%). This procedure is carried out at temperatures ranging from 300 to 800 degrees Celsius, with heating rates of 5 to 10 ° C  $min^{-1}$ . In slow pyrolysis, vapor residence time can range from a few minutes to many hours. Pyrolysis usually involves physical and chemical processes that are highly dependent on the reactor conditions, operation parameters, and feedstock composition. Such variables have a strong influence on biochar produce and characteristics. A low pyrolysis temperature and a slow heating rate, for example, result in a high yield of solid product. A high pyrolysis temperature, fast heating rate, and long residence time, on the other hand, have an impact on biochar's carbon percentage, surface area, and high heating value (HHV). The amount of fixed carbon in biochar normally rises as the pyrolysis temperature increases. This can be explained in the following way. Volatile matter such as Carbon monoxide, Carbon dioxide, Hydrocarbons, Water, Hydrogen Cyanide, and Ammonia are randomly released from the biomass feedstock during the early stages of pyrolysis (i.e., at low temperatures). The yield of biochar is reduced by the random emission of volatile substances. As the pyrolysis temperature rises, the release of carbon-rich compounds ( $C_xH_yO_z$ ) diminishes, but other volatile chemicals (Carbon Monoxide, Carbon dioxide, Hydrogen cyanide, etc.) continue to be released, resulting in an increase in the fixed carbon content of the residual biochar. Aside from biochar yield and fixed carbon content, pyrolysis temperature affects textural aspects of biochar (e.g., surface area and pore size distribution). For example, increasing the pyrolysis temperature causes more volatile materials to be released from the biomass surface, resulting in a biochar with higher surface area and pores. (Becidan et al., 2007)

It has been discovered that, in addition to pyrolysis temperature and heating rate, high moisture contents (e.g., 40%-60%) boost biochar output at high pressures. This shows that high-moisture biomass may be favored for biochar production, but it may be energy intensive. This shows that high-moisture biomass may be favored for biochar

production, but it may be energy intensive. Another significant parameter for biochar synthesis is the chemical composition of biomass (i.e., the composition of cellulose, hemicellulose, lignin, and inorganics). Inorganic species found in biomass feedstock, such as alkaline earth metals, have catalytic effects on the degradation of biomass and the generation of char. When biomass feedstock is processed with hot water or acid, the output of biochar is reduced. (Manya,2012)

Herbaceous biomass-derived biochar has a low carbon content and a high ratio of PAHs to total organic carbon among the samples. Biochar made from woody biomass, on the other hand, has a high carbon content and low PAH-to-TOC ratios. Furthermore, the pyrolysis temperature has the greatest impact on the surface area and pH of biochar, whereas the total organic carbon content, mineral concentration, ash content, and carbon sequestration capacity of biochar are mostly impacted by the type of biomass feedstock. Table 2 summarizes some of the information available on biochar characteristics as a function of temperature and feedstock type. The results show that by carefully selecting the feedstock and pyrolysis temperature, biochar characteristics may be tailored for certain uses. (Zhao et al.,2013)



Table 2: Characteristics of Biochar Produced by Pyrolysis of Different Biomass Feedstocks

Feedstock	Production Temp. (°C)	Yield (%)	Total C (% Dry Basis)	Fixed C (% Dry Basis)	Volatile Matter (% Dry Basis)	Ash (%)	pH	BET-N <sub>2</sub> Surface Area, (m <sup>2</sup> g <sup>-1</sup> )	Cation Exchange Capacity (cmol kg <sup>-1</sup> )
Wheat straw	200	99.3	38.7	22.5	70.2	7.2	5.4	2.5	32.1
	300	52.5	59.8	53.2	31.3	14.7	8.7	3.5	87.2
	400	29.8	62.9	63.7	17.6	18.0	10.2	33.2	95.5
	500	26.8	68.9	72.1	11.1	16.2	10.2	182.0	146.0
Sawdust	500	28.3	75.8	72.0	17.5	9.9	10.5	203.0	41.7
Grass	500	27.8	62.1	59.2	18.9	20.8	10.2	3.3	84.0
Peanut shell	500	32.0	73.7	72.9	16.0	10.6	10.5	43.5	44.5
Pig manure	200	98.0	37.0	12.6	50.7	35.7	8.2	3.6	23.6
	300	57.5	39.1	34.7	27.4	37.2	9.7	4.3	49.0
	400	38.5	42.7	40.2	11.0	48.4	10.5	47.4	82.8
	500	35.8	45.3	19.2	10.7	69.6	10.8	42.4	132.0
Cow manure	500	57.2	43.7	14.7	17.2	67.5	10.2	21.9	149.0
Shrimp hull	500	33.4	52.1	18.9	26.6	53.8	10.3	13.3	389.0
Chlorella	500	40.2	39.3	17.4	29.3	52.6	10.8	2.8	562.0
Bone dregs	500	48.7	24.2	10.5	11.0	77.6	9.6	113.0	87.9
Waste paper	500	36.6	56.0	16.4	30.0	53.5	9.9	133.0	516.0
Wastewater sludge	500	45.9	26.6	20.6	15.8	61.9	8.8	71.6	168.0
Waterweeds	500	58.4	25.6	3.8	32.4	63.5	10.3	3.8	509.0

As a result of recirculating gaseous products and recovering liquid products to improve the efficiency of slow pyrolysis, pyrolysis is an environmentally friendly and cost-effective biochar production process. The yield and qualities of biochar are strongly linked to the parameters of the specific process and the type of feedstock. (Zhang et al., 2010)

## 2.10 Wastewater

Water that has been contaminated by domestic, industrial, or commercial use is referred to as wastewater. As a result, the composition of all wastewaters is continually changing and extremely varied, making it impossible to pin down a single definition of the term. The composition of wastewater is 99.9% water, with the remaining 0.1 percent being removed. Organic materials, bacteria, and inorganic chemicals help compensate

0.1 percent. Wastewater effluents are discharged into lakes, ponds, streams, rivers, estuaries, and seas, among other places. Storm runoff comprises dangerous elements that wash off roadways, parking lots, and rooftops. Raw wastewater, often known as raw sewage, is a type of wastewater. 3 types of wastewaters domestic, industrial, and commercial. (TUSER, 2020)

### 2.10.1 Wastewater Domestic

Water that is discarded from homes, workplaces, and industry is referred to as wastewater. It was previously known as sewage and originates from toilets, sinks, showers, washing machines, and industrial operations. Wastewater is the water that is dumped from households, businesses, and industries. Because it comes from toilets, sinks, showers, washing machines, and industrial processes, it was originally known as sewage. (ILIAS 3, 2005)

Table 3: Type of wastewater

- <b>yellow water:</b>	human urine
- <b>brown water:</b>	human faeces with flushed water (can include paper if used)
- <b>black water:</b>	human faeces (brown water) mixed with urine (yellow water), in general: wastewater from toilets. It contains human waste and can be a public health risk if not treated properly. (Sometimes, water used in kitchen is also classified as black water)
- <b>grey water:</b>	water used in the kitchen, bathroom including sinks, baths, showers and laundry, etc. or any water that has been used at home. except water from toilets

As illustrated in Table 3, wastewater components can be classified into several categories. If released into the environment, they can have a negative impact on aquatic life. (ILIAS 3, 2005)

Table 4: Wastewater Component

<b>Component</b>	<b>Of special interest</b>	<b>Environmental effect</b>
Microorganisms	Pathogenic bacteria, virus and worms eggs	Risk when bathing and eating shellfish
Biodegradable organic materials	Oxygen depletion in rivers and lakes	Fish death, odours
Other organic materials	Detergents, pesticides, fat, oil and grease, colouring, solvents, phenols, cyanide	Toxic effect, aesthetic inconveniences, bioaccumulation in the food chain
Nutrients	Nitrogen, phosphorus, ammonium	Eutrophication, oxygen depletion, toxic effect
Metals	Hg, Pb, Cd, Cr, Cu, Ni	Toxic effect, bioaccumulation
Other inorganic materials	Acids, for example hydrogen sulphide, bases	Corrosion, toxic effect
Thermal effects	Hot water	Changing living conditions for flora and fauna
Odour (and taste)	Hydrogen sulphide	Aesthetic inconveniences, toxic effect
Radioactivity		Toxic effect, accumulation

### 2.10.2 Wastewater Industrial

Means non-toxic, non-hazardous wastewater from commercial establishments that is normally comparable in composition to residential wastewater but occasionally exceeds typical domestic values in one or more elements. Waters from commercial and institutional food service operations, commercial laundry facilities with no more than four washing machines, animal holding facilities (such as kennels, veterinary hospitals, and animal grooming facilities), and beauty salons are all included in this definition, if no toxic, hazardous, or industrial wastes are introduced into the system. (Commercial Wastewater Definition, 2019)

### 2.10.3 Wastewater Commercial

Industrial wastewater is defined as water- or liquid-carried waste from any industry, manufacturing operation, trade, or business that includes any combination of process wastewater, cooling water, contaminated storm water, contaminated leachates, or other waters, and is subject to regulation under Federal Categorical Pretreatment Standards, the State Waste Discharge Permit Program, or this chapter.

#### 2.10.4 Characteristic Wastewater

Characteristic of wastewater have three characteristics first is physical, chemical, and biological. Temperature, solids, odor, and color are all physical properties of wastewater. (Jamal, 2017)

- Temperature: Due to home and industrial operations, the temperature of sewage is frequently higher than that of water. The average yearly temperature of sewage varies by geographical area and ranges from 10 to 21°C. Because of its impact on chemical reaction rates and aquatic life, sewage temperature is an important parameter. The presence of fish species in water bodies may alter when the temperature rises. Similarly, oxygen is less dissolved in warm water, and the population of some aquatic species rises with temperature, creating increased demand for oxygen and resulting in dissolved oxygen depletion in the summer. (Jamal, 2017)

- Color: The color of fresh sewage is a light brownish grey. Sewage will transform from fresh to old in 2 to 6 hours if the temperature is over 20 °C. Due to anaerobic activity, old sewage becomes a dark grey or black hue, which is known as an old or toxic color. Domestic wastewater is colored by some industrial effluent. The grey, dark grey, and black colors are caused by sulfide, which is formed under anaerobic conditions and interacts with the metals in the wastewater. (Jamal, 2017)

- Odor: The odor of fresh residential sewage is somewhat soapy or oily. Stale sewage has a strong Hydrogen Sulfide odor (H<sub>2</sub>S). At low concentrations, the odor has no impact, but at high concentrations, it produces a loss of appetite, decreased water consumption, difficulty breathing, vomiting, and other symptoms. (Jamal, 2017)

- Solids: Matter suspended or dissolved in water and wastewater is referred to as solids. Solids are split into many fractions, and their concentrations give significant information for wastewater classification and treatment process management. (Jamal, 2017)

Secondly is chemical wastewater. Organics make around 75% of suspended particles and 40% of filterable solids in sanitary wastewater. Animals, plants, and people

all contribute to the production of these solids. Protein (40-60%), carbohydrates (25-50%), lipids and oils are among the organic components present in wastewater (10%). Wastewater contains pollutants as well. Inorganic compounds can also be found in wastewater. Chemical features of wastewater are determined by tests such as BOD, COD, nitrogen, phosphorus, alkalinity, and others. (Wastewater Characteristics, 2009). Chemical characteristics of sewage is organic matter, chloride, biological oxygen demand (BOD), Dissolved oxygen (DO), pH, Nitrogen and Oxidation Reduction (O-R) potential: (Karki, 2019)

- Organic Matter: Wastewater includes a considerable quantity of organic stuff in general. However, the amount of organic matter in wastewater is determined by the kind and quality of the wastewater. Organic matter in wastewater can be detected as dissolved chemicals, colloidal materials, suspended particles, or sedimented particles. (Karki, 2019)

- Chloride: Humans produce a substantial quantity of chloride in the form of sodium chloride (8-15 gm/day), mostly through urine and perspiration. As a result, the chloride content of residential wastewater from the toilet and bathroom is increased. (Karki, 2019)

- Sulfide: During the anaerobic breakdown of organic waste by anaerobic bacteria, sulphatic in the form of H<sub>2</sub>S (hydrogen sulfide) is produced in wastewater. Wastewater has a foul odor due to Hydrogen sulfide. (Karki, 2019) (Karki, 2019)

- Biological oxygen demand (BOD): Wastewater typically has a high BOD due to the significant quantity of organic waste present. BOD concentrations range from 100 mg/liter in extremely diluted wastewater to 600 mg/liter or higher in concentrated wastewater including industrial effluent mix. (Karki, 2019)

- pH: Wastewater is slightly alkaline in pH. (Karki, 2019)

- Nitrogen: Fresh sewage is mostly made up of organic nitrogen, with very little inorganic nitrogen. (Karki, 2019)

Wastewater biologicals contain bacteria, Algae, Fungi, Virus, and Protozoa.

- Algae: Chlorella phormium, Ulothrix, and other algae found in sewage. In sewage treatment plants, algae are employed in trickling filters. (Karki, 2019)

- Fungi: In wastewater, fungi such as Fusarium and Sporidium are discovered, and they play a vital function in the trickling filter. (Karki, 2019)

- Virus: Some viruses that cause human disease, such as Poliovirus, Rotavirus, Hepatitis A and E, are present in sewage and are transmitted to patients through their feces. (Karki, 2019)

- Protozoa: Some protozoa that cause intestinal sickness enter the sewage system with the patient's feces. Pathogenic protozoa include Entamoeba histolytica, Giardia, and Balantidium coli, among others. In trickling filters, a few protozoa such as Vorticella and Opercular can be detected. (Karki, 2019)

## **2.11 Total suspended solid**

Total suspended solids, or TSS, are waterborne particles larger than 2 microns in diameter. A fully dissolved solid, on the other hand, is defined as any particle less than 2 microns (TDS). Although inorganic materials comprise most total suspended solids, algae and bacteria can also be included. TSS can be sand, silt, or plankton, as well as anything else that floats or "suspends" in water. The organic particles released into the water when some water sources are contaminated with decaying plants or animals are usually suspended solids. While some sediment settles at the bottom of a water source, TSS floats on the surface or remains suspended somewhere in the middle. TSS affects water's clarity, so the higher a water source's TSS content, the less clear it will be.

### **2.11.1 Effect of Total Suspended Solid**

TSS levels above a certain threshold can block light from reaching submerged vegetation. Photosynthesis slows when the amount of light passing through the water decreases. Plants release less dissolved oxygen into the water when photosynthesis rates are reduced. Bottom-dwelling plants will stop producing oxygen and die if light is fully blocked. Bacteria will use even more oxygen from the water as the plants decay. Fish kills can occur when there is a lack of dissolved oxygen. Because suspended particles

absorb heat from the sun, high TSS can induce an increase in surface water temperature. TSS causes a decrease in water quality, which might hinder fish's ability to perceive and catch food. Suspended debris can block fish gills, lower growth rates, impair illness resistance, and inhibit the development of eggs and larvae. Suspended particles can choke freshly born insect larvae and smother the eggs of fish and aquatic insects as they drop to the bottom of a water body. Settling sediments can fill up crevices between rocks that aquatic organisms could have used as habitats. TSS levels beyond a certain level can indicate greater bacterium, nutrient, pesticide, and metal concentrations in the water. These contaminants may attach to sediment particles on the bottom and then be transferred into water bodies by storm water. Pollutants may be released from the sediment or transported further downstream in the water. High TSS can clog or scour pipes and machines, posing a challenge for industrial application. (Murphy, 2007)

### **2.11.2 Factors Affecting Total Suspended Solids**

**High Flow rate:** TSS concentrations are influenced by the flow rate of the water body. Fast-moving water can carry more particles and debris of a greater size. Sand, silt, clay, and organic particles (such as leaves, dirt, and tire particles) can be picked up by heavy rains and carried to surface water. TSS can be affected by changes in flow rate, if the speed or direction of the water current changes, particulate particles from the bottom sediments may be resuspended. (CAMPBELL, 2021)

**Soil Erosion.** The disturbance of a land surface causes soil erosion. Building and road construction, forest fires, logging, and mining can all contribute to soil erosion. Stormwater can transport eroded soil particles to surface water. The TSS of the water body will rise because of this.

**Urban Runoff.** Soil particles and trash from streets, industrial, commercial, and residential areas can be washed into streams during storm occurrences. Infiltration is reduced, velocity is increased, and natural settling places have been removed because of the huge volume of pavement in urban areas. Sediment is delivered straight to streams and rivers via storm drains.

Human Pollution. TSS levels in water sources across the United States are due to human activity. Pathogens and heavy metals that are dissolved in water can adhere to suspended water particles, lowering water quality. Pesticides, lead, bacteria, and mercury are examples of common human pollution contaminants. (CAMPBELL, 2021)

## **2.12 pH**

The pH scale determines how acidic or basic water is. The range is 0 to 14, with 7 representing neutrality. Acidity is indicated by a pH of less than 7, whereas baseness is indicated by a pH of higher than 7. PH is a measurement of the proportion of free hydrogen and hydroxyl ions in water. While water with more free hydroxyl ions is basic, water with more free hydrogen ions is acidic. Since chemicals in the water may change pH, pH is a crucial sign that the chemical composition of the water is changing. "Logarithmic units" are used to report pH. Each number corresponds to a 10-fold difference in the water's acidity or basicity. The pH of the water will fluctuate depending on the aquatic life in the pond. When aquatic creatures respire, carbon dioxide is released into the water, generating an acidic response due to all species' respiration and CO<sub>2</sub> synthesis, and the pH value drops at night. Phytoplankton and other aquatic plants take CO<sub>2</sub> from the water through photosynthesis throughout the day, raising the pH of ponds. Waters with moderate alkalinity have a stronger buffering capacity and reduced pH volatility. The pH levels in the pond fluctuate. (Pal, 2017)

## **2.13 Color Admi**

The ADMI color scale is developed to measure the color of wastewater as an indicator of water quality. ADMI color is a metric quantity based on the Adams Nickerson color difference between the APHA/Pt-Co/Hazen liquid standards and distilled water. (Suwanboriboon et al., 2018)



## **2.14 Chemical Oxygen Demand**

The chemical oxygen demand (COD) is a metric for measuring the quality of water and wastewater. The COD test is frequently used to measure the performance of water treatment plants. This test is because, in acidic conditions, a powerful oxidizing agent can completely oxidize practically any organic substance to carbon dioxide. COD is the amount of oxygen used to convert organic water pollutants to inorganic end products through chemical oxidation. Under acidic conditions, COD is frequently evaluated using a strong oxidant (e.g., potassium dichromate, potassium iodate, potassium permanganate). The oxidant is introduced to the sample at a level that is known to be excessive. After the oxidation process is complete, the amount of oxidant left in the solution is measured to determine the concentration of organics in the sample. Titration with an indicator solution is commonly used to accomplish this. The mass of oxygen consumed per liter of solution is measured in milligrams per liter of COD.

## **2.15 Summary**

An overview of a literature review comprises relevant information methodologies, and hypotheses based on prior study. This chapter will examine recent research on the general topic of biochar in dye wastewater treatment. This literature review will give information research regarding the type of wastewater, pyrolysis, type of biomass that being using to make biochar and the parameters such as pH, COD, SS, Color Admi. This literature review will give detailed information about this project modified biochar from waste biomass to treat dye wastewater.

## **CHAPTER 3**

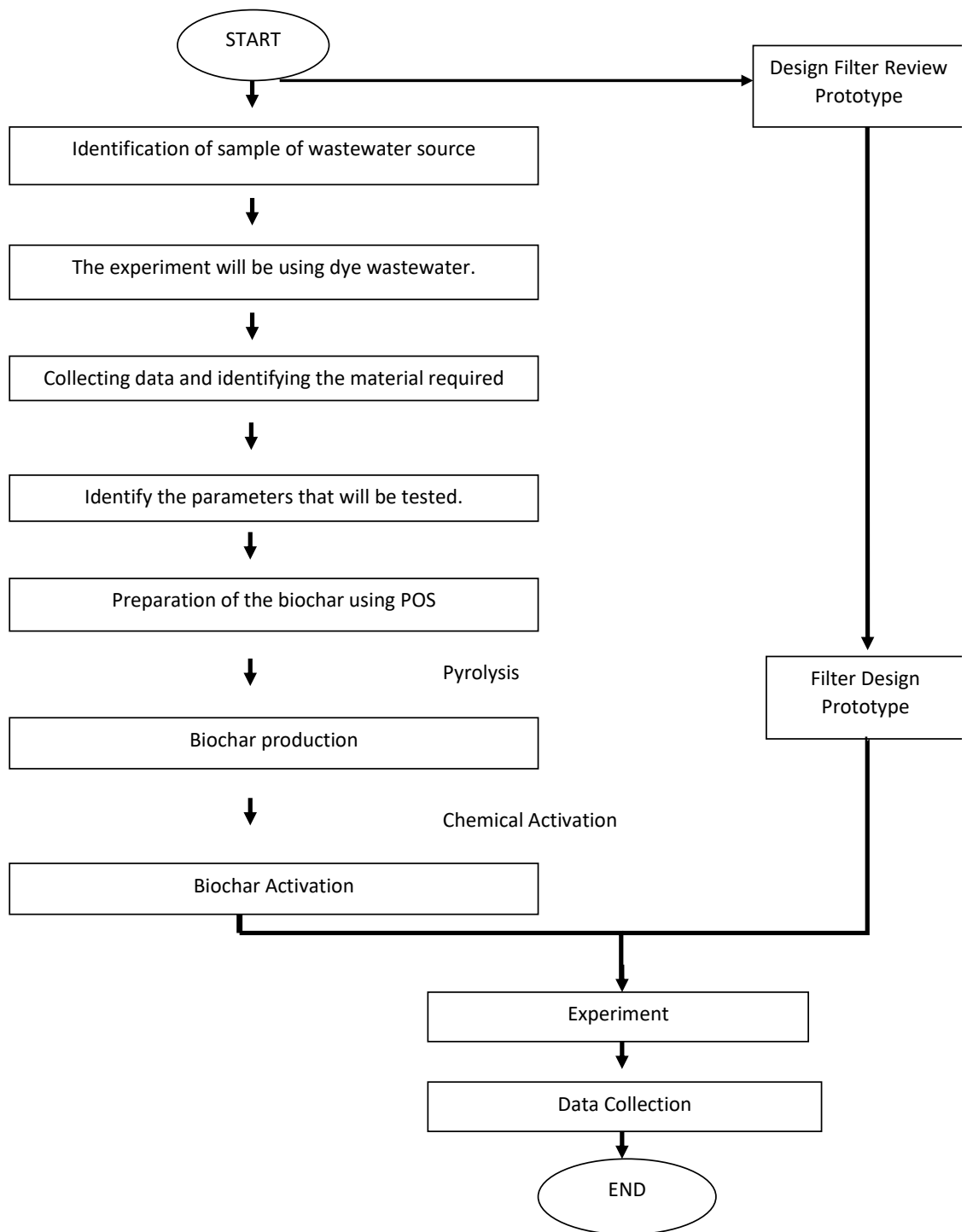
### **METHODOLOGY**

#### **3.1 Introduction**

Methodology is a way to find out the result of a given problem on specific research. This chapter will describe the method to identify and justify the sampling method and demonstrate the techniques and instrument used for qualitative and quantitative of the expected result for given research problem. This research is to make biochar using Palm oil sludge and to design a filter for domestic wastewater treatment.

#### **3.2 Research Approach**

Figure show the research emphasized on both field study analysis and laboratory method. In-situ analysis involved in this research includes the mechanism of fast pyrolysis from Palm oil sludge.



### 3.3 Biochar production from Palm Oil Sludge (POS)

#### 3.3.1 Palm Oil Sludge



Figure 1: Collect Pome (decanter cake)



Figure 2: Put Pome in Container



Figure 3: POME in container

The palm oil mill sludge (decanter cake) was collected at Lepar Hilir FGV Palm Factory. The pome (decanter cake) was gathered and dried in the sun to minimize moisture. It took about three days to dry out in the sun. After drying in the sun, it was baked in an oven at 120°C for 5 hours to eliminate any remaining moisture from the sludge. Then it was crushed into fine particles to make it easy to fill it into a porcelain bowl before it was inserted into the furnace to go for the pyrolysis process. After that, it will be kept in an airtight container to minimize moisture buildup and fungal infestation.

### 3.3.2 Chemical Reagent

The choice of activating agents is based on the application of biochar. Biochar is activated with acid for better adsorption capacity. The reagent that being used to activate biochar is phosphoric acid. Phosphoric acid is being used in chemical activation. Concentration for the phosphoric acid is 80%.

### 3.3.3 TGA

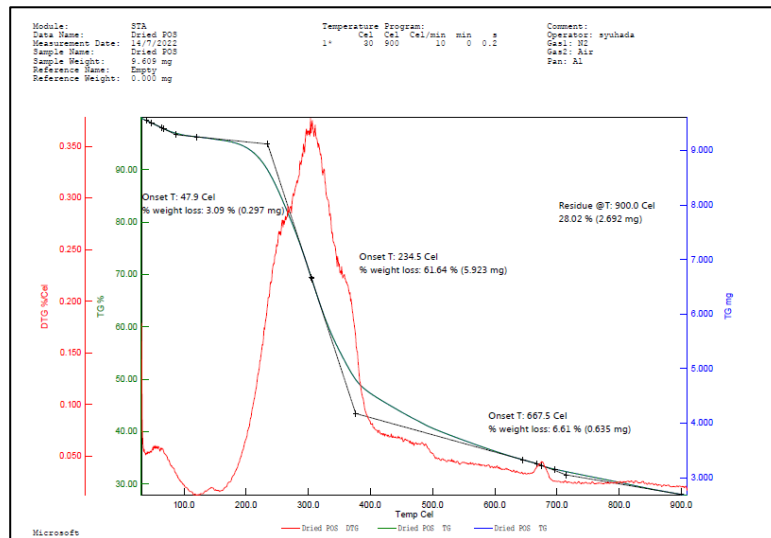


Figure 4: TGA Result

Send the sample POS to the lab to obtain the TGA results after obtaining it. Thermogravimetric analysis (TGA), an analytical technique, can be used to determine a material's thermal stability and the fraction of volatile components by watching the weight change that occurs while a sample is heated at a constant rate. Utilize TGA to determine the pyrolysis process' ideal temperature. Gas nitrogen is used. The TGA result for the sample POS is shown in Figure 3.

### 3.3.4 Fast Pyrolysis



Figure 5: POS in Porcelain bowl



Figure 6: Sample in Furnace



Figure 7: Sample after taking it out from furnace

Based on the TGA results, determine the furnace's temperature. Three samples, each with a different temperature, were pyrolysis at 250°C, 300°C, and 400°C. The procedure was carried out with a small amount of undiluted air. A sample of dried sludge weighing 400g was placed in each porcelain bowl, followed by the addition of 50g to the furnace. Based on samples 1, 2, and 3, the furnace oven's temperature is set. 250°C for two hours is used for sample 1. Another sample 2 (300°C) and sample 3 (400°C) underwent the same procedure.



### 3.3.5 Chemical Activation



Figure 8: Sample soaked in phosphoric acid



Figure 9: Wash sample with distilled water

The activation of biochar using chemical processes. The reagent chemical is Phosphoric acids. Chemical activation with Phosphoric acid was carried out in a laboratory. The sample(char) was mixed with Phosphoric acid with a mass ratio of 1:2 .200 ml of phosphoric acid is impregnated to the biochar. The sample soaked in

phosphoric acid for two hours before being washed with distilled water. The sample was heated in the oven for one hour at 120 C. The sample should be taken out and put in a container and kept in a drying cabinet.

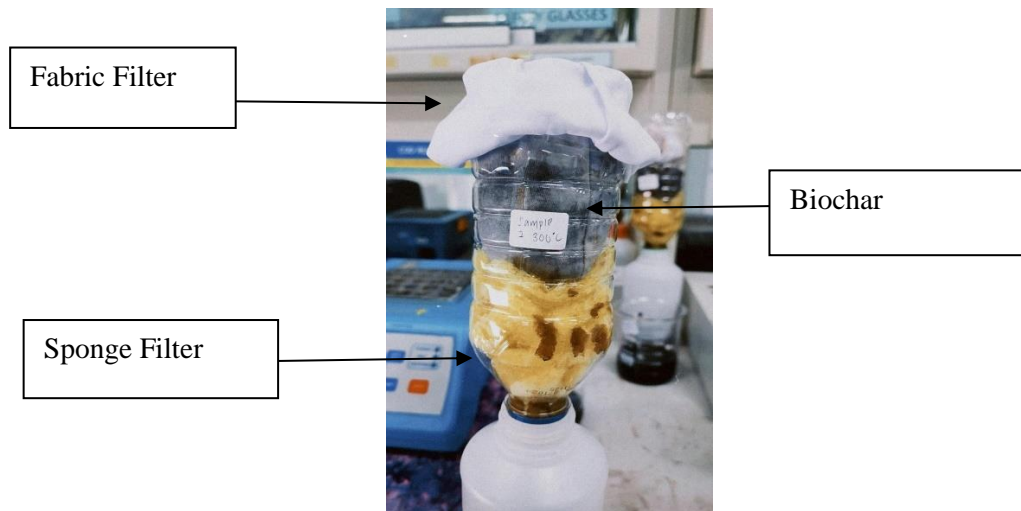
### 3.3.6 Collected Dye Wastewater



Figure 10: Sample of dye water

The dye wastewater was kept in a 3Liter sample bottle and was collected from RM Batik. After that the dye wastewater will put in the chiller

### 3.3.7 Design Filter



The filter material consists of fabric filter, filter and activated carbon biochar, according to the filter design scheme. Charcoal filters and fabric filters will be used to filter dye wastewater. The dye wastewater will be put in the filter and the dye wastewater will pass through the biochar and filter. The water that has been filtered will go into the bottle. Filtered water will be used for testing. Each sample will have 500ml of filtered water.

### 3.3.8 Parameter

The purpose of this experiment was to demonstrate the potency of biochar as an adsorbent for water filters. The parameter chosen based on characteristics of dye wastewater.

### 3.3.8.1 Biological Oxygen Demand (BOD) and Dissolved (DO)



Figure 11: DO sample

The accuracy of BOD test totally depends upon the proper bacterial growth, present in the water sample. pH of the diluted sample should be adjusted 7.00 before the incubation for five days for proper results. The sample should be neutralized in the following manner. Take 100 ml of sample. Measure the pH of the solution by using the calibrated pH meter. Add the sulfuric acid to neutralize pH. Take diluted water 400 ml and sample water 100 ml into a beaker before fill into the BOD bottles. Take the result using DO meter. Then, immediately close the bottles when filled and there should not be any air bubble in the bottle. Mark the bottles as blank and sample. Put in the incubator for 5 days. Repeat the steps with another sample. After 5 days take out the sample in the incubator and let the sample in room temperature. Take the DO result using DO meter. Calculate the DO result to get BOD result. Using formula BOD.

$$BOD \frac{mg}{l} = \frac{D_i - D_f}{p}$$

$D_i$  = initial dissolved O<sub>2</sub> concentration

$D_f$  = final or 5-day dissolved O<sub>2</sub> concentration

$p$  = Volumetric fraction of wastewater

### 3.3.8.2 COD



Figure 12: Sample COD High Range

First, gather the sample in the proper container size and preserve it with the correct amount of sulfuric acid. Then provide enough prepared COD vials with the appropriate labels (without mercury for non-regulatory samples) to allow for the digestion of all samples and standards. Third, make sure the COD reactor is adjusted to 150°C and turn it on. By blending or shaking, homogenize the sample. Diluted the sample with distilled water ratio is 1:10. Replace the cap after adding 2 mL of the sample diluted. Place the vials containing the samples or standards in the reactor after it has reached the right temperature, and then set the timer for two hours. Next, remove the vials from the reactor after two hours so they may cool to room temperature. Lastly, read the absorbance after setting the spectrophotometer to the correct wavelength. Result needs to be calculate using formula Dilution Factor.

$$\text{Dilution Factor} = \frac{\text{Final Volume of Diluted Solution}}{\text{Initial Volume of the Undiluted Solution}}$$

### 3.3.8.3 Total Suspended Solid



Figure 13: Some of apparatus need to use



Figure 14: Wash disc



Figure 15: Disc in the oven



Figure 16: Disc into the desiccator

Switch on oven  $105^{\circ}\text{C}$ . Insert filter disc onto base and clamp. On vacuum and wash disc with 20ml distilled water. Remove all traces of water by shake slowly the suction flask. Switch off vacuum and remove funnel from base. Place the filter disc on the watch glass. Dry in an oven  $105^{\circ}\text{C}$  for 1hour. Take out the filter paper from the oven and put the filter disc into the desiccator 20 minute – 30 minutes, after that takeout filter disc in analytical balance and take reading and write the data. Place the filter on base and

clamp on funnel and shake sample. Pour the sample 100ml into measuring cylinder and wash measuring cylinder with distilled water. Pour into funnel and wash funnel using distilled water. Wait few second and switch off vacuum and remove filter paper. Dry in oven at 1 hour at 105°C. Take out filter disc from oven and put desiccator about 20 minute – 30 minutes. Wash the filter disc in analytical balance and take reading and write data.

#### **3.3.8.4 Colour ADMI**

The ADMI colour scale is to measure the colour of wastewater as an indicator of water quality. ADMI colour is a metric quantity based on the Adams Nickerson colour difference between the APHA/Pt-Co/Hazen liquid standards and distilled water. The Adams Nickelson colour formula is obtained by transforming CIE tristimulus colour indices into a uniform metric colour scale that is independent of hue. If two colours of different hues are different to the same degree from the reference colourless water, then the difference will be the same. For a general-purpose spectrophotometer with a measuring range of 360 nm to 830 nm and the wavelength resolution of 1 nm, we developed an ADMI colour application based on APHA 2120F weighted-ordinate spectrophotometric method for measuring the colour of wastewater in the range of 0 ADMI to 500 ADMI.



## **CHAPTER 4**

### **RESULTS AND DISCUSSION**

#### **4.1 Introduction**

This chapter will focus on the results and findings obtained through the experiments conducted. The results are tabulated and presented in a graphical manner. Based on chapter 1, the experiment was made to determine whether biochar using palm oil sludge can help in the treatment of dye wastewater. In the experiment, the variable used is temperature. Thermogravimetric analysis (TGA) is used to determine the temperature of pyrolysis.

## 4.2 Result Thermogravimetric analysis (TGA)

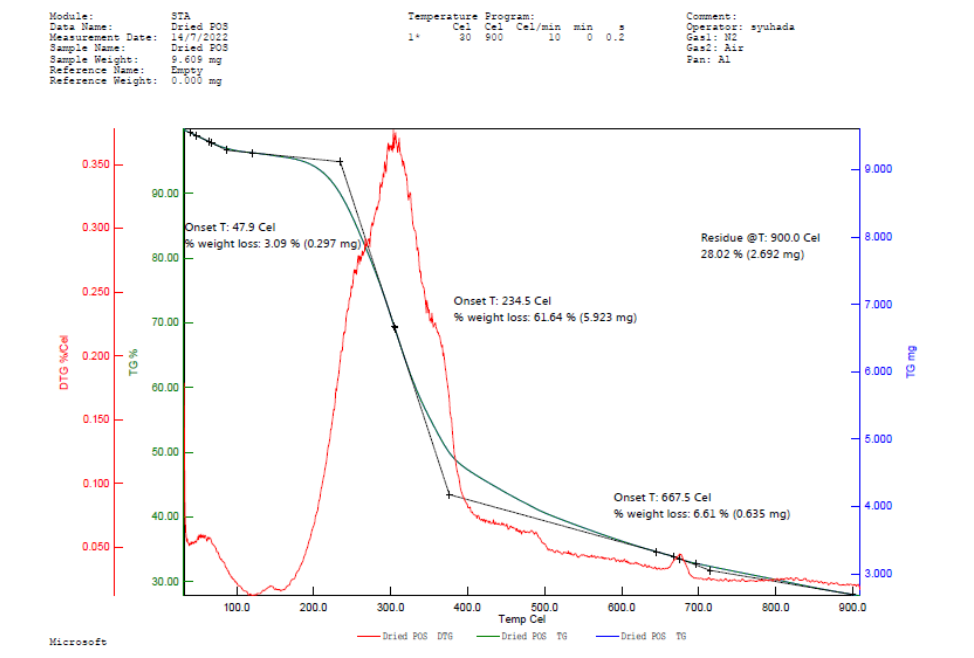


Figure 17: TGA Result

To investigate the pyrolysis mechanism of substances, thermogravimetric analysis (TGA) is frequently utilised. Thermodynamic information and kinetic parameters of the decomposition process can be obtained through TGA, which enables the investigation of thermal stability and thermal breakdown mechanisms. Pyrolysis kinetics is a frequently utilised tool for investigating biomass. The degradation of three pseudo components linked to the three primary components of lignocellulosic biomass—cellulose, hemicellulose, and lignin—is generally the focus of biomass pyrolysis kinetic research on TGA. Additionally, the peak thermal degradation activation energy varies for each of these biomass components (Kristanto et al., 2021). The results of the TGA experiment illustrating the thermal decomposition and heat flow of cellulose are displayed in Figure 15. Between 200°C and 600°C, volatile weight loss and moisture removal take place. A good reaction temperature for the pyrolysis process may be seen in the rising temperature graph. The rising temperature graph shows a desirable reaction temperature for the pyrolysis process. Three temperatures have been selected for this

experiment based on the TGA result. In table 5, the three temperatures are shown. (Kristanto et al., 2021)

Table 5: Sample Temperature

NO.	Sample	Temperature
1.	Sample initial	
2.	Sample 1	250°C
3.	Sample 2	300°C
4.	Sample 3	400°C

### 4.3 Effect of temperature

The temperature at which biochar is produced through pyrolysis can have an impact on its efficiency for treatment of wastewater. This is because the temperature can affect the physical and chemical properties of the biochar, such as its surface area, pore structure, and chemical composition, which can impact its ability to adsorb pollutants from wastewater. The temperature of the pyrolysis to process the biochar can be varied and have different effects on the biochar. To activate the carbon, phosphoric acid was utilized as a chemical activator. A few experiments have been carried out to determine the influence of different temperatures on the activation of carbon. COD, BOD, TSS, pH, and color admitt test are all part of the test.

#### 4.3.1 Effect of temperature biochar on Chemical Oxygen Demand (COD)

Chemical oxygen demand (COD) is a measure of the amount of oxygen required to oxidize all the organic and inorganic matter in a water sample. Based on result dye wastewater, the COD is higher due to the presence of high levels of organic compounds, such as dyes, which are released into the water during the dyeing process. These dyes and other organic compounds can be difficult to oxidize, leading to an increased demand for oxygen to remove them from the water. The high COD levels in dye wastewater can also

cause problems in treatment systems, as they can lead to decreased oxygen levels in the water, which can harm aquatic life and create anaerobic conditions that can support the growth of harmful bacteria.

In this study the dye wastewater has higher chemical oxygen demand since its from industrial wastewater. Below is the result of industrial wastewater that being record. Sample 0 is the initial sample, which has not been treated in any way. The sample dye wastewater needs to dilute because it over measuring. The diluted factor is 1/10.

Table 6: Effect of Biochar Temperature on COD value

<b>SAMPLE</b>	<b>INITIAL CONCENTRATION (MG/L)</b>	<b>FINAL CONCENTRATION (MG/L)</b>	<b>REMOVAL EFFICIENCY, E (%)</b>
<b>0</b>	3210	3210	0.00%
<b>1</b>	3210	2540	20.87%
<b>2</b>	3210	2170	32.39%
<b>3</b>	3210	1240	61.37%

Table 6 shows the COD level for dye wastewater after interacting with the biochar adsorbent using palm oil sludge. The final COD level for the adsorbent temperature of 250°C is 2540 mg/L but for the adsorbent dosage of 300°C and 400°C, COD level is slightly decreasing to 2170 mg/l to 1240 mg/L. The standart COD level for the dye wastewater or industrial wastewater is below 250 mg/L which make it the susitable condition for discharge.

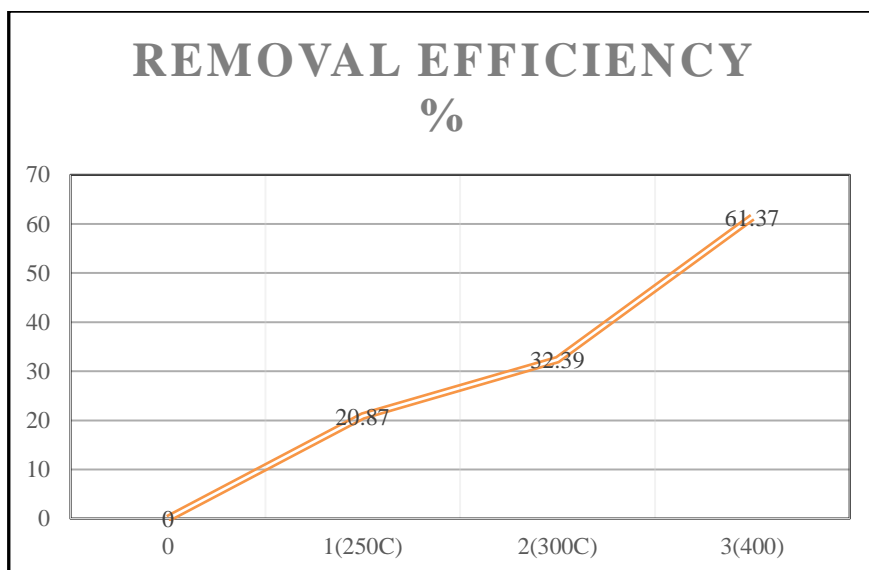


Figure 18: Effect of Biochar Temperature on COD value

For figure 18, the removal efficiency of COD level was steadily increased from 20.87% at biochar temperature of 250°C and 32.39% with temperature of 300°C. Then for the temperature of 400°C, the percentage of COD removal efficiency is 61.37%. From this figure, the percentage of removal efficiency on COD is increased. Although the standard of DOE is 250 mg/l is not achieved, the removal efficiency is more than 60% which means that biochar can reduce more than 60% of the chemical in the dye wastewater.

#### 4.3.2 Effect of temperature biochar on Biochemical Oxygen Demand

Biochemical oxygen demand (BOD) is a measure of the amount of oxygen required to break down organic matter in water. Based on the result of dye wastewater treatment, the BOD is higher due to the presence of high levels of organic compounds, such as dyes, which are released into the water during the dyeing process. These compounds can be difficult to break down, leading to an increased demand for oxygen to remove them. The high BOD levels in dye wastewater can also cause problems in treatment systems, as they can lead to decreased oxygen levels in the water, which can harm aquatic life and create anaerobic conditions that can support the growth of harmful

bacteria. To reduce the BOD levels in dye wastewater, various treatment processes can be used, such as oxidation, biodegradation, and chemical precipitation, which can help to break down the organic compounds and remove them from the water.

Biochemical oxygen demand (BOD) parameter is used as an identification of required oxygen quality for oxidation of biodegradable organic matter in dye wastewater. The BOD test is mainly based on determining and measuring the dissolved oxygen (DO) before and after a 5-day period of incubation for the sample at 20 °C to determine the amount of oxygen used biochemically. Therefore, the biochemical oxygen demand under this condition is sometimes referred to as BOD. Since bacteria and nutrients are present in all natural waterways, therefore, any introduced waste compound into such waterways will start biochemical reactions. Such biochemical reactions are because of what is known as the biochemical oxygen demand (BOD).

Table 7: Effect temperature of biochar on BOD value

<b>SAMPLE</b>	<b>INITIAL CONCENTRATION (MG/L)</b>	<b>FINAL CONCENTRATION (MG/L)</b>	<b>REMOVAL EFFICIENCY, E (%)</b>
<b>0</b>	36.10	36.10	0.00%
<b>1</b>	36.10	35.85	0.70%
<b>2</b>	36.10	34.65	4.04%
<b>3</b>	36.10	34.25	5.13%

Table 7 shows the results of BOD, it is observed for five days. BOD level for dye wastewater after interacting with the biochar adsorbent. The final BOD level is for the biochar temperature 250°C is 35.85 mg/L and for biochar temperature 400°C is 34.65 mg/l. For the BOD level in sample biochar 400°C is 34.25 mg/L. The standard BOD level for the industrial wastewater of dye wastewater is below 50 mg/L, which means it is an acceptable condition for discharge.

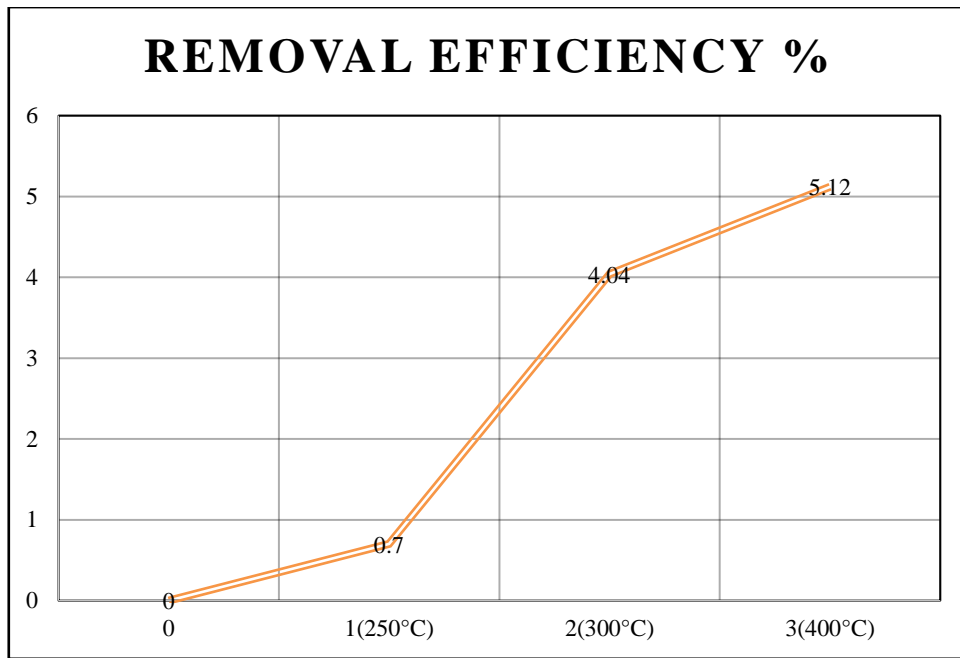


Figure 19: Effect temperature biochar on BOD value

Based on figure 19, BOD percentage of removal efficiency interacts with biochar with different temperatures. Removal efficiency of sample biochar 250°C is 0.7% and for removal efficiency of sample biochar 300°C is 4.04%. Finally, removal efficiency for sample 400°C is 5.12%. Based on the figure, the percentage of removal efficiency for biochar has increased. The removal efficiency percentage only increases slightly because dye wastewater or textile wastewater does not have much biochemical oxygen demand (BOD). Biochar produced from palm oil sludge may have lower removal efficiency for BOD due to its lower lignin content and lower surface area compared to other feedstocks, such as water hyacinth. Lignin is an important component of biochar that provides a source of carbon for the growth of microorganisms that can break down organic matter. The lower lignin content of palm oil sludge biochar may limit its ability to support the growth of these microorganisms and reduce its efficiency for BOD removal.

### 4.3.3 Effect of temperature biochar on TSS

Total suspended solids (TSS) refer to the number of solid particles that are suspended in a water sample and can be seen with the naked eye. In dye wastewater, the TSS levels are typically higher due to the presence of dye particles, fibers, and other solid materials that are released into the water during the dyeing process. These solid particles can cause problems in treatment systems and can lead to decreased water quality, as they can clog filters, interfere with the performance of treatment processes, and contribute to the formation of sludge. Additionally, the high TSS levels in dye wastewater can create favorable conditions for the growth of harmful bacteria and other microorganisms, which can cause further degradation of water quality.

TSS measurement was carried out by filtering the samples influent (raw wastewater) and effluent (the supernatant of the treated sample after settling) using 0.45  $\mu\text{m}$  filter papers.

Table 8: Effect of biochar temperature on TSS

<b>SAMPLE</b>	<b>INITIAL CONCENTRATION</b>	<b>FINAL CONCENTRATION</b>	<b>REMOVAL EFFICIENCY, E (%)</b>
<b>0</b>	405	405	0.00%
<b>1</b>	405	257	36.54%
<b>2</b>	405	231	42.96%
<b>3</b>	405	120	63.33%

Based on the table shown, we can conclude that the result is decreasing. From sample raw materials the total suspended solid is 405 and for the first sample filter using biochar 250C is 257. The result for sample 2 is 231 and for sample 3 is 120. This shows show, the biochar filter can reduce among of total suspended solids. The standard from DOE to discharge dye wastewater is 100. Although the result is not achieved the standard DOE, the percentage of removal efficiency is more than 63%.



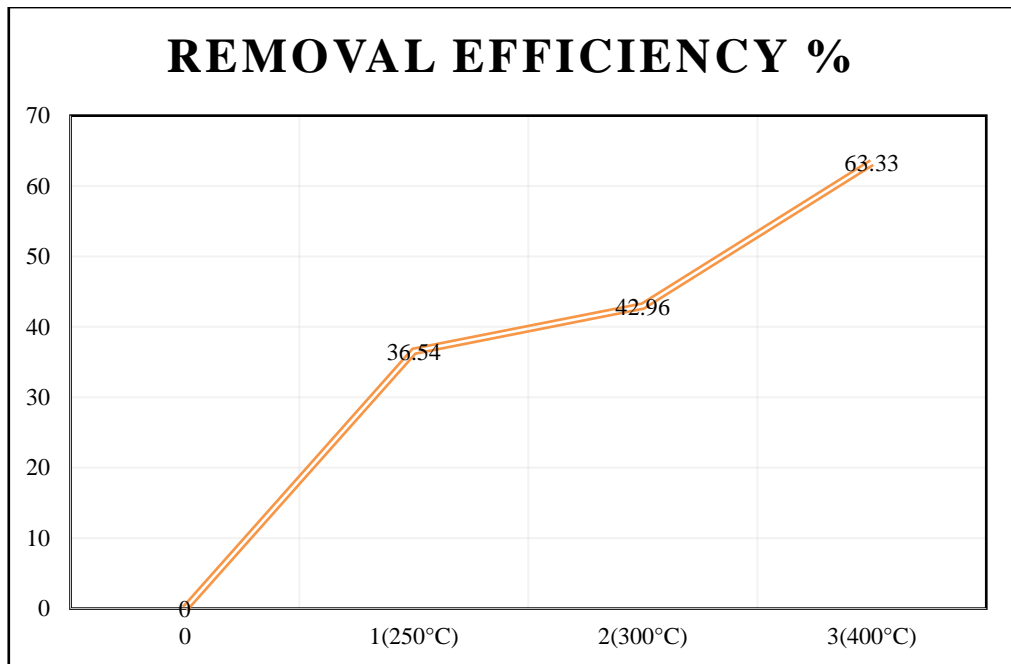


Figure 20: Effect of biochar temperature on TSS

The percentage of removal efficiency from this figure, the percentage of removal efficiency is increased. For the sample one is 36.54% and for the sample two is 42.98% and finally for the sample 3 is 63.3%. Which means this biochar has good interaction. The higher the temperature of biochar, the more increased the percentage of removal efficiency for total suspended solids.

#### 4.3.4 Effect of temperature of biochar on Color Admi

Based on result dye wastewater, the color admi is higher due to the presence of high levels of dyes, which are released into the water during the dyeing process. These dyes can give the water a distinctive color, making it difficult to achieve the desired water quality standards for discharge or reuse. The high color admittance levels in dye wastewater can also create aesthetic problems and can impact the local water environment, as the dyes can discolor the water, making it less appealing to the human eye and reducing its overall value.

Color (American Dye Manufacturers Institute (ADMI) unit, the scale used to measure the color removal of wastewater as an indicator of water quality). The ADMI of the sample was determined using a spectrophotometer.

Table 9: Effect of biochar temperature on Color ADMI

<b>SAMPLE</b>	<b>INITIAL CONCENTRATION</b>	<b>FINAL CONCENTRATION</b>	<b>REMOVAL EFFICIENCY, E (%)</b>
<b>0</b>	22200	22200	0.00%
<b>1</b>	22200	20100	9.50%
<b>2</b>	22200	11800	46.85%
<b>3</b>	22200	9600	56.75%

Based on the table show that result for dye wastewater before and after filter using biochar by different temperature. For raw dye wastewater is 22200. Sample 1 shows the difference between raw dye wastewater and after filter. The first sample result is 20100 and second sample is 11800 and for the last sample is 9600. Although the result did not achieve the color standard from DOE, the removal efficiency is quite effective. The result is not achieved the standard DOE because as we know the dye wastewater has high of color admi since the wastewater is from textile wastewater.

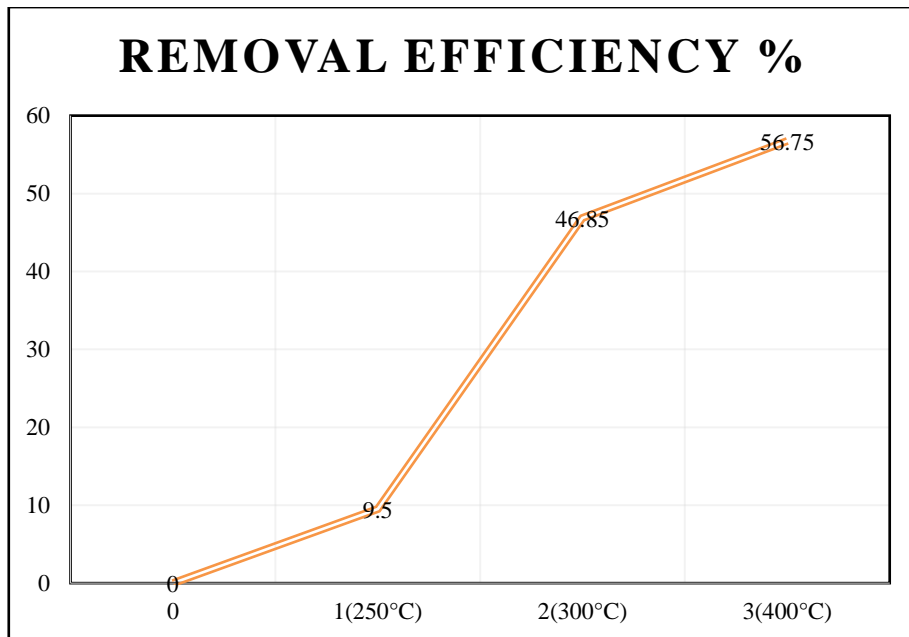


Figure 21: Effect of biochar temperature on Color ADMI

Based on the graph, we can conclude that the biochar is effective for color admi since the percentage of removal efficiency is more than 56%. For sample 1 the percentage of removal efficiency is 9.5% and sample 2 is 46.85% and lastly for sample 3 is increase to 56.75%. As we know the dye wastewater has high color admi since it is from textiles wastewater. The standard of DOE is not achievable, but the efficiency of biochar is more than 50%. This experiment only does for 50gram of biochar, so for my preference if put some more biochar can achieve the standard.

#### 4.4 Removal Efficiency Palm Oil Sludge and Water Hyacinth

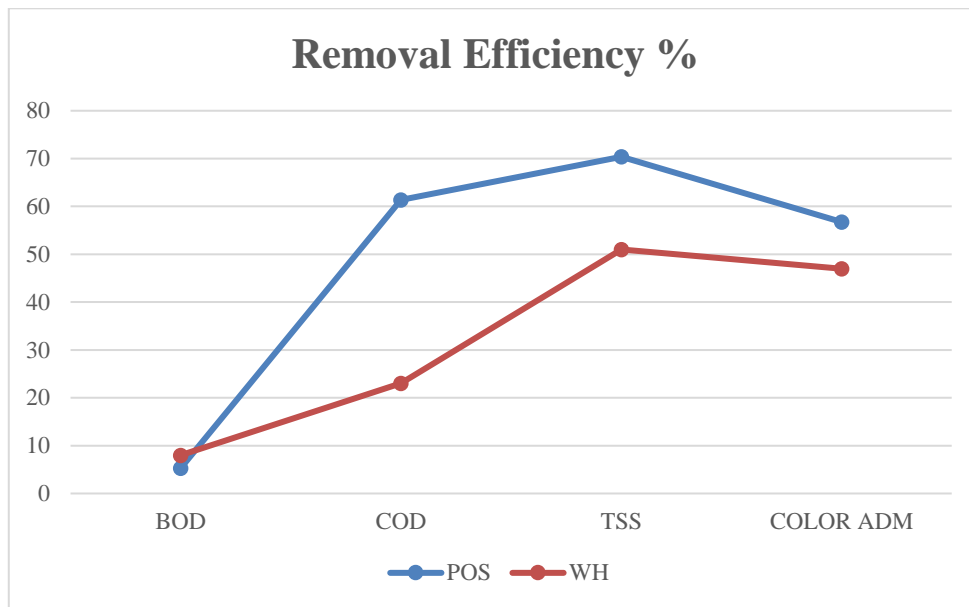


Figure 22: Removal Efficiency POS vs WH

The efficiency of biochar for wastewater treatment can vary depending on the feedstock used to produce it. Both palm oil sludge and water hyacinth can be used to produce biochar, but they may have different properties and efficiencies for wastewater treatment.

Palm oil sludge is a by-product of the palm oil industry and contains high levels of organic matter, making it an attractive feedstock for biochar production. Biochar produced from palm oil sludge tends to have high surface areas and high adsorption capacities for pollutants, making them more efficient for wastewater treatment.

Water hyacinth is an aquatic plant that grows in large quantities in many parts of the world and can be used as a feedstock for biochar production. Biochar's produced from water hyacinth tend to have lower surface areas and lower adsorption capacities for pollutants compared to biochar's produced from palm oil sludge. Additionally, water hyacinth biochar's may contain lower levels of volatile compounds and minerals, which can reduce their overall efficiency for wastewater treatment.

Biochar produced from water hyacinth has been shown to have high removal efficiency for BOD due to its high lignin content, which can provide a source of carbon for the growth of microorganisms that can break down organic matter. Biochar produced from palm oil sludge also has the potential to remove BOD from wastewater, but its efficiency may be lower compared to water hyacinth biochar due to its lower lignin content.

In summary, the efficiency of biochar for wastewater treatment depends on several factors, including the feedstock used, the temperature and processing conditions used to produce the biochar, and the specific contaminants present in the wastewater. While palm oil sludge biochar is generally considered to be more efficient than water hyacinth biochar for wastewater treatment, the optimal choice of feedstock will depend on the specific treatment needs and requirements.

## CHAPTER 5

### CONCLUSION

#### 5.1 Conclusion

The temperature at which biochar is produced through pyrolysis can have an impact on its efficiency for treatment of wastewater. This is because the temperature can affect the physical and chemical properties of the biochar, such as its surface area, pore structure, and chemical composition, which can impact its ability to adsorb pollutants from wastewater.

Based on the result, high temperatures can result in the production of biochar with high surface areas, which can increase their adsorption capacity for pollutants. On the other hand, high temperatures can also result in the loss of volatile compounds and minerals from the biochar, reducing its effectiveness as a wastewater treatment agent. Lower temperatures, on the other hand, can result in the preservation of volatile compounds and minerals, but may also produce biochar with lower surface areas, reducing their adsorption capacity.

The optimal temperature for biochar production will depend on the specific contaminants present in the wastewater and the desired properties of the biochar, such as its adsorption capacity, pore structure, and chemical composition. In general, temperatures in the range of 250-400°C are commonly used for biochar production, but the optimal temperature will depend on the specific feedstock, processing conditions, and end-use applications. It is important to consider the trade-offs between surface area, pore structure, and chemical composition when selecting the temperature for biochar production for wastewater treatment.

For this study, palm oil sludge is used as an adsorbent for biochar removal. Biochar is used in the filter to treat dye wastewater. Biochar can reduce some of the substances that have in dye wastewater such as biochemical oxygen demand BOD,

chemical oxygen demand COD, Total suspended solids, and colour adm. Based on the result, the temperature of biochar during the process of pyrolysis really affects the result of BOD, COD, TSS, and Colour Admi. Based on the result, the most effective sample biochar is sample 3 at 400°C. The removal efficiency of biochar sample 3 is higher compared to sample 1 and sample 2.

In a conclusion to this study, prove that biochar can remove some substance in dye wastewater such as BOD, COD, TSS, and Colour Admi. The removal efficiency for COD is 61.37%, BOD is 5.13%, TSS is 63.3%, and the last for Colour adm is 57%.

A large volume of palm oil sludge used as biochar may remove a larger number of pollutants. Palm oil sludge, which is a by-product of the palm oil production process, can have potential hazards if not properly handled. The sludge contains high levels of organic matter, heavy metals, and other pollutants that can be harmful to the environment and to human health if they are not treated or disposed of properly. It can be used as a good filter to preserve the environment. Lastly, biochar is basically a biomass product, and it can be gotten easily. If the experiment is expanded, it can be an affordable filter that can be used by others.

## **5.2 Recommendation**

Future research could further examine a difference in chemical reagents that are used to activate biochar. Instead of using phosphoric acid, use a different chemical to activate the biochar. This study can show if different chemical reagents can affect effectiveness of biochar.

Then, using a different type of wastewater, such as shrimp wastewater, domestic wastewater, sewage and others. This is to determine whether the biochar is good or not in treating the different types of wastewaters.

Only 50 grams of biochar were utilized in this study. In future research can use more biochar such as 100gram, 200gram or more. Increasing the weight of the biochar in the filter can improve the removal efficiency.

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## **APPENDICES**

## Appendix A: Preparation of Activate Carbon for Biochar



## Appendix B: Preparation of Experiment

