MODIFIED BIOCHAR FROM WASTE BIOMASS (WATER HYACINTH) TO TREAT TEXTILE DYE WASTEWATER

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ABSTRAK

Industri tekstil merupakan salah satu industri berkembang dengan pesat dan juga merupakan salah satu penyumbang kepada pertumbuhan ekonomi di Malaysia. Pewarna batik atau kain yang dilepaskan kea lam sekitar merupakan salah satu punca utama penyebabnya pencemaran air. Sistem rawatan individu sama ada dalam jenis atau bentuk fizikal, biological mahupun kemikal sering di jual pada harga yang tinggi dan menghasilkan keladak. Dalam pada masa yang sama, Keladi Bunting atau nama saintifiknya *Eichhornia crassipes* merupakan tumbuhan invasive atau tumbuhan yang bersifat ceroboh. Tumbuhan ini selalunya akan tumbuh dengan banyak dan akan menghalang sinaran matahari dari tembus ke dalam air, sehingga kadar oksigen berkurangan, menyebabkan kualiti air menurun dan akhirnya menjejaskan ekosistem di dalam kawasan perairan tersebut. Penggunaan keladi bunting dipilih untuk dijadikan sebagai 'activated carbon'. Keladi bunting tersebut akan dikumpul, dikeringkan dan akan melalui proses pirolisis dan dibahagikan mengikut tiga suhu yang berbeza, iaitu 250°C, 300°C dan 400°C. kemudian setiap sampel tersebut akan direndam dalam asid fosforik menggunakan ratio 2:1 untuk pengaktifan kimia. Data akan dikumpul dan direkod. Parameter yang diambil adalah *Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Total Suspended Solids (TSS)* dan *Color ADMI*. Hasilnya, keputusan menunjukkan bahawa '*activated carbon*' dengan suhu 400°C mempunyai peratusan penyingkiran yang tinggi berbanding dengan sampel yang lain, dengan 23% dalam COD, 51% dalam TSS dan 47% untuk colour ADMI

ABSTRACT

One of the industries with the quickest growth rate and a major contribution to Malaysia's economic expansion is the textile industry. One of the main causes of water pollution is the discharge of dyes into the environment during the dyeing and finishing procedures for textile fibres. Individual wastewater treatment, whether it be physical, biological, or chemical, is frequently quite expensive and produces a lot of sludge. In the meanwhile, water hyacinth (WH) or Eichhornia crassipes is an invasive plant that will block the water surface from letting the sunlight goes through the water, causing oxygen levels to reduced, water quality to deteriorate and ecosystem lifeforms to suffer. To filter the wastewater, a substance for the filter is needed. Thus, the use of water hyacinth as activated carbon was suggested. The water hyacinth was collected, dried undergoes pyrolysis in three different temperatures, which is 250°C, 300°C and 400°C. Then, the WH were impregnated in phosphoric acid with 2:1 ratio for chemical activation. The results were then taken and recorded. The parameter taken were Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Total Suspended Solids (TSS) and Colour ADMI. Results shown that activated carbon with 400°C has higher removal efficiency with 23% in COD, 51% in TSS and 47% for colour ADMI.

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LIST OF ABBREVIATIONS

WH	Water Hyacinth
TGA	Thermogravimetric Analysis
DTG	Derivative Thermogravimetry
BOD	Biochemical Oxygen Demand
COD	Chemical Oxygen Demand
TSS	Total Suspended Solids

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CHAPTER 1

INTRODUCTION

1.1 Background Study

Water hyacinth or *Eichhornia crassipes* is an invasive floating plant that can be found in bodies of water all over the world. The plant can be recognised by its blue-purple or lilac coloured with a yellow spot flower. The leaf stalk is thick and spongy and helps to keep the plant buoyant with a mass of fine roots hang in the water underneath the plant (Hiralal Jana, 2015). It reproduces swiftly and jams slow-moving streams frequently. Invasive alien species are considered the second most serious danger to biodiversity after habitat loss. Phenotype plasticity, or the ability to change growth shape to suit current conditions, is a unique feature that allows these invasive alien species to effectively overrun a specific area (Dewitt T. J, 1994). Species were transported to other parts of the world, including Malaysia and Indonesia, as a result of growing global trade and cross-border activity. These countries provide habitats that allow living organisms to thrive in a humid and warm tropical environment. Some species colonise and adapt well to their surroundings, while others become invasive. In Malaysia, aquatic plant species considered as noxious weeds are water hyacinth (*Eichhorniacrasspies*), Lemna and hydrilla (*Hydrilla verticillata*) (Mashhor M, 1994).

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 adaption. 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 colonised 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 m3 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. The majority of invasive aquatic plants (146 species, or 96.05%) thrive in freshwater. *Eichhorniacrasspies*, hydrilla (*Hydrilla verticillata*), alligatorweed (*Alternanthera philoxeroides*), Eurasian watermilfoil (*Myriophyllum 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 a number of 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.

Water hyacinth deprives phytoplankton of nutrients by absorbing and utilizing them. Phytoplankton, zooplankton, and fish stocks are all affected. Under the floating water hyacinth mats, however, when the huge volumes of organic material produced by senescent water hyacinth decay, oxygen deprivation and anaerobic conditions result. These anaerobic circumstances have been a direct cause of fish death and alterations in the fish community, with most species being eliminated at the expense of air-breathing species. Water hyacinth mats that remain stationary shade out bottom-growing flora, depriving some fish species of food and reproductive habitats (Ayanda, 2020). The impact on fish diversity might be substantial. The conditions provided by water hyacinth stimulate the vectors of various human diseases, as well as the majority of mosquito vectors, including those that transmit malaria.

However, there is also advantage of water hyacinth. The green parts and inflorescence can be used as vegetable. It also can be used for compost and vermicompost making (Ali ElNaggar, 2019). Water hyacinth also can be used as an alternative source of biomass and also in charcoal briquetting. Thanks to its roots, the water hyacinth can act as phyto-accumulators, whereby the roots absorb toxic pollutants and chemicals at higher concentrations in water bodies (Mukesh Dwivedil). It has the ability to absorb a lot of nitrogen and phosphorus, as well as other nutrients and minerals. The ability of water hyacinth to suck up heavy metals has led to the notion that it could be used to clean industrial effluent from water.

Bio-char is a by-product charcoal made from pyrolysis, a biomass thermochemical process with a temperature about 700°C in the absence or limited supply of oxygen. It can be used as a soil amendment to increase soil health, store carbon in soil, and improve soil qualities by burying it in fields, among other things (Mohammed, 2011). Furthermore, bio-char reduces carbon emissions into the atmosphere by stabilizing carbon in a form similar to charcoal. Furthermore, bioenergy created by the pyrolysis process might be used to replace fossil fuels. Biochar from water hyacinth has been used in several application. Water hyacinth biochar has been reported to remove heavy metal and to function in soil amendment.

1.2 Problem Statement

Water hyacinth (*Eichhorniacrasspies*) is considered as invasive species. Invasive alien species are considered the second most serious danger to biodiversity after habitat loss. Phenotype plasticity, or the ability to change growth shape to suit current conditions, is a unique feature that allows these invasive alien species to effectively overrun a specific area (Dewitt T. J). Species were transported to other parts of the world, including Malaysia and Indonesia, as a result of growing global trade and cross-border activity. These countries provide habitats that allow living organisms to thrive in a humid and warm tropical environment. Some species colonise and adapt well to their surroundings, while others become invasive. In Malaysia, aquatic plant species considered as noxious weeds are water hyacinth (*Eichhorniacrasspies*), Lemna and hydrilla (*Hydrilla verticillata*) (Mashhor M).

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 adaption. 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 colonised 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 m3 of water per hectare, leading reservoir water levels to fall more quickly during dry periods (Wittenberg R).

Water hyacinth has a direct impact on the chemistry of the water. It has the ability to absorb a lot of nitrogen and phosphorus, as well as other nutrients and minerals. The ability of water hyacinth to suck up heavy metals has led to the notion that it could be used to clean industrial effluent from water. Water hyacinth deprives phytoplankton of nutrients by absorbing and utilising them. Phytoplankton, zooplankton, and fish stocks are all affected. Under the floating water hyacinth mats, however, when the huge volumes of organic material produced by senescent water hyacinth decay, oxygen deprivation and anaerobic conditions result (Hiralal Jana, 2015). These anaerobic circumstances have been a direct cause of fish death and alterations in the fish community, with most species being eliminated at the expense of air-breathing species. Water hyacinth mats that remain stationary shade out bottom-growing flora, depriving some fish species of food and reproductive habitats. The impact on fish diversity might be substantial. The conditions provided by water hyacinth stimulate the vectors of various human diseases, as well as the majority of mosquito vectors, including those that transmit malaria (Ayanda, 2020).

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 filters out there, there are so many types of filters used in treating waste water. Most of the filters are usually very expensive because of its complexity and usually have more downtime to fix clogged filter. This is because there are many layers in the filters. Those layers included is gravel, fine sand, silica sand and etc.

That's how we come up to control the growth of water hyacinth and take advantage of its ability to absorb the heavy metals, nitrogen, etc and turn it to biochar. By using slow pyrolysis to produce biochar and apply it to filter out the suspended solids and COD level from the sample of wastewater. Therefore, there will be need of understanding on how to synthesis the biochar from the water hyacinth and to design the filter system so that it is able to filter out the parameters stated.

1.3 Objective

The objective of this research study is:

1. To synthesis activated carbon from water hyacinth (WH)
2. To design a filtration system prototype by using recyclable materials and using activated carbon from water hyacinth
3. To treat chemical oxygen demand (COD), biochemical oxygen demand (BOD), total suspended solids (TSS) and colour of dye wastewater

1.4 Scope of Study

The scope of study is focused on the production of biochar from palm oil sludge (POS) and water hyacinth through pyrolysis and to biochar. The water hyacinth will be collected, crushed and heated from 100°C – 110°C 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 undergoes a process called fast pyrolysis by taken three temperatures within the temperature acquired from TGA analysis for one hour. Then, the biochar produced is then activated through a process called activation, which the biochar is impregnated with phosphoric acid. Next, the filter is design and fabricate 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 identify the parameter, that is COD level, total suspended solids, BOD level and the colour of dye wastewater.

1.5 Significant of Study

Biochar is a carbon-rich substance created by pyrolysis of organic waste. The amount of biochar produced is determined on the type of feedstock used and the pyrolysis conditions. Biochar has been proposed as a potential alternative for improving energy access and waste management. It's a useful system that can be put to a lot of different uses. Most of the biochar are usually used in agriculture. Through repeated crop harvesting, modern agriculture is contributing to nutrient mining and a decline in soil organic matter levels. The use of soil amendments in the form of fertilisers containing the three key nutrients is the most widely used solution to this deficit. The most limiting ingredient for plant growth is nitrogen, which is required for protein synthesis, structure, hormones, chlorophyll, vitamins, and enzymes. Biochar is a carbon-sequestering material that can be added to soils to improve soil health, fertility, and carbon sequestration.

CHAPTER 2

LITERATURE REVIEW

An overview of a subject called a "literature review" comprises relevant information, methodologies, and hypotheses based on prior study. This chapter will examine recent research on the general topic of activated carbon in dye wastewater treatment.

2.1 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 adaption. 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).

2.2 Biochar Production

The primary component of solid biomass, organic waste, has a significant potential for producing biochar. Crop residues from forestry, agriculture, and municipal solid trash, as well as food waste and animal manures, are examples of biomass waste sources that can be used to make biochar (Ali El-Naggar, 2019). The carbon content of biochar produced from biomass via thermal combustion in an oxygen-limited atmosphere is quite high (Sanjay K. Mothany, 2018). Biochar is unique in that it has a wide surface area, a high porosity, functional groups, a high cation exchange capacity, and stability, all of which make it useful for a variety of uses. A few benefits of biochar are its quick and simple preparation, eco-friendliness, reusability, and affordability. (R. Gayathri,

2021)

The process parameters are primarily responsible for determining biomass yield. Temperature, biomass type, residence time, heating rate, pressure, and other parameters are among those considered. Temperature is the most important factor influencing biochar properties (M. Ahmad, 2014). Pyrolysis, hydrothermal carbonization, gasification, flash carbonization, and torrefaction are common thermochemical techniques used for biochar production. Pyrolysis is the most commonly used method for producing biochar. In an oxygen-limited environment, the organic compounds in biomass decompose at a specific temperature. Process temperature, residence time, biomass type, and heating rate are all factors that influence the pyrolysis product (J. S. Cha, 2016).

2.2.1 Pyrolysis

. Pyrolysis is the thermal decomposition of organic materials in an oxygen-free environment at temperatures ranging from 250°C – 900°C (J.I. Osayi, 2014). This method is an alternative method for converting waste biomass into value-added products such as biochar, syngas, and biooil. At specific temperatures, lignocellulosic components such as cellulose, hemicellulose, and lignin undergo reaction processes such as depolymerization, fragmentation, and cross-linking, resulting in different states of products such as solid, liquid, and gas. Char and bio-oil are solid and liquid products, while carbon dioxide, carbon monoxide, hydrogen, and syngas are gaseous products (C1-C2 hydrocarbons). Various types of reactors such as paddle kiln, bubbling fluidized bed, wagon reactors and agitated sand rotating kilns are used for biochar production. The biochar yield during the pyrolysis process depends on the type and nature of biomass used. Temperature is the main operating process condition that decides the product efficiency (J. Wei, 2019).

Pyrolysis can be classified as a fast and slow pyrolysis process, depending on the heating rate, temperature, residence time and pressure. Carbonization is another term for slow pyrolysis. It does not condense the pyrolysis products and operates at a slow rate, with low heating rates and a longer residence time. During the slow pyrolysis process, the biomass is heated and decomposed at a moderate temperature of 350-500 °C, over an adequate residence time (Dhyani and Bhaskar, 2018). Slow pyrolysis biochar has a high carbon content, which is an important criterion for determining whether biochar is of high quality or not. Some pyrolysis parameters, such as higher pyrolysis temperature, lower heating rate, longer residence time, presence of the catalyst, particle size, and the pyrolysis atmosphere, must be considered when producing high carbon content biochar. Furthermore, the biomass feedstock is an important factor in determining the quality of the biochar (Wang. L, 2020).

Because the reaction time in the process is so short, it is also known as a continuous process. Because of the high heat used in this process, biomass particles decompose instantly, producing biochar and pyrolysis vapour. The pyrolysis vapour is then processed further. The feedstock can be heated to produce desired products by setting optimal parameters such as high heating rates (1000 °C/min), pyrolysis temperature (500 °C), and short residence time (Wang. L, 2020). These are the factors that play an important role in determining the chemical constituents of the product obtained during the reaction:

1) Mass and heat transfer

2) Chemical kinetics

Table 1: Thermochemical conversion technique and their process condition

Technique	Temperature (C)	Residence time	Yield of biochar (%)	Yield of bio-oil (%)	Syngas production (%)	References
Pyrolysis	300-700 (slow) 500-1000 (fast)	< 2 s (slow) Hour-day (fast)	35 (slow) 12 (fast)	30 (slow) 75 (fast)	35 (slow) 13 (fast)	K.B.Cantrell et al
Hydrothermal carbonization	180-300	1-16 h	5080	5-20	2-5	A Funke et al
Gasification	750-900	10-20 s	10	5	85	Castaldi et al
Torrefaction	290	10-60 min	80	0	20	W.R Zwart et al
Flash carbonization	300-600	< 30 min	37	–	–	S.R Wade et al

2.2.2 Biochar production

To determine whether biochar can remove pollutants or be put to other uses, biochar is characterised. The structural and elemental analysis also helps to foretell how biochar may affect the environment. Additionally, metals and biochar interact in ways that are influenced by pH, such as:

1. Biochar function changes with pH

2. Metal contaminant ion speciation changes with pH

These characteristics of biochar showed that it can function as a very efficient adsorbent to get rid of the bulk of soil pollutants. Biochar is characterised by its structure, surface functional groups, and elemental analysis (Brewer, 2014).

2.2.3 Thermo-gravimetric Analysis (TGA)

TGA is a type of thermal analysis that assesses the material's physical and chemical characteristics in relation to temperature. For a very long time, thermogravimetric analysis (TGA) has been used to demonstrate and assess the heated behaviour of diverse materials. The aim of this study was to investigate the igniting characteristics of biochar and biomass/biochar blends using thermogravimetric analysis. In TGA, a sample is subjected to a controlled temperature program in a high-precision balance while measuring its weight changes. The data collected can be used to determine the onset of degradation, the amount of volatile components, and the residual weight after heating. The results of TGA are often presented as a thermogravimetric curve, which shows the relationship between the weight change and the temperature or time.

Additionally, the normal weighted average of each component was looked at to determine whether there was any synergy between the blend's component elements. The results could contribute to a better understanding of the warm technique, case characteristics, and testing of tests in general (Yao. Y, 2014). During this process, the temperature of the biochar is heated from room temperature to 1000 °C.

2.3 Pre – treatment of Biochar

Depending on their physical and chemical characteristics, biochar can be pre – treated in a variety of ways. These characteristics aid in deciding where biochar should be used, such as for the elimination of contaminants (organic pollutants, heavy metals, dyes, etc.). The quality of raw biochar is improved through biochar alteration to boost its effectiveness in eliminating contaminants at higher concentrations.

2.1.1 Chemical Activation

Chemical activation of biochar is described as simultaneous carbonization of biomass and activation by chemical agent (chemical activation). This activation is typically inexpensive and completed in a shorter amount of time at a lower temperature (Rangabhashiyam and Balasubramanian, 2019). Chemical activation involves adding chemicals to biochar, such as agents, bases, and oxidants (Enaime. G, 2020). Chemical activation enhances the carbon in biochar, decreases mineral matter, improves the functional groups (cation and anion exchange characteristics) on the surface of the biochar, and increases microporosity.

Chemical activation of biochar can be achieved by employing activating agents such as phosphoric acid, potassium hydroxide, or zinc chloride. During the process of chemical activation, biochar is subjected to activating agents, treated with heat between 300 °C and 500 °C, and then the activating agent is removed by means of washing with an acid or base and water. The washing step of this process leads to the increase in surface area and highly porous pore structure. (AlcañizMonge & Illán-Gómez, 2008)

CHAPTER 3

METHODOLOGY

3.1 Introduction of Methodology

Methodology is the study of research methodologies, or more officially, a contextual framework for research, a coherent and logical scheme based on perspectives, attitudes, and values that directs researchers' [or other users'] decisions. It entails a theoretical examination of a branch of knowledge's corpus of procedures and principles, with methodologies from different disciplines differing depending on their historical evolution.

This chapter will explain and justify the production process, as well as demonstrate the planning and equipment used to produce a high-quality result for the specific study topic. To achieve an acceptable result, there are three major components:

- Planning
- Implementation
- Performance analysis

These three components are important as it explain the overall process of the production of activated carbon from water hyacinth. The first component is describing on how the research for the activated carbon and the filter is made. Also, the preparation and selection of the materials and products needed to conduct and build the activated carbon and the filter respectively.

After the planning is made, then comes the second component, that is implementation. After the activated carbon is produced and characterized, the activated carbon is put in the filter stacked with fine sand, silica sand, coarse silica sand. The activated carbon produced is originally biochar from water hyacinth (WH). The WH is collected, dried and undergoes pyrolysis process through fast pyrolysis at 250°C - 400°C then sieved into 1 – 2 mm. After that, the biochar under goes chemical activation, where the sieved biochar is impregnated with phosphoric acid, thus the production of activated carbon. Then the test is conducted to filter out the contaminant in the wastewater taken from waterbodies in domestic areas located in Paloh Hinai, Pekan, Pahang.

The performance analysis, which is the last components in methodology is made. Before the test is conducted, the data of each parameter is taken and compared with final data, which is the data taken after the test is conducted. Then, both of the data is compared and the analysis is conducted. There should be reduction of contaminant in each of the parameter after the test.

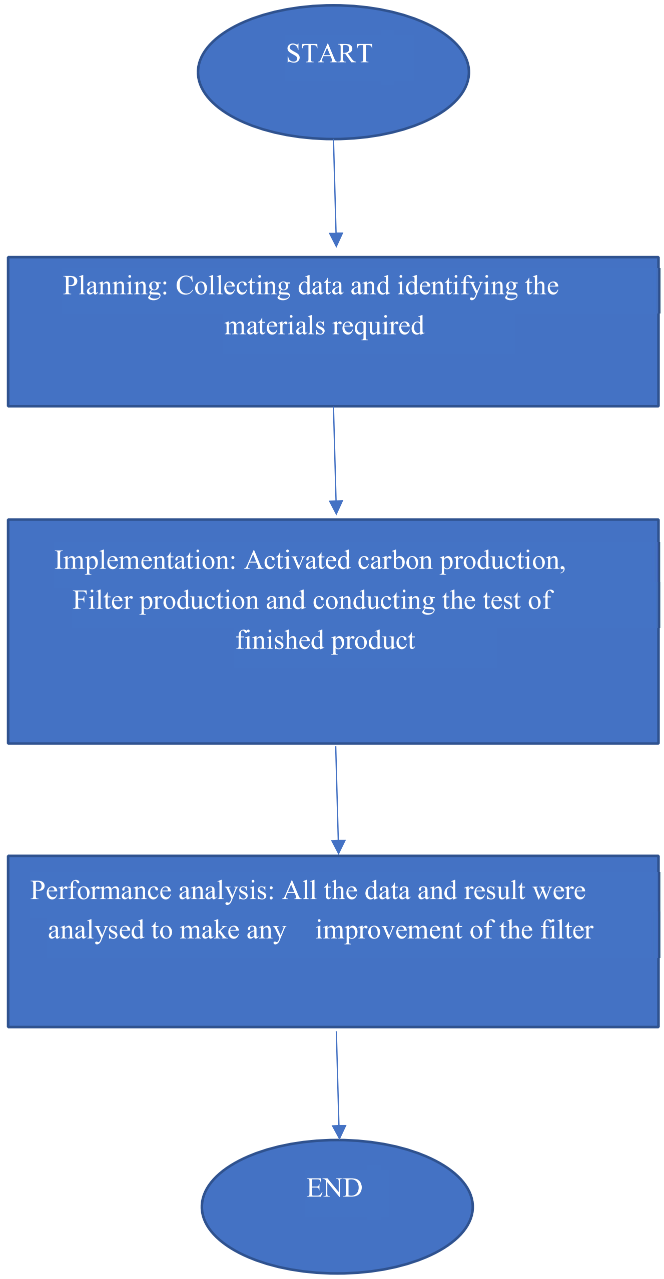

Figure 1: General flowchart of the research

3.2 Project Flowchart

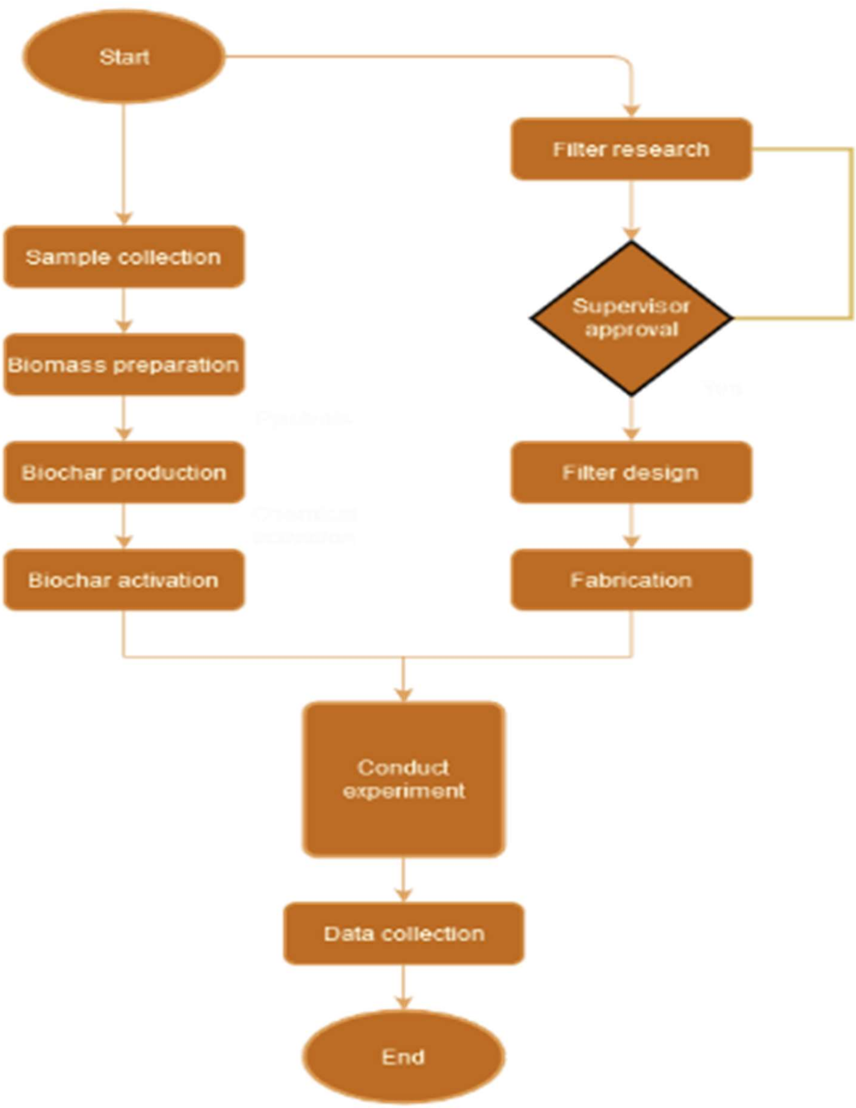

Figure 2: Overall flowchart of the research

3.3 Biochar from Water Hyacinth

3.3.1 Material

The material used in the filtration system is biochar. The biochar will be used from water hyacinth that will be collected from swamps located around Paloh Hinai, Gambang. The collected water hyacinth will be cleaned to get rid of dust and algae.

3.4 Preparation of Water Hyacinth (WH) Biochar

The biochar that will be used in this study will be prepared from WH. Initially, the collected WH will be cleaned from any contaminant from the swamps. Then, the collected WH will be dried in an oven at 110°C for five hours to remove the moisture content. Then, it will be let to cool at room temperature. Next, the biochar will be placed in the crucible and put in the furnace and undergoes pyrolysis at three different temperatures range from 250°C – 400°C for 1 hour. After cooling, the biochar will be kept in sealed bottles for further use.

3.5 Characterization of Biochar

The suitable temperature for pyrolysis process will be determined by using thermogravimetric analysis (TGA).

3.5.1 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) measures weight changes in a material as a function of temperature (or time) under a controlled atmosphere. Its principle uses include measurement of a material's thermal stability, filler content in polymers, moisture and solvent content, and the percent composition of components in a compound. In a TGA analysis, a sample is heated incrementally in a furnace while its weight is recorded on an analytical balance that is kept outside the furnace. If a heat event results in the loss of a volatile component, mass loss is detected in TGA. Mass losses occur during chemical processes like burning but not during physical processes like melting. To depict thermal transitions in the material, such as the loss of solvent and plasticizers in polymers, water of hydration in inorganic materials, and finally material disintegration, the weight of the sample is plotted versus temperature or time.

3.6 Activated Carbon Activation

As opposed to biochar that has been activated, raw biochar has less of a tendency to absorb or adsorb. This is noteworthy in terms of the substance's surface area, pore size, pore volume, and total number of pores. Without the proper activation treatments, the resulting biochar has many intermolecular spaces as a result of the breakdown of organic component bonds, clogged pores that cause tar generation, insignificant pore size that results in less surface area distribution, and contaminants like condensates and ashes that reduce pore size and volume (Hagemann N, 2018). Therefore, activation is necessary to enhance the sorption capacity and eliminate the pollutants, and it can be accomplished through physical or chemical treatments.

3.6.1 Chemical Activation

Activating biochar with phosphoric acid increases its surface area and porous structure, which can increase its capacity to absorb contaminants and other impurities. One mole of phosphoric acid, which is frequently employed as a biochar activator due to its inexpensive cost and high reactivity, was used in this particular experiment. The biochar samples were impregnated in phosphoric acid in a 2:1 ratio, which means that there was 1 part of biochar for every 2 parts of phosphoric acid. For the next two hours, this mixture was vigorously agitated, allowing the phosphoric acid to permeate and interact with the biochar.

Because it helps the phosphoric acid disperse evenly throughout the biochar and makes it possible for the acid to reach every area of the biochar, including its internal structure, stirring is crucial to this process. The phosphoric acid and biochar react to produce new functional groups and increase the surface area of the biochar, which increases its capacity to absorb pollutants and other contaminants. The activation process can also improve the stability of the biochar, which is important for its long-term use in environmental applications. The impregnated biochar can then be dried and used as a low-cost and effective adsorbent for a range of pollutants, including heavy metals, organic compounds, and other impurities.

3.7 Filter Design

According to the filter design schematic, only cotton and activated carbon biochar are used to form the filter's material. The water filter will let the dye wastewater through the filter.

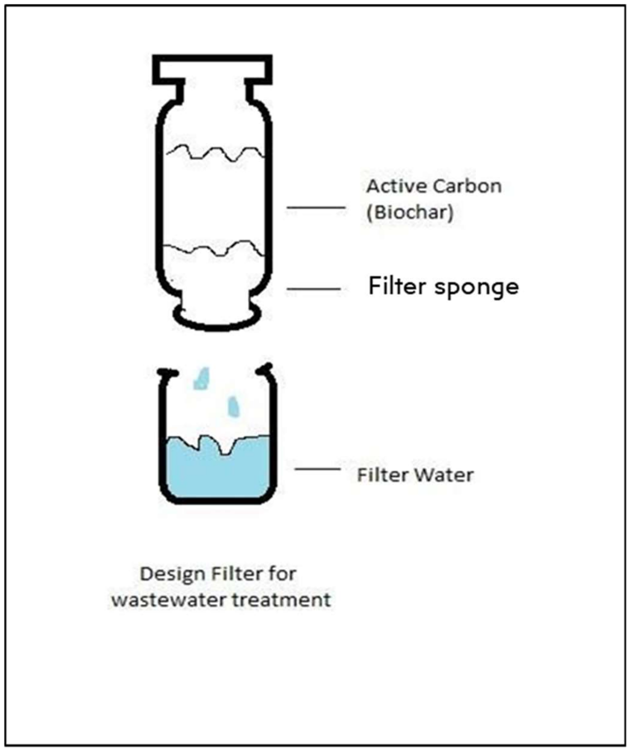

Figure 3: Design of the filter system

3.8 Parameter Analysis

Biochemical oxygen demand (BOD), chemical oxygen demand (COD), total suspended solids (TSS), and colour ADMI are the variables utilized in the test to determine the efficacy of activated carbon generated from water hyacinth in cleaning dye wastewater. The degree of the organic pollution, the quantity of solid particles in the wastewater, and the intensity of the wastewater's colour are all indicated by these metrics, in that order. Researchers can evaluate the success of the treatment and the potential effects of the treated wastewater on the receiving water bodies by assessing these factors.

3.8.1 Chemical Oxygen Demand (COD) Test

COD quantifies the amount of DO that is consumed under regulated circumstances during the oxidation of organic material and inorganic substances like ammonia or nitrite. In order to test for COD, the water sample is normally digested for two hours at 150°C with potassium dichromate and sulfuric acid in a sealed vial. Results are read from vials in a spectrophotometer.

The following steps are used to determine COD in dye textile wastewater utilizing water hyacinth-derived activated carbon:

1. Sample Collection: Take a sample of the wastewater from the facility that treats wastewater from dyed textiles.
2. Pre – treatment: Filter the sample to get rid of any contaminants and suspended solids.
3. Adsorption: Add water hyacinth-derived activated carbon to the filtered sample and carefully combine. The sample's organic contaminants are adsorbed by the activated carbon.
4. Filtration or centrifugation should be used to separate the sample from the activated carbon.
5. COD Determination: Mix the filtrate well after adding the COD reagents. The COD reagents use oxygen while oxidizing the organic contaminants in the sample. The sample's COD value determines how much oxygen is consumed.
6. Measure the amount of oxygen the COD reagents use and use a standard formula to determine the COD value.
7. Results: To assess the efficacy of the activated carbon from water hyacinth in eliminating the organic pollutants from the dye textile wastewater, compare the COD value of the sample before and after adsorption.

After the data is taken from the test, the COD value were calculated using formula as follow:

$$COD\ (mg/L) = (V1 - V2) \times (N / 1000)$$

Where:

V1 = volume of oxygen consumed by the COD reagents in the blank solution (mL)

V2 = volume of oxygen consumed by the COD reagents in the sample solution (mL)

N = normality of the COD reagents (mg of oxygen per liter of solution)

3.8.2 Biochemical Oxygen Demand (BOD) Test

The Biochemical Oxygen Demand (BOD) test calculates how much oxygen microorganisms need to break down organic contaminants in wastewater. The BOD test estimates the amount of biodegradable organic matter in wastewater and serves as a gauge for the wastewater's pollutant load.

The steps to perform a BOD test are as follows:

1. Take a representative sample of the wastewater that will be examined.
2. Dilution: Adjust the sample's concentration by diluting it with deionized water. A common dilution ratio is between 1:2 and 1:10.
3. Fill two BOD bottles with the diluted sample, then add an oxygen source (such magnesium sulfate) to each bottle for incubation. To make sure that the bacteria in the sample are actively growing, put a tiny amount of seed inoculum from a wastewater treatment facility into one of the bottles. For five days, in an incubator that is dark and warm (20–22°C), seal both bottles.
4. Initial Oxygen Concentration Determination: Using a dissolved oxygen meter, determine the dissolved oxygen (DO) levels in each bottle. As the initial oxygen concentration, note the DO concentration of the inoculated container (BOD1).
5. Calculating the Final Oxygen Concentration: Measure the DO concentration in both bottles once more after five days. As the ultimate oxygen concentration, note the infected bottle's DO concentration (BOD2).
6. BOD calculation: The following formula is used to determine BOD: (BOD1 - BOD2) x = BOD (mg/L) (Dilution Factor).
7. Results: To assess if wastewater is appropriate for disposal into the environment, compare the sample's BOD value to accepted BOD standards.

The BOD test is a labour and time-intensive method that calls for meticulous sample handling and strict adherence to incubation conditions in order to produce reliable findings.

3.8.3 Colour ADMI

The "American Dye Manufacturers Institute" (ADMI) is the source of the ADMI colour system, which, like the Pt/Co colour system, is based on the hue of hexa-chloroplatinate solutions. The ADMI value is calculated by measuring the transmittances (transmittances) in the wavelength spectrum between 400 and 700 nm. This method can also be used to calculate ADMI values for hues outside the yellow-orange range of the Pt/Co scale because the final step in the calculation is dependent on the sample's E value in relation to water. The procedure is similar to that in the APHA 2120 F standard. The procedure for colour ADMI procedure:

1. To achieve an accurate measurement of colour, prepare the surface to be measured by cleaning it if necessary. As differences in illumination can alter the colour measurement, ensure that the lighting conditions are constant and under control.
2. Prepare the sample in a way that is appropriate for measuring colour if necessary. Apply the sample to a substrate that is suitable for the kind of colour measurement being done, for instance, or cut it to a specified size or shape.
3. Calibration: Use a reference standard or a set of standards to calibrate the colour measuring device. Making sure the device is measuring colour accurately requires this step.
4. Measurement: Insert the sample into the device and measure the colour. Depending on the instrument being used, this may entail shining light on the sample and measuring the light that is reflected, transmitted, or absorbed.
5. Data analysis: Keep track of the colour measurement data and examine it to ascertain the sample's colour. This could entail comparing the measurement data to a set of reference standards, computing colour characteristics using software, or evaluating the colour using visual aids.

3.8.4 Total Suspended Solids (TSS)

Small solid particles that are suspended in water as a colloid or because the water is moving are referred to as suspended solids. If the suspended solids are quite large or dense, they can be removed either by filtering or sedimentation. It serves as a gauge for the strength of sewage or wastewater in general as well as the quality of the water. It serves as a key design factor for sewage treatment procedures. Sedimentation and/or water filters are typically used to remove suspended particles from water (usually at a municipal level). The large amount of water is typically made near to drinkable quality by removing the majority of the suspended particulates in a water source. After that, any pathogens that may be free floating or connected with the negligible quantity of suspended solids are disinfected to guarantee that they are made inert. Below is the procedure for TSS test:

1. Water is sampled by being placed in a pristine, sterile container. The sample must be true to the water body being examined.
2. Filtration: To remove the suspended particulates, the water sample is filtered through filter paper that has been pre-weighed. After that, the filter paper is dried in an oven for two hours at 103 2°C in order to eliminate the water and preserve the suspended solids.
3. Weighing: To calculate the weight of the suspended materials, dry filter paper is weighed. It is important to note the filter paper's weight both before and after drying.
4. Calculation: The weight of the dry filter paper is subtracted from the combined weight of the filter paper and the suspended solids to determine the weight of the suspended solids. The weight of the suspended particles is then divided by the volume of the water sample to determine the TSS concentration.
5. Reporting: Milligrams per litre (mg/L) or other suitable units of measurement should be used to express the results of the TSS analysis. The TSS findings are used to evaluate the water's quality and spot any pollutants that might be dangerous to the environment or people's health.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

A result refers to an outcome or consequence of an action, event, or process. It can be the outcome of a calculation, experiment, competition, or survey, among other things. Results can be expressed in various forms, such as numbers, statistics, or observations, and they can be used to make decisions, draw conclusions, or compare different scenarios. This chapter will explain the results obtained from the experiment.

4.2 Thermogravimetric Analysis (TGA)

TGA analysis data were taken and recorded. The data taken were used to determine the temperature range in pyrolysis process.

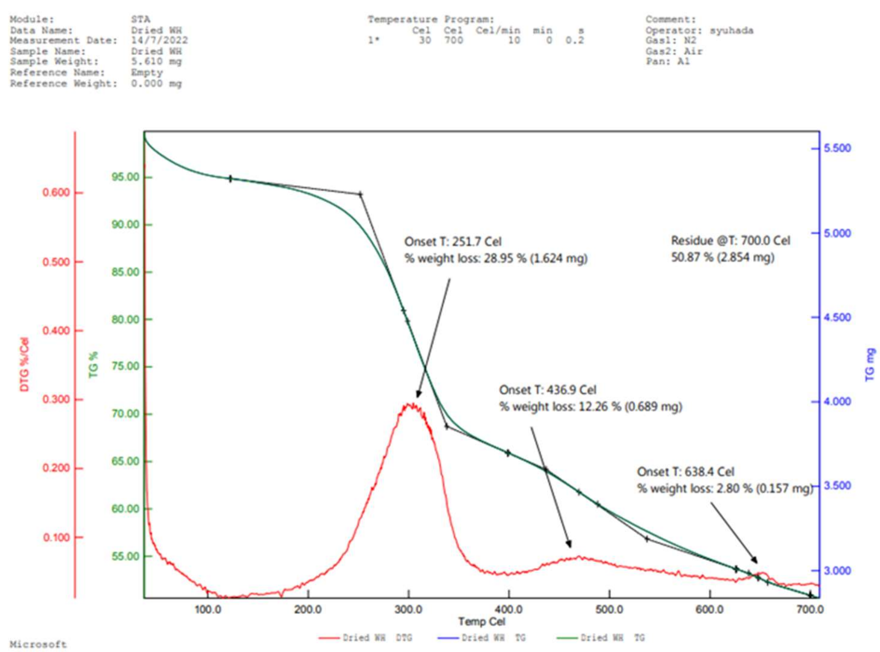

Figure 4: Data of thermogravimetric analysis

The sample Using a Perkin Elmer Thermal Analyzer (STA 6000), the dried water hyacinth sample's thermal degradation behaviour was studied under a nitrogen environment. 5 – 6 mg sample weights were used. The water hyacinth sample was heated with nitrogen gas flow rate of 20 mL/min to maintain inert atmosphere for the thermal degradation. The temperature range for the pyrolysis investigation was 30°C to 950°C with a heating rate of 10°C/min.

The WH TG shows that there are weight loss respects to temperature starting from 200°C until 600°C. A considerable change in the sample's weight is indicated by the graph's steep gradient at 300°C. The gradient, according to the information given, reflects a weight loss of 28.95%. This indicates that 28.95% of the sample's initial weight was lost as the temperature rose to 300°C. This quick process, which resulted in the large weight loss, may have been caused by thermal deterioration, oxidation, or evaporation, as shown by the steep gradient. Understanding the reason for this weight loss can reveal crucial details about the sample's characteristics and thermal stability. According to the information given, the sample under investigation lost another amount of weight between 400 and 500°C. This weight reduction helped the sample lose 12.26% of its initial weight. Understanding the sample's thermal behaviour and its characteristics and stability under various situations can be helped by the knowledge of the weight loss between 400 and 500°C. Last weight loss occurs between 600°C and 700°C which contributed to 2.80% weight loss.

The graph of the DTG shows the activities occurs during TGA. DTG is a sort of thermal analysis that makes reading the weight versus temperature thermogram peaks that appear close together easier by plotting the rate of material weight changes as a function of temperature. The graph shows that the peak of the weight loss of the sample were between 200°C until 400°C. The amount of material left over after heating a sample to a specific temperature or to a specific point in the heating process is referred to as the residue from a Thermogravimetric Analysis (TGA). When a TGA experiment yields 50.87% residue, it signifies that after heating, 50.87% of the original sample is still present, while the remaining 50.87% has been lost owing to evaporation, breakdown, or oxidation. The residue percentage can reveal details about a material's thermal stability, decomposition temperature, and other crucial characteristics.

4.3 Pyrolysis Process

The temperatures of 250, 300, and 400□C were chosen for the pyrolysis process from the temperature range established by the Thermogravimetric Analysis (TGA). Pyrolysis is a thermal process in which a substance is heated in an inert environment to decompose its chemical structure into more basic, smaller molecules. Based on the findings of the TGA analysis, which showed that weight loss happened at various temperatures, the temperature range chosen for the pyrolysis procedure was probably based on those findings. The pyrolysis process can target particular components of the material for breakdown and generate particular products by utilizing the temperatures at which weight loss occurred.

Each sample of WH were burned/heated at different temperature which is 250, 300, and 400□C for 2 hours in furnace oven. The physical and chemical characteristics of the biochar can be enhanced by this procedure, making it more useful for a range of applications. Increased Surface area is one benefit of physical activation via pyrolysis. By increasing the surface area of biochar, physical activation via pyrolysis can improve its suitability for adsorption and catalysis. Next, improved pore structure: Physical activation can also improve the pore structure of biochar, which will increase its capacity to retain nutrients and water in soil. Reactivity: biochar's reactivity can also be raised through physical activation, which makes it more efficient for catalytic processes and the generation of energy. Last but not least, improved chemical stability: Biochar that has undergone physical activation has a chemical structure that is more lasting and stable, making it more resistant to deterioration over time.

Figure 5: Sieved WH in furnace for pyrolysis process

Figure 6: WH biochar after pyrolysis process

4.4 Chemical Activation

The surface area and reactivity of the water hyacinth biochar can increase as a result of chemical activation using 60 ml of 1 mol of phosphoric acid in a 2:1 ratio for 2 hours. The surface functional groups on the water hyacinth biochar can react with phosphoric acid to produce new surface functional groups and increase the biochar's overall surface area. Because of its larger surface area, activated biochar may have better catalytic and adsorption abilities, increasing its usefulness in agriculture, energy production, and environmental clean-up.

Additionally, contaminants like ash and metals that may reduce the efficiency of biochar can be eliminated through chemical activation using phosphoric acid. This may produce a type of biochar that is purer and more stable and is better suited for particular uses.

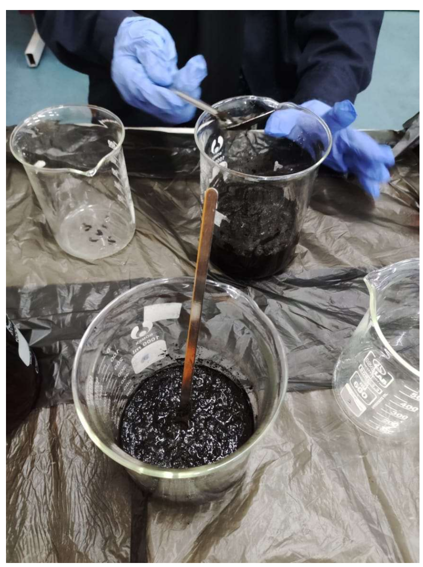

Figure 7: WH biochar impregnated in phosphoric acid

4.5 Parameter Analysis

4.5.1 Chemical Oxygen Demand

The amount of oxygen needed to oxidize the organic contaminants found in wastewater is measured by a term called chemical oxygen demand, or COD. Results in the table below shows decreased of the concentration of COD as the temperature of each sample is different. Dye wastewater CODs were recorded as 1332 mg/L. Sample 1 shows that there is reduction which is 1290 mg/L, same goes with sample 2 and sample 3 which the reading is 1140 mg/L and 1021 mg/L respectively. The water hyacinth-derived activated carbon's ability to bind to organic contaminants is what causes the COD in dye textile wastewater to drop. Since it has a huge surface area and is very porous, activated carbon can efficiently absorb organic contaminants found in wastewater. The activated carbon from water hyacinth can absorb the organic contaminants found in dye textile effluent, lowering the COD of the wastewater. The organic contaminants in the wastewater are drawn to and adhere to the surface of the activated carbon during the adsorption process. As a result, the organic contaminants are removed from the wastewater, which lowers the quantity of oxygen needed to oxidize them and lowers the COD value.

Table 2: COD test result

Sample	Concentration, mg/L	
Sample (Dye Wastewater)	133.2 mg/L	1332 mg/L
Sample 1 (250)	129.0 mg/L	1290 mg/L
Sample 2 (300)	114.0 mg/L	1140 mg/L
Sample 3 (400)	102.1 mg/L	1021 mg/L

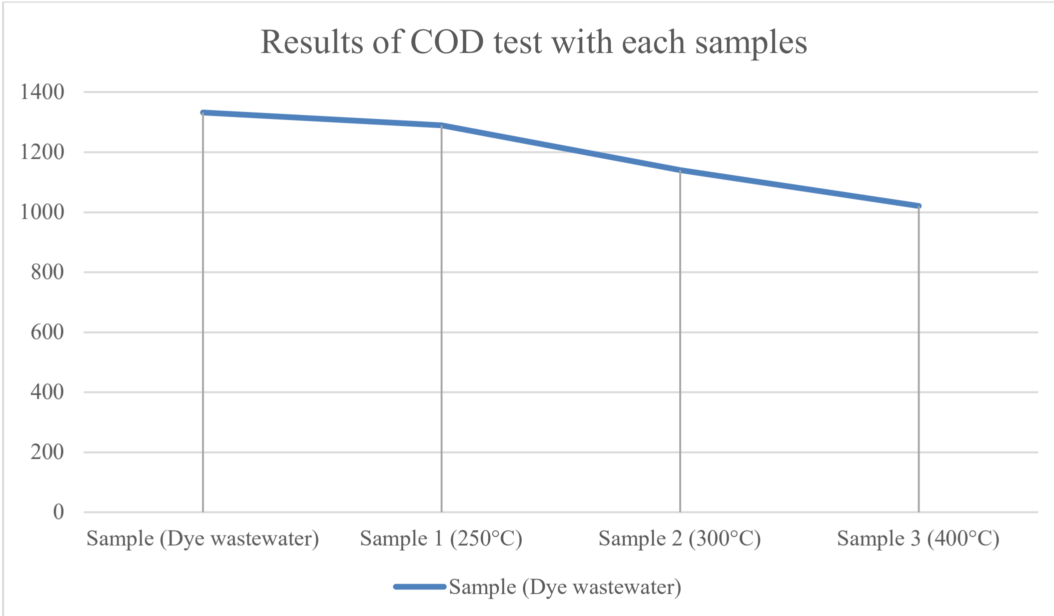

Figure 8: Graph of COD test result

4.5.2 Biochemical Oxygen Demand (BOD)

Pyrolysis is the thermal degradation of organic material in the absence of oxygen, and the temperature during pyrolysis can affect the efficiency of the process. As the temperature during pyrolysis increased from 250°C to 300°C and then to 400°C, it caused an increase in the decomposition of the organic matter present in the water hyacinth, leading to a more porous and active carbon with higher surface area.

As a result of the activated carbon's increased surface area being able to absorb more organic contaminants, there is less organic matter accessible for microorganisms to break down, which lowers BOD. BOD dropped from 42.95 mg/l to 38.75 mg/l and subsequently to 39.0 mg/l as a result of the pyrolysis temperature being raised.

Table 3: BOD test result

	DO	BOD5
Sample (Dye Wastewater)	8.59 mg/L	42.95 mg/L
Sample 1	0.84 mg/L	38.75 mg/L
Sample 2	0.79 mg/L	39.0 mg/L
Sample 3	0.73 mg/L	39.3 mg/L

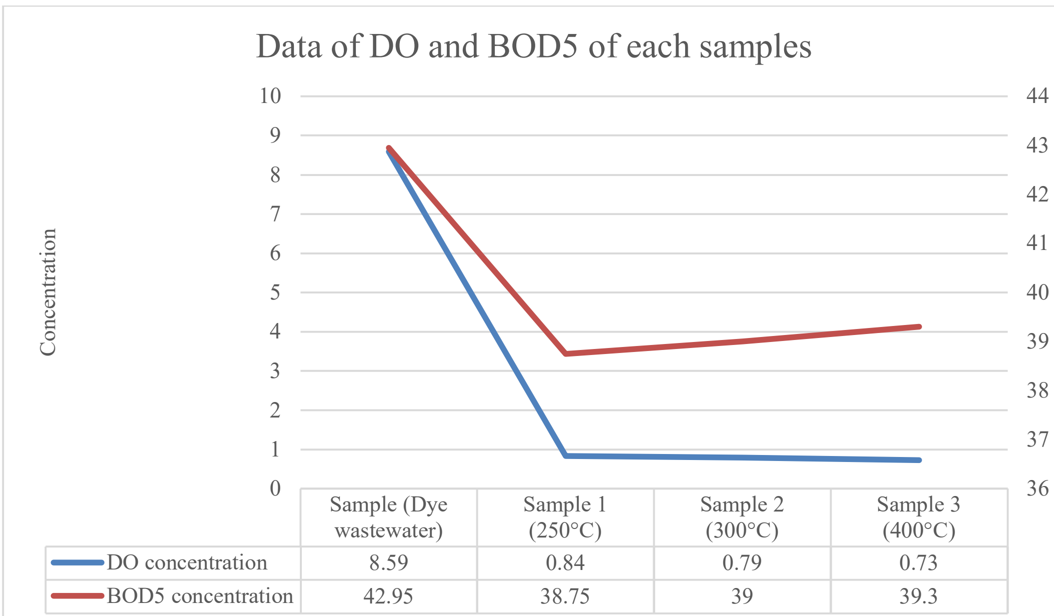

Figure 9: Graph of BOD result

4.5.3 Total Suspended Solids (TSS)

The table below shows decreasing of concentration from sample 1 until sample 3 as the temperature of each sample increase from sample 1 to sample 3. The adsorption of these particles onto the surface of the activated carbon can be used to explain why the TSS concentration in the water hyacinth sample decreased. The pollutants can be drawn to and held onto by the activated carbon during the adsorption process, which lowers their concentration in the water. Due to this, the TSS reading in sample 1 drops from 19.17 mg/L and 617 NTU to 18.593 mg/L and 329 NTU in s

Table 4: TSS result

Sample (Dye Wastewater)	Concentration		
	Mg/L		NTU
Sample 0	19.2317 mg/L	19.17 mg/L	617
Sample 1	19.0911 mg/L	19.1457 mg/L	546
Sample 2	18.8513 mg/L	18.9013 mg/L	500
Sample 3	18.5602 mg/L	18.5931 mg/L	329

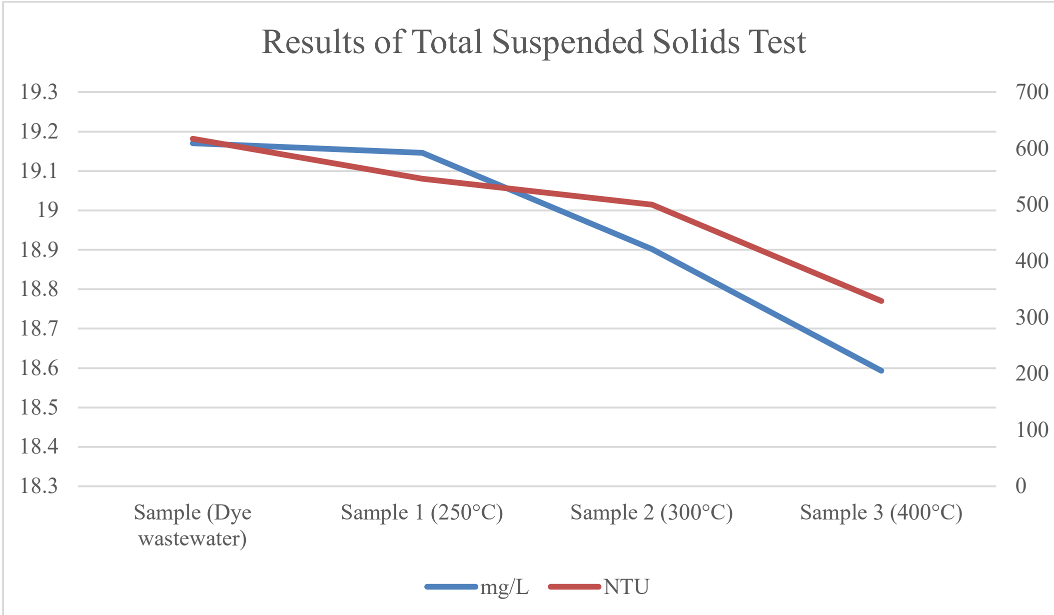

Figure 10: Graph of TSS result

Total Suspended Solids (TSS) refers to the concentration of solid particles in water, and the decrease in TSS in dye wastewater denotes this. Since high levels of TSS might signal the presence of contaminants and organic matter in the water, a decrease in TSS is typically an indication of improving water quality. The elimination of these particles can make wastewater more suitable for release into the environment or for further treatment while also lowering its toxicity. Because dye wastewater can increase the levels of TSS in the water, a decrease in TSS can also signify that the dyes have been successfully removed from the wastewater.

4.5.4 Colour ADMI

The dye wastewater's colour ADMI has decreased, which suggests that there is less dye in the wastewater overall. In other words, as observed from samples 1 (2220) to 4, the colour intensity of the wastewater is decreasing over time (1180). According to the declining colour ADMI measurements, the wastewater treatment process (in this example, activated carbon made from water hyacinth) is successful in removing the dye. The results are as the table below.

Table 5: Colour ADMI result

	Color ADMI
Sample (Dye Wastewater)	2220
Sample 1	1730
Sample 2	1650
Sample 3	1180

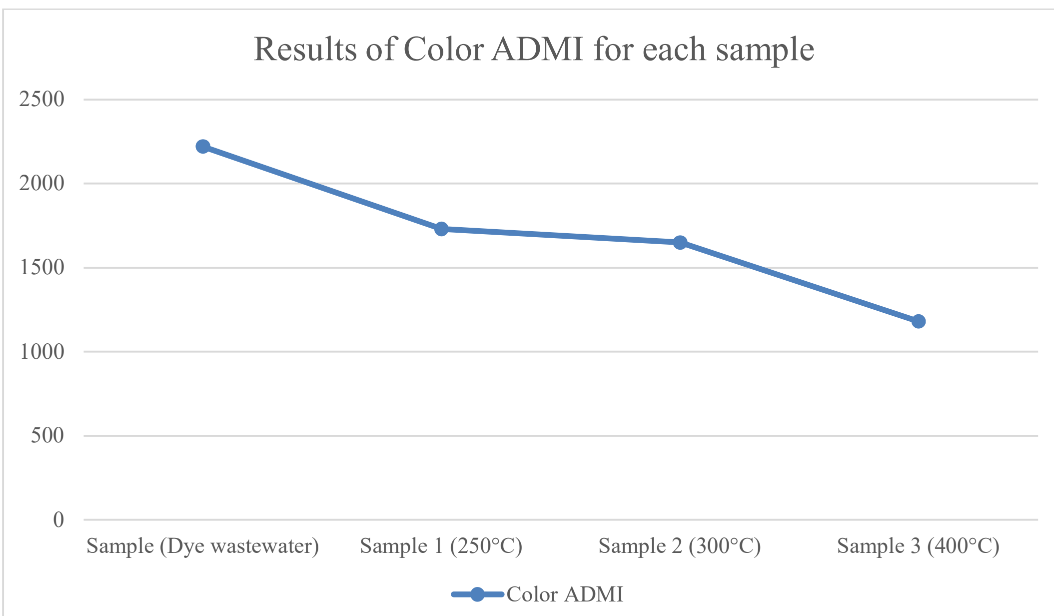

Figure 11: Graph of colour ADMI result

An indicator of a specific colour's intensity in wastewater is the colour ADMI. The amount of dye in the wastewater must be reduced for the ADMI to decrease, and this reduction in dye content immediately relates to a decrease in colour intensity. In this instance, the water hyacinth-derived activated carbon is successfully removing the dye from the wastewater, which is causing a drop in the colour ADMI measurements.

CHAPTER 5

CONCLUSION

In conclusion, research has been done on the activation of water hyacinth-derived carbon using phosphoric acid and its application to the treatment of dye wastewater. The objective of the research are achieved. TGA analysis was used to characterize the activation process and find the ideal pyrolysis temperature. The effectiveness of the treatment was assessed by measuring different parameters like total suspended solids (TSS), biochemical oxygen demand (BOD), chemical oxygen demand (COD), and colour ADMI after the activated carbon had been employed to treat dye wastewater. The results demonstrate that water hyacinth-derived activated carbon is an efficient substance for the treatment of dye wastewater as it greatly lowers the colour levels of ADMI, BOD, COD, and TSS. Plus, the design of the filter system is working well and can be improved. The potential of water hyacinth as a sustainable and affordable source of activated carbon for the treatment of wastewater containing organic contaminants is highlighted by this study.

The use of water hyacinth as an activated carbon for dye wastewater treatment is a promising approach due to its ability to absorb pollutants, including dyes. Here are a few recommendations for future studies in this area:

1) Further study is required to improve the activation process in order to increase the effectiveness of colour removal by water hyacinth activated carbon. It is important to adjust variables including temperature, timing, and activating agent kind.

2) Comparison with other biochar: To ascertain which biochar is more efficient at removing dye, it would be interesting to compare the performance of water hyacinth activated carbon with that of other biochar, such as rice straw and sawdust.

3) Adsorption kinetics and mechanism: Improving our comprehension of adsorption kinetics and mechanism will aid in process optimization. It is possible to do research to find out how variables like pH, initial dye concentration, and temperature affect the adsorption process.

4) The creation of cost-effective techniques will be crucial to the universal acceptance of this strategy for the synthesis and regeneration of water hyacinth activated carbon.

5) Integrating water hyacinth activated carbon with other treatment techniques, like as coagulation, flocculation, and oxidation, may increase the effectiveness and cost-effectiveness of waste removal.

These recommendations can help guide future research and development of water hyacinth as an activated carbon for dye wastewater treatment.

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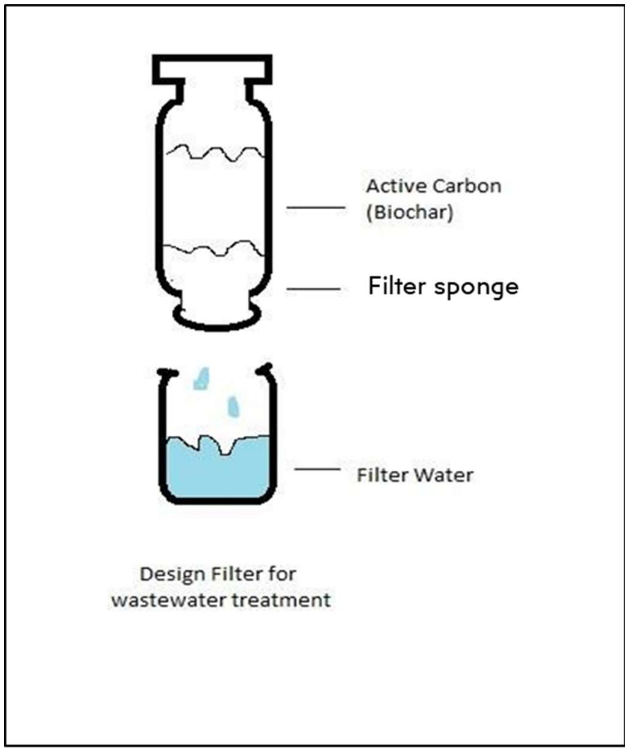
APPENDICES

APPENDIX A: FILTER SYSTEM DESIGN



APPENDIX B: ACTICATED CARBON PRODUCTION PROCESS