

TREATMENT OF TEXTILE WASTEWATER
USING
BIO-CHAR BASED PALM OIL
MILL SLUDGE (POMS)

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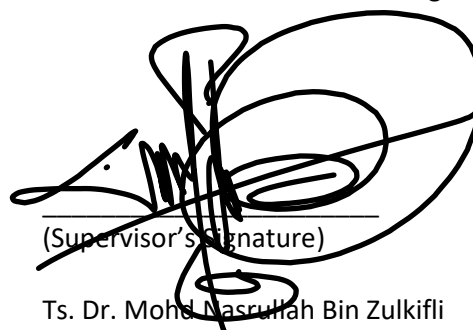
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ABSTRAK

Tujuan penyelidikan ini adalah untuk menyediakan biochar, sebuah produk tambah nilai daripada produk sampingan enap cemar yang dihasilkan melalui proses pirolisis sisa pepejal minyak kelapa sawit (POMS) melalui kaedah pengaktifan kimia. Biochar juga disebut arang aktif adalah bentuk karbon yang diproses untuk memiliki pori-pori kecil, isipadu rendah yang meningkatkan luas permukaan yang tersedia untuk adsorpsi atau reaksi kimia. Biochar biasanya digunakan untuk rawatan air, air sisa dan resapan untuk menghilangkan bau dan logam berat. Ia sangat murah dan mudah didapati dalam pasaran, digunakan untuk merawat bahan pencemar yang terdapat dalam air sisa dan untuk pelbagai aplikasi industri. Produk sampingan yang dihasilkan dalam proses ini, iaitu enap cemar, ditukarkan kepada karbon aktif. Enap cemar efluen kilang kelapa sawit digunakan pendahulu untuk menyediakan karbon aktif. Proses aktivasi and parameter proses menentukan sifat fizikal karbon aktif. Proses penyediaan enapcemar diikuti dengan pengeringan dan pirolisis dengan ketiadaan oksigen, prosedur pengeringan pertama kali dijalankan dalam ketuhar pada 105°C selama 24 jam untuk menjadikan (POMS) menjadi karbohidrat yang sangat tinggi. Biochar yang terbentuk diaktifkan menggunakan asid dan reagen alkali untuk menghasilkan biochar diaktifkan kimia dengan keliangan dan keberkesanan yang tinggi. Kemudian, biochar diaktifkan sedang menjalani proc pirolisis. Sifat permukaan dan komposisi unsur biochar yang diperolehi sebagai dicirikan. Dengan menggabungkan adsorbent dan air sisa dalam kelalang kon dan menggegarkannya pada 200 rpm selama 10 minit, biochar berasaskan POMS digunakan untuk merawat air sisa pewarna tekstil. Hasil penyelidikan mendapati pengaktifan berasaskan biochar (enapcemar kilang kelapa sawit) boleh digunakan sebagai calon penjerukan untuk merawat permintaan oksigen kimia (COD), warna, pH daripada air sisa tekstil

ABSTRACT

The aim of this research is to prepare activated biochar, a value added product from waste the by-product means sludge produced from palm oil mill sludge (POMS) through chemical activation. Biochar has been used in a variety of applications since its discovery as a solid and effective adsorbent. It is widely used to extract odour and certain heavy metals from sewage, drainage, and leachate, it is also inexpensive and widely available in the marketplace. With its highly micro porous structure, it is reliable and effective for extracting contaminants from aqueous solutions. The sludge generated as by product of this process is recovered and converted into activated biochar. POMS sludge was used as main precursor to prepare the biochar. The physical properties of biochar are determined by the activation mechanism and process parameters. The sludge preparation process followed by drying and pyrolysis with the absence of oxygen, drying procedure was first conducted in an oven at 105°C for 24 hours to turn the (POMS) into a highly carbonaceous substance. The biochar formed is activated using acid and alkaline reagent to generate chemically activated biochar with a high porosity and effectiveness. Then, the activated biochar is undergone pyrolysis process at different temperature as 500°C, 600°C and 700°C for 60 minutes. The surface properties and elemental compositions of the biochar obtained as **characterised**. By combining the adsorbent and wastewater in a conical flask and shaking it at 200 rpm for 10 minutes, the POMS based biochar was used to treat textile dye wastewater. The research finding the activation of biochar based (palm oil mill sludge) could be used as an adsorbent candidate for treating chemical oxygen demand (COD), colour, pH from textile wastewater.

TABLE OF CONTENT

DECLARATION	
TITLE PAGE	
ACKNOWLEDGEMENTS	ii
ABSTRAK	iii
ABSTRACT	iv
TABLE OF CONTENT	v
LIST OF TABLES	vi
LIST OF FIGURES	vii
LIST OF SYMBOLS	viii
LIST OF ABBREVIATIONS	ix
LIST OF APPENDICES	X
CHAPTER 1 INTRODUCTION	13
1.1 Project Background	13
1.2 Problem Statement	14
1.3 Objectives	15
1.4 Research Scopes	15
1.5 Significant of the Study	16
CHAPTER 2 LITERATURE REVIEW	19
2.1 Pyrolysis	19
2.2 Biochar	19
2.2.1 Biochar Based Palm Oil Mill Sludge (POMS)	19
2.2.2 Biochar Production	18
2.2.3 Raw Material for Production of Biochar	20
2.2.4 Characteristics of Sludge Based Biochar	22

2.2.5	Classification of Biochar	22
2.2.6	Chemical Structure of Biochar	23
2.2.8	Preparation of Biochar	24
2.2.9	Pores of Biochar	24
2.3	Textile Dye Wastewater	25
2.3.1	Properties of Textile Dye Wastewater	28
2.3.2	Classification of Textile Dyes	28
2.3.3	Toxicity of Textile Dyes Wastewater	29
CHAPTER 3 METHODOLOGY		32
3.1	Introduction	32
3.2	Preparation of Activated Biochar	34
3.2.1	Carbonization of POMS	34
3.2.2	Chemical Activation of POMS	34
3.3	Analysis of Textile Wastewater	35
3.3.1	Chemical Oxygen Demand (COD) Analysis	35
3.3.2	Colour Analysis	35
3.3.3	Total Suspended Solid (TSS) Analysis	36
3.3.4	pH Analysis	36
3.4	Textile Wastewater Dye Wastewater	36
3.4.1	Preparation and Characteristics	36
3.5	Adsorption	37
3.5.1	Batch Adsorption	37
CHAPTER 4 RESULTS AND DISCUSSION		38
4.1	Introduction	38
4.2	Adsorption of Biochar Based Palm Oil Mill Sludge (POMS)	39
4.2.1	pH Analysis for adsorption of biochar based POMS	39
4.3	Performance of Biochar Based palm oil mill sludge (POMS) as adsorbent	40
4.3.1	Removal Performance of Chemical Oxygen Demand (COD)	40

4.3.2	Removal Performance of Total Suspended Solid (TSS)	41
4.3.3	Removal Performance of Colour	43
CHAPTER 5 CONCLUSION		45
5.1	Introduction	45
5.2	General Conclusion	45
5.1	Recommendation	47
REFERENCES		50

LIST OF TABLES

Table 2.2	Differences between the one-step and two-step pyrolysis processes	26
Table 4.2	pH of textile wastewater after subjecting biochar based (POMS)	40
Table 4.3	COD of textile wastewater after subjecting biochar based POMS	42
Table 4.4	(TSS) of textile wastewater after subjecting biochar POMS	43
Table 4.5	Colour of textile wastewater after subjecting biochar based POMS	44

LIST OF FIGURES

Figure 2.1	Schematic diagram of structure of activated carbon	27
Figure 2.2	Number of articles based on the chemical activator used for the preparation of activated carbon from agricultural residues	28
Figure 3.1	Flow chart of research methodology	35
Figure 4.1	pH vs Biochar	41
Figure 4.2	Chemical Oxygen Demand (COD) vs Biochar	42
Figure 4.3	Total Suspended Solid (TSS) vs Biochar	44
Figure 4.4	Colour vs Biochar	45

LIST OF SYMBOLS

LIST OF ABBREVIATIONS

POMS	Palm Oil Mill Sludge
DS	Dried Sludge
GAC	Granular Activated Carbon
PAC	Powdered Activated Carbon
POME	Palm Oil Mill Effluent
POMS	Palm Oil Mill Sludge
SS	Suspended Solids
TSS	Total Suspended Solid
IR	Impregnation Ratios

LIST OF APPENDICES

Appendix A: Source of palm oil mil sludge (POMS) from Lepar Hilir.	56
Appendix B: Wash palm oil mill sludge(POMS).	56
Appendix C: Drying process of (POMS).	57
Appendix D: Dried palm oil mill sludge(POMS).	57
Appendix E: Chemical activation of POMS with (KOH) & (H ₃ PO ₄).	58
Appendix F: Wash chemically activated POMS with distil water.	58
Appendix G: Carbonization using furnace of (POMS) after chemical activation.	59
Appendix H: Carbonized palm oil mill sludge (POMS).	59
Appendix I: Crushing and sieving of carbonized palm oil mill sludge (POMS).	60
Appendix J: Collection of textile wastewater from Tenun Diraja Pahang.	60
Appendix K: After applying activated biochar based (POMS) into textile wastewater.	61
Appendix L: Overall colour adsorption of biohar based (POMS).	61
Appendix M: Ideal activated biochar based (POMS).	62

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Palm oil is the second largest vegetable oil in the world, according to global demand. Palm oil is a natural resource that thrives in Malaysia's tropical climate. In 2017, Malaysia had a total of 454 palm oil mills with a production capacity of approximately 112 million tonnes of fresh fruit bunches. After Indonesia, it is the world's second-largest producer of the product. Malaysia currently produces 39 percent of the world's palm oil and exports 44 percent of it (Begum, 2019). In 2004, palm oil became the most important oil product in the world in terms of production, trade, and consumption. According to the United Nations' Food and Agriculture Organization (FAO), the oil palm planting area in Southeast Asia accounted for roughly 71 percent of the overall oil palm planting area in the world in 2017 (FAO, 2019).

However, the continuous expansion of oil palm plantations not only leads to large changes in land use types, but also seriously threatens the environment, including reduced biodiversity in forest ecosystems, increased greenhouse gas emissions, and water pollution (Li, 2020). In this country alone, 43.29 million cubic metres of POME are generated per year. (Hayawin, Jalani, et al., 2017) and constitutes to a serious environmental pollution by releasing it to the environment, especially in waterways. The release of untreated or inadequately treated POMS into the environment will result in serious consequences such as groundwater pollution, methane release, and an unwanted odour into the atmosphere. (Chavalparit, 2016). All carbonaceous materials could be used as raw materials in the preparation of carbonaceous adsorbent. Palm oil sludge's high carbon content makes it ideal for the manufacture of carbonaceous adsorbents. Apart from resolving issues with POMS treatment and disposal, biochar made from POMS sludge may be used to reduce a variety of contaminants in water.

Adsorption has received considerable attention among the several dye removal strategies presented because of its capacity to remove various kinds of dye and provide high quality treated water, ease of operation, simplicity of design, insensitivity to hazardous contaminants, and lack of formation of dangerous compounds (Crini, 2016). Biochar is one of the most researched materials for textile dye adsorption. Biochar is a cost-effective substance in addition to being very efficient (Herrera-González, Caldera-Villalobos, & Peláez-Cid, 2019).

Biochar is the most effective adsorbent known for the treatment of domestic, municipal, agricultural and industrial wastewater due to its small particle sizes and good adsorption capacity. The use of POMS sludge as activated carbon to treat dye waste water is unexplored in published literature. As a result, the aim of this study was to characterise the possible use of sludge-activated carbon from POMS. The activated carbon from sludge of palm oil mill effluent is produced by using physical activation. A two-step approach was used to convert palm oil mill effluent into activated carbon, which represents an abundantly usable oil palm waste in Malaysia. The surface properties, as well as the elemental and proximate concentrations, of the activated carbon obtained will be thoroughly investigated. The effect of experimental factors such as interaction time and temperature on the sorption experiment was also studied.

1.2 Problem Statement

The palm oil sector in Malaysia is expanding rapidly, and there is a huge amount of palm oil sludge available to be processed for the production of oil palm products. Alternative ways for managing with this substantial amount of palm oil sludge must be devised. Furthermore, humanistic activities in Malaysia involve significant consideration as they discharged tonnes of harmful materials into the aquatic environment, containing toxic substances. The increase in palm oil production for food and products has raised major concerns on greenhouse gas (GHG) emission. Studies have shown that agricultural and plantation acreage are one of the biggest contributors to the total global GHG emission, accounting for 17%. This is alarming for Malaysia's reputation as the second largest palm oil producer, which currently contributes 39% of world palm oil production and 44% of world exports. While most studies have focused on the GHG emission from palm oil mill sludge (POMS) and GHG emission from oil

palm peat, little attention is given to GHG emission arising from operation during oil palm planting production activities.

Textile industry wastewater usually has a high concentration of total dissolved solids and suspended solids. Due to dyestuff and suspended solids, the wastewater is often heavily colour and viscous (Yaseen, DA and Scholz, M, 2018). For the effluent to be safely discharged, the wastewater must be effectively treated and not only does the textile wastewater contain colour pigment, but it also includes other metallic particles, a high pH fluctuation, and suspended solids with a high chemical oxygen demand (COD). One of the possible causes of defilement and waste to the atmosphere is the discharge of effluents from these applications and these contaminants in water are not only unsightly, but they are also harmful to organic species, human beings, and ecosystems. As there's metal contamination in textile effluent due to the presence of dye and additive used during textile manufacturing steps. Cobalt, copper, and chromium are the most common metals found in dye chromophores in textile effluents (Yaseen, 2018). Whereas chromium, zinc, iron, mercury, and lead are the primary metals that cause environmental problems. Textile wastewater contamination prevents biological processes from performing the roles that society requires and puts natural maintainability in jeopardy. Owing to increasingly strict environmental standards, the textile industry has been forced to manage its effluents (Yang et al., 2020). To find an economical and efficient way to treat textile wastewater, many techniques such as biological treatment, precipitation, coagulation/flocculation, flotation, oxidation, and adsorption are the most common methods used to treat textile wastewater ("The Textile Industry," 2013).

Adsorption is a commonly used process of physiochemical wastewater treatment, in which wastewater is combined with a porous material such as activated carbon or bio-char, allowing the wastewater to pass through a granular filter bed. Adsorption is good method for removing toxin from wastewater because of the high efficiency of activated carbon because of their demonstrated efficacy in removing organic a mineral contaminant, as well as cost considerations. This adsorption procedure has recently gained popularity due to their effectiveness in removing harmful contaminants emitted during the traditional process and adsorption is a low cost& practical method that yields a superior product. It is now widely accepted as a reliable and cost-effective approach for water decontamination and separation analysis. It's a

fantastic colour evacuation technique that doesn't need any special equipment aside from the adsorbent and is most straightforward to utilize.

1.3 Objectives

The aim of this study is to produce biochar from sludge from palm oil mill sludge (POMS). The following specific objectives are developed:

- Produce bio-char based palm oil mill sludge through pyrolysis process.
- Compare the effectiveness between raw bio-char and chemical activated bio-char.
- Able to treat textile wastewater by utilizing the produced bio-char based Palm oil mill sludge (POMS).

1.4 Project Scope

In this research, the raw material palm oil mill sludge (POMS) was procured from Lepar Hilir 3 Palm Oil Mill as biomass raw material for the mass production of biochar by way of pyrolysis and the preparation of biochar via chemical activation method. While for the sample of textile wastewater was obtained from nearest location which is from 'Tenun Diraja Pahang'. The carbonization process was conducted out in a muffle furnace with flow of nitrogen atmosphere inside it along with certain range of temperature of 500°C, 600°C and 700°C.

Then, the formulated biochar based POMS was then experimented for its performance. The best carbonization temperature adsorbent was tested by batch adsorption by varying several parameters. Prior proceeding with the batch adsorption wastewater sample of textile wastewater was collected from Tenun Diraja Pahang.

Following that, the effluent of textile wastewater was used as an adsorbent to prove the efficacy of biochar created based palm oil mill sludge (POMS). Refer to the previous testimony, by the end of test made a comparison between the raw bio-char and both chemically activated biochar. The effect of adsorbent dosage on the four parameter independent are pH, Chemical Oxygen Demand (COD), Total Suspended Solid (TSS) and colour. The investigational specific was important in determination of the adsorption effectiveness of both acid and alkaline activated based palm oil mill sludge (POMS) biochar.

1.5 Significance of the Study

Palm oil production is vital for the economy of Malaysia, which is the world's second-largest producer of the commodity after Indonesia and palm oil mill sludge (POMS) is the main pollutant produced in palm oil mills in Malaysia. These circumstances bring about several environmental pollution and hazards if there is no appropriate governance, as it may lead to main source of air pollution and water pollution in the future. This is due to palm oil mill sludge (POMS) contains high nutrient, organic and carbon contents despite having high biochemical oxygen demand (BOD) and chemical oxygen demand (COD) content. As a remedy to the above issue, the sludge in the palm oil mill sludge (POMS) is utilized as a raw material for the creation of bio-char which is effective in number of process application. Mostly biochar is generated from feedstock as agricultural waste, animal manures and paper products. The importance of these wastes for the creation of biochar is a constructive mechanism to bring about waste to useful and value added substance. This study evaluates the impacts of creation of biochar based palm oil mill sludge (POMS) by pyrolysis in terms of environment, economy and society.

Besides that, eventually the biochar benefits the environment by sorb major environmental pollutants for the water and soil. Many organic pollutants are being cut off by utilizing biochar to alter the effects on the environment eventually. Due to its resisting nature towards microorganisms and its extraordinary sorption affinity, biochar acts as a critical binding phase for the different organic pollutants in the environment. The creation of biochar by pyrolysis for treatment motive could greatly benefit the economy of the country as it requires less cost and energy compared to that of commercial treatments. Additionally, the simplicity of the design insensitivity to toxic pollutants and not resulting in the formation of harmful substances along with the treatment process makes it extremely favourable.

Then, the utilization of bio-char impacts the society by allocate them with knowledge and understanding on its application and productions. Any carbonaceous materials could be utilized as the raw material for the production of biochar and many researches have been done on the production of bio-char based biomass as like plant residue, wood chips and organic waste to be converted into bio-char. Biochar's

production cost is also a significant constraint in its broad promotion and application. As a result, low cost, high-efficiency biochar research and development is an unavoidable driving force in the development in this field. Invasive plants exist in a range of shapes and sizes, are widely spread, and are renewable, these plants are more cost-effective and readily accessible as charcoal feed stocks than some other biomass waste. Furthermore, investigations have indicated substantial structural, composition, adsorption performance variations between biochar formed by invasive plants and other raw materials (Fan et al., 2020). As a potential adsorbent, biochar has prospects as a low-cost substitute to activated carbon for wastewater treatment (Perez-Mercado, Lalander, Berger and Dalahmeh, 2018).

The combined usage biochar in wastewater treatment solves today's wastewater management problems and the benefit of employing it. On the other hand, varies based on its kind and features, which are reliant on the biomass and pyrolysis circumstances. Due to unique properties of the biochar, including as adsorption efficiency, specific surface area, microporosity, and ion exchange capacity as biochar provides a wide range of uses in water along wastewater treatment (Ahmad et al., 2014). The interactions of various contaminants with various properties of biochar, which are dependent on pyrolysis temperature, input type, regulate their removal pathways along the temperature at which biochar is pyrolyzed has a significant effect on its own attributes. Biochar with a higher carbon content, hydrophobicity, aromaticity, surface area, and microporosity does have a higher pyrolysis temperature.

Thus, this study provides details and references about generating bio-char based palm oil mill sludge (POMS), the methods and results can be referred as guideline ad direction for further advancement and exploration.

CHAPTER 2

LITERATURE REVIEW

2.1 Pyrolysis

Biomass, as an effective source of energy, is one of the alternatives to fossil fuels, which can be utilised by direct burning or indirect thermal treatment. Thermal degradation processes, like pyrolysis or gasification, were designed to convert the organic matter into a variety of products including gases, oils and chars (Zhang, 2020). During pyrolysis, biomass is thermally decomposed in the oxygen-depleted conditions with temperatures between 300 and 1200°C. This temperature range is seen as a main advantage when compared with gasification and combustion processes (Pawel, 2017).

Utilisation of waste biomass materials in pyrolysis process is a part of a current trend of closed cycle production to achieve more sustainable processes and products. The residue charred material formed during pyrolysis has much higher carbon content (% wt.) in comparison to the substrate, since moisture, volatiles, and most of the compounds with non-carbon heteroatoms are removed during the thermal treatment. Char with low commercial value formed from waste biomaterials can be activated to produce adsorbents for various industrial processes or used directly as a fuel. The final surface properties and functionalities of the char are determined by the nature of pyrolyzed precursors and the type of activation process. In this regard, a variety of raw materials, as well as methods to enhance properties of the char, were investigated to produce effective and low-cost products with high sorption properties (Amaya, 2017).

The high surface area and ability to influence the pore size and distribution during the production process makes adsorbents from waste biomass suitable for areas as different as the separation and purification of gases, water treatment, energy storage and catalysis, among others (Tan, 2017).

2.2 Biochar

2.2.1 Biochar Based Palm oil mill sludge (POMS)

Biochar is a carbon-rich dry biomass obtained through pyrolysis, a heating process. For example, waste from industry activities such as wood, dung, or rubbish. Biochar has recently gained popularity since it may be used in a variety of industries, including soil restoration, agriculture, and pollution control. It can be used to increase agricultural yields as well as carbon sequestration. Biochar, on the other hand, is an effective method for reducing carbon emissions into the atmosphere. Furthermore, by burying bio-char in the soil, biochar can improve soil characteristics and store carbon. Because of its propensity to absorb, biochar can also be used to remove pollutants from aqueous solutions (Gwenzi, Chaukura, Noubactep & Mukome, 2017).

The left solid phase product after treatment of the palm oil mill effluent (POME) discharge from the entire palm oil manufacturing process is known as palm oil mill sludge (POMS). After the POME treatment, it is made up of suspended solids and dissolved solids. In order to make high quality biochar, POMS traits and attributes must be examined and learned. Physical and chemical parameters have a big impact on the quality of biochar made from POMS.

2.2.2 Biochar Production

Palm oil residues are one of the most abundant biomasses from agriculture, biochar a black carbon rich material obtained from the pyrolysis of organic biomass under oxygen-limited conditions. It has been prevalently recognised as a readily available, environmentally friendly sorbent for contaminant management in water and soil. It is confirmed that the sorption effects of pollutants by biochar are closely related to its physicochemical properties as like surface area, high cation exchange capacity and surface functional groups which significantly depend on the biomass source and pyrolysis conditions. Recently, macroalgae have been used as the precursor for preparing biochars because of their massive abundance and easily acquired nature. Moreover, the conversion of biomass to biochar as one potential end-use will improve the resource utilisation of macroalgae, especially for the 'pest' species for an example

green tide algae that exhibit rapid growth and environmental tolerance in eutrophic environments (Cheng, 2021).

Biochar is a widely available material, holds promises for pollutant adsorption. So far, biochar has been found to be effective for multiple purposes, including carbon sequestration, nutrient storage, and water-holding capacity. However, its limited porosity restricts its use in water treatment. Biochar is a carbon rich solid substance derived from biomass through pyrolysis, a thermochemical process. During pyrolysis, the feedstock's lignin, cellulose, hemicellulose, fat, and starch are thermally broken down, yielding biochar (solid), bio-oil (partly condensed volatile matter), and non-condensable gases such as CO₂, CO, CH₄, and H₂ (Suliman et al., 2016). Bio-oil and gases can be captured to provide energy and valuable coproducts such as wood preservatives, food flavours, adhesives, or biological compounds, depending on the feed. However, the yield of biochar and its properties are determined by the pyrolysis condition.

Slow pyrolysis at a moderate temperature (350–500°C) and slow heating rate produces a greater yield (30%) of biochar than fast pyrolysis (600–700°C and fast heating rate) or gasification (temperature 700°C or above), which yields around 10% or less. Surface area, polarity, atomic ratio, pH, and elemental composition all alter significantly depending on the feedstock type and pyrolysis condition used to produce biochar. Biochar's efficacy in wastewater treatment is determined by these qualities. Physicochemical activation process is appropriate for the production of highly porous materials. As well, the morphological and chemical structure of feedstock together with pyro-gasification operating conditions for the biochar production can greatly impact the porosity of the final materials.

The effectiveness of activated biochar as adsorbent depends on porosity and on some functional groups connected to its structure, both of these are developed during activation. This study provides a comprehensive synthesis of the effect of several activated biochars when applied to the treatment of organic and inorganic contaminants in water. Results show that high aromaticity and porosity are essential for the sorption of organic contaminants, while the presence of oxygen-containing functional groups. Optimum pH are crucial for the sorption of inorganic contaminants especially metals. Although activated biochar is a promising option for the treatment of contaminants in

water, further research is required to evaluate its performance with real effluents containing contaminants of emerging concern (Flavia, 2018)

Activated biochar is an amorphous carbon based substance that comes in powdered or granular form and is structured in an indefinite shape. Carbonization and activation are used to create it, which results in a high level of porosity along with an increased inter-particulate surface area. Activated biochar is a good non polar adsorbent because of its huge specific surface area and micropore, good adsorption performance and recycling it can be used together with a variety of substances to adsorb various properties of wastewater. This paper mainly introduces the structure, adsorption mechanism, modified activated biochar, microbial bound activated biochar, microwave bound activated biochar and the future research direction of activated biochar.

2.2.3 Raw Materials for Production of Biochar

Activated biochar may be made from a variety of low-cost materials and biomass, including waste biomass, coconut shell, and so on (Girods et al., 2019). Previously, readily available carbon sources such as wood, peat, and vegetable waste were commonly used as raw materials (Ao et al., 2018). However, several low-cost and effective alternatives are being investigated these days. Various agricultural and industrial waste are being investigated as source materials for biochar production (Nabais, Carrott, Carrott, Luz, & Ortiz, 2008).

The final surface properties and functionalities of the char are determined by the nature of pyrolyzed precursors and the type of activation process. In this regard, a variety of raw materials, as well as methods to enhance properties of the char, were investigated to produce effective and low-cost products with high sorption properties. Chars were produced from waste of wooden and non-wooden origin, including agricultural residues, rice husk, palm shells, coconut shells, apple and cherry pulp, plum pulp and stones, and olive stones among others. The high surface area and ability to influence the pore size and distribution during the production process makes adsorbents from waste biomass suitable for areas as different as the separation and purification of gases, water treatment, energy storage and catalysis, among others.

2.2.4 Characteristics of Sludge Based Biochar

The physical and chemical properties of biochar, as well as other material characteristics, must be learned during the biochar preparation process in order to create a superior biochar output. In order to assure the precision and dependability of biochar, both physical and chemical qualities must be examined. Chemical properties such as moisture content, ash content, volatile matter, fixed carbon, and function group of biochar provide data in terms of biochar yield, surface area, surface morphology, carbon content, and physical structure, while functional group and chemical content provide data in terms of biochar yield, surface area, surface morphology, carbon content, and physical structure. Biochar has diverse properties depending on the biomass feedstock.

2.2.5 Classification of Biochar

According to the size and form of the particles, activated carbon is divided into three categories: granular, power, and extruded activated carbon. Each type has its own variety of purposes and functions. Powdered activated biochar (PAB) comes in the form of fine granules or powders with an average diameter of 15-25 μ m and a size of less than 100 μ m. It has an apparent density of 23 to 46 lb/ft². This characteristic demonstrates that PAB has a large interior surface area and a short diffusion distance. PAB's crushed or ground carbon particles will be sorted using mesh sieves or sifters to certain mesh sizes. PABs are primarily used for adsorption in the liquid phase. PAB has a lot of advantages, including reducing processing costs and increased efficiencies (Parvathi, Maruthavanan, & Prakash, 2019).

In comparison to PAB, granular activated biochar (GAB) has a larger particle size range of 200m to 5000m, resulting in a smaller external surface. They're also tougher and last longer than PAB. It is recommended for most gases and vapours adsorption due to its high kinetics of adsorption and rapid diffusion rate. GAB is available in two forms: granular and extruded. The most common sizes for liquid phase applications are 8x20, 20x40, or 8x30, whereas the most common sizes for vapour phase applications are 4x6, 4x8, or 4x10. The most widely utilised ones are 12x40 and 8x30, which have an excellent mix of size, surface area, and head loss properties. Furthermore, GAB is constantly revived and recycled. During the reaction, only 5-15% of the materials are wasted. (Cheremisinoff, 2016).

As a consequence of fusion between powdered activated carbon and extruded activated carbon with a binder, cylindrical shaped activated carbon block with diameters ranging from 800- 1300m are extruded. It is often applied in gas phase applications because to its low dust percentage, low pressure fall, and excellent mechanical strength.

2.2.6 Chemical structure of Biochar

The inside of activated biochar has crystal and pore structures, while the surface of activated biochar contains chemical structure as well. The chemical structure of activated biochar surface affects the adsorption effectiveness of it as well as the physical (pore) structure of activated biochar. The edge chemical bond of the aromatic sheet created during the carbonization step breaks during the manufacture of activated carbon, resulting in an edge carbon atom with unpaired electrons. These marginal carbon atoms contain unsaturated chemical bonds and can create diverse surface groups by reacting with heterocyclic atoms like oxygen, hydrogen, nitrogen, and sulphur. The presence of these surface groups impacts the adsorption capabilities of activated carbon without a doubt. Acidic, alkaline, and neutral surface groups exist on activated carbon (Jiang, 2017).

Biochar is constantly made up of surfaces that are positively and negatively charged. cation exchange capacity (CEC) is aided by negatively charged functional groups, whereas anion exchange capacity (AEC) is aided by O-containing functional groups (oxonium heterocycles) in biochar. Because they carry a negative charge and serve as Lewis bases for cation sorption, oxygen (O) containing alcohol, carbonyl, and carboxylate functional groups is believed to contribute to biochar cation exchange capacity (Lawrinenko and Laird, 2015). In biochars, oxonium functional groups are believed to contribute to pH-independent anion exchange, while pyridinic functional groups and nonspecific proton adsorption by condensed aromatic rings are thought to contribute to pH-dependent anion exchange.

2.2.7 Preparation of Biochar

Any carbonaceous material could be used to create biochar. The strong adsorptive characteristics of these materials have attracted a lot of researchers. Carbonization is the way of transforming organic compounds to elemental carbon through a series of pyrolysis processes. Activation is then performed on the resulting char to achieve a desirable level of porosity (Bakkaloglu, 2014).

There two methods for creating biochar are one-step pyrolysis and two-step pyrolysis. Physical activation is a two-step pyrolysis process in which carbonization and activation occur separately, whereas chemical activation is a one-step pyrolysis method in which carbonization and activation occur simultaneously. In a two-step activation process, the carbonaceous raw material is first carbonised to generate carbonaceous char, then activated at a high temperature with a suitable activating agent (Hapazari, Ntuli, & Parawira, 2011). Carbonization takes place at temperatures between 400-800°C, whereas activation takes place at temperatures between 600-900°C (Cetin, Moghtaderi, Gupta, & Wall, 2004). However, such a procedure takes longer and consumes a lot of energy. Bio-char obtained by a single-step pyrolysis technique is a simple, quick, and effective way to obtain activated carbon from carbonaceous materials. It eliminates the need for a separate carbonization phase and uses less resources and costs than two-stage pyrolysis (Lee, Ooi, Othman, & Yeoh, 2014).

Meanwhile, Zubrik et al (2016) found that the two-stage pyrolysis technique is substantially more efficient than the one-step pyrolysis in a recent research on preparation of chemically activated bio-char from waste biomass by single-stage and two-stage pyrolysis. The volume of micropores in activated carbon produced by two-step pyrolysis is four times that of one-step pyrolysis. Furthermore, two-step pyrolysis activated carbon has improved physiochemical characteristics and a larger sorption capacity. In Table 2.2 summarises the differences between the one-step and two-step pyrolysis processes.

Table 2.2: Differences between the one-step and two-step pyrolysis processes

	One-step Pyrolysis	Two-step Pyrolysis
Diagram	<pre> graph TD A[Raw carbonaceous material] --> B[Activation (physical/chemical)] B --> C[Washing and Drying] C --> D[Activation Carbon] </pre>	<pre> graph TD A[Raw carbonaceous material] --> B[Carbonization in inert atmosphere] B --> C[Activation (physical/chemical)] C --> D[Washing and Drying] D --> E[Activation Carbon] </pre>
Type of process	Single stage process whereby carbonization and activation occurs simultaneously	Two stage process whereby carbonization and activation occurs separately
Cost	Low	High
Energy Consumption	Low	High
Duration	Short	Long
Characteristics	Modest physio-chemical properties and higher sorption capacity	High physio-chemical properties and sorption capacity

2.2.8 Pores of Biochar

The pores of biochar are generally divided into three clusters:

- Micropores with pore size less than 2nm;
- Mesopores with pore size between 2-50nm;
- Macropores with pore size more than 50nm

All three types of pore sizes has its own function in the adsorption process. Micropores determine the comprehensive adsorptive capacity of activated carbon due to its large surface area and volume. Under sufficiently high pressure, condensation takes place in mesopores. Mesopores also provide a pathway for the micropores to the outer surface. The amount of adsorbate engaged in mesopores are relatively

insignificant due to its smaller surface area. However, when the activated carbon is used as a catalyst support, it serves as a site for catalyst depositions (Madala.S, 2009). The most widely used activated carbons are microporous and have high surface areas, and thus, show high efficiency for the adsorption of low molecular weight compounds and for larger molecules. The pore size of the activated carbon enhances the removal efficiency. This is a distinct property. Besides, the pore-size distribution and the relative size of the adsorbate molecules have a significant effect on the adsorption capacity (Patel & Himanshu., 2018).

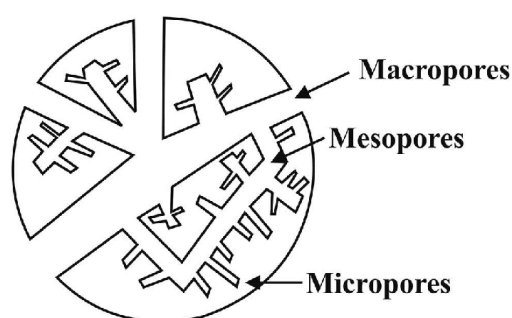


Figure 2.1: Schematic diagram of structure of activated carbon (source : Himanshu , 2018)

2.2.9 Carbon Activation Process

The quality of biochar is enhanced using a physical activation after carbonation process. The applications of activated carbon are numerous, from air cleaning to water treatment. Both processes of carbonation and activation could be done using microwave assisted process with temperature ranging up to 900 °C. The temperature in the reactor was closely controlled by a temperature controller. Studies show that chemical activation give higher BET surface areas using less MW power. Also, despite using lower MW power other parameters also contribute the development of the porosity in the activated carbon, such as activated time, temperature, impregnation ratio and agent (Farid, 2016).

Activation processes increase porosity, enhance surface areas and modify chemical properties by introducing a variety of surface sites such as carboxylic, phenolic, hydroxylic and carbonyl groups. The most important reason for the use of different techniques of activation is to increase a partially blocked porous surface by using agents to remove the trapped products of incomplete pyrolysis. For the chemical

activation of biochar KOH, ZnCl₂, NaOH, H₃PO₄ and K₂CO₃ impregnation can be applied with accompanied by high temperatures. By using this method, the hydrophilic properties of biochar can be improved and the N/C and H/C (aromaticity) ratio increased. Chemical activation with alkaline reagents, especially KOH, is widely used, providing materials with very high surface area and defined micropore size distribution. The complete text analysis of 81 articles was approved. Among the chosen articles, the number of articles based on the chemical activator applied for the manufacture of activated carbon from agricultural wastes is shown in Figure 2.3.

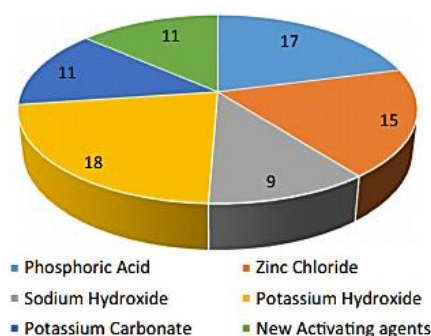


Figure 2.2: Number of articles based on the chemical activator used for the preparation of activated carbon from agricultural residues (source: Zoha, 2020)

Chemical activation is one of the most common methods for producing porous bio-char which involves doping char with a chemical agent, is the most common method for changing surface functionalities, but its mechanism is unknown. Chemical activation was accomplished by immersing or suspending a pyrolysis biochar in a chemical agent's solution at a ratio of up 1:5 as biochar: acid, base or salt) at room temperature up to 120 for a specified period of time. To achieve the desired adjusted bio-char, secondary thermal treatment may be used. Thermal treatments of functional group rich char at high temperatures fully carbonize the organic matter, resulting in the development of new nano pores and an increase in surface area. Then, the biochar was washed with acid or base and deionized water until the acidic/alkaline pH is neutralized otherwise the biochar's functionality and compatibility is jeopardized. Washing also gets rid of the chemicals left in the carbonized sample, resulting in the formation of new pores.

Chemical activation with alkaline reagents, especially KOH, is widely used to produce materials with a high surface area and a well-defined micropore size

distribution (Borhan et al., 2013). KOH and pyrolysis increased surface area, which increased Cd adsorption from a aqueous solution through surface complexation Hamid et al. (2014) provided additional evidence that Cu sorption on KOH activated bio-char was caused by chemical adsorption, as sorption kinetics match pseudo second order model and thermodynamics studies suggested a spontaneous endothermic method. Chemical reduction is often referred to as the alkali modification process. To increase the non-polarity of bio-char, the reducing agent was utilized to minimize functional groups on its surface (Yang et al., 2019). Meanwhile, chemical modification of bio-char will increase its porosity and precise surface area. Finally, biochar's adsorption capacity for pollutants is improved, especially for non-polar adsorbates.

The increased surface area caused by KOH modification of bio-char improves the adsorption of oxyanions from solution. The use of various reagents results in the formation of various surface functional groups. As a result, the chemical activation of bio-char can be categorized depending on either the chemical agents or the functionalities obtained. Surface oxidation using activating agents is the process normally used for creating oxygen-containing functional groups on the surface of bio-char. Carboxyl, phenolic hydroxyl, lactones, and peroxides are the most common oxygen functional groups formed on bio-char after oxidation. Among these groups, hydroxyl and carboxyl groups remarkably improve the adsorption capacity when bio-char is utilized for heavy-metal removal.

By adding a number of sites as mentioned previously such as carboxylic, phenolic, hydroxylic and carbonyls groups, chemical activation process increase porosity, increase surface areas and change chemical properties (Shen et al., 2010). The main reason to utilize various chemical activation techniques is to increase a partially blocked porous surface by using agents to extract trapped products of incomplete pyrolysis (Figueiredo et al., 2020). The chemical activation carried out in two different techniques which are acidic modification through chemical oxidation and alkaline modification through chemical reduction. Whereby, acidic modification is chemical activation by utilizing acid reagent while alkaline modification is chemical activation by utilizing alkaline reagent. Oxidation can be accomplished by using either a gas such as steam, CO₂, ozone, air, or NO, during pyrolysis or treatment with an acidic/basic solution.

2.3 Textile Dye Wastewater

2.3.1 Properties of Textile Dyes Wastewater

Dyes can be characterized as substances that, when applied to a substrate, produce colour by altering the crystal structure of the colour substances, at least temporarily. Textile, pharmaceutical, food, cosmetics, plastics, photography, and paper industries all use substances with significant coloring ability. By forming covalent bonds or complex with salts or metals, physical adsorption, or mechanical retention, the dyes may bind to suitable surfaces (Drumond Chequer et al., 201). Dyes are categorized based on their use and chemical composition, and are made up of chromophores, which are responsible for the dye's colour.

The functional groups azo, anthraquinone, methane, nitro, arilmethane, carbonyls, and others are used to create these chromophore-containing centres. Chromophores are the part of molecules which are responsible for colour of a particular dye as N=N- and pi-bonds while auxochromes are the part of molecules which provide support to chromophore for fix colour on the fiber and increase intensity of colour. Auxochromes are electrons that withdraw or donate substituents to produce or amplify the colour of chromophores. Auxochromes such as amine, carboxyl, sulfonate, and hydroxyle are the most common.

Over 10,000 different dyes and pigments are expected to be used in industry, with over 7×10^5 tons of synthetic dyes manufactured each year (Drumond Chequer et al., 2013). Batch, continuous dyeing processes are available for textile materials. The types of process used is determined by a number of factors, including the type of material, such as fiber, yarn, cloth, fabric construction, and garments, as well as the generic type of fiber, dye lot size, and dyed fabric quality specifications. The batch process is the most common method for dyeing textile materials among these methods.

2.3.2 Classification of Textile Dyes

The Colour Index contains information on over 8,000 different dyes (CI) and dyes are divided into two categories as like industrial application or dye end use, and chemical composition. The dyes are classified by their chromophore in terms of

chemical composition. The three sections of the CI generic names of dyes are recognized usage class, colour hue, and serial number.

The dyes are graded as follows based on their chemical composition:

- I. Azo dye
- II. Nitro dye
- III. Anthraquinone dye
- IV. Triarylmethane dye
- V. Indigo dye

The following is a list of dye classifications based on their industrial applications:

- I. Mordant dye
- II. Direct dye
- III. Disperse dye
- IV. Vat dye
- V. Ingrain dye

2.3.3 Toxicity of Textile Dyes Wastewater

Among the other chemicals in the textile industry that need to be eliminated, textile dyes are considered the most important pollutants. Every day, the textile dyeing industry generates a large volume of effluents, soil slurry, and solid waste ingredients. The textile industry is the biggest consumer of dyestuffs and industry-generated wastewater is one of the major sources of water contamination. Toxic dyes, organic matter, and heavy metals are all present in the wastewater. The metal contained in wastewater has been discovered to be carcinogenic. Because of their non-biodegradable complex chemical composition, dyes, used in the textile industry pose a pollution risk. Temperature, colour, sanity, COD, BOD and suspended solids are all strong in dye-laden wastewater.

Heavy metals like iron, lead, nickel, copper, zinc, and chromium are found in trace quantities in textile dyeing effluents. Synthetic azo dyes are carcinogenic and harmful to humans, posing a serious health risk (Islam & Goslan, 2018). These dyeing effluents are discharged into nearby waterways, farming areas, irrigation channels,

outside water, then at last into water bodies such as rivers and seas. Textile and dye industrial effluents can alter physical, chemical and biological nature of the aquatic environment by causing continuous change in turbidity, odour, noise, temperature, PH, and other that are harmful to human health, livestock, wildlife, fish and biodiversity. Along the process involved in textile manufacturing which is sizing, de-sizing, scouring, bleaching, mercerizing, dyeing, printing, and finishing technique all contribute to major contaminants in textile wastewater discharge (Yaseen & Scholz, 2018b).

Moreover, the Colour Index lists over 8000 chemical items related to the dyeing process, including many structural types of soluble dyes, which include acid, mordant, metal complex, direct, simple, and reactive dyes, can also be categorized as acid, mordant, metal complex, direct, basic, and reactive dyes. Azoic, sulfur, vat and disperse dyes are the examples of insoluble dyes (R Ananthashankar, 2013). Dyeing is the most complicated of the wet procedures, involving hundreds of dyes as well as auxiliary chemicals like fixing agents, acids and alkalis. The dyeing method primarily employs synthetic dyes. This method alone generates around half of the total amount of wastewater. Dyeing effluents are distinguished by their dark colour, strong BOD, suspended solids and dissolved solids. Dye effluents may contain chemicals that are teratogenic, radioactive, mutagenic, or carcinogenic (Suresh, 2015). Because of their complex aromatic molecular structures and synthetic nature, most dyes remain in the atmosphere for a long time and are difficult to degrade. As the amount of available water is limited due to contamination from both point and non-point sources from industry, problems arise. Dye-bath effluents has a difficult time removing its colour, when released into the water, it blocks light penetration in the water body, disrupting aquatic biological process.

Dyes in surface and subsurface water make them not only appealingly intolerable, but also a source of a variety of water-borne diseases. The usual functioning of cells is a disturbed and this, in turn, may cause an alteration in the physiology and biochemical mechanisms of animals are resulting in the injury of important function like respiration, osmoregulation, reproduction and even mortality due to the toxic nature of the textile dyeing effluent (Buthelezi et al., 2012). The textile materials can cause allergic reactions and it can also cause serious health problem exposed for an extended

period time, necessitating urgent care. Respiratory problems caused by dye particles inhaled are the most common. When such particles invade the respiratory tract, the body responds drastically, and the immune system can be harmed.

Concerns about the toxicity and carcinogenicity of textile dyes have piqued researchers' interest in their emission potential. This is due to the fact that many dyes are produced from established carcinogens like benzidine and other aromatic compounds, which can all be converted by microbial metabolism. It has also been said that azo and nitro compounds are reduced in sediments and the intestinal system, leading to the regeneration of the parent toxic amines. Azo dyes are described as having one or more azo bonds (-N=N-) in conjunction with one or more aromatic structures (Sarkar et al., 2017). They think it's important to have high photolytic stability and resistance to oxidizing agents. Chemical reduction of azo dyes can result in the formation of carcinogenic arylamines.

These dyes have been found to be carcinogenic and to be highly stable in terms of chemical and photochemical properties. Azo dyes are extremely toxic and can have both acute and long-term effects on species. They have the potential to degrade water sources, change soil physical and chemical properties, and damage the environment. The death of soil microorganisms caused by azo dye toxicity has a significant impact on agricultural activities. (Ventura-Camargo, 2015)

CHAPTER 3

MATERIALS AND METHODS

3.1 Introduction

The experimental methodologies, adsorbent, and textile dye wastewater preparation are all covered in this chapter. The precursor for biochar was palm oil mill sludge (POMS). The samples were taken from Felda Lepar Hilir 3, the first palm oil mill first effluent in Gambang, Pahang Malaysia. By using the biochar based palm oil mill sludge (POMS) utilized to treat textile wastewater and which the textile wastewater sample was collected from Tenun Diraja Pahang. They were then stored at temperatures of less than -20C to avoid bacterial contamination and the flowchart of the research methodology is illustrated below in Figure 3.1. To make a comparison at the end of the research, produced biochar was modified by chemical activation method using both acid and alkaline reagent. Aside from that, the use of biochar adsorbent in textile wastewater treatment is demonstrated by experimenting it for analysis as like chemical oxygen demand (COD), Total Suspended Solid (TSS), colour withal pH.

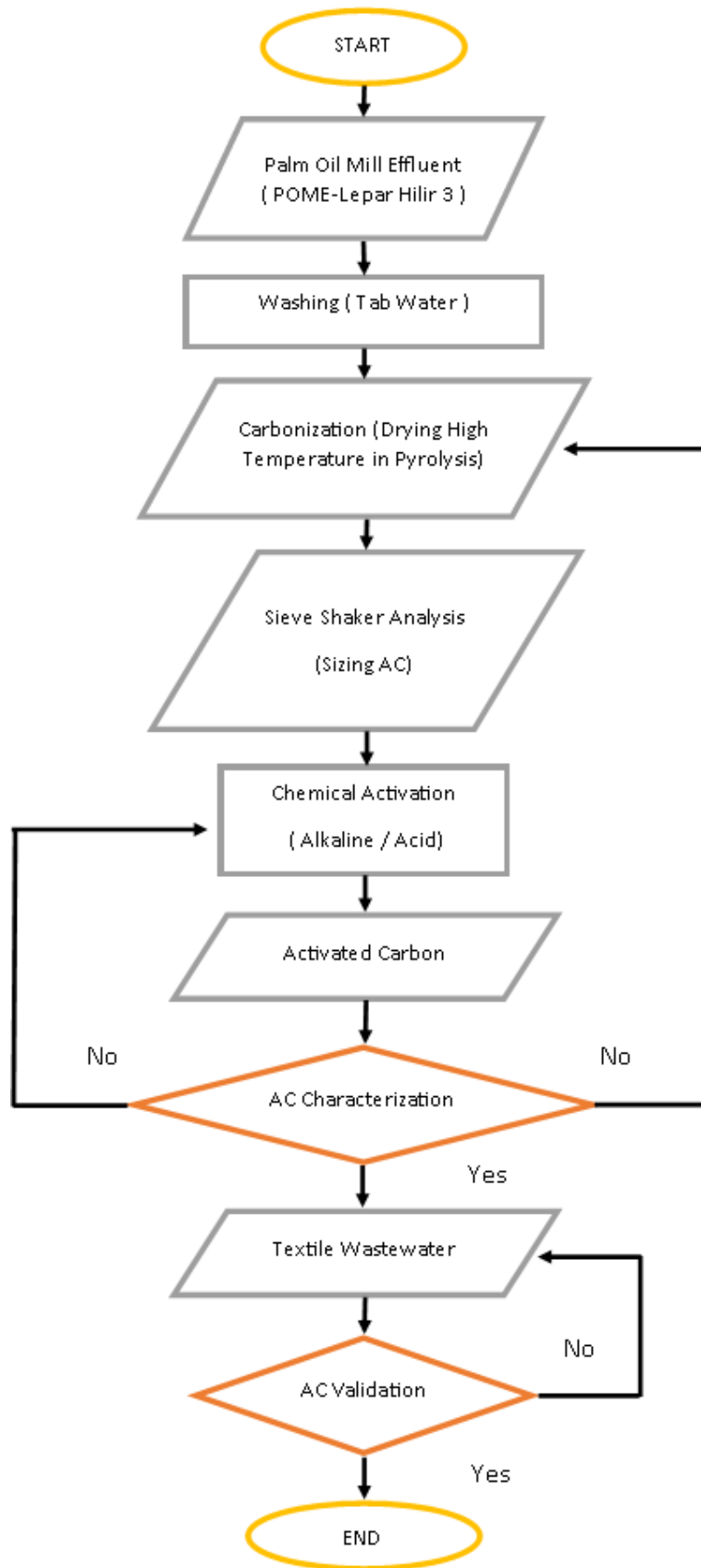


Figure 3.1: Flow chart of research methodology

3.2 Preparation of Activated Biochar

3.2.1 Carbonization of POMS

Palm oil mill sludge (POMS) was collected and washed thoroughly with distilled water to remove impurities and dirt on the fruits. To achieve a consistent weight and eliminate moisture from the content, the raw material from Palm Oil Mill Sludge (POMS) was dried at 105°C for 24 hours and the dried (POMS) was left outside to let it cool in room temperature. Before the carbonization procedure, the dry sludge was employed to perform chemical activation for about 24 hours. Then, it will washed thoroughly with distilled water before place in oven to dry it once again about 105C for 12 hours. Besides that, the dried sludge will then be carbonized at three different temperature start with 500°C, 600°C and 700°C in a furnace for 60 minutes to enhance the pore development for adsorption. The biochar from the carbonization process was left to cool down before being crushed into small pieces with a crusher and pestle. It was then sieved through 2 mm to have a homogeneous structure. The biochar was housed in an airtight polyethylene container and dried in a drying cabinet. At last, the resulting activated biochar utilized for testing with textile wastewater.

3.2.2 Chemical Activation of POMS

In chemical activation, the carbonized POMS was activated with Phosphoric acid reagent (H_3PO_4) and alkaline reagent Potassium hydroxide (KOH) as the activating agent. Prior the chemical activation the dry sludge (POMS) was derive equally to 3 different temperature sample which is 500C,600C and 700Cfor an activated biochar, same goes for (H_3PO_4) phosphoric acid based biochar and (KOH) potassium hydroxide based biochar. Afterwards, with the prepared solution of H_3PO_4 and also KOH the dry sludge is impregnated into relative solution based on impregnation ratio. Impregnation ratio that was used to combine the carbon formed with the activating agent solution (IR). The mass of impregnate in relation to the mass of the biochar is referred to as IR. The impregnation ratios (IR) employed in this experiment was 5:1. After that, the dry sludge (POMS) was impregnated with activating agent mixture for 24 hours. To eliminate any activating agent compounds, after 24 hours the mixture was well rinsed

with distilled water and the step was repeated for several time until the washing solution reaches a neutral pH value. Again, the impregnated sludge will be dried for another 24 hours at 105°C to produce a constant mass. Besides that, the sludge was undergone pyrolysis process to the dry sludge (POMS) into biochar based (POMS). The produced biochar through pyrolysis was crushed and sieved to a size of 2mm before being labelled as raw biochar, H₃PO₄ activated biochar and KOH activated biochar based (palm oil mill sludge) POMS.

3.3 Analysis of Textile Wastewater

3.3.1 COD Analysis

The textile wastewater's Chemical Oxygen Demand (COD) initial and after adsorption was measured using the HACH method 8000. The DRB200 reactor was preheated to 150 degrees Celsius. Using a clean pipette, 2mL of textile wastewater sample was added to the vial containing reagent. 2mL of deionized water was applied to the second vial with reagent to prepare the blank. The vials were tightly closed and gently inverted to allow the solutions to combine. They were then placed inside the preheated DRB200 reactor. The reactor lid was closed, and the vials were heated for 2 hours. After 2 hours, the vials were cooled in the reactor for around 20 minutes to a temperature of 120°C or less. After that, the vials were put in a test tube rack to cool to room temperature.

3.3.2 Colour Analysis

The initial colour of textile wastewater and the colour measurement after subjecting the biochar produced based palm oil mill sludge (POMS), was measured using spectrophotometer DRB 600. Two vials were prepared where one vial for zero which is filled with distilled water and the other want with textile wastewater. After measured colour for initial textile wastewater, same steps was repeated to measure colour after subjecting biochar into textile wastewater. For each sample the reading was taken for three times to make sure the data obtain is accurate.

3.3.3 Total Suspended Solid (TSS)

Total suspended solid are defined as solids in water that will be trapped by a filter paper applied in the middle of the vacuum flask. To measure initial total suspended solid (TSS) of the textile wastewater, the textile wastewater sample is filtered through a pre weighed filter paper. The residue retained on the filter paper was dried in oven at 105°C for 1 hour and the increase in weight of the filter paper represent the total suspended solid (TSS) exists in the textile wastewater. Again, the same procedure repeated for each sample biochar employed into textile wastewater.

3.3.4 pH Analysis

The initial and treated textile wastewater with biochar based palm oil mill sludge (POMS) of textile wastewater's pH was measured using potentiometric pH meter. The electrode tip was first rinsed with distilled water before placed into the surface of textile wastewater. It will take several minutes or seconds for it to calibrate the reading and as it indicates square root on monitor, the final reading was obtained. Same procedure maintained for the rest of sample for pH measurement.

3.4 Textile Dye Wastewater

3.4.1 Preparation and Characteristics

Textile wastewater contains dye combined with a number of pollutants in varying concentrations. The resulting activated carbon was used to treat textile dye wastewater with a high concentration of suspended solids. Textile wastewater is also a significant cause of contamination in surface waters. To treat these carcinogenic effluents, various methods have been used. They are, however, both inexpensive and inefficient in terms of energy consumption. As a result, adsorption using activated carbon has been investigated as one of the most promising treatment methods.

This experiment used wastewater obtained from Pusat Tenun Diraja Pahang to validate the efficacy of the resulting activated carbon. The samples were collected in order to exclude any suspended solids. The samples were transported in a 4L container

to the UMP laboratory for examination. To avoid contamination, the wastewater sample was held in a freezer at 20°C in the chemistry laboratory UMP. There were no contaminants applied to the wastewater sample that was collected. The wastewater sample was collected and treated in accordance with the standard operating procedures.

3.5 Adsorption

3.5.1 Batch Adsorption

For each biochar based palm oil mill sludge (POMS), nine conical flasks with varied temperatures of pyrolyzed biochar based POMS (500°, 600°C, 700°C for raw biochar, H₃PO₄ modified biochar, and KOH modified biochar) were used. Each conical flask had 200mL of textile effluent added to it, along with varying temperatures of biochar based POMS 500°C, 600°C, 700°C of raw and chemically activated biochar. Then, before mixing the textile wastewater with a glass rod, 10g of biochar-based POMS was added to each conical flask that already contained textile wastewater. Using an Orbital Shaker, the samples were shaken at 120 rpm for 5 hours at 20°C. The activated biochar samples were retrieved from the orbital shaker and left for 30 minutes to settle down. Following that, samples were filtered using filter paper and then evaluated for several water quality characteristics. Standard methods were used to test water quality parameters such as pH, COD (Chemical Oxygen Demand), TSS (Total Suspended Solid), and colour.

CHAPTER 4

RESULT & DISCUSSION

4.1 Introduction

The results are explained in detail and analysed in this chapter in order to achieve the study's main purpose. The features of activated biochar made from palm oil mill sludge (POMS) are investigated in terms of its ability to treat textile wastewater as an adsorbent. The activated biochar was tested for its ability to remove Total Suspended Solids (TSS), Chemical Oxygen Demand (COD), pH, and colour from textile dye wastewater in order to achieve standard (A) discharge quality.

4.2 Adsorption of Biochar Based Palm Oil Mill Sludge (POMS)

4.2.1 pH Analysis for adsorption of biochar based POMS

Biochar	pH Value
Raw 500°C	7.9
Raw 600°C	7.5
Raw 700°C	7.5
KOH 500°C	7.7
KOH 600°C	8.0
KOH 700°C	7.9
H3PO4 500°C	7.2
H3PO4 600°C	7.3
H3PO4 700°C	7.3

Table 4.2: pH of textile wastewater after subjecting biochar based (POMS)

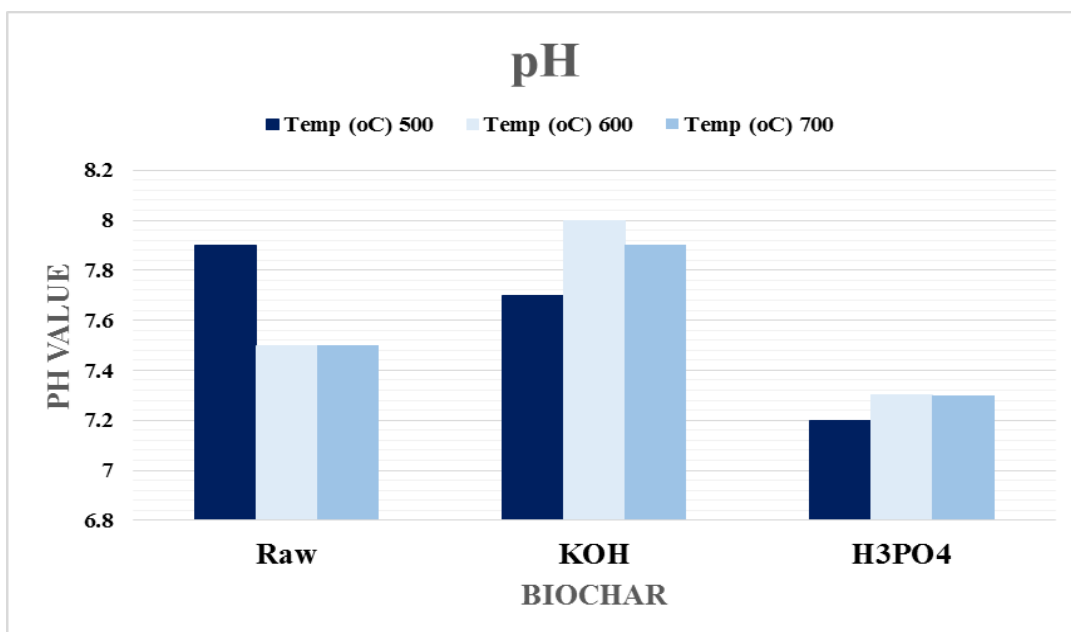


Figure 4.1: Ph vs Biochar

At first the initial Ph of textile wastewater was measured and obtain value of Ph about 8.4, as all sample of biochar mixed well into textile wastewater in different conical flasks. The after Ph was measured as all the data obtained have been presented on table 4.- above. Based on the result obtained biochar based POMS perform well with the Ph range of 7.2 until 7.5. This is because based on the result obtained for raw biochar based (POMS), (H_3PO_4) acid based biochar and (KOH) alkali based biochar performs efficiently as the value of Ph in range of 7.2 until 7.5. Other Ph value which is above 7.5 seems to be less competent for the adsorption of biochar based POMS. The ph range of 7.2 until 7.5 obtain for all 600°C raw, KOH, H_3PO_4 pyrolyzed biochar as well as for (500°C) H_3PO_4 , 700°C (H_3PO_4) and also raw (700°C). Overall, the Ph value of for all H_3PO_4 acid modified biochar based POMS falls in the Ph range of 7.2 until 7.5, and for raw biochar belongs to 600°C and 700°C also falls in the range. Additionally, all 600°C pyrolyzed biochar based POMS reduce high number total suspended solid (TSS) achieve in the range of Ph 7.2 until 7.5 while high number of COD reduction obtain with Ph range of 7.3 until 7.9 for all biochar belongs to 700°C. The optimum Ph for colour removal was 7.3 until 8.0 with all biochar pyrolyzed at 600°C. The Figure above shows the bar chart of Ph value belongs to raw, KOH and H_3PO_4 biochar based (POMS).

4.3 Performance of Biochar Based palm oil mill sludge (POMS) as adsorbent

4.3.1 Removal Performance of Chemical Oxygen Demand (COD)

Biochar	Chemical Oxygen Demand (COD)	Percentage of COD Reduction (%)
Raw 500°C	34	43
Raw 600°C	22	63
Raw 700°C	15	76
KOH 500°C	62	1
KOH 600°C	60	0
KOH 700°C	52	13
H3PO4 500°C	31	49
H3PO4 600°C	12	80
H3PO4 700°C	3	84

Table 4.3: COD of textile wastewater after subjecting biochar based POMS

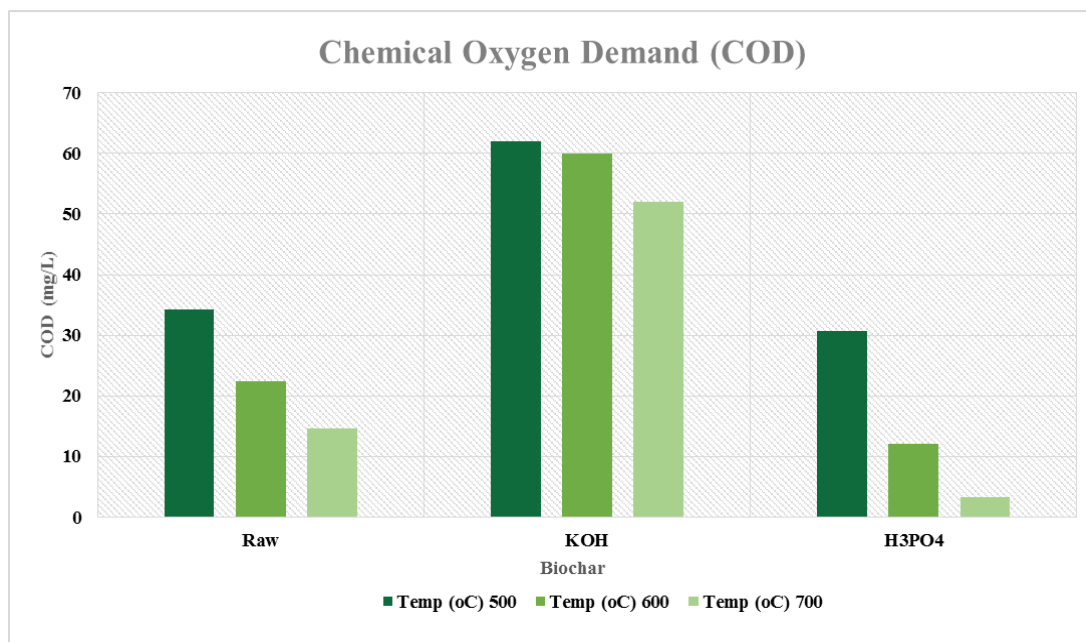


Figure 4.2: Chemical Oxygen Demand (COD) vs Biochar

The initial chemical oxygen demand (COD) of the textile wastewater was 60 mg/L and in the table above represent the data of COD reduction after subjecting the biochar based POMS into the textile wastewater. Based on the aftermath acquired for the COD reduction, biochar based POMS belongs to 600°C raw, (H₃PO₄) and (KOH) seems to achieve high number of COD reduction in the textile wastewater. Hereby, the highest performance percentage of COD reduction was 84%, 80% and 76% belongs H₃PO₄ (700°C), H₃PO₄ (600°C) and raw (700°C). To be conclude that the highest percentage performance is meet when employing biochar based POMS belongs to H₃PO₄ (700°C) compared to raw (700°C). KOH (700°C) seems not competent maybe there's no strong interaction between the biochar based POMS and also the chemical reagent employed which potassium hydroxide. That's what leads to poor COD reduction for KOH (700°C) compared to the H₃PO₄ (700°C) and raw (700°C). Again, it's a solid prove that phosphoric acid is very competent with biochar based POMS to be presented as activated biochar to treat textile wastewater treatment. Moreover, the raw biochar based POMS also performs efficiently in term of chemical oxygen demand (COD) reduction but it is just slightly lower in performance percentage of COD compared to H₃PO₄ based biochar.

4.3.2 Removal Performance of Total Suspended Solid (TSS)

Biochar	Total Suspended Solis (TSS) (mg/L)	Percentage of TSS Removal (%)
Raw 500°C	0.4101	25
Raw 600°C	1.6306	82
Raw 700°C	0.972	55
KOH 500°C	0.3834	28
KOH 600°C	0.5628	30
KOH 700°C	0.4149	24
H3PO4 500°C	1.0638	58
H3PO4 600°C	1.2418	68
H3PO4 700°C	0.7459	50

Table 4.4: (TSS) of textile wastewater after subjecting biochar POMS

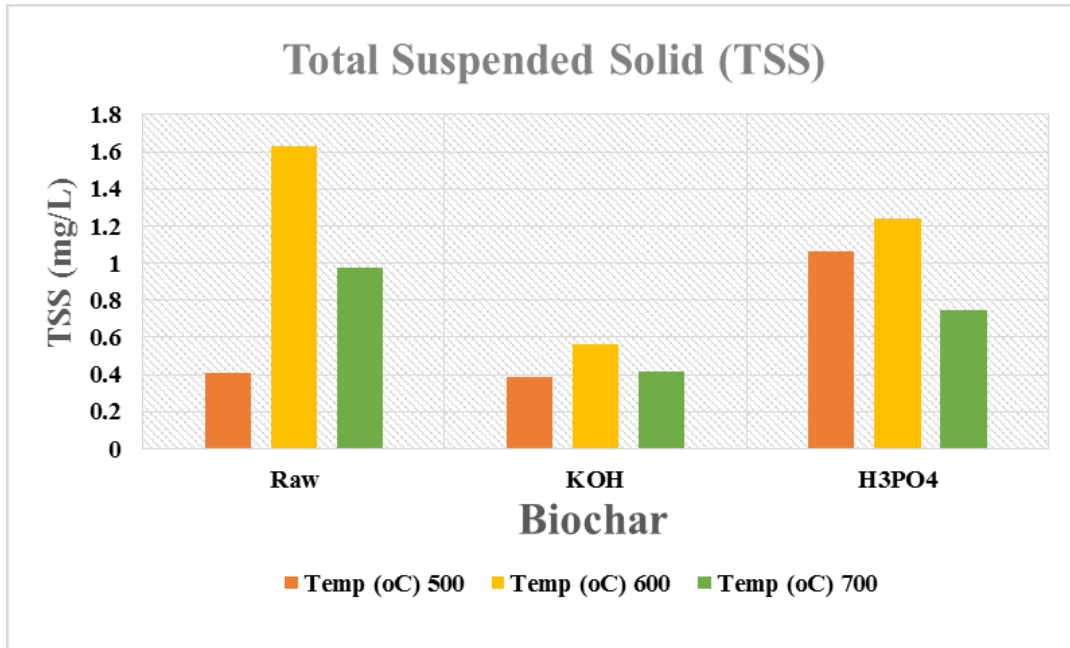


Figure 4.3: Total Suspended Solid (TSS) vs Biochar

The initial reading of Total Suspended Solid (TSS) was 0.1095 mg/L the same procedure was repeated to measure the total suspended solid (TSS) after subjecting biochar based POMS into textile wastewater. Based on the result obtained, all biochar based POMS belongs to 600°C performs wells to remove high number of (TSS) from textile wastewater. The highest percentage performance of TSS removal achieved by raw biochar based POMS modified about 82% and 68% by biochar modified by phosphoric acid (H₃PO₄). Biochar modified by potassium hydroxide (KOH) belongs to 600°C also able to achieve 30% TSS removal compared KOH (500°C) about 28% and 24% by KOH (700°C). For TSS removal, raw biochar based POMS carbonized at 600°C appear to be more productive compared to biochar modified by phosphoric acid (H₃PO₄) and potassium hydroxide (KOH) refers 500°C and 700°C. This is because biochar modified by phosphoric acid (H₃PO₄) refers to 600°C less effective compared to raw biochar of 600°C as mention earlier the raw biochar attained more percentage removal. Even so the biochar modified by phosphoric acid (H₃PO₄) still attained high number percentage removal of TSS 58% for 500°C, 68% for 600°C and 50% for 700°C thus its works but just less effective compared to raw biochar 600°C. Potassium hydroxide (KOH) modified biochar based POMS is not effective as the percentage removal of TSS in textile wastewater is low compared to raw biochar and also phosphoric acid (H₃PO₄) modified biochar.

4.3.3 Removal Performance of Colour

Biochar	Colour (pt/co)	Percentage of Colour Removal (%)
Raw 500C	99	32
Raw 600C	32	78
Raw 700C	122	16
KOH 500C	114	22
KOH 600C	83	43
KOH 700C	136	8
H3PO4 500C	28	81
H3PO4 600C	23	86
H3PO4 700C	71	52

Table 4.5: Colour of textile wastewater after subjecting biochar based POMS

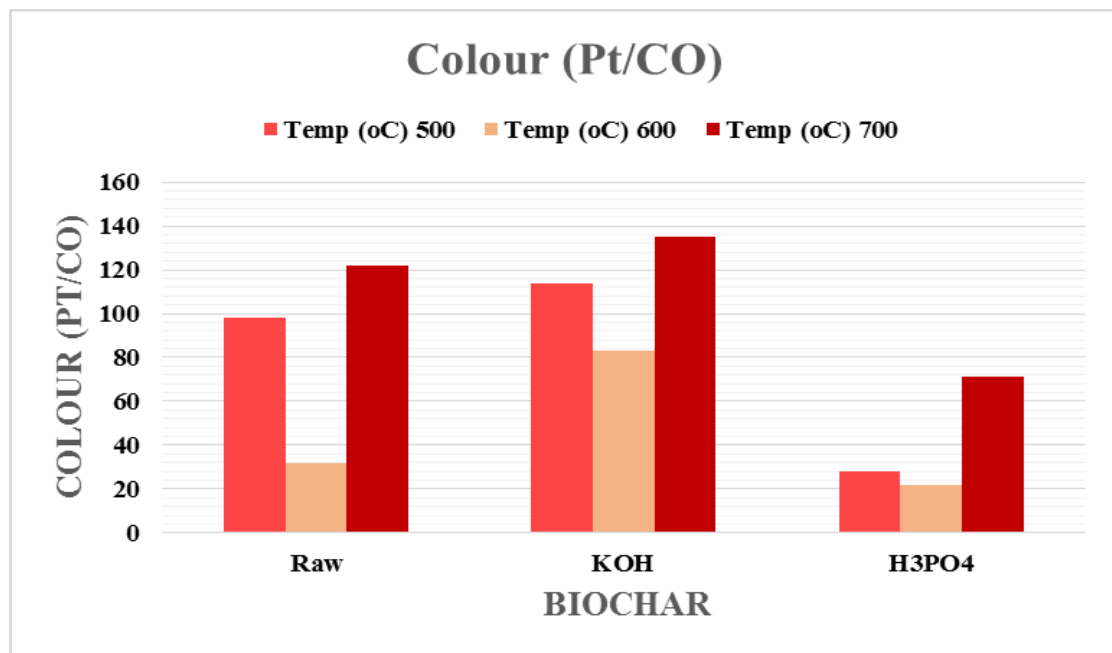


Figure 4.4: Colour vs Biochar

Before initiate the test for colour analysis after applied biochar based POMS into textile wastewater the initial colour of textile wastewater was measured which is about 145pt/co. After employing biochar based POMS high number of colour removal can be observed through the result obtained. Extensive removal of colour obtained for biochar based POMS 600°C belongs to raw, H₃PO₄ and also KOH. Raw 600°C reduce the colour by 32pt/co from its initial value of colour in the textile wastewater which is 145 pt/co. while H₃PO₄ (600°C) reduce to 23pt/co and 83% for KOH (600°C). From here, high performance percentage which is 86% was achieved as employing H₃PO₄ (600°C), 81% by H₃PO₄ (500°C), 78% by raw 600°C. Whereas, the 600°C biochar based POMS belongs to KOH seems to be less in performance percentage compared to H₃PO₄ and raw which is about 43%. This is because as already mention before this, KOH is not competent to be employed as a alkali based chemical reagent for chemical activation of biochar based POMS as the POMS itself is already a alkali based group thus when interacting alkali substance with alkali chemical reagent the performance is not efficient. H₃PO₄ biochar based POMS belongs to 500°C also performs in colour removal as its reduced to 28pt/co compared to initial colour of textile wastewater with 81% second highest percentage performance of colour adsorption compared to raw 600°C and KOH (600°C) with 32pt/co and 83pt/co.

CHAPTER 5

CONCLUSION

5.1 Introduction

The primary goal of this study is to create activated biochar from palm oil mill sludge (POMS). The manufacture of chemically activated biochar based POMS sludge based activated carbon was verified in this segment. Some suggestions for improving the efficiency of the activated biochar produced using POMS were also made.

5.2 General Conclusion

As can be seen from the literature reviewed above, biochar was an excellent choice to remediate wastewater dyes. Biochar formed in biomass hydrothermal processing would operate as an effective adsorbent after surface modification which can remove dyes from synthetic and also real industrial wastewater. Agricultural waste, algae biomass, sludge, plant residue, etc. for biochar production are most popular ways of pyrolysis and hydrothermal carbonization for the treatment of different types of feed stocks. The chemisorption mechanism exhibits most of the bio-char-based adsorption process. Because biochar not only has good adsorption performance and low price, but also can recycle and other excellent performance, it is widely used in water treatment. The application of activated biochar is not good enough, which shows that there is still a long way to go.

Biochar can be used either alone or in combination with other water treatment technologies. In order to maximize the adsorption capacity of activated carbon in water treatment, better service for water treatment, saving cost, reducing energy consumption and improving efficiency become a basic research criterion. In the modification of biochar, combined with microbial properties and microwave-activated carbon research, the application performance of activated carbon in future water treatment will be higher. After surface modification, biochar formed through hydrothermal processing of biomass would operate as an effective adsorbent that could remove colours from

synthetic and actual industrial effluent. For processing various types of feedstocks (agricultural waste, algae biomass, sludge, plant residue, etc.) for biochar synthesis, pyrolysis and hydrothermal carbonization have been the most popular processes. Chemisorption is present in the majority of biochar-based adsorption methods.

Activated biochar is made up of a variety of carbonised materials with a high porosity and surface area. It has several applications in water purification, home and industrial wastewater treatment, desalination, refining and separating gases, odour and pollution removal, and medical applications in many regions of the world due to its unique features. Activated biochar is now made from a variety of industrial wastes, activated biochar is activated in two ways: physically and chemically. Chemical activation is more cost-effective than physical activation because it requires a lower activation temperature, a shorter processing time, and a higher carbon efficiency. In addition, the growth of porous structures in activated carbon is larger when using the chemical activation method. The role of various activation parameters in the performance and efficiency of activated biochar produced from diverse precursors was investigated in this study. Because the cost of production and the adsorption capacity of various contaminants make activated biochar a significant tool in industrialised countries, choosing the appropriate activating agents in the manufacture of activated carbon is critical. The current study's discussion revealed that several activating agents increase the adsorption capacity of activated biochar. The use of phosphoric acid for activation results in reduced environmental and toxicological pollution.

In general, when using alkali materials for chemical activation, potassium hydroxide produces a better result in terms of surface area and performance in various applications. In addition, activated biochar with potassium hydroxide has a higher adsorption efficiency than other activators. As previously said, a variety of factors influence the activation of activated biochar, necessitating several studies to better understand the adsorption mechanism and increase the adsorption of pollutants utilising activated carbon generated on a global scale. The potential for employing biochar as an adsorbent in the future is enormous. Engineered biochar and selective tweaks will boost biochar's efficiency. Embedding metal frameworks produces catalyst-quality biochar, which can be employed as an inexpensive catalyst in a variety of sophisticated oxidation processes.

Furthermore, regeneration and reuse of used biochar adsorbents is a hot topic in biochar adsorbent research. There have been few research on the use of waste biochar as a soil amendment and additive in biological degradation processes. After being employed as adsorbents, biochar can be used as biocarriers. The problem of dye contamination in water is becoming more prevalent in most nations throughout the world, hence studies on biochar dye interaction will be vital.

5.3 Recommendation

- ✚ To discover the best optimum for producing the most effective activated biochar, a variety of activation time and temperature should be investigated.
- ✚ Analysis of leached elements from POME sludge should be performed to identify and predict components that may be released from the sludge-adsorbent, so that appropriate post-treatments may be applied to overcome the leached elements.
- ✚ Because contaminants in organic waste and accumulated solid impurities that are not removed by washing processes are responsible for ash production when heated in the presence of O₂, the carbonization and activation process should be carried out in an inert atmosphere.
- ✚ To get a lower particle size, the activated biochar must be sieved using a mechanical shaker.
- ✚ After chemical activation of biochar, further washing procedures should be applied to eliminate any contaminants and make it neutral.
- ✚ In order to extend the study to practical applications, it is critical to analyse the adsorption and kinetics behaviour of the adsorbates and adsorbents.
- ✚ Prior to any examination, the biochar should be dried at 105°C for 24 hours to remove any moisture.
- ✚ To obtain optimum result for adsorption the sample must be placed in orbital shaker must for about 12 hours this is because the adsorbent/ biochar applied will mixed into the wastewater surface.

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APPENDICES

Appendix A: Source of palm oil mil sludge (POMS) from Lepar Hilir.



Appendix B: Wash the raw material collected which is palm oil mill sludge (POMS).



Appendix C: Drying process of palm oil mill sludge (POMS) in oven.



Appendix D: Dried palm oil mill sludge (POMS).



Appendix E: Chemical activation of potassium hydroxide (KOH) and Phosphoric acid (H₃PO₄).



Appendix F: Wash chemically activated POMS with distil water.



Appendix G: Carbonization using furnace of (POMS) after chemical activation.



Appendix H: Carbonized palm oil mill sludge (POMS).



Appendix I: Crushing and sieving of carbonized palm oil mill sludge (POMS).



Appendix J: Collection of textile wastewater from Tenun Diraja Pahang.



Appendix K: After applying activated biochar based (POMS) into textile wastewater.



Appendix L: Overall colour adsorption of biochar based (POMS).



Appendix M: Ideal activated biochar based (POMS).

