SYNTHESIS OF TREATMENT TEXTILE WASTE WATER BY USING BIOCHAR BASED PALM OIL MILL SLUDGE (POMS) BY CHEMICAL ACTIVATION (POTASSIUM HYDROXIDE)

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SYNTHESIS OF TREATMENT TEXTILE WASTE WATER BY USING BIOCHAR BASED PALM OIL MILL EFFLUENT (POME) BY CHEMICAL ACTIVATION (POTASSIUM HYDROXIDE)

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ABSTRAK

Matlamat kajian ini adalah untuk mencipta karbon teraktif, produk bernilai tinggi, daripada enap cemar yang dijana oleh efluen kilang kelapa sawit (POME) menggunakan pendekatan pengaktifan kimia. Karbon teraktif telah digunakan dalam pelbagai aplikasi sejak penemuannya sebagai penjerap yang kuat dan berkesan. Ia biasanya digunakan untuk menghilangkan bau dan logam berat tertentu daripada air, air sisa, dan larut lesap. Ia murah dan boleh diakses secara meluas di pasaran. Oleh kerana strukturnya yang sangat mikroliang, tahap kereaktifan permukaan yang tinggi, dan luas permukaan zarah yang besar, ia boleh disesuaikan dan praktikal untuk membuang bahan cemar daripada larutan akueus. Enap cemar yang dihasilkan oleh kilang dikumpul dan diubah menjadi karbon teraktif. Untuk membuat karbon teraktif, enap cemar POME digunakan sebagai permulaan. Ciri fizikal karbon teraktif ditentukan oleh proses pengaktifan dan parameter proses. Enap cemar diikuti dengan pengkarbonan dan pengaktifan dengan karbon dioksida, CO2. Pengkarbonan karbon pada mulanya dijalankan dalam relau pada 500°C, 600°C dan 700°C selama 60 minit untuk menukar enap cemar menjadi bahan berkarbon tinggi. Karbon yang tercipta selepas itu diaktifkan menggunakan pendekatan fizikal untuk menghasilkan karbon teraktif dengan keliangan dan keberkesanan yang tinggi. Sifat permukaan dan kandungan unsur karbon teraktif yang diperoleh dicirikan. Selepas menggabungkan penjerap dan air sisa dalam kelalang kon dan digoncang pada 150 rpm selama 15 minit, penjerap enap cemar digunakan untuk merawat air sisa pewarna tekstil. Keputusan menunjukkan bahawa enap cemar POME mungkin digunakan sebagai pilihan penjerap yang menjanjikan untuk penyingkiran pepejal terampai daripada efluen pewarna tekstil.

ABSTRACT

The goal of this study is to create activated carbon, a high-value product, from sludge generated by palm oil mill effluent (POME) using a chemical activation approach. Activated carbon has been used in a variety of applications since its discovery as a powerful and effective adsorbent. It is commonly used to eliminate odour and certain heavy metals from water, wastewater, and leachate. It is inexpensive and widely accessible in the marketplace. Because of its very microporous structure, high degree of surface reactivity, and large particle surface area, it is adaptable and practical for removing contaminants from aqueous solutions. The sludge produced by factory is collected and transformed into activated carbon. To make the activated carbon, POME sludge was employed as a preliminary. The physical characteristics of activated carbon are determined by the activation process and process parameters. The sludge was followed by carbonization and activation with carbon dioxide, CO2. Carbonization of the carbon is initially conducted in a furnace at 500°C, 600°C and 700°C for 60 minutes to turn the sludge into a highly carbonaceous substance. The carbon that is created after that is activated using a physical approach to produce activated carbon with a high porosity and effectiveness. The surface properties and elemental contents of the activated carbon obtained are characterised. After combining the adsorbent and wastewater in a conical flask and shaking it at 150 rpm for 15 minutes, the sludge adsorbent was utilised to treat textile dye wastewater. The results indicate that POME sludge might be used as a promising adsorbent option for the removal of suspended solids from textile dye effluent.

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LIST OF ABBREVIATIONS

AC	Activated Carbon
CS	Carbonised Slugde
COD	Chemical Oxygen Demand
DS	Dried Slugde
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
DOE	Department of Environmental

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

According to worldwide demand, palm oil is the world's second biggest vegetable oil. In Malaysia's tropical environment, palm oil is a natural resource that grows. Malaysia has 454 palm oil mills in 2017, with a combined output capacity of 112 million tonnes of fresh fruit bunches. It is the world's second-largest producer of the substance, behind Indonesia. Malaysia produces 39 percent of all palm oil in the world and exports 44 percent of it (Begum, 2019). In 2004, palm oil replaced olive oil as the world's most significant oil product in area and production, trade, and consumption. According to the Food and Agriculture Organization of the United Nations (FAO), oil palm planting area in Southeast Asia accounted for around 71% of global oil palm planting area in 2017 (FAO, 2019). POME contain 95–96% water; 4–5% total solids, of which 2–4% are suspended solids; and 0.7–0.9% residual palm oil. They have high chemical and biological oxygen demand because of lignocellulose and hemicellulose components in the oil-palm fresh fruit bunches (FFB) (S.N.H Abu, 2020).

On the other hand, continuous growth of oil palm plantations not only results in significant changes in land use types, but also represents a substantial danger to the environment, including decreased biodiversity in forest ecosystems, increased greenhouse gas emissions, and water pollution (Li, 2019). 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 POME into the environment will result in serious consequences such as groundwater pollution, methane release, and an unwanted odour into the atmosphere. (Chavalparit, 2006).

In the manufacture of carbonaceous adsorbent, any carbonaceous substance might be employed as a raw material. The high carbon concentration of palm oil sludge makes it perfect for making carbonaceous adsorbents. Biochar generated from POME sludge may be used to minimise a number of pollutants in water. Biochar is one of the most studied compounds for dye adsorption in textiles. Biochar is both a cost-effective and a highly efficient material (Li, 2019).

Along with its microscopic particle sizes and high adsorption capacity, biochar is the most effective adsorbent for the treatment of domestic, municipal, agricultural, and industrial wastewater. In the published literature, there is no mention of using POME sludge cake as activated carbon to treat dye waste water. As a result, the goal of this research was to characterise the potential utilisation of POME sludge-activated carbon. Physical and chemical activation is used to create activated carbon from palm oil mill effluent sludge cake. Palm oil mill effluent was converted into activated carbon using a two-step process, representing an abundantly useable oil palm waste in Malaysia. The surface characteristics of the activated carbon produced, as well as the elemental and proximate concentrations, will be carefully examined. The influence of experimental variables like interaction duration and temperature on the sorption experiment was also investigated. Moreover, biochar is porous, and it has a large surface area and a variety of functional groups on the surface (Banu, 2021)

1.2 Problem Statement

Malaysia is the world's top producer and exporter of palm oil, producing 10.55 million tonnes in 1999, corresponding to 54 percent of global production, and more than doubling production to 21.8 million tonnes in 2000. Half of the world's palm oil production, or 10.8 million tonnes, is produced in Malaysia, making it the world's top producer and exporter of palm oil during this time period. On 4,500,000 hectares of land utilised for palm oil planting, Malaysia produces 17.7 million tonnes of palm oil. Palm oil has shown rapid and consistent expansion in the worldwide market over the last four decades, with average annual output in the nation expected to reach 15.4 million tonnes between 2016 and 2020.

The oil palm sector is directly related to the country's ecosystem, economics, and agriculture. In terms of the environment, it offers a number of benefits, but it also has certain problems, such as the fact that oil palm mills contribute considerably to destruction of the environment. As a result of its production and processing activities, the oil palm mill greatly contributes to environmental degradation by generating vast amounts of industrial waste, wastewater, and air pollution. Solid wastes (PKS) include empty fruit brunch (EFB), mesoscarp fruit fibres (MF), and palm kernel shells, while liquid waste is formed when palm oil is extracted in a decenter. Palm oil mill effluent is created when this liquid waste is combined with waste from the cooling water and sterilizing (POME). The stench discharged into the environment as a result of POME digestion pollutes the air in the lagoon area (Abdullah & Sulaiman, 2013).

The presence of these wastes from oil palms has generated a significant disposal problem as waste recycling and minimization, energy recovery, waste disposal are the fundamental concepts of waste management. To achieve the best solution for using oil palm wastes, a combination of technical, economic, energy balance, and environmental considerations must be maintained since the residues cannot be disposed of without adequate care while a more cost-effective option exists (Abdullah & Sulaiman, 2013).

Total dissolved solids and suspended solids are typically high in textile industry effluent. The wastewater is commonly coloured and viscous due to dyestuff and suspended particles (Yaseen, DA and Scholz, M, 2018). The wastewater must be adequately treated in order for the effluent to be safely released, and textile wastewater contains not only colour pigment, but also other metallic particles, a significant pH fluctuation, and suspended solids with a high chemical oxygen demand (COD). The discharge of effluents from these applications is one of the possible sources of pollution and waste to the atmosphere, and these impurities in water are not only unpleasant, but also damaging to organic species, humans, and ecosystems.

Due to the presence of dye and additives used during the textile manufacturing operation, there is metal contamination in textile effluent. The most frequent metals identified in dye chromophores in textile effluents are cobalt, copper, and chromium (Yaseen, DA and Scholz, M, 2018). The principal metals that create environmental concerns include chromium, zinc, iron, mercury, and lead. Textile wastewater pollution prohibits biological processes from completing their societal responsibilities and reactions that occur natural maintainability. The textile sector has been pushed to regulate its effluents due to increasingly severe environmental rules (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," 2003).

Adsorption is a physiochemical wastewater treatment method in which wastewater is mixed with a porous substance such as activated carbon or biochar, allowing it to flow through a granular filter bed. Because of the high efficiency of activated carbon and their established effectiveness in removing organic and mineral contaminants, as well as economic concerns, adsorption is an useful solution for eliminating toxin from wastewater. Adsorption is a low-cost, practical technology that produces a best performance, and it has lately gained favour due to its effectiveness in eliminating dangerous particles released during the conventional process. For water purification and separation analysis, it is now commonly recognized as a reliable and cost-effective method. It's a wonderful colour removal procedure that requires no additional equipment other than the adsorbent and is very uncomplicated to apply.

1.3 Objectives

The purpose of this research is to make biochar from palm oil mill effluent sludge (POME). The particular objectives listed below have been determined :-

- Produce bio-char based palm oil mill sludge cake through chemical activation and pyrolysis process
- Examine the effectiveness of raw biochar and alkaline biochar.
- Able treat textile wastewater effluent with the biochar based POME sludge.

1.4 Scope of Project

The raw material palm oil mill sludge (POMS) was obtained from Lepar Hilir 3 Oil Palm Mill and was used as a biomass raw material for mass production of bio-char through pyrolysis and manufacture of bio-char by chemical activation technique in this study. While a sample of textile wastewater was collected from the nearest location, 'Tenun Pahang Diraja.' The tests were carried out in a muffle furnace with a nitrogen environment flowing through it and a different temperature range.

Following that, the effluent of textile wastewater was used as an adsorbent to prove the efficacy of bio-char. Refer to the previous testimony, by the end of test made a comparison between the raw bio-char, acid based bio-char and also alkaline based bio-char. The effect of adsorbent dosage on total suspended solid, COD, colour and the investigational specific was important in determination of the adsorption effectiveness of both raw and alkaline based bio-char.

1.5 Significant of Study

Palm oil production is critical to Malaysia's economy, since the country is the world's second-largest producer after Indonesia, and palm oil mill effluent (POME) is the principal pollutant produced in Malaysia's palm oil mills. If no effective governance is in place, these situations will result in a variety of environmental pollutants and problems, since they will certainly become the major source of air and water pollution in the future. This is because, despite having high biochemical oxygen demand (BOD) and chemical oxygen demand (COD), POME includes rich nutritional, organic, and carbon content.

As a solution to the stated problem, the sludge in the (POME) is used as a raw material for the production of bio-char, which is used in a variety of processes. Agricultural waste, animal manures, and paper products are the most common feedstocks for bio-char production. The utility of these wastes in the production of bio-char is a constructive process for converting waste into a useful and valuable commodity. This study assesses the environmental, economic, and sociological implications of pyrolysis to produce bio-char based palm oil mill effluent (POME).

Aside from that, bio-char eventually helps the environment by absorbing major contaminants in the water and soil. Many organic pollutants are being cut off as a result of the use of bio-char, which will gradually change the consequences on the environment. Bio-char functions as a crucial binding phase for many organic contaminants in the environment due to its resistance to microorganisms and excellent sorption attraction.

The use of pyrolysis to create bio-char for treatment purposes might considerably assist the country's economy because it involves less money and energy than commercial treatments. Furthermore, the design's simplicity, insensitivity to hazardous pollutants, and lack of creation of dangerous compounds during the treatment process make it particularly attractive.

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 bio-char 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.

As a consequence, this research gives information and references on how to make bio-char from palm oil mill sludge (POMS), and the techniques and outcomes may be used as a guideline and direction for further research & innovation.

CHAPTER 2

LITERATURE REVIEW

2.1 Pyrolysis

Biomass, as a practical energy source, is one of the alternatives to fossil fuels that may be used directly or indirectly through thermal treatment. Thermal decomposition techniques such as pyrolysis and gasification were created to transform organic matter into a range of products such as gases, oils, and chars (Zhang, 2010). Biomass is thermally decomposed in oxygen-depleted conditions at temperatures ranging between 300 to 1200°C during pyrolysis. When compared to gasification and combustion processes, this temperature range is viewed as a major benefit (Pawel, 2017).

Biochar is the solid residue of biomass pyrolysis and it is characterized by high amount of carbon in aromatic form (de Caprariis et al, 2015). When properly activated, this material presents sorption capacity and surface area similar to that of activated carbon. The activation procedure aims to improve the biochar characteristic needed in the adsorption process

Pyrolysis is a prominent thermo-chemical primary treatment for converting biomass into biochar. Biochar's physical, synthetic, and basic qualities are highly dependent on the source material and pyrolysis temperature. Biochar is produced as a result of insufficient ignition and has a unique aromatic quality that makes it stable in nature (Kumar, 2020).

Attributed to relatively large surface area and capacity to modify pore size and distribution throughout the manufacturing process. Adsorbents made from waste biomass are useful for a variety of applications, including gas separation and purification, water treatment, energy storage, and catalysis (Tan, 2017).

2.2 Biochar

2.2.1 Biochar Production.

Palm oil wastes are one of the most prevalent agricultural biomasses. Biochar, a black carbon-rich substance produced by pyrolyzing organic biomass under oxygen-limited condition, is universally recognized as a cost-effective and ecologically sustainable sorbent for pollutant removal in water and soil. The physicochemical parameters of biochar (surface area, high cation exchange capacity, and surface functional groups), which are highly dependent on the biomass source and pyrolysis conditions, are proven. Because of their massive abundance and flexibility of availability, macroalgae have recently been adopted as a precursor for the production of biochar. Furthermore, transforming biomass to biochar as one possible end-use will improve macroalgal resource consumption, especially for 'pest' species (green tide algae) that have rapid growth and environmental tolerance in eutrophic environments (Chenghu Y, 2021).

Along with its abilities to absorb contaminants onto it and into its surface through chemical and physical processes, adsorption has emerged as the most promising technology for eliminating landfill leachate pollutants. It possesses a porous structure, low acid/base reactivity, thermostability, strong surface reactivity, and a low cost of inclusion. Commercially available adsorbents are expensive and mostly come from non-renewable resources such as coal, so using traditional materials like agricultural waste or biomasses that are readily available in large quantities locally, such as bamboo, pecan shell, oil palm fibre, and sugarcane bagasse, could be the best alternative. These materials may be chemically and physically transformed to create low-cost adsorbents.

As a commonly accessible substance, biochar has great potential of pollutant adsorption. Biochar has been proven to be beneficial for a number of functions, including storing carbon, nutrient storage, and water content. However, its insufficient porosity as limits its application in water treatment. When activated at a high temperature (900°C) and in the presence of particular chemicals (H3P04, KOH) and gases (CO2, steam), biochar increase pores growth by selectively gasifying carbon atoms. The physicochemical activation technique is ideal for making extraordinarily porous materials. In addition, the porosity of the final materials can be strongly influenced by the morphological and chemical structure of the feedstock, as well as the pyro-gasification working conditions for biochar formation.

Porosity and several functional groups connected to the structure of activated biochar determine its efficiencies as an adsorbent, both of which are developed during activation. The impacts of several activated biochar on the treatment of organic and inorganic contaminants in water are investigated in the present study. The results show that high aromaticity and porosity are required for organic contaminant sorption, while the presence of oxygen-containing functional groups and an optimal pH are required for inorganic contaminant sorption, particularly metal sorption. Finally, while activated biochar appears to be a promising option for treating contaminants in water, more research is needed to assess its performance with real effluents containing contaminants of emerging concern (Flavia, 2018). Hu Zian discovered that activated carbon is an excellent adsorbent in water treatment and is a green, pollution-free substance in and of itself. It has good purifying ability due to its loose porous environment and high specific surface area (Hu Z.A, 2018)

Activated biochar is a carbon-based amorphous material that occurs in powdered or granular form and has an amorphous shape. It is made through carbonization and activation, resulting in a high level of porosity and an enhanced inter-particulate surface area. Biochar that has been activated is an excellent non-polar adsorbent. It may be used with a range of substances to adsorb diverse qualities of wastewater due to its large specific surface area and micropore, high adsorption performance, and recycling. The structure, adsorption process, modified activated carbon, microbial bound activated carbon, microwave bound activated carbon, and future research directions of activated carbon are all covered in this study.

2.2.2 Raw Materials for Production of Biochar

Waste biomass, palm oil waste, coconut shell, and other low-cost materials and biomass can all be used to make activated biochar. Previously, raw resources such as wood, peat, and vegetable waste were extensively used. However, a number of low-cost and effective alternatives are now being researched. Various agricultural and industrial wastes are being researched as biochar source production (Ao et al., 2018).

The nature of the pyrolyzed precursors and the type of activation procedure affect the char's ultimate surface characteristics and functions. In order to manufacture effective and low-cost products with high sorption capabilities, a variety of raw materials as well as ways to increase char qualities were examined. Agricultural residues, rice husk, palm shells, coconut shells, apple and cherry pulp, plum pulp and stones, and olive stones, among others, were used to create chars. Adsorbents derived from waste biomass are effective for a variety of applications, including gas separation and purification, water treatment, energy storage, and catalysis, due to their large surface area and ability to control pore size and distribution throughout the manufacturing process.

2.2.3 Classification of Biochar

Activated carbon is classified into three types based on particle size and shape: granular, power, and extruded activated carbon. Different types have a specific list of standards and functions. Activated carbon powder (PAC) is made up of tiny granules or powders with an average diameter of 15-25 m and a size of less than 100 m. Its apparent density ranges from 23 to 46 lb/ft2. PAC has a large internal surface area and a short diffusion distance, as seen by this feature. Crushed or ground carbon particles from PAC will be separated into certain mesh sizes using mesh sieves or sifters. PACs are generally utilised for liquid phase adsorption. PAC provides a number of benefits, including lower processing costs and more effectiveness.

Granular activated carbon (GAC) has a larger particle size range over PAC, ranging from 200 to 5000 microns, resulting in a smaller external surface. They're also more durable and long-lasting than PAC. With it's high kinetics of adsorption and quick diffusion rate, it is suited for most gases and vapours adsorption. GAC comes in two different forms: granular and extruded. 8x20, 20x40, or 8x30 are the most frequent sizes for liquid phase applications, whereas 4x6, 4x8, or 4x10 are the most common sizes for vapour phase applications. The most often used sizes are 12x40 and 8x30, which offer a good balance of size, surface area, and head loss. GAC is also renewed and recycled on a regular basis. Only 5-15 percent of the materials are wasted during the reaction (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.

By employing a blender and sieve, the dried oil palm was crushed to a particle size of 0.3 to 1.18 mm (KJ Lau, 2021). According Banu, biochar samples made from date palm fronds and leaves were crushed and sieved to a particle size of around 0.15 mm and being ground and sieved to 2 mm in size (Izzudin, 2021).

2.2.4 Chemical Structure of Biochar

Activated carbon features crystal and pore structures on the inside, as well as chemical structure on the outside. The adsorption efficiency of activated carbon as well as the physical (pore) structure of activated carbon are both affected by the chemical structure of the surface. During the manufacturing of activated carbon, the edge chemical bond of the aromatic sheet formed during the carbonization process breaks, resulting in an edge carbon atom with unpaired electrons. These marginal carbon atoms have unsaturated chemical bonds and can react with heterocyclic atoms like oxygen, hydrogen, nitrogen, and sulphur to form a variety of surface groups. Without a doubt, the existence of these surface groups has an influence on the adsorption properties of activated carbon. On activated carbon, there are acidic, alkaline, and neutral surface groups (Jiang, 2017). Chemical changes are introduced to biochar during the pyrolysis at various temperatures. As the biomass gets dehydrated, aliphatic bonds are converted into aromatic bonds which are consolidated into stable graphene structures (Banu, 2021).

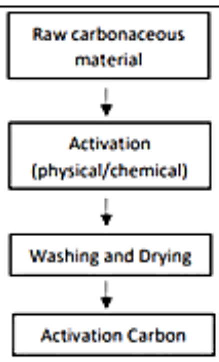
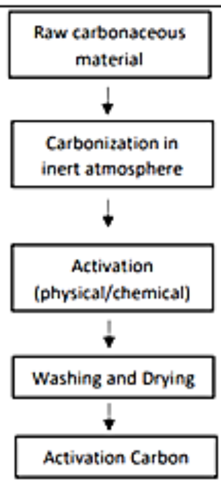
2.2.5 Preparation of Biochar

Biochar might well be made from any carbonaceous material. Many researchers have been attracted to these materials because of their high adsorptive properties. Carbonization is the method of converting organic molecules to elemental carbon is through a sequence of pyrolysis reactions. The resultant char is then activated to produce the desired amount of porosity (Bakkaloglu, 2014).

One-step pyrolysis and two-step pyrolysis are the two procedures for producing AC. Chemical activation is a one-step pyrolysis procedure in which carbonization and activation produce the same results, whereas physical activation is a two-step pyrolysis process in which carbonization and activation take place separately. The carbonaceous raw material is first carbonised to produce carbonaceous char, then activated at a high temperature with an appropriate activating agent in a two-step activation process. Carbonization takes place at temperatures between 400-800^{o}C, whereas activation takes place at temperatures between 600-900^{o}C (Cetin, Moghtaderi, Gupta, & Wall, 2004). However, such a technique is time-consuming and power. A single-step pyrolysis methodology for obtaining activated carbon from carbonaceous materials is a simple, fast, and effective way to acquire activated carbon. It consumes less resources and expenses than two-stage pyrolysis since it does not require a second carbonization process. (Lee, Ooi, Othman, & Yeoh, 2014).

In a recent study on Preparation of Chemically Activated Carbon from Waste Biomass by Single-stage and Two-stage Pyrolysis, Zubrik et al (2016) discovered that the two-stage pyrolysis approach is even more efficient than the one-step pyrolysis technique. Two-step pyrolysis produces four times the volume of micropores in activated carbon than one-step pyrolysis. Furthermore, activated carbon from two-step pyrolysis has better physiochemical properties and a higher sorption capacity. The differences between the one-step and two-step pyrolysis methods are summarised in Table 2.1.

Table 2.1 summarises the differences between the one-step and two-step pyrolysis processes

	One-step Pyrolysis	Two-step Pyrolysis
Diagram		
Type of process	Single stage process whereby carbonization and activation occurs simultaneously	Two stage process whereby carbonization and activation occurs seperately
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

The impacts of feedstock and pyrolysis temperatures on the removal efficiencies were studied, as well as the physical and chemical characteristics of date palm leaf/frond biochar formed at varied pyrolysis temperatures 400°C, 500°C, and 600°C (Banu, 2021). Palm oil empty fruit brunch was carbonized using a tightly closed muffle furnace to provide a low oxygen atmosphere at 450°C for 30 min (Izzudin, 2021). Other journal, the raw materials POMS utilised in this study were created in a wastewater treatment plant for the palm oil sector in Chumphon Province, Thailand. The original material was sun dried for around five days before being pyrolyzed for two hours at 500°C (Kalasee, 2021).

Temperature has the greatest impact on biochar quality. The optimal temperature for successful biochar synthesis, according to Spokas et al., (2012), is between 300°C and 700°C, which is within the slow carbonization process. Free radical production and water evaporation occur at temperatures below 350°C. When the temperature is between 350°C and 450°C, glycosidic connections in polysaccharides are broken by replacement, and when the temperature is over 450°C, dehydration, rearrangement, and fission of sugar units occur (Jahirul et al., 2012). The lignin component of biomass begins to decompose at temperatures above 600°C, reducing biochar output. Biochar produces liquids and gases at temperatures above 700°C.

Table 2.2 Current carbonization technologies for oil palm biomass and its biochar

Carbonization type	Biomass source	Combustion temperature (°C)	Sample size	Reactor capacity	Exhausted air flow rate	Retention time	Calorific value (MJ/kg)	Char yield (%)	Reference(s)
Two Fixed bed reactor	oil palm stone char/palm kernel cake char	500 -700 °C	6 - 10 mm	150 -200 g	618 – 1484 kg/m² kg	92 min	27 - 28	18-30	(Razuan et al., 2010)
Fluidized bed bench	EFB char	450	300-355 μm	-	-	-	-	26	(Abdullah et al., 2010)
	EFB fibers	110		18.66	-	-	18.66	-	
Pilot kiln	EFB char	600	-	-	-	-	-	24.8	(Hooi et al., 2009)
Kiln reactor	Palm Kernel	400-600	-	-	-	-	-	-	(Elham, 2001)
Tube Furnace	palm long fibre, palm shells and palm stone	850	0.5-1	-	-	3.5 hrs	-	29.5	(Lua and Guo, 1998)
Fluidized bed bench	oil palm stone	850-950	5 mg	-	791-1187 kg/m² hr	-	-	-	(Razuan et al., 2011)
Microwaves carbonization	EFB	600-900	-	-	-	-	Pitch-14, Branch-18	-	(Omar et al., 2011)
Fluidized bed carbonization	EFB fibre	400	-	0.4kg	-	2-5s	40	-	(Xu et al., 2011)
Quartz fluidized fixed bed	EFB fibre	300	91 – 106 um	2g	-	10 min	42	-	(Sukiran et al., 2009)
Muffle furnace	OPEFB	300-500	< 1.8 mm	-	-	-	18.46	-	(Sugumaran P, 2009)
Fluidized fixed bed	OPEFB	300-600	91 – 106 μm	-	-	-	25.98	-	(Sukiran et al., 2011)

Source from Z.Nahrul 2020.

2.2.6 Pores of Biochar

Each of the three types of pore diameters has a different purpose in the adsorption process. Because of its large surface area and volume, activated carbon's complete adsorptive ability is determined by micropores. Condensation occurs in mesopores when the pressure is high sufficiently. Mesopores also create a pathway for micropores to reach exterior surface. Because of the lower surface area, the quantity of adsorbate involved in mesopores is limited. When activated carbon is utilised as a catalyst support, however, it acts as a catalyst deposition site.

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

The most often used activated carbons are microporous and have a large surface area, allowing them to adsorb low molecular weight chemicals and larger molecules with good efficiency. The activated carbon's pore size improves the removal efficiency. This is a unique characteristic. Furthermore, the adsorption capacity is influenced by the pore-size distribution and the relative size of the adsorbate molecules (Patel & Himanshu., 2018).

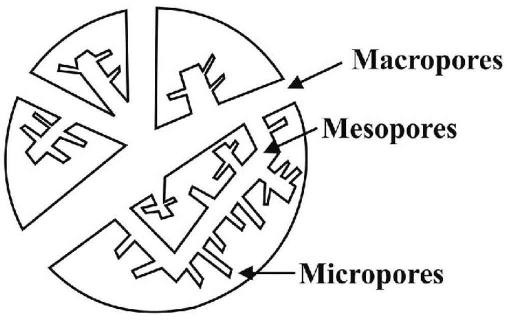


Figure 2.1: Schematic diagram of structure of activated carbon (source: Patel & Himanshu, 2018)

2.2.7 Carbon Activation Process of Biochar

Physical activity throughout the carbonation process improves the quality of biochar. Activated carbon has a wide range of uses, from improving air quality to the water treatment. Microwave supported carbonation and activation techniques with temperatures up to 900 °C might be used for both processes. A temperature controller kept the reactor's temperature under tight control. Chemical activation produces greater BET surface areas with less MW power, according to studies. In addition, despite the lower MW power, other factors such as activated duration, temperature, impregnation ratio, and agent all had a role in the development of porosity in the activated carbon (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 technics 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 char, KOH, ZnCl2, NaOH, H3PO4 and K2CO3 impregnation can be applied, 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 text research of 81 articles was accepted in its entirety. Figure 2.2 illustrates the number of items selected based on the chemical activator used in the manufacture of activated carbon from agricultural wastes.

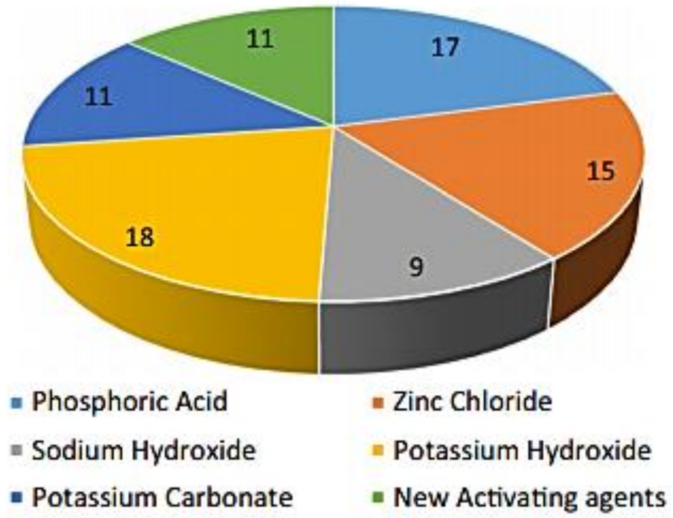


Figure 2.2 Number of articles based on the chemical activator used for the preparation of activated carbon from agricultural residues (source :Zoha, 2020)

Chemical activation, which includes doping char with a chemical agent to change surface properties, is one of the most frequent procedures for making porous bio-char, although its mechanism is unclear. Chemical activation was achieved by immersing or suspending pyrolysis bio-char in a chemical agent's solution at a ratio of up to 1:5 (bio-char: acid, base, or salt) for a given amount of time at room temperature up to 120C. Secondary heat treatment may be employed to get the necessary modified bio-char. Thermal treatments at high temperatures totally carbonise functional group-rich char, resulting in the formation of new nanopores and an increase in surface area. The bio-char was then rinsed with acid or base and deionized water until the acidic/alkaline pH was neutralised, failing which the bio-functioning char's and compatibility would be endangered. Washing also removes the chemicals that have remained in the carbonised sample, allowing new pores to develop.

Chemical activation using alkaline reagents, particularly KOH, is commonly applied to generate materials with a large surface area and 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). 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, bio-char's adsorption capacity for pollutants is improved, especially for ono-polar adsorbates.

The main reason to utilize various chemical activation techniques is to increase a partially blocked porous surface by suing agents to extract trapped products of incomplete pyrolysis (Figueiredo et al., 1999). Chemical activation is achieved through two methods which is acidic modification during chemical modification and alkaline modification through chemical reduction. Acidic modification is the chemical activation of a substance using an acid reagent, whereas alkaline modification is the chemical activation of a substance using an alkaline reagent. During pyrolysis, oxidation can be achieved by employing a gas such as steam, co2, ozone, air, or NO, or by treating 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. Chemicals having substantial colouring capacity are used in the textile, pharmaceutical, food, cosmetics, plastics, photography, and paper industries. The dyes may attach to suitable surfaces by creating covalent bonds or complexes with salts or metals, physical adsorption, or mechanical retention (Drumond Chequer et al., 2013).

Textile Industry is predicted to utilize over 10,000 different dyes and pigments, with over 7105 tonnes of synthetic dyes produced per year (Drumond Chequer et al., 2013). 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. Among these processes, the batch procedure is the most prevalent for dyeing textile fabrics.

2.3.2 Classification of Textile Dyes

The Colour Index (CI) provides data on approximately 8,000 dyes, which are classified into two categories: industrial application or dye end use, and chemical composition. In terms of chemical composition, dyes are categorized according to their chromophore. Recognized usage class, colour hue, and serial number are the three components of the CI general designations of dyes.

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

- Mordant dye
- Direct dye
- Disperse dye
- Vat dye
- Ingrain dye

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

- Azo dye
- Nitro dye
- Anthraquinone dye
- Triarylmethane dye
- Indigo dye

2.3.3 Toxicity of Textile Dyes Wastewater

Textile dyes are the most critical pollutants among the various chemicals in the textile sector that need to be removed. The textile dyeing industry generates a large amount of effluents, soil slurry, and solid waste substances every day. The textile industry is the largest user of dyestuffs, and wastewater created by the industrial is one of the most significant contributors of water pollution. The effluent contains toxic chemicals, organic debris, and heavy metals. It has been determined that the metal found in wastewater is carcinogenic. Dyes used in the textile sector offer a pollution issue due to their non-biodegradable complicated chemical composition. In dye-laden wastewater, temperature, colour, sanity, COD, BOD, and suspended particles are all excessive.

Textile dyeing effluents contained trace amounts of heavy metals such as iron, lead, nickel, copper, zinc, and chromium. Humans are exposed to carcinogenic and toxic synthetic azo dyes, providing a major health concern (Yaseen, D.A, 2018). These dyeing effluents are released into neighbourhood streams, farming lands, irrigation channels, outside water, and eventually rivers and oceans. Textile and dye industry effluents can disrupt the physical, chemical, and biological aspects of the aquatic environment by creating constant changes in turbidity, odour, noise, temperature, PH, and other factors that are hazardous to human health, livestock, ecology, fish, and biodiversity. The sizing, de-sizing, sourcing, bleaching, mercerizing, dyeing, printing, and finishing techniques used in textile production all contribute to key pollutants in textile wastewater outflow (Yaseen & Scholz, 2018b).

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). Most dyes remain in the atmosphere for a long time and are difficult to dissolve due to their complex aromatic molecular structures and synthetic nature. Due to contamination from both point and non-point sources from industry, the amount of usable water is restricted, causing issues. When dye-bath effluents are discharged into the water, they have a difficult time losing their colour and prevent light penetration in the water body, affecting aquatic biological processes.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter includes experimental methodologies, adsorbent, and textile dye wastewater preparation. Apart from that, the usage of adsorbent in wastewater treatment is also shown. In this research, variety of temperature in pyrolysis process have been used to produce activated carbon from 500°C to 700°C. The Chemical Oxygen Demand test, Total Suspended Solid (TSS), Turbidity was employed in this experiment.

Raw palm oil mill effluent sludge cake has been used as a preliminary for activated carbon (POME). The samples were obtained from the initial effluent of the Felda Lepar Hilir 3 Palm Oil Mill in Gambang, Pahang, Malaysia. To minimise bacterial contamination, they were then refrigerated at temperatures below -20°C. Figure 3.1 demonstrates the flowchart of the study methodology.

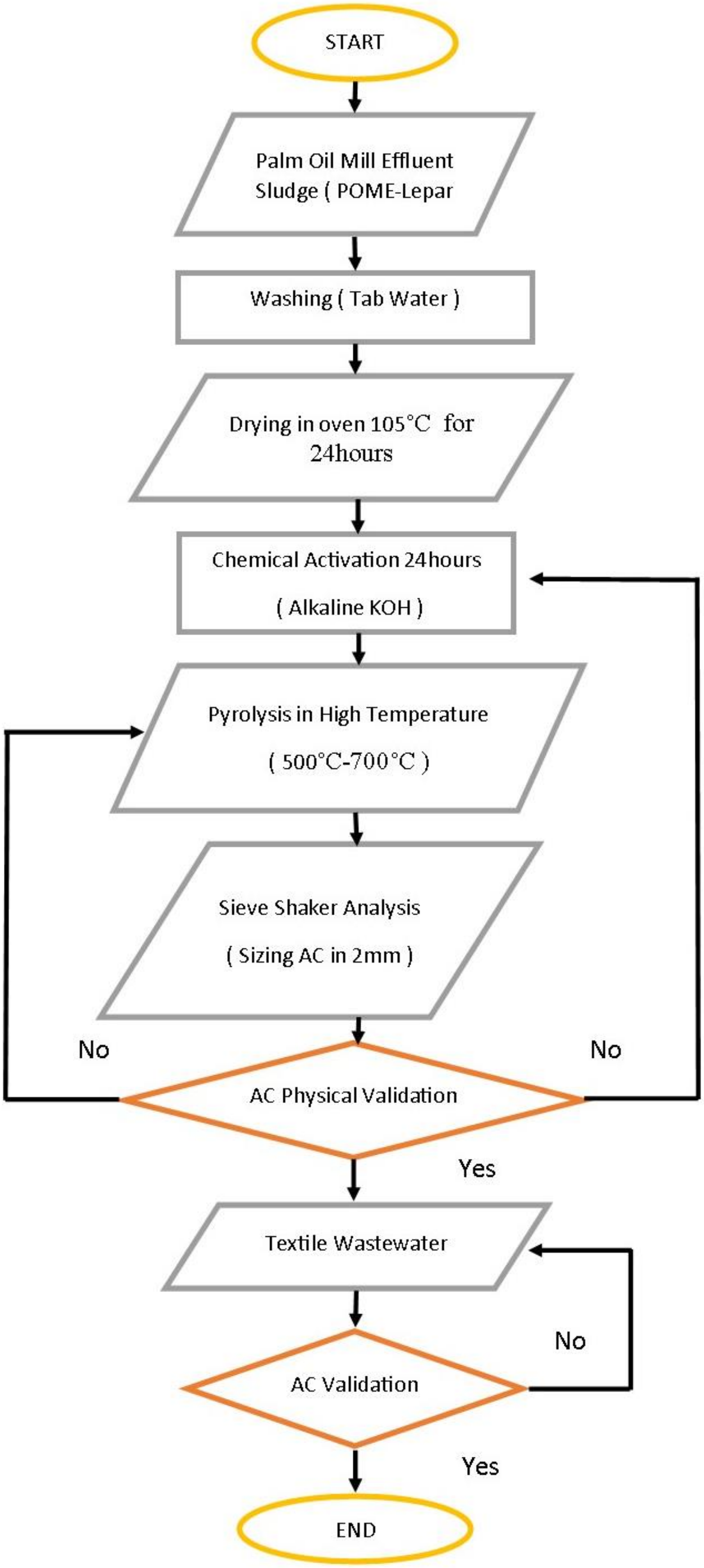


Figure 3.1: Flow chart of research methodology

3.2 Preparation of Activated Carbon

3.2.1 Carbonization of POMES

The raw material from Palm Oil Mill Sludge (POME) was dried at 105°C for 24 hours to create a uniform weight and remove moisture from the substance. Oil palm estate in Bintulu were washed with water to remove dirt from its surface and then dried in an oven at 105 o C for 1 day to remove excess water and allow the phosphoric acid to absorb entirely (KJ Lau, 2021). Dry sludge was used for the carbonization process. Then, using a mechanical sieve shaker, the material will be sieved to a uniform size of 2mm. After that, the dried sludge will be carbonised in a furnace for 60 minutes at range temperature from 500°C to 700°C. The formed char will next be chemically activated.

3.2.2 Chemical Activation of POME

Alkaline Potassium Hydroxide (KOH) were used to activate the activated carbons POME in chemical activation. To mix the carbon produced with the activating agent solution, several impregnation ratios will be required (IR). IR refers to the mass of impregnant in proportion to the bulk of charcoal. In this experiment, the impregnation ratios (IR) used were 5:1 (300g Biochar | 60ml chemical). The carbon and activating agent combination was then stored for another 24 hours. The mixture will be washed with distilled water to remove any activating agent compound. This process will be continued until the pH of the washing solution is neutral. To get a consistent mass, the product will be dried for another 24 hours at 105°C.

3.3 Textile Dye Wastewater

3.3.1 Preparation and Characteristic

Dye is mixed with a variety of contaminants in various amounts in textile effluent. The activated carbon that resulted was used to treat textile dye effluent with a high suspended solids concentration. Textile effluent is also a major source of pollution in surface waterways. Various approaches have been explored to treat these hazardous effluents. In terms of energy usage, they are, nevertheless, both cheap and inefficient.

As a result, activated carbon adsorption has been studied as one of the most promising treatment interventions.

This experiment employed wastewater collected from Pusat Tenun Pahang Diraja to evaluate the efficiency of the activated carbon produced. Suspended solids were removed from the samples. The samples were sent to the laboratory in UMP in a 4L container for analysis. To avoid contamination, the wastewater sample was kept in a freezer at -20°C at the chemical laboratory UMP. There were no chemicals applied to the wastewater sample that was taken. The wastewater sample was collected and processed in compliance with the standard operating procedures.

3.3.2 Experimental Technique for Wastewater Treatment

The validation procedure is carried out in order to better understand the activated carbon's adsorption properties and application. Batch experiments were used to conduct the experiment. The adsorption test was carried out in a 500 mL sealed beaker containing 50 mL of aqueous solution with an initial concentration of 78 mg/L at 27 1 °C and pH 9. First, the pH level of textile dye effluent was determined. The pH was not changed and was tested as it was. The textile dye wastewater was treated with POME sludge adsorbent in 10 g adsorbent/L sample dosages and the solution was agitated at room temperature at a mixer speed of 150 rpm for 15 minutes to ensure the AC was meticulously mixed with the solution and to establish equilibrium.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

The results are explained in depth and analysed in this chapter in order to achieve the study's main objective. The surface properties, elemental, and proximate compositions of activated carbon generated by CO2 are investigated. The activated carbon was tested for its ability to remove suspended particles from textile dye wastewater in order to fulfil river water quality requirements.

4.2 Introduction

Treatment of Textile Wastewater can be conduct of various analysis which is:

- Total Suspended Solid
- Chemical Oxygen Demand
- Color

4.3 POME Sludge-Adsorbent Performance

To determine the adsorbent's performance, the effects of adsorbent dosage was examined. As a result, the trend might well be explained to the available adsorptive capacity of POME sludge adsorbent, which was not fully used at increasing adsorbent dose. This might be due to the equilibrium concentration difference, where a low driving force for adsorption occurred at higher adsorption dosage. (Chong, Lee, Chieng, & Ramli, 2009).

Aside from that, the absorption of suspended solids onto the POME sludge adsorbent is not affected by surface area. The presence of functional groups may be causing the adsorption.

4.4 Standard discharge limit by DOE

Table 4.0 illustrates the amounts of BOD, COD, TSS, and colour of textile industry discharge using dual approach bioadsorbent materials, which were compared to the Department of Environment Malaysia's standard discharge limit (1982). Even though all of the reference studies showed great removal efficiency, there is a standard value limit for POME before release into rivers to avoid destroying the environment and to fulfill the zero-emission concept. Because BOD, TSS, and colour concentrations are more stringent than COD concentrations to achieve standard discharge standards, we may conclude that bioadsorbent from oil palm mill sludge successfully meets the requirement for textile industrial effluent.

. The result indicated that the adsorbent is most efficient at Raw Biochar 600°C, reducing Chemical Oxygen Demand from 60 mg/L to 22 mg/L, passing the river water quality standard of 50 mg/L for COD (DOE) allowing it to be discharged into the environment. However, for color reducing by using POMS as biochar can indicated is also from Raw Material 600°C, reduce color from 145 PtCo to 32 PtCo.

Table 4.1 Comparison of Textile Wastewater discharge after treatment with bioadsorbent and the requirement for standard discharge limit

AC Material	**TSS (mg/L)**	**COD (mg/L)**	**Color ADMI / PtCo**
Textile Wastewater	**0.1095**	**60.0**	**145.0**
Raw 500°C	0.4101	34.3	96.6
Raw 600°C	1.6306	22.3	32.0
Raw 700°C	0.972	14.6	311
KOH 500°C	0.5628	122.0	235.3
KOH 600°C	0.3834	82.0	113.6
KOH 700°C	0.4149	52.0	82.6
Standard A DOE	**50**	**50**	**100**
Standard B DOE	**100**	**100**	**200**

CHAPTER 5

CONCLUSION

5.1 Introduction

The goal of this research is to create activated carbon from POME sludge. The production of POME sludge-based activated carbon is completed in this chapter. Some suggestions for improving the efficiency of the activated carbon generated were also presented.

5.2 General Conclusion

The potential of palm oil mill sludge generated biochar to remove suspended solids, COD, and dyes solutions was examined using chemical activation and pyrolysis temperatures of 500, 600, and 700 °C. When comparing biochar pyrolyzed at higher temperatures to biochar pyrolyzed at lower temperatures, it was discovered that biochar pyrolyzed at higher temperatures did not generate statistically significant colour removal efficiencies. In terms of cost, it was determined that producing biochar from POMS at a lower temperature consumes less energy and is more cost effective.

However, both surface chemical and physical properties were shown to be affected by pyrolysis temperature. In comparison to the chemical activation, biochar that has been pyrolyzed at high temperatures has lost the majority of its functional qualities. Due to the volatilization of organic matter, the surface area and pore volume increased as the pyrolysis temperature increased, and they became more porous and showed deep channels. Carbon concentration increased as the pyrolysis temperature raised.

The palm oil mill sludge as biochar are effective low-cost absorbents for removing colour and dyes. Utilizing palm waste materials to produce biochar is a sustainable solution due to its negative impact on Global Warming Potential (GWP), cost effectiveness and waste management perspective. These study results also can involve the development of an appropriate technology for the palm oil industry, using commonly available equipment and a simple method that can be easily implemented to solve the biochar's lack of adsorption performance and promote the technology's adoption, especially in the palm oil industry.

Physical activation with CO2 generated POME sludge-based activated carbon with excellent adsorptive abilities from Palm Oil Mill Effluent in this study. The high ash concentration is thought to be responsible for the AC's small surface area. POME sludge based activated carbon produced in this study was successful in removing suspended particles from textile dye wastewater. The result indicated that the adsorbent is most efficient at Raw Biochar 600°C, reducing Chemical Oxygen Demand from 60 mg/L to 22 mg/L, passing the river water quality standard of 50 mg/L for COD (DOE) allowing it to be discharged into the environment. However, for color reducing by using POMS as biochar can indicated is also from Raw Material 600°C, reduce color from 145 PtCo to 32 PtCo. POME -sludge-based activated carbon is a superior alternative for POME sludge handling and disposal from an environmental and economic viewpoint, and it might be utilised for a variety of wastewater treatment applications. In conclusion, this research project has justified that POMS is suitable to be used to produce AC that can treat Textile Waste water very efficiently.

5.3 Recommendations

AC is recognized as one of the most efficient and successful materials in the treatment of water and wastewater. It is affordable and widely accessible in the industry. However, there are certain enhancements that might be performed to improve the AC's performance and efficiency. The following are some suggestions for further research:

1. To produce a nanoparticle size, the activated carbon should be sieved to a uniform size.
2. A variety of activation durations and temperatures should be investigated to find the greatest combination for producing the most effective activated carbon.
3. Prior to any analysis, the AC should be dried at 105oC for 24 hours to eliminate any moisture content.
4. After turning on the air conditioner, a multiple of washing procedures should be done to eliminate any pollutants.
5. Experiments with other well-characterized adsorbents will be beneficial in understanding the function of carbon structure in SAC adsorption.
6. Analysis of leachable elements from POME sludge should be performed to identify and determine components that may be released from the POME sludge adsorbent, so that appropriate post-treatments may be conducted to eliminate the leachable elements.
7. In goal is to expand the study to practical applications, it is important to analyze the adsorption and kinetics behaviour of the adsorbates and adsorbents.
8. The carbonization and activation processes should be carried out in an inert atmosphere because impurities in organic waste and deposited solid impurities that are not removed by washing procedures cause ash to form when heated in the presence of these impurities.

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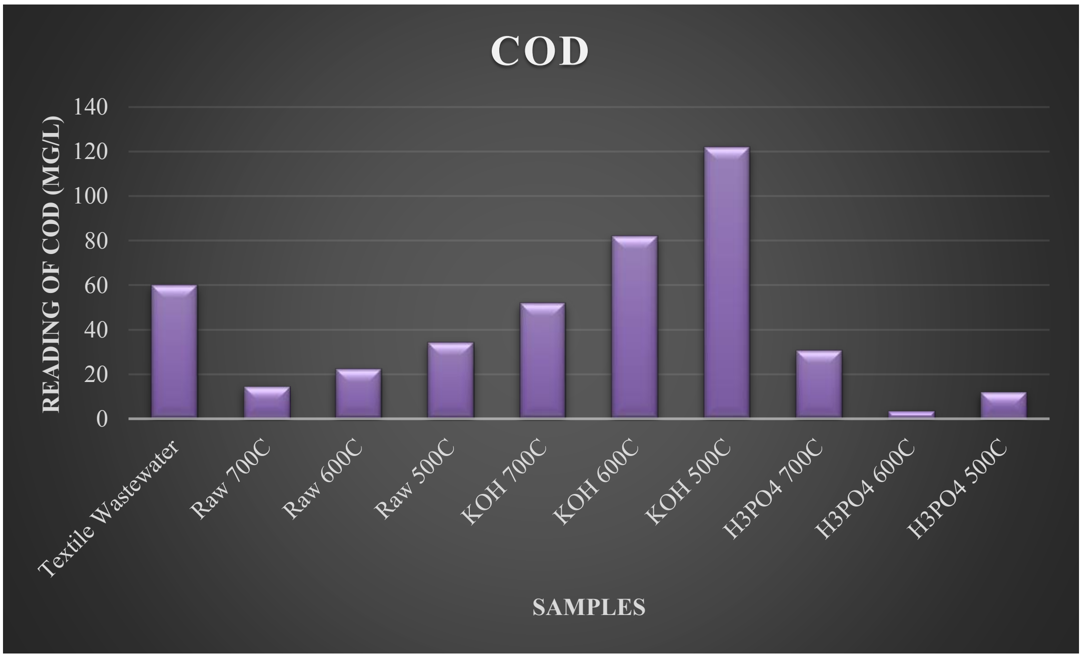
APPENDICES

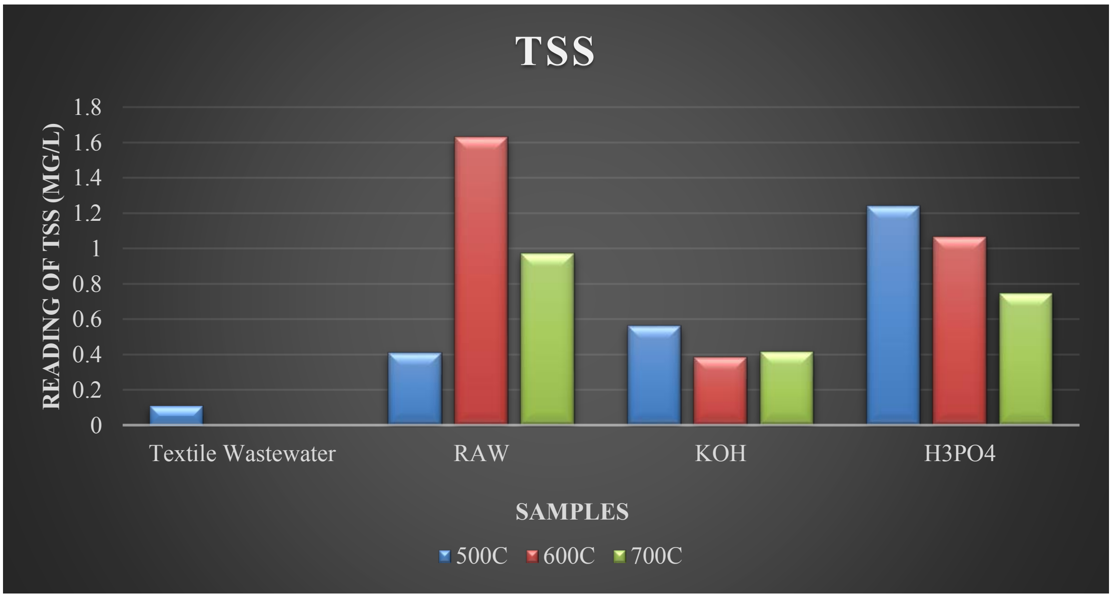
Appendix A:

Data Chart of Chemical Oxygen Demand



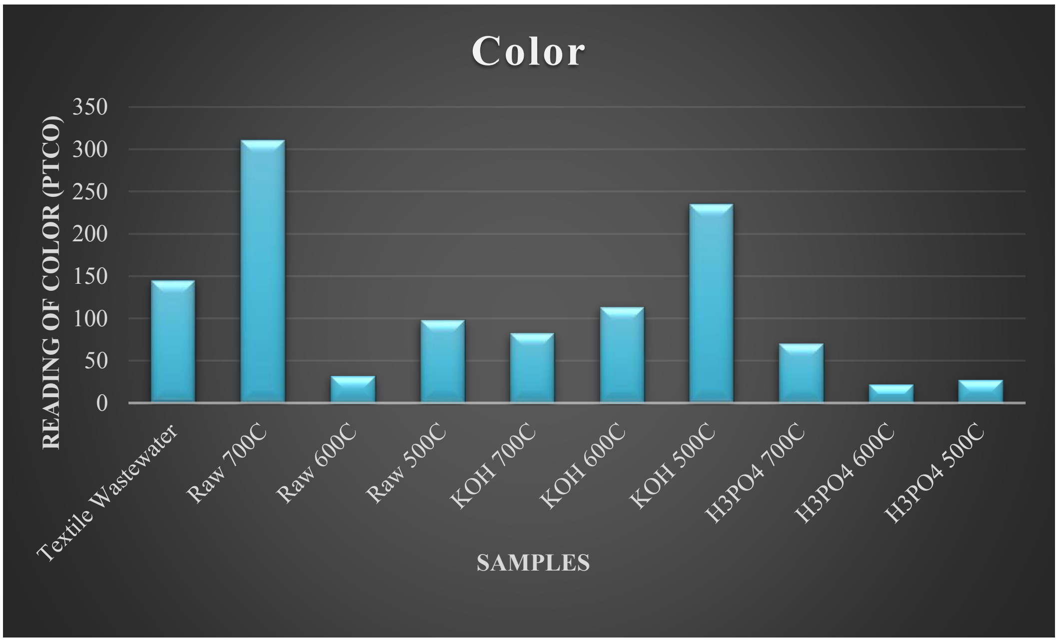
Appendix B:

Data Chart of Total Suspended Solid



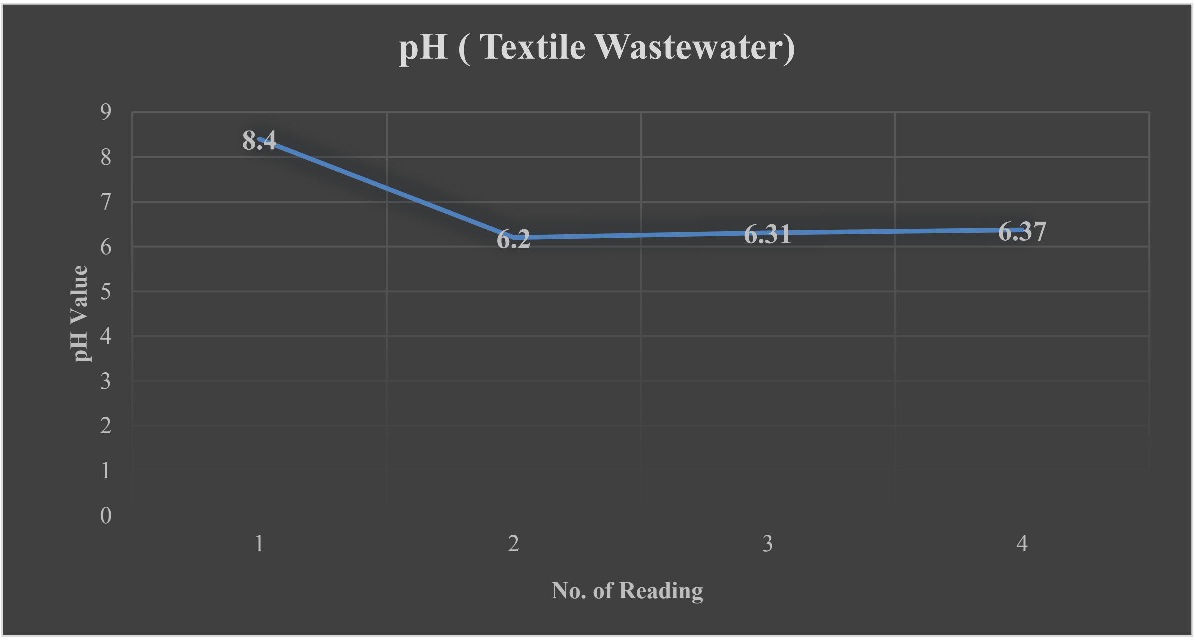
Appendix C:

Data Chart of Color



Appendix D:

Data Chart of pH Textile Wastewater



Appendix E:

Result after apply biochar into Textile Wastewater

Appendix F:

Source of Textile Wastewater at Tenun Pahang Diraja, Sungai Soi

Appendix G:

During Chemical Activation Process on Biochar

Appendix H:

Applying Biochar in Textile Wastewater

Appendix I :

Source of Palm Oil Mill Effluent Sludge at FGV Holding Berhad , Lepar Hilir 3

Appendix J :

Crushed and sieved process

Appendix K :

Carbonization and Pyrolysis Process