PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM OIL PALM TRUNK FIBER BY CHEMICAL ACTIVATION

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by

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LIST OF SYMBOLS

Symbol	**Description**	**Unit**
SA	Surface area	m^2/g
Ce	Concentration of adsorbate at equilibrium	mg/L
Co	Initial adsorbate concentration	mg/L
q_e	Amount of adsorbate adsorbed per unit mass of adsorbent at equilibrium	mg/g
q_m	Maximum adsorption capacity of adsorbent	mg/g
K_F	Freundlich isotherm constant related to capacity	mg/g
K_L	Langmuir adsorption constant	L/mg
R^2	Correlation factor	-
R_L	Separation factor	-
n_F	Freundlich isotherm constant related to adsorption intensity	-
W	Mass of adsorbent used	mg
V	Volume of dye solution	cm^3/s
t	Time	hr
M_F	Final concentration of adsorbate	mg/L
V_F	Final volume of adsorbate	L
M_I	Initial concentration of adsorbate	mg/L
V_I	Initial volume of adsorbate	L
W_{AC}	Weight of activated carbon	mg
W_I	Weight of precursor	mg

LIST OF ABBREVIATION

Symbol	**Description**
AC	Activated Carbon
BET	Brunauer-Emmet-Teller
SEM	Scanning Electron Microscope
MO	Methyl Orange
H_3PO_4	Phosphoric Acid
OPTF	Oil Palm Trunk Fiber
UV	Ultraviolet
NaOH	Sodium Hydroxide
HCl	Hydrochloric Acid
N_2	Nitrogen
IR	Impregnation Ratio
P	Parenchyma
VB	Vascular Bundle

PREPARATION AND CHARACTERIZATION OF ACTIVATED CARBON FROM OIL PALM TRUNK FIBER BY CHEMICAL ACTIVATION

ABSTRACT

Industrial activated carbon has been a favoured adsorbent for the removal of a variety of contaminants, but its relatively expensive cost has limited its broad application. As the commercial activated carbon produced from non-renewable and unsustainable resources, this situation led to study on the possible alternative and low-cost replacements on raw materials to produce activated carbon. Due to the extensive discharge of pollutants, activated carbon produced from biomass waste by various processing methods and conditions is gaining popularity as a promising adsorbent for ecological treatment. Methods of manufacturing activated carbon, the type of lignocellulosic biomass waste, and the interaction between adsorbent and adsorbate are essential elements that must be considered in order to develop an effective adsorbent. Oil Palm trunk fibre (OPTF) is one of the major oil palm biomass wastes with a high carbon content that can be used as a precursor to produce activated carbon (AC) by utilising phosphoric acid (H_3PO_4) that can be turned into activated carbon with a large surface area and porous structure. This project's objective is to investigate how the impregnation ratio (IR) of OPTF and H_3PO_4 affects the porosity development and surface area of AC. Various physiochemical parameters such as pH of adsorbent solution and amount of activated carbon used were investigated in a batch adsorption technique. aximum surface area (1521.9 m^2/g) was found at IR of 2, which demonstrates a type IV isotherm, that is contains micropores and mesopores. At three different pH values of 4, 7, and 10, adsorption equilibrium isotherms for methyl orange (MO) adsorption onto the AC were collected and nonlinearly fitted using the Langmuir and Freundlich isotherm models, with the Langmuir

isotherm providing a better fit. The absence of repulsive electrostatic interaction between the AC surface and MO can account for the greater adsorption capacity at lower pH. In addition, the removal efficiency was not significantly improved by increasing the amount of activated carbon.

PENYEDIAAN DAN PENCIRIAN KARBON TERAKTIF DARIPADA BATANG KELAPA SAWIT MENGGUNAKAN PENGAKTIFAN KIMIA

ABSTRAK

Karbon teraktif industri telah menjadi penjerap yang digemari untuk penyingkiran pelbagai bahan pencemar, tetapi kosnya yang agak mahal telah mengehadkan penggunaannya yang luas. Ini telah membawa kepada kajian tentang kemungkinan penggunaan bahan mentah yang alternatif dan kos rendah bagi penghasilan karbon teraktif. Disebabkan oleh pelepasan bahan pencemar semakin meluas, karbon teraktif yang dihasilkan daripada sisa biojisim melalui pelbagai kaedah dan keadaan pemprosesan berkembang sebagai penjerap yang menjanjikan dalam rawatan ekologi. Kaedah pembuatan karbon teraktif, daripada sisa biojisim lignoselulosa, dan interaksi antara penjerap dan bahan yang diserap adalah elemen penting yang mesti dipertimbangkan untuk membangunkan penjerap yang berkesan. Gentian batang kelapa sawit (OPTF) adalah salah satu sisa biojisim kelapa sawit utama dengan kandungan karbon tinggi yang boleh digunakan sebagai prekursor untuk menghasilkan karbon teraktif (AC) dengan menggunakan asid fosforik (H_3PO_4) yang boleh ditukar menjadi karbon aktif dengan luas permukaan yang besar dan struktur berliang. Maka, objektif projek ini adalah untuk menyiasat bagaimana nisbah impregnasi (IR) OPTF dan H_3PO_4 mempengaruhi pembangunan keliangan dan luas permukaan AC. Luas permukaan maksimum (1521.94 m^2/g) didapati pada nisbah impregnasi 2, yang menunjukkan isoterma jenis IV, iaitu mengandungi liangmikro dan liangmeso. Pada tiga nilai pH berbeza iaitu 4, 7, dan 10, isoterma keseimbangan penjerapan untuk penjerapan MO pada AC telah dikumpulkan dan menggunakan model isoterma Langmuir dan Freundlich, yang membuktikan isoterma Langmuir memberikan kesesuaian yang lebih baik. Ketiadaan interaksi elektrostatik yang menolak antara permukaan AC dan metil jinga (MO) boleh

menyumbang kepada kapasiti penjerapan yang lebih besar pada pH yang lebih rendah. Di samping itu, kecekapan penyingkiran tidak meningkat dengan ketara dengan meningkatkan jumlah karbon teraktif.

CHAPTER 1

INTRODUCTION

1.1 Activated Carbon (Ac)

Activated carbon (AC) is a solid carbonaceous material characterized by large internal surface areas and well-developed structure, used as adsorbents to remove organic and inorganic pollutants toward purifying liquids and gases in various applications, including food and drink industry, aroma elimination, and industry environment protection. AC are also capable of absorbing chemicals from carbon dioxide (CO_2), urea, and other gases (Reza et al., 2020). Moreover, AC can also absorb colors, heavy metals, petroleum hydrocarbons, medicines, insecticides, and other organic compounds(Hubetska et al., 2020). The ability of AC to behave as an adsorbent is the result of carbonation and activation activities that produce pores on the precursor surface.

According to Observatory of Economic Complexity, Malaysia exported $51.5M worth of activated carbon in 2020. Japan ($9.45M), Singapore ($7.21M), Netherlands ($3.98M), Thailand ($3.98M), and Germany ($2.16M) were the top destinations for Malaysia's exports of activated carbon. More than ten firms produce between 300 and 1000 tons of AC every month in Malaysia(Saad et al., 2020). According to the market research, the Asia-Pacific activated carbon market size is anticipated to reach $4,307.9 million by 2028, up from $2,192.1 million in 2021, at a compound annual growth rate (CAGR) of 10.0% between 2021 and 2028. This analysis demonstrates that activated carbon is a prominent industrial adsorbent worldwide.

Generally, AC is produced from raw material with high carbon and organic content. (Saleem et al., 2019). Due to the excessive reliance on crude oil resources and the development of environment issues, biomass has been identified as a sustainable feedstock for the manufacture of AC. (Popp et al., 2014). There are multiple potential biomass sources for the manufacture of AC in Malaysia such as agricultural waste, forest residues, and municipal waste. Among them, biomass seems to have the best potential of a sustainable alternative to the existing production of AC.

Currently, the production of AC depends on physical activation and chemical activation(Saleem et al., 2019). Pyrolysis of the precursor material in inert gas or carbon dioxide flow are the physical activation process. (Saleem et al., 2019). The development of the highly porous AC is accomplished by removing a substantial quantity of internal carbon mass. In addition, highly porous carbons will only be produced at high activation temperature. In contrast, the chemical activation process involves impregnating the raw material with activation agents. The formation of pore by chemical activation is due to the removal of volatile content matter with chemical agent by dehydration technique. Due to the dehydrogenation technique, the chemical agent facilitate the production of cross-link, resulting in the formation of a hard matrix.(Saleem et al., 2019). Besides, the chemical activation can produce activated carbon at lower activation temperature and short activation time which is cost effective(Saleem et al., 2019). This clearly shows that the chemical activation method possesses significant advantages and produces a larger surface area and better developed porosity for AC compared to physical activation method.

1.2 Biomass from the Oil Palm Industries

Malaysia is the world's second-largest producer of palm oil. (Alam et al., 2015). These products are critical to Malaysia's economic development. As the sector expands in size and importance, vast quantities of oil palm waste are produced, creating a disposal concern for biowaste. Lignocellulosic biomass, which is generated after the oil palm harvesting, include oil palm fronds (OPF), empty fruit bunches (EFB), and palm pressed fibers (PPF), palm shells and palm oil mill effluent (POME) (Abdullah and Sulaiman, 2013). Since oil palm trees need to be replaced with new oil palm trees after 25 years due to low production, this created another source of oil palm biomass which is oil palm trunk fiber. The oil palm trunk fiber (OPTF), which is biodegradable, non-toxic, and rich in carbon, has been found as the precursor for the manufacture of AC. (Abdullah and Sulaiman, 2013)

At the moment, the disposal of oil palm trunk is still a major problem facing the plantation industries. The presence of Ganoderma Boninense, a pathogen that causes oil palm Basal Stem Rot (BSR) may further complicates this problem. The illness is one of the industry's most important obstacles. Although BSR is often detected in mature stands, sick palms have been discovered as early as 12–24 months after planting (Bivi et al., 2016). Infection with this disease can result in significant yield loss and eventually results in the destruction of basal tissue, resulting in the death of palm tree. These problems tend to burden operators with control problems and increase producing costs. Thus, fully removal of the oil palm waste after 25 years is required to prevent formation of Ganoderma Boninense in new oil palm trees. This indicates that oil palm plantations created a vast quantity of old chopped oil palm trunks, which must be handled with promptly due to their volume. For the utilization of OPTF, numerous applications, such as saccharification and fermentation for bioethanol, chemical activation for nanocellulose, thermal decomposition for bio-oil, and physical manufacturing for composites, such as wood material, plywood, and particleboard, were investigated.(Prawitwong et al., 2012). In addition, the OPTF has great potential in many applications in reducing the dependency on fossil fuels.

1.3 Research Objectives

The objectives of this research:

1. To prepare oil palm trunk-based activated carbon by chemical activation with chemical activating agent, which is phosphoric acid (H_3PO_4).
2. To characterize the physical properties of the synthesized activated carbon.
3. To study the performance of the synthesized activated carbon for Methyl Orange removal at different pH, Impregnation ratio and amount of activated carbon.

1.4 Problem Statement

According to previous literatures, it was found that the major challenges faced by commercial activated carbon production are the availability of carbon precursor, activation pathways, activating agents and processing conditions(Gao et al., 2020). The production of activated carbon conventionally uses petroleum-based resources as one of the precursors. However, the uses of petroleum-based resources are non-renewable and unsustainable. Therefore, the use of lignocellulose biomass as carbon precursor instead of fossil resources such as coal in the production of AC can be more environmentally friendly. Oil palm trunk fiber, one of the lignocellulosic-rich biomasses is a promising feedstock for AC production due to its flexibility, availability, efficiency, and economical advantages(Lim et al., 2020). In addition to that, OPTF has a high volatile matter required in the production of AC with high adsorption capabilities and good pore size, pore structure and surface area characteristics. Significant amounts of oil palm trunks are produced in Malaysia's extensive oil palm plantations and therefore it is a readily available precursor for AC production.

AC is widely applied as adsorbent for removal of organic chemicals and metal ions from air, gases, potable water and wastewater. Either by selecting a low-priced raw material or by employing an efficient manufacturing method, it is possible to lower the cost of producing activated carbon. Nevertheless, it is still a challenge to produce activated carbons with porous structure and high surface area. which are suitable for adsorption. Consequently, it is of the utmost importance to identify low-cost precursors that are economically viable and capable of producing activated carbons with good adsorption capability. Recently, phosphoric acid activation has been developed to provide high surface areas and well-developed porosity(Luo et al., 2019). Other than that, this activation method is able to produce high adsorption capacity AC for removal of dye.

Methyl orange is one of the most often employed colors as an adsorbate in measuring the adsorption capacity of AC from aqueous solution. However, the optimal conditions of the adsorption process must be determined in order to achieve the maximum dye removal in terms of the pH of the aqueous solution and the amount of AC. Consequently, the purpose of this work is to examine the viability of preparing AC from biomass abundantly available in Malaysia, especially oil palm trunk, via chemical activation with well-developed pores, high surface area, and high adsorption capacity. The performance of the synthesized AC will be tested for Methyl Orange adsorption at different pH, impregnation ratio and amount of AC.

1.5 Scope of Research

Lignocellulosic biomass is a green alternative to replace the presence of non-renewable and unsustainable feedstock. The performance of lignocellulosic biomass as a precursor for the manufacture of AC is outstanding. This is owing to the fact that it has a high concentration of hydrocarbon molecules and is readily available. Many types of lignocellulosic biomasses, such as wood, corncob, rice husk, and many others, can be utilized as precursor for AC production, but in this research project, oil palm trunk fiber (OPTF) is used as precursor for AC production due to its availability and lack of utilization.

This research has decided to produce activated carbon from OPTF using the chemical activation process. In chemical activation, the carbonization and activation are achieved in a single step by impregnating raw material with activating agents. The activating agent is selected is Phosphoric acid (H_3PO_4). Phosphoric acid works as an chemical agent, which facilitates breakage reaction (Yang et al., 2020). After the impregnation stage, the precursor was combusted in furnace using inert gas (N_2) with preserving operational parameters settings such as activation temperature and activation period.

Many factors affect the performance of AC, such as the impregnation ratio of chemical agent and OPTF, pH of aqueous solution, and amount of AC used(Yahya et al., 2018). The pH can influence the performance of AC, which has a significant effect on surface charge of aqueous solution(Cavalcante et al., 2022). For instant, the surface of activated carbon is positively charged at low pH, while the surface of activated carbon is negatively charged at high pH. Usually, the activated carbon must be neutral as the adsorption process is highly pH-sensitive and pH driven(Yang et al., 2020). However, the alkalinity and acidity of the working solution have a substantial impact on the adsorption process. In addition, the amount of AC used also affects the performance of adsorption. As the molecular weight increases, the solubility of the molecules in water increases, enhancing the absorptivity of AC. The impregnation ratio also affects the performance of AC. The impregnation ratio is the quantity of the chemical agent (H_3PO_4, KOH, etc.) divided by the mass of organic material that must be transformed into activated carbon (often biomass, but it can also be non-activated porous carbon). According to previous research, the volume and depth of activated carbon pores formed increase as the activator concentration increases.

1.6 Thesis organization

This work is comprised of five chapters. The first chapter briefly presents the research context, problem statement, research objectives, and study scope of the current project. The second chapter for literature review will examine the raw material available in Malaysia, classification of activated carbon, dye, the adsorption process, and parameters that effect adsorption. This chapter also includes batch adsorption and isotherm studies. In chapter three, materials, and methods section, all the materials and chemicals utilized in this study were explained in detail. The production of activated carbon (AC) procedures as well as the characterization techniques will also be given. The fourth chapter contains the results and discussion of the characterization and batch tests and isotherm studies for the removal of MO dye. This study's findings are summarized in the fifth chapter, along with recommendations for future research.

CHAPTER 2

LITERATURE REVIEW

Green approaches for activated carbon manufacturing have garnered a lot of interest in a range of industrial applications, as it makes up a huge and significant class of porous solids. Because of its versatility, efficiency, and cost-effectiveness, the oil palm trunk is indeed a possible carbon precursor for activated carbon production(Lin et al., 2020). In this way, the carbon precursor derived from biomass has a significant advantage over the carbon precursor derived from fossil resources. Utilizing neutral, acid, basic, and self-activating agents, AC can be produced. (Gao et al., 2020). In this instance, the chemical agents react with the cellulose, hemicellulose, and lignin in the carbon precursor, leading to the development, expansion, interaction, and collapse of pores. Chemical activation seems to be the most frequent method used for developing of porous structure on AC due to its lower temperature and reduced energy use. In this context, chemical activation methods for the production of AC have the potential to produce AC with a larger surface area and well-developed pores at lower temperatures than physical activation methods, providing considerable advantages.

2.1 Definition of activated carbon

Activated carbon (AC) is an odorless, black carbon-containing material with a large internal surface area and pores size. Amorphous carbon is typically used for cokes, chars, and activated carbon. Applications of AC depend mostly on its surface chemistry and pore structure. According to Benedetti et al., activated carbon is a carbonaceous substance with an amorphous structural solid, a high degree of porous structure consisting micro-,meso- and macropores, high surface area and availability of functional groups such as carboxylic acids and carbonyl group, make it versatile material with a wide range of applications.(Benedetti et al., 2018). In addition, it is the conventional name for a group of solid adsorbing agents with developed inner pore structures that make carbon contains more adsorption capacity. AC may be distinguished from amorphous carbon by removing all non-carbon contaminants and oxidizing the carbon surface. (Gao et al., 2020). These distinctive properties of activated carbon are determined by the nature of precursor used in its synthesis and the activation process.

In industry, activated carbon is a critical and rapidly developing cleaning agent that is extensively used to cleanse wastewater for a variety of dangerous metal ions and organic liquids, as well as trap harmful gases produced during incineration and recycling. AC is appropriate for industrial usage due to its high carbon content and low inorganic content, as it is derived from biomass, an inexpensive raw material(Parab et al., 2009). AC is also an organic compound characterized by a mostly graphitic structure.(Yahya et al., 2018). The basic properties among all AC are graphite-like planes with varying degrees of disorientation, the spaces between these planes that constitute porosity, and the unit made up of condensed aromatic rings(Jurkiewicz et al., 2018).

AC is a black, crystalline material made primarily of carbon with trace amounts of ash, volatile chemical compounds, and water, according to all classifications. In its simplest form, activated carbon exhibits physical and chemical characteristics such as surface functional groups, surface morphology, and surface area. The processed form of activated carbon includes microscopic, low-volume pores that enhance the available surface area for adsorption process.

2.2 Characterization and Properties of Activated Carbon

Characterization of AC is crucial for classifying it for certain applications. Basically, AC is characterized by chemical and physical properties(Gao et al., 2020). The characteristics of AC depend on the physical and chemical properties of the precursor as well as on the activation method(Ahmed and Theydan, 2012). Physical characteristics of AC, includes surface area and surface morphology while chemical characteristics is represented by moisture content, ash content and functional group of surfaces. These characteristics may influence the use of adsorbent and influence its suitability for particular applications. Additionally, AC has a porous surface structure that is divided into three types: microporous, mesoporous, and microporous(Maulina and Handika, 2019).

2.2.1 Moisture Content

The moisture content of activated carbon produced depends on chemical activation agent. Different types of chemical activation agent used will affect the moisture content of the resulting AC. According to Phansiri Moonsria et al., the moisture content of activated carbon produced with KOH as activator reached 1.65%(Jjagwe et al., 2021). In contrast, H_3PO_4 produced moisture content of 2 to 3%(Anisuzzaman et al., 2015). On the other hand, moisture content decreased along with activation temperature due to the decreasing amount of the pores formed in the activated carbon. This phenomenon results in reduced hygroscopic capability of activated carbon(Maulina and Handika, 2019). The impregnation ratio influences the generated moisture content as well. Increased water levels indicate that additional organic molecules are degraded by the activator during carbonization. The higher the impregnation ratio, the higher the moisture content of activated carbon, which promotes the dissolution of organic minerals on activated carbon. As most of the precursor's moisture should have been removed during activation, the moisture content can be attributed to the absorption of moisture from the surrounding atmosphere during the activation process. Although they may absorb moisture from their environment, it should appear to be dry. Lower moisture content in AC is better, because water vapours compete in adsorption process and fill the adsorption sites within the pores, thus reducing the efficiency of the AC(Zhou et al., 2001).

2.2.2 Ash Content

When carbonaceous material is burnt, a residue of carbon ash is produced. Carbon combustion will result in an increase in the amount of ash on AC(Maulina and Iriansyah, 2018). Since AC is composed of inorganic elements derived from primary sources and chemical agents, the overall quantity of inorganic constituents will fluctuate and will appear in the same form as they do when the carbon is in the ash(Saleem et al., 2019). If the ash content of the precursors is more than AC, organic components in the precursor will generate metal oxides, resulting in an increase in ash content. With rising of carbonization temperature, AC ash content also increased. These results show that when the carbonization temperature rises, the vaporization of particular volatile chemicals contained inside the particles increases(Zhang et al., 2011).

The ash level of AC is a good indication of its quality. The proportion of ash in commercial activated carbon exceeds 10%(Bernardo et al., 1997). Using biomass as a precursor, activated carbons with an ash concentration of less than 7% were generated(Jaguaribe et al., 2005). Chemical activation produces AC with very low ash content. These results suggest that chemical activating agents may be better in maintaining heat during carbonization, hence limiting further oxidation of organic matter and the formation of undesirable compounds such as ash(Son et al., 2005). As the chemical activation concentration increased, the ash content increased proportionally. The increased surface area of activated carbon caused by the high activator concentration results in the development of additional pores. As a result of the enhanced pore formation, more ash is generated(Tani et al., 2015).

2.2.3 Surface area

Activated carbon is a valuable carbonaceous material with a large surface area and a controllable pore structure that is frequently used in the industry as an adsorbent for the removal of a wide variety of organic and inorganic pollutants(Luo et al., 2019). The higher the specific surface area of AC, the higher the adsorption capacity of the AC and will result in good performance of AC. AC with a surface area of between 1000 to 1500 m^2 /g can be produced after treated with steam and heat in the physical activation process(Cevallos Toledo et al., 2020). In comparison, AC that is produced using chemical activation has a surface area varying from 736 to 2342 m^2/g(Heidarinejad et al., 2020). This comparison demonstrated that chemical activation produces a greater surface area than physical activation. The Brunauer-Emmett-Teller (BET) method is most frequently used to calculate the specific surface area (m^2 /g) of activated carbon(Mistar et al., 2020).

2.2.4 Surface Morphology

A method for determining the surface morphology of AC is required since the performance of AC is highly dependent on surface properties such as pore structure and roughness. Because the adsorption capacity of AC is positively related to surface characteristics such as pore structure and roughness, a technique for measuring the surface morphology of AC is required to verify and generate activated carbon that fulfils specifications(Mistar et al., 2020). The most common method for determining activated carbon's shape and surface morphology is using a scanning electron microscope (SEM). Good development surface morphology, such as pore structure, pore-volume, and surface area of the material, relates to effective results from using activated carbon for pollutant removal(Mistar et al., 2020). The surface morphology of activated carbon changed during adsorption for both chemical activation agents, indicating the accumulation of adsorbate following the adsorption process due to changes in the external and internal surface area and the degree of porosity of activated carbon. This demonstrates that the adsorbed adsorbate can replace existing molecules on the surface, resulting in a better adsorption performance than untreated activated carbon that remains the same after adsorption.

2.3 Porous Structure

Pores can be classified according to their available space between the pore walls using the IUPAC classification system as shown in Figure 2.1(Rouquerol et al., 2007). Micropores have pore widths less than two nanometers, mesopores have pore widths between two and fifty nanometers, and macropores have pore widths greater than fifty nanometers. Micropores and mesopores are used for adsorption, while macropores serve as a transport channel. Mesopores can be generated in carbon by enlarging micropores, such as reactivity with oxidizing gases, as in activated carbon(Maulina and Handika, 2019).

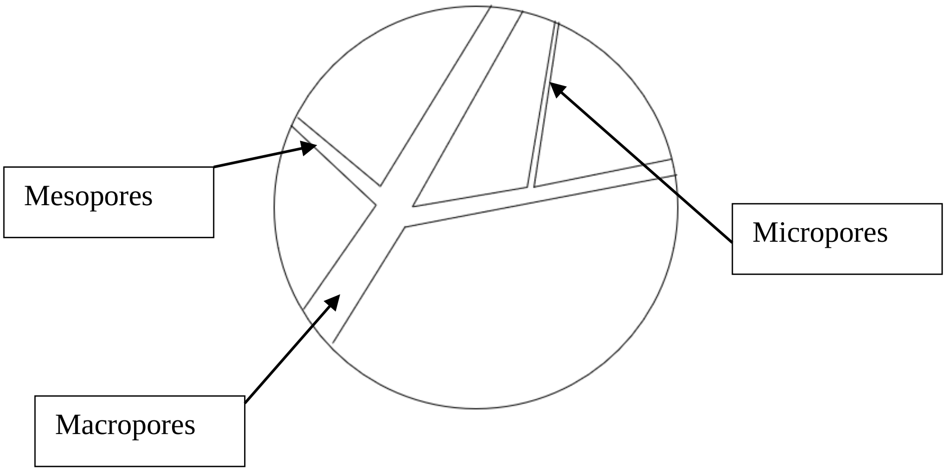

Figure 2. 1: Schematic of active carbon pore structure

The easiest way to characterize the structure of activated carbon is as crumpled carbon sheets. Activated carbons may be easily synthesized by opening and developing the pores of a variety of carbonaceous precursors(Celzard et al., 2007). Physical activation is a process that progressively gasifies the material across a temperature range of 700 to 1000°C while employing a mild oxidizing agent such as water vapor, carbon dioxide, or even air(Ahmed and Theydan, 2012). If the precursor lacks sufficient carbon, activation may be carried out in two processes, with the material first pyrolyzed in an inert environment to form char, which is subsequently oxidized to increase its porosity. This procedure simultaneously opens and expands all types of pores. Chemical activation, by contrast, requires heating the carbonaceous precursor to between 400 and 800°C. and combining it with a chemical agent(Ahmed and Theydan, 2012). The chemicals utilized (often strong mineral acids, metallic salts, or hydroxides) operate as dehydrating, cross-linking, and oxidizing agents simultaneously, resulting in the formation of porous carbons. This process produces very tiny or medium-sized holes, depending on the precursor and activating agent used(Ahmed and Theydan, 2012). For example, during the activation process, the loss of volatile components from carbon in the form of gas may result in the formation of a porous structure. When phosphoric acid was utilized as a chemical activator, the reticulated structure of oxygen and phosphorus prolonged the polyaromatic cross-linking, therefore increasing the porosity and surface area of AC(Yorgun and Yıldız, 2015).

2.4 Processing of activated carbon

Carbonaceous natural and synthetic precursors are pyrolyzed and activated to produce activated carbons. In the absence of air, pyrolysis of any carbonaceous substance results in the destruction of organic molecules, the evolution of tarry and gaseous products, and finally the formation of a solid porous carbon mass(Devi et al., 2020). Carbonization begins with the removal of the majority of non-carbon elements, hydrogen, and oxygen, from the starting materials by pyrolytic breakdown, and the free atoms of elemental carbon are clustered into structured crystallographic forms known as elementary graphite crystallites(Manocha, 2003). Carbonization is the process of heating a carbon-rich material and turning it to pure carbon. Lignin, cellulose, and hemicellulose are all components of biomass. Lignin, is perhaps the most thermally resistant compared to other components, which becomes the most abundant component of burned materials. Thus, the ratios of the three components in the initial raw material and at the end of the carbonization stage are crucial for determining the properties of charred materials, their behaviour during activation, and finally the yield and adsorptive qualities of AC.(Contescu et al., 2018). The only way to generate an adsorbent with a highly developed porosity and correspondingly large surface area is to activate the carbonized material, either physically or chemically.

2.5 Hysteresis loop isotherm

The formation of adsorption hysteresis loops can be related with the formation of micropores and mesopores, that allows for further textural characterization possibilities. Additional investigations of the isotherm hysteresis offer information regarding the pore structure and connectivity of the porous structure, which is crucial for adsorption capacity and transport qualities. Moreover, inert gas sorption methods employing for both low- and high-pressure measurements, may directly assess the amount of gas that can be stored in a nanostructure as well as its potential for gas adsorption-desorption isotherm. According to IUPAC categorization, there are four different types of isotherms for micropores and mesopores. In fact, IUPAC has classified the various forms of hysteresis loops with relatable shape. Type I demonstrates the usual hysteresis loop, whereas Type II demonstrates desorption pore blocking effects. These hysteresis loops show the micropores make up the majority of AC's pores. Type III displays hysteresis indicating desorption pore blocking effects, whereas Type IV displays hysteresis indicating cavitation in the desorption branch. These hysteresis loops characterize the majority of mesopores in AC.

2.6 Dye

Dye is one of the most widely used compounds that may colorize other substances and has been utilized in numerous sectors, including textiles, leather, paint, plastics, and paper. The visual, thermal, and physicochemical stability of these dyes is provided by their inorganic structures with complex aromatic structures. The complex aromatic structure of dyes makes them more persistent and hard to eliminate from effluents discharged into waterbodies. (Zaharia and Suteu, 2012). The presence of a chromophore group gives a dye its color. A chromophore group consists of conjugated double bonds in a radical configuration. In industry, Methyl orange (MO) and methylene blue (MB) are two colors found in high concentrations in textile effluent(Lau et al., 2015). MO is an anionic dye that contains the stable double bond N=N in its structure and is widely used in the printing, textile, culinary, and pharmaceutical industries, whereas MB is a cationic dye with adjacent aromatic rings that is used to color wool, cotton, and silk(Elgarahy et al., 2021). These colors have the potential to cause allergic eczema, skin conditions, mutations, and cancer. It is well recognized that color has a substantial impact on public perception of water quality. The first contaminant discovered in wastewater is the color. The presence of even low amounts of some colors in water, less than 1 ppm, is very apparent(Adegoke and Bello, 2015). Current techniques for removing dyes from industrial effluents are presented in Table 2.1(Foo and Hameed, 2011). Among these methods, adsorption is the most effective method for treating wastewater. AC is one of the most effective absorption materials for wastewater treatment. It has been determined that adsorption is more adaptable to other wastewater treatment approaches in terms of starting cost, simplicity of design, ease of operation, insensitivity to harmful compounds, and complete removal of colors even from dilute solutions(Elgarahy et al., 2021).

Table 2. 1: The current methods of dye removal industrial effluents(Foo and Hameed, 2011).

Method	**Method description**	**Advantages**	**Disadvantage**
Ozonation	Oxidation reaction using ozone gas	Application in gaseous state: no alteration volume	Short half-life (Max 20 min)
Electrokinetic coagulation	Addition of ferrous sulphate and ferric chloride	Economically feasible	High sludge production
Ion exchange	Ion exchange resin	Regeneration: no adsorbent loss	Not effective for all dyes
Membrane filtration	Physical Separation	Removal of all dye types	Concentrated sludge production
Activated Carbon	Dye removal by adsorption	Good removal of a wide variety of dye	Regeneration difficulties

2.7 Factor Affecting Activated Carbon Production

2.7.1 Raw material

The vast majority of carbon-rich organic compounds that do not merge during carbonization can be used to produce AC. Several parameters are considered throughout the feedstock selection process for the production of porous carbon. The elements are:

i) High carbon content
ii) Low in inorganic content (i.e., low ash)
iii) High density and sufficient volatile content
iv) The availability of raw material
v) Potential extent of activation

The most typically utilised precursor for the synthesis of activated carbon is lignocellulosic material. Coconut shell and palm oil are particularly common raw materials for various forms of AC, since their relatively high density, hardness, and volatile content make them excellent for AC synthesis(Jaya and Khair, 2021). On the other hand, low organic content is necessary to generate AC with a low ash percentage, a somewhat high volatile content is also required for process control. According to the previous study, lignin-derived charcoal has a greater total pore volume and a higher BET surface area compared to cellulose-based charcoal(Gao et al., 2020). In contrast, lignin has a lower reactivity than cellulose and therefore, it is as easier to activate precursor with high cellulose content. It is also found that, lignin activation produced mostly micropores, while during cellulose activation produce a mixture of pore size(Gao et al., 2020). Oil palm trunks fiber (OPTF), known as significant lignocellulosic-rich materials, contains high lignocellulose compounds. Table 2.2 shows the lignocellulose compounds in oil palm trunk(Jaya and Khair, 2021).

Table 2. 2: The lignocellulose compounds in oil palm trunk(Jaya and Khair, 2021).

Lignocellulose Compounds	**Contain (%)**
Lignin	12.50
Cellulose	25.88
Hemicellulose	16.40
Holocellulose	12.28
Pentose	10.06

Due to the high carbon content of lignin in OPTF, it has the potential to function as phenolic structure. It can be considered an ideal one precursor to activated carbon(Hussein et al., 2001). However, non-uniform sizes and shapes of the oil palm trunk represent a significant drawback to its use compared to the others biomass precursors and contribute to the high pre-production costs of activated carbon(Srivaro et al., 2018). In addition, the oil palm trunk has high ash content(Jaya and Khair, 2021).

Oil palm trunk contains a major microstructure of two tissues, which are the parenchyma (P) and vascular bundle (VB). The P has only a thin primary wall and no secondary wall, which fine powder forms. Along with vascular tissue, VB cells are referred to as transportation tissue(Kamyab, 2021). Vascular tissue is composed of various plant cells and is also known as a transport system in which granular particles form(Kamyab, 2021). Both tissues can be used as the precursor of activated carbon. However, the parenchyma showed higher extractive content than the vascular bundle as parenchyma contains a high percentage of lignin, which has high carbon content(Lamaming et al., 2015). Moreover, the fine powder provides much more surface area for adsorption than granular particles.

2.7.2 Chemical activation agent

The activation process is heavily reliant on activating agents, and the various chemicals utilized respond differently depending on the type of biomass and the temperatures used. Chemical activating agents' reactivity with biomass materials at elevated temperatures results in an intermolecular reaction that results in efficient AC generation. Surface textural investigation demonstrates that increasing the ratio of activating agents increases the efficiency proportionately until the optimum range is achieved, resulting in AC with increased pore capacity upon activation(Yang et al., 2020). Phosphoric acid (H_3PO_4) classified as an acid activating agent capable of removing carbon precursor contaminants, such as ketones, alcohols, acids, and aldehydes, which will enlarge the surface area of AC so that adsorption capacity will increase. H_3PO_4 might act as a chemical agent which facilitates the release of CO_2, an oxidant which reacts with carbon after dehydration, and a reagent which enters AC through C–O–P bonds(Li et al., 2015). According to the literature, the mechanism of chemical activation of H_3PO_4 on precursor surface involve three stages(Zięzio et al., 2020); Initial activation involves the breakdown of organic matter into semi-finished products with a lower molar mass and the release of volatile matter in gaseous compounds. In this step, H_3PO_4 begins to degrade, and precursors react with P_2O_5 to cause pores to open and expand.

2.7.3 Activation temperature

Temperature, especially the ultimate activation temperature, has an effect on the activated carbon's characteristics. Generally, for industrial activated carbon, the process is carried out at temperatures more than 800 ^{O}C in a steam/CO_2 combination. Recently, research has focused on improving the ultimate activation temperature in order to reduce manufacturing costs and activation times. According to various publications, activation temperature has a substantial effect on the yield and surface area of activated carbon. Temperatures as low as 200 ^{O}C and as high as 1100 ^{O}C were utilized.

The optimal temperatures have been found to be between 400 and 500 ^{O}C by the majority of prior studies, regardless of the activation period or impregnation ratio for various raw materials(Baysal et al., 2018). In prior research, the yield of activated carbon dropped when the activation temperature was increased from 400 to 600 °C. This outcome is due to the fast rate of interaction between carbon and activator chemicals, which results in the release of volatile components, as well as the growing textural features of activated carbon, which results in the formation of pores and carbon combustion(Maulina and Handika, 2019). Additionally, moisture content fell in lockstep with carbonization temperature owing to the shrinkage of activated carbon pores. As a consequence of this event, activated carbon's hygroscopic capacity is lowered(Maulina and Handika, 2019). With increasing carbonization temperature, the ash content of activated carbon rose. This result implies that when the carbonization temperature rises, the vaporization of particular volatile chemicals contained inside the particles increases as well(Maulina and Handika, 2019). Additionally, carbon combustion results in an increase in the ash content of activated carbon(Maulina and Handika, 2019). The volatile matter concentration dropped as the carbonization temperature increased, indicating that a greater carbonization temperature results in more complete breakdown of non-carbon molecules in activated carbon. According to prior study, when heat was applied to the precursor during the activation process, the functional groups from the raw material spectrum were released as volatile materials. This demonstrated that the activation process was successful. This result shows that volatile substances are being produced and carbon is being burned, resulting in a low volatile matter concentration.

2.7.4 Activation time

The activation time influences the carbonization process and activated carbon characteristics. According to prior research, the typical activation time for palm shell and coconut shell was between one and three hours(Tani et al., 2015). As time passed, the yield % declined significantly, but the BET surface area grew. This conclusion is possible because of the volatilization of organic molecules in raw materials, resulting in the creation of activated carbon. When excessive activation occurs, the amount of the drop in product yield is shown to be decreasing(Maulina and Handika, 2019).

2.7.5 Impregnation Ratio

The impregnation ratio (IR) is the weight ratio of chemical activation to biomass(Mistar et al., 2020). The IR increases when the activating agent concentration is increased. The impregnation ratio influences the activation process and its features. According to previous research, activated carbon with an impregnation ratio in the range of 1-3 is the most effective(Ziȩzio et al., 2020). By using H_3PO_4 as chemical agent, when the impregnation ratio is higher, the chemical activation provides more acidity for etching the surface and results in the pores becoming larger. Therefore, it may result information of porous structure. On the other hand, the volume and depth of activated carbon pores formed increases as the activator concentration increases. Eventually, this event will improve the hydroscopic properties of the material, allowing it to absorb water from the air(Tani et al., 2015). Additionally, the ash content rise in proportion to the high activator concentration. The high activator concentration increases the surface area of activated carbon, resulting in the development of more pores. Increased pores result in increased ash generation(Tani et al., 2015). A high activator concentration will dissolve the silica in the activator, increasing the activated carbon's surface area. Finally, this reaction results in an increase in pore formation(Tani et al., 2015).

2.8 Factor Affecting Activated Carbon Performance

2.8.1 Adsorption

Adsorption is the process by which adsorbate moves from a gas or liquid phase to a solid or liquid condensed phase and forms a monomolecular surface layer on the adsorbate. In an adsorption process, molecules, atoms, or ions in a gas or liquid diffuse to the surface of a solid, where they connect with the surface or are held by weak intermolecular forces. The adsorbate is the deposited solute, while the adsorbent is the solid surface on which adsorption occurs. Figure 2.2 shows the adsorption process. The Figure 2.2 represents that the adsorbate adsorbs on the surface of adsorbent.

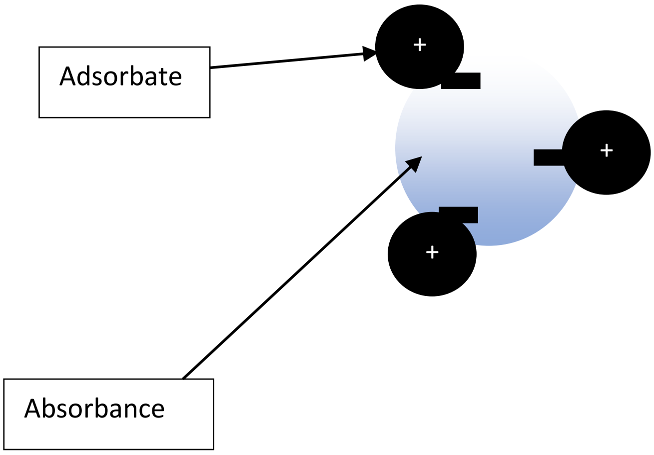

Figure 2. 2: Schematic diagram of adsorption process

To obtain high adsorbate removal, the adsorbent may have well-developed pores and a large surface area(Mistar et al., 2020). On the other hand, adsorption is the preferred method for dye removal due to its simplicity of operation and low cost. The adsorption process can be classified into two groups based on the different adsorption forces, which are physical adsorption and chemical adsorption. When intermolecular attractive interactions between solid and gas molecules are stronger than those between gas molecules, physical adsorption from a gas occurs. Physical adsorption is surface area dependent. As surface area grows, so does the extent of adsorption. For chemical adsorption, chemical bonds are formed between the adsorbent and the adsorbate in a monolayer, typically accompanied by a heat release greater than the heat of vaporization. Chemical adsorption is dependent on the surface functional group of AC. For chemical, adsorption, it involves formation of chemical bonds between adsorbent and adsorbate in a monolayer, frequently with a release of heat larger than the heat of vaporization. Using H_3PO_4 as chemical agent, the surface of activated carbon consists of H^+ that easily attracted with anionic adsorbate such as methyl orange, which consists of NH ions(Cavalcante et al., 2022). Therefore, adsorption efficiency of AC depends on activation process.

2.8.2 pH of solution

The pH solution of adsorbate influences the adsorption of activated carbon. Typically, the activated carbon should be washed with tap water to neutralize the pH. However, the effect of the adsorbate solution's pH on the adsorptive uptake of dyes is related to its effect on the surface binding sites of the adsorbent(Cavalcante et al., 2022). The pH of the adsorbate solutions affects the surface chemistry of the adsorbent and the ionic strength; hence, overall efficiency of an adsorption process is reliant on the pH of the solution. As pH of adsorbate solution decrease, the availability of additional hydrogen ions (H^+) increases(Cavalcante et al., 2022). In contrast, as the initial pH of the adsorbate solution increases, the positive surface charge decreases, and the number of negatively charged sites increases which provide more OH^- (Padmavathy et al., 2016).