

CHARACTERIZATION OF ACTIVATED CARBON FROM CORN COBS BASED ON POTASSIUM HYDROXIDE IMPURITIES AND TEMPERATURE

NUR IZZATY SHAFIQAH BINTI MOHAMED TARMIZI B092110025

BACHELOR OF MECHANICAL ENGINEERING TECHNOLOGY (REFRIGERATION AND AIR CONDITIONING SYSTEM) WITH HONOURS

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

Faculty of Mechanical Technology and Engineering

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DECLARATION

I declare that this Choose an item. entitled "Characterization of Activated Carbon from Corn Cobs based on Potassium Hydroxide Impurities and Temperature" is the result of my own research except as cited in the references. The Choose an item. has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

izzaty Signature : Name Nur Izzaty Shafiqah Binti Mohamed Tarmizi Date 10/01/2025

APPROVAL

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the Bachelor of Engineering Technology (Refrigeration and Air Conditioning Sytem) with Honours.



DEDICATION

To my beloved parents, Mohamed Tarmizi Bin Mohamed Nor, Norhawati Binti Salleh. Thank you for your gentleness in caring for me, supporting me, advising me, and loving me. Thank you to my supervisor, Ts. Dr. Amir Abdullah Bin Muhamad Damanhuri and also to my friends for helping me out. Thank you to the Faculty of Mechanical Technology and Engineering for providing the opportunity to use the machines and equipment to conduct this

research.

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ABSTRACT

Activated carbon (AC) is highly advantageous man-made material due to its advanced pore structure. It is used in air and water purification, gas separation, catalyst support, and energy storage electrodes. AC absorb contaminant from air and water, making it ideal for purifying these hazardous-contaminant. AC can be made from carbon-rich materials including coal, wood, and coconut shell. This study focusses on the development of activated carbon from corn cob. Factorial design methods were employed in this project which utilizes potassium hydroxide (KOH) activation process. Three factors were set to investigate significant contribution to the porosity and carbonization namely (A) KOH purities (90 - 95.5%), (B) temperature (200 °C - 500°C), and (C) KOH weight (10 - 100 gram). Scanning electron microscopy (SEM) and energy dispersive x-ray spectroscopy (EDX) were used to identify activated carbon's porosity, and carbonization element. In this study, sample 5 with (A) 95.5% KOH purities, (B) 500 °C temperature and (C) 10g KOH weight shows best activated carbon which is pores size in average distribution of 1.8 µm and the carbonization elemental composition which is 70.3%. Based on this study, activated carbon should have excellent adsorption and chemical removal properties. This study ensures that activated carbon meets these applications' criteria and performance standards.

ABSTRAK

Karbon teraktif (AC) adalah bahan buatan manusia yang sangat berfaedah kerana struktur liang termajunya. Ia digunakan dalam penulenan udara dan air, pemisahan gas, sokongan pemangkin, dan elektrod simpanan tenaga. AC menyerap bahan cemar daripada udara dan air, menjadikannya ideal untuk membersihkan bahan cemar berbahaya ini. AC boleh dibuat daripada bahan kaya karbon termasuk arang batu, kayu dan tempurung kelapa. Kajian ini memberi tumpuan kepada pembangunan karbon teraktif daripada tongkol jagung. Kaedah reka bentuk faktorial digunakan dalam projek ini yang menggunakan proses pengaktifan kalium hidroksida (KOH). Tiga faktor telah ditetapkan untuk menyiasat sumbangan penting kepada porositi dan pengkabonan iaitu (A) ketulenan KOH (90 - 95.5%), (B) suhu (200 °C - 500°C, dan (C) berat KOH (10 - 100 gram). Mikroskopi elektron pengimbasan (SEM) dan spektroskopi sinar-x penyebaran tenaga (EDX) digunakan untuk mengenal pasti karbon teraktif. keliangan, dan unsur pengkarbonan Dalam kajian ini, sampel 5 dengan (A) ketulenan KOH 95.5%, (B) suhu 500 °C dan (C) berat KOH 10g menunjukkan karbon teraktif terbaik iaitu saiz liang dalam nilai purata 1.8 µm dan komposisi unsur pengkarbonan iaitu 70.3%. memastikan bahawa karbon teraktif memenuhi kriteria dan piawaian prestasi aplikasi ini.

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LIST OF SYMBOLS AND ABBREVIATIONS

AC Activated Carbon _ PAC Powder Activated Carbon GAC Granular Activated Carbon _ Potassium Hydroxide KOH _ SEM Scanning Electron Microscopic _ S Energy of Dispersive X-ray EDX A Volatile Organic Compound VOC Design of Experiment DOE

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CHAPTER 1

INTRODUCTION

1.1 Background

Activated carbon (AC) is a versatile synthetic substance with a sophisticated pore arrangement, making it useful in various applications such as air and water purification, gas separation, catalyst support, and energy storage systems. Its exceptional capacity to absorb contaminants from the atmosphere and water makes it an excellent option for cleansing these resources. AC also serves as an efficient gas separator, transporting catalysts and promoting chemical reactions. It has been used as an electrode material in energy storage devices like supercapacitors and batteries due to its exceptional surface area and electrical conductivity. activated carbon is an incredibly versatile material that has found extensive use in various fields due to its unique physical and chemical properties (Kumar Misha et al, 2024).

The current process of AC utilizes CO^2 as the activation gas due to its cleanliness and ease of handling. CO^2 has also been commonly used as an activation gas in laboratory settings due to its ability to facilitate control over the activation process, as it reacts slowly at temperatures around 800 °C (T. Zhang et al., 2004).

Activated carbon (AC) is produced from carbon-rich materials like coal, wood, and coconut shell, with the highest adsorption forces and porosity globally. Available in granular, powder, and pellet forms, it has the most potent physical adsorption forces. The primary use of these pellets is in gas phase applications due to their ability to maintain low pressure, high durability, and minimal dust particles (Rahman et al., 2012). Pelletized activated carbon is characterized by its cylindrical shape, primarily used in gas phase applications due to its low pressure, high durability, and minimal dust particles. Figure 1.1 (a), (b) and (c) illustrates the

type of forms of activated carbon based on powdered, granular and pallet.



(c) Pellet Activated Carbon

Figure 1.1 Type of Activated carbon

These by products have the ability to produce high-quality AC with excellent absorption capabilities, impressive durability, and minimal ash content (Ioannidou & Zabaniotou, 2007). Activated carbon has the ability to capture contaminant, making it a valuable material for the control of air pollution and the treatment of waste water (Fan et al., 2004). Researchers have discovered a method to produce activated carbon from decomposed plant matter, which effectively enhancing water purification efficiency and cost-effectiveness. People are still looking for even more ways to make activated carbon from things that don't cost too much and can clean up lots of different kinds of dirt (Hussain et al., 2023).

Design of Experiment (DOE), is referred to as sample optimization, is a very strong tool for studying samples in a systematic and thorough manner, taking into consideration the potential internal problems that may arise. In the wide application of development, production, and research, DOE has emphasized that random experimentation yields random results, with an emphasis on controlled variables and experimental conditions (Ahmed Badr Eldin, 2011; Sundberg, 1994). Two important data factors of DOE associated software are replication run and repetitive run that allow multiple responses at the same factor level. It is a systematic approach to complement the advancement in the water purification process like activated carbon from decomposed plant matter. While effective, traditional activated carbon is costly and environmentally taxing, inspiring ongoing efforts to produce it more affordably and sustainably, using diverse raw materials to address various contaminants (Hussain et al., 2023). Together, these innovations promise more efficient and economical solutions to critical challenges.

1.2 Problem Statement

Activated carbon is a versatile substance used in various industrial sectors especially in water and air filtration, gas separation, and energy storage. However, current process of activated carbon and constraints its ability to address specific contaminant. To overcome these constraints, novel strategies are needed to develop advanced activated carbon materials with enhanced characteristics, cost efficiency, and sustainability.

Large quantities of corn cobs, a renewable agriculture waste product, are currently disposed of, creating a stain on landfill space and potentially leading to environmental issues. Conversion of this waste into a valuable product, like activated carbon, offer significant contribution in air filtration field.

Research and development efforts are needed to address rising pollution levels in air filtration which focusing on developing activated carbon compounds with enhanced adsorption

capacity and efficiency. Optimizing likewise materials (potassium hydroxide), activation procedures (carbonization), and post-treatment techniques is crucial to enhance AC capability. Heat or chemical treatments can enhance the adsorption capability, while improving the resistance to fouling and regeneration protocols can improve durability and lifespan. Activated carbon materials must possess specific qualities to address unique adsorption applications, such as improving surface chemistry and pore structure. Therefore, the optimization of activation parameters-activation temperature, KOH concentration, and activation time-is usually performed using a one-factor-at-a-time (OFAT) approach or even based on trial-and-error, which has several limitation and consume a lot of sample and time. Given that, the approach of design of experiments (DOE) specifically factorial design techniques will be adopted in this study. The application of this method should be able to reduce number of experiments while systematically identifying the interaction effects of variables to optimize the activation conditions for improved pore development and adsorption capacity in the production of activated carbon.

1.3 Research Objective

The main aim of this research is to develop the activated carbon from waste source based on KOH activation process. Specifically, the objectives are as follows:

- a) To characterize morphology and elemental composition of activated carbon from corn cobs using different impurities of potassium hydroxide (KOH) and different carbonization temperature based on factorial design method.
- b) To investigate interaction and significant factor between weight KOH, temperature and purities that contribute to the pores diameter and carbonization.

1.4 Scope of Research

vi.

The scope of this research are as follows:

- Corn cobs as a raw material is use in this project which collected from local supplier based on central region planting.
- ii. Chemical activation method by carbonization as potassium hydroxide (KOH) is main agent to produce activated carbon.
- iii. Activated carbon is produced specifically for respirator application where air filtration activity.
- iv. Morphological structure was analyzed using Scanning Electron Microscope (SEM).
- v. Energy Dispersive X-ray (EDX) were used to evaluate elemental composition of activated carbon made form corn cob.
 - Design expert version 13 were used to design factorial design method based of Design of Experiment (DOE) concepts.

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CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Exploring the use of inexpensive carbon-containing substances like lignocellulosic materials, biopolymers, coal, char, and fruit peels to produce activated carbons, which have diverse functional groups and are widely used in gas separation, solvent recovery, and water adsorption. (Jawad et al., 2019). Researchers are exploring the use of agricultural waste resources like almond tree dry leaves, spent tea leaves, pineapple leaves, mango leaves, dead Ashoka leaves, and aloe vera waste leaves for activated carbon production (Obayomi et al., 2022). In recent years, biochar, a carbon-rich substance, has garnered much interest and earned the nickname "black gold". Biochar, produced through pyrolysis or gasification in oxygen-restricted environments, offers renewability, high carbon content, accessibility, and stability, making it ideal for large-scale environmental operations. These applications include soil remediation, carbon sequestration, water and wastewater treatment, catalysts and activators, gas adsorbents and storage, supercapacitors, electrode materials, and electrode modifiers (Faraji & Saidi, 2023).

Nowadays, DOE software is often used as one of the alternatives for sample data collection. It is chosen because of its systematic method in the process of determining the relationship between the factors that affect a process and the output of the process. Moreover, it is very important as it helps in the input management process to optimize the output (Sundararajan, 2016). The DOE input methodology concept, is divided into 6 factors, namely, it can be hypothesis testing, interaction, controlled and uncontrolled, replication,

response, and blocking. The DOE input methodology concept, is divided into 6 factors, namely, it can be controlled and uncontrolled, response, hypothesis testing, blocking, replication, and interaction (Whitcomb, 2005). For design and analysis, there are two ways, the first way through comparison between two or more levels in a factor it serves to compare the variance between the ways of different factor levels with individual variance. Whereas, the second way is to have two levels or stages namely through the size of the experiment from other designs and the interaction of factor effectiveness. The latter method is more widely used in experiments because it can involve many factors, simple and versatile (Montgomery, 2013).

2.2 **Description** Physical Activation

In order to avoid the introduction of chemical impurities and contamination, it is more advantageous to activate carbon via physical methods rather than employing extra chemical processes. One way to accomplish this is by employing gentle oxidizing agents such as steam or carbon dioxide on biochar produced by biomass pyrolysis or gasification. While steam activation may be more suitable for certain types of biomass, carbon dioxide activation typically results in a higher specific area due to its promotion of micropore development (Colomba et al., 2022).

Activated carbon is primarily produced through two essential methods: gasification and physical activation. Gasification is the process of converting carbonaceous materials or biomass into syngas, which is a compound in the form of gas. Physical activation refers to the process of activating carbonaceous materials by exposing them to gases like steam or carbon dioxide at high temperatures. The utilization of these techniques is essential for the production of activated carbon with precise pore architectures and exceptional adsorption characteristics.

In the middle of gasification, the transformation of biomass or carbon-based substances

takes place through the application of thermochemical reactions, intermingled with oxygen or steam. The outcome yields syngas, containing carbon monoxide, hydrogen, and various other gases (Basu, 2013). Physical activation is a process that involves using activating chemicals like steam or carbon dioxide at high temperatures to change carbonaceous materials. This process eliminates volatile components and creates new pores, resulting in a porous structure. This method produces activated carbon with a vast surface area, interlinked pore network, and remarkable adsorption ability (Harry Marsh & Francisco Rodríguez-Reinoso, 2006). Figure 2.1 shows the physical activation process.



2.3 Chemical Activation

Activated carbon (AC) has gained popularity in recent years because to its desirable characteristics, such as a large surface area, well-defined porous structure, and enhanced surface functions. This permeable carbon material is extensively utilized as an adsorbent for the elimination of contaminants from wastewater and for the separation and purification of gases. In the chemical activation process the two steps are carried out simultaneously, with the precursor being mixed with chemical activating agents, as dehydrating agents and oxidants. Chemical activation, a single step process combining carbonization and activation at lower temperatures, offers advantages like improved porous structure, but raises environmental concerns (Ioannidou & Zabaniotou, 2007).

Chemical activation is a technique used to generate alternating current (AC). It entails combining activating substances such as sodium hydroxide, potassium carbonate, potassium hydroxide, phosphoric acid, or zinc chloride with the raw material to create a porous network. The chemical reaction between the activating agents and the precursor material results in increased surface area and pore volume, leading to advantages over physical activation methods such as higher carbon yields, larger surface areas, better development of porous structure, and the presence of surface functional groups (Kumar et al., 2022).

Chemical activation is superior to physical activation because it operates effectively at lower temperatures and requires less time. Factors such as concentration, temperature, and activation time significantly influence the organic acid reaction. This method prevents the formation of extra volatile organic compounds and increases the surface area of the activated carbon. Moreover, chemical activation offers an alternative to physical activation by producing adsorbents with unique physicochemical properties. Chemically activated carbon, unlike carbonized and physical activated carbon, exhibits a narrow micropore distribution, high surface area, and strong adsorption capacity.

Chemicals that are commonly used in chemical activation include alkalis like KOH, K2CO3, NaOH, and Na2CO3, as well as alkali earth metal salts such as AlCl3 and ZnCl2, and some acids like H3PO4 and H2SO4. These dehydrating agents play a crucial role in influencing the process of pyrolytic decomposition and preventing the formation of tar. Researchers have shown a particular interest in the effects of KOH and ZnCl2 on the carbonization of precursors, and ZnCl2 in particular is widely used in the production of activated carbon (Ozdemir et al., 2014).

The conventional method for manufacturing activated carbon (AC) often entails utilizing an external heat source such as an electric boiler. Nevertheless, microwave energy has experienced a surge in popularity in recent years, particularly in diverse scientific and industrial sectors. Despite the significant impact that microwaves have on carbonaceous materials, there are only a limited number of studies that explore the use of microwaves in the production and regeneration of AC (J. Li et al., 2010).





Figure 2.2 Chemical Activation Process (Sujiono et al., 2022)

2.4 Effect of parameters according to previous study

A parameter is something that will affect the sample during the electrospinning process. process. Parameter care is very essential. Without proper parameters, care will be affected by the sample-making process. If a sample is damaged owing to inappropriate care of the parameters, it shall can have a significant impact on other processes that use the sample. Table 2.1 shows the previous study of activated carbon.

N		
Authors & Year	Materials	Description/Finding
(Lua, A.C., and Yang, T.)	Coconut Shell	The effectiveness of KOH as an
2004.		activating agent for forming
		microporous structures was
		highlighted in the study.
(Xu, X., Cao, X., and Zhao, L.)	Biochar	Excellent adsorption capabilities of
2013.		the activated biochar were shown
		for organic contaminants and heavy
		metals.
(Wang, J., and Kaskel, S.)	Biopolymers	These materials worked well for gas
2012		storage and separation because of
		their large surface areas and high
		micropore concentration.
(Prahas, D., Kartika, Y.,	Banana Peel	The resultant activated carbon
Indraswati, N., and Ismadji,		showed promise for adsorption
S.) 2008		applications, especially the removal
		of colour from wastewater, with a
		large surface area and significant
		pore volume.

Table 2.1 Previous Study of activated carbon

2.5 Application of Activated Carbon

Most reviews on activated carbon have focused on its use in energy, catalytic, and liquid and gas phase applications. Nevertheless, the findings of our study can be beneficial in utilizing waste types for the production of activated carbon (AC), which serves as a very efficient adsorbent, especially in the field of water treatment and purification. Our research can serve as a valuable reference for enhancing AC processes to increase its efficiency and economic viability in water treatment, specifically in the elimination of heavy metals and other harmful substances (Muttil et al., 2023). Activated Carbon is a versatile substance that finds extensive applications in catalyst carriers, blood purification, supercapacitors, lifesaving clothing, purification of hazardous gases, and absorption of heavy metal pollutants. Its widespread use is a testament to its efficacy and reliability in diverse domains (Lan et al., 2023).

2.5.1 Coal Based Activation Carbon

Coal is a plant matter substance formed through decomposition and increased pressure and temperature. It consists of hydroaromatic groups and aromatic rings, linked by aliphatic and ether linkages. The degree of coalification affects carbon content, with geological movements affecting quality. Processing yields various products for power generation, fuel, and carbon materials. The utilization of coal for the creation of practical carbon materials has been recognized as an emerging area of study in the realms of coal chemistry and carbon science.

This has led to significant gains, but also presents challenges due to the complex composition and structure of coal. To overcome these challenges, molecular chemical engineering strategies must be employed to convert the aromatic basic structural units in coal into active precursors for constructing functional carbon materials (H. Li et al., 2022).

2.5.2 Removal of Heavy Metal

The extensive dissemination of harmful contaminants, consisting of metals and metalloids, in the environment as a result of industrialization and urbanization, is a critical concern. The existence of these detrimental compounds in aquatic environments offers a substantial peril to global human well-being. Although tiny quantities of specific metal ions are essential for biological functions, a high concentration of these ions can be harmful. However, hazardous metal ions like lead, cadmium, mercury, and arsenic are very detrimental to

biological processes and are classified as priority pollutants because of their significant toxicity, even at low concentrations. Addressing this issue is crucial to safeguarding human health (Pawar et al., 2018).

Metals in water can exist in various forms, including complex complexes and particles in solutions. The transport and dispersion of high-density metallic elements in water are primarily impacted by phenomena such as dilution, dispersion, sedimentation, and adsorption/desorption. The main contributors to water pollution are diverse human activities, such as the discharge of industrial and domestic wastewater, agricultural practices, air pollution, and the presence of outdated landfills. Exposure to heavy metals can exert detrimental effects on human health, specifically affecting vital functions such as the neurological system, liver, kidneys, and respiratory system. Additionally, environmental impacts of toxic metals can be severe, posing risks to wildlife health and leading to the accumulation of such metals in aquatic species like fish (Zbair et al., 2019).

2.5.3 Volatile Organic Compound (VOC) and Dyes Removal

Volatile organic compounds (VOCs), including benzene, toluene, and o-xylene, are recognized as hazardous air pollutants that can adversely affect human health and the environment. These substances are regarded as poisonous, mutagenic, and carcinogenic, underscoring the seriousness of the problem. Enhancing the efficacy of activated carbon in eliminating volatile organic compounds (VOCs) or dyes can be achieved by customizing its characteristics during the activation procedure. carbon for VOC or dye removal can be improved by tailoring its properties during the activation process. This allows for creating a more suitable pore structure and surface chemistry for the target contaminant (Vivo-Vilches et al., 2013). With the increasing concentration of volatile organic compounds (VOCs), regulations have gotten more stringent, creating an urgent requirement to decrease VOC

emissions. Dyes, however, are organic molecules that exhibit resistance to light, water, and antioxidants, rendering them recalcitrant to degradation upon their introduction into aquatic systems. The efficacy of adsorbent in eliminating dyes is primarily contingent upon the surface chemistry of the material. ACS plays a crucial role in the process of adsorbing and removing dyes. AC has been widely used to adsorb and recover a variety of VOCs, such as alkanes, alcohols, ethers, ketones, aldehydes, esters, and aromatics.

The ability of activated carbon (AC) to adsorb volatile organic compounds (VOCs) varies from a few to several hundred milligrams per gramme, according to a variety of applications. This is dependent on factors such as surface area, pore size, pore volume, chemical functional groups, as well as the properties of the VOCs such as their molecular size and polarity, and the adsorption conditions like temperature and moisture (X. Zhang et al., 2017). Physical adsorption mechanisms are the primary reason for the high adsorption capacity of AC with a large surface area and rich pore structure. In contrast, AC with chemical functional groups on its surface is an effective adsorbent for polar VOCs through chemical adsorption (Zbair et al., 2019).

2.5.4 Adsorption of Gas Storage

The adsorption of gases onto ACS is a favorable technique for storage because of its hydrophobic characteristics and capacity to remain stable in damp environments. An example of utilizing this technique is the extraction of carbon dioxide (CO2) from diverse origins, such as thermal power plants, with the aim of diminishing the quantity of CO2 present in the atmosphere. The adsorption capacity of ACS for CO2 is affected by its surface characteristics. The phenolic resin spheres undergo CO2 activation to form highly microporous carbon spheres. These carbon spheres have a substantial surface area of 2930 m2/g and a micropore volume of 1.12 cm3/g, with a diameter of 200nm. The carbon spheres exhibit efficient CO2

adsorption, with a significant absorption of 8.05 and 4.55mmol/g at 0 and 25°C at 1 bar pressure, respectively. At low CO2 pressures, the uptake capacity increases, resulting in an uptake of 1.42 mmol/g at 0.2 bar and 25°C (Tripathi, 2018).

The researchers synthesized sulfur-doped porous materials by sulfonating, oxidizing, carbonizing, and activating polystyrene divinylbenzene. After heating, sulfonic acid was introduced to the carbon materials in order to enhance the intermolecular bonding of the polymer molecules. The resultant materials have dimensions in the millimeter range.

2.5.5 Activated Carbon as Medical Use

The growing demand for medical and personal care goods has resulted in an increase in the quantity of pharmaceutical chemicals being discharged into the environment. These compounds are often employed and, because of their properties, they pose challenges for removal through conventional wastewater treatment procedures. As a result, they end up in water sources, causing significant environmental and health concerns (Rocha et al., 2020).

Recent research emphasizes the significance of water quality monitoring due to the over utilization of disinfectants, antibacterial agents, and medicines, resulting in an accumulation of persistent and organic waste in wastewater. To tackle this problem, activated carbons are increasingly being favored because of their efficacy in adsorption. These materials are extremely adaptable, productive, and economical, frequently derived from industrial waste. Researchers have conducted numerous studies on the synthesis of activated carbon using various biodegradable materials as alternatives (Januário et al., 2022).

Adsorption modelling is essential for process design and scalability, however the understanding of pharmaceutical adsorption mechanisms using standard models is typically inadequate. The Langmuir model has a limitation in that it assumes adsorption occurs through the formation of a single layer where one pharmaceutical molecule becomes trapped per active site, regardless of the specific adsorbent-pharmaceutical system. Nevertheless, the idea of monolayer formation during pharmaceutical adsorption may not hold true in various instances (Yazidi et al., 2019).

The existence of micropollutants, medicines, personal care products (PPCPs), and endocrine-disrupting chemicals (EDCs) in the environment is worrisome because of their potential for causing harm. These compounds provide challenges for conventional wastewater treatment methods due to their persistence and limited biodegradability, resulting in their ongoing discharge into the environment. Various studies have demonstrated the harmful impacts of medications and their interactions with other pollutants. For example, diclofenac, a common pharmaceutical, can interact with other inorganic and organic contaminants as well as its own metabolites, potentially creating new emerging contaminants (Zhao et al., 2023).

2.6 Advances and Future Perspective of Activated Carbon

Activated carbon has shown notable progress and shows excellent prospects for diverse applications. Surface modification techniques have become a crucial development, enabling the improvement of adsorption capacity and selectivity. Functional groups and nanoparticles have been introduced to activated carbon surfaces, boosting their efficiency in removing emerging contaminants from water and wastewater (Singla, 2019).

Additionally, innovative precursor materials and activation methods have enabled the production of activated carbons with unique properties, such as hierarchical pore structures and surface chemistries (Singla et al., 2019). Activated carbon has also found utility as a catalyst or catalyst support, synergizing adsorption and catalytic activities for applications such as biodiesel production, pollutant degradation, and energy conversion (Hu, 2013).

The combination of activated carbon with sophisticated technologies like as membrane filtration and electrochemical systems offers promising prospects for hybrid systems that exhibit enhanced performance and efficiency. Future research will focus on tailoring properties, developing sustainable precursors, optimizing activation methods, and exploring new activation techniques to further enhance the potential of activated carbon (Singla, 2019).

2.7 Research Design of Experiment

Design of Experiment (DOE) is statistical tool deployed in various types, process and product design, development and optimization. It is a multipurpose tool that can be used in various situations such as design for comparisons, variable screening, transfer function identification, optimization and robust design (Durakovic, 2017). Its use has also been widely used in the industry to assist in the decision-making process throughout the development of new products made, the overall manufacturing process of the product as well as product improvement. Furthermore, it is not only used in engineering but also medicine such as pharmaceuticals (Paulo & Santos, 2017) and hospitals, chromatography (Hibbert, 2012), administration, architecture (Schlueter & Geyer, 2018), marketing, energy, and food industries (Yu et al., 2018). DOE is a structured approach to investigating the relationship that exists between factors, or input variables, and the response or output of a process. The major objective of DOE is thus to identify, quantify, and optimize the relationships in order to improve performance, quality, or efficiency. The major components of DOE are factors, which represent controllable or manipulable variables such as temperature and pressure; levels, which are the different settings or values of factors, such as high and low temperatures responses, which are the measurable outcomes of an experiment, such as product yield; and interactions, in which the effect of one factor depends on the level of another.

Factorial design is a useful technique to investigate the main and interaction effects of the variables chosen in any design of experiment. This technique is helpful in investigating interaction effects of various independent variables on the dependent variable or process output (Rahman, 2018).

Among the common approaches in DOE, factorial design involves a study of all possible combinations of the levels of factors in order to study the individual effects and interactions of factors. Factorial designs are generally classified into two broad classes: full factorial and fractional factorial. Full factorial design studies the combination of all levels of factors and provides full information about main effects and interactions. Consider a simple example if there are two factors, A and B, each at two levels, the experiment would include four combinations (A low, B low), (A low, B high), (A high, B low), and (A high, B high). In contrast, a fractional factorial design evaluates only a subset of these combinations, reducing the number of experiments needed. This becomes particularly useful in the study of a large number of factors when not all interactions may be of interest.

Factorial designs are useful because they allow several factors and their interactions to be studied simultaneously with efficiency and precision. These designs also help in showing whether factors influence each other's performance, and thus a powerful understanding of the effects of the factors on the response is determined. This becomes very important during thesis research, as it provides one with a framework to study processes and optimize them in a systematic way.

2.8 Summary of Research

Activated carbon is essential in multiple sectors because of its extensive surface area and exceptional adsorption characteristics. Recent progress has focused on enhancing the adsorption capacity and selectivity of activated carbon by emphasizing its surface features, including the introduction of functional groups and nanoparticles.

As a result, the removal efficiency of emerging pollutants from water and wastewater has been enhanced. In addition, the use of novel precursor materials and activation techniques has resulted in the creation of activated carbons that possess distinct characteristics, such as hierarchical pore architectures and surface chemistries.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter aims to achieve the study's objectives by conducting a literature review and Design of Experiment (DOE). The experimental approach involves using raw materials like corn cob and potassium hydroxide (KOH) to carbonize the material. The temperature and duration of carbonization are determined, and the sample is prepared. The raw material is dried and pulverized to remove moisture. The carbon is then activated using an activating agent, which is then washed with distilled water and dried again. Efficiency and timeliness are crucial for successful research on activating agents. The AC sample is prepared, and the activated carbon is analyzed using laboratory techniques like Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray (EDX) to investigate its characteristics. The findings are meticulously examined, and a comprehensive presentation of the study is provided, along with a detailed description of the factors and components used in the research. Figure 3.1 illustrates the research flowchart:


Figure 3.1 Research Flowchart

3.2 Design of Experiment (DOE)

Design of Experiment (DOE) is a method of data analysis that assists the process of planning, conducting, analyzing, and interpreting controlled tests to determine factors influencing product quality, stability, or other key process attributes. Rather than experimenting with one parameter at a time, DOE can help speed up the process and identify important interactions by manipulating multiple factors at the same time. In the context of DOE, replication runs and repeated runs are both multiple response readings taken at the same factor level. The DOE also has a function of how to handle the case. The choice of replication or repeated runs can have a significant impact on the analysis of experimental data. In the case of replicas, showing an interaction between the two factors had a significant effect on the experimental sample. In the case of repetition, both factors and interactions were insignificant. When using multiple observations for all experimental treatments, all runs must be random (ReliaSoft Corporation, 2009).

In this study, 3 factors were applied to 8 samples. The first factor was conducted on samples with concentration of 90% and 95% of impurities. Meanwhile, second factor were conducted on samples with concentration of different temperature. The last factor was weight of KOH using ratio 1:1. Table 3.1 illustrates the Design of Experiment (DOE) table:

Std	Run	Factor 1	Factor 2	Factor 3
		A: KOH Purities	B: Temperature	C: KOH Weight
		(%)	(°C)	(g)
3	1	95	500	10
2	2	95.5	200	10
5	3	95	200	100
6	4	95.5	200	100
4	5	95.5	500	10
7	6	95	500	100
8	74	95.5	500	100
1	8	95	200	10

Table 3.1 Design of Experiment

3.3

Experimental Setup

This experiment must be carefully organized according with the planning outlined in the study flowchart. This experiment requires four crucial components the source material, activating agent, carbonization temperature, and carbonization duration. The experiment is well planned in accordance with the study flowchart, involving four main variables: source material, activating agent, carbonization temperature, and carbonization time. Among all these raw materials, corn cobs will be the major raw material used, collected from northern Malaysia. This cost-effective approach transforms agricultural waste into assets by making use of the ease of availability and the potential of agricultural byproducts in producing activated carbon with superior absorption, durability, and minimum ash residue. Potassium hydroxide (KOH) is used as an activating agent due to its strong alkaline properties, which assists in developing a highly porous structure (Ioannidou & Zabaniotou, 2007). Using KOH with 90% and 95.5% purity enables the production of activated carbon with a large surface area and pore size distribution, suitable for diverse adsorption applications (Dias, 2007). The carbonization process involves combustion in the absence of oxygen, using CO2 as the activation gas for its cleanliness, ease of handling, and ability to allow controlled activation at temperatures between 200–500 °C. The carbonization duration significantly impacts the properties of the activated carbon, with extended periods (30 minutes to 3 hours) leading to higher carbonization levels, reduced volatile matter, and increased carbon content. This enhances the activated carbon's surface area and adsorption efficiency, making it effective for various applications (Harry Marsh & Francisco Rodríguez-Reinoso, 2006).

3.4 Sample Preparation

Before starting the procedure, it is important to properly plan and highlight certain significant variables. The maize seeds and corn cobs must be manually separated. This method must be carried out while the maize is in a state of freshness. Subsequently, the corn cobs will be placed on the scale and their weight will be measured utilizing a weight scale. Additionally, it is necessary to closely monitor the corn cobs to maintain a correct separation between the corn seed and the cobs, and to prevent any seeds from being left on the cobs. Figure 3.2 illustrates the sample preparation:



Figure 3.2 Sample Preparation flowchart

3.4.1 Drying process (Raw Material)

The corn cobs as in the raw material process need be dried before the pyrolysis process happen. The researcher done this process about 72 hours according to (Sujiono et al., 2022). The proposed of this step is let the raw material (Corn Cobs) to be dried in the drying oven for 24 hours. This will let the raw material in dried condition with low moisture content and prevent from microorganism such as mold and fungus produce on the surface of the raw material (Corn Cobs).



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3.4.2 Crushing Process

After the drying process happens, the raw material will be proceeded by the process of crushing the corn cobs. The previous study done the crushing process 2mm to 3mm (Sujiono et al., 2022). The raw material will be crushed to a specific size range, preferably between 2 mm and 4 mm, or until it reaches a coarse powdery consistency. This task will be manageable due to the furnace's small capacity, which means that the powdered material does not take up much room.



Figure 3.4 Crushing Process

3.4.3 Sieving Process

Sieving plays an important part in both the production and purification of activated carbon. It enables the classification of particles into discrete size categories, ensuring that the final product meets stringent size criteria. Activated carbon can be manufactured with a constant particle size distribution by using the sieving method. This ensures that it has the right physical qualities to work optimally in various applications. From the past studies, the recommended the sieving size of 200 mesh (Sujiono et al., 2022). As we proposed the size of the mesh is 0.2mm - 0.5 mm to gain the activated carbon in the powder condition.



Figure 3.5 Sieving Process 26

3.4.4 Carbonization Process

Utilize an electrical boiler with a 3-phase power supply (415 volts) capable of reaching temperatures up to 1000 degrees Celsius. Carbonization is the pivotal starting point. Following the crushing process, the raw material derived from corn cobs will be introduced into the furnace, where it will be subjected to temperatures ranging from 200 °C to 500 °C. It is transformed into biochar. The correlation between the carbonization temperature and duration is a critical determinant in the manufacturing process of activated carbon. Increased temperatures and extended durations typically enhance the carbonization process, resulting in the creation of char that possesses favorable characteristics for the manufacture of activated carbon. However, the optimal temperature and duration conditions depend on various factors and require careful optimization to achieve the desired properties of the final activated carbon product (Harry Marsh & Francisco Rodríguez- Reinoso, 2006).



Figure 3.6 Carbonization Process

3.4.5 Activation Process

The biochar needs to be activating to become activated carbon. The process of creating pore structures in biomass-based carbon materials using KOH activation is a crucial step in determining the performance of activated carbon (Y. Zhang et al., 2022). When producing carbon materials from corncobs, it is crucial to meticulously evaluate the precise circumstances of KOH activation to get activated carbon with superior performance. Potassium hydroxide (KOH) is frequently employed as an activating agent during the activation process of activated carbon. This is because it is highly successful in generating a highly porous structure and improving the adsorption capabilities of the carbon.

Chemical agent	Composition	Types of
		Chemical
H3PO4	85%	Acidic
0	1000 mg/L	Alkaline
UNIVERSITI TEKI NaOH	10 to 300 ALAYSIA	MELAKA
	mg/L	
КОН	50%	Alkaline

Table 3.2 Composition of chemical to mix with activated carbon (Rahman et al., 2012)

3.4.6 Washing the Sample

The activated carbon sample must be rinsed with distilled water. When utilizing activated carbon in applications that need pH neutrality, such as water treatment or pharmaceutical operations, it is imperative to ensure the pH of the activated carbon is stabilized. An effective method to accomplish this is by rinsing it with distilled water. This process helps to eliminate any substances that could alter its pH and ensures that the activated carbon maintains the desired pH characteristics (Sujiono et al., 2022).

3.4.7 Drying Process

After the soaking and washing with distilled water. The sample needs to be dried once again in the oven at 130 °C for 3 hours (Sujiono et al., 2022). The objective of drying the sample is to remove moisture while preserving the structure and properties of the activated carbon. To prevent the carbon from undergoing thermal degradation or damage, it is necessary to carry out the drying process at a carefully regulated temperature to avoid overheating.

3.4.8 Sample of AC prepared

Once the sample is created, it is placed in a container and stored in a dry area to prevent any moisture from affecting the activated carbon's moisture content. Prior to the sample being subjected to observation or analysis in the study. The sample must be cooled down to reach the same temperature as the room, which is between 24 °C and 26 °C.

3.5 Characterization Study NIKAL MALAYSIA MELAKA

The activated carbon sample will go through characterization testing. This approach will assess the capacities of activated carbon created from waste sources, such as maize cobs. In addition, we suggested that the sample should undergo testing using Scanning Electron Microscopy (SEM) with the Hitachi model-SU 500 equipment in the mode named Secondary Electron Imaging-SEI and Energy Dispersive X-ray (EDX). This stage must be executed accurately to ensure that the obtained results are valid for discussion in the subsequent phase of this research. This stage can also identify the ash, volatile, and moisture content in the sample derived from the activated carbon of maize cobs.



Figure 3.7 Analysis Process by SEM machine

3.5.1 Coating

Coating is one of the important steps in preparing samples for Scanning Electron Microscope (SEM) analysis, especially for non-conductive or poorly conductive materials. The primary purpose of coating is to enhance the conductivity of the sample, which will prevent the charging of electrons on its surface during SEM imaging. Charging can distort the electron beam, leading to poor image quality. A conductive coating also improves the quality of secondary and backscattered electron signals, enhancing contrast and resolution of the SEM images. Coating also helps to dissipate heat from the electron beam an effect quite important for protecting delicate samples during high-magnification imaging.

The materials used for coating vary in type, chosen based on requirements for imaging. Traditional methods to treat non-conductive materials have been to either sputter coat or evaporate a thin film of metals such as gold, gold-palladium, or platinum, or to evaporate a thin layer of conductive carbon on the sample surface (*Sample Coating for SEM*, n.d.). Gold has been the material most often utilized for sputter coatings because it possesses relatively high conductivity and relatively small grain size and is suitable for high-resolution imaging. For EDX analysis, carbon is preferred and used as a general sputter coating material since neither does it interfere with the X-ray peak of any other element (Erik Luyk, 2019). The coating can be done by various methods of sputter coating, thermal evaporation, and electron beam evaporation. Sputter coating is the most common way to coat biological and non-conductive samples. It produces a very thin, even layer of conductive material.

Thin layer is usually defined to be in the range 5-50nm, that is, not thick enough to mask the surface morphology but still providing a conductive coating to dissipate charging artifacts, any resultant heat buildup, and minimize beam damage (*Sample Coating for SEM*, n.d.) This ensures that the coating is sufficiently thick to totally eliminate charging effects but not so thick as to obscure fine surface details. Only then can coated samples be tested in the SEM for highquality imaging and stability during analysis. Proper coating not only improves image clarity and contrast but also preserves structural integrity and the durability of the sample during imaging.



Figure 3.8 Coating Process

3.5.2 Porosity analysis

ImageJ is an open-source software released by NIH for image processing, which has grown to be quite versatile for general applications. Many scientists and research professionals find ImageJ particularly valuable in studying porosity, as its applications provide precise quantifications of pore sizes and distribution patterns along with their total porosity percentage. High-resolution images of the surface are normally taken through means of SEM or other highmagnification imaging, then uploaded into ImageJ. Resolution and contrast should be managed appropriately at the time of capture to ensure correct analyses. These images are subjected to preprocessing-grayscale conversion for enhancing the pores, binary thresholding to differentiate pores from the solid structure, and application of filters like the median or Gaussian for noise reduction to view clearly. Porosity measurement involves calculating the total pore area by summing all black regions in the binary image, determining the porosity percentage as the ratio of the pore area to the total sample area, and using tools like "Analyze Particles" to measure pore sizes and generate a size distribution histogram. Determination of the fractal dimension by plugins such as FracLac, or 3D reconstructions for volumetric analyses from micro-CT images, are higher-order analyses for the complexity and connectivity in pore networks. The program also provides various visualization options, including overlays and color-coded maps, with possibilities to export the results in graphical and tabular formats for statistical analysis. It is cost-effective, open source, customizable, has a wide community support system, and-most importantly-it can generate accurate and reproducible results for porosity studies in materials like activated carbon, which greatly helps researchers to optimize structural properties responsible for adsorption and other applications.

3.5.3 Element composition analysis

Energy Dispersive X-Ray Spectroscopy (EDX) is an analytical tool very common in detecting and identifying the elements present in a sample. It works on the principle of analyzing the unique X-rays given off by the elements excited by an electron beam, hence allowing researchers to deduce the presence and concentration level of elements present in a material. When the electron beam from a scanning electron microscope or a transmission electron microscope strikes a specimen, it may eject electrons from the inner shells of the atoms in the specimen. This creates a vacancy that is filled by an electron from a higher energy shell, and the energy difference between the two shells is emitted as an X-ray. Each element has a unique set of energy levels that emit characteristic X-rays with specific energies, serving as elemental signatures. The EDX detector collects these X-rays and creates a spectrum plotting intensity against energy. In this spectrum, the peaks represent specific elements, whose identification is therefore enabled, while the height of these peaks provides information on the relative abundance of elements present in the sample.



Figure 3.9 EDX element

3.6 Summary

Every process must be executed flawlessly in order to achieve optimal outcomes. If each step is performed incorrectly. The obtained result may not be satisfactory, as it can influence the outcome of the characterization study. The characterization study is crucial for comprehending the features and capabilities of the activated carbon used in air filtration. The constraint of this approach is that we can only determine the physical characteristics of activated carbon by utilizing maize cobs as the source material. Moreover, the inability to conduct the carbonization and activation process will result in the inability to produce samples for Scanning Electron Microscope (SEM) and Energy Dispersive X-ray (EDX)

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

The results would center on developing activated carbon for the attainment of specific properties and performance benchmarks. The main objectives include a large increase in the surface area for the purposes of augmenting the number of active adsorption sites, the optimum pore size distribution with a good balance of micropores, mesopores, and macropores to ensure efficient adsorption, and satisfactory capacity and effectiveness of activated carbon in terms of the high adsorption rate of removal of target substances. Testing would also be done to assess how well the activated carbon fits with such applicative domains as water treatment, air purification, gas separation, or energy storage to satisfy the standards of performance. The outlined results would define a guiding line and serve as means for evaluating the design process efficiency for varied industrial and environmental applications.

Activated carbon has been recognized as one of the most effective adsorbents for organic and inorganic pollutants in both aqueous and gaseous media. Such exceptional adsorption by activated carbon is attributed to its large specific surface area, huge pore volume, and great variation in surface chemistry, all of which depend largely on the type of raw materials and the method of activation. Among various methods, the processes of chemical activation have indeed demonstrated their viability in tailoring porosity and thereby enhancing the adsorption properties of activated carbons still, there is considerable interest in understanding the play of activation parameters to optimize the pore structure for specific applications.

Agricultural residues, such as corncobs, are one of the most abundant and inexpensive feedstocks for the production of activated carbon. Despite their potential, studies on corncobderived activated carbons remain scant. Chemical activation of corncobs leads to a highly porous material with a significant amount of mesopores, which is essential for the adsorption of larger molecules. Several investigations have confirmed the efficiency of activation agents for the development of microporous structures in corncob series-derived activated carbons. In most cases, previous investigations focus on the direct activation of raw biomass without considering the advantages that can be derived through a two-step process involving carbonization followed by activation.

The preparation and characterization of activated carbons derived from corncob char using KOH as an activation agent. In this work, we systematically vary the KOH to char ratio to demonstrate the fine control of structural properties in the resulting activated carbons, such as BET surface area, pore size distribution, and pore volume. Their adsorption performances were further tested for dyes and phenolic compounds in aqueous solutions. In the manuscript, scanning electron microscopy and adsorption isotherms revealed that the peculiar structural features evidenced these carbons as attractive materials in applications pertaining to environmental remediation and other sophisticated uses in supercapacitors.

The interrelation of the activation parameters with the carbon structure is an interesting platform for the design of high-performance activated carbons from renewable and low-cost materials. Besides opening a route to handle increasing municipal wastes in a more sustainable way, this approach also significantly furthers efforts toward versatile adsorbents that can meet wide-ranging demands in pollution control and energy storage.

4.2 **Design of Experiment (DOE)**

This table shows a DOE intended to study three factors-KOH purity, temperature, and KOH weight-on the variations of two responses: creation of pores and carbonization. Each line describes one experiment where the variables have, for each experiment, been intentionally and systematically changed from one experiment to the other in order to study their effects. It involves pure KOH, 90, 95.5, and 99% pure KOH at two temperatures 200° and 500°C (KOH weighing 10g and 100g, respectively).

The combined effect of these factors is reflected in the responses. For pore formation, the maximum observed value was 63.375 at 90% KOH purity, 200°C, and 10 g KOH weight. The same was the case with the highest carbonization of 70.3 with KOH purity of 95.5%, 500°C, and 10 g KOH weight. These data reflect that KOH purity significantly influences both responses; low KOH purity favors pore formation. Among the conditions for carbonization, temperature is the most important, and the higher the temperature, the better. The weight of KOH affects both responses but influences pore formation even more usually, lower weights give more pores. This systematic experimental design identifies the best combination of factors that provide the best results concerning pore formation and carbonization and thus provides insight into process optimization. Table 4.1 below show the Design of Experiment. The activated carbon well prepared as follows as the Design of Experiment (DOE). Figure 4.1 below shows the activated carbon prepared.

Std	Run	Factor 1	Factor 2	Factor 3	Response 1	Response 2
	-	A: KOH	В:	C: KOH Weight,	Pores	Carbonization
		Purities, %	Temperature, °C	g		
3	1	95	500	10	1.332	61
2	2	95.5	200	10	19.749	57.1
5	3	95	200	100	15.97	53.3
6	A AL	95.5	200	100	15.083	51.2
4	5	95.5	500	10	1.82	70.3
7	6	95	500	100	1.566	21.7
8	7	95.5	500	100	6.137	58.9
1	8/10	95	200	10	63.375	38.5
	ا ملاك	ىل ملىسى	کنگ	ستى ئەچ	اوينوم	

Table 4.1 Design of Experiment (DOE)

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Figure 4.1 Samples of activated carbon

4.3 **Pores size analysis of samples**

An important parameter influencing its adsorption capacity is the pore structure, which varies depending on activated carbon performance for the different applications. Scanning Electron Microscope-SEM techniques of samples for the study of the size and morphology of activated carbon pores were taken with the Hitachi model-SU 500 equipment in the mode named Secondary Electron Imaging SEI and that allows providing a very valuable morphological and topographic surface details end.

Tests by the machine were done at x10.0k, x1.00k, x5.00k, and x3.00k and 500 magnifications to allow both macroscale and microscale observations of the pore structure. Some key test parameters included the electron current, maintained at 10 kV, with the working distance maintained within 10 mm \pm 0.1 mm to ensure a sharp focus on high-resolution images. Sample to detector distances were 5 µm, 10 µm, 50 µm, and 100 µm, thus capturing the various perspectives of the surface features. The Figure 4.2below shows the result of SEM with the use of SEI and ImageJ and Table 4.1 below shows the value of the average for each sample.

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Figure 4.2 SEM Image

Experimental results are shown studying the effects of purities of KOH, carbonization temperature (°C), sample length, on a measured property. Data are summarized with averages for each experimental condition. Each sample is numbered to track/compare. The activating agent used was potassium hydroxide in two levels of purity, 90 % and 95.5%. The temperature set for carbonization was either at 200 °C or 500 °C. Individual measurements for each sample are outlined under the "Length" column, while their averages are under the "Average" column. From this table, one notices that with low KOH purity of 90% used in samples 1, 3, 6, and 8, there is a great variability in average measurement, reflecting the performance variability arising due to such factors as temperature and other ambient conditions.

From samples 2, 4, 5, and 7, the KOH purity of 95.5% generally showed close or the same value, which suggested that higher purity provides better homogeneity. Another crucial factor is the temperature; in samples 2, 3, 4, and 8, the 200°C samples frequently displayed an average value with the highest of sample 8 at 63.375. This could be interpreted as a function of lower carbonization temperatures yielding higher measured properties, possibly due to milder activation conditions that preserve more structure. By contrast, samples processed at 500°C-1, 5, 6, and 7-generally show lower average measurements, possibly reflecting the more aggressive carbonization in terms of material loss or denser structure formation.

The range of each sample gives a good indication of the variability within. For example, sample 1 has values ranging between 0.809 and 1.883, giving it an average of 1.332. Sample 8 also has a great range, from 52.453 to 128.257, which leads to its large average. This could be due to experimental inconsistencies, heterogeneity in the material, or actual differences in the activation behavior.

In general, the results suggest that the purity of KOH, temperature, and experimental factors are influential in the measured outcomes. The lower temperatures, 200°C, and specific

purities, 90%, tend to be favorable for higher averages, while higher temperatures, 500°C, tend to result in smaller averages. These could further be analyzed in relation to material properties such as porosity, yield, or adsorption capacity.

No Sample	KOH Purities %	Temperature °C	Length	Average
1	90	500	1.883	_
			0.809	_
			1.034	1.332
			1.797	_
			1.137	-
2	95.5	200	23.385	19.749
			16.112	
3	90 🏹	200	12.137	
			26.64	15.97
			9.132	-
4	95.5	200	14.111	
			22.856	
			10.359	15.083
			14.834	=
			13.253	
5	95.5	500 SIKAL MALAY	1.552	
			1.979	-
			\$ 1.834	LAKA 1.82
			1.419	_
			2.316	-
6	90	500	1.533	
			1.721	-
			1.112	1.566
			2.023	-
			1.443	-
7	95.5	500	4.827	
			6.533	-
			6.962	6.137
			5.087	-
			7.257	-
8	90	200	52.453	
			63.316	63.375
			72.85	-
			128.257	_

Table 4.2 Result of Scanning Electron Microscope (SEM)

The statistical analysis of the pore size in a sample under the influence of different factors is put into the following table KOH impurities (A), temperature (B), KOH weight (C), and their interactions (AB, AC, BC, and ABC). From the analysis, it's quite obvious that temperature B has the greatest influence on pore size, with a contribution of 44.7791% to the overall variation. With a highly negative standardized effect of -25.8305, temperature plays a very important role in controlling the activation process and pore formation. The contribution of KOH weight to the variation is 9.47202% with a standardized effect of -11.88, which shows moderate impact on the pore structure and activation level. By contrast, the impurities of KOH, A, are the smallest effects among the major factors, contributing only 6.52938% to the variation and a standardized effect of -9.8635, which proves that the purity of KOH plays only a minor role in the development of pores.

Also, there are some interactions that stand out. The interaction of temperature and KOH weight (BC) contributes the most at 13.4481% to underline the fact that the combined effect of these two parameters is much stronger than the separate effects and that these parameters should be optimized together. The interaction between KOH impurities and temperature (AB) has a contribution of 10.3077% with a standardized effect of 12.393, indicating that although impurities per se have very small effects, their interaction with temperature can appreciably affect pore size. Likewise, the interaction of KOH impurities with KOH weight (AC) contributes 9.1958% to the variation, indicating a moderate effect if these two factors are combined. Lastly, the three-way interaction of KOH impurities, temperature, and KOH weight (ABC) has the least contribution among the interactions at 6.26792%, which has a minor but not negligible effect on pore size.

Term	Standardized Effect	Sum of Square	% Contribution
A-KOH Impurities	-9.8635	194.577	6.52938
B-Temperature	-25.8305	1334.43	44.7791
C-KOH Weight	-11.88	282.269	9.47202
AB	12.393	307.173	10.3077
AC Solution	11.7055	274.037	9.1958
BC	14.1555	400.756	13.4481
ABC	-9.664	186.786	6.26792

Table 4.3 Pores Size Analysis of sample

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In the normal probability plot of the standardized effects, several key revelations concerning the factors affecting pore size variation in samples are obtained. The most significant negative factor is temperature, B, with a standardized effect of -25.83, showing that increased temperatures drastically reduce pore sizes; this is further emphasized by its contribution of 44.7791% to pore size variation. In contrast, the KOH weight (C) and KOH impurities (A) have moderate negative effects on pore size, with standardized effects of -11.88 and -9.86, respectively. Although their effect is not as pronounced as that of temperature, they do contribute to a decrease in pore size.

The positive interactions BC interaction between Temperature and KOH Weight-are colored orange and lie on the positive side of the x-axis with a standardized effect size of 14.16, indicating that it is synergistic in increasing pore size. This, therefore, calls for optimization of this interaction between the two variables.

Shapiro-Wilk test W-value = 0.715, and for P-value = 0.017, indicating deviation from perfect normality, so non-linear relationships by which the mentioned factors take part in controlling the pore size may be produced in some of these factors. These factors fall at the furthest rank, including Temperature B, BC interaction; accordingly, the bigger the influence or effect of temperature variation on pore dimensions. On the other hand, closer factors KOH weight, and KOH impurities (C and A), have very negligible relevance.

Conclusion, the major factor affecting the pore size is Temperature (B), and therefore this parameter needs careful control, whereas the interaction, such as BC, can potentially be optimized. The KOH weight (C) and KOH impurities (A) would also contribute in adjusting the pore size.



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AB interaction contributes 10.3077% with a standardized effect of 12.393, indicating a moderate impact. It means that the impurities as such have little effect, while in combination with temperature, a great effect regarding the pore size can be the result. AC interaction between impurities and weight contributes 9.1958%, which shows that while these factors have limited effects individually, their combination can moderately affect the pore structure. BC interaction of temperature and weight of KOH contributes 13.4481%. This result indicates that, compared to their individual effects, the effect of their synergy is more considerable, so one needs to work on both parameters in combination to obtain an optimum pore diameter. Figure 4.4 below show the interaction graph for pores size.





Figure 4.4 Pores analysis of AB, AC and BC

4.4 Element composition analysis of samples

Comparing sample 1 and sample 2 of activated carbon, significant differences in elemental composition appear, most probably due to different conditions of the activation process and efficiency of washing. Sample 1 consists of 43% carbon, 22.6% oxygen, 30.2% potassium, and 4.1% gold showing the retention of residues of activation agent (potassium from KOH) and trace metals, probably absorbed during the process. Sample 2 would be made up of 50% carbon and 50% oxygen, with undetectable traces of potassium and gold. This would then indicate that sample 2 had a far more extensive cleaning or purification step in which residual chemicals and metals had been washed off the material effectively. Or it may imply a modification in activation parameters of temperature, time, KOH ratio in sample 2, or a combination thereof to result in a minimum amount of residue retention.



Figure 4.5 Sample 1 of EDX result



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In the case of sample 3 and sample 4, the elemental constitutions of these activated carbons are different regarding conditions of activation and contamination. Thus, sample 3 was constituted by 38.9% carbon, 35.2% oxygen, 0.2% sodium, 24.3% potassium, and 1.5% gold, whereas sample 4 had a percentage of carbon lower at 35.5%, 33.4% oxygen, considerably higher potassium of 27.8%, and 3.3% gold. The presence of sodium, though in trace amounts, is unexpected in sample 3. This could be due to the contamination during its preparation, either from impurities of the raw material, corn cobs, or the used chemicals, or surrounding environmental factors affecting it during handling and storage. Sample 4 had a higher gold content than sample 3, which suggests that the way these samples absorbed or retained trace metals must have differed due to differences in porosity, surface area, or exposure conditions. These compositional variations underline the influence of activation parameters and the importance of maintaining strict control over the process to ensure consistency and minimize contamination.



Figure 4.7 Sample 3 of EDX result



Figure 4.8 Sample 4 of EDX result

The elemental composition of sample 5 and sample 6 of activated carbon shows marked differences, especially for carbon, oxygen, potassium, palladium, and gold. Palladium was not expected in either of the samples. Sample 5 contains 54% carbon, 22.5% oxygen, 18% potassium, 0.7% palladium, and 4.7% gold, reflecting a high carbon content and moderate retention of potassium and gold. In contrast, sample 6 contains a much lower carbon content of 21.7%, much higher oxygen at 42.7%, a much-increased potassium at 22.2%, and notably higher levels of palladium 2.3% and gold 11.1%. The sudden appearance of palladium during activation or handling, probably with the use of Pd-containing materials, equipment, or chemicals. Sample 6 could have been exposed for a longer time, or its higher oxygen content meant a better surface functionalization, which resulted in a more substantial absorption of palladium. Changes in the activation/washing step may also have led to better retention of the metal. Moreover, sample 6 had lower carbon content and higher oxygen content, indicating less efficiency during carbonization and the presence of oxygen-rich functional groups that might increase the adsorption of metals. These results highlight the importance of the tight control of

contamination sources and standardization of activation and handling procedures to achieve consistent results and avoid unwanted elemental inclusions.



Figure 4.10 Sample 6 of EDX result

The comparison of sample 7 and sample 8 of activated carbon shows marked differences in elemental composition, with distinct implications for their preparation and possible sources of contamination. Sample 7 contains 44.4% carbon, 32.3% oxygen, 21.1% potassium, and 2.3% niobium, while for sample 8, the carbon content is 24.3%, oxygen is higher at 35.1%, and potassium is remarkably high at 40.6%, there is no detectable amount of niobium or gold was found on either sample. The detection of niobium in Sample 7 is rather strange and points toward contamination, possibly from raw materials, activation chemicals, or equipment used in the process. For example, the presence of niobium may indicate that there was an introduction, either by way of an activation system involving materials or components that contained niobium or feedstock exposed to a niobium-rich environment. The absence of gold in the two samples reflects a different contamination or adsorption mechanism compared with previous samples. The activated carbon could, therefore, be exposed to other sources that contain gold; or there was some other form of variation on the surface chemistry that reduced its capacity for retaining trace metals such as gold.



Figure 4.11 Sample 7 of EDX result





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Table 4.4 Energy-dispersive X-ray (EDX) analysis results, giving the elemental composition of various samples. The table gives the weight percentage of a number of different elements identified in each sample, along with the atomic percentage. Major elements observed from samples include Carbon (C), Oxygen (O), and Potassium (K), while other elements detected but with less content, include Gold (Au), Palladium (Pd), and Niobium (Nb). Each sample represents a different combination of these elements, based on the material structure and experimental conditions. For example, Sample 1 is composed of major elements like Carbon, Potassium, and Oxygen, while Gold is in trace amounts, probably because of sample coating. Sample 2 has an equal distribution of Carbon and Oxygen, indicating that organic and inorganic compounds are well balanced within the sample composition. Sample 5 have a greater amount of Carbon and comprise Palladium and Gold, suggesting more the metal type. Sample 7 has a remarkable weight percentage of Niobium, which hints at the addition of some specific metals for certain purposes. EDX analysis showed that in most samples, Potassium plays an important role, and its weight percent lies in the range of 18% to 40.6%. The fact that Gold (Au) was constantly detected in different samples indicates that its presence is related to the preparation process. The general elemental analysis provides an idea about the possible applications these materials might have in catalysis, adsorption, or any other industrial process and that the samples could be organic-metallic composites.
	Sample	Element	Weight %	Atomic %	
-	1	Carbon, C	43	61.9	
		Oxygen, O	22.6	24.4	
		Potassium, K	30.2	13.3	
		Gold, Au	4.1	0.4	
-	2	Carbon, C	50	57.1	
		Oxygen, O	50	42.9	
	3	Carbon, C	38.9	53.3	
		Oxygen, O	35.2	36.2	
		Sodium, Na	0.2	0.1	
		Potassium, K	24.3	10.2	
ANI MA		Gold, Au	1.5	0.2	
	LAYSIA	Carbon, C	35.5	51.2	
		Oxygen, O	33.4	36.2	
		Potassium, K	27.8	12.3	
		Gold, Au	3.3	0.3	
	5	Carbon, C	54	70.3	
		Oxygen, O	22.5	22	
		Potassium, K	18	7.2	
		Palladium, Pd	0.7	0.1	
	<u> </u>	Gold, Au	4.7	0.4	
ملاک NIVE	6	Carbon, C	21.7	47.3	
		Oxygen, O	42.7	21.6	
		Potassium, K	22.2	29	
		Palladium, Pd	2.3	0.6	
	KƏH	Gold, Au	11.1	1.5	
	7	Carbon, C	44.4	58.9	
		Oxygen, O	32.3	32.1	
		Potassium, K	21.1	8.6	
		Niobium, Nb	2.3	0.4	
	8	Carbon, C	24.3	38.5	
		Oxygen, O	35.1	41.8	
		Potassium, K	40.6	19.7	

Table 4.4 Element Composition Analysis of sample

The following table represents a factorial experiment by which one looks at multiple factors and the interaction between them on a response of interest. In this case, the factors A - KOH Impurities, B – Temperature, C - KOH weight, with their interactions AB, AC, BC, and ABC. Standardized Effect, Sum of Squares, and % Contribution have been presented by columns and signify the order of magnitude of every factor that may have changed the response variable. From these factors, the one having the highest standardized effect of 15.75 with significant contribution 31.1993% is the main factor A - KOH impurities. C - KOH weight, also has an important role to play with an effect of -10.45 and a contribution of 13.7346%, while the effect of B (Temperature, 2.95) comes out to be relatively small, with a contribution of 1.09453%, indicating little influence. Among the interactions, BC (-14.9, 27.9226%) is a powerful influencing factor-meaning the interacting effect of both B (Temperature) and C (KOH Weight) would significantly affect the response.

Furthermore, the interaction of ABC is 12.15, 18.5668%, indicating that all three factors are important in combination. The AB interaction has a medium importance with 7.5, 7.07467%, while AC is the least influential with 1.8, 0.407501%. In summary, A, KOH Impurities, BC, and ABC interactions are the most influential factors B, Temperature, and AC interaction have the least influence. The results from this analysis will provide valuable information on process optimization, mainly focusing on the most influential parameters.

Term	Standardized Effect	Sum of Square	% Contribution
A-KOH Impurities	15.75	496.125	31.1993
B-Temperature	2.95	17.405	1.09453
C-KOH Weight	-10.45	218.405	13.7346
AB MALAYS/4	7.5	112.5	7.07467
AC	1.8	6.48	0.407501
BC	-14.9	444.02	27.9226
ABC	12.15	295.245	18.5668
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Table 4.5 Carbonization Analysis of samples

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This is plot of the standardized effects, showing the comparative importance of several factors and their interactions involved in carbonization. The x-axis represents the standardized effects, while the y-axis represents the cumulative probability on a normal scale. On the plot, the factors with orange-colored squares denote positive effects, while blue-colored squares denote negative effects. The red-colored diagonal line is just for reference and provides an indication of where effects would have fallen had they been purely random or not significant.

From the plot, the most important factor is C (KOH weight), as its blue square is furthest to the left, indicating that it drives the response variable down on average. The other significant negative effect belongs to the BC interaction - the interaction of Temperature and KOH weight. On the other side, the A (KOH purities) and the ABC interaction have large positive effects with orange squares far to the right. These factors contribute positively to the response variable and are crucial in optimizing outcomes. In contrast, Temperature (B) lies near the reference line, suggesting its minimal influence compared to other factors. Generally, from the plot, factor C (KOH weight) was mainly an acting negative factor it pointed out the positive contributions of factor A (KOH purities) and the interaction ABC, while the temperature seems to be less influential in the process. This visualization underlines how important it is to keep these factors under control with a view to getting an optimal outcome.



Figure 4.13 Carbonization Analysis



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The interactions between factors have a significant effect on carbonization and optimization of pore structure. The AB interaction (temperature and KOH purities) contributes 10.3077% with a standardized effect of 12.393, showing a moderate impact. While temperature and KOH purities individually have minimal effects, their combination enhances carbonization, with higher levels of both leading to increased carbonization due to their synergistic effect. The AC interaction of KOH purities and KOH weight contributes 9.1958%, which is a reasonable effect where increasing both factors at the same time improves carbonization. The maximum contribution is from the interaction of temperature and KOH weight in the BC interaction, which was 13.4481%. This means variation in both highly affects carbonization. The sloping surface and contour lines emphasize that the optimization of both parameters together yields the best results, since changing only one parameter is not sufficient to achieve the desired pore structure. Figure 4.14 below shows the interaction graph for carbonization.





Figure 4.14 Carbonization analysis of AB, AC and BC

4.5 Summary

Characterization of corn cobs-based activated carbon by using potassium hydroxide as the activating agent: how purity of KOH, carbonization temperature, and weight of KOH affect pore size and carbonization efficiency. Higher temperatures at 500°C and reasonable purity at 95.5% for KOH resulted in higher carbonization. Sample 5 had the highest carbonization efficiency at 70.3%. Larger pore sizes favored low temperatures at 200°C and lower purities of KOH at 90%, where Sample 8 had an average diameter of 63.375 µm. Besides, SEM analysis detailed morphology differences in pores, and EDX analysis detailed residual potassium, the more surprising trace metals contamination, or process inconsistency. In statistical analysis, it was confirmed that among the factors influencing pore size, the highest factor was temperature, followed by the interaction of temperature with KOH weight. These results confirm that optimization of the activation parameters is required for obtaining high-quality activated carbon, which is imperative in air and water filtration applications.

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CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The present research has successfully determined the feasibility of corn cobs for the production of activated carbon by systematic studies on the activation parameters, including purity of KOH, temperature, and weight. Indeed, these variables are found to strongly influence the characteristics of the prepared activated carbon regarding pore size distribution, carbonization yield, and elemental composition. Low KOH purity and low temperatures favored higher pore formation, creating an ideal structure for adsorption applications. On the other hand, high temperatures and a moderate weight of KOH optimized carbonization to higher carbon content with reduced impurities in the activated carbon.

Characterization through SEM and EDX illustrated the development of highly porous structures with variations in elemental composition. Some of the challenges that were observed included variability in pore size and elemental contamination, including residual potassium and unexpected metals such as palladium. Again, these results point to the need for tight control of activation parameters and post-processing techniques, especially washing, in terms of product consistency and performance reliability.

This is a promising area of research where corn cobs are to be considered a cost-effective and eco-friendly precursor for activated carbon, aiding in waste management and environmental sustainability. The optimization of activation conditions would allow the process to be tailored according to specific needs, increasing its applicability in air and water purification, energy storage, and industrial filtration systems. Future research should focus on refinements in activation techniques, improvements in contamination control, and performance testing of activated carbon under real conditions. This study lays a very strong foundation for further exploration and industrial-scale implementation that would address not only environmental but also economic challenges effectively.

5.2 **Recommendations**

For improvement for future activated carbon applications can be improved as follows:

i. Further refinement of the ratio between KOH and corn cobs and of the conditions for activation in such a way as to balance effectively pore formation against carbonization is also part of optimization of the activation parameters. Modelling and optimization might be performed by advanced statistical tools, such as response surface methodology.

ii. The advanced characterization techniques such as the BET surface area analysis,FTIR spectroscopy, and XRD would give, beyond SEM and EDX, detailed information on the structural and chemical properties of the activated carbon.

5.3 Project Potential

These results can be extended further to several other parameters to help researchers formulate network models for air filtration applications in diverse studies. The application of air filtration is important because it eliminates floating particles, pollutants, and microorganisms in the air that are harmful to both human health and ecosystems. In addition, filtration systems are very crucial in industries to maintain the quality of products and materials while protecting highly valued equipment from possible damage. Optimized activated carbon made from corn cobs in this study offers a cost-effective and sustainable solution to enhance air filtration efficiency across various domains.

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APPENDICES

NO. ACTIVITY WEEKS PSM 1 Image: 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 PSM 2 22 23 24 25 26 27 28 29 30 Tittle selection 1. Planning and research 2. Background, problem 3. statement Objective, scope 3. 4. Literature review (Study jurnal) Research on corn cobs 5. Flowchart 6. 7. Survey corn cobs Purchasing corn cobs 8. 9. Purchasing activating agent 10. Sample preparation Drying and crushing process 11 12. Carbonization and sieving process 13. Activation process Washing and drying process 14. 15. Product testing 16. Report preparation Presentation Weekly E-Logbook 17. 18. 19 Final report submission 1/NU

APPENDIX A Gantt Chart

ونيوم سيتي تيكنيكل مليسيا ملاك

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

APPENDIX B PORES SIZE FROM SCANNING ELECTRON MICROSCOPE (SEM)



Sample 2



Sample 4



Sample 6



Sample 8

APPENDIX C CARBONIZATION ELEMENT FROM ENERGY DISPERSIVE X-RAY



Sample 1



Sample 2



Sample 3

EDAX APEX test 1 Author: User Apex Creation: 12/18/2024 3:21:12 PM Sample Name: activated carbon Area 3 eZAF Quant Result - Analysis Uncertainty: 8.95 % Weight % Atomic % Element с 35.5 51.2 33.4 36.2 0 27.8 12.3 κ 3.3 0.3 Au /test 1/activated carbon/Area 3/Full Area 1/ kV: 10 Mag: 1000 Takeoff: 34.4 Live Time(s): 20 Amp Time(µs): 7.68 Resolution:(eV) 126.9 0 0.99K C 0.88K 0.77K 0.66K 0.55K 0.44K 0.33K 0.22K Au 0.11K Au 0.00K 0.00 1.34 2.01 0.67 2.68 3.35 4.02 4.69 5.36 6.03 Det: Octane Elect Super

Sample 4



Sample 5

EDAX APEX test 1 Author: User Apex Creation: 12/18/2024 3:38:20 PM Sample Name: activated carbon Area 5 eZAF Quant Result - Analysis Uncertainty: 9.02 % Weight % Atomic % Element 0 42.7 69.0 43.9 29.0 к Ρd 2.3 0.6 Au 11.1 1.5 /test 1/activated carbon/Area 5/Full Area 1/ Mag: 1000 Takeoff: 35.8 Live Time(s): 20 Amp Time(µs): 7.68 Resolution:(eV) 126.9 kV: 10 1.20K 1.08K 0.96K 0.84K 0.72K 0.60K 0.48K 0.36K 0.24K 0.12K Pd 0.00K 0.00 1.34 2.01 2.68 0.67 3.35 4.02 4.69 5.36 6.03 Det: Octane Elect Super

Sample 6



Sample 7



Sample 8

APPENDIX D TURNITIN

Turnitin Report PSM 1 Nur Izzaty

