ENHANCED CO_2 ADSORPTION AND DESORPTION IN CHEMICALLY ACTIVATED CARBON DERIVED FROM CORN COB

A Thesis Submitted to the Department of Environmental Science, Faculty of Science and Technology, Bangladesh University of Professionals for
Partial Fulfilments of the Requirements for the Degree of BSc in Environmental Science

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DEDICATION

This thesis paper is dedicated to my parents

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First and foremost, I would like to praise Allah the Almighty, the Most Gracious, and the Most Merciful for the blessings bestowed upon me throughout my studies and in the successful completion of this thesis. With deep gratitude and honor, I acknowledge my thesis supervisor, Professor Dr. Sirajul Hoque, Department of Soil, Water, and Environment, University of Dhaka. The constant support and guidance of my supervisor have significantly influenced exploration of my interested topic, while his professionalism has facilitated my academic work. The consistent motivation provided by him has been a guiding force throughout this journey. Sir has provided both professional guidance and shelter, consistently being accessible when required. I am deeply grateful for his invaluable contributions to my academic growth.

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DECLARATION

I hereby declare that the research work entitled **“Enhanced CO_2 Adsorption and Desorption in Chemically Activated Carbon Derived from Corn Cob”** has been carried out under the Department of Environmental Science, Faculty of Science and Technology, Bangladesh University of Professionals in fulfillment of the requirement for the Degree of BSc in Environmental Science. This thesis has been prepared utilizing original research findings derived from experiments conducted by me, supplemented by references from existing literature. The present work has not been previously submitted, either in part or in its entirety, to any other academic institution for the purpose of obtaining any other degree. **I also certify that this thesis does not contain any plagiarized content (Maximum 20%).**

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CERTIFICATE OF THE SUPERVISOR

This is to certify that Mirana Tahsin carried out her Thesis under my guidelines and supervision, and hence prepared the thesis entitled “**Enhanced CO_2 Adsorption and Desorption in Chemically Activated Carbon Derived from Corn Cob”.** So far, I am aware, the researcher duly acknowledged the other researchers’ materials and sources used in this work. Further, the thesis was not submitted to any other Universities or institutions for any other degree or diplomas.

It is thus recommended that the thesis be submitted to the Department of Environmental Science, Faculty of Science and Technology, Bangladesh University of Professionals, in fulfillment of the requirements for the award of the degree of BSc in Environmental Science. **I also certify that there is no plagiarized content in this thesis (Maximum 20%).**

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CERTIFICATE OF THE CO-SUPERVISOR

This is to certify that Mirana Tahsin carried out her Thesis in my laboratory of Institute of Fuel Research & Development (IFRD), BCSIR under my guidelines and supervision, and hence prepared the thesis entitled **“Enhanced CO_2 Adsorption and Desorption in Chemically Activated Carbon Derived from Corn Cob”**. Based on my knowledge the researcher appropriately acknowledged the materials and sources used from other researchers in this work. Additionally, the thesis was not submitted to any other universities or institutions for the purpose of obtaining any other degrees or diplomas.

Therefore, it is recommended that the thesis be submitted to the Department of Environmental Science, Faculty of Science and Technology, Bangladesh University of Professionals, in fulfillment of the requirements for the award of the degree of BSc in Environmental Science. **I also certify that there is no plagiarized content in this thesis (Maximum 20%).**

22/12/2023

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Abstract

Air pollution accelerates the process of global warming. CO_2 is the primary greenhouse gas responsible for global warming. The need to address global carbon emissions has led to the advancement of solid sorbents, such as activated carbons, for the purpose of CO_2 capture. The effectiveness of adsorption is influenced by the choice of preparation and source material. The cost-effectiveness of producing activated carbons for carbon capture from agricultural byproducts, such as corn cobs, has been demonstrated. This study utilises corn cob biomass, a cost-effective and readily available raw material, for the production of activated carbon. The biomass was subjected to activation using KOH at ratios of 1:1 and 1:3, at a temperature of 850°C. The Modified Activated Carbon (MAC) samples, namely MAC-1 and MAC-3, were produced by treating biochar with a 1:1 and 1:3 ratio of KOH solution, respectively. In contrast, the MAC-2 method involved the direct addition of solid KOH pellets to biochar in a 1:1 activation ratio. The adsorption experiments were conducted for durations of 5, 10, and 20 minutes, and were subsequently followed by a 60-minute desorption process at a temperature of 105°C. The adsorption rate initially increased from 5 to 10 minutes, followed by a gradual decrease until reaching CO_2 saturation at 20 minutes. The desorption of CO_2 exhibited a consistent decline throughout the selected adsorption time intervals. Both MAC-1 and MAC-2 reach their maximum adsorption levels after 10 minutes. Both samples exhibited peak desorption at 5 minutes. The CO_2 adsorption capacity of MAC-2 was found to be 1.523 mmol/g or 0.067 g/g, indicating the promising potential of activated carbon derived from corn cob.

Keywords: *Activated carbon, corn cobs, biochar, adsorption, desorption, carbon dioxide*

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List of Acronyms and Abbreviations

Acronym/Abbreviations	**Expressions**
BET	Brunauer-Emmett-Teller
FTIR	Fourier Transform Infrared
SEM	Scanning Electron Microscope
EDS	Energy Dispersive Spectroscopy
BCSIR	Bangladesh Council of Scientific and Industrial Research
IFRD	Institute of Fuel Research and Development

CHAPTER ONE

INTRODUCTION

Introduction

1.1. Background of the Study

Air pollution is a notable environmental challenge that significantly contributes to the broader problem of global warming. The primary factors responsible for this situation are greenhouse gases, including carbon dioxide (CO_2), methane (CH_4), chlorofluorocarbons (CFCs), and nitrous oxides (NOx). Additionally, indirect contributors such as sulphur dioxide (SO_2) also contribute to the occurrence of acid rain (Xu et al., 2009). This issue arises due to the emission of harmful gases and particulate matter, originating from both natural sources like volcanoes, and human activities such as the release of dust from industrial processes. Industrialization is a prominent human-driven factor that greatly contributes to the release of harmful substances. In response to the detrimental impacts of air pollution, nations across the globe have established specialized organizations to enforce international environmental regulations. Carbon dioxide (CO_2) is a significant greenhouse gas that has a significant impact on global climate change (Wang et al., 2015). Power plants are significant sources of industrial CO_2 emissions as they heavily depend on fossil fuels for generating electricity. The release of CO_2 into the atmosphere can lead to acid rain and the corrosion of transportation and storage infrastructure due to its acidic nature. Researchers are actively investigating new methods and materials to improve the efficiency of CO_2 capture technologies in order to address the urgent need to reduce carbon dioxide emissions (Promraksa & Rakmak, 2020). The International Energy Agency (IEA) highlights the crucial role of CO_2 Capture and Storage (CCS) technology in the pursuit of limiting global warming to less than 2°C. According to the IEA, projections indicate that by 2050, CCS technology could contribute approximately 19% towards the goal of reducing CO_2 emissions, highlighting its crucial role in addressing the global need for CO_2 reduction. The IPCC predicts a greater than 50% probability of global temperatures rising by 1.5°C or more from 2021 to 2040, based on analysis of different scenarios. If greenhouse gas emissions continue at their current rate, there is a concerning chance that this threshold could be exceeded sooner, potentially happening within the span of 2018 to 2037. n a hypothetical scenario characterized by substantial carbon emissions; it is projected that the average global temperature could witness a rise of 3.3 to 5.7°C by the year 2100 (Schumer, 2023). The illustration **Figure 1.1** below depicts the carbon budgets for global mean temperature targets of 1.5°C, 1.7°C, and 2°C.

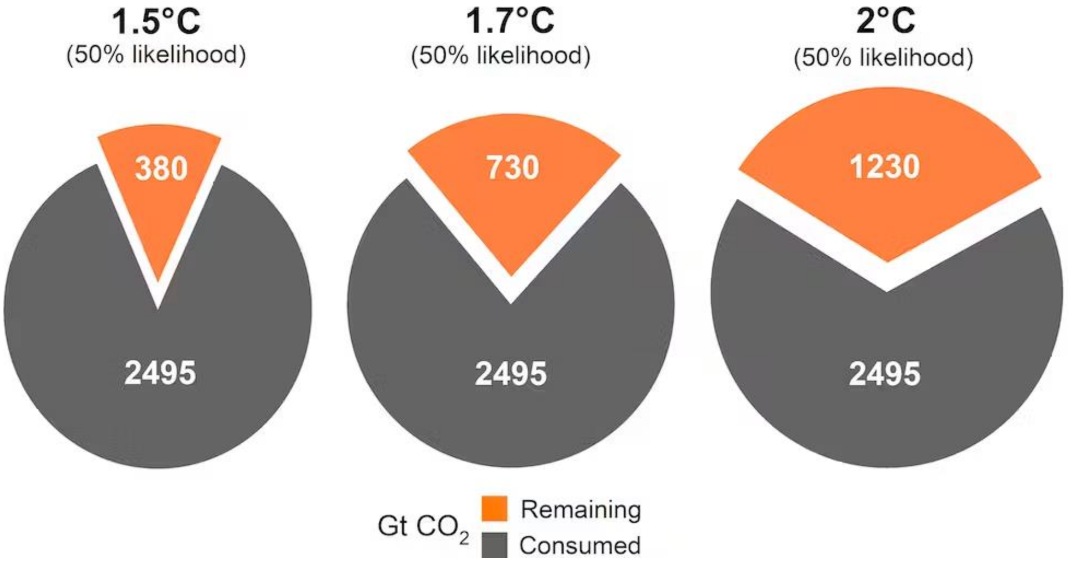


Fig 1.1. Global mean temperature carbon budgets to 1.5°C, 1.7°C, and 2°C

Dhaka is currently ranked as the second-most polluted city worldwide according to the latest World Air Quality Index (AQI) Report. The city's high population density requires significant dependence on personal transportation, particularly cars, motorcycles, and trucks, which increases its susceptibility to air pollution. Unfortunately, a significant number of these vehicles do not meet strict engine efficiency or fuel standards, thereby exacerbating air quality issues. In addition, Bangladesh has a significant number of brick-making kilns that operate primarily during the dry season and release a range of industrial pollutants. Coal and wood-powered kilns release particulate pollution, sulphur oxides, and volatile organic compounds. These kilns are also essential for cement production (UNEP, 2001). According to a study conducted by the Centre for Global Studies (CGS, 2023), the implementation of the metro rail system in Dhaka has worsened air pollution in the city. The investigation yielded a report emphasizing the worrisome impact. Hoque et al. (2020) highlight air pollution as a substantial environmental issue in Bangladesh. The country's environmental performance, as indicated by the Bangladesh Environmental Performance Index (2022), is below average, with a CO_2 growth ranking of 148 out of 180 neighbouring countries. **Figure 1.2** presents a bar graph depicting the trend of carbon dioxide (CO_2) emissions in Bangladesh over the period from 2013 to 2020.

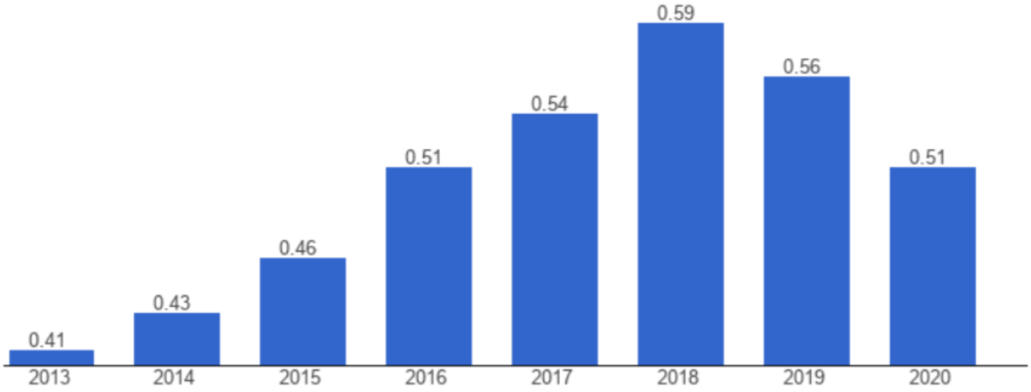


Fig 1.2. CO_2 emission per capita in Bangladesh

Extensive research has been aimed to mitigating and controlling CO_2 emissions due to their detrimental effects on the atmosphere. Rezaei et al. (2015) investigated the effectiveness of different adsorbents, including activated carbon (AC), zeolite, mesoporous silica, and metal oxides. Activated carbon (AC) is highly effective in adsorbing gases due to its physical properties, including a high adsorption capacity, large specific surface area, well-developed micro-pores, excellent repeatability, and ease of processing. Various capture methods, such as membranes, solvents, and solid sorbents, are used to separate and remove carbon dioxide from flue gas and waste gas streams. These methods are effective for material separation, absorption, and adsorption. Solvent-based absorption systems face significant technological challenges due to corrosion. However, emerging solid sorbents show promise in efficiently and reversibly capturing carbon dioxide from flue gas streams. These technologies have clear advantages compared to other methods of separating CO_2. They demonstrate features such as lower energy requirements for regeneration, a high capacity for capturing CO_2, selectivity, and user-friendly interfaces. The materials commonly used in carbon capture and storage include activated carbons (ACs), zeolites, metal-organic frameworks (MOFs), polymers, and metal oxides. These materials are used for adsorption to improve the efficiency of CO_2 adsorption (Pardakhti et al., 2019). Rochelle (2009) predicts that the most feasible technology for CO_2 capture before 2030 will be chemical absorption using aqueous alkanolamine solutions. Aqueous alkanolamine solutions pose challenges such as equipment corrosion, energy consumption during regeneration, and the requirement of a large absorber volume. Solid adsorption techniques are being investigated as a potential solution to address the challenges associated with chemical absorption. **Figure 1.3** illustrates a range of carbon dioxide (CO_2) capture methods.

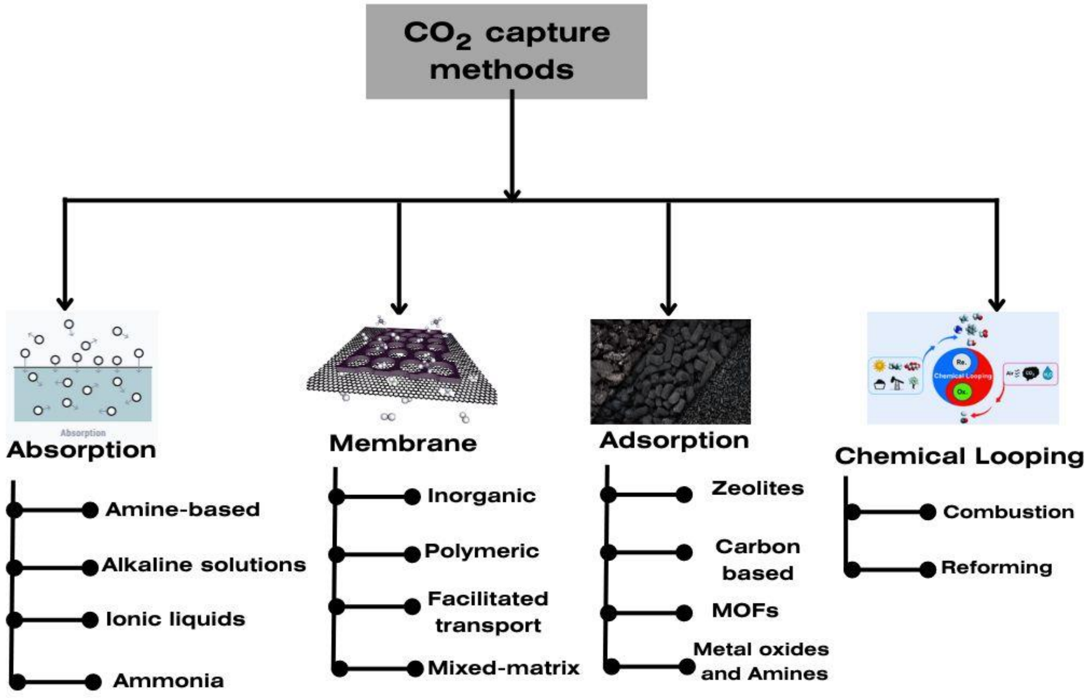


Fig 1.3. Various CO_2 capture methods

Researchers are increasingly studying solid adsorbent adsorption for potential performance enhancements and cost reductions in the medium to long term. The high cost of commercial activated carbon has limited its widespread usage, despite its impressive adsorption capabilities. Biochar, derived from waste biomass, shows promise as a viable alternative to commercial activated carbon due to its abundant availability, cost-effectiveness, and environmental sustainability. The main goal of this approach is to find a cost-effective and efficient adsorbent for capturing CO_2 (Cheng et al., 2013).

Biochar has gained significant attention due to its high carbon content, increased specific surface area, cation exchange capacity, nutrient retention ability, and stable structure. Sakhiya et al. (2020) suggest various applications of biochar in areas including agricultural productivity, water purification, alternative fuels, air purification, catalysts, and biogas production. **Figure 1.4** presented below demonstrates several applications of activated carbon.

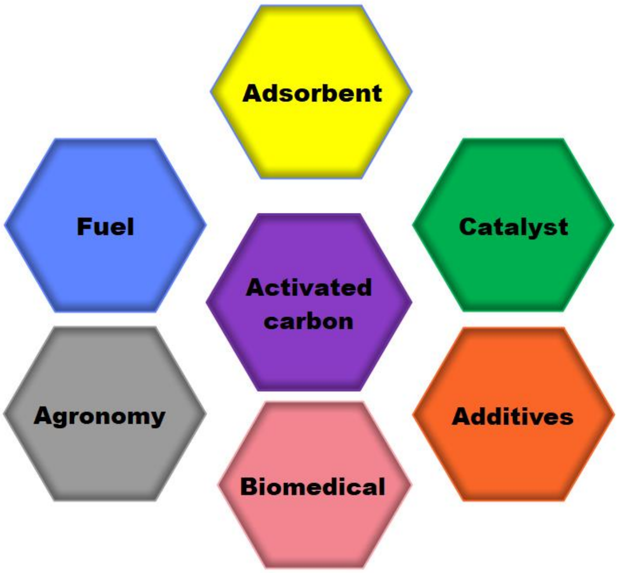


Fig 1.4. Application of Activated Carbon

There is a noticeable increase in academic interest in the field of biochar research. According to Sun et al. (2016), biochar is a type of activated carbon that possesses distinct properties including porosity, a large specific surface area, surface functional groups, and a high mineral content. Xu et al. (2016) investigated the adsorption capabilities of biochar derived from various sources, such as rice husk, sewage sludge, and dairy cow manure. Their study confirmed the inherent adsorption characteristics of these biochar materials. Several studies have examined the complex process of carbon integration into activated carbon. This material is known for its diverse functions, such as separation, purification, deodorization, storage, and catalysis of gaseous species. Tsai et al. (1998) attributed these functions to the microporous nature and large surface area of activated carbon. Active carbons have been extensively studied by various research teams as efficient sorbents for carbon dioxide, using both experimental and theoretical approaches. The motivation for this exploration stems from the wide availability and cost-effectiveness of activated carbon's primary components. Aksoylu et al. (2001) highlight the advantageous characteristics of activated carbons, including their low cost, chemical stability in acidic and basic environments, and economic benefits due to their ability to be regenerated and reused.

Dissanayake et al. (2020) examined two activation strategies: KOH activation and KOH-CO_2 activation. Both methods were found to significantly increase the CO_2 adsorption capacity of gasification biochar, regardless of the feedstock type. The effectiveness of these methods was consistently observed across different types of feedstocks. Extensive research has focused on increasing the surface area of carbon adsorbents to enhance gas adsorption and separation. Ahmadpour (2014) states that activated carbons can be synthesized using physical or chemical techniques, with the former being a commonly used method. Ben-Mansour et al. (2016) have suggested various activated carbons made from agricultural waste for the purpose of adsorbing CO_2 in both pure and mixed streams.

This study examines the utilization of corncob as a primary material for the production of activated carbon. The objective is to establish a relationship between the preparation conditions of activated carbon and the properties of carbon derived from biomass. The main goal is to thoroughly analyze microporous carbons and evaluate their ability to adsorb and desorb carbon dioxide. Our primary objective is to assess the capacity of this economically feasible carbonaceous material to reduce carbon dioxide levels through adsorption**.**

1.2. Problem Statement

Fossil fuels are widely recognized as the primary energy source in the foreseeable future, with a consensus that this trend will continue for at least 50 years. The emission of CO_2 from fossil fuel usage is a major driver of global climate change (AEO, 2015). The main sources of air pollution in Bangladesh include brick kilns, open-air burning, automotive emissions, riverbank sand fields, coal and biomass burning, and industrial emissions (Masum et al., 2020).

According to Dr. Salam, a renowned air quality specialist, there was a significant decrease of around 80% in air pollution caused by transportation during the early months of the COVID-19 pandemic until April of the previous year. Following the cessation of the nationwide lockdown, the situation progressively worsened (CGS, 2023). The rapid urbanization, industrialization, and motorization in cities in Bangladesh have been found to be major factors in the escalation of pollution levels. As a result, the increase in pollution has resulted in the release of more hazardous pollutants into the environment. Hoque et al. (2020) anticipate a significant rise in the occurrence of air pollution-related diseases, such as asthma, bronchial disease, pulmonary diseases, and lung cancer, in the coming years. This trend has important implications for public health. According to the World Health Organization (WHO), approximately 80% of urban residents are exposed to air pollution levels that surpass the recommended standards for acceptable air quality. The issue's significance is underscored by the discovery that a considerable proportion of cities in both low-middle income countries (98%) and high-income countries (56%) fail to meet the standards established by the World Health Organization (2016). This is applicable irrespective of the country's level of income. According to Masum and Pal (2020), their research project forecasts that the worldwide annual mortality rate caused by ambient air pollution will surpass 4.2 million people. This mortality is linked to various health conditions including cardiac arrest, cerebral stroke, cancer, and chronic respiratory diseases. Given that fossil fuels currently account for over 85% of global energy production, it is expected that air pollution will deteriorate. The combustion of fossil fuels emits carbon dioxide (CO_2), nitrogen oxides (NO, NO_2), and sulfur oxides (SO_2) into the atmosphere. The rising levels of carbon dioxide pose a significant concern due to their contribution to urban pollution and the acidification of precipitation. According to Lee et al. (2003), increased atmospheric concentrations of these gases can result in various health problems, including hair loss, throat irritation, vision impairment, respiratory disorders, and severe infections. Although certain CO_2 adsorption methods have demonstrated potential, additional research is necessary to investigate economically feasible alternatives for CO_2 replacement, despite the accompanying expenses.

1.3. Rationale of the Study

The major environmental impacts of greenhouse gases are primarily caused by carbon dioxide (CO_2). Examining the link between energy production and greenhouse gas emissions requires innovative technological solutions. Potential solutions can be explored in the domains of energy production and greenhouse gas capture and sequestration (Çağlayan & Aksoylu, 2013). Chemical absorption and adsorption are currently considered the most viable technologies for capturing CO_2 (Yu et al., 2012). This study aims to evaluate the feasibility of using activated carbon derived from corncob biochar for CO_2 mitigation. Our aim is to analyze the relationship between activation ratios, porosity generation, and surface chemistry, as these factors play a crucial role in the adsorption mechanisms of CO_2. The present study examines the adsorption characteristics of CO_2 gas on chemically activated carbon. It explores the impact of this modification on several properties of activated carbon, such as texture, surface functional groups, morphology, and thermal stability. The objective is to enhance the efficiency and performance of carbon dioxide (CO2) capture while analysing the desorption by employing activated carbon obtained from corncobs. The study's findings might have implications for future research in this field.

1.4. Research Questions

The following possible research question might arise:

- What is the most effective chemical activation ratio for producing high-quality activated carbon?
- To what extent is the efficacy of the generated activated carbon demonstrated in the adsorption and desorption of CO_2?

1.5. Research Objectives

The study aims to achieve the following specific objectives:

i. To assess the quality of activated carbon produced using different chemical activation ratios.
ii. To evaluate the efficacy of the activated carbon in adsorbing and desorbing CO_2.

1.6. Limitation of the Study

The key limitations of this investigation are briefly stated as follows:

- During the execution of experiments using a digital balance, it is susceptible to both human and instrumental errors to occur during the weighing process.
- The round trip from my residence to BCSIR covered 46 km and used to take 5-6 hours per day, posing a significant obstacle to research productivity.

1.7. Outline of the Study

This study is organized into seven chapters:

- **Chapter One: Introduction**

This chapter provides an overview of the study, including the background, problem statement, rationale, objectives, and research questions.

- **Chapter Two: Literature Review**

This chapter provides a review and summary of relevant local and international articles pertaining to the thesis topic. It also identifies any gaps in the existing research.

- **Chapter Three: Conceptual Framework**

This chapter presents the abstract representation of the research study, which is aligned with the research study goal of collecting and analyzing data.

- **Chapter Four: Methodology**

This chapter provides a comprehensive overview of data collection and data analysis methodology.

- **Chapter Five: Data Collection and Analysis**

In this chapter, the processes of data collection and data analysis have been carried out.

- **Chapter 6: Results and Discussion**

This chapter presents a detailed interpretation and discussion of the results obtained from the data analysis.

- **Chapter 7: Conclusion and Recommendations**

This chapter provides a summarized conclusion and recommendations for mitigating the problems.

- **Reference**

This section includes the references.

CHAPTER TWO

LITERATURE REVIEW

Literature Review

2.1. Literature Review

This chapter provides a comprehensive literature review revealing important insights into the current study and the existing research on the adsorption of carbon dioxide by activated carbons.

Activated carbon is widely used for separation and purification owing to its ability to adsorb different organic compounds. Activated carbon is the most commonly used adsorbent. The carbonaceous material is carbonized in an inert atmosphere and then activated in an oxidized environment to produce activated carbon. Volatile substances are eliminated through thermal decomposition, leading to the formation of fixed carbon (FC) with inherent pores. The duration of the activation process affects the formation of the porous carbon network (Ahmad & Idris, 2013).

2.1.1. Pre-treatment of biochar

Biochar is the residue formed from the carbonization of biomass in the absence of oxygen. The widespread use of this substance in soil treatment is attributed to its high carbon content and limited surface area (Ok et al., 2015). The current biomass drying process lacks energy recovery, resulting in the evaporation of moisture content. Biomass materials often have varying moisture content, which necessitates the use of pre-treatment methods to alter the properties of biochar prior to pyrolysis. Typical pre-treatment methods include soaking raw materials in a solution and decreasing the size of biomass particles. Specifically, the latter has been shown to improve biochar production. Pine wood biomass was subjected to pre-treatment using immersion in a mild acidic solution, resulting in significant reduction in tar levels (Yaashikaa et al., 2020).

Chen et al. (2019) conducted research on the effects of hydrochloric acid washing on sweet sorghum bagasse. The treatment aimed to reduce metal levels and increase the concentration of three main constituents in sweet sorghum bagasse. The acid treatment significantly influenced the surface structure of the cell walls, resulting in a porous and well-structured microstructure in the treated bagasse. The process effectively reduced metal content in the material while increasing the concentration of volatile substances.

2.1.2. Carbonization of biochar

The characteristics of activated carbon, including surface area and pore properties, are significantly affected by key factors such as carbonization temperature, duration, gas flow rate, and impregnation ratio, as discussed by Rahma et al. (2019). Varying the carbonization duration produces biochar and activated carbons with different pore sizes, suitable for diverse applications. Extended carbonization periods, lasting more than one to two hours, have no effect on temperature or chemical composition. It is advisable to restrict the carbonization of this material to temperatures exceeding 605°C, as this is the point at which the highest rate of change is observed and a slight reduction in weight occurs after a two-hour carbonization period. Recent research has shown that the conversion of biochar into activated carbon results in a decrease in hydrogen and oxygen content, while simultaneously increasing carbon and nitrogen levels. Hamza et al. (2015) found that carbonization and activation processes improve pore network expansion and increase pore spaces, as observed through scanning electron microscope analysis. Zhang et al. (2015) observed that carbonization temperature has a notable impact on the characteristics of biochar generated via pyrolysis.

Rahma et al. (2019) employed a horizontal tube furnace with a carbonization process conducted under an argon flow rate of 200 cc/min. The temperature was increased to 600°C and held for 3 hours. Elevated temperatures result in the deterioration of organic matter and the production of volatile compounds. Liu (2018) found that increasing the carbonization temperature of biochar leads to enhanced porosity, decreased yield, H/C and O/C ratios, and elevated pH and EC levels. The surface areas of chars obtained at temperatures of 800 and 900°C were measured to be 91 and 549 m^2/g, respectively. This indicates that the char produced at the higher temperature has a more favorable pore structure. Higher temperatures can reduce the activation time for burn-off and counteract the negative effects of longer activation times needed to achieve a larger surface area. Higher temperatures can improve the production of activated carbon from agricultural waste like corn cobs, resulting in greater adsorption capacity (Chang et al., 2000).

2.1.3. Activation of biochar

Activated carbon can be manufactured through either chemical or physical activation methods. In the carbonization process, carbonaceous materials undergo activation at temperatures ranging from 800 to 1000°C, achieved by oxidizing gases like carbon dioxide or steam. On the other hand, chemical activation involves the interaction between a precursor and a chemical agent, followed by pyrolysis in an oxygen-restricted environment at temperatures between 400 and 600°C. Biniak et al. (2001) highlighted the benefits of chemical activation, including the integration of carbonization and activation processes, lower operating temperatures, and an enhanced porous structure. Considering environmental impacts is crucial, and Tsa et al. (1997) illustrated the feasibility of obtaining additional chemicals. **Figure 2.1** illustrates the various ways employed for the activation of biochar.

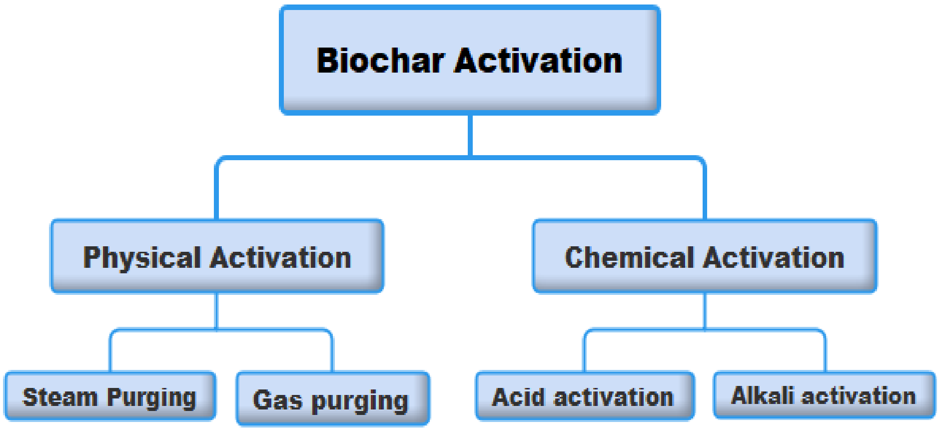


Fig 2.1. Types of biochar activation methods

Physical activation

In order to promote the inception of the physical activation process of char, oxidizing agents such as steam, carbon dioxide, or a mixture thereof are introduced at temperatures ranging from 700 to 950°C. During the course of this procedure, carbon atoms are removed from the substance, resulting in the production of $CO+H_2$ or CO (Rodríguez-Reinoso et al., 1995).

Activation of biochar with steam

Steam is widely recognized as the primary activator in the process. The method involves introducing steam into the char's internal structure, which triggers a carbon reaction resulting in pore enlargement. Azargohar (2009) suggests that the enlargement of pores during steam activation may lead to the deterioration of carbon pore walls, ultimately causing the creation of mesopores.

Figure 2.2 depicts a schematic diagram of the steam activation process. Biochar activation using steam was conducted in a fixed bed reactor made of Inconel, measuring 870 mm in length, 25.4 mm in outer diameter, and 22 mm in inner diameter. Applied Test System Inc. produced the 3210 series vertical furnace, which has a maximum temperature of 1200 degrees Celsius and includes a controlled reactor temperature feature. A K-type thermocouple was positioned at the bed's center for temperature calibration. The Eurotherm 2416 and Omega CN 7500 temperature controls were used to monitor the temperatures of the furnace and boiler. Genie Kent metering pumps were employed to introduce water into the boiler for steam production. The process is contained within a stainless-steel tube with aluminum lining and insulation.

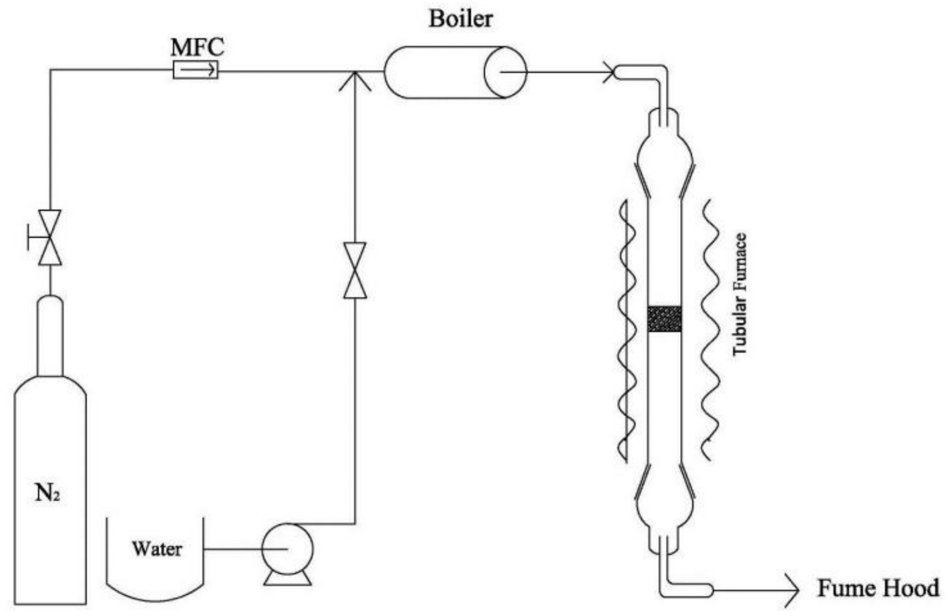


Fig 2.2. Schematic diagram of steam activation set up

<u>Activation of biochar with CO_2</u>

The process of CO_2 activation involves the utilization of carbon dioxide (CO_2) as an oxidizing agent to selectively remove carbon atoms, induce porosity, and generate carbon monoxide (CO) inside char materials (Azargohar, 2009).

Figure 2.3 depicts the continuous setup utilized for CO_2 biochar activation in the experiment. An Inconel fixed-bed reactor was used to activate biochar with carbon dioxide. The reactor had dimensions of 660 mm (length), 25.4 mm (outer diameter), 22 mm (inner diameter), and a total length of 25.4 mm. Applied Test System Inc. employed a 3210 series vertical furnace to control the temperature of the reactor at 1200°C. The Eurotherm 2416 temperature controller was used for boiler temperature regulation. The flow of argon and carbon dioxide was controlled using a Brooks Instrument 5850S A/B mass flow controller.

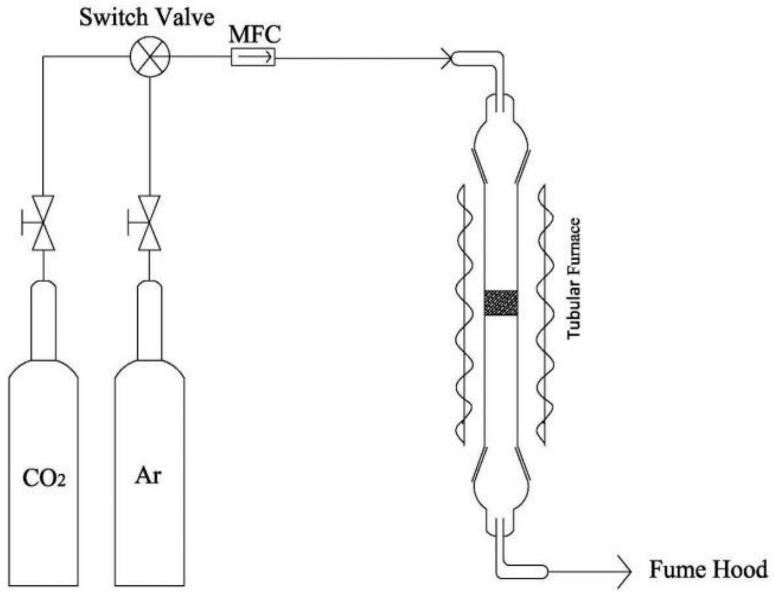


Fig 2.3. Schematic diagram of CO_2 activation set up

Chemical activation

The use of KOH and NaOH in chemical activation increases surface area and pore volume, resulting in activated carbon with well-developed micropores and mesopores (Azargohar, 2009). The KOH activation method, first introduced by Wennerberg and O'Grady in 1978 (Romanos et al., 2011), entails the combination of charcoal with a chemical activator. This mixture is then subjected to carbonization under an inert gas at temperatures between 400 and 900°C. The melting point of potassium hydroxide (KOH) is 360°C.

Chemical absorption is favored over physical absorption in post-combustion power plants for CO_2 capture, primarily due to the low CO_2 partial pressure in flue gas and the need to handle large quantities of CO_2. Several studies have investigated the impact of chemical modifications on high-surface-area solid materials in relation to their ability to enhance CO_2 adsorption and selectivity. This study focuses on the analysis of amines, organic compounds, alkali metals, alkali-earth metals, and inorganic metal oxides. Covalent bonds are established between acidic CO_2 molecules and modified basic active sites on the surface, which aids in the process of CO_2 adsorption. This interaction arises from the attraction between CO_2 molecules with acidic properties and active sites with basic properties (Yu et al., 2012). Chemical absorption is more efficient than physical absorption. Chemical activation is superior to steam activation in terms of porosity development, BET surface area, and total pore volume. Song et al. (2013) found chemical activation to be a cost-effective method. The activation of biochar improves its ability to adsorb heavy metals and polycyclic aromatic compounds. Several chemicals have been identified as activators, such as $ZnCl_2$ (Tsai et al., 1998), H_3PO_4 (Puziy et al., 2002), KOH (Guo et al., 2000), K_2CO_3 (Hayashi et al., 2002), and water vapor (Nakagawa et al., 2002).

The **Table 1** provided presents the textural characteristics of activated carbons produced from corn cobs at various carbonization and activation temperatures, as reported by Song et al. (2013). Previous studies conducted by Wang et al. (2020) and Rahma et al. (2019) commonly utilized a 1:3 ratio of corn cobs to KOH for activation. The activation process was conducted at a temperature of 800°C for a duration of 120 minutes, and was subsequently followed by carbonization at 400°C. This particular procedure resulted in the attainment of the highest BET surface area, measuring 1600 m^2g^{-1}. When the activation time was reduced to 90 minutes at the same temperature, the activated carbon exhibited a larger pore diameter of 2.16 nm when carbonized at 450°C.

Table 1. Corn cobs activated carbons based on their textural characteristics

Carbonation Temperature(°C)	**Activation Temperature(°C)**	**Activation Time (min)**	**Carbon/Alkali Ratio**	**BET surface area (m^2g^{-1})**	**Pore diameter (nm)**	**Pore volume (cm^3g^{-1})**
450	800	90	1:3	810	2.16	0.45
400	800	120	1:3	1600	2.01	0.62
500	850	90	1:3	932	2.12	0.49
600	900	120	1:3	855	2.14	0.46

According to Zaini and Kamaruddin (2013), the decision to undergo pre-carbonization is influenced by the chemical composition of the raw material and the activators used. Pre-carbonization, regardless of the atmosphere used (air or nitrogen), results in higher specific surface area and the formation of pores at an early stage. This enables convenient interaction between KOH molecules and the outer layer of the char. In addition, the activation level of the material increases when organic components undergo transformation into carbon structures upon treatment with KOH. The synthesized char demonstrates increased reactivity with Potassium Hydroxide (KOH), leading to the formation of more mesopores and micropores.

The lack of sufficient evidence makes it difficult to draw a definitive comparison between direct chemical activation and char-impregnated chemical activation. $ZnCl_2$ has been observed to enhance structure and crystallite size, while KOH is linked to optimal surface morphology. Rahma et al. (2019) found that decreasing the concentration of $ZnCl_2$ results in increased pore sizes in activated carbon. KOH-activated biochar exhibits a unique pore structure that enables clear visualization of the pore channels. Moreover, the activated carbon demonstrates a consistent and highly porous structure with a noticeable honeycomb pattern (G. Wang et al., 2020). Char-impregnated chemical activation is considered superior to direct chemical activation for producing activated carbon. The material obtained shows enhanced reactivity with potassium hydroxide (KOH), resulting in the formation of a greater quantity of mesopores and micropores (Hui & Zaini, 2015).

2.1.4. Chemistry of carbon dioxide

Carbon dioxide (CO_2) is a colorless and odorless gas with non-polar properties. Carbon dioxide is formed by the covalent bonding of one carbon atom with two oxygen atoms, utilizing a combined total of 16 valence electrons. The molecule exhibits C=O bonds with consistent lengths of 1.16 °A, and has a total diameter of 3.30 °A (Greenwood et al., 2012). Carbon dioxide (CO_2) is a nonpolar molecule despite the polar nature of its carbon-oxygen bonds, owing to its symmetrical molecular structure. The system exhibits a zero-dipole moment due to the close alignment of two dipoles with equal magnitudes. The infrared (IR) spectrum displays two vibrational bands: an antisymmetric stretching mode at 2350 cm^{-1} and a degenerate pair of bending modes at 667 cm^{-1} (House et al., 2004).

The CO_2 molecule possesses a significant quadrupole moment of -13.71E-40 Coulomb/m^2, resulting in the presence of overlapping positive charges. Wilcox (2012) states that the quadrupole moment interacts with the carbon lattice, aiding the gas molecule's penetration into the pores. Carbon dioxide has larger quadrupole moments compared to nitrogen, hydrogen, oxygen, and methane in flue gas. The disparity in quadrupole moments between carbon dioxide and activated carbon facilitates their effective interaction with each other (Liu & Wilcox, 2012). The physical properties of Carbon dioxide are presented in **Table 2** (Freund et al., 2006).

Table 2. Physical properties of CO_2

Molecular weight (g/mol)	44.01
Critical temperature (°C)	31.1
Critical pressure (bar)	73.9
Boiling point at 1.013 bar (°C)	-78.5
Specific volume at STP	0.506 m^3kg^{-1}
Gas density at STP	1.976 kgm^{-3}
Viscosity at STP	13.72μNsm^{-2}
pH of saturated CO_2 solutions	3.7

2.1.5. Adsorption of CO_2

Different types of activated carbon have been studied for their ability to adsorb CO_2 gas. This study investigated the adsorption of CO_2 on unmodified activated carbon (UMAC), acid-modified activated carbon (AMAC), and base-modified activated carbon (BMAC) derived from sugarcane bagasse. The adsorption was analyzed using gravimetric and weight differential measurement techniques. The morphological structures and surface functional groups of the adsorbent were assessed before and after adsorption. The findings Mohammed et al. (2017) suggest that BMAC has better pore structure and unique surface properties, leading to higher efficiency in CO_2 adsorption compared to AMAC and UMAC.

Chiang and Juang (2017) have indicated that alkali has the ability to enhance the CO_2 capture capabilities of carbon materials. In their study, Igalavithana et al. (2020) observed that the inclusion of KOH resulted in enhanced CO_2 extraction from food waste/wood biochar. Dissanayake et al. (2020) found that gasification of WCM biochar improved its CO_2 adsorption capacity, surface area, micropore volume, CO_2 selectivity, adsorption speed, and regeneration potential. Wood chips treated with potassium hydroxide (KOH) exhibited a significant tenfold augmentation in both surface area and porosity. The combination of wood chips and chicken dung resulted in a significant increase in surface area and porosity, with a fivefold enhancement observed. In their study, Drage et al. (2009) examined the efficacy of activated carbon adsorbents in removing CO_2 from syngas. They observed that these adsorbents demonstrated exceptional adsorption performance when subjected to high-pressure conditions.

Artificially synthesized activated carbon demonstrates notable CO_2 adsorption capabilities, which are influenced by factors such as surface area, composition, and inherent properties. Elevated temperatures can reduce adsorption capacity, while the adsorption rate is enhanced by higher concentrations of CO_2 and temperature. According to Balsamo et al. (2013), regenerated carbon retains its ability to adsorb CO_2 even after undergoing multiple cycles of adsorption and desorption. Ogungbenro et al. (2017) found that the optimal pyrolysis temperature for the physical activation method using date seeds was 800°C. Additionally, the highest CO_2 adsorption capacity was observed at 900°C. Activated carbons are economically efficient and exhibit high thermal stability, while also displaying reduced susceptibility to moisture. Temperature increases can reduce CO_2 uptake because the adsorption process is exothermic (Samanta et al., 2011). In a study conducted by Zhang et al. (2004), it was observed that the addition of polyethyleneimine (PEI) to activated carbon resulted in an increased capacity for adsorbing CO_2. Li and Xiao (2019) found that rice husk char, treated with K_2CO_3 and activated with CO_2, exhibited comparable CO_2 adsorption capabilities to carbons activated with KOH. **Figure 2.4** displays a composition diagram depicting the typical process of adsorption and desorption for CO_2 collection, as presented by Aouini et al. (2012).

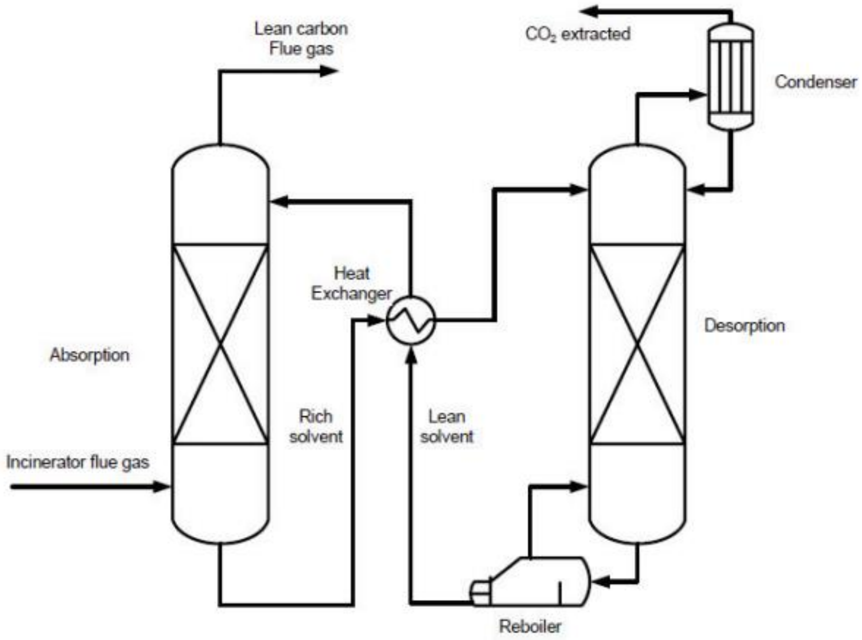


Fig 2.4. Typical CO_2 capture adsorption and desorption composition diagram

2.2. Research Gap

Previous literature and investigations indicate that acid digestion with HCl was not employed in the preparation of biochar from corn cobs. Instead, it was used after the carbonization of the biochar. Raw carbonaceous materials, such as wood, coconut shells, peat, coal, or corn cobs, typically contain impurities like minerals, ash, and non-carbonaceous components. The removal of these impurities can occur during material processing. HCl digestion facilitates the dissolution and removal of contaminants. The addition of HCl to carbonaceous material leads to a reaction with the minerals present, resulting in the formation of soluble chlorides. Following the completion of the reaction, a purified carbon residue remains. Moreover, it can lead to an increased tar content. The presence of tar in biochar can adversely affect its quality and suitability for specific applications. Tar can fill the pores of biochar, reducing its surface area and ability to adsorb substances. Tar can contain hazardous substances, including polycyclic aromatic hydrocarbons (PAHs) and other toxic compounds. Acid digestion or treatment can potentially decrease tar levels. Previous studies have investigated the carbonization of corn cobs to produce biochar at temperatures of 450°C (Cao et al., 2006) and

600°C (G. Wang et al., 2020) (Song et al., 2013). However, there is currently no available research on the carbonization preparation of biochar from corn cobs at 800°C. Most investigations on chemical activation of activated carbon did not include post-pyrolysis washing. Washing the activated carbon is a crucial step in the activation process following pyrolysis. Activated carbon can be produced from carbonaceous materials like coal, wood, or coconut husks and corn cobs by subjecting them to heating and the application of different chemicals. The washing stage aims to eliminate contaminants, residual chemicals, and fine particles from the surface of the activated carbon. Washing is necessary to eliminate any residual chemical activation agents, such as hydrochloric acid (HCl) or potassium hydroxide (KOH), that may remain after the activation process. The presence of residues can negatively impact the quality of activated carbon products, especially if they are released into the environment.

CHAPTER THREE

CONCEPTUAL FRAMEWORK

Conceptual Framework

3.1. Preface

This study aims to address a significant problem through a systematic process of identifying the issue and developing strategies for improvement. A thorough review of the literature has established the foundation and rationale for this research. Navigating unknown areas, developing research questions, and setting clear objectives to guide efforts in reducing adverse impacts. During the study's course, a series of experiments were conducted to study the adsorption and desorption of CO_2. These experiments involved the production of activated carbon using corn cobs as the raw material. This study will investigate the field of data processing, analysis, and interpretation, leading to a discussion and summary of important findings.

3.2. Conceptual Framework

The framework has been specifically developed to facilitate the execution of research activities by adhering to the sequential procedures outlined as follows in **Figure 3.1**.

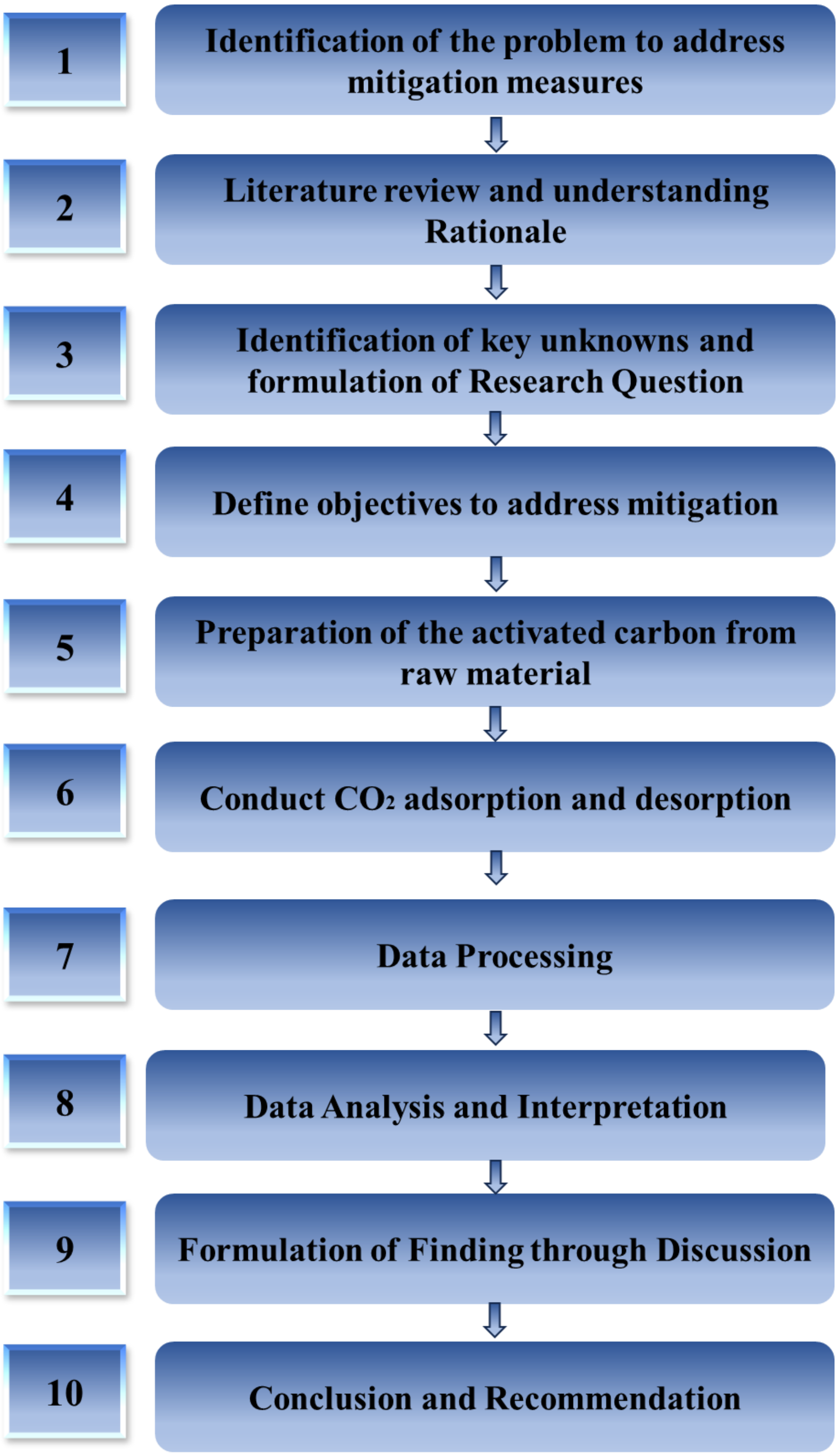


Fig 3.1. Conceptual Framework

CHAPTER FOUR

METHODOLOGY

Methodology

3.1. Chemicals and materials

Corn cobs were collected from Kawran Bazar, Dhaka for the purpose of preparing activated carbon. All chemicals used in this study were of GR grade and were sourced from Germany. Additionally, all compounds were accessible at the BCSIR and used without further modification. The chemicals, together with their respective origins, are presented in **Table 3.**

Table 3. List of chemicals used in this study

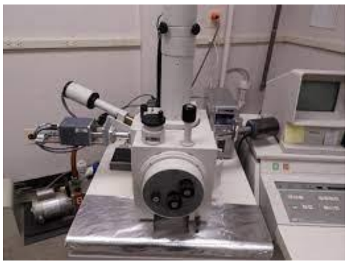
Name	Source	Purity
KOH (Potassium hydroxide)	Merck (Germany)	GR grade (>99%)
HCl (Hydrochloric acid)	Merck (Germany)	Purity: 37%
Water		Distilled

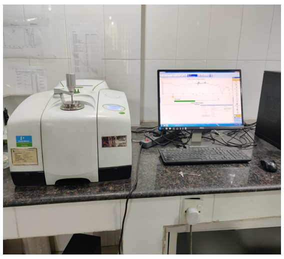
3.1.2 Instruments

Every instrument was accessible and available at the Institute of Fuel Research and Development (IFRD) department of BCSIR. **Table 4** presents a comprehensive compilation of the instruments employed in the current study.

Table 4. List of instruments used in this study

Blender	The blender (Philips-7710, Netherlands) was used to crush the corncob	 **Fig 4.1. Philips blender**
Oven	The oven (Memmert-UN 55 in Germany) was used at 105°C to dry the samples	 **Fig 4.2. Memmert oven**
Tube Furnace	Tube Furnace (Nabertherm, Germany) was used for carbonization and pyrolysis (800-850°C)	 **Fig 4.3. Tube Furnace**

Digital Balance	Digital balance (Shimadzu, Philippines) was used for measurement purposes	 **Fig 4.4. Digital balance**
Scanning Electron Microscope (SEM)	Scanning Electron Microscope **(**EVO-18, Carl Zeiss, Germany) for surface characterization	 **Fig 4.5. SEM machine**
Energy Dispersed Spectroscopy (EDS)	Energy dispersive spectroscopy analysis (EDS; JEOL 7610F, Japan) for EDS analysis	 **Fig 4.6. EDS machine**

Fourier Transform-Infrared Spectroscopy (FTIR)	Fourier Transform-Infrared Spectroscopy (Frontier, PerkinElmer, UK) was performed for characterization	 **Fig 4.7. FTIR machine**

3.2. Preparation of 5% HCl

In the laboratory, a 37% hydrochloric acid (HCl) solution was initially present and subsequently diluted to a 5% concentration for experimental use. A 5% hydrochloric acid (HCl) solution was prepared using a standard dilution method. A 100 ml of a 37% hydrochloric acid (HCl) solution was added to with 640 ml of distilled water.

3.3. Biochar Preparation

The biochar was produced by implementing slight modifications to the methodology provided by Roschat et al. (2016) and Hossain et al. (2021). Initially, the corn cobs were cut into smaller, more manageable pieces. Next, the pieces were blended using a blender (Philips-7710, Netherlands) and later subjected to a drying process in an oven (Memmert-UN 55, Germany) at a temperature of 105°C, for a duration of 24 hours.

In order to enhance the acid digestion process, the powdered corn cobs was pretreated with a previously prepared 5% (1.4 M) HCl solution. This pretreatment was conducted in a water bath (Memmert, Germany) at a temperature of 80°C for a duration of 5 hours. Subsequently, the slurry that had been created using hydrochloric acid (HCl) underwent filtration and then washed with distilled water till the filtrate achieved a state of neutrality. Afterwards, the pH of the solution was determined through the utilization of blue litmus. Following the filtration process the product was placed in an oven set to 105°C for the duration of the drying process. The powders were eventually dried and carbonized at a temperature of 800°C (Gong et al., 2019) for a duration of four hours using a tube furnace (Nabertherm, Germany) under a N_2 flow. The schematic diagram of biochar preparation is depicted below in **Figure 4.8**

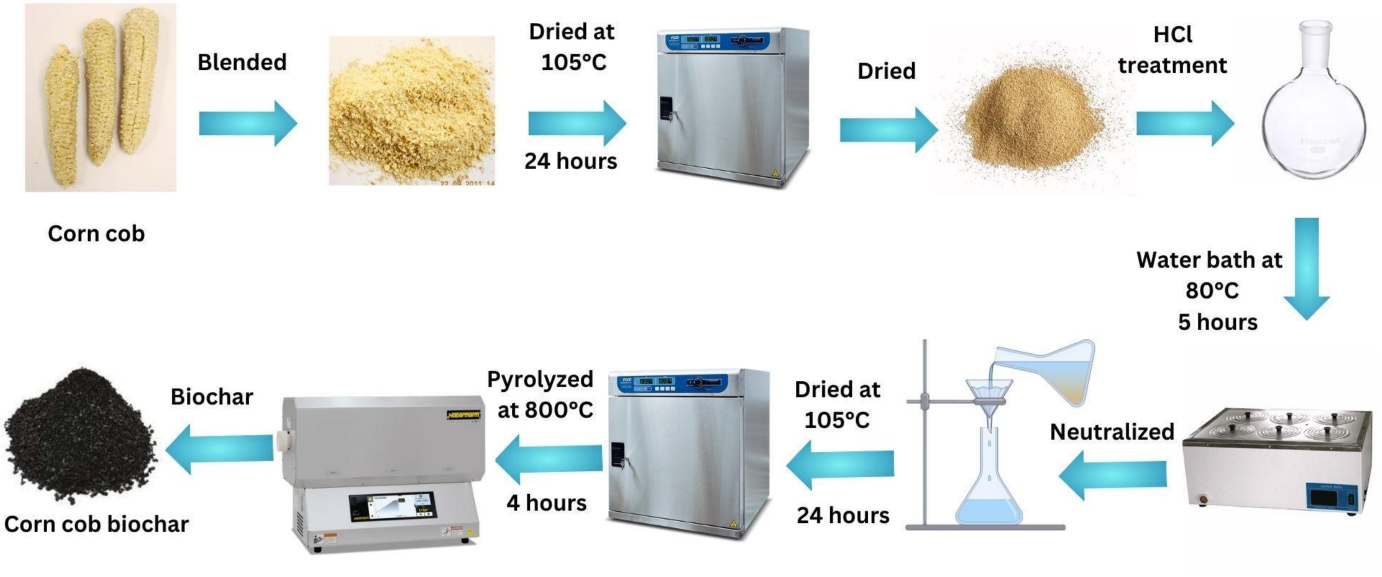


Fig 4.8. Schematic diagram of biochar preparation

3.4. Activated Carbon preparation

Biochar was obtained and afterwards subjected to treatment with potassium hydroxide (KOH) utilizing two distinct methodologies and two varying ratios. The mass ratios for the activation agents were selected as 1:1 and 1:3. The molarity of a chemical solution is directly influenced by the mass of the chemical agent, with a larger mass resulting in a higher molarity (Rahma et al., 2019). **Table 5** presents the activation ratios together with their corresponding temperatures.

Table 5. Sample activation ratios with respective temperatures

Sample	KOH/Biochar ratio	Activation Temperature(°C)
	1:1	850
MAC-2	1:1 (without KOH solution preparation)	850
MAC-3	1:3	850

MAC-1 sample preparation

In the first ratio and procedure, a 1:1 ratio of KOH pellets and biochar (1:1, wt/wt), was immersed in an appropriate quantity of distilled water to promote the breakdown of the KOH pellets and the concurrent immersion of the biochar (MAC-1) (Hossain et al., 2021).

MAC-2 sample preparation

The second method entailed the direct incorporation of solid KOH pellets (1:1, wt/wt) into the biochar, known as (MAC-2), as an activator for KOH, as described by Cao et al. (2006).

MAC-3 sample preparation

The second ratio of potassium hydroxide (KOH) pellets (1:3, wt/wt) was immersed in a precise volume of distilled water that was sufficient to dissolve both the KOH pellets and the specific biochar referred to as (MAC-3) (G. Wang et al., 2020). **Figure 4.9** depicts a schematic diagram illustrating the process of activated carbon preparation.

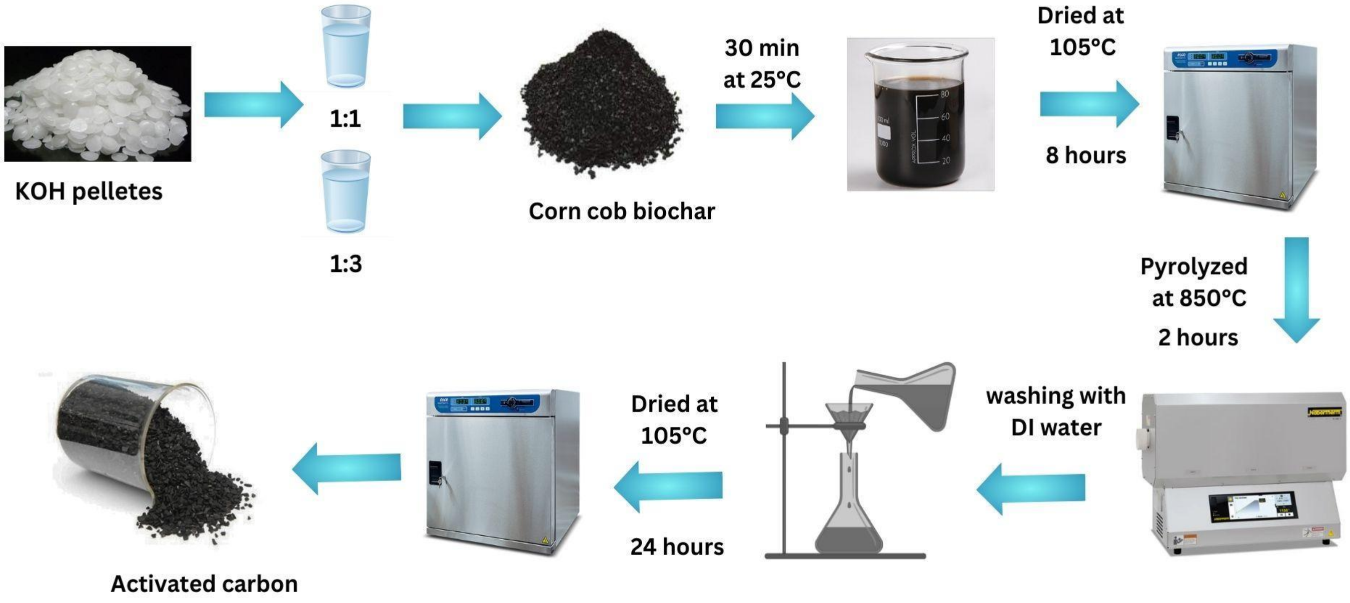


Fig 4.9. Schematic diagram of activated carbon preparation

The mixtures were allowed to sit at room temperature for 30 minutes and subsequently dried in an oven at 105°C overnight. The KOH-impregnated biochar was subjected to a temperature of 850°C for a duration of two hours in a tubular furnace, with the presence of nitrogen gas (Hossain et al., 2021). The activated carbons MAC-1, MAC-2, and MAC-3 were cooled and rinsed with hot and cold distilled water until the pH of the rinse water reached 7, as determined by red litmus testing. The activated carbons were later dried at 105°C in an oven. The activated carbons were finally enclosed in a vial and stored in a desiccator until needed. **Figure 4.10** presents a flowchart illustrating the process of preparing activated carbon from raw corn cobs.

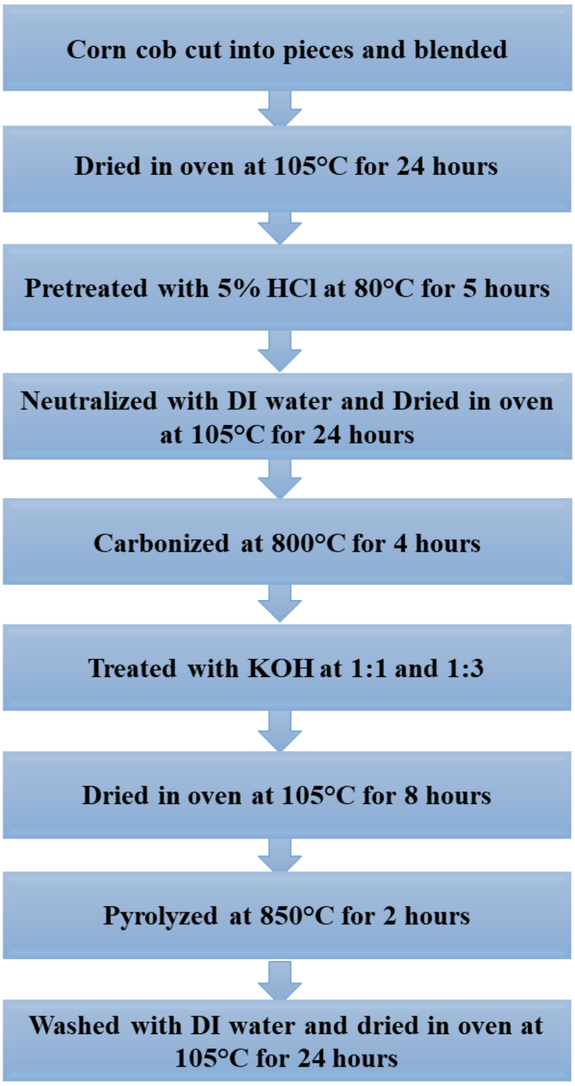


Fig 4.10. Flowchart of Activated carbon preparation from raw corn cobs

The laboratory procedure for the manufacture of activated carbon from raw corn cobs is illustrated in **Figure 4.11**.

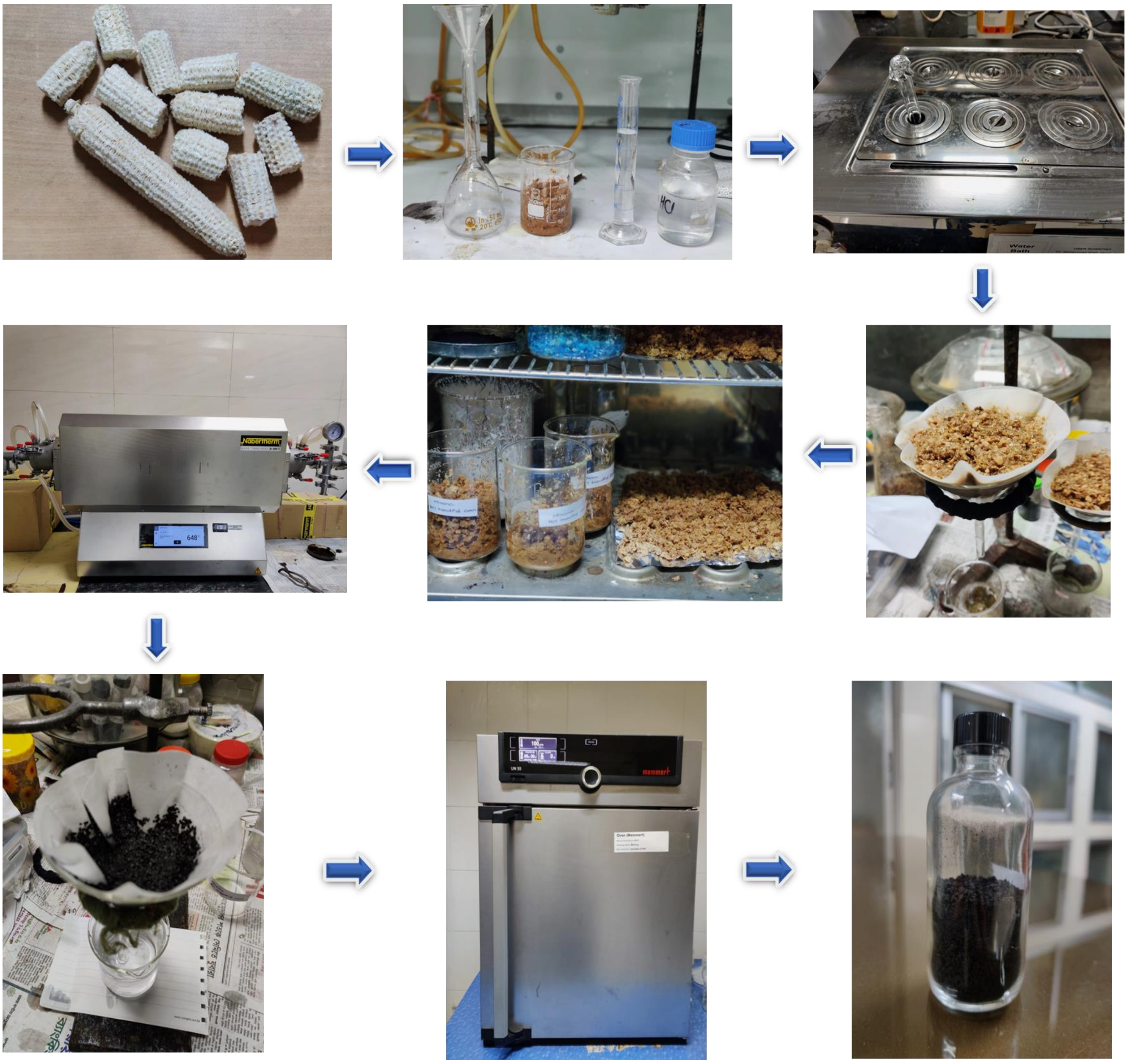


Fig 4.11. Activated carbon preparation from raw corn cob in laboratory

3.5. CO_2 adsorption

The CO_2 adsorption glass column was supported by a foam structure and connected to a gas balloon by means of a delivery pipe. Parafilm was applied to the opposite end of the column to prevent leakage. The upper section of the column featured a vent that was equipped with a cap to facilitate the release of excess gas.

After adding a certain amount of the sample or activated carbon (AC), the adsorption column was tightly sealed. The CO_2 pipe's valve was opened to release the gas, which was subsequently held at room temperature and atmospheric pressure for durations of 5, 10 and 20 minutes. The samples' weights were measured using an electronic weighing balance both before and after the flow of CO_2. This study aimed to assess the weight variation of the samples. The process continued until a stable weight was achieved, indicating that the sample had reached its maximum adsorption capacity. The same procedure was repeated for sample MAC-1 and MAC-2

3.6. CO_2 desorption

The desorption process was conducted for the stated samples at time intervals of 5, 10 and 20 minutes, employing a similar approach as the adsorption process. Following the measurement of weight, the samples were subsequently subjected to a desorption process by being exposed to a temperature of 105°C within an oven for a duration of 60 minutes. Soon after a 60-minute duration, the samples were then taken to a dessicator for an interval of time in order to facilitate cooling. Subsequently, the samples were re-weighed to determine the amount of desorption. The procedure has been repeated for all the samples. **Table 6** presents a list of the sample types and corresponding reaction times employed for the processes of adsorption and desorption and the process of CO_2 adsorption-desorption experiment is illustrated in **Figure 4.12.**

Table 6. List of sample and reaction time used for adsorption and desorption

Reaction Time(min)	Activated Carbon sample used
5	MAC-1
10	MAC-1
20	MAC-1
5	MAC-2
10	MAC-2
20	MAC-2

Fig 4.12. CO_2 adsorption-desorption experiment run

CHAPTER FIVE

DATA ANALYSIS

Data Analysis

5.1. Adsorption and Desorption experiment

The experiment involved conducting adsorption and desorption tests on samples MAC-1 and MAC-2 at different time intervals (5, 10, and 15 minutes) under ambient temperature conditions. To minimize error, two replications were performed for each sample at each time interval following the initial experiment.

5.2. Characterizations

Scanning Electron Microscope (SEM)

The scanning electron microscopy (SEM) **(**EVO-18, Carl Zeiss, Germany) was employed to determine the visual characteristics, structural composition, and development patterns of pore spaces present in both biochar and activated carbon materials. (accelerating voltage: 15kV, magnification: 1.00K-5.00K and width-9.5mm).

Fourier Transform Infrared (FTIR)

FT-IR spectrometry is a non-destructive and real-time analytical method used to identify unknown compounds and determine their concentration. It can be applied to organic and inorganic substances in solid, liquid, or gas samples. FT-IR spectrometry is applicable to solid, liquid, and gas samples (Ţucureanu et al., 2016). A Fourier Transform-Infrared Spectroscopy (FT-IR) analysis was performed (FTIR; Frontier, PerkinElmer, UK). The present investigation analyzed and characterized the surface functions and bond stretching related to the adsorption and desorption processes of MAC-1 and MAC-2 to determine the bonds present within the spectra ranging from 4000 cm^{-1} to 450 cm^{-1}, with a data interval of 1 cm^{-1}

Energy Dispersive Spectroscopy (EDS)

Energy dispersive spectroscopy analysis (EDS; JEOL 7610F, Japan) was performed on samples prior to CO_2 flow to determine the elemental composition as a baseline. The analysis was performed using an accelerating voltage of 10kV and a magnification of 200X. The process of analyzing the samples after adsorption and desorption allows for the monitoring of changes in composition. This provides valuable insights into the dynamic transformations that occur during the interaction with CO_2. The dual-stage EDS approach helps in understanding the structural and elemental changes that occur during CO_2 adsorption and desorption.

5.3. Statistical Analysis

The quantification of the adsorption and desorption process of carbon dioxide (CO_2) has been conducted for each sample at specific time intervals. The Excel spreadsheet application was used to analyze the graphical representation of CO_2 adsorption and desorption, including their respective standard deviations, through statistical analysis. The objective of this analysis is to compare and observe the rate of change over time. The raw Fourier Transform Infrared (FTIR) data was acquired from an FTIR instrument and exported into Microsoft Excel 2019. Subsequently, the processed data was analyzed by creating graphs using Origin 2023b software. The graph displayed the wavenumber on the X-axis, measured in units of cm^{-1}, and the transmittance on the Y-axis, represented as a percentage.

The figure below (**Figure 5.1 to 5.4)** illustrates the procedure for preparing pellets using potassium bromide (KBr) and subsequently performing Fourier transform infrared spectroscopy (FTIR).

Fig 5.1. KBr and activated carbon mixture

Fig 5.2. Hydraulic pellet press

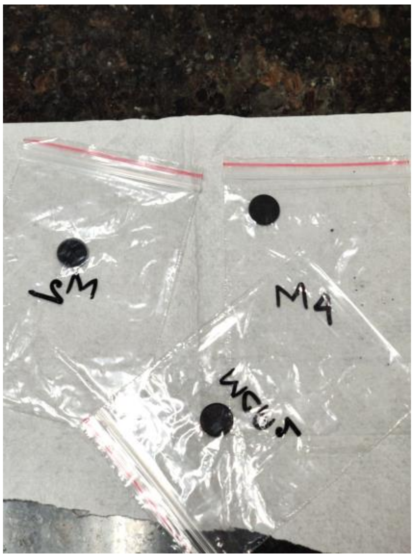


Fig 5.3. sample

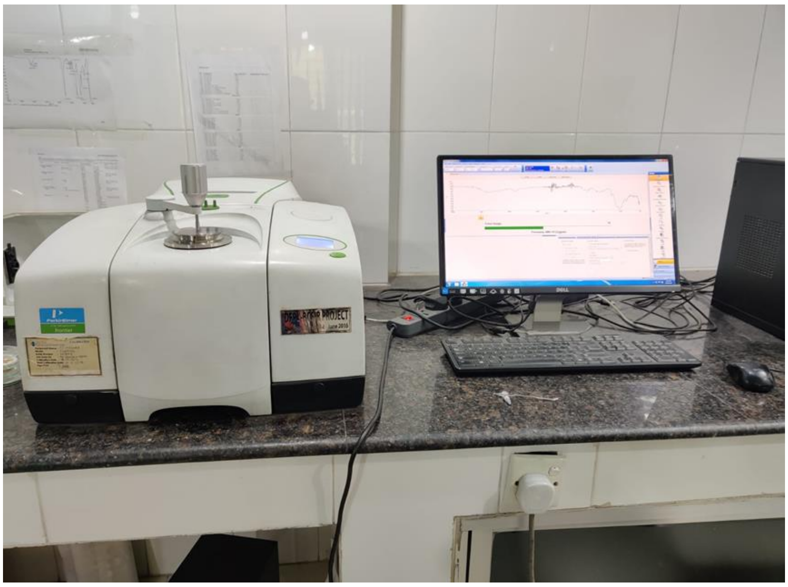


Fig 5.4. FT-IR machine analysis

CHAPTER SIX

RESULT AND DISCUSSION

6.1. Adsorption and desorption analysis of carbon dioxide

The experiments were performed in three different time intervals: 5, 10, and 30 minutes. In each reaction, a quantity of 1 gram of activated carbon from samples MAC-1 and MAC-2 was utilized. Replication was conducted in order to minimize the probability of errors. MAC-3 was not used in the reactions due to its insignificant activated carbon quality in comparison to MAC-1 and MAC-2. The results of the processes of adsorption and desorption are presented in **Table 7.**

Table 7. Adsorption and desorption amount with their standard deviation

Sample name and Time (min)	Adsorbed amount (gm)	Adsorbed amount (gm)	Adsorbed amount (gm)	Average Adsorbed amount	Standard deviation of adsorption	Desorbed amount (gm)	Desorbed amount (gm)	Desorbed amount (gm)	Average Desorbed amount	Standard deviation of desorption
MAC-1 (5 min)	0.0317	0.0425	0.0302	0.035	0.007	0.0348	0.0426	0.03240	0.054	0.005
MAC-1 (10 min)	0.0775	0.0528	0.06112	0.064	0.013	0.0385	0.0362	0.03500	0.037	0.002
MAC-1 (20min)	0.05226	0.0534	0.06561	0.057	0.007	0.0317	0.0297	0.02220	0.028	0.005
MAC-2 (5 min)	0.0436	0.0352	0.0373	0.039	0.004	0.0324	0.0337	0.02690	0.031	0.004
MAC-2 (10 min)	0.0777	0.0635	0.0584	0.067	0.010	0.0233	0.0182	0.02120	0.021	0.003
MAC-2 (20 min)	0.0529	0.0468	0.0447	0.048	0.004	0.0206	0.0224	0.01780	0.020	0.002

The literature analysis of various articles revealed that the measurement of CO_2 uptake is typically expressed in millimoles per gram (mmol/g). However, it is important to note that our experimental procedure involved the use of grams (gm) as the unit of measurement. In order to facilitate comparative comparison, the uptake values in the table have been expressed from grams (g) to millimoles per gram (mmol/g). **Table 8** displays the conversion of adsorption and desorption in mmol/g.

Table 8. Adsorption and desorption in mmol/g

Sample name and Time (min)	Average Adsorbed amount (gm)	Average Adsorbed amount (mmol/g)	Average Desorbed amount (gm)	Average Desorbed amount (mmol/g)
MAC-1 (5 min)	0.035	0.795	0.054	1.227
MAC-1 (10 min)	0.064	1.455	0.037	0.841
MAC-1 (20 min)	0.057	1.295	0.028	0.636
MAC-2 (5 min)	0.039	0.886	0.031	0.705
MAC-2 (10 min)	0.067	1.523	0.021	0.477
MAC-2 (20 min)	0.048	1.090	0.020	0.455

The graph illustrated below shows an initial upward trend, which is then followed by a subsequent decline. Eventually, after a duration of 20 minutes, the graph reaches a state of saturation. The graph depicts the increase in the adsorption capacity of activated carbon derived from corn cobs for CO_2 over a period of 5 to 10 minutes. The MAC-2 sample demonstrated the highest CO_2 absorption at the 10-minute mark, with a measurement of 1.523 mmol/g. In contrast, the samples collected over a duration of 20 minutes clearly demonstrated a decrease in adsorption. Over time, a gradual saturation of carbon dioxide (CO_2) uptake is observed, typically occurring after approximately 20 minutes. The **Figure 6.1** below depicts the adsorption of CO_2 at different time intervals, specifically 5, 10, and 20 minutes.

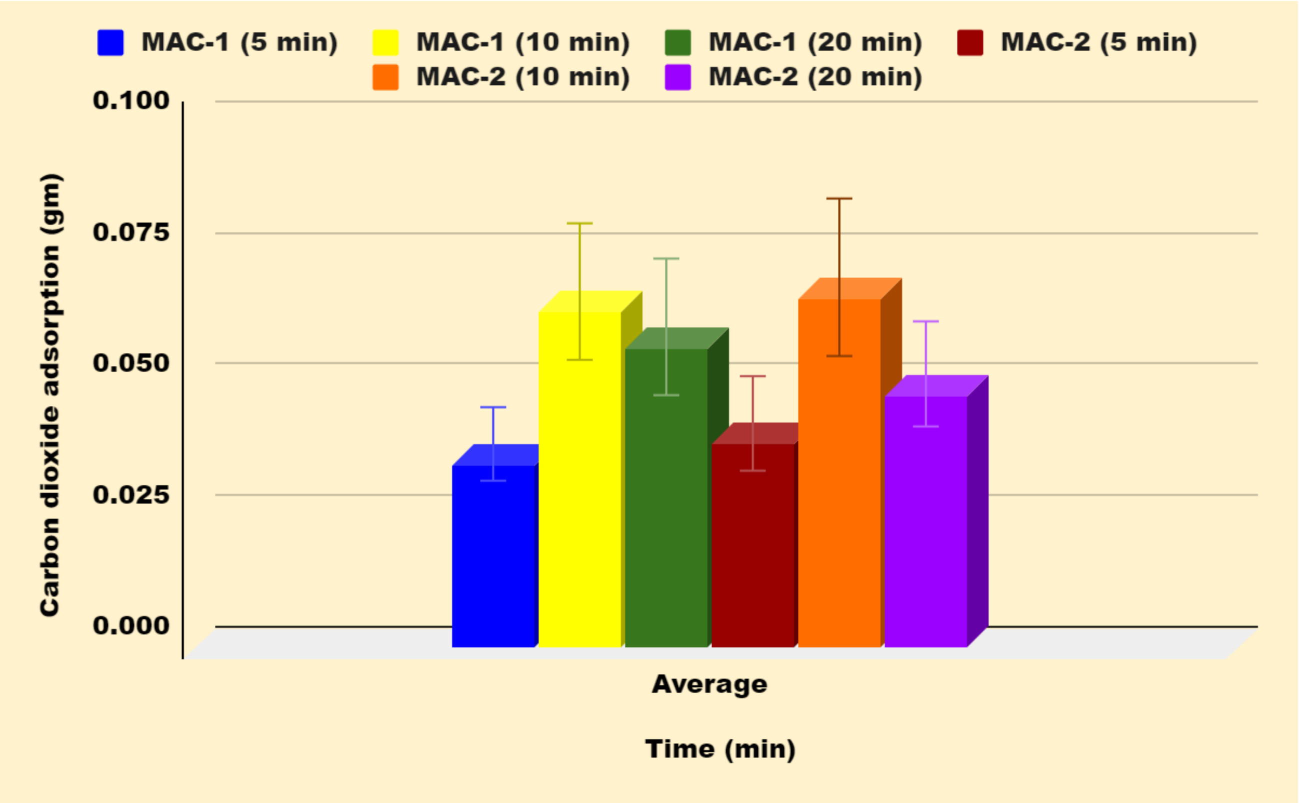


Fig 6.1. Adsorption of CO_2 at different time intervals

The graph depicted below visually represents the process of carbon dioxide desorption. The bar chart presented clearly illustrates a decline in the desorption capacity of activated carbon derived from corn cobs for CO_2 over a time span of 5 to 10 minutes, as well as at the 20-minute mark. During the 5-minute duration, the MAC-1 sample exhibited the highest level of carbon dioxide (CO_2) desorption, measuring at 1.227 mmol/g. **Figure 6.2** displays the graphical representation of the process of CO_2 desorption at various time intervals.

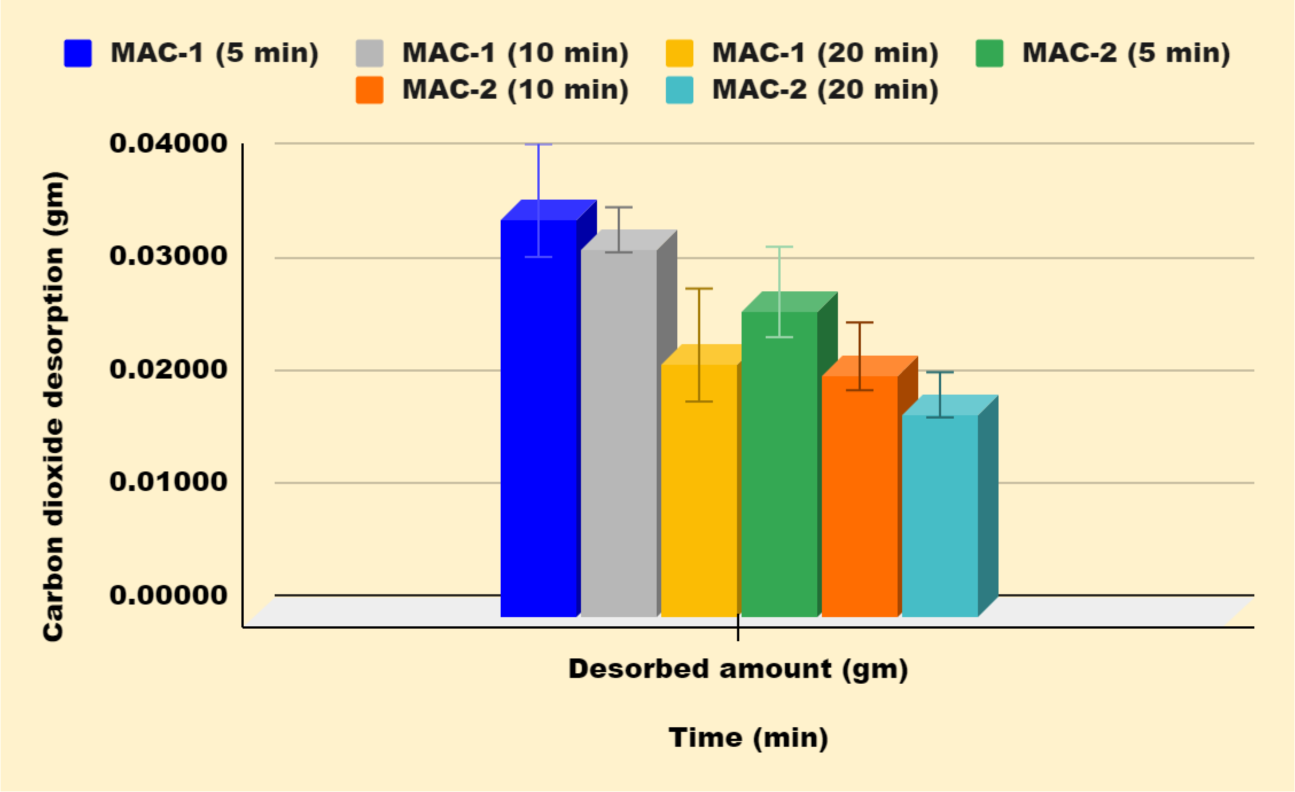


Fig 6.2. Desorption of CO_2 at different time intervals

The graph given below depicts that the adsorption demonstrated a noticeable enhancement in the rate of adsorption during the time period ranging from 5 to 10 minutes. In contrast, the desorption phase exhibited a progressive decrease in adsorption over time. The desorption process was conducted for a duration of 60 minutes at a temperature of 105 °C for all samples. However, the desorption values exhibited variation as a result of the adsorption process, which was carried out for different time intervals of 5, 10, and 20 minutes. Therefore, these time intervals were also taken into consideration during the desorption phase**. Figure 6.3** shows the graph depicting the combined result of adsorption and desorption of carbon dioxide (CO_2).

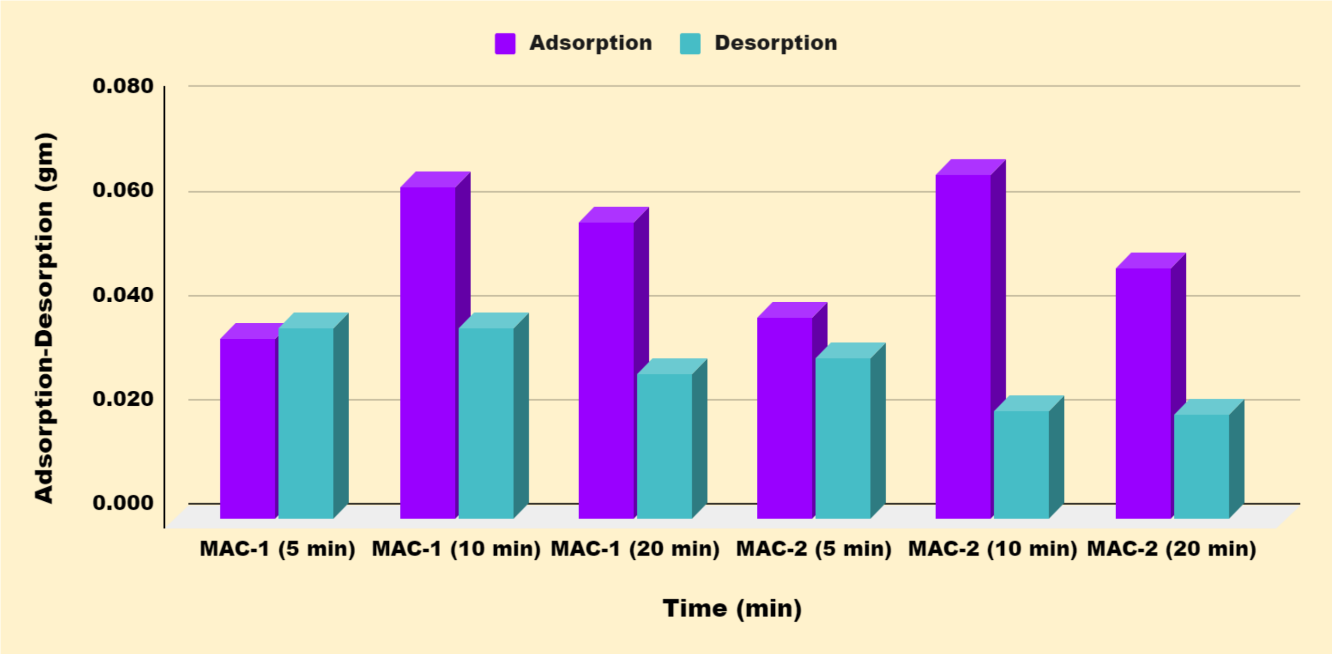


Fig 6.3. CO_2 adsorption-desorption graph

6.2. Comparison with previous research studies

Song et al. (2013) investigated the effects of subjecting corn cobs to a temperature of 800°C and activating it using a KOH activation ratio of 1:3. The study found that the activated corn cobs exhibited a notable CO_2 absorption capacity of 1.5 mmol/g. In contrast, the present research subjected the corn cobs to a higher activation temperature of 850 degrees Celsius and activated using a 1:1 ratio. The experimental condition yielded a maximum adsorption capacity of 1.523 mmol/g, surpassing the findings of Song et al. (2013) who used a higher activation ratio. This indicates that a higher KOH activation ratio in study of Song et al. (2013) did not result in a corresponding increase in CO_2 adsorption. However, the higher activation temperature observed in this study appears to affect the adsorption capacity.

Wu et al. (2021) conducted a research experiment to examine corn kernels using pyrolysis and carbonization techniques. The corn kernels underwent treatment with KOH at temperatures ranging from 800 to 900 °C. The carbon material derived from corn kernels demonstrated a high capacity for adsorbing CO_2. The CKC-900-4 sample demonstrated the highest carbon dioxide (CO_2) adsorption capacity, measuring 3.63 mmol/g at a temperature of 25 °C over a 2-hour period. CKC-900 refers to carbon obtained from corn kernels, which underwent activation at 900°C with a KOH activation ratio of 1:4. This study employed corn cobs as the raw material and subjected it to activation at a temperature of 850°C using a KOH activation ratio of 1:1. The resulting adsorption capacity was measured at 1.523 mmol/g within a 10-minute adsorption period. This study demonstrates more effective adsorption performance within a 10-minute timeframe, in contrast to the 2-hour experiment conducted by Wu et al. (2021).

According to a recent study conducted by Khuong et al. (2021), the adsorption capacity of bamboo carbon for CO_2 exhibited a significant improvement when activated for varying durations between 0 and 15 minutes. The bamboo samples that underwent activation for a duration of 10 minutes had the highest level of carbon dioxide absorption. During the current study, it was revealed that the corn cobs demonstrated the maximum level of adsorption within a 10-minute time frame.

In the study conducted by Lee et al. (2013), the researchers applied a coating of 2-amino-2-methyl-1,3-propanediol onto the surface of activated carbon that was obtained from palm shells. The experiment's results demonstrated that the adsorption capacity was 64.0 mg/g, which is comparable to 0.064 g/g. In contrast, the activation of corn cobs using potassium hydroxide (KOH) resulted in a carbon dioxide (CO_2) adsorption capacity of 0.067g, as observed in the present work.

Rashidi et al. (2013) conducted a study to investigate the adsorption capacity of activated carbon derived from coconut by-products for CO_2. The experimental procedure involved examining carbon samples at temperatures of 25, 50, and 100 °C. The synthesized activated carbon demonstrated a maximum sorption capacity of 80 mg/g for carbon dioxide (CO_2) over a 2-hour period, as determined by the researchers. However, this study achieved a high adsorption capacity of 67 mg/g in just 10 minutes. It is crucial to consider that both research had an equal sample size of 1 gram

In the study conducted by Wang et al. (2022), an analysis was carried out to determine the adsorption capacity of KOH-activated corncob. The experiment was conducted at a temperature of 20°C using an online fixed bed adsorption device. The observed adsorption capacity was identified to be 3.49 mmol/g. In contrast, the present study successfully attained a maximum adsorption capacity of 1.523 mmol/g at ambient temperature, without the utilization of a fixed bed adsorption system or equipment.

6.3. Scanning Electron Microscope (SEM) analysis of biochar and activated carbon

A scanning electron microscopy (SEM) analysis was performed in order to examine the surface morphology of biochar and activated carbon subsequent to KOH activation. The SEM images of biochar **(Figure 6.4, 6.5, and 6.6)** demonstrate a lesser pore structure compared to the significant pore structure developed by MAC-1, 2, and 3. The surface morphology of MAC-2 **(Figure 6.10, 6.11)** closely resembles a honeycomb structure with evenly distributed pores. This suggests that the chemical agent is uniformly spread over the surface. The pore spaces of MAC-1 (**Figure 6.7 and 6.8)** exhibit improved quality and clearly defined structure. The unwashed samples **(Figure 6.9, 6.12, and 6.15**) exhibit the presence of developed pore spaces, although they are predominantly blocked by chemical agents. In the washed samples, the pores exhibit open, distinct, and well-defined characteristics.

The pore size of carbon is influenced by the impregnation ratio. Rahma et al. (2019) found that increasing the molarity of KOH led to the formation of carbon with larger and more defined pore sizes. The SEM images of this study indicate that the sample with a 1:3 ratio (MAC-3) exhibited unevenly distributed and partially closed pore spaces, in contrast to the samples MAC-1 and MAC-2. Both MAC-1 and MAC-2 were activated at a 1:1 ratio, despite employing separate activation methods. However, the resulting activated carbon exhibited better pore quality compared to the activated carbon produced at a 1:3 ratio (MAC-3).

The observed alterations in the morphology and arrangement of pores indicate that these materials are responsive to activation. MAC-1 and MAC-2 exhibit better pore characteristics compared to MAC-3.

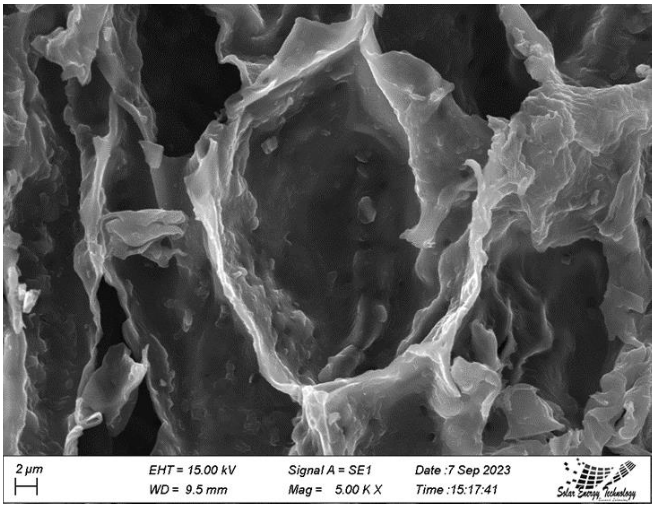


Fig 6.4. SEM of Biochar with 5k magnification

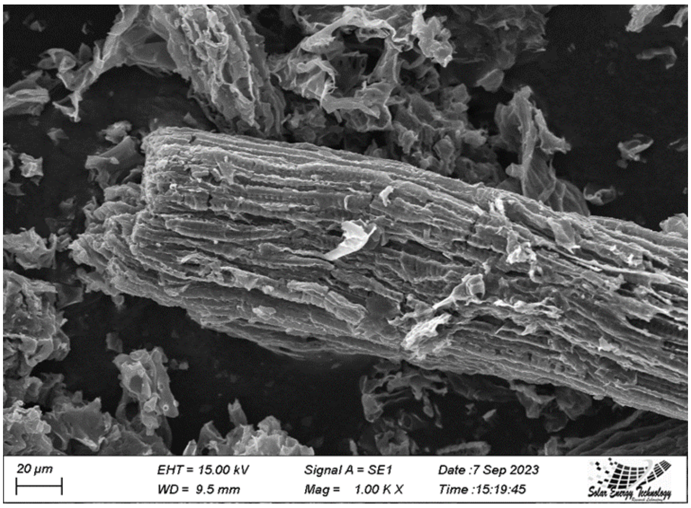


Fig 6.5. SEM of Biochar with 1k magnification

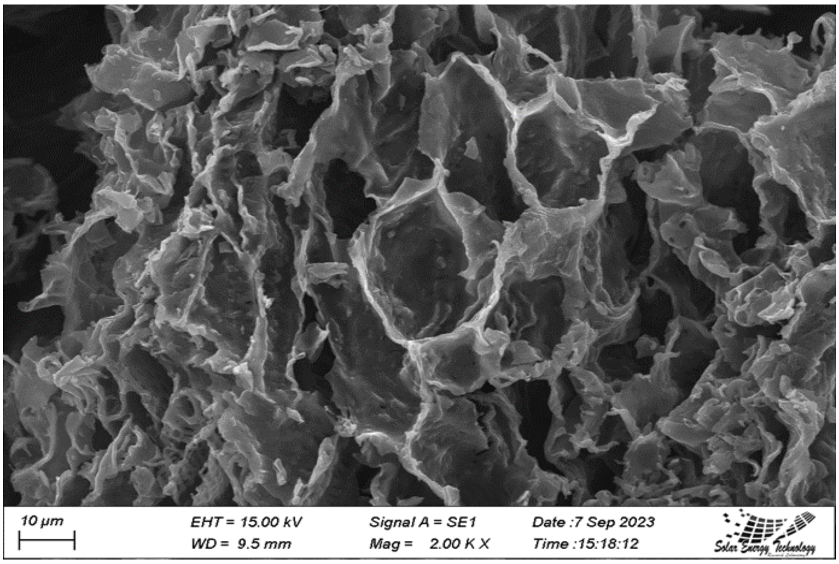


Fig 6.6. SEM of Biochar with 2k magnification

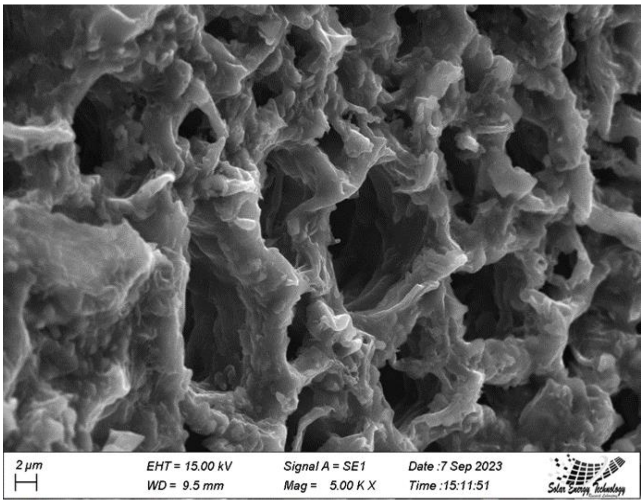


Fig 6.7. SEM of MAC-1 with 5k magnification

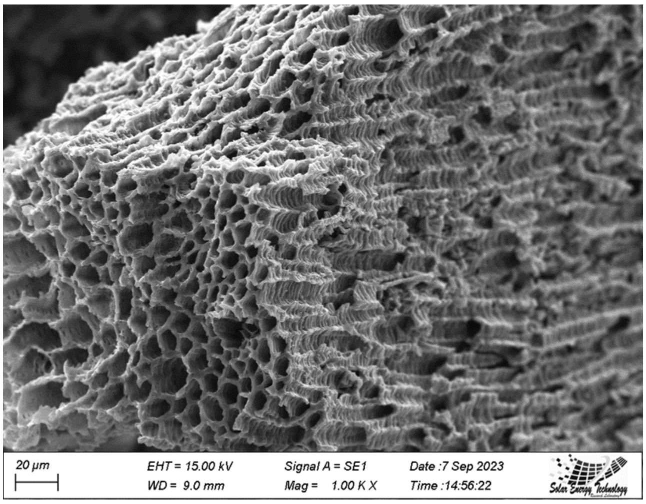


Fig 6.8. SEM of MAC-1 with 1k magnification

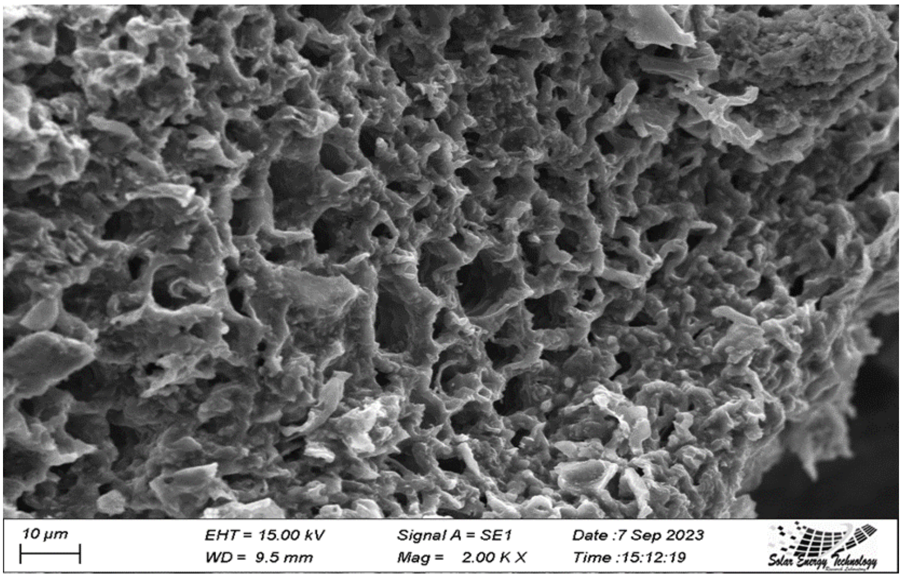


Fig 6.9. SEM of MAC-1(unwashed) with 5k magnification

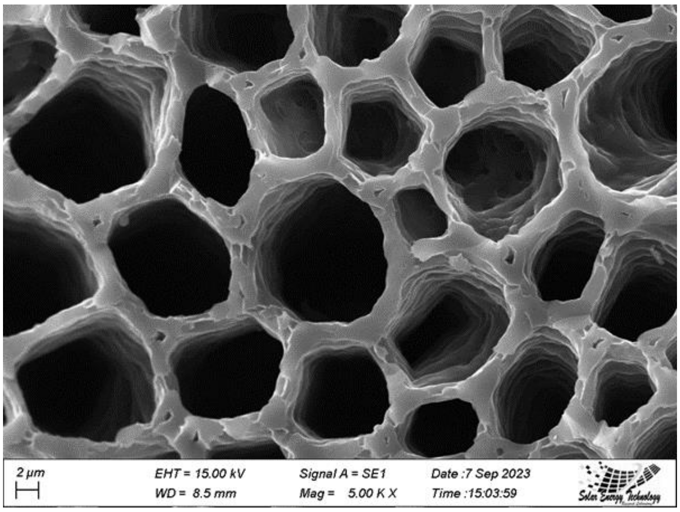


Fig 6.10. SEM of MAC-2 with 5k magnification

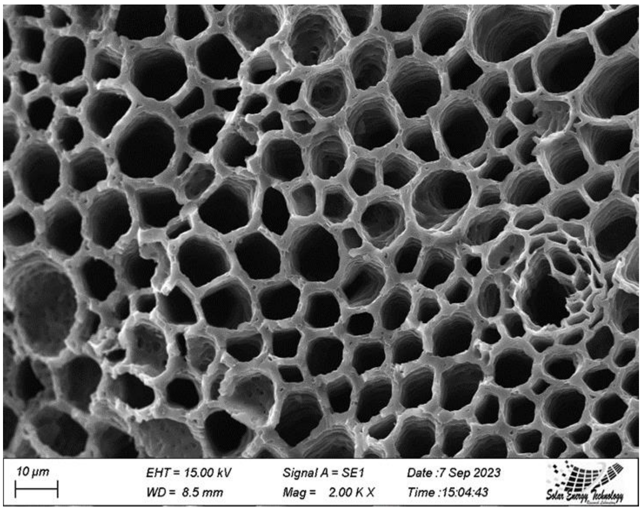


Fig 6.11. SEM of MAC-2 with 2k magnification

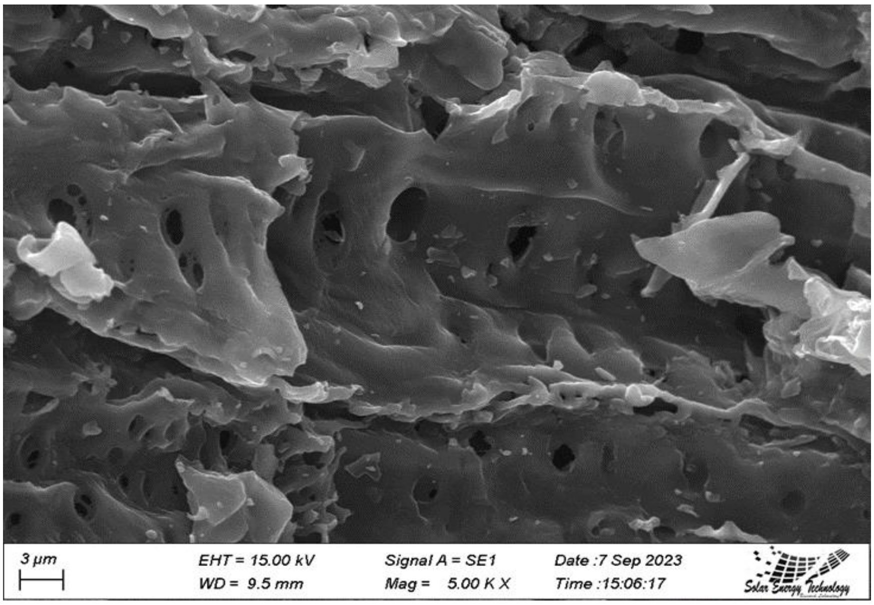


Fig 6.12. SEM of MAC-2 (unwashed) with 5k magnification

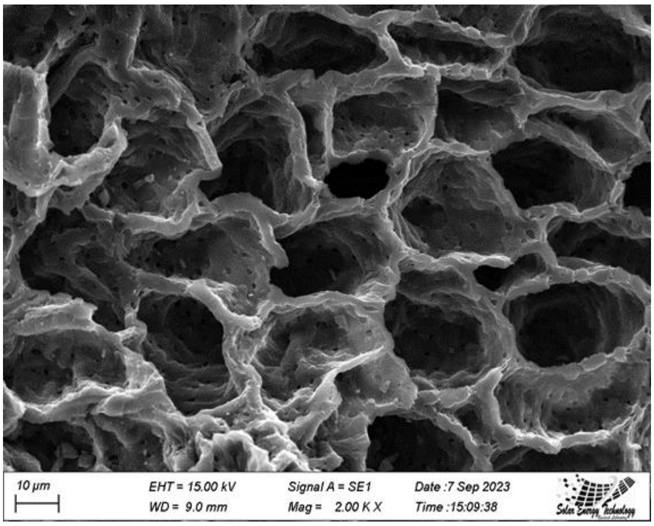


Fig 6.13. SEM of MAC-3 with 2k magnification

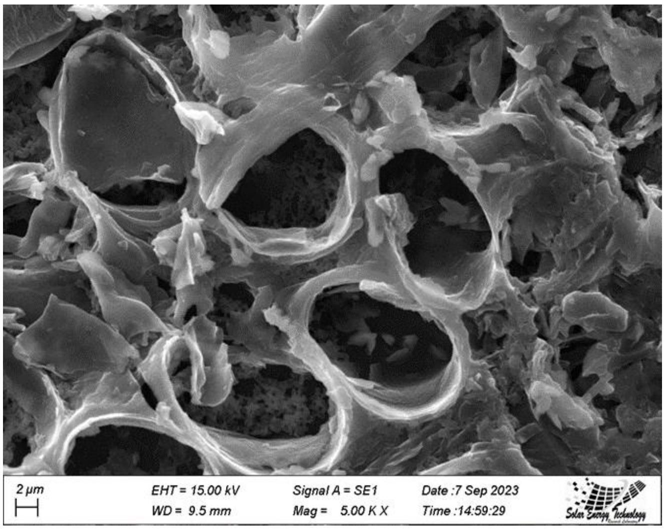


Fig 6.14. SEM of MAC-3 with 5k magnification

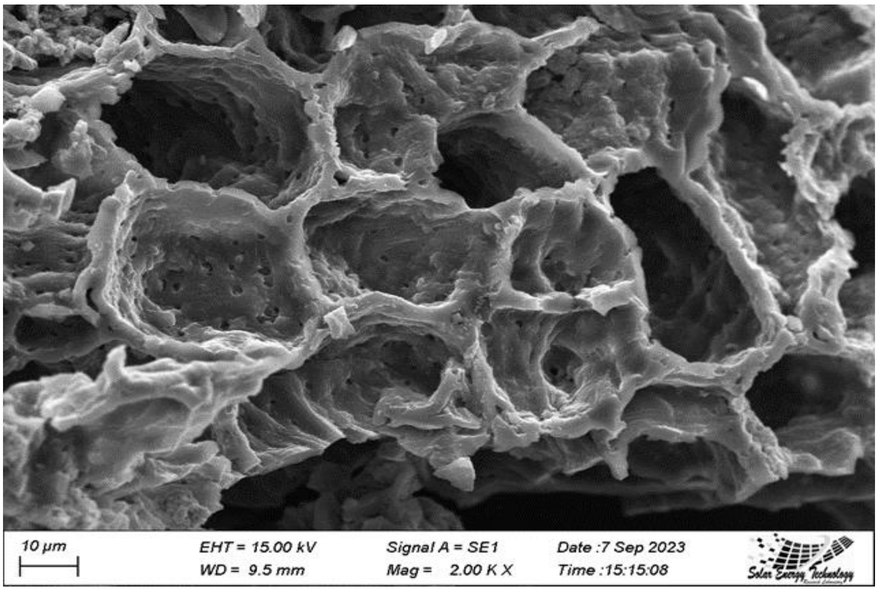


Fig 6.15. SEM of MAC-3 (unwashed) with 2k magnification

6.4. Scanning Electron Microscope (SEM) analysis post CO_2 adsorption and desorption

The SEM images **(Figure 6.16, 6.17, and 6.18)** taken after adsorption indicate that CO_2 has predominantly filled the pore spaces, resulting in reduced visibility of these spaces in the images. When CO_2 molecules are adsorbed onto the surface of activated carbon, they accumulate in the pores. This accumulation results in an increased concentration of CO_2 within the material and changes in the surface morphology.

In contrast, during the process of desorption, the images **(Figure 6.19, 6.20, and 6.21)** reveal that the pore spaces become minimally visible. During the desorption process, the removal of CO_2 from activated carbon results in the creation of vacant pore spaces that were previously occupied by CO_2 molecules. The liberation of CO_2 from the activated carbon structure facilitates the creation of pore spaces within such a structure. However, as depicted in **Figure 6.20**, the pores have exhibited a greater degree of expansion of pores.

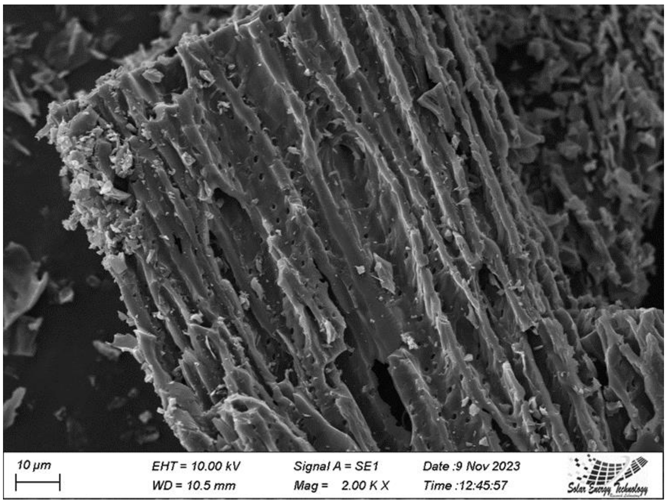


Fig 6.16. SEM of MAC-1 (20 min adsorption

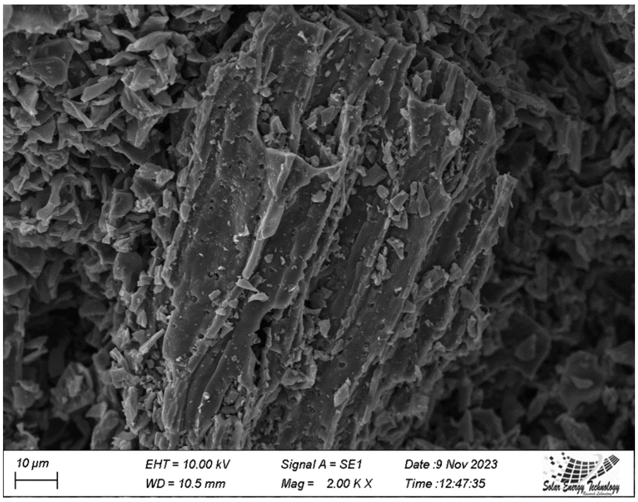


Fig 6.17. SEM of MAC-2 (20 min adsorption)

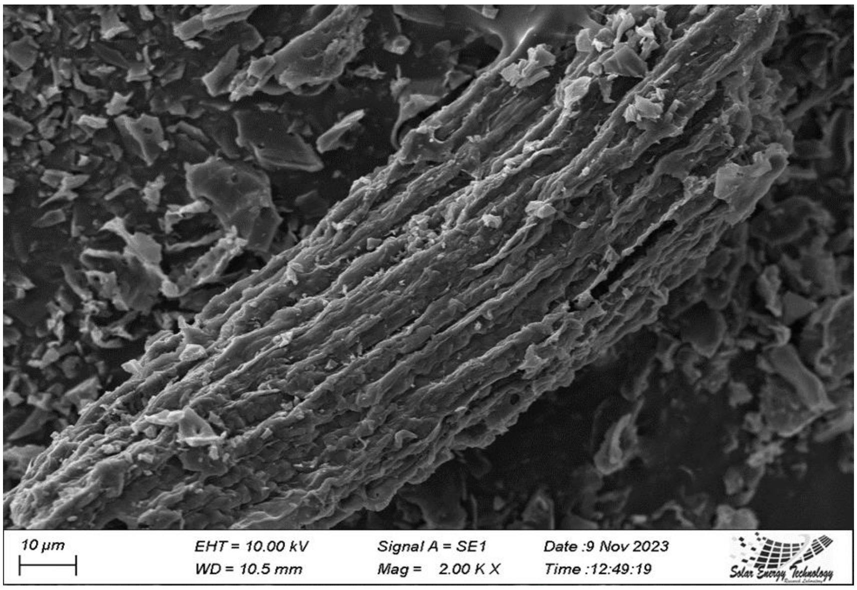


Fig 6.18. SEM of MAC-1 (10 min adsorption)

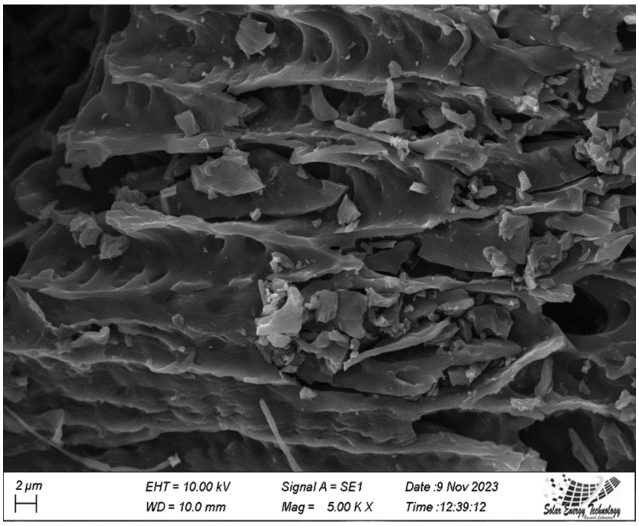


Fig 6.19. SEM of MAC-1 (20 min desorption)

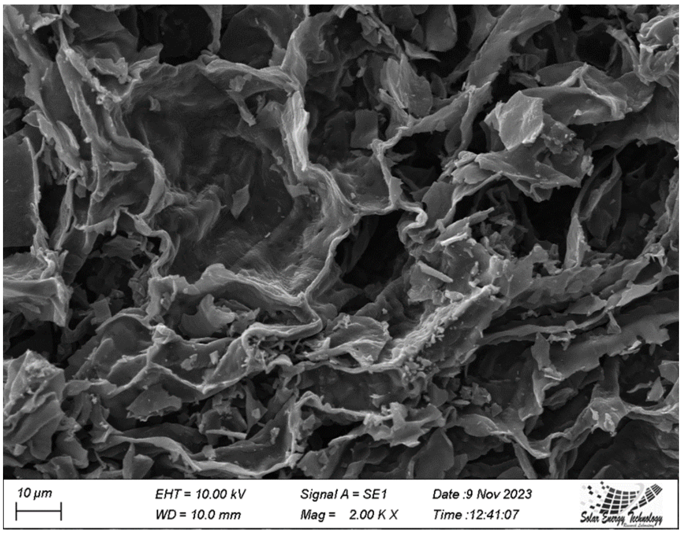


Fig 6.20. SEM of MAC-2 (20 min desorption)

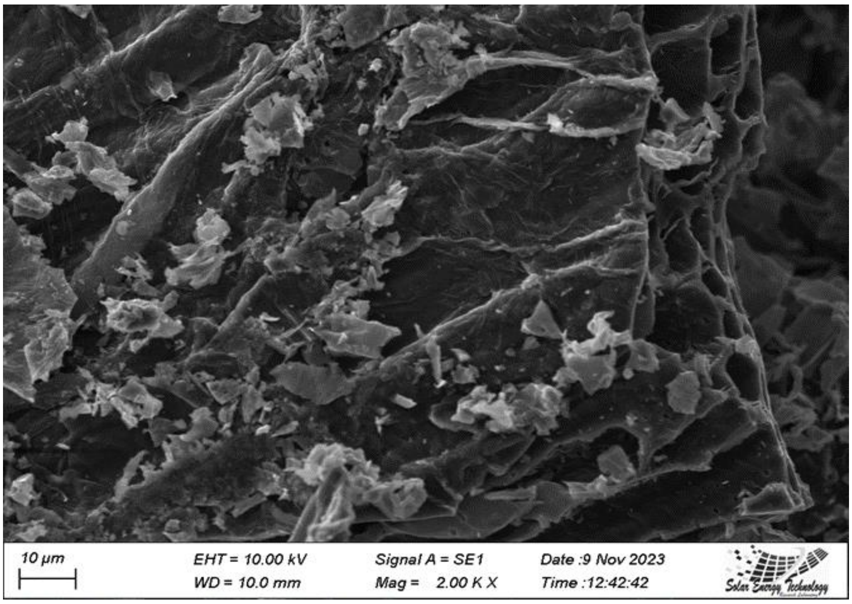


Fig 6.21. SEM of MAC-1 (10 min desorption)

In brief, scanning electron microscopy reveals the dynamic structural changes of activated carbon during the process of CO_2 adsorption and desorption. During the process of adsorption, there is a strong interaction between CO_2 molecules and the substance, resulting in the filling of pore holes. Desorption reverses the adsorption process, restoring visible pore spaces and indicating the cycle's reversibility. These discoveries enhance the optimization of material performance in carbon capture and storage by providing insights into its behavior.

6.5. Fourier Transform Infrared (FTIR) analysis after CO_2 adsorption and desorption

Based on the analysis of the FT-IR graphs, it is evident that there are broad bands detected within the spectral range of 3122-3697 cm^{-1} (as depicted in **Figure 6.22 - 6.29**). The observed broad bands appearing within the range of 2500-3620 cm^{-1} can be attributable to the stretching vibrations of O-H bonds (Figueiredo et al., 1999). This indicates the presence of O-H functional groups in all of the analyzed materials. In contrast, the occurrence of spectral bands ranging from 1384 to 1394 cm^{-1} can be ascribed to the stretching of the C=O functional group (Ramadoss & Muthukumar, 2015). It is noteworthy that this particular band exhibits a narrow pattern throughout all analyzed samples.

The graphs depicted in **Figures 6.24, 6.25, and 6.27** exhibit bands within the range of 1630-1650 cm^{-1}, indicating the existence of pronounced C=C stretching (Kamalini et al., 2018). The figures depicted in **Figures 6.23, 6.24, 6.25, 6.27, 6.28, and 6.29** have observable bands within the range of 980-1060 cm^{-1}, which suggest the occurrence of C-O stretching (Saleh et al., 2014). The presence of KOH impregnation gives rise to a K-O stretching band within the range of 600-702 cm^{-1} (Hossain et al., 2021). **Figures 6.26, 6.28, and 6.29**, which depict desorption graphs, indicate the absence of K-O bonds due to the desorption process occurring at a temperature of 105 °C. The bonds and their corresponding ranges are depicted in **Supplementary Table 1**.

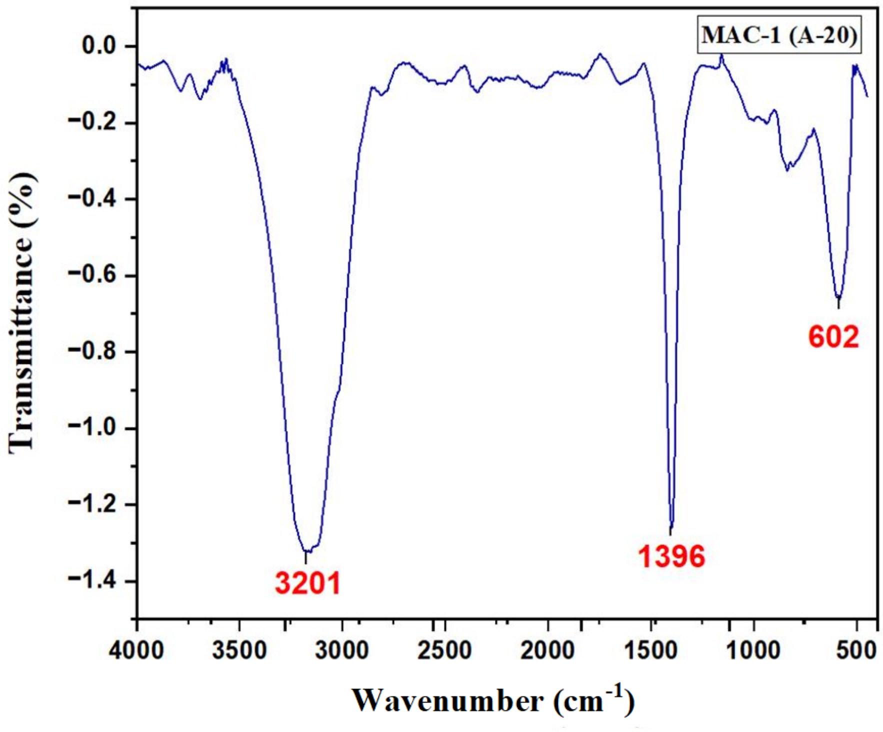


Fig 6.22. FTIR of MAC-1 (20 min adsorption)

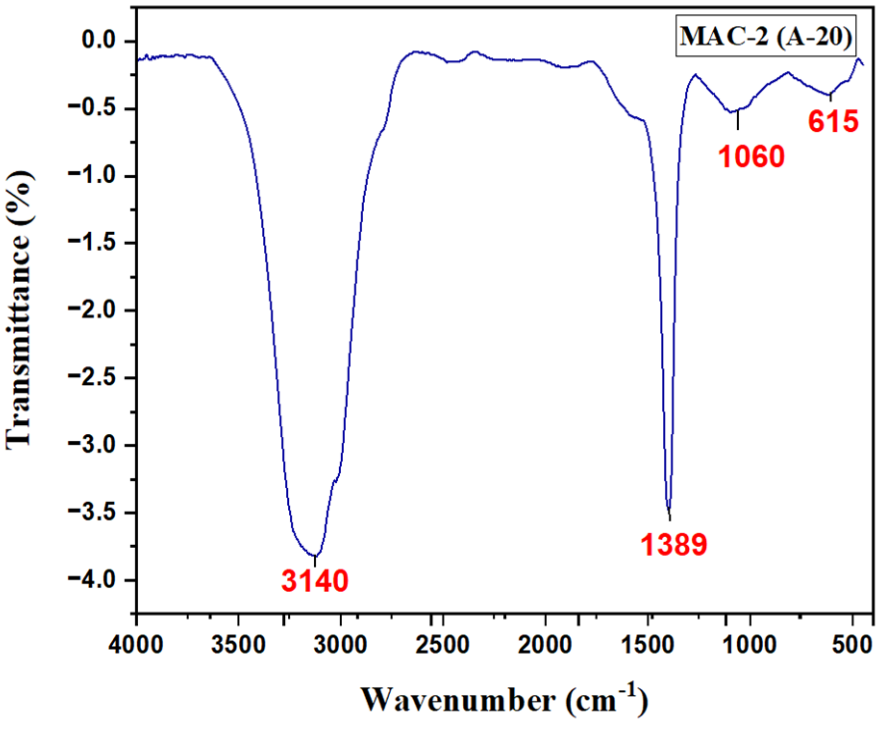


Fig 6.23. FTIR of MAC-2 (20 min adsorption)

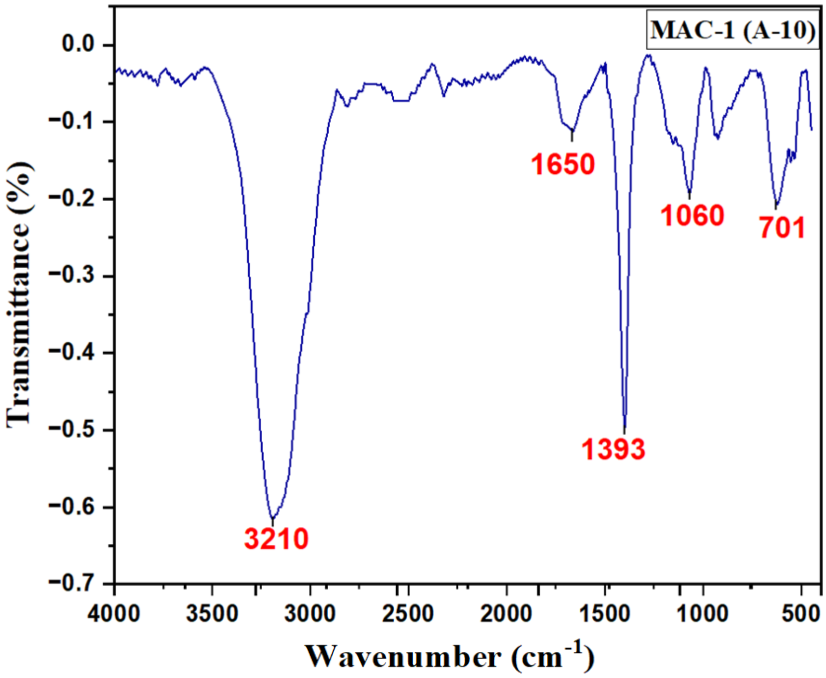


Fig 6.24. FTIR of MAC-1 (10 min adsorption)

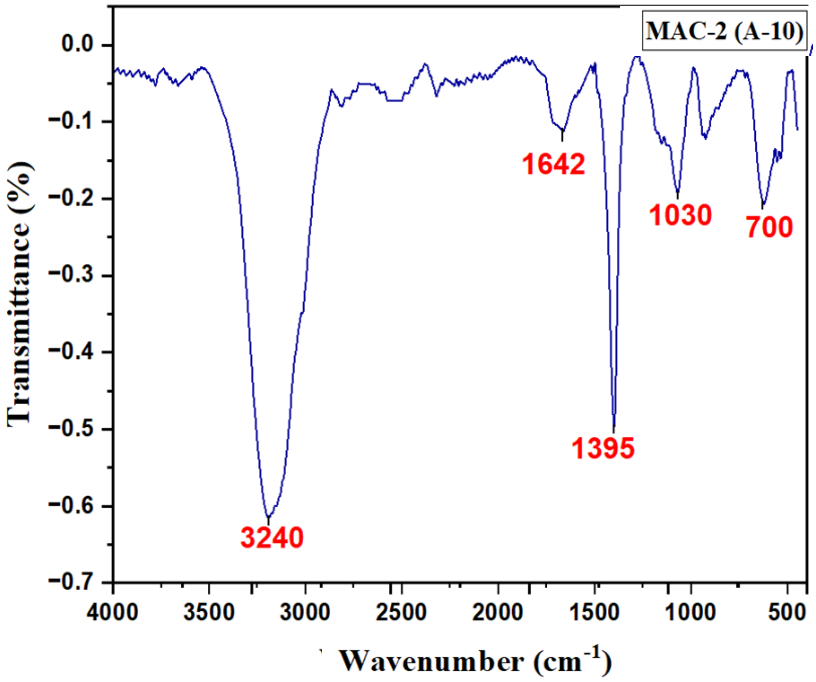


Fig 6.25. FTIR of MAC-2 (10 min adsorption)

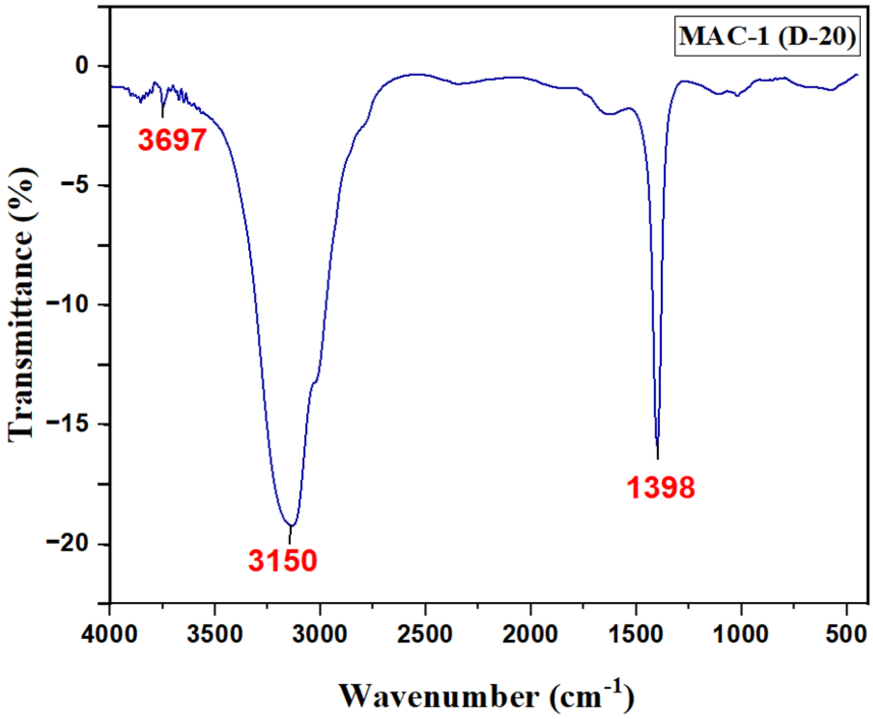


Fig 6.26. FTIR of MAC-1 (20 min desorption)

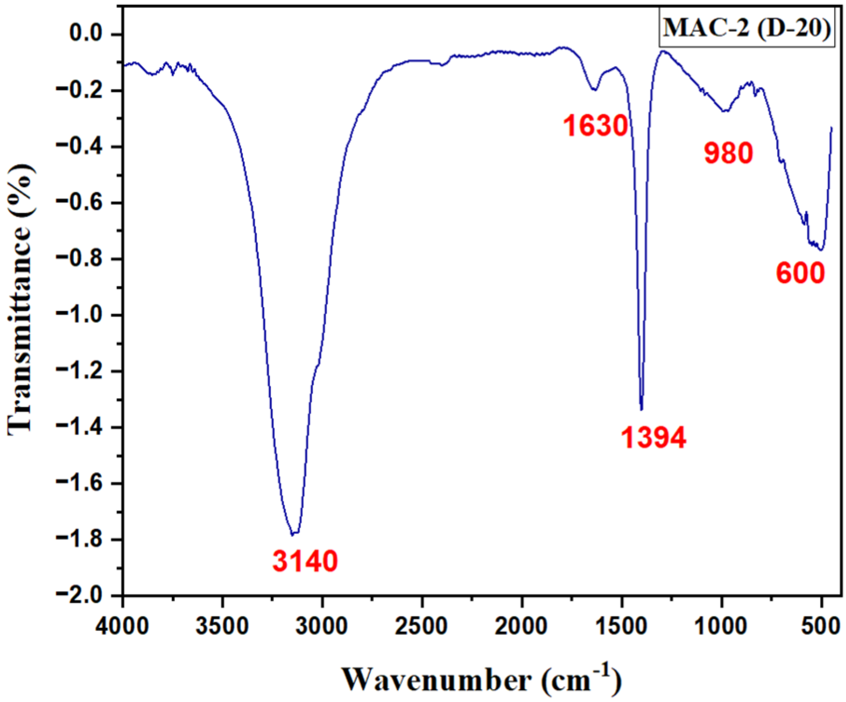


Fig 6.27. FTIR of MAC-2 (20 min desorption)

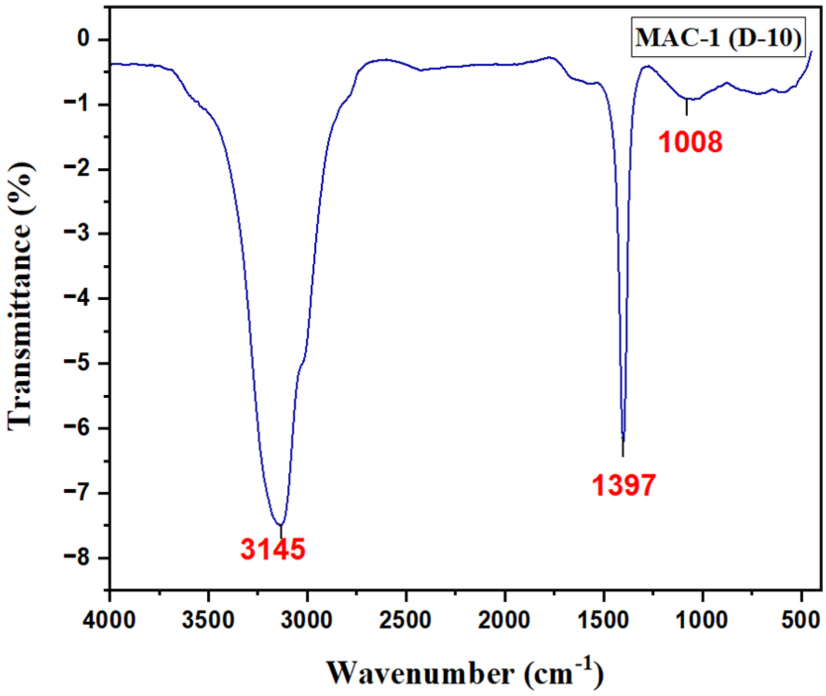


Fig 6.28. FTIR of MAC-1 (10 min desorption)

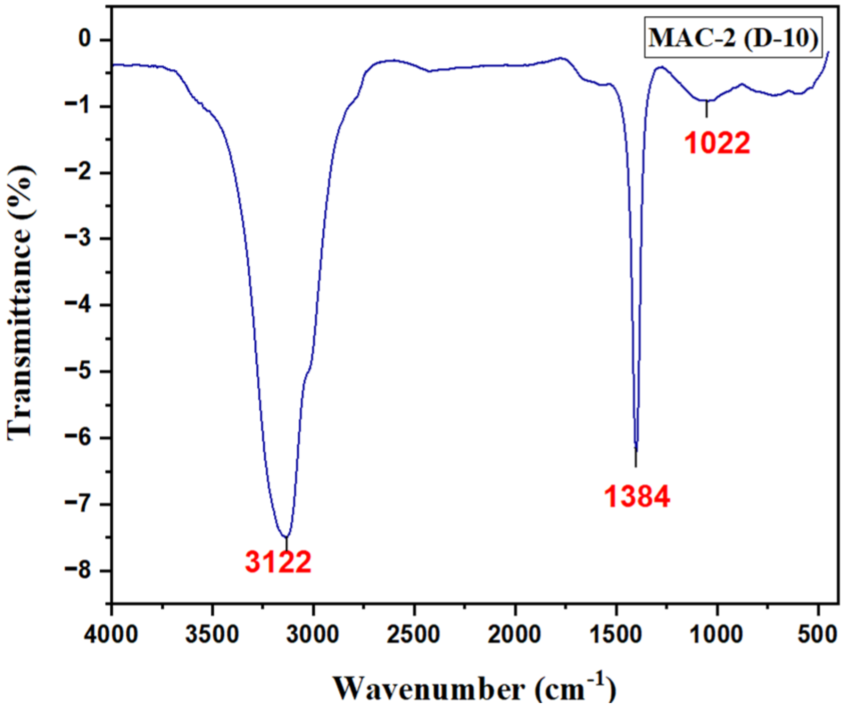


Fig 6.29. FTIR of MAC-2 (10 min desorption)

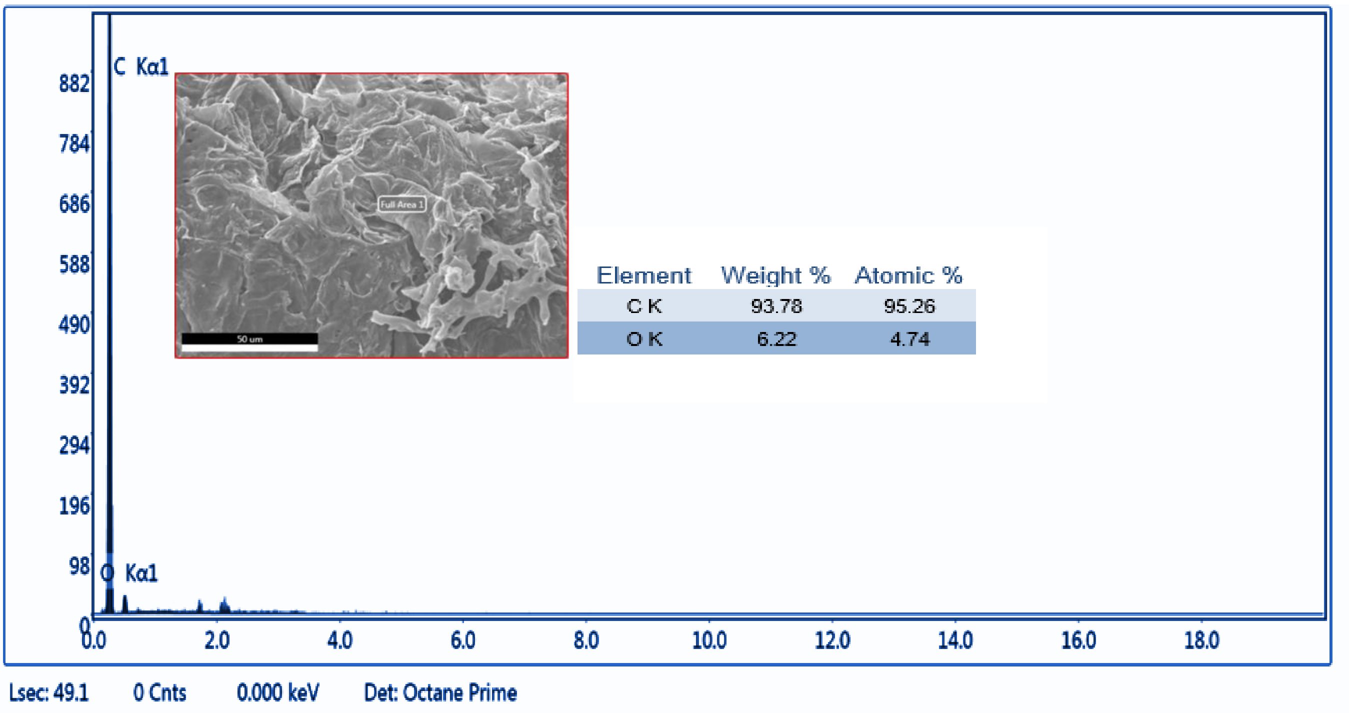
6.6. Energy Dispersive Spectroscopy (EDS) analysis

The EDS spectrum reveals that the samples exhibit a significant presence of Carbon and Oxygen elements, while also displaying a minor quantity of Potassium, which can be considered negligible. After the process of adsorption, the obtained samples exhibited Carbon contents of 95.26% and 92.84%, along with Oxygen contents of 4.74% and 7.16%. The desorption samples exhibit the presence of 95.89% and 96.26% carbon, along with 4.11% and 3.74% oxygen. The presence of Carbon and Oxygen in the samples indicates the incorporation of CO_2. Following the desorption process, there is a slight drop in the quantity of oxygen. The presence of Potassium in activated carbon is negligible as a result of the washing process following activation with KOH. **Table 9** presents the atomic weight (%) of the elements as determined by EDS.

Table 9. EDS elemental atomic weight (%)

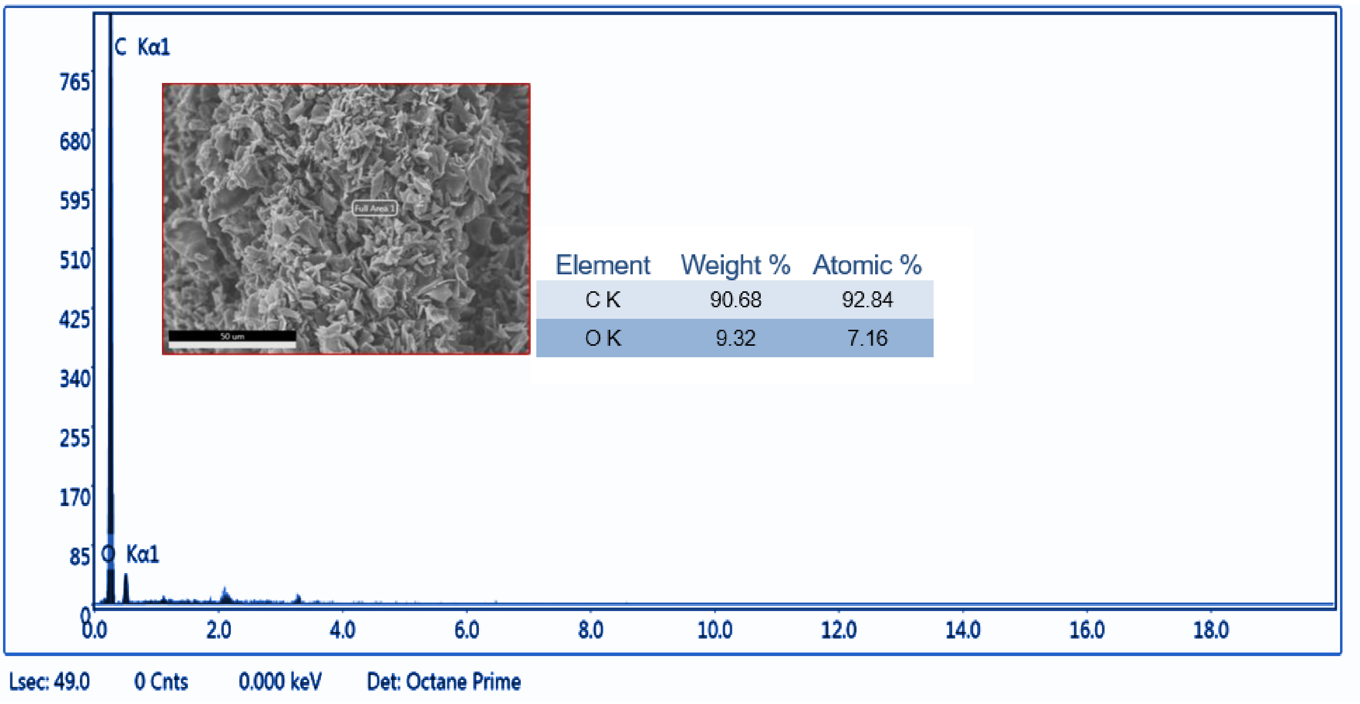
Type	Sample	Carbon (%)	Oxygen (%)
Adsorption	MAC-1	95.26	4.74
	MAC-2	92.84	7.16
Desorption	MAC-1	95.89	4.11
	MAC-2	96.26	3.74

Figures 6.30 to 6.33 depict the Energy Dispersive Spectroscopy (EDS) results obtained from the samples subjected to adsorption and desorption processes.



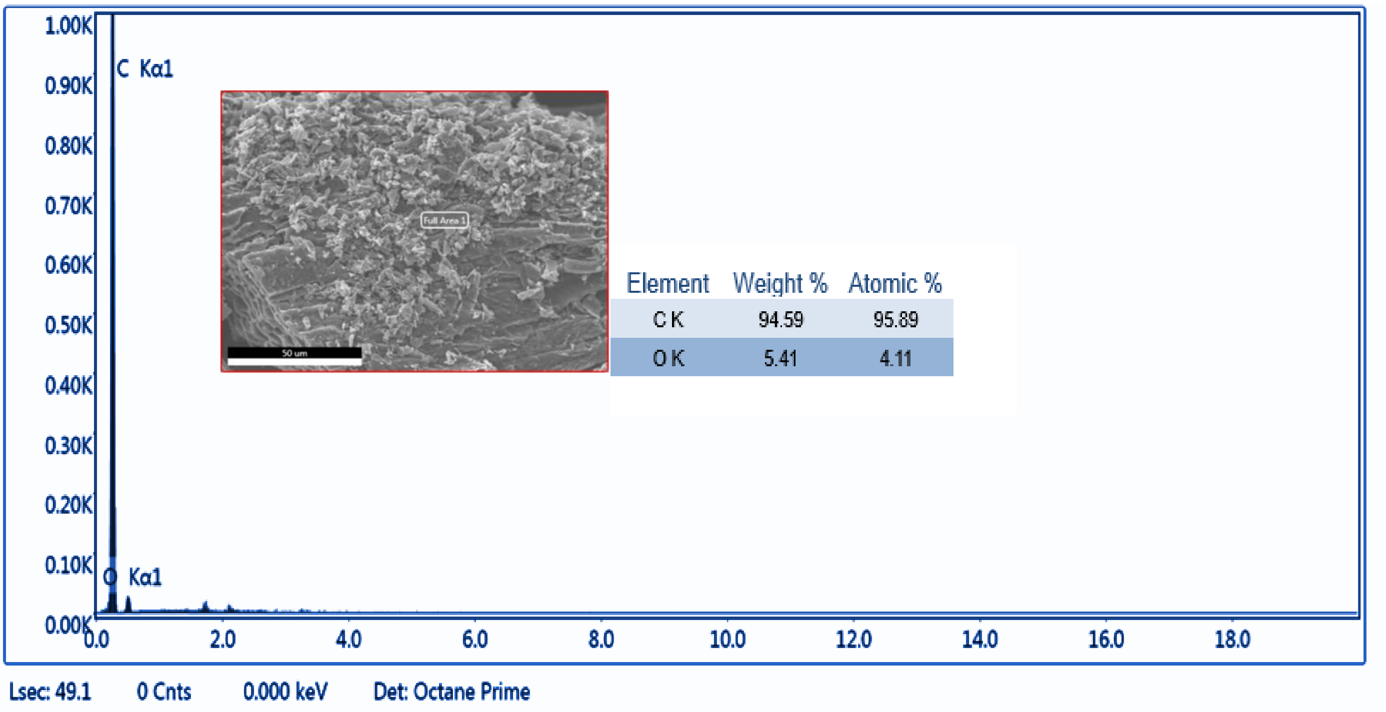
Element	Weight %	Atomic %
C K	93.78	95.26
O K	6.22	4.74

Fig 6.30. EDS of MAC-1 (20 min adsorption)



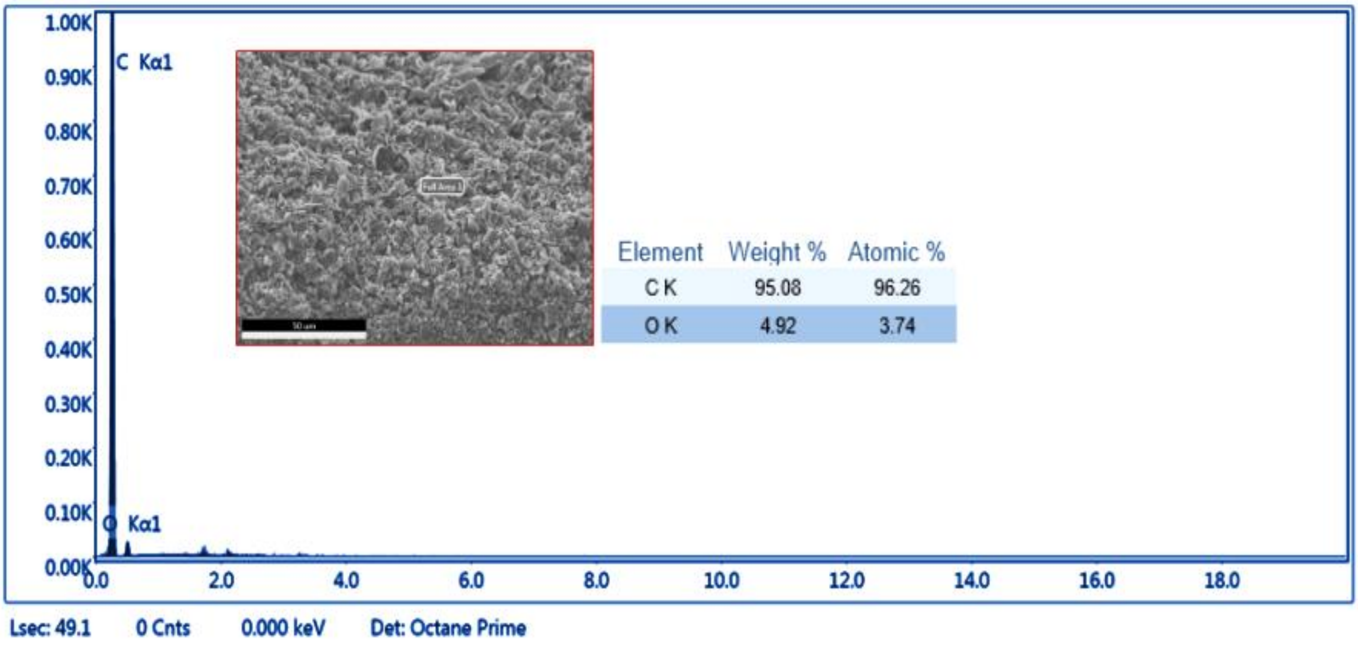
Element	Weight %	Atomic %
C K	90.68	92.84
O K	9.32	7.16

Fig 6.31. EDS of MAC-2 (20 min adsorption)



Element	Weight %	Atomic %
C K	94.59	95.89
O K	5.41	4.11

Fig 6.32. EDS of MAC-1 (20 min desorption)



Element	Weight %	Atomic %
C K	95.08	96.26
O K	4.92	3.74

Fig 6.33. EDS of MAC-2 (20 min desorption)

CHAPTER SEVEN

CONCLUSION AND RECOMMENDATIONS

Conclusion and Recommendations

7.1. Conclusion

The reduction of carbon dioxide (CO_2) emissions represents a pivotal approach employed to address the pressing environmental issue of global warming. The unique texture of activated carbon has led to its recognition as a promising adsorbent for carbon dioxide (CO_2) in several applications. Biochar was produced from corn cobs using a carbonization process conducted at a temperature of 800°C for a duration of 4 hours. This work focuses on the characterization of activated carbon derived from corncobs utilizing a chemical agent, specifically potassium hydroxide (KOH), at two distinct ratios (1:1 and 1:3). Two alternative methodologies were employed to carry out the activation process. The study of the activated carbons using scanning electron microscopy (SEM) reveals the presence of distinct pore structures resembling honeycombs, which exhibit a uniform distribution across the material. The distinct nature of pore spaces is greater in the 1:1 activation method as compared to the 1:3 activation method. This contradicts the hypothesis that increasing the molarity of KOH results in the formation of carbon with larger and more well-defined pore sizes. The activation of activated carbon through the use of KOH has the potential to greatly enhance its structural characteristics. The adsorption and desorption studies were conducted using samples with a 1:1 ratio.

The present study also involved an analysis of the adsorption and desorption experiment of carbon dioxide (CO_2). Adsorption experiments were conducted for three different durations: 5, 10, and 15 minutes, using samples MAC-1 and MAC-2. The experimental results revealed that MAC-2 had the greatest adsorption capacity, measuring 1.523 mmol/g, after a duration of 10 minutes. It was also observed that as the duration of time increased, there was a noticeable decline in the rate of adsorption, ultimately leading to the saturation of CO_2 after a period of 20 minutes. The process of desorption was conducted, followed by subsequent adsorption at a temperature of 105°C for a duration of 60 minutes. The graph depicting the desorption process exhibited a noticeable downward trend as the duration of the process increased. The scanning electron microscopy (SEM) analytical images demonstrate that the pore spaces experience occupancy by CO_2 during adsorption, and then exhibit a partial regaining of visibility upon desorption. The Fourier Transform Infrared (FT-IR) analysis revealed the existence of several functional groups following the processes of adsorption and desorption. Notably, the O-H group was seen in all of the samples. The EDS spectra indicated that Carbon (C) was the predominant element present in all of the samples. The presence of Potassium (K) was difficult to determine due to the post-washing of the samples following activation. The activated carbon demonstrated promising CO_2 adsorption properties, implying its potential as an absorbent for CO_2 capture. The present study exhibits a cost-effective and sustainable method for producing porous carbon materials that can efficiently capture CO_2 from biomass. The high surface area and porosity of the prepared activated carbons (ACs), along with their excellent dynamic CO_2 adsorption capacities (> 1 mmol g^{-1}), make them advantageous for capturing CO_2 at ambient temperatures.

7.2. Recommendations

- To acquire a comprehensive understanding of the impact of KOH on pore structure, it is recommended to conduct additional experiments using varying activation ratios. This method can optimize the properties of activated carbon to meet specific requirements.

- Further investigation should be conducted to examine the influence of activation methods on pore structure. Specifically, future research should consider additional activation parameters, including duration and temperature, in order to enhance the development of distinct and well-defined pore structures in activated carbon.

- Extended adsorption experiments conducted under different conditions can provide insights into the enduring stability and effectiveness of the material. This has the potential to enhance the applicability of the process. Continuous monitoring exceeding 20 minutes may indicate higher levels of adsorption and saturation.

- A study should be conducted to determine the extent to which the production method may be modified in order to meet prospective industrial applications.

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APPENDIX

Supplementary table 1: Bonds present in the sample from FTIR analysis

Bond	Range in experiment	References
O-H	3122-3697 cm^{-1}	(Figueiredo et al., 1999)
C=O	1384 to 1394 cm^{-1}	(Ramadoss & Muthukumar, 2015)
C=C	1630-1650 cm^{-1}	(Kamalini et al., 2018)
C-O	980-1060 cm^{-1}	(Saleh et al., 2014)
K-O	600-702 cm^{-1}	(Hossain et al., 2021)