# Development of Electrochemically Modified Biochar Adsorbents for Enhanced Recovery of Phosphorus

A

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Submitted by

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# Certificate

This is certified that the thesis entitled "**Development of Electrochemically Modified Biochar Adsorbents for Enhanced Recovery of Phosphorus**" submitted by **Srishti (Roll No. 302101027)**, a postgraduate student of the Department of Biotechnology in partial fulfilment for the award of the degree of **Master's of Science** at Thapar Institute of Engineering and Technology, Patiala, Punjab 147004, India, is a record of student's own work carried out under my supervision and guidance. This report has not been submitted for the award of any other degree or certificate in this institute or any other university or institute.

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# Declaration

I hereby declare that the work presented in the dissertation entitled "**Development of Electrochemically Modified Biochar Adsorbents for Enhanced Recovery of Phosphorus**" in partial fulfilment of the requirement for the award of the degree of Master of Science in Biotechnology, Department of Biotechnology, Thapar Institute of Engineering and Technology (TIET) Deemed to be University, Patiala, is a genuine record of my own work during the period from January 2023 to June 2023, under the supervision of Dr. Bunushree Behera, Department of Biotechnology, TIET. The matter incorporated in this thesis has not been submitted to any other university or institute for the award of any degree in India or abroad.

Srishti.

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Srishti

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# List of Symbols And Abbreviations

| **Symbol / Abbreviation** | **Meaning** |
|---|---|
| -COOH | Carboxyl group |
| -OH | Hydroxyl group |
| °C | Degree Celsius |
| % | Percentage |
| $\theta$ | Theta |
| µS/cm | Micro siemens per centimetre |
| A | Ampere |
| A/cm$^2$ | Amperes per square centimetre |
| BC 400 | Biochar produced at 400°C |
| BET | Brunauer-Emmett-Teller |
| BM | Biomass |
| C | Carbon |
| $Ca(OH)_2$ | Calcium hydroxide |
| cc/g | Cubic centimetres per gram |
| cm$^{-1}$ | Inverse centimetres |
| cm$^2$ | Square centimetres |
| Cu(II) | Copper with a +2 charge |
| DC | Direct Current |
| dil. | Dilute |
| EC | Electrical Conductivity |
| EDS | Energy-dispersive X-ray spectroscopy |

| | |
|---|---|
| Eqs | Equations |
| et al. | And all |
| eV | Electronvolt |
| FTIR | Fourier-transform infrared spectroscopy |
| g | Gram |
| g/L | Grams per litre |
| H | Hour |
| $H_3PO_4$ | Phosphoric acid |
| $K_2CO_3$ | Potassium carbonate |
| KCl | Potassium chloride |
| KOH | Potassium hydroxide |
| kV | Kilovolt |
| M | Molar |
| $m^2/g$ | Square meters per gram |
| mg | Milligram |
| $mg\ g^{-1}$ | Milligrams per gram |
| mg/L | Milligrams per litre |
| $MgCl_2$ | Magnesium chloride |
| $MgNH_4PO_4.6H_2O$ | Magnesium ammonium phosphate hexahydrate |
| ml | Millilitre |
| N | Nitrogen |
| N | Normality |
| NaOH | Sodium hydroxide |
| $NH_4HCO_3$ | Ammonium bicarbonate |

| | |
|---|---|
| nm | Nanometre |
| O | Oxygen |
| OD | Optical Density |
| P | Phosphorus |
| Pb(II) | Lead with a +2 charge |
| pH | Potential of Hydrogen |
| Post-EAC BC 400 | Biochar produced at 400°C with Post Electrochemical treatment |
| Pre-EAC BC 400 | Biochar produced at 400°C with Pre Electrochemical treatment |
| rpm | Rotations per minute |
| SEM | Scanning Electron Microscopy |
| Si | Silicon |
| V | Volt |
| XRD | X-ray diffraction |
| $ZnCl_2$ | Zinc chloride |

# Abstract

The overall aim was to develop efficient biochar-based adsorbents using an easy and time-efficient approach for improving the performance of recovering nutrients from sewage wastewater for subsequent real-time application. To achieve a sustainable nutrient recovery and utilization, natural adsorbents from carbonaceous rich biochar has gained attention. The study focussed to develop an electrochemically modified nano-biochar for enhanced removal of nutrients from sewage wastewater. Biochar was produced via pyrolysis of wheat straw at 400°C. To enhance biochar's adsorption capacity, selectivity, and reusability, the biochar was modified electrochemically. Several other forms of analysis, including elemental analysis, surface area analysis, and proximate analysis, were used to characterize the biochar samples. According to the findings, the elemental composition, surface area, pore structure, and physicochemical properties of the biochar adsorbents that had been electrochemically modified were significantly altered. On comparison with the untreated biochar, the modified biochar exhibited a greater specific surface area, increased porosity, and enhanced capacity for phosphorus adsorption. These improved structural characteristics were primarily responsible for more efficiency of biochar adsorbents that have been modified electrochemically. The adsorption capacity of pre-treated biochar at 400°C was calculated to be 0.43 mg/g and for post-treated biochar, the adsorption capacity was found to be 0.2 mg/g, however it was comparatively low for untreated biochar (0.43 mg/g). These results support the potential application of electrochemically modified biochar in sustainable wastewater treatment for phosphorus recovery.

**Keywords :** Electrochemically modified biochar, Adsorption, Phosphorus recovery, Sewage wastewater, Wheat Straw, Pyrolysis

# Chapter 1 : Introduction

The introduction chapter of this thesis, "Development of Electrochemically Modified Biochar Adsorbents for Enhanced Recovery of Phosphorus from Sewage Wastewater," gives a comprehensive account of the research and the importance of the study. The introduction emphasizes the need for sustainable technologies to remove and recover phosphorus from wastewater.

## 1.1 Background of the Study

The development of electrochemically modified biochar adsorbents for improved phosphorus recovery from sewage effluent is the main objective of this study. Rapid urbanization and population expansion have increased the production of wastewater, which frequently contains high levels of phosphorus. The excessive discharge of high-phosphorus effluent into natural water bodies results in eutrophication and ecological imbalances. **(Kamilya et al., 2022)**. Phosphorus is an essential component for plant growth and for the production of food and other agricultural products. However, due to its limited reserves and improper fertilizer use, there is growing concern about the depletion of phosphorus **(Alewell et al., 2020)**. Phosphorus scarcity can have a detrimental influence on food security because of the dependence of agricultural output on phosphorus-derived fertilizers **(Vu et al., 2023)**. A few successful strategies for phosphorus recovery have already been discovered **(Zahed et al., 2022)**. For phosphorus recovery, a variety of technologies are being used, such as chemical precipitation, biological phosphorus removal, innovative chemical precipitation techniques, and other wastewater and sludge-based techniques. **(Law et al., 2016)**. However, these technologies are not economical and have not yet been scaled up to the industrial level.

Burning crop residue in India contributes to poor air quality and imposes a health burden on the population **(Lan et al., 2022)**. Sustainable techniques, such as composting, biochar production, and mechanization, can be used as alternatives to crop residue burning **(Bhuvaneshwari et al., 2019)**.

Biochar is produced when organic materials are thermally broken down (pyrolyzed) at high temperatures (300–800 °C) in the absence of oxygen. Biochar is proposed to be an efficient adsorbent because of its huge surface area (200–1000 $m^2/g$), low density, and high porosity. **(Perez-Mercado et al., 2018)**. Biochar can be used to enhance the physical, chemical, and fertility qualities of soil. **(Kapoor et al., 2022)**.

Biochar can also be used to remove phosphorus. Biochar produced from sesame straw through pyrolysis at 300 °C had phosphorus adsorbing efficiency of 62.6 mg $g^{-1}$, 90.6 mg $g^{-1}$ at 400 °C, 118.1 mg $g^{-1}$ at 500 °C and 168.2 mg $g^{-1}$ at 600°C **(Park et al., 2015)**. $Ca(OH)_2$ pre-treated rice straw was used to prepare calcium-rich biochar, which was employed to remove phosphate from aqueous solutions. The highest phosphate sorption capacity of the synthesized biochar composites was 197 mg $g^{-1}$ **(Liu et al., 2019)**. However, the implementation of chemical activation procedures may be costly and difficult. Heat-intensive techniques and high temperatures are frequently used for chemical activation.

In recent years, studies have been conducted on electrochemical activation methods for the modification of biochar. The electrochemical modification method is cost-effective and less time-consuming. It enhances the characteristics of biochar to improve its adsorption efficiency **(Mahdi et al., 2019)**.

This study is basically about how biochar could be used as an efficient adsorbent to recover phosphorus from wastewater.

# Chapter 2 : Review of Literature

## 2.1 Introduction

The increasing global demand for phosphorus and the environmental consequences of its inefficient use and discharge into water bodies have prompted research into sustainable and efficient phosphorus recovery technologies. Phosphorus is a non-renewable resource that is essential for agricultural productivity **(Wu et al., 2021)**. Discharging sewage wastewater into aquatic ecosystems has resulted in eutrophication, endangering human and environmental health **(Hu et al., 2020)**.

Researchers are investigating methods of phosphorus recovery from wastewater effluent. Phosphorus can be removed and recovered cheaply and sustainably using biochar adsorbents. Biochar, a carbonaceous material made from the pyrolysis of organic waste, is a promising phosphorus adsorbent due to its high surface area, porous structure, and adsorption capacity **(Sun et al., 2022)**.

This literature review evaluates research on electrochemically modified biochar adsorbents and other conventional methods for producing activated biochar for phosphorus recovery from sewage wastewater. This review will assess the sustainability of biochar adsorbents modified electrochemically for phosphorus recovery by examining the current state of knowledge, key findings, and research gaps.

The review will go through the effects of phosphorus recovery from sewage wastewater on the environment and the economy. The special characteristics of biochar and phosphorus adsorption mechanisms will be covered. The studies done so far on electrochemical modification to improve biochar adsorption are studied in the review.

The review will summarise the essential findings and identified research gaps, laying the groundwork for future research on electrochemically modified biochar adsorbents for efficient phosphorus recovery from sewage wastewater. This analysis will enhance wastewater treatment technologies, reduce phosphorus pollution, and promote the circular economy by converting waste into resources.

### 2.1.1 Phosphorus Recovery from Wastewater

In recent years, wastewater phosphorus recovery has gained popularity due to phosphorus pollution and the growing demand for a finite resource. Diverse methods for removing and recovering phosphorus from wastewater have been investigated to reduce eutrophication and promote sustainable phosphorus use **(Kaljunen et al., 2022)**.

The efficacy of chemical precipitation, biological treatment, and membrane filtration for phosphorus removal from wastewater has been evaluated **(Khan et al., 2023)**. These procedures are effective, but generate much sediment and require much energy. Consequently, alternative phosphorus recovery techniques such as crystallization and precipitation are widespread **(Daneshgar et al., 2018) (Li et al., 2019)**.

Phosphorus is recovered via struvite precipitation. In effluent, magnesium and ammonium ions precipitate struvite ($MgNH_4PO_4.6H_2O$) **(Ha et al., 2023)**. The recovery of struvite removes phosphorus and generates fertilizer **(Vasa et al., 2021)**. To optimize struvite precipitation, numerous studies have examined various conditions.

Membrane filtration and ion exchange, have also been studied for phosphorus recovery **(Kekre et al., 2023)**. Phosphorus compounds can be selectively separated and concentrated for recovery through reverse osmosis and nanofiltration. By selectively absorbing phosphorus ions, ion exchange resins can regenerate and reuse effluent **(Xia et al., 2021), (Chen et al., 2023)**.

Electrochemical and biological techniques for phosphorus recovery are promising. Phosphorus can be precipitated, adsorbed, and electrochemically reduced by electrocoagulation and electrooxidation **(Lagum et al., 2021)**. EBPR and microalgae cultivation utilize natural organisms to recover phosphorus from effluent **(Trebuch et al., 2023)**.

Phosphorus can be extracted and reclaimed by sorbents **(Zhang et al., 2022)**. Phosphorus absorption in wastewater has been evaluated using activated carbon, biochar, and modified clays **(Lagum et al., 2021) (Priya et al., 2022)** . These materials have large surface areas and unique chemical properties, which allow for efficient phosphorus binding **(Sun et al., 2022) (Qiu et al., 2022)** .

Technologies for phosphorus recovery are selected based on effluent characteristics, product quality, economic feasibility, and environmental considerations **(Canziani et al., 2023)**. Understanding the advantages, disadvantages, and applicability of phosphorus recovery methods is crucial for developing sustainable and efficient wastewater treatment systems **(Yu et al., 2022) (He et al., 2022)**.

### 2.2 Biochar Production through Pyrolysis

Pyrolysis is a widely used technique for the production of biochar from a variety of biomass types. Pyrolysis is a thermal decomposition process that converts biomass to biochar, a carbon-rich solid residue **(Hung et al., 2023)**. The process involves the decomposition of organic compounds present in the biomass at a particular temperature and in an oxygen-limited environment. The pyrolysis product is affected by process temperature, residence time, biomass type, and heating rate, among other variables **(Kim et al., 2015)**. Various reactor types, such as paddle kilns, bubbling fluidized beds, wagon reactors, and agitated sand rotating kilns, are used for biochar production **(Yaashikaa et al., 2020)**. The biochar yield during the pyrolysis process is influenced by the type and composition of the biomass used. Temperature is the most critical operating process condition that determines product efficacy. Increasing the temperature during the pyrolysis process generally reduces the biochar yield **(Li et al., 2023)** **(Chen et al., 2022)**.

The yield of biochar produced from lignocellulosic biomass from a few different studies has been summarized in Table 1. These studies also lead to the conclusion that an increase in pyrolysis temperature results in a decrease in biochar production yield.

***Table 1*** *: BC Production Yield of Lignocellulosic Biomasses under different pyrolysis conditions.*

| **S. No.** | **Biomass** | **Pyrolysis Temperature** | **Yield (%)** | **Reference** |
|---|---|---|---|---|
| 1 | Rice Straw | 400°C | 36.19 | Zong et al., 2021 |
| 2 | Rice Straw | 550°C | 32.68 | Zong et al., 2021 |
| 3 | Wheat straw | 400°C | 35.76 ± 0.11 | Zhang et al., 2020 |
| 4 | Wheat straw | 600°C | 31.55 ± 0.28 | Zhang et al., 2020 |
| 5 | Rice straw | 600°C | 37.6 | Gao et al., 2023 |
| 6 | Rice straw | 700°C | 36.6 | Gao et al., 2023 |

### 2.3 Applications of Biochar

Biochar, a carbon-rich material produced through pyrolysis, has gained wide attention in recent years due to its range of applications in various fields **(Danesh et al., 2022)**. This review aims to provide an overview of the literature regarding the applications of biochar and its potential benefits.

Majorly, biochar is applied in agriculture. Biochar has positive effects on soil fertility and agricultural output **(Wang et al., 2022) (Jeffery et al., 2017)**. Biochar's porous structure helps plants retain water and nutrients, reducing irrigation and fertilizer requirements **(Zhang et al., 2018)**. Biochar can also enhance soil structure and microbial activity, promoting plant health and nutrient cycling **(Zhang et al., 2023)**.

Biochar has the potential for environmental remediation. Due to its large surface area and absorption capacity, it removes contaminants from the air, water, and soil. Biochar can remove heavy metals, organic contaminants, and even pharmaceuticals **(Ji et al., 2022)** (**Liu et al. 2017) (Yao et al. 2018)**. Biochar can be activated or impregnated with functional groups to increase its pollutant removal capacity **(Zeghioud et al., 2022)**.

Biochar has energy and agricultural applications **(Hamidzadeh et al., 2023)**. Biochar, a by-product of bioenergy production, sequesters carbon and reduces emissions of greenhouse gases. Biochar can be used as a sustainable fuel either directly or as biomass granules or briquettes **(Sarker et al., 2023) (Mishra et al., 2023)**. These energy applications offer a sustainable alternative to fossil fuels and contribute to controlling carbon emissions and global warming **(Zahed et al., 2021)**.

Biochar has also been investigated in the contexts of water filtration, animal husbandry, and waste management**(Ahmad et al., 2018)**. Biochar-based filters remove contaminants from wastewater affordably and without affecting the environment **(Kammann et al., 2018)**.

Biochar as a feed supplement enhances digestive health and reduces cattle methane emissions **(Man et al., 2021)**. Additionally, biochar can remediate organic waste and decontaminate contaminated landfills **(Kanwar et al., 2023) (Li et al., 2019)**.

In conclusion, biochar has multiple applications. Agriculture, energy, environmental remediation, animal husbandry, and waste management; all utilize biochar. However, further research is required to determine the economic viability, manufacturing techniques, and long-term effects of biochar-based applications **(Mishra et al., 2023)**.

### 2.4 Properties of Biochar

Biochar's efficacy in various applications is primarily determined by its physical properties. Biochar's water-holding capacity, nutrient retention, and microbial activity are substantially influenced by its porosity and surface area **(Ghorbani et al, 2023) (Adhikari et al., 2022)**. The pore structure, particulate size distribution, and specific surface area of biochar are significant factors in determining its adsorption capacity, gas exchange, and overall stability **(Liao et al., 2022) (Jeffery et al., 2017)**. In addition, the presence of biochar in soil has been shown to improve soil structure, resulting in improved water infiltration, decreased soil erosion, and increased soil fertility **(Gholamahmadi et al., 2023) (Maisyarah et al., 2023)**.

Biochar's chemical properties are predominantly determined by the feedstock composition and pyrolysis conditions **(Cao et al., 2022)**. Biochar possesses a complex carbon matrix consisting of hydroxyl, carboxyl, and phenolic functional groups, contributing to its interactions with contaminants and nutrients **(Wang et al., 2016) (Majumder et al., 2023)**. The pH and electrical conductivity of biochar are crucial factors in determining its behaviour in soil environments and its capacity to alter soil pH **(Tan et al., 2022)**. In addition, the presence of phosphorus, potassium, and calcium in biochar contributes to its fertilization potential and nutrient-cycling capabilities **(Fachini et. al., 2023)**.

It has been demonstrated that biochar has significant effects on soil microbial communities, plant growth, and soil health as a whole. The unique physicochemical properties of biochar, which provide a suitable habitat for microorganisms, account for its ability to increase soil microbial activity and diversity **(Kuzyakov et al., 2019)**. Increased abundance and diversity of beneficial microorganisms, such as mycorrhizal fungi and nitrogen-fixing bacteria, have been recorded in biochar-amended soils **(Qi et al., 2022) (Chan et al., 2018)**. In addition, biochar amendments have been shown to increase nutrient availability, stimulate plant growth, and increase crop yield in various agricultural systems **(Hou et al., 2022) (Chen et al., 2022)**.

### 2.5 Activation of Biochar

Activation of biochar is the process of improving its physical, chemical, and biological properties by applying external agents or modifications **(Ambika et al., 2022) (Chen et al., 2022)**. The activation of biochar may include physical, chemical or biological treatment in order to modify its structure and properties. These modifications improve its surface area, pore structure, and functional groups, resulting in enhanced adsorption capacity, pollutant removal, soil fertility, and carbon sequestration capability **(Elkhlifi et al., 2023) (Tan et al., 2022)**.

#### 2.5.1 Physical and Chemical Activation

Physical and chemical activation techniques have been applied to activate biochar and their effects on its physicochemical properties.

Physical activation modifies the structure and surface area of biochar. Thermal treatment, steam activation, and gasification are the standard techniques. Thermal treatment, such as high-temperature carbonization, removes volatile compounds and increases biochar porosity **(Tang et al., 2019)**. Utilizing steam as an activating agent creates additional pores in biochar, thereby increasing its surface area and adsorption capacity **(Zhang et al., 2017)**. The partial oxidation of biochar during gasification results in the formation of highly porous structures and enhanced reactivity **(Sun et al., 2022)**. These physical activation methods have been discovered to considerably enhance the adsorption capacity and catalytic properties of biochar, making it suitable for applications such as water treatment and soil remediation.

Chemical activation modifies the surface properties and functional groups of biochar through the impregnation of chemical agents. Alkaline compounds like potassium hydroxide (KOH), sodium hydroxide (NaOH), and phosphoric acid (H3PO4) are frequently used as chemical activators **(Neme et al., 2022) (Thithai et al., 2020)**. These chemicals react with biochar, to create micropores and mesopores and introduce functional groups **(Sajjadi et al., 2019)**. Chemical activation can considerably increase the biochar's surface area, pore volume, and absorption capacity **(Park et al., 2021)**. In addition, it can modify the surface chemistry of biochar to enhance its selectivity and performance in specific applications, such as heavy metal removal and gas adsorption **(Yang et al., 2019)**.

Overall, both physical and chemical activation techniques can effectively enhance biochar's performance and expand its possible applications. Table 2 presents a summary of several studies focused on the utilization of physical and chemical activation techniques.

*Table 2 : Review of Literature on Physical and Chemical Activation of Biochar*

| **S. No.** | **Biomass Used** | **Pyrolysis Temperature** | **Electrochemical Method** | **Key Finding** | **Reference** |
|---|---|---|---|---|---|
| 1 | Sawdust | 300°C | Steam activation | Amount of Phosphorus adsorbed :<br>Biochar : 62.7 mg $g^{-1}$<br>Biochar-Steam Activated : 64.0 mg $g^{-1}$ | Lou et al., (2016) |
| 2 | Sawdust | 550°C | Steam activation | Amount of Phosphorus adsorbed :<br>Biochar : 69 mg $g^{-1}$<br>Biochar-Steam Activated : 78.6 mg $g^{-1}$ | Lou et al., (2016) |
| 3 | Oil-palm Shells | 350°C | Chemical activation with $K_2CO_3$ | Amount of Phosphorus adsorbed : 0.59 mg/g | Munar-Florez et al., (2021) |
| 4 | Oil-palm Shells | 650°C | Chemical activation with $K_2CO_3$ | Amount of Phosphorus adsorbed : 0.89 mg/g | Munar-Florez et al., (2021) |
| 5 | Oil-palm Shells | 750°C | Chemical activation with $K_2CO_3$ | Amount of Phosphorus adsorbed : 0.46 mg/g | Munar-Florez et |

|  |  |  |  |  |  |
|---|---|---|---|---|---|
|  |  |  |  |  | al., (2021) |
| 6 | Sewage sludge | 600-900°C | $NH_4HCO_3$ activation | Improved adsorption capacity for Pb(II) ions (92.80 mg $g^{-1}$) | Xia et al., (2019) |
| 7 | Switchgrass | 500-800°C | Potassium acetate activation | Increased surface area and micropore volume of biochar.<br>Adsorption capacity increased to 209.65 mg/g. | Liu et al., (2022) |

### 2.5.2 Limitations of Physical and Chemical Activation

Physical activation techniques, such as steam or carbon dioxide activation, can produce biochar adsorbents with a limited surface area. This can impact the total phosphorus adsorption capacity **(Panwar et al., 2020)**. Physical activation techniques may result in a range of pore diameters that may not be optimal for phosphorus adsorption. The size and distribution of fissures within the biochar matrix can influence phosphorus adsorption **(Panahi et al., 2020)**. Physical activation processes may not introduce functional groups to the surface of biochar, thereby limiting the number of interaction sites for phosphorus adsorption **(Zhang et al., 2020)**. Chemical activation methods frequently involve the use of environmentally unfriendly chemicals, such as strong acids or alkalis, which may necessitate additional waste management steps **(Baskar et al., 2022)**. The chemical activation process can leave residues on the surface of biochar, which may have an adverse effect on the biochar's phosphorus adsorption capacity or may introduce contaminants into the treated effluent **(Jellali et al., 2021)**. Chemical activation procedures can result in the loss of biochar's carbon content, thereby decreasing its phosphorus adsorption capacity **(Zhang et al., 2017)**.

Because of these restrictions, it is necessary to investigate alternative methods, such as electrochemical modification, in order to increase the adsorption capacity and selectivity for the purpose of phosphorus recovery from wastewater.

### 2.5.3 Electrochemical Activation of Biochar

In recent few years, the electrochemical activation of biochar has been an area of interest. It entails biochar to an electrochemical procedure, which results in the modification and improvement of its properties. Several studies have reported significant effects of this technique on the physicochemical properties of biochar, as well as its potential environmental remediation and agricultural applications **(Mukjerjee et al., 2022)**.

The modification of biochar's surface properties is one of the primary effects of electrochemical activation. The electrochemical treatment results in the formation of functional groups, such as oxygen-containing groups (-OH, -COOH), on the surface of biochar, which substantially increase its surface area and porosity **(Liu et al., 2022)**. This enhanced adsorption capacity makes biochar a promising material for water and soil remediation **(Smith et al., 2018) (Wang et al., 2019)**.

In addition, biochar's electrochemical behaviour is altered by its electrochemical activation. The improved electrochemical activity makes electrochemically activated biochar a suitable electrode material for use in energy storage devices, such as supercapacitors and batteries **(Li et al., 2020) (Zhang et al., 2021)**.

In addition to its physical and electrochemical modifications, biochar's chemical composition is also altered by its electrochemical activation. Various bioactive substances, such as organic acids, phenolic compounds, and plant growth regulators, can be released from biochar as a result of this process **(Chen et al., 2017) (Sun et al., 2020)**. These substances may increase soil fertility, promote plant growth, and stimulate microbial activity in the rhizosphere.

Overall, electrochemical activation of biochar has demonstrated tremendous promise for enhancing the properties and applications of biochar. Its ability to increase surface area, adsorption capacity, electrochemical behaviour, and release bioactive substances makes it an attractive material for environmental and agricultural applications **(Liu et al., 2021) (Garcia et al., 2022)**. A summary of a few studies based on electrochemical activation is given in Table 3.

***Table 3*** *: Review of Literature on Electrochemical Activation of Biochar*

| S. No. | Biomass Used | Pyrolysis Temperature | Electrochemical Method | Key Finding | Reference |
|---|---|---|---|---|---|
| 1 | Brown marine macroalgae | 450°C | Electro-modification using aluminium electrode | Adsorption with Biochar : 8.23 mg $g^{-1}$<br>Adsorption with Electro Modified Biochar : 31.28 mg $g^{-1}$ | Jung et al., (2015) |

| | | | | | |
|---|---|---|---|---|---|
| 2 | Marine brown algae | 500°C | Aluminium-electrode based electro-modification | Adsorption of Phosphate with Modified Biochar : 273.9 mg/g | Jung et al., (2016) |
| 3 | Marine brown algae | 600°C | Aluminium-electrode based electro-modification | Adsorption of Phosphate with Modified Biochar : 345.1 mg/g | Jung et al., (2016) |
| 4 | Marine brown algae | 900°C | Aluminium-electrode based electro-modification | Adsorption of Phosphate with Modified Biochar : 460.3 mg/g | Jung et al., (2016) |
| 5 | Corn straw | 600°C | Electrochemical activation with ferric chloride ($FeCl_3$) | Adsorption with Biochar : 99.7 mg $g^{-1}$<br>Adsorption with EM Biochar : 113.0 mg $g^{-1}$ | Yang et al., (2019) |

# Chapter 3 : Research Gaps And Objectives

## 3.1 Research Gaps

This chapter addresses the research gaps in the field of phosphorus recovery from sewage effluent using biochar adsorbents that have been modified electrochemically. This dissertation aims to advance strategies for sustainable phosphorus recovery from sewage wastewater by addressing these research gaps. Producing effective and affordable electrochemically modified biochar adsorbents could significantly enhance phosphorus recovery from wastewater.

A few of the research gaps which could be observed so far are:

### (i) Limited studies for electrochemical active lignocellulosic biomass

In the existing literature, only a few investigations on electrochemically active lignocellulosic biomass have been found. This research gap demonstrates a need for more studies and comprehension of the electrochemical properties and potential applications of lignocellulosic biomass in various electrochemical processes. Addressing this research void will contribute to the development of renewable energy technologies and the use of biomass resources in sustainable electrochemical systems.

### (ii) Limited studies to compare electrochemically treated biochar and untreated biochar

A comparative analysis needs to be done to compare the characteristics and properties of pyrolyzed biochar and modified biochar. This indicates the need for further research. Characterization could result in more reliable and accurate electrochemical activation. To resolve these gaps and enhance the application of electrochemical studies, it is necessary to characterize the electrochemically activated biochar.

## 3.2 Research Objectives

The primary objective of this research is to conduct a comprehensive study and then develop a cost-effective method for the synthesis of electrochemically activated biochar. Biochar, derived from biomass, has high carbon content and properties that make it appropriate for use in soil improvement, retention of carbon, and water purification. Electrochemical activation is a novel method for enhancing biochar's properties and applications.

Wheat straw, a readily available agricultural by-product, has been selected as the primary source material for this study. It is easily accessible crop residue after cereal harvesting, and its use for biochar production corresponds with the goals of sustainable waste management and utilization of resources.

The purpose of this entire study has been divided into two objectives, which are:

### (i) To Produce Electrochemically Activated Biochar from Wheat Straw

The primary objective of this study is to develop a technique for electrochemically activating wheat straw biochar. It includes designing and optimising an electrochemical activation system that converts biochar to electrochemically modified biochar through electrolysis control. The objective is to explore the influence of various parameters, such as electrolyte composition, electrode materials, current density, and activation duration, on the production of electrochemically activated biochar and, further, on the properties of biochar.

### (ii) To Comparatively Evaluate the Physicochemical Properties of Biochar

The other main objective is to analyse the biochar on the basis of its physicochemical properties. It includes the pH, EC, surface area, pore size distribution, elemental composition and functional groups. The objective is to compare the characteristics of electrochemically activated biochar with pyrolysis-produced biochar. This comparison considers how electrochemical activation affects the properties of biochar.

# Chapter 4 : Materials And Methods

## 4.1 Feedstock

Waste wheat straw was gathered from Ablowal village in Patiala city. The wheat straw was oven dried at 100°C for 24 h. The moisture content was discovered to be approximately 10%.

## 4.2 Biochar Production via Pyrolysis

The dried biomass was ground using a mixer grinder until it passed through a sieve with a size of 500 microns. In a lab-scale electrically heated Muffle Furnace, the biomass was pyrolyzed at 400°C with a heating rate of 10°C/min. The biochar was packed in ceramic crucibles. A lid and aluminium foil were used to seal the crucibles. The dried biomass samples were heated for a total of two hours **(Behera et al., 2020)**. After pyrolysis, the sample was once rinsed with dil. HCl (0.5N) to remove any ashes that might have remained. The sample was then repeatedly washed with distilled water until the pH reached 7.

## 4.3 Electro-Chemical Modification of Biochar

0.5N $MgCl_2$ was prepared in order to modify the biochar **(He et al., 2022)**. In a 500 ml beaker, 10g of biomass was added to 400 ml of the prepared $MgCl_2$ solution. A programmable DC power supply (32 V and 1 A) was used to apply the proper current density for electro-modification. Two stainless steel electrodes with an effective surface area of 12 $cm^2$ each were used for electro-modification **(Li et al., 2022) (Jung et al., 2016)**. The electrodes were fixed at a distance of 3 cm from each other in the beaker containing biomass dissolved in $MgCl_2$

solution. A current density of 0.083 A/cm$^2$ was delivered for 5 min while swirling the beaker continuously at 700 rpm with the help of a magnetic stirrer. The activated biomass was subsequently filtered using a Whatman filter paper. The sample was then oven-dried at 100°C for 4-5 hours. Later, the activated biomass was stored in a zip-lock bag. This biomass was further used to produce biochar through pyrolysis at 400°C. In a similar way, the biochar produced at 400°C was electro-chemically modified.

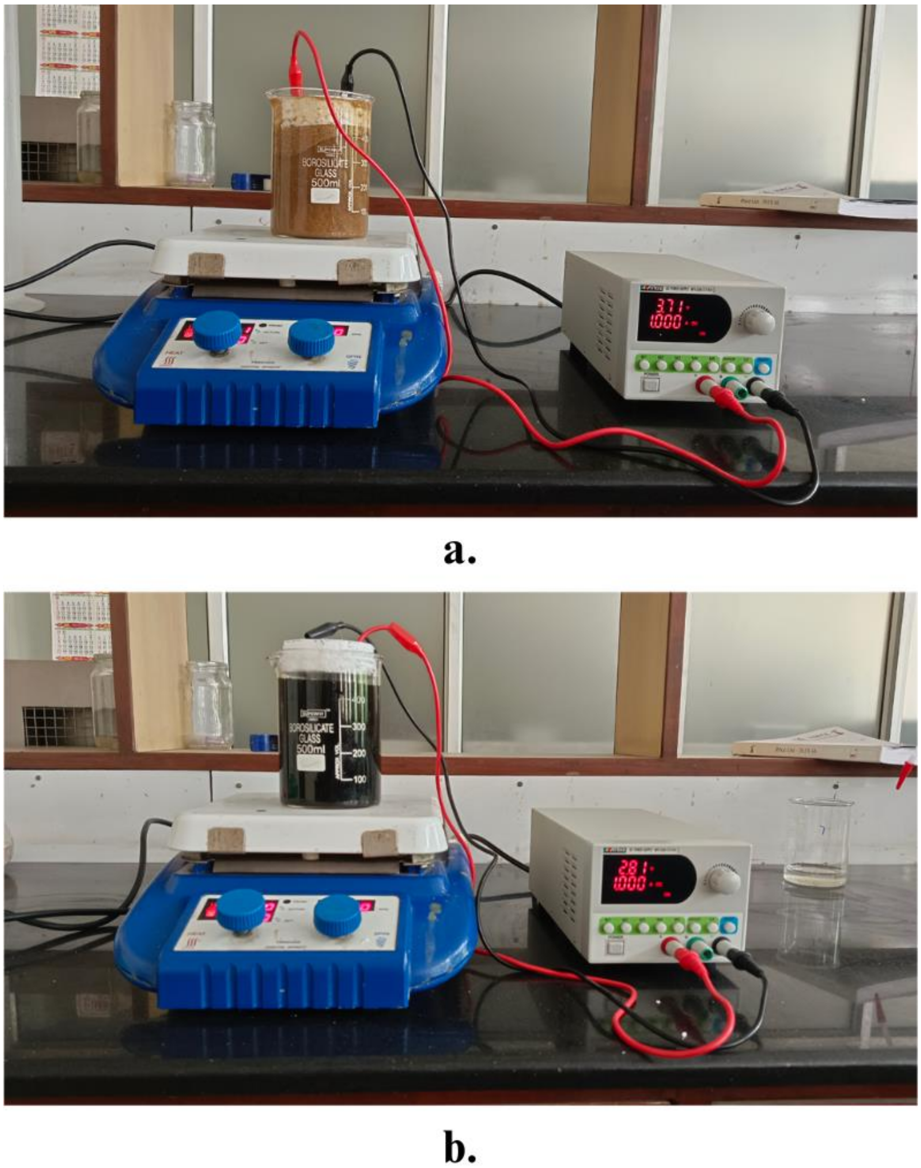

***Figure 1** : **a.** Electrochemical activation of BM **b.** Electrochemical activation of BC 400*

### 4.4 Characterization of Biochar

The chemical characterization of biochar includes a variety of techniques for elucidating its elemental composition, functional groups, chemical bonds, and crystalline structure **(Zong et al., 2021)**. Energy-dispersive X-ray spectroscopy (EDS) is used to determine the elemental composition, providing quantitative data on the presence and abundance of different elements. Fourier-transform infrared spectroscopy (FTIR) is used to identify functional groups and chemical bonds, thereby facilitating the evaluation of biochar's chemical composition and reactivity **(Hamissou et al., 2023)**. X-ray diffraction (XRD) analysis is utilized to investigate biochar's crystalline structure and mineralogy, providing information regarding its structural properties and potential interactions with other substances. BET (Brunauer-Emmett-Teller) characterization is a common technique used to determine biochar's specific surface area and porosity **(Zhong et al., 2023)**. These characterization techniques are crucial in obtaining comprehensive information on biochar's chemical properties and structure, elucidating its various applications and facilitating further scientific research.

#### 4.4.1 SEM-EDS

SEM-EDS was used to characterize the biochar samples. The biochar samples were coated with a fine layer of gold using a sputter coater before SEM-EDS analysis. The biochar samples were imaged at 100x, 250x, and 500x magnifications using a high-resolution scanning electron microscope (Carl Zeiss Sigma 500) with a resolution of 1.0 nm @ 30 kV.

The EDS analysis was conducted to determine the biochar's elemental composition by detecting characteristic X-ray emissions. EDS was conducted using a Bruker, QUANTAX 200 instrument with an Energy resolution of 129 eV. Biochar samples were analyzed under high vacuum conditions to ensure precise elemental analysis.

The interpreted SEM images and EDS spectra were then used to evaluate the biochar's morphological characteristics, such as shape and surface structure, as well as its elemental composition, which included carbon, oxygen, hydrogen, nitrogen, and any potential trace elements. The SEM-EDS characterization provided invaluable insights into the biochar's physical and chemical properties, facilitating the comprehension of its structure and composition for future analysis and potential applications.

### 4.4.2 BET Analysis

Using BET analysis, the specific surface area of biochar was determined. The biochar samples were deposited in a high-precision instrument capable of gas adsorption measurements according to the BET theory. Nitrogen gas was used as the adsorbate because of its inertness and ability to interact with the biochar surface. The BET instrument progressively exposed biochar samples to variable pressures of nitrogen gas, allowing adsorption to occur **(Thomas et al., 2013)**. The quantity of gas adsorbed at various pressures was then measured, and the obtained data were analyzed using the BET equation to determine the biochar's specific surface area. This method of characterization provides important data on the porosity and surface area of biochar.

### 4.4.3 XRD Analysis

XRD analysis was conducted using a Rikagu, SmartLab SE with Multipurpose θ-θ X-ray diffractometer with built-in intelligent Guidance. The biochar samples were placed on glass slides in the XRD chamber. The X-ray beam, generated by Cu-anode, was directed onto the biochar sample at an incident angle of range of 10°-80° at a scan rate of 10° per min with a step size of 0.01 min. The XRD instrument detected the X-rays diffracted by the biochar's crystal lattice.

The obtained XRD patterns were then analysed to determine the biochar's crystalline structure and mineral composition. Comparing the XRD patterns with known reference patterns enabled the identification of the diffraction peaks. The 2θ values associated with the peaks were recorded.

The XRD analysis provided valuable insights into the crystalline structure and degree of crystallinity of the biochar samples, contributing to a thorough characterization of the biochar material.

### 4.4.4 FTIR Analysis

Fourier transform infrared spectroscopy (FTIR) (High-performance high-resolution software) was used to examine the spectral properties of the raw feedstock and biochar samples. FTIR spectra with 0.3 $cm^{-1}$ resolution were acquired at wavelength range of 400 to 4000 $cm^{-1}$ with a combination of 128 scans. The spectral peak functional group characteristics for biochar samples were interpreted based on their characteristic vibrations.

## 4.5 Proximate Analysis

Proximate analysis is a widely used technique that offers valuable insights into biochar's physical properties. It involves determining approximate parameters such as fixed carbon, volatile matter, moisture content, and ash content. These parameters provide crucial information about biochar's thermal stability, combustion behaviour, and nutrient retention capacity. One can evaluate the suitability of biochar for specific applications, such as carbon sequestration, water purification, and soil fertility improvement, by employing proximate analysis. Consequently, a comprehensive characterization of biochar through proximate analysis is crucial for optimizing its production processes and investigating its diverse environmental and agricultural applications.

### 4.5.1 Moisture Content

The crucibles were washed, dried, and cleaned prior to the experiment. Empty crucibles were carefully weighed, and approximately 1 g of the ground sample was added to each crucible. The crucibles containing the samples were then placed in an oven set at 105°C for a duration of 2 hours to facilitate drying. After drying, the samples were transferred to a desiccator for an additional hour to reach equilibrium with the surrounding moisture levels. Finally, the crucibles with the dried samples were weighed to determine their final mass. These procedures followed standard scientific protocols and aimed to ensure accurate and precise measurements for the thesis **(Aller et al., 2017)**.

Moisture % = $[(A - B)/A] * 100$

Where: A = grams of air-dry sample used

B = grams of the sample after drying at 105°C

### 4.5.2 Volatile Matter

The muffle furnace was heated to a temperature of 950°C. Crucibles utilized for moisture determination were preheated, with lids securely in place, together with the sample. The preheating process involved placing the crucibles, with the furnace door open, on the outer edge of the furnace for 2 minutes, maintaining a temperature of 300°C. Subsequently, the crucibles were moved to the edge of the furnace and allowed to preheat for an additional 3 minutes at a temperature of 500°C. The samples were then transferred to the rear of the furnace and kept there for 6 minutes, ensuring the muffle door remained closed throughout this period. Continuous observation of the samples was facilitated through a tiny peephole in the muffle door to identify any occurrences of sparking, which could have impacted the accuracy of the results. Following the completion of the heating process, the samples were allowed to cool in a desiccator for 1 hour, after which their weights were determined **(Enders et al., 2017)**.

Volatile % = [(B – C)/B] * 100
Where: B = grams of the sample after drying at 105°C
C = grams of the sample after drying at 950°C

### 4.5.3 Ash Content

The crucibles employed for the determination of volatile matter were uncovered and positioned within the muffle furnace at a temperature of 750°C for a duration of 6 hours. Following the pyrolysis process, the crucibles were allowed to cool within a desiccator for 1 hour to attain thermal equilibrium. Subsequently, the crucibles were weighed to obtain the final mass of the samples **(Aller et al., 2017)**.

Ash % = (D/B) * 100
Where: B = grams of the sample after drying at 105°C

D = grams of the residue

### 4.5.4 Fixed Carbon

Fixed carbon refers to the portion of carbonaceous material that persists in a sample following the removal of volatile substances and moisture through heating or combustion. It is the proportion of carbon that does not readily vaporize or endure significant chemical changes during pyrolysis or combustion. Fixed carbon percentage can be calculated by subtracting the sum of moisture content %, Volatile matter % and Ash content % from 100.

Fixed Carbon % = 100% – (Moisture content % + Volatile matter % + Ash content %)

## 4.6 Physicochemical Properties of Biochar

The pH and electrical conductivity (EC) of biochar are important physicochemical properties that influence its functionality and interactions with the surrounding environment. The acidity or alkalinity of a substance is measured by its pH. pH is determined by the concentration of hydrogen ions present. EC, on the other hand, reflects a material's ability to conduct electrical current and is affected by the ion concentration in a solution **(Chellappan et al., 2018)**.

In this study, pH was measured with a pH meter. In contrast, electrical conductivity (EC) measurements were taken with a Digital Conductivity meter.

In order to prepare the biochar samples for analysis, 1g of each sample was immersed in deionized water with a solid-to-solution ratio of 1:10. Using a rotary agitator with a speed of 70 rpm for a duration of one hour. The samples were thoroughly mixed. The suspensions were then allowed to settle undisturbed for an additional hour before measurements were taken.

Before measuring pH, the pH meter was calibrated using standard pH buffers with pH values of 4, 7, and 9.2. Similarly, the EC meter was calibrated using 0.001 M KCl and 0.01 M KCl solutions. All analyses were conducted in triplicate, and then the mean values were reported **(Sahoo et al., 2021)**.

## 4.7 Batch Adsorption

In the batch adsorption experiments, 50 mg of biochar sample adsorbent was added to 50 ml of aqueous solution of phosphorus ($KH_2PO_4$) of concentration 1g/L in a 250 ml flask. To facilitate the process of adsorption, flasks were set on an incubator shaker and agitated at a controlled speed of 120 rpm. The temperature of the incubator shaker was set at 37°C. At regular intervals of 20 min, samples were collected and analysed for phosphorus concentration using the colourimetric spectroscopy technique. The readings were taken for 280 mins. The OD was recorded at 690 nm. The adsorption capacity was calculated using Eqs *(1)-(3)* **(Munar-Florez et al., 2017)**.

$$VC_0 = VC + qM \tag{1}$$

$$qM = (C_0 - C)V \tag{2}$$

$$q = (C_0 - C)/C_0 \tag{3}$$

where, M is the weight of biochar in each sample (g), V is the solution volume (l), $C_0$ is initial concentration of adsorbate (mg/l), C is final concentration of adsorbate (mg/l) and q is the adsorption capacity ($mg_{adsorbate}/g_{biochar}$)

# Chapter 5 : Results And Discussion

## 5.1 Biochar production yield

The production of biochar depicted an inversely proportional relationship with the pyrolysis temperature. In this study, the yield of the wheat straw biochar production at 400°C was found to be 34.68%. In a similar study, biochar was produced from lignocellulosic biomass and the yield was found to be 36.19% **(Zong et al., 2021).** Higher temperature is required to break down lignocellulosic components of biomass and produce biochar **(Kim et al., 2016)**. During pyrolysis, the time and temperature have a crucial effect on amount of biochar produced. Very high temperature can cause loss of useful organic compounds and lead to low quality biochar **(Muzyka et al., 2023) (He et al., 2021)**. During the pyrolysis process, some losses may occur, like the release of volatile organic molecules and the formation of ash **(de Almeida et al., 2022)**. These loses may influence the final amount of biochar produced. The yield of biochar produced has been summarized in Table 6.

***Figure 2*** *: BC Produced after Pyrolysis at 400°C*

## 5.2 Characterization of Biochar

### 5.2.1 SEM-EDS

The surface morphology of the biochar samples was identified using SEM. Under SEM, wheat straw typically has a fibrous, rough surface. It consists of a network of tangled fibres with irregular sizes and forms **(Singh et al., 2011)**. On pyrolysis, the SEM structure of biochar displays a network of irregular fibres and fissures. Biochar has a porous structure. **(Kong et al., 2021)**. After activation, biochar developed a high surface area and an effective porous structure. These large-scale pores are developed as a result of the wheat straw's hollow structure **(He et al., 2018)**. The results for the SEM are shown in Figure 3.

The elemental composition of the biochar is quantitatively reported by the EDS analysis. From the results, it can be observed that in comparison to biochar samples, biomass had a lower carbon content. On electrochemical activation, the carbon content significantly increased **(Jiang et al., 2020)**. One possible reason behind increased carbon content could be electromigration. During electrochemical treatment, the applied electric current can move charged particles within the biochar structure. Electromigration can cause carbon-rich molecules to build up in the biochar, making it have higher carbon content **(Liu et al., 2020)**. The pattern for oxygen and magnesium was also comparable. After pyrolysis, silicon composition was high; however, after electrochemical treatment, it drastically decreased. When exposed to an electric current, silicon molecules can dissolve in the electrolytic solution. This can reduce the amount of silicon in the biochar **(Sattar et al., 2022)**. The results for the EDS are summarized in Table 4.

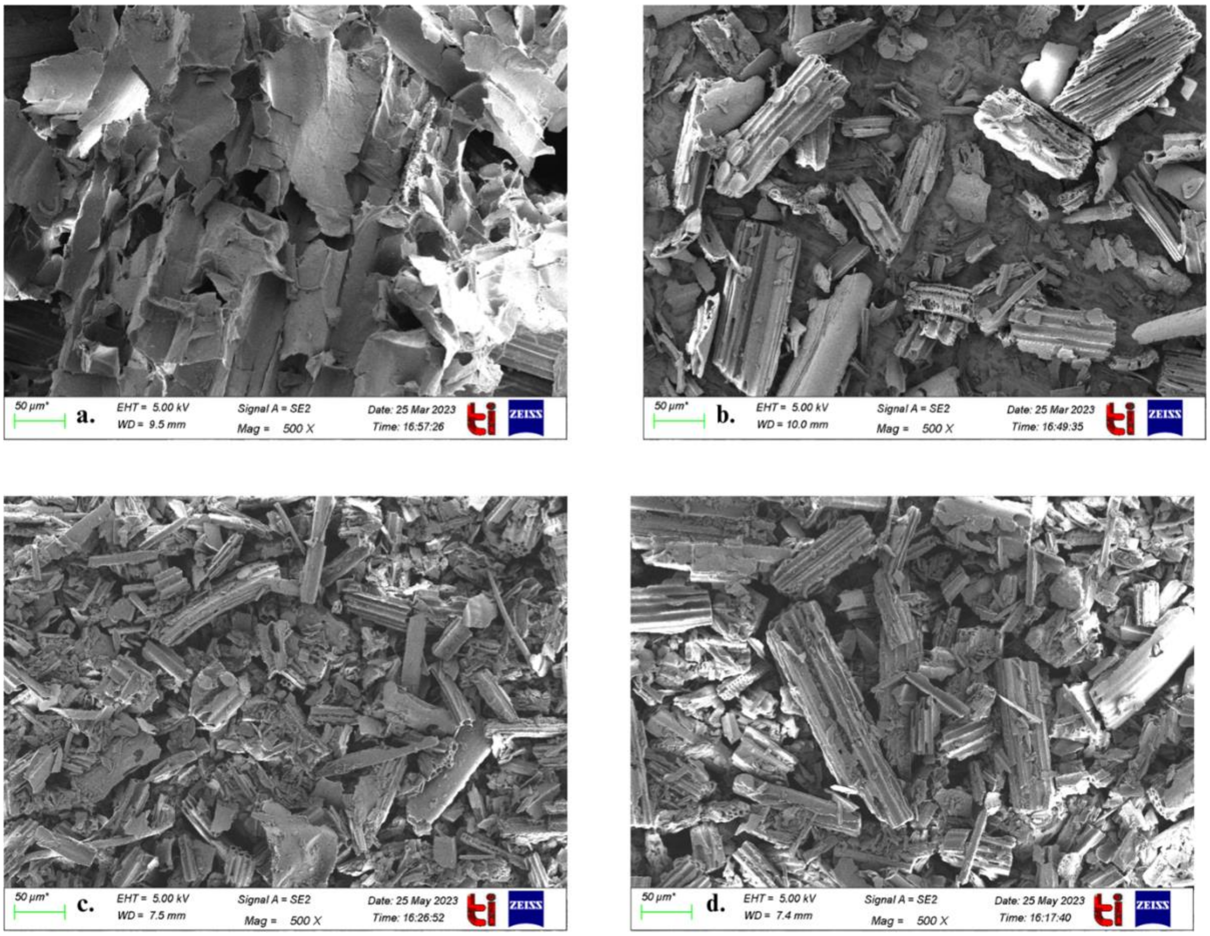

***Figure 3*** *: SEM Images of Samples at 500x.* ***a.*** *BM* ***b.*** *BC 400* ***c.*** *Pre-EAC BC 400* ***d.*** *Post-EAC BC 400*

***Table 4*** *: Elemental Composition of the Samples via EDS*

| Sample | C (%) | O (%) | Si (%) | Mg (%) |
|---|---|---|---|---|
| **BM** | 19.01 | 23.70 | 16.65 | 0.865 |
| **BC 400** | 45.12 | 16.43 | 35.35 | - |
| **Pre-EAC BC 400** | 59.06 | 32.74 | 3.13 | 5.08 |
| **Post-EAC BC 400** | 58.25 | 32.71 | 6.23 | 2.81 |

The EDS graphs depicting the elemental composition analysis of the samples are displayed in Figure 4. The EDS graphs provide elemental composition information, showcasing the presence and relative abundance of different elements in the analysed samples.

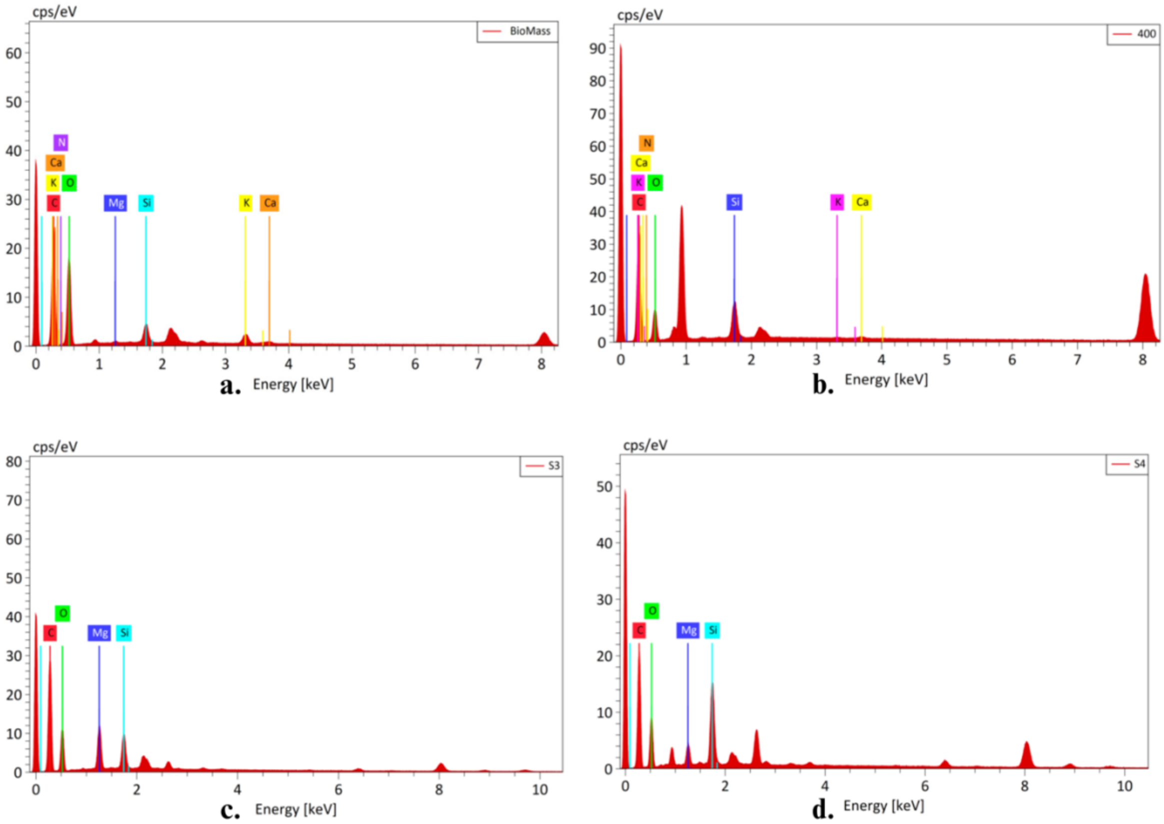

***Figure 4*** *: Graphs illustrating EDS analysis of samples.* ***a.*** *BM* ***b.*** *BC 400* ***c.*** *Pre-EAC BC 400* ***d.*** *Post-EAC BC 400*

### 5.2.2 BET Analysis

The BET analysis was performed to determine the specific surface area and pore characteristics of the biochar. After electrochemical modification, the surface area of the biochar significantly increased compared to untreated biochar, as demonstrated by the results. When biochar is treated electrochemically, the carbon structure may get activated, which can lead to the formation of new sites and functional groups **(Rawat et al., 2023)**. Carbon materials usually get activated either by oxidizing or reducing them, which adds oxygen-containing groups (such as carboxyl or hydroxyl) or may remove certain carbon atoms **(Yang et al., 2022) (Liu et al., 2023)**. These active sites and functional groups lead to formation of greater surface area of biochar as more sites for binding or chemical reactions to happen are present **(Raja et al., 2023)**. Since modified adsorbents have more surface area, they can absorb much more **(Benis et al., 2021)**. The study of pore size distribution showed that the modified adsorbents had mesopores and macropores. This means that phosphorus can be absorbed more easily. The BET study also showed that the total pore volume of the modified adsorbents had increased, which means they could hold more phosphorus. **(Dai et al., 2020)**. The enhanced porosity and surface area of electrochemically modified biochar adsorbents provided a more significant number of active sites for phosphorus binding, thereby enhancing the efficacy of phosphorus removal from effluent wastewater **(Yang et al., 2019)**. The results of the BET analysis of the wheat straw biomass and biochar samples are displayed in Table 5.

***Table 5*** *: BET Analysis of the Samples*

| Sample | Surface area ($m^2/g$) | Pore size (nm) | Pore volume (cc/g) |
|---|---|---|---|
| **BM** | 2.777 | 1.11284e + 01 | 7.727e - 03 |
| **BC 400** | 5.409 | 1.22949e + 01 | 1.663e - 02 |
| **Pre-EAC BC 400** | 12.974 | 1.02072e + 01 | 3.311e - 02 |
| **Post-EAC BC 400** | 12.956 | 1.18593e + 01 | 3.841e - 02 |

### 5.2.3 XRD Analysis

The prominent peaks at 22° and 15° in the XRD pattern of unprocessed wheat straw indicate the presence of crystalline cellulose. These peaks correspond to crystalline cellulosic planes. The presence of crystalline cellulose in unprocessed biomass indicates its well-preserved structure prior to pyrolysis **(Naz et al., 2020)**.

A broad peak was observed at approximately 24.5° in the XRD patterns of pyrolyzed biochar. This peak is commonly linked to the formation of turbostratic carbon crystallites. Turbostratic carbon refers to a disordered form of carbon characterized by haphazardly oriented layers that produce broad diffraction peaks. The presence of turbostratic carbon crystallites suggests the conversion of biomass into amorphous carbon during pyrolysis. Moreover, the XRD analysis of the pyrolyzed biochar revealed a distinct diffraction peak at $2\theta = 20.9°$, indicating the presence of the cellulose graphite microcrystalline crystal plane. This peak reflects the stratified structure of cellulose within biochar **(Mohanty et al., 2018)**.

Changes in XRD patterns were observed following the addition of MgCl2 after electrochemical modification. As the pyrolysis temperature increased, the intensity of the $2\theta = 20.9°$ diffraction peak increased, indicating an increase in the crystallinity of the cellulose microcrystals. Moreover, the peak shifted to a higher angle, indicating a decrease in the layer spacing of the cellulose microcrystals. Biochar's cellulose graphite microcrystals underwent structural modifications as a result of pyrolysis. The decrease in layer spacing and increase in superposition density led to an increase in crystallinity and the transformation of the biochar's chemical structure into a more stable carbon compound **(Quin et al., 2020)**.

The XRD analysis of biomass, biochar, and activated biochar revealed significant structural changes that occurred during the pyrolysis and activation processes. The presence of crystalline cellulose in the unprocessed biomass attested to the integrity of its structure. The formation of turbostratic carbon crystallites in biochar was indicative of the transformation of biomass into amorphous carbon. The diffraction peak of the precursor biochar at 2θ = 20.9° confirmed the presence of cellulose graphite microcrystals, and the alterations induced by the activation process led to an increase in crystallinity and a decrease in layer spacing. This suggests that the activation process altered the structural properties of the biochar, potentially enhancing its stability and adsorption properties. The graph illustrating XRD Pattern of each of the sample is displayed in Figure 5.

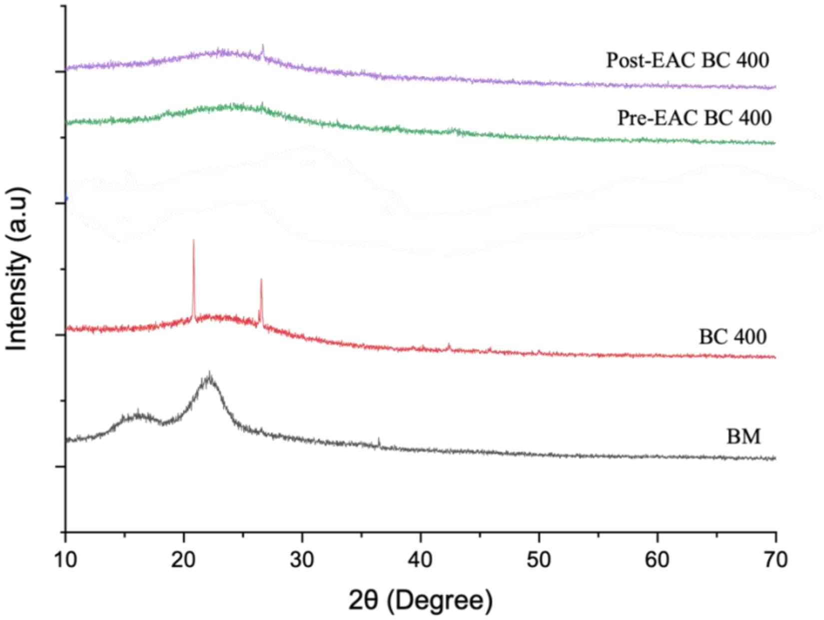

***Figure 5** : Graph illustrating XRD Patterns of Samples*

### 5.2.4 FTIR Analysis

The more notable peak at 1375 cm−1 is indicative of phenolic groups (O–H bending), while the large peak at 1581 $cm^{-1}$ is associated with amine groups (N-H bending) **(Pradhan et al., 2022)**. The peaks attributed to C–O stretch of carboxylate ions, hydroxyl bending vibration, CO stretching vibration of the alcoholic groups and aromatic compounds were observed at

1375, 1317, 1049 and 780 cm$^{-1}$ respectively. The band intensity around 1029 cm$^{-1}$ was characteristic of C-OH vibration of carboxylic acids **(Jiang et al., 2022)**. It was observed the formation of many phosphate functional groups bonded to O (O-P-O, P-O, P=O), which probably occurred due to the increase of O in the biochars after impregnation. Bands of -OH present in biochar may have contributed to the adsorption of P, since they disappear after adsorption **(Jiang et al., 2018)**. The band at 1082 and 1036 cm$^{-1}$ is assigned to C-O-C group, and the peaks of 874, 811 and 749 cm$^{-1}$ can be attributed to the aromatic C-H out-of-plane bending vibrations.

The FTIR result clearly showed that some peaks were shifted owing to the introduction of alginate and chitosan (dashed line in Figure 6), which may affect their sorption capacity **(Ahmad et al., 2014)**. Moreover, more peaks were observed after modification, meaning more oxygen-containing functional groups formed, in accordance with the results of element analysis. The graph depicting FTIR Spectra of each of the sample is displayed in Figure 6.

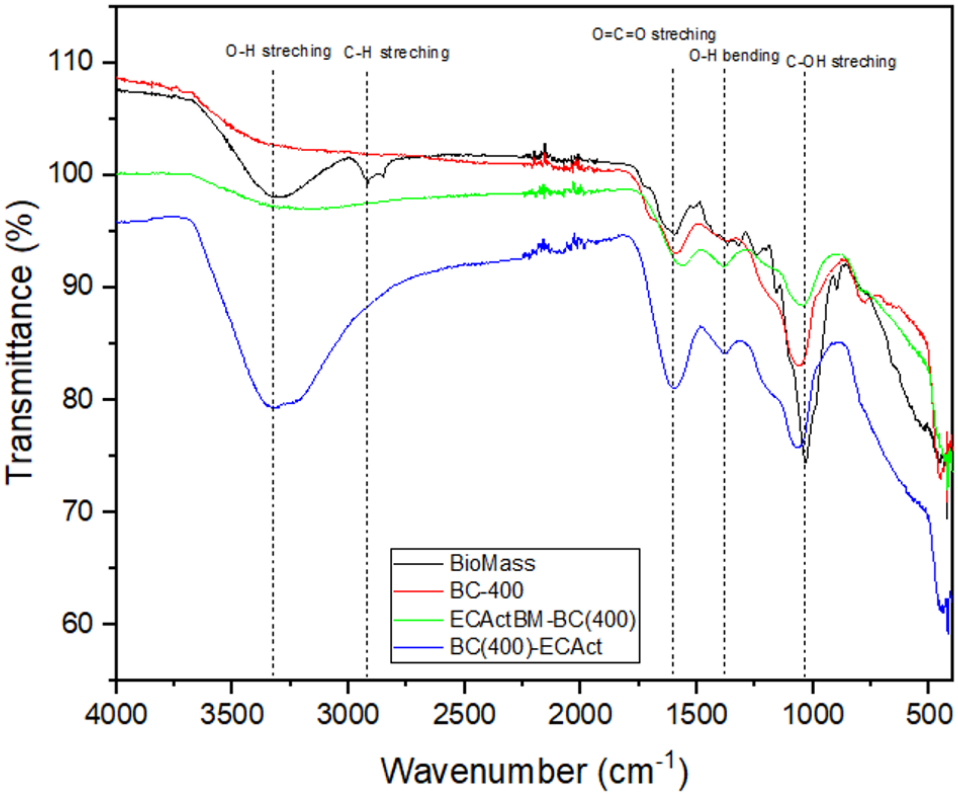

***Figure 6** : Graph illustrating FTIR Spectra of Samples*

### 5.3 Proximate Analysis

To evaluate the composition and properties of pyrolyzed biochar and electrochemically modified biochar adsorbents, proximate analysis was performed. The results revealed that the activated biochar adsorbents had a significantly different composition than the unmodified biochar adsorbents. It was observed that the moisture content of all the samples ranged from 7.498 ± 0.12% to 10.703 ± 0.17%. The activated biochar samples had slightly lower moisture content indicating enhanced stability and decreased susceptibility to microbial degradation **(Kiasson et al., 2017)**. The increase in ash content suggests the presence of inorganic compounds due to electrochemical modification. The volatile matter content decreased, indicating that the thermal stability of the modified biochar has improved **(Murtaza et al., 2022)**. The fixed carbon content increased significantly, indicating that the modified adsorbents have greater carbonaceous content and enhanced adsorption capacity. These findings indicate that the electrochemical modification of biochar significantly affects its chemical composition, enhancing its stability, carbonaceous content, and adsorption properties. This preliminary analysis provides valuable insight into the structural changes that occur in biochar during electrochemical modification, laying the groundwork for future research into the adsorption performance and phosphorus recovery capabilities of modified biochar adsorbents.

The results of the proximate analysis of the wheat straw biomass and biochar samples are displayed in Table 6.

## 5.4 Physiochemical Properties of Biochar

pH and EC of the biomass and biochar samples were measured. The pH of the biomass and biochar samples was approximately 7 before modification. After electrochemical activation, pH shifted upward. The increase in pH observed in modified biochar could be attributed to the removal of acidic components during electrochemical activation of the samples. Using biochar with a higher pH for soil amendment in areas having acidic soils could be advantageous **(Jung et al., 2016)**. EC of the samples also increased after activation. The activation process can introduce or remove certain ions, which results in alterations in the EC value. This indicates that the activation process modified the surface chemistry and ionic composition of the biochar particles, potentially improving its adsorption capacity or other desirable properties **(Panwar et al., 2020)**. The results of the physicochemical characteristics of the wheat straw biomass and biochar samples are displayed in Table 6.

***Table 4*** *: Yield, Physiochemical Properties, Proximate Analysis of the Samples*

| Sample | Yield (%) | pH | EC (µS/cm) | Moisture (%) | Ash (%) | Volatile (%) | Fixed Carbon (%) |
|---|---|---|---|---|---|---|---|
| **BM** | - | 7.33 ± 0.07 | 65.18 ± 2.33 | 10.703 ± 0.17 | 3.67 ± 1.34 | 86.84 ± 5.65 | 3.77 ± 2.85 |
| **BC 400** | 34.68 ± 1.72 | 6.73 ± 0.06 | 59.747 ± 1.55 | 9.550 ± 0.34 | 35.14 ± 6.13 | 34.53 ± 4.75 | 27.31 ± 12.52 |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| **Pre-EAC BC 400** | 34.27 ± 0.27 | 10.5 ± 0.06 | 254.915 ± 3.27 | 8.524 ± 0.24 | 25.87 ± 4.72 | 40.95 ± 5.60 | 17.48 ± 1.70 |
| **Post-EAC BC 400** | - | 8.9 ± 0.05 | 211.46 ± 2.67 | 7.498 ± 0.12 | 32.97 ± 5.80 | 39.33 ± 2.18 | 20.43 ± 12.49 |

### 5.5 Preliminary Adsorption

***Table** 7 : Adsorption Capacity of the Biochar Samples*

| **Sample** | **Adsorption Capacity (mg/g)** |
|---|---|
| BC 400 | 0.098 |
| Pre-EAC BC 400 | 0.43 |
| Post-EAC BC 400 | 0.2 |

The preliminary adsorption tests demonstrated that the biochar samples had good adsorption capacity. The adsorption efficacy of biochar adsorbents was determined by conducting batch adsorption experiments with a solution of known phosphorus concentration (1 g/L). The adsorption capacity for the biochar samples was calculated using Eqs (1)-(3). The results for the adsorption capacity of different samples are shown in Table 7. The adsorption capacity of pre-treated biochar was calculated to be 0.43 mg/g and for post-treated biochar, the adsorption capacity was found to be 0.2 mg/g. In a similar study, the adsorption capacity of lanthanum-modified biochar was found to be 0.34 mg/g **(Zhao et al., 2021)**.The results indicate that the adsorption capacity increased significantly after the electrochemical modification of biochar.

The electrochemically modified biochar exhibited higher phosphorus removal efficiency due to the surface modifications achieved through the electrochemical treatment **(Munar-Florez et al., 2017)**. The electrochemical treatment can alter the surface charge, pore volume and introduce new functional groups to the surface of biochar, increasing its affinity for phosphorus ions **(Tomin et al., 2021)**. However, the adsorption capacity of Post-treated biochar was still less than that of Pre-treated biochar.

Overall, the results of the preliminary adsorption study on electrochemically modified biochar adsorbents were promising. The biochar adsorbents modified electrochemically exhibited high absorption capacities than the untreated ones. These findings provide an adequate foundation for further optimization and scaling up the adsorption process for wastewater treatment and phosphorus recovery systems. The graph for the preliminary adsorption experiments is displayed in Figure 7.

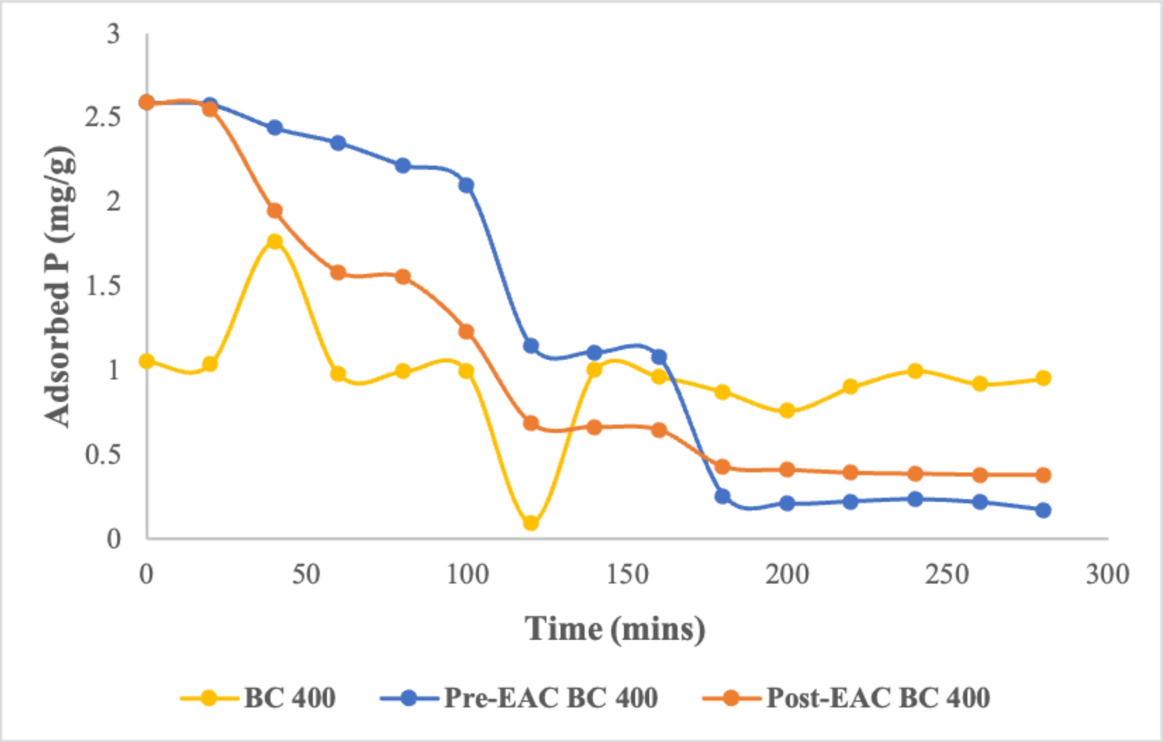

***Figure 7** : Graph for preliminary adsorption experiments*

# Chapter 6 : Conclusions And Future Recommendations

## 6.1 Conclusion

The purpose of the thesis entitled "Development of Electrochemically Modified Biochar Adsorbents for Enhanced Recovery of Phosphorus" was to examine the potential of electrochemically modified biochar adsorbents for enhanced phosphorus recovery in wastewater treatment. The outcomes of various analyses and experiments provide basis for the efficacy of the electrochemical modification method.

After electrochemical modification, the characterization of biochar samples using SEM-EDS revealed significant changes in surface structure. There were irregular fibres which led to increased surface area and greater pore size. The EDS report showed that the pre-and post-electrochemically modified biochar adsorbents contained more carbon and less oxygen, silicon, and magnesium. This change in elemental composition indicates that the biochar surface has been successfully modified, which may improve its efficiency for adsorption.

The BET analysis provided insight into the adsorbent's surface area, pore size, and pore volume. Compared to untreated biochar, the electrochemically modified biochar adsorbents exhibited significantly increased surface area and pore volume. These structural modifications suggest enhanced phosphorus removal accessibility and absorption capacity in sewage effluent.

In addition, the biochar samples' physicochemical analysis revealed variations in yield, pH, electrical conductivity (EC), moisture content, ash content, volatile matter, and fixed carbon. The modified biochar samples exhibited altered physicochemical properties, indicating that electrochemical modification affected the composition and behaviour of the biochar.

The results of this study suggest that the electrochemical modification of biochar adsorbents is promising for enhancing phosphorus recovery in effluent wastewater treatment. The structural modifications observed via elemental composition, BET analysis, and physicochemical characterization contribute to a better comprehension of the potential of the modified adsorbents. To investigate the maximum potential and practical applications of these electrochemically modified biochar adsorbents in sustainable wastewater treatment processes, additional research and optimization are required.

This thesis contributes to expanding knowledge regarding wastewater remediation and resource recovery. The results provide valuable insights for the further optimization and practical application of electrochemically modified biochar adsorbents. In order to promote environmental welfare and resource conservation in wastewater management, it is anticipated that the findings of this study will guide future efforts to develop efficient and sustainable technologies for phosphorus removal and recovery.

## 6.2 Future Directions

Based on the findings and conclusions of this thesis regarding the production of electrochemically modified biochar adsorbents for increased phosphorus recovery from sewage effluent, numerous future recommendations may be made to advance this field of study. Initially, the electrochemical modification procedure needs to be optimized. If applied voltage, electrolyte composition, and modification time are well-known, biochar adsorbents may have a greater surface area, porosity, and adsorption capacity.

Additionally, the durability and reusability of modified adsorbents must be evaluated. Experiments conducted over an extended period of time and involving multiple adsorption-desorption kinetics can provide knowledge on the durability and efficacy of electrochemically modified biochar adsorbents.

Biochar adsorbents that have been modified electrochemically also need to be evaluated in real-world applications. Trials on an industrial scale or in the field can provide practical information on wastewater treatment plants' performance, viability, and cost-effectiveness. Evaluating adsorbent effectiveness in the presence of additional pollutants prevalent in sewage effluent should be part of all comprehensive tests.

Electrochemical modification in collaboration with membrane filtration or biological processes can enhance phosphorus removal and recovery. Synergistic methods can boost treatment efficacy while decreasing their environmental impact.

Overall, future research should optimize the electrochemical modification procedure, evaluate adsorbents' long-term durability and reusability, conduct field-scale experiments, and investigate synergistic treatment strategies. These proposals will promote the development and use of biochar adsorbents that have been electrochemically modified, thereby aiding in the maintenance of wastewater treatment and the efficient recovery of phosphorus from sewage effluent.

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