

**EVALUATION OF BIOCHAR DERIVED FROM MANGO AND  
BANANA PEELS FOR THE REMOVAL OF SELECTED ENDOCRINE-  
DISRUPTING COMPOUNDS FROM WASTEWATER**

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**A Dissertation Submitted in Fulfilment of the Requirements for the Award of the  
Degree of Doctor of Philosophy in Environmental Science and Engineering of the  
Nelson Mandela African Institution of Science and Technology**

**Arusha, Tanzania**

**January, 2026**

## ABSTRACT

Wastewater pollution by endocrine-disrupting compounds poses a significant environmental and public health risk due to their ability to interfere with hormonal systems in living organisms even at trace levels. Conventional removal methods of endocrine-disrupting compounds from wastewater often fail to effectively eliminate these pollutants, encouraging the search for sustainable and eco-friendly alternatives. This study evaluated the potential of biochar derived from banana and mango peels, both in mono- and hybrid forms, to remove progesterone and bisphenol A from synthetic and real wastewater. It also examined the impact of the coexistence of progesterone and bisphenol A on hybrid biochar removal efficiency. Biochar was produced through pyrolysis at temperatures of 300, 550, 700 and 800°C for a duration of two hours. Batch adsorption experiments in varying conditions revealed that biochar produced at 700°C yielded the most effective adsorbent. Optimal removal was achieved with a 0.5 g dose of biochar, pH 8, an adsorbate concentration of 12.50 mg L<sup>-1</sup>, a temperature of 25°C and agitation of 140 rpm. Under these conditions, the hybrid biochar demonstrated the highest removal efficiency of 97.80% of progesterone and 91.80% of bisphenol A, compared to banana peels biochar (92.80% and 87.90%) and mango peels biochar (80.50% and 90.40%), respectively. Adsorption kinetics followed a pseudo-second-order model, indicating chemisorption as the rate-limiting step. Isotherm models showed that bisphenol A followed the Freundlich model ( $R^2 = 0.99$ ), indicating multilayer adsorption on heterogeneous surfaces, while progesterone fit the Langmuir model ( $R^2 = 0.98$ ), indicating monolayer adsorption. The hybrid biochar had a Brunauer-Emmett-Teller surface area of 652 m<sup>2</sup> g<sup>-1</sup>, outperforming banana (481 m<sup>2</sup> g<sup>-1</sup>) and mango (562 m<sup>2</sup> g<sup>-1</sup>) biochar. Fourier Transform Infrared Spectroscopy confirmed the presence of functional groups and Scanning Electron Microscopy revealed a highly porous structure that facilitates the entrapment of pollutants. Thermodynamic analysis showed that adsorption was spontaneous, endothermic and involved both strong physisorption and weak chemisorption. The hybrid biochar maintained high performance over four reuse cycles, although it shows slightly less efficiency in real wastewater due to the presence of competing solutes. Therefore, hybrid agro-waste biochar is a promising, reusable material for removing endocrine-disrupting compounds. Further research is recommended to optimize its properties and evaluate its performance against a broader range of organic pollutants in full-scale field applications.

## DECLARATION

I, Hildegard R. Kasambala, do hereby declare that this thesis entitled “*Evaluation of Mango and Banana Peels-Derived Biochar for Removing Selected Endocrine-Disrupting Compounds from Wastewater*” is my own original work and that it has neither been submitted nor being concurrently submitted for degree award in any other institution.

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## CERTIFICATION

The undersigned certify that they have read and hereby recommend for acceptance by the Senate of the Nelson Mandela African Institution of Science and Technology the thesis entitled “*Evaluation of Mango and Banana Peels-Derived Biochar for Removing Selected Endocrine-Disrupting Compounds from Wastewater*” in Fulfilment of the Requirements for the Award of the Degree of Doctor of Philosophy in Environmental Science and Engineering at the Nelson Mandela African Institution of Science and Technology.

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## ACKNOWLEDGEMENTS

First, I would like to express my sincere gratitude to God Almighty for granting me strength, good health and perseverance throughout my study. I am deeply thankful to my supervisors Prof. Mwemezi J. Rwiza, Dr. Nelson Mpumi and Prof. Karoli N. Njau whose guidance, expertise and unwavering support have been instrumental in shaping the direction and depth of this research. Their critical insights and on-time and constructive feedback challenged me to grow as a scholar and remain committed to pursuing clarity, precision and rigor.

I extend my thanks to the Nelson Mandela African Institution of Science and Technology (NM-AIST), specifically the School of Materials, Water and Environmental Sciences (MEWES), whose resources, facilities and academic environment provided a solid foundation for this work. Also, my heartfelt appreciation goes to the former Dean of the School of MEWES, Prof. Kelvin Mtei. If it were not for his good leadership as Dean during my studies by then, I would have not attained this stage by now. I am particularly thankful to the technical staff and laboratory team, under the leadership of Dr. Mwema Felix Mwema as laboratory manager and co-researcher, whose assistance contributed to the success of this thesis.

I would like to extend my heartfelt appreciation to my colleagues and fellow researchers, Ms. Vaileth Hance and Mr. Optatus Mwalongo, for their friendship and intellectual exchange. The many thought-provoking discussions and shared explorations have enriched this thesis and made the research process truly collaborative.

I am profoundly grateful to my family, especially my lovely husband, Mr. Beda Pius Mtwewe, for his love, encouragement, and understanding throughout this endeavour. His patience during long hours of research and writing, and their unwavering belief in my capabilities, have been a source of constant strength. Furthermore, I would like to thank my children, Careen Mtwewe, Maureen Mtwewe, Beatrice Mtwewe, Diana Mtwewe and Brayden Mtwewe, and my lovely father, Mr. Raphael Kasambala, and my lovely mother, Ms. Veronica Kakwaya, for their persistence and deep prayers throughout the period of my study.

I am also grateful to the Higher Education for Economic Transformation (HEET) project under Mbeya University of Science and Technology (MUST) for providing full financial support which made me comfortable during the whole period of the study time. Finally, I acknowledge the inspiration drawn from the environmental challenges facing our communities as directed by the Environmental Research Group (ERG) at NM-AIST.

## **DEDICATION**

This thesis is dedicated to my beloved parents, Mr. Raphael Kasambala, Ms. Veronica Kakwaya, and my parents-in-law, Mr. Johan Pius Mtweve and Ms. Ernesta Sanga, whose unwavering love, support and prayers have been the foundation of my journey. I also dedicate this work to my husband Mr. Beda Pius Mtweve and our children, Careen Mtweve, Maureen Mtweve, Beatrice Mtweve, Diana Mtweve and Brayden Mtweve, who stood by me with patience and understanding throughout this academic endeavour. Lastly to my sister Ms. Sebastiana Kasambala, thank you for assisting me in taking care of my children throughout my study journey.

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## LIST OF ABBREVIATIONS AND SYMBOLS

AC	Activated Carbon
$A_e$	Adsorption Efficiency
AOM	Algae Organic Matter
AOP	Advanced Oxidation Processes
ATRC	African Technical Research Centre
ATRC	African Technical Research Centre
BET	Brunauer, Emmett and Teller
BOD	Biological Oxygen Demand
BP	Banana Peels
BPA	Bisphenol-A
BPB	Banana Peels Biochar
C	Carbon
C=O	Carbonyl group
$Ca^{2+}$	Calcium
CAO	Chemical Advanced Oxidation
$Cd^{2+}$	Cadmium
Ce	Concentration at Equilibrium
C <sub>0</sub>	Initial Concentration
COD	Chemical Oxygen Demand
COOH	Carboxyl group
$Cr^{3+}$	Chromium ion
C <sub>t</sub>	Concentration at a Time
DDT	Dichlorodiphenyltrichloroethane
E <sub>2</sub>	Estrone
EC	Electric Conductivity
EDA	Electron Donor Acceptor
EDCs	Endocrine Disrupting Compounds
EDX	Energy Dispersive X-Ray Spectroscopy
EE <sub>1</sub>	Ethinyl Estradiol
EIA	Environmental Impact Assessment
FAO	Food and Agriculture Organization
FTIR	Fourier Transform Infrared Spectroscopy

GACS	Granular Activated Carbon
GC-MS	Gas Chromatography Mass Spectrometer
H	Hydrogen
H <sub>2</sub> O <sub>2</sub>	Hydrogen Peroxide
HCl	Hydrochloric Acid
HNO <sub>3</sub>	Nitric Acid
HPLC	High Performance Liquid Chromatography
K <sup>+</sup>	Potassium ion
MEWES	School of Material, Energy, Water and Environmental Sciences
MnO <sub>4</sub> <sup>-</sup>	Permanganate ion
MP	Mango Peels
MPB	Mango Peels Biochar
N	Nitrogen
NaCl	Sodium Chloride
NaOH	Sodium Hydroxide
NM-AIST	Nelson Mandela African Institution of Science and Technology
NO <sub>2</sub> <sup>-</sup>	Nitrite
NO <sub>3</sub> <sup>-</sup>	Nitrate
OH	Hydroxyl Group
PAH	Polycyclic Aromatic Hydrocarbons
Pb <sup>2+</sup>	Lead
PCBS	Polychlorinated Biphenyl
PFAS	Per- and Polyfluoroalkyl Substances
PO <sub>4</sub> <sup>3-</sup>	Phosphate
PZC	Point of Zero Charge
q <sub>e</sub>	Adsorption Capacity at Equilibrium
q <sub>t</sub>	Adsorption capacity at Time
R-CO-R	Ketone group
Rpm	Rotation per minute
SEM	Scanning Electron Microscopy
SO <sub>4</sub> <sup>•-</sup>	Per sulphate Radical
TARI	Tanzania Agricultural Research Institution
UV	Ultraviolet
WHO	World Health Organization

WSP	Waste Stabilization Ponds
XRF	X-Ray Fluorescence
$\Delta G^\circ$	Standard Gibbs free energy
$\Delta H^\circ$	Standard Enthalpy change
$\Delta S^\circ$	Standard Entropy Change

## CHAPTER ONE

### INTRODUCTION

#### 1.1 Background of the Problem

Endocrine-disrupting compounds (EDCs) have emerged as a significant environmental concern, primarily due to their ability to disrupt the endocrine systems in humans and animals. Globally, it is estimated that around 1000 chemicals possess endocrine-disrupting characteristics (Yilmaz *et al.*, 2020; Puri *et al.*, 2023). This diverse group of compounds encompasses various industrial chemicals, pharmaceuticals, personal care items, pesticides, fungicides, plasticizers, nonylphenols and metals (Gore *et al.*, 2015; Yilmaz *et al.*, 2020). Even at low concentrations, EDCs can cause adverse health effects, including reproductive defects, obesity, cancer and metabolic disorders (Gore *et al.*, 2015; Yilmaz *et al.*, 2020; Werkneh *et al.*, 2022).

Endocrine disrupting compounds originate from both point sources, such as pharmaceutical products and non-point sources, including agricultural runoff, storm water and effluents from treatment plants (Ismanto *et al.*, 2022; Ravikumar *et al.*, 2022). Its extensive environmental applications result in its presence across various environmental matrices, including surface water, groundwater, wastewater, soil, plants and air (Yilmaz *et al.*, 2020), underlining its potential health impacts on ecosystems. Among the most significant endocrine disruptors are bisphenol A (BPA) and progesterone, both of which have far-reaching consequences for human health and environmental stability (Puri *et al.*, 2023; Gore *et al.*, 2024).

Progesterone is widely used in the pharmaceutical industry, particularly in contraceptives and plays a role in dairy processing as a hormonal cattle induction agent (De Graaff & Grimard, 2018; Dias *et al.*, 2023). Additionally, agricultural practices involving livestock manure contribute to progesterone contamination in water systems, particularly when manure is applied to the fields (Kasambala *et al.*, 2019). Bisphenol A, on the other hand, is commonly found in food and beverage packaging (Flint *et al.*, 2012; Cimmino *et al.*, 2020). It can leach from polycarbonate plastics and epoxy resins, particularly in canned foods, water bottles and baby bottles (Vilarinho *et al.*, 2019; Khalili-Sadrabad *et al.*, 2023).

The presence of BPA and progesterone in the environment raises substantial health concerns, including reduced sperm quality, impaired sexual function, menstrual irregularities, cancer and cardiovascular diseases (Diamanti-Kandarakis *et al.*, 2009; Gore *et al.*, 2015; Vilarinho *et al.*,

2019; Yang *et al.*, 2019). This is due to their ability to disrupt hormonal functions in both humans and wildlife. Despite the ecological implications of progesterone and BPA, their industrial usage remains important. Various techniques, including reverse osmosis, chlorination, photocatalytic treatment, ultrafiltration, adsorption, biodegradation and advanced oxidation, applied to remove organic and inorganic contaminants from polluted water (Katibi *et al.*, 2021; Ghazal *et al.*, 2022).

However, reverse osmosis, chlorination, photocatalytic treatment and advanced oxidation are chemical strategies which are not environmentally friendly, leading to secondary pollution (Zhang *et al.*, 2019; Luo *et al.*, 2022). Furthermore, a lack of expertise makes these methods difficult to apply. Likewise, conventional wastewater treatment facilities, primarily located in developing countries, often face difficulties to effectively remove these emerging micropollutants. Despite the significant roles played by conventional treatments in reducing the concentration of EDCs in aquatic environments, they still appear in large amounts in wastewater effluents (Archer *et al.*, 2017; Liang *et al.*, 2021). The lack of solid regulations, especially in Africa (Zamri *et al.*, 2021), allows pollutant-rich effluents to be released into the receiving environment, becoming a source of secondary pollution to the aquatic environment and posing a problem for ecosystems.

Furthermore, the inadequacy of advanced tertiary treatment technologies poses significant challenges, especially for developing countries (Yilmaz *et al.*, 2020). Since EDC removal depends on the physical and chemical properties of specific compounds as well as the specific environment, the lack of advanced tertiary treatment technologies poses a significant problem, especially for developing countries (Yilmaz *et al.*, 2020). Therefore, given these limitations, there is an urgent need for a more efficient, environmentally friendly and sustainable solution utilizing agro-waste biochar for the removal of EDCs.

Agro-waste is an abundant and renewable resource that has promising properties for biochar production (Dong *et al.*, 2023). Biochar, a carbon-rich material derived from the pyrolysis of organic matter, has brought considerable interest due to its potential in environmental remediation. A significant amount of agricultural produce, particularly fruits and vegetables, is wasted post-harvest (Bhavani *et al.*, 2023; Daffalla *et al.*, 2024). For instance, worldwide mango production is approximately 50.60 million tons per year, with mango peels contributing 7-14% (Jahurul *et al.*, 2015). On the other hand, banana production is estimated at 250 million tons per year, with its peels contributing about 20% of this amount (Daffalla *et al.*, 2024). Given this substantial biomass availability, co-pyrolysis of banana and mango peels as biosorbents

owing to their richness in lignin, cellulose and hemicellulose (Orozco *et al.*, 2014), adds value to agricultural by-products and contributes to effective waste management.

Furthermore, co-pyrolysed biochar, produced from various agro-waste sources, can exhibit superior adsorption properties due to the synergistic effects of different feedstocks. For instance, blending feedstocks can diversify the chemical and physical properties of biochar, thereby enhancing its efficacy (Wang *et al.*, 2020; Monica *et al.*, 2023). Most existing studies have focused on conventional biochar produced from single feedstocks (Kapoor *et al.*, 2022; Vieira *et al.*, 2022; Ripanda *et al.*, 2023), often neglecting the unique properties and performance of hybrid biochar derived from diverse agricultural wastes, especially banana and mango peels.

Previous studies have shown that banana and mango peels are excellent low-cost precursors for biochar because their lignocellulose structure produces porous carbon materials with high surface area during pyrolysis. They contain functional groups ( $-OH$ ,  $-COOH$  and phenolic) and minerals (K, Ca and Mg) that enhance surface reactivity and adsorption capacity for EDCs (Munagapati *et al.*, 2020). These properties justify their selection as sustainable biochar materials for effective removal of EDCs from wastewater. Single feedstocks typically produce homogeneous pores and are mainly used to treat wastewater from similar pollutants (Weber & Quicker, 2018). This method can benefit industries and regions that consistently generate specific waste materials.

Several studies have demonstrated that mixed feedstocks can enhance biochar quality and increase the abundance of oxygen-containing functional groups (Suliman *et al.*, 2016; Hoslett *et al.*, 2020). Co-pyrolysis of different feedstocks for biochar production increases the aromatic groups, resulting in high  $\pi$ - $\pi$  interactions with aromatic groups of pollutants (Ahmad *et al.*, 2020). Mixed feedstocks also provide biochar with a heterogeneous surface morphology, featuring pores of varying sizes, which effectively removes diverse micropollutants compared to a single feedstock (Tong *et al.*, 2019; El Barkaoui *et al.*, 2023).

Therefore, this study hypothesized that agro-waste-derived hybrid biochar exhibits significantly higher efficiency in EDCs removal from wastewater compared to conventional single-feedstock biochars. This increased efficiency is anticipated to result from enhanced surface characteristics and functional group availability due to the hybridization process, allowing for better interaction with EDCs. Competitive adsorption among co-existing EDCs may reduce overall efficiency, yet hybrid biochar is hypothesized to maintain superior

selectivity and stability in complex wastewater matrices. Finally, the optimized biochar is expected to be regenerable over multiple cycles with minimal loss of performance.

Therefore, this study aims to evaluate an agro-waste-derived hybrid biochar made from banana peels (BP) and mango peels (MP) as a novel approach for efficiently removing EDCs from contaminated water sources. By combining banana and mango peels as feedstocks, the study aims to enhance the adsorption properties of biochar without chemical modifications. The novelization can optimize surface area, functional groups and porosity, thereby improving adsorption efficiencies for a broader range of EDCs compared to single feedstock biochars. The synergistic effects of combining different agro-wastes on biochar performance could offer a sustainable solution to water pollution. Consequently, these findings could pave the way for developing cost-effective and environmentally friendly solutions to mitigate EDCs in water and aquatic environments, while also promoting effective environmental management.

## **1.2 Statement of the Problem**

Wastewater contamination by EDCs presents a significant environmental and public health challenge due to their ability to interfere with hormonal systems even at trace concentrations (Gore *et al.*, 2014; Kumar *et al.*, 2020; Cheng *et al.*, 2021). These compounds are highly persistent, resistant to natural degradation processes and toxic to both aquatic organisms and humans, where long-term exposure has been associated with reproductive disorders, developmental abnormalities and endocrine-related diseases (Gore *et al.*, 2022). Endocrine-disrupting compounds are also widely detected in municipal and industrial wastewater effluents worldwide due to continuous discharge from pharmaceuticals, personal care products, plastics and agricultural activities.

Conventional wastewater treatment methods, particularly biological treatment systems, are often limited in their ability to completely remove EDCs because many of these compounds are recalcitrant, poorly biodegradable due to complex structures and may inhibit microbial activity (Gao *et al.*, 2020; Elangovan *et al.*, 2022; Chaturvedi *et al.*, 2023). As a result, EDCs can pass through treatment plants and enter natural water bodies, posing long-term ecological and health risks.

Subsequently, there is growing interest in applying sustainable and environmentally friendly physical treatment techniques based on biochar derived from agricultural wastes for wastewater remediation. Most existing studies have focused on biochars produced through conventional pyrolysis of single biomass sources such as wood, coconut shells, banana peels or mango peels

in isolation, with limited investigation into advanced physical modification techniques such as co-pyrolysis or thermal activation to enhance pore structure, surface area and adsorption efficiency (Wang *et al.*, 2021; Kapoor *et al.*, 2022; Vieira *et al.*, 2022; Ripanda *et al.*, 2023).

Very limited research has explored hybrid biochars made by co-pyrolysing mixed fruit peels. Since banana and mango peels have different biochemical compositions, combining them could create a biochar with improved surface properties and a combined adsorption behaviour (Munagapati *et al.*, 2020). These properties justify their selection as sustainable biochar materials for effective removal of EDCs from wastewater. However, this potential has not been sufficiently investigated, especially for unmodified hybrid biochar, which is environmentally friendly and sustainable materials.

As a result, there is a clear knowledge gap regarding how mixing banana and mango peels affects biochar properties and whether such a hybrid material can outperform single-feedstock biochars in removing persistent EDCs from wastewater. This research aims to: (a) Evaluate the adsorption efficiency of mango, banana and hybrid peel biochars for removing progesterone and BPA from wastewater, (b) optimize operational parameters, and (c) investigate the adsorption mechanisms involved, thereby contributing to the development of eco-friendly wastewater treatment solutions.

### **1.3 Rationale of the Study**

Increasing the presence of EDCs such as progesterone and BPA in wastewater poses significant environmental and health challenges. These compounds interfere with hormonal systems in humans and aquatic organisms, resulting in reproductive issues, developmental abnormalities and potential long-term ecological consequences. Conventional wastewater treatment plants are largely inefficient in completely removing these micropollutants because EDCs are often persistent, biologically active and resistant to biodegradation. Their continuous release into water bodies through domestic, industrial and agricultural effluents has resulted in widespread contamination of surface and groundwater resources, particularly in developing countries where treatment infrastructure is limited.

At the same time, the increasing generation of agricultural and fruit-processing wastes, particularly banana and mango peels, presents both an environmental challenge and an untapped resource. These biomasses are abundantly available in tropical regions, including many parts of Africa yet they are often discarded without valorisation, contributing to solid waste management problems. Converting these wastes into biochar not only offers a

sustainable waste management strategy but also produces environmentally friendly adsorbent materials for water treatment applications. While several studies have explored biochar for the adsorption of organic pollutants, limited research has specifically focused on the use of mango and banana peel-derived biochar for the removal of EDCs.

Furthermore, comparative evaluation of single-feedstock biochars and hybrid or combined biochars from mixed fruit peel sources remains insufficiently studied. There is also a lack of detailed understanding of how the physicochemical properties of these biochars such as surface area, pore structure, functional groups and surface chemistry influence their adsorption performance toward specific EDCs.

Therefore, this study is rationalized by the need to develop low-cost, locally available and sustainable adsorption materials for mitigating EDC pollution in wastewater, while simultaneously addressing fruit waste disposal challenges. By evaluating the performance of banana and mango peel-derived biochar in the removal of selected EDCs, this research contributes to both environmental remediation and circular economy initiatives. Moreover, its findings will provide scientific insights into adsorption mechanisms and offer practical solutions for improving water quality management in resource-limited settings.

## **1.4 Research Objectives**

### **1.4.1 General Objectives**

The general objective of this study was to examine the effectiveness of banana and mango peels biochar in removing progesterone and bisphenol A from wastewater.

### **1.4.2 Specific Objectives**

- (i) To evaluate the effectiveness of banana and mango peels biochar in isolated components for the removal of progesterone and bisphenol A.
- (ii) To investigate the efficiency of mango and banana peels in a hybrid form in removing progesterone and bisphenol A from wastewater.
- (iii) To evaluate the competitive adsorption behaviour of co-existing progesterone and bisphenol A in synthetic and real wastewater matrices using the optimized hybrid biochar.

- (iv) To assess the effect of co-existing endocrine disruptors and wastewater matrix on the adsorption performance of hybrid biochar in synthetic and municipal wastewater
- (v) To assess the regeneration and reusability of the developed hybrid biochar materials for sustainable wastewater treatment applications.

### **1.5 Research Questions**

- (i) How effective are banana and mango peels biochar in removing progesterone and bisphenol A from wastewater when applied in singular form?
- (ii) What is the efficiency of banana and mango peels biochar in removing selected pollutants from wastewater when applied in a hybrid form?
- (iii) Can the hybrid biochar remove EDCs in the complex matrix?
- (iv) How do co-existing endocrine disruptors and wastewater matrix components affect the adsorption performance of hybrid biochar in synthetic and municipal wastewater?
- (v) How many regeneration cycles can the biochar undergo before significant loss of performance?

### **1.6 Significance of the Study**

The significance of this study lies in its investigation of the utilization of banana and mango peels, agricultural waste products, as low-cost and sustainable materials for biochar production. The research corresponds with sustainability and waste management principles by transforming waste into valuable water treatment material using these abundant waste resources.

Furthermore, the investigation addresses the worldwide requirement for environmentally beneficial, cost-effective, and accessible technologies that effectively eliminate hazardous pollutants from wastewater. This technique will be advantageous for water authorities to consider as an alternative and appropriate advanced wastewater treatment method when facing the economic challenges of developing countries.

The study also assists environmental agencies in fostering public engagement in environmental protection. The research is also expected to benefit the individual at the family level, enabling them to utilize biochar from banana and mango peels to purify water since the method is cost-

effective and the reusable materials are readily available and locally sourced. Following the study, a policy brief on environmentally friendly wastewater treatment methods will also be developed. Also, the study will assist the municipal local authorities implement measures to enhance reuse applications and increase waste recycling. The study also advances a more comprehensive understanding of biochar's potential in environmental applications, facilitating future research on agricultural reuse to mitigate water pollution.

## **1.7 Delineation of the Study**

The study focuses on removing two specific EDCs, progesterone and BPA, from wastewater using biochar derived from banana and mango peels. The study is confined to using biochar produced solely and mixed from these two agricultural wastes, prepared through a defined pyrolysis process. Neither other materials nor modifications were applied in biochar synthesis. The scope of the study is further narrowed to targeting progesterone and BPA as the endocrine disruptors of interest, leaving the analysis of other contaminants beyond the study's boundaries.

The methodology included the pyrolysis process for producing biochar, detailing temperature settings, duration and other pre-treatment procedures. Moreover, pollutant removal analysed in this research is adsorption, focusing on understanding the materials' adsorption kinetics, isotherms, reusability and mechanisms involved in the interaction between the biochar and the EDCs.

The study was conducted at a laboratory scale, under controlled conditions, simulating wastewater environments and comparing with municipal wastewater without extending to full-scale field applications or other types of sewage. The physical and chemical characteristics, such as surface area, porosity, functional groups and chemical composition, were evaluated to correlate biochar properties with adsorption efficiency.

Additionally, the study compared the adsorption efficiency of biochar derived from banana peels, mango peels and a mixture of both to determine which material performs better in removing progesterone and BPA. The research remains focused on these parameters and broader studies involving other pollutants, different biochar materials or large-scale applications are outside the scope of this investigation.

## CHAPTER TWO

### LITERATURE REVIEW

#### 2.1 Endocrine Disrupting Compounds

In the mid-20<sup>th</sup> century, many substances known as EDCs, especially synthetic chemicals, were discovered to interfere with the endocrine system of living organisms (Di Pietro *et al.*, 2023; Gore *et al.*, 2024). Endocrine-disrupting compounds are chemicals that interfere with the normal functioning of the endocrine system in living organisms (Choi *et al.*, 2021; Gore *et al.*, 2024). Usually, the endocrine system produces and regulates hormones essential for the proper development, growth and function of various bodily systems (Kim & Ji, 2022). Any interference with the endocrine system may lead to abnormalities in the living organism's body.

Due to the increased industrialisation, different EDCs have been discovered over time (Gore *et al.*, 2024). Around the 1960s, Dichlorodiphenyltrichloroethane (DDT) insecticide was the first EDCs to accumulate in the body tissue of birds, reducing egg viability and depopulating bird species (Matsushima, 2018). Likewise, per- and polyfluoroalkyl substances (PFAS) are among the EDCs that have become a significant concern in manufacturing industries since the mid-20<sup>th</sup> century (Hepburn *et al.*, 2019). They are currently used in various products, including firefighting foams, non-stick cookware, cosmetics, paper goods, food packaging and water repellents (Cousins *et al.*, 2019; Glüge *et al.*, 2020).

The presence of PFAS in the aquatic environment can lead to health problems, including hormonal disruption, cancer and reduced sperm count and quality in aquatic organisms (Gonsioroski *et al.*, 2020; Petersen *et al.*, 2020; Thacharodi *et al.*, 2023). The EDCs are widely spread in most environments. For instance, phthalates are found in some personal care products (Koniecki *et al.*, 2011) polychlorinated biphenyls (PCBs) are found in electrical equipment (Reddy *et al.*, 2019), dioxins, are found in burning wastes and industrial processes (Ross, 2004), progesterone are found in municipal and livestock's wastewater (Kasambala *et al.*, 2019), and BPA mostly found in plastics materials (Hernández-Abreu *et al.*, 2021). While specific hormones typically bind to designated receptor sites, these receptors can also interact with EDCs (Gore *et al.*, 2024). This interference disrupts hormone-receptor interactions, ultimately altering the endocrine system's response (Gore *et al.*, 2014).

Mechanisms of interaction between EDCs and hormones involve EDCs mimicking natural hormones, which can trigger a response that modifies the body's sensitivity to various hormonal

signals (Diamanti-Kandarakis *et al.*, 2009; Amir *et al.*, 2021). Additionally, EDCs may stimulate the production of hormones or accelerate their breakdown, leading to the depletion of specific hormones essential for normal body functions (Wang *et al.*, 2021; Wan *et al.*, 2022). Furthermore, some EDCs, such as BPA, Phthalates and PCBs, interfere with enzymatic activity by modifying the chemical structure of hormones, preventing them from binding to their corresponding receptor sites and thereby disrupting hormonal signalling (Schantz & Widholm, 2001). Moreover, progesterone and BPA are also other classes of EDCs of their kind that occur in the environment at high levels, posing alarming problems to ecosystems (Vilarinho *et al.*, 2019; Di Pietro *et al.*, 2023; Dias *et al.*, 2023).

Due to their wide range of applications, they are found in various environmental matrices, including surface water, groundwater, wastewater, soil, plants and air (Xiao *et al.*, 2020; Yilmaz *et al.*, 2020). They originate from point sources, such as pharmaceutical and plastic products and non-point sources, including agricultural runoff, stormwater and wastewater from treatment plants (Ismanto *et al.*, 2022; Ravikumar *et al.*, 2022). Wastewater from municipal and agricultural runoff contributes to the high EDC levels in the receiving rivers (Kasambala *et al.*, 2019; Radovic *et al.*, 2023), and their presence in the environment indicates potential health effects on ecosystems (Šauer *et al.*, 2018). Therefore, finding a novel, environmentally friendly technique for EDCs removal is paramount.

## **2.2 Environmental Progesterone and Bisphenol A Health Implication**

Progesterone and BPA appeared in different environment with health implications (Fig. 1). Progesterone is explicitly used as an oral contraceptive and sometimes used for hormone replacement therapeutics (De Quattro *et al.*, 2012; Gore *et al.*, 2024). Additionally, it is utilized in dairy processing as a hormonal cattle induction agent (Maggioni *et al.*, 2013; De Graaff & Grimard, 2018). However, progesterone either used as contraceptive or sourced from the environment, has several health implications. For instance, the study of Gore *et al.* (2024) has shown that even a low progesterone concentration can impact reproductive health and ecosystem development. According to Payus *et al.* (2021), birth control pills with gestagens are suspected to cause vaginal and breast cancers. Moreover, Frankel *et al.* (2018) has shown that environmental exposure to progesterone in humans affects sperm motility and fecundity and causes masculinization in the fathead minnow.

On the other hand, BPA is a synthetic chemical primarily used to produce polycarbonate plastics and epoxy resins (Vilarinho *et al.*, 2019; Xiao *et al.*, 2020). It is widely found in various

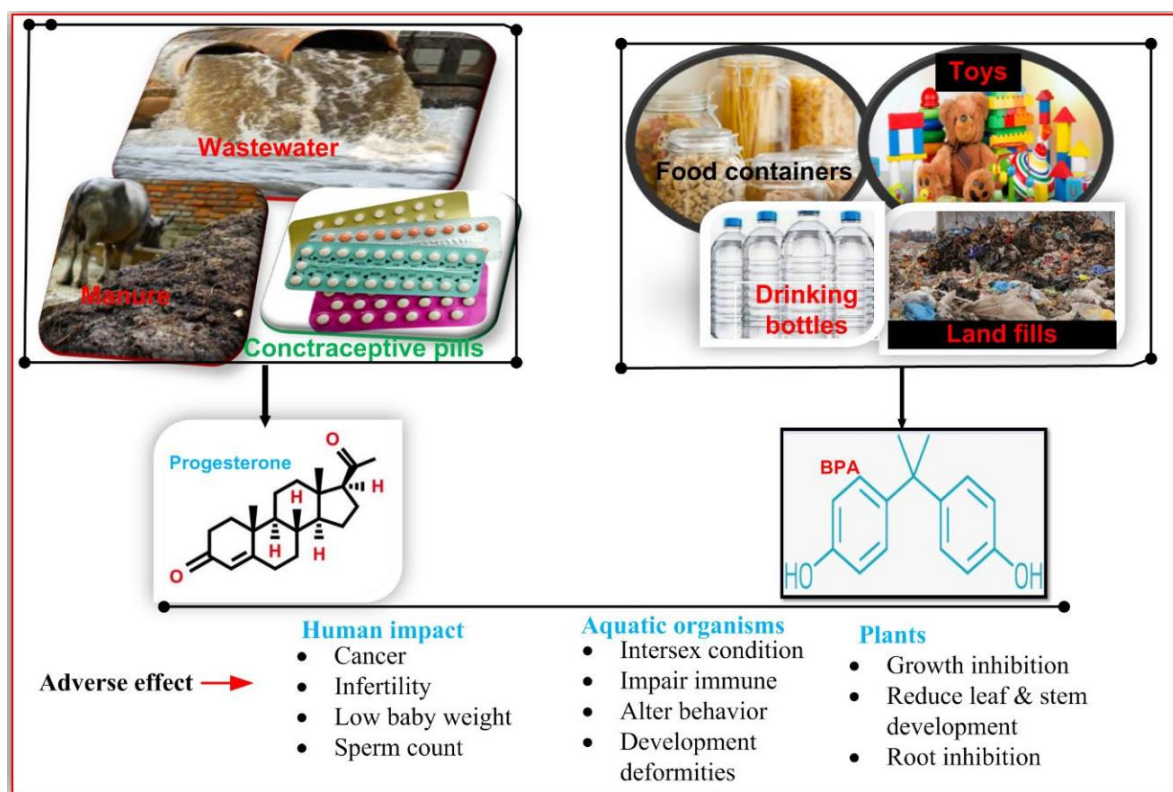
consumer products, particularly those related to food and beverages (Cimmino *et al.*, 2020). Most individuals' primary source of BPA exposure is diet, particularly from canned foods, polycarbonate containers, drinking water through leaching pipes and wastewater (Xiao *et al.*, 2020; Hernández-Abreu *et al.*, 2021; Nie & Wang, 2022). It was known that exposures to BPA, even in a low concentration (below 1 mg L<sup>-1</sup>), can cause low birth weight in male offspring (Flint *et al.*, 2012; Cimmino *et al.*, 2020), and it may also cause infertility, diabetes, breast, prostate and oral thyroid cancers (Vilarinho *et al.*, 2019; Xiao *et al.*, 2020; Uldbjerg *et al.*, 2022). That is why some countries, such as Canada, Europe, Sweden, the United States and Malaysia, have banned the use of BPA in baby bottles (Flint *et al.*, 2012; Gao *et al.*, 2020). Understanding the effects and sources of EDCs is crucial for identifying potential removal techniques to mitigate their impact on living organisms.

Environmental BPA and progesterone have a significant impact on aquatic organisms through hormonal disruption and toxicity. Bisphenol A, a known endocrine disruptor, causes acute toxicity, developmental deformities and reproductive issues in fish and invertebrates (Oetken *et al.*, 2004). Furthermore, it altered growth patterns and intersex conditions, with effects extending across generations (Mills & Chichester, 2005; Celino-Brady *et al.*, 2021). Bisphenol A can lead to abnormalities and adverse effects on cardiovascular health and survival, especially during early life stages (Wu & Seebacher, 2020; Wu *et al.*, 2023).

Progesterone can also influence reproductive behaviours and embryonic development; elevated levels may disrupt regular reproductive cycles (Akram *et al.*, 2022). Both compounds can impair immune function, alter normal behaviours and reduce reproductive success, ecosystems (Gore *et al.*, 2014; Kanda, 2019; Langston, 2020), ultimately threatening population stability and disrupting aquatic ecosystems. Furthermore, BPA and progesterone have diverse effects on plant growth and development (Fig. 1). Bisphenol A exhibits a hormesis effect, where low concentrations (1.50-3 mg L<sup>-1</sup>) can promote root growth and enhance levels of growth hormones like zeatin and gibberellin, leading to increased root length (Xiao *et al.*, 2020). However, higher concentrations (above 30 mg L<sup>-1</sup>) result in significant growth inhibition, causing root necrosis, reduced stem and leaf development and impaired photosynthesis due to oxidative stress (Wang *et al.*, 2015; Zhang *et al.*, 2015).

Conversely, progesterone at low doses can stimulate shoot growth and early seedling development, while higher concentrations may inhibit root proliferation and overall growth (Dumlupinar *et al.*, 2011). Both substances can influence hormonal balances within plants, affecting their physiological responses to environmental stressors (Mlynarcikova & Scsukova,

2018; Xiao *et al.*, 2020). The effects of these compounds on aquatic and terrestrial organisms and plants make this study necessary to find the best techniques for removing them from the environment.



**Figure 1: Summary of progesterone and BPA sources and their impact on the living organisms**

### 2.3 Treatment Techniques and Endocrine Disrupting Compounds Removal Challenges

Air, water, soil and biota are contaminated with EDCs, where wastewater effluents play a significant role (Anderson *et al.*, 2022). Even though the Aquatic environment is polluted with emerging micropollutants such as EDCs, most research focuses on analysing water quality and removing contaminants, such as nutrients, faecal coliform and heavy metals (Gahlan *et al.*, 2023; Natarajan *et al.*, 2023). However, few studies that have been done on the removal of such emerging micropollutants revealed that the techniques used for their removal are less effective (Kasambala *et al.*, 2019; Vu & Wu, 2022). Likewise, lack of analytical tools and expertise, on emerging organic contaminants removal (Sheriff *et al.*, 2020) are anticipated causes of the limited information on removal techniques. This section reviews the techniques used and their strengths and weaknesses in removing EDCs from wastewater.

### 2.3.1 Electrochemical Oxidation

Chemical advanced oxidation (CAO) is a treatment method in which strong oxidants transform contaminants in wastewater into nontoxic or low-toxic substances through oxidation reactions (Zhang *et al.*, 2019). Depending on the precise electrochemical method being used for EDCs removal, this causes the formation of reactive species, such as hydroxyl radicals ( $\cdot\text{OH}$ ) or persulfate radicals ( $\text{SO}_4\cdot^-$ ) (Plumlee *et al.*, 2009; Zhang *et al.*, 2019). These reactive species are capable of oxidizing EDCs molecules. Chemical oxidation methods, including ozonation, chlorination and catalytic oxidation such as Hydrogen peroxide ( $\text{H}_2\text{O}_2$ ), Permanganate ( $\text{MnO}_4^-$ ) and their combinations, such as UV/ $\text{O}_3$  and UV/ $\text{H}_2\text{O}_2$ , (Zhang & Li, 2014; Gao *et al.*, 2020). Although many chemical methods effectively remove successfully contaminants from wastewater, they sometimes fail to completely degrade EDCs and can lead to the formation of harmful by-products (Ahmed *et al.*, 2017; Ahmed *et al.*, 2021). For example, chlorination may not significantly reduce the estrogenic activity of certain compounds, such as BPA, resulting in the persistence of these contaminants in treated water (Lee *et al.*, 2004; Werkneh *et al.*, 2022).

Likewise, combining ozonation and UV with other oxidants requires capital (Werkneh *et al.*, 2022), which hinders their application, especially in developing countries. Moreover, the use of chemical treatments can also pose environmental risks. For instance, residual chemicals from treatment processes can re-enter ecosystems and disrupt aquatic life, complicating the overall impact on environmental health (Gadupudi *et al.*, 2021). Additionally, the efficiency of chemical treatments can be highly dependent on specific operational conditions, such as pH and temperature (Boczkaj & Fernandes, 2017). For example, using the ozonation method is pH dependent; any pH variation disturbs the treatment process (Martins *et al.*, 2015), affecting the consistency of different treatment setups. Hence, finding an environmentally friendly method is necessary for this study.

### 2.3.2 Anion Exchange Resins and Endocrine Disrupting Compounds Removal

Anion exchange resins effectively remove certain EDCs from wastewater through electrostatic interactions and adsorption (Del Moral *et al.*, 2020). These resins, particularly those with open structures and high water content, excel at capturing either negatively charged or ionisable contaminants such as BPA and acidic EDCs (Vilarinho *et al.*, 2019; Devaisy *et al.*, 2023). Ion exchange resins with quaternary ammonium groups (strong base) or amine groups (weak base) attract negatively charged EDCs such as sulfonated compounds or ionised BPA via

electrostatic forces (Ali *et al.*, 2023). In contrast, non-polar regions of EDCs interact with the resin's polymeric matrix, enhancing removal efficiency for uncharged molecules such as progesterone (Zaggia *et al.*, 2016). Furthermore, Del-Moral *et al.* (2020) study showed that anion exchange resins have functional groups that can swap out PFAS ions from wastewater and resin can be regenerated after becoming saturated with EDCs. The advantage of using ion exchange resin is based on its high selectivity for charged contaminants and regenerable with NaCl or NaOH solutions, therefore this property allows it to be used repeatedly (Harmer & Sun, 2001).

### **2.3.3 Biodegradation**

Moreover, the use of microbes in EDCs removal is the most common technique, especially in developing countries, due to the scarcity of resources. Biological treatment is the controlled natural process of using microorganisms to degrade or transform EDCs into less harmful substances (Ahmed *et al.*, 2017; Ismanto *et al.*, 2022). In most developing countries, biodegradation is the principal method of removing pollutants in wastewater (Anderson *et al.*, 2022; Ismanto *et al.*, 2022). In certain environments, some bacteria can degrade certain PFAS and thereby, lowering the levels of PFAS in effluents (De Silva *et al.*, 2021; Araújo *et al.*, 2022). Biological methods involve biodegradation, membrane bioreactor, phytoremediation and biofilms (Liu *et al.*, 2020; Antony *et al.*, 2022).

Mostly, biological methods show low efficiency in removing EDCs (Qiang *et al.*, 2013; Ahmed *et al.*, 2017), because many EDCs are naturally resistant to biodegradation due to their complex chemical structures. Most EDCs such as BPA, PFAS, progesterone and certain pharmaceuticals exhibit low biodegradability, making it difficult for microorganisms to break down effectively (Yuniarto & Hadibarata, 2024), leading to high levels of pollutants in aquatic environments. Furthermore, some EDCs in high concentrations can lead to toxicity in microbial communities, reducing their efficiency in degrading organic matter and affecting treatment performance (Chaturvedi *et al.*, 2023).

Likewise, wastewater often includes a complex combination of EDCs and other contaminants, which can interact differently (Gadupudi *et al.*, 2021; Antony *et al.*, 2022). Multiple pollutants can hinder the biodegradation process, as some compounds may compete for microbial resources or inhibit the degradation of others (Husain & Qayyum, 2013; Ojogoro *et al.*, 2017). Furthermore, the microbial transformation of EDCs can lead to the release of other by-products, which may also be EDCs (Liu *et al.*, 2020).

For instance, progesterone can degrade into estrone under certain conditions through aromatization (Ojoghoru *et al.*, 2017), while BPA can produce intermediate by-products such as 4-hydroxybenzoic acid. Therefore, finding the best ways and environmentally friendly techniques for completely removing EDCs is paramount. Hence, the environmental impact of using chemical and biological methods leaves physical methods, such as biochar adsorption, prone to EDC removal.

### **2.3.4 Biochar as a Suitable Adsorbent for Wastewater Treatment**

Biochar is a carbon-rich material produced from the pyrolysis of biomass under limited oxygen conditions (Dong *et al.*, 2023). It is an environmentally friendly carbonaceous material with promising advantages for removing various organic and inorganic pollutants from the environment (Lu *et al.*, 2014). Its unique properties make it an effective adsorbent for wastewater treatment, removing various pollutants, including heavy metals and EDCs (Anderson *et al.*, 2022). The materials used for biochar synthesis include agro-waste from agricultural and forestry materials, such as animal manures, algae, fungi, coconut shells, waste wood chips, paper wastes, corn husks, rice husks, cotton stalks, bamboo and cone cobs (Lu *et al.*, 2014; Rwiza *et al.*, 2018; Gao *et al.*, 2020; Wang *et al.*, 2021).

Feedstock solid wastes from plants and animals are commonly available, affordable and renewable resources (Foong *et al.*, 2022). Feedstocks are generated in vast quantities yearly and disposal is frequently challenging in most African countries (Hikal *et al.*, 2022; Hussin *et al.*, 2022). Biochar synthesis using waste materials reduces waste accumulation in the environment (Rynk *et al.*, 2022) while purifying water to ensure living organisms' health. Biochar mainly uses adsorption techniques to remove pollutants from wastewater (Wang *et al.*, 2024). Adsorption is a mass transfer method that occurs through hydrophobic and electrostatic interactions between the adsorbate and the adsorbent (Gao *et al.*, 2020). This technique has emerged as a prominent approach due to its simplicity in operation, flexibility, ability to produce non-toxic by-products and higher removal efficiency (Achak *et al.*, 2009; Dong *et al.*, 2023).

Adsorption involves the accumulation of molecules from a liquid or gas onto a solid surface (Ahmad *et al.*, 2020). Endocrine disrupting compounds removal through adsorption can be either by sorption, where EDCs adhere to the surface of adsorbents (Vieira *et al.*, 2022) or biodegradation, where adsorbents can facilitate biological processes that break down EDCs (Ismanto *et al.*, 2022). Chemical reactions can also be involved, and specific adsorbents may

engage in chemical reactions that contribute to EDC removal (Aris *et al.*, 2020). Sometimes the terms biochar and activated carbon (AC) are used interchangeably as carbon based adsorbents (Gęca *et al.*, 2022). Both are low-cost materials with large surface areas used to remove pollutants (Vercruyssen *et al.*, 2023). However, they differ in their production, structure and intended applications (Ahmed *et al.*, 2017).

Biochar is obtained through biomass pyrolysis, whereas activated carbon is a type of biochar that has undergone chemical or physical activation (Ahmed *et al.*, 2021; Nadew *et al.*, 2023). Additionally, biochar has appeared as a promising material for wastewater treatment due to its exceptional advantages. Biochar is an economical alternative to activated carbon because it requires less energy for its production (Wang *et al.*, 2020; Gęca *et al.*, 2022). Also, utilizing waste materials for biochar production reduces disposal costs and provides a valuable resource for wastewater treatment, which is an economic advantage (Buss *et al.*, 2022).

Furthermore, biochar has the potential to sequester carbon in the soil, thereby contributing to climate change mitigation (Qambrani *et al.*, 2017). Biochar's structure often allows it to maintain some level of adsorption capability even after use, making it potentially more sustainable over time (Werkneh *et al.*, 2022). Availability of various functional groups on the biochar makes it easy for ion exchange capacity to occur over a minimal amount, which provides a high ability to interact with diverse pollutants beyond simple adsorption (Nadew *et al.*, 2023).

#### **(i) Banana Peels Biochar and Pollutant Removal**

Banana peels are widely generated as agricultural waste globally, yet their potential remains underutilized mainly, leading to environmental disposal concerns (Hikal *et al.*, 2022). One effective approach to reducing waste is converting fruit by-products into valuable resources (Sirohi *et al.*, 2021). Research indicates that fruit waste can be converted into high-value products such as biochar for wastewater treatment and soil enhancement (Hussin *et al.*, 2022) or pharmaceuticals for medicinal applications (Hikal *et al.*, 2022).

Transforming agricultural waste, including banana peels, into biochar not only improves waste management (Ahmad *et al.*, 2020), but also fosters environmental sustainability by recycling organic materials (Li *et al.*, 2021). This process helps minimize landfill usage and reduces pollution from improper waste disposal. According to the Food and Agriculture Organisation (FAO), banana production is estimated at 250 million tons worldwide (Daffalla *et al.*, 2024). Nevertheless, approximately 114.08 million metric tons of peeled waste are generated (Bhavani

*et al.*, 2023). Banana peels waste are primarily found in African markets in huge quantities due to poor monitoring of the products (Mohd-Zaini *et al.*, 2022).

Currently, banana peels are utilized for animal feed, cosmetics, medicine, biofuel production, beverages and textile and fibre production (Bhavani *et al.*, 2023). However, vast amounts of waste are still generated, especially in African markets. Therefore, adding value to these peels by making biosorbent does not solve the endocrine disruptor problem only; it will also be used as a waste management solution to the environment.

Banana peels biochar is produced through a process known as pyrolysis. The produced biochar usually has a high surface area and adsorption capacity, making it a promising material for various applications, including wastewater treatment (Hu *et al.*, 2021; Daffalla *et al.*, 2024). On the other hand, banana peels have a high lignocellulose content, ensuring a high carbon composition. According to Orozco *et al.* (2014), banana peels contains 25.52, 11.45 and 9.82% by weight of hemicellulose, cellulose and lignin, respectively. Banana peels biochar are rich in functional groups such as hydroxyl, carbonyl and amino, aromatic rings and alkyl groups, which play a more significant role in binding EDCs pollutants (Munagapati *et al.*, 2020). The functional groups on the progesterone molecule may interact with the surface functional groups of the biochar (Yang *et al.*, 2019).

For example, the hydroxyl groups on the biochar surface can form hydrogen bonds with the hydroxyl groups on the BPA molecule (Flint *et al.*, 2012). Similarly, the ketone and methyl groups on the progesterone and hydroxyl groups in BPA may interact with the oxygen-containing functional groups on the biochar surface (Havens *et al.*, 2020; Xiao *et al.*, 2020). These interactions can lead to the adsorption of progesterone and BPA onto the surface of the banana peels biochar, making it crucial for managing EDCs in the environment. Furthermore, the large surface makes the banana peels more valuable to accommodate more pollutants during the adsorption process (Patel *et al.*, 2021; Farias *et al.*, 2023).

Banana peels biochar is highly efficient in removing organic and inorganic pollutants. Kapoor *et al.* (2022) used banana peels biochar to remove the reactive black 5 dye from aqueous solutions. From that research, the banana peels biochar showed about 97% removal efficiency achieved at pH 3 with a concentration of 75 mg L<sup>-1</sup> adsorbents at 500°C pyrolysis temperature. In a study by Achak *et al.* (2009), the banana peels biochar showed the highest removal capacity of 688.60 mg g<sup>-1</sup> for phenolic compounds from wastewater. Moreover, a study by Patel *et al.* (2021), showed that ciprofloxacin and acetaminophen were removed by banana peels biochar.

Additionally, under chemical modifications, banana peels biochar remove methylene blue, atrazine and glyphosate (Farias *et al.*, 2023).

Furthermore, Table 1 indicates progesterone and BPA removed with other feedstocks by adsorption processes. Vieira *et al.* (2022) indicated that fungiculture wastes can effectively adsorb progesterone. At the same time, the modified sweet prickly pear was shown to remove progesterone, testosterone, estriol and BPA, as well as  $\alpha$ -Oestradiol, from wastewater with moderate and low capacities for removing BPA and progesterone, respectively (Amusat *et al.*, 2023).

Additionally, Amusat *et al.* (2023) demonstrate that progesterone and BPA can be effectively removed by modifying the African star apple shell, with removal efficiencies of 86% and 87%, respectively, for progesterone and BPA. Since using banana peels biochar as an adsorbent has potential applications in water treatment and environmental remediation, mixing it with mango peels and other feedstocks provides more potent properties effective for removing EDCs from wastewater.

**Table 1: Variations of adsorbent adsorption capacity and efficiency for endocrine-disrupting compounds**

S/No	Biosorbent	Pyrolysis (°C)	Adsorbate	q <sub>max</sub> (mg g <sup>-1</sup> )	Removal (%)	References
1.	Fungiculture	600	Progesterone	232.64	91	Vieira <i>et al.</i> (2022)
			17 $\alpha$ -ethinylestradiol (EE2)	138.98	89	
2.	Modified sweet prickly pear	550	Estriol, BPA, $\alpha$ Oestradiol, $\beta$ -Oestradiol, testosterone progesterone	14.53, 9.75 14.53, 10.58, 12.50, 5.73 5.63	84 - 89	Amusat <i>et al.</i> (2023)
3.	Banana peels modified	600	methylene blue, atrazine, and glyphosate	33, 3.26, and 3.02	66, 91, 97	Farias <i>et al.</i> (2023)
4.	Banana peels biochar	750	ciprofloxacin and acetaminophe	20.42, 57.30	-	Patel <i>et al.</i> (2021)
5.	Banana peels biochar	500	Black 5 dye	7.58	97	Kapoor <i>et al.</i> (2022)
6.	Banana peel	-	Phenolic compounds	688.60	88	Achak <i>et al.</i> (2009)
	Modified the African star apple shell	550	Progesterone, BPA	40.50, 40.50	86, 87	Amusat <i>et al.</i> (2023)
7.	Banana peels biochar	700	Progesterone, BPA	43.42, 40.89	92.80, 80.50	This study

## (ii) Mango Peels Biochar and Pollutant Removal

Mango peels are often considered waste materials in many African countries. However, efforts are underway to find alternative uses for this waste product, including fuel, fertiliser, animal feedings, biochar, fruit powders and natural dyes (Ajila *et al.*, 2007; Rind *et al.*, 2022; Aung *et al.*, 2024). Mango peels biochar is a highly porous material with a large surface area, making it an excellent candidate for various environmental applications. According to Tapia-Orozco *et al.* (2016), mango peels are rich in carbon content due to hemicellulose, cellulose and lignin, which are 14.51, 9.19 and 4.25 wt%, respectively.

The presence of functional groups influences the high removal efficiency of mango peels for many micropollutants such as carboxylic acid (-COOH), hydroxyl and carbonyl groups (-C=O), which facilitate pollutant interaction (Razali *et al.*, 2022). These functional groups can interact with various contaminants in soil and water, such as heavy metals and organic pollutants, through adsorption and ion exchange mechanisms (Devaisy *et al.*, 2023). The carboxylic acid groups on mango peels biochar can form strong bonds with heavy metals, such as lead and Cadmium (Gao *et al.*, 2019; Geça *et al.*, 2022).

The hydroxyl and carbonyl groups can interact with organic compounds such as Polycyclic Aromatic Hydrocarbons (PAHs), BPA and pesticides (Sun *et al.*, 2022). Hydroxyl, ketones, C=C bonds and the alkyl chain of progesterone and BPA make it possible to interact by hydrogen bonding, pi-pi ( $\pi$ - $\pi$ ) and hydrophobic interaction with the aromatic surface of mango peels biochar (Ajila *et al.*, 2007). These interactions can lead to the removal or sequestration of contaminants from the environment, thereby improving soil and water quality.

Table 2 illustrates that mango peels biochar can effectively remove various types of pollutants from wastewater. The adsorption capacity of mango peels biochar was revealed to be 13.28 mg g<sup>-1</sup> for Cd (Zhang *et al.*, 2020), while Chen *et al.* (2022) obtained a high efficiency of 27.89 mg g<sup>-1</sup> at a pyrolysis temperature of 700°C. Mango peel, sometimes modified in various ways, exhibits a high capacity for removing heavy metals, drugs, nutrients and pesticides from wastewater and contaminated soil (Shehzad *et al.*, 2016; Rind *et al.*, 2022). The ability of mango peels to remove nutrients such as phosphorus and nitrates makes them suitable for controlling eutrophication pollution problems (Gao *et al.*, 2024). Few studies have been identified on using chemically modified mango peels biochar to remove heavy metals, with limited studies on removing EDCs.

**Table 2: Variation of mango peels adsorbent on removal efficiency and adsorption capacity for different pollutants**

S/No	Biosorbent	Pyrolysis (°C)	Adsorbate	q <sub>max</sub> (mg g <sup>-1</sup> )	(%) Removal	References
1.	Mango peel	500	Cd(II)	13.28	67.7	Zhang <i>et al.</i> (2020)
2.	Thermally activated Mango peel	400	Pb(II)	3831	-	Rind <i>et al.</i> (2022)
3.	Sea mango-activated biochar	400-800	Color, COD, NH <sub>3</sub> -N	83.33 Pt-Co, 35.71, 500	95.10, 84.90, 95.77	Shehzad <i>et al.</i> (2016)
4.	Mango peels hydrochar	400	Cd(II), Pb(II)	453.50	97.10	Rind <i>et al.</i> (2022)
5.	Mango kernel waste	700	Cd(II)	27.81	-	Zhang <i>et al.</i> (2022)
7.	Mango peel	700	Progesterone BPA	37.80, 41.01	87.80, 90.40	This study

Color - Pt-Co, COD - mg/L

### **(iii) Hybrid Synthesized Biochar and Pollutant Removal**

Using mixed feedstock for biochar preparation offers several advantages for the adsorption of organic and inorganic pollutants, primarily due to the synergistic effects of combining different biomass types (Sewu *et al.*, 2017). Biochar synthesized from mixed feedstocks enhances the surface area, and its porosity facilitates the formation of more heterogeneous active sites for the adsorption of diverse pollutants from various sources, compared to biochar synthesized from a single feedstock (Hoslett *et al.*, 2020; Wang *et al.*, 2020). However, single feedstocks provide homogeneous pores and are mainly applied for wastewater treatment, targeting the same sources of contaminants (Weber & Quicker, 2018).

This method can benefit industries and regions that consistently produce certain waste materials. Using mixed feedstocks can enhance biochar quality and increase the presence of oxygen-containing functional groups (e.g., hydroxyl, carboxyl, carbonyl) on the biochar surface (Hoslett *et al.*, 2020). These groups enhance the chemical interactions with organic contaminants, facilitating better adsorption through different mechanisms (Wang *et al.*, 2020). Additionally, material stability is strengthened due to the different thermal stability of the mixed feedstocks (Engamba-Esso *et al.*, 2022). This can produce biochars that are more resistant to decomposition and retain their pollutant adsorption efficiency (Sewu *et al.*, 2017).

Similarly, mixed feedstocks can enhance and better alter the pH of biochar; for instance, combining acidic and alkaline feedstocks can help modify the pH to suit the specific pollutants being targeted (Wang *et al.*, 2020). For example, acidic biochars might be better for adsorbing basic pollutants, while alkaline biochars may perform better with acidic pollutants (Yin *et al.*, 2019). Furthermore, mixing different biomass materials can facilitate the coexistence of multiple adsorption mechanisms such as physical adsorption, ion exchange and electrostatic interactions (Kazak, 2021). This makes biochar more effective in adsorbing a broader range of organic pollutants, including both polar and non-polar compounds (Ahmed *et al.*, 2018). Therefore, using hybrid biochar helps in waste management by converting diverse organic residues into valuable biochar at once.

Different studies have proven the pronounced properties of co-pyrolysis biochar in various applications. A study by Liu *et al.* (2018) found that biochar produced from a mixture of rice straw and pig manure had a higher surface area, pore volume and cation exchange capacity than biochar produced from a single feedstock. Furthermore, the combination of wheat straw and chicken litter exhibits higher pH, carbon, inorganic and ash content compared to its

individual components (Taherymoosavi *et al.*, 2016). Mixing wheat straw with basalt provides biochar with high ash content and low fixed carbon, volatile organic matter, electric conductivity (EC) and dissolved organic matter compared to its single feedstocks (Taherymoosavi *et al.*, 2018).

Compared to their single feedstocks, mixed feedstocks exhibit high performance in removing a diverse range of pollutants, including dyes, heavy metals, phenols and pesticides (Sewu *et al.*, 2017; Siyal *et al.*, 2018; Gao *et al.*, 2019). Nielsen *et al.* (2015), also indicated that the mixture of sewage sludge and fish waste shows high efficiency in removing carbamazepine. Sewu *et al.* (2017) compared the efficiency of a single mushroom and a mixed spent mushroom with seaweed kelp in removing crystal violet, with the  $q_{\max}$  of 282.90 and 610.10 mg g<sup>-1</sup>, respectively. Nielsen *et al.* (2015) studied the removal of carbamazepine using fish waste mixed with sewage slug and single sewage slug with  $q_{\max}$  of 37.10 and 18.10 mg g<sup>-1</sup>, respectively. According to Li *et al.* (2021), the co-pyrolysis of cyanobacterial and polyethylene waste exhibits a maximum adsorption capacity ( $q_{\max}$ ) of 490 mg g<sup>-1</sup> for removing methylene blue from synthetic wastewater.

Likewise, blended corn stalk and polyethylene at 50 mg L<sup>-1</sup> of adsorbate, 0.10 g of adsorbent and pH of 4.50 show a high  $q_{\max}$  of 99.50 mg g<sup>-1</sup> for the removal of Pb (Fan *et al.*, 2020). Additionally, combining different granular activated carbons (GACs) increased the removal of algal organic matter (AOM) from 25 - 44% for single GACs and from 52% to 74% pesticide removal (Sichrova *et al.*, 2025). Furthermore, only two studies have been identified on using co-blended feedstocks for the adsorption of pollutants from real wastewater. First, the co-pyrolysis of red mud and sucrose at an adsorbent of 2 mg L<sup>-1</sup>, pH of 2.10 and 120 min with a  $q_{\max}$  of 20.41 mg g<sup>-1</sup> for Chromium removal (Kazak, 2021). Second, another study indicates that wood and plastic waste can remove phosphates and turbidity from wastewater. It was reported that the efficiency removal for phosphate and turbidity were 6.36% and 78.25%, respectively (Omoniyi & Salami, 2018).

Mixing feedstocks is the best method for increasing the possibility of high efficiency of biochar, because different adsorbents possess varied surface properties, pore structures and functional groups (Retnam *et al.*, 2022). When combined, these features can complement each other, leading to better overall adsorption performance and broader contaminant coverage (Sichrova *et al.*, 2025). Furthermore, what to mix is also necessary to avoid the emission of toxic products like PAHs, dioxins and PCBs (Zama *et al.*, 2018). The amount of dioxins produced by incomplete dehydrochlorination can be decreased by avoiding the use of specific

feedstocks, such as those containing copper or chlorine (Lu *et al.*, 2019). In contrast, PAHs can be reduced by avoiding feedstocks that contain plastics (Sophonrat *et al.*, 2019).

Therefore, mixing feedstocks having different properties and functions and varying pyrolysis temperatures, generally provides high-quality biochar suitable for specified applications (Taherymoosavi *et al.*, 2016). To the best of knowledge, no published study has investigated the co-pyrolysis of banana and mango peels feedstock biochar for removing EDCs from wastewater. Therefore, this study contributed to understanding the use of hybrid biochar to mitigate the risk of EDCs to human health while preserving the environment.

#### **2.4 Interaction Mechanisms Between Biochar and Endocrine Disrupting Compounds**

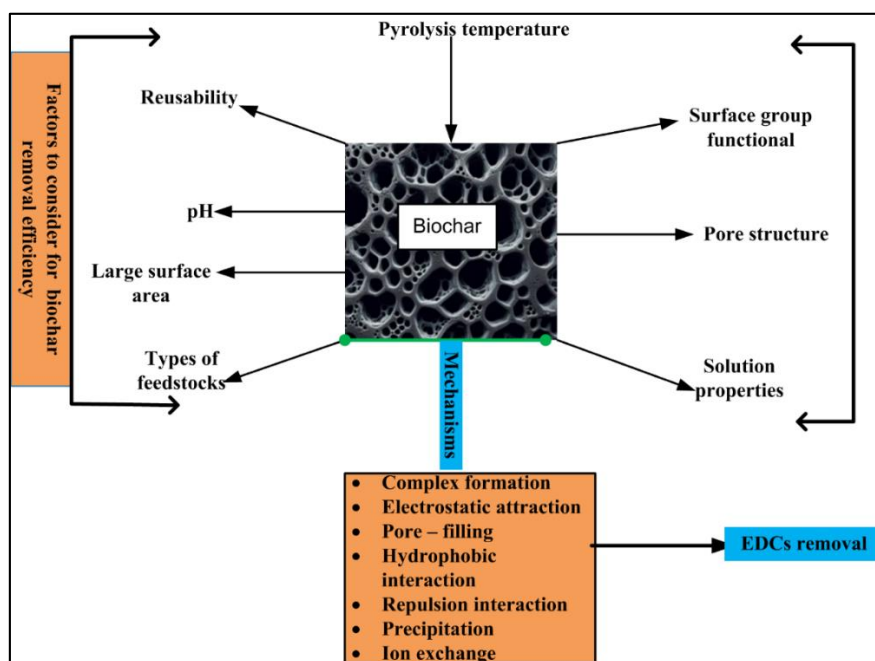
The interaction mechanisms between biochar and EDCs are a critical area of study due to the increasing concern over EDCs in water sources and their potential health impacts (Fig. 2). Different mechanisms are involved during the interaction between biochar and EDCs (Ambaye *et al.*, 2021). Electrostatic interactions are among the mechanisms involved during EDCs' adsorption. The surface charge of biochar can attract oppositely charged EDCs, facilitating their adsorption (Cimmino *et al.*, 2020). This is particularly relevant for charged contaminants, where the electrostatic attraction enhances binding efficiency (Ahmad *et al.*, 2020b). For instance, electrostatic attraction mechanisms are vital in progesterone adsorption processes (Esmaeeli *et al.*, 2017). Under basic conditions, the C-O group of the biochar surface deprotonates, creating a negative charge surface that attracts the positively charged region of the progesterone, leading to electrostatic attraction (Esmaeeli *et al.*, 2017; Šauer *et al.*, 2018).

Also, the availability of a hydrogen atom in the hydroxyl group and the polarity of the carbonyl group in progesterone cause the O-H groups found on the biochar surface to form hydrogen bonds with progesterone (Amir *et al.*, 2021; Dias *et al.*, 2023). Likewise, pore filling can be applied to the porous structure of biochar to allow EDCs to be physically trapped within its pores, increasing the overall adsorption capacity (Dias *et al.*, 2023). The size and distribution of these pores can significantly influence how effectively different EDCs are retained (Ahmed *et al.*, 2018). For example, the porous structure of the biochar combines with the C-O group of pollutants by van der Waals forces, enhancing the entrapment of pollutants into the biochar pores (Valenzuela-Calahorra *et al.*, 2004).

Additionally, EDCs removal from wastewater may be facilitated by ion exchange processes. This occurs when cations in the biochar exchange with positively charged sites on the EDCs molecule or its associated ions in solution (Peiris *et al.*, 2020). This mechanism can

significantly enhance the removal efficiency of EDCs by increasing the effective concentration of binding sites available on the biochar (Ahmed *et al.*, 2018; Dias *et al.*, 2023). Moreover, metal ions on the biochar surface at the basic medium cause EDCs to form a surface complexation with metal ions or carbonates on the biochar surface (Peiris *et al.*, 2020), forming a stable complex that enhances the hormone's retention on the biochar surface, leading to improved removal rates.

Many EDCs are hydrophobic; thus, their adsorption onto biochar can occur through hydrophobic interactions, where non-polar molecules preferentially associate with the non-polar surfaces of biochar (Cimmino *et al.*, 2020). For instance, progesterone and BPA are hydrophobic molecules; thus, their removal can be enhanced through hydrophobic interaction mechanisms with the graphene structure of biochar (Amir *et al.*, 2021; Hernández-Abreu *et al.*, 2021). The biochar's aromatic rings and hydrophobic surfaces provide favourable conditions for such interactions, accompanied by pollutant diffusion into pores and non-carbonised materials (Amir *et al.*, 2021). Therefore, understanding these mechanisms is critical for optimising biochar production and application strategies in environmental remediation efforts targeting EDCs.



**Figure 2: Critical factors affecting biochar efficiency and selected EDCs removal mechanisms**

## 2.5 Factors Influencing the Adsorption Efficiency of Biochar

The effectiveness of biochar in adsorption depends on various intrinsic and extrinsic factors that govern its structural, chemical and surface properties (Fan *et al.*, 2021; Li *et al.*, 2021).

These factors influence the interaction between biochar and contaminants (Ahmad *et al.*, 2020). Understanding these factors is essential for optimising biochar production and modifying its properties for specific environmental applications. The following are factors as summarized in Fig. 2, determining the effectiveness of biochar for sorption purposes.

### **2.5.1 Effect of Pyrolysis Temperature and Type of Pollutants Adsorbed**

Pyrolysis is the thermal decomposition of organic materials without oxygen, primarily occurring at 300°C to 850°C under limited air supply (Engamba-Esso *et al.*, 2022). While co-pyrolysis involves blending feedstocks, producing a hybrid biochar with a combination of properties (Sichrova *et al.*, 2025). Hybrid characteristics are influenced by the synergistic effect contributed by single feedstocks. The synergistic co-pyrolysis impact is positive or negative depending on the yield characteristics (Sewu *et al.*, 2017). If the interaction of the hybrid feedstocks produces a total value higher than that of the single feedstocks, it is termed as positive synergism. While negative synergistic effects happen when the yield or property of hybrid biochar deteriorates compared to single feedstocks (Abnisa *et al.*, 2015; Yin *et al.*, 2019).

However, the synergistic effect of co-pyrolysis depends on various factors, including pyrolysis temperature, feedstock type, blending ratio, residence time, reactor type, reaction temperature and catalyst (Sewu *et al.*, 2017; Yin *et al.*, 2019; Engamba-Esso *et al.*, 2022). The end product of pyrolysis is chars and gases, with biofuel as the main product depending on the pyrolysis temperature (Abnisa *et al.*, 2015). A slower pyrolysis rate with a slow temperature leads to biochar production (Mandal *et al.*, 2017), while a high heating rate with a longer residence time maximizes the production of biofuel (Abnisa *et al.*, 2015). For biochar application, pyrolysis temperature significantly influences the physical and chemical properties of biochar for removing pollutants from wastewater (Weber & Quicker, 2018). High pyrolysis temperatures lead to larger surface areas and enhanced porosity, which facilitate the adsorption of contaminants (Dias *et al.*, 2023). However, low pyrolysis temperatures may result in slower sorption rates due to the presence of condensed organic phases (Liu *et al.*, 2018).

The effects of pyrolysis temperature depend on the type of pollutants adsorbed. For instance, chromium Cr (VI), a heavy metal, showed a decrease in removal efficiency by coconut shell as the pyrolysis temperature increases from 350 to 650°C (Hou *et al.*, 2022). At low temperatures, there is an availability of functional groups that enhance electrostatic attraction for complexation and chemical bonding with metal ions (Weber & Quicker, 2018). On the

other hand, naphthalene, an organic pollutant, shows high removal efficiency at 600°C due to its increased surface area and pore structure (Hou *et al.*, 2022).

According to Wang *et al.* (2020), for organic adsorption, the pyrolysis should range from 600-700°C, maintaining the balance between sufficient functional groups while increasing the surface area and aromaticity. This balance is crucial for maximizing the removal efficiency of a wide range of organic and inorganic pollutants such as sulfamethoxazole, thiacloprid, PO<sup>3-</sup>, Cr (VI) (Zhang *et al.*, 2018; Yin *et al.*, 2019; Kazak, 2021). Therefore, finding the best pyrolysis temperature and well-feedstock combinations provides a biochar with the best properties for diverse functions.

### 2.5.2 Availability of Surface Functional Groups

The functional groups on the surface of biochar play a crucial role in its ability to adsorb various pollutants. These functional groups, such as Carboxyl (-COOH), Hydroxyl (-OH), Carbonyl (C=O) affect the interaction between the biochar and different pollutants through different mechanisms (Šauer *et al.*, 2018). These involve hydrogen bonding, electrostatic interactions, surface complexation and ion exchange (Ahmed *et al.*, 2018; Wang *et al.*, 2020). The biochar with a high carboxyl content- incredibly rich in cellulose or hemicellulose- forms strong complexes with heavy metal cations such as Pb<sup>2+</sup> and Cd<sup>2+</sup> (Gao *et al.*, 2019). The carboxyl groups provide negatively charged sites that attract and bind positively charged metal ions (Ahmed *et al.*, 2018).

Likewise, hydroxyl groups on the biochar can form hydrogen bonds with organic pollutants, such as BPA and progesterone (Xiao *et al.*, 2020). This occurs when the hydroxyl group donates a hydrogen bond to the oxygen atom in the pollutant, facilitating adsorption onto the biochar surface (Peiris *et al.*, 2020). For instance, biochar made from coconut husks tends to have hydroxyl groups that can help remove phenols from wastewater streams (Wang *et al.*, 2021). These groups contribute to the biochar's cation exchange capacity (CEC) by providing oxygen atoms that can interact with positively charged contaminants (Smith *et al.*, 2015). Furthermore, some biochar contain phenolic compounds, particularly those with aromatic ring structures, which enhance the adsorption of non-polar organic pollutants (Qiu *et al.*, 2022). The phenolic groups can form  $\pi$ - $\pi$  interactions with the aromatic rings in organic molecules (Zhang *et al.*, 2021).

For instance, due to its phenolic groups, biochar produced from peanut shells may effectively adsorb atrazine from soil and water (Xiao *et al.*, 2020). Most biochar contains diverse

functional groups, increasing the possibilities of adsorbing many types of pollutants (Dias *et al.*, 2023). Thus, mixing different feedstocks provides diverse functional groups contributed by both biochars for various applications, including wastewater treatment from multiple sources and soil remediation.

### 2.5.3 Surface Area and Porosity

Biochar's specific surface area and pore volume are vital for its adsorption abilities (Amir *et al.*, 2021). A larger surface area generally provides more sites for pollutants adsorption (Patel *et al.*, 2019). The greater the surface area and pores volume, the more pollutants or molecules can interact with the biochar, increasing its adsorption capacity (Tan *et al.*, 2021). A large surface area increases the biochar porosity, which provides the biochar with various pore structures, including microporous (<2 nm), necessary for adsorbing small molecules (Gao *et al.*, 2019), mesoporous (2–50 nm) and macroporous (>50 nm) to handle larger pollutant molecules such as EDCs (Nielsen *et al.*, 2015). Similarly, according to Sun *et al.* (2022), a higher pore volume biochar can hold more adsorbate because it provides more physical space for trapping molecules (Tan *et al.*, 2021).

Studies show that banana peels biochar with a surface area of 9.65 m<sup>2</sup> g<sup>-1</sup> removes Congo red dye effectively from wastewater (Daffalla *et al.*, 2024). Likewise, the activated banana peels biochar with a surface area of 432 mg g<sup>-1</sup> shows effective dye adsorption (Buss *et al.*, 2022). Furthermore, the maximum surface area of mango peels was attained at 731 m<sup>2</sup> g<sup>-1</sup> with phosphoric acid activation (Majumder & Das, 2022). Similarly, high adsorption efficiency of BPA in porous carbon appeared at 1000°C pyrolysis temperature with a surface area of 300 m<sup>2</sup> g<sup>-1</sup> and a pore volume of 0.15 cc g<sup>-1</sup> (Tong *et al.*, 2019). While the fungiculture with 246 m<sup>2</sup> g<sup>-1</sup> removes progesterone effectively from wastewater (Vieira *et al.*, 2022).

However, the adsorption efficiency of biochar is influenced by the balance between micropores, mesopores and macropores, which determines its ability to capture different molecules (Nielsen *et al.*, 2015). Furthermore, a well-developed pore structure allows for better accessibility of adsorbates, facilitating their retention within the adsorbent matrix (Li *et al.*, 2021). Well-connected pores enhance mass transfer, facilitating rapid diffusion of adsorbates to active sites (Bian *et al.*, 2024), whereas poorly connected pores hinder adsorption efficiency (Wang *et al.*, 2024). To optimize pollutant removal, mixing biochar with varied pore sizes creates a hybrid material capable of accommodating diverse contaminants without requiring chemical modifications.

#### 2.5.4 Types of Feedstocks

The feedstock type also determines biochar's ability to remove pollutants. Different feedstocks, such as wood, agricultural residues, sludge, etc., produce biochar with varying pore structures, surface functional groups and ash content (Nielsen *et al.*, 2015; Rwiza *et al.*, 2018; Wang *et al.*, 2024). Type of feedstocks used influence the ability of biochar in different ways. For instance, nutrient-dense feedstocks such as animal waste, sludge and manure produce biochar with higher adsorption capacities for the removal of heavy metals and organic pollutants (Havens *et al.*, 2020). This is because such feedstocks often contain more nitrogen, oxygen, phosphorus and potassium, enhancing functional group and element availability on the biochar surface and improving pollutant interaction (Ahmed *et al.*, 2018; Anderson *et al.*, 2022).

Similarly, feedstock type can affect the polarity of the biochar; for instance, according to Pipiška *et al.* (2022) biochar produced from high-lignin content feedstocks such as woody materials are more hydrophobic and stable with high carbon content, while Yadav and Singh (2023) study indicates feedstocks rich in proteins or starches like food waste can yield biochar with more hydrophilic properties, which may be better suited for adsorbing polar compounds. Therefore, finding the best feedstock materials provides adsorbents with the best properties for adsorption of diverse micropollutants in wastewater.

#### 2.5.5 Contact Time and Initial Sorbate Concentration

Adequate contact time is crucial for maximizing pollutant removal in biochar. Longer contact times allow maximum adsorption (Ahmad *et al.*, 2020), but after a certain point, equilibrium is reached and no more adsorption occurs (Dias *et al.*, 2023). Extended contact times can lead to lower desorption rates, indicating stronger binding of contaminants to biochar (Liu *et al.*, 2018). The maximum contact time for heavy sorption by using nanocomposite iron-modified biochar is 360 min, while Kumar *et al.* (2023) reveal a contact time of 110 min for the removal of Zn(II) from aqueous solution onto *Argemone maxicana*-derived biochar.

Furthermore, organic pollutants do not show a difference in the contact time, with optimal removal observed at 60 minutes (Ambaye *et al.*, 2021; Vieira *et al.*, 2022). Likewise, sorbate concentration (the adsorbate) significantly influences biochar's adsorption capacity for various pollutants (Qiu *et al.*, 2022). Higher initial concentrations can enhance the driving force for mass transfer, leading to increased adsorption rates (Ndoun *et al.*, 2021). As sorbate concentration increases, more molecules are available to interact with the biochar surface

(Batool *et al.*, 2023). This typically leads to an increase in the amount of pollutant adsorbed until a saturation point is reached (Ni *et al.*, 2011).

For example, studies have shown that as Nickel (Ni) ion concentration increased from 20 to 100 ppm until saturation was achieved (Batool *et al.*, 2023). Beyond a specific concentration, the adsorption rate may plateau or decrease because all available active sites on the biochar surface become occupied (Yu *et al.*, 2021; Wang *et al.*, 2024). This indicates that further increases in sorbate concentration do not significantly enhance removal efficiency due to saturation.

### **2.5.6 Effect of solution pH**

On the other hand, the pH of the solution plays a crucial role in influencing the adsorption capacity of biochar for various pollutants (Wang *et al.*, 2024). Xiao *et al.* (2020) verified that the pH effect is due to changes in biochar's surface charge and the contaminants' speciation in their ionic forms. The pH can enhance the adsorption capacity through the Point of Zero Charge (PZC), a point at which the biochar surface charge is neutral (Xiong *et al.*, 2021). Below this PZC, biochar carries a positive charge; hence, it influences the adsorption of negatively charged species such as phosphates (Shehzad *et al.*, 2016). While pH above it, biochar becomes negatively charged, which influences the adsorption of positively charged species such as ammonia and heavy metals through electrostatic attraction processes (Rind *et al.*, 2022). The pH can also affect the dissociation of an adsorbate. Many adsorbates, such as heavy metals or organic compounds, can exist in ionized or non-ionized forms depending on the pH of the solution (Ahmed *et al.*, 2018).

For instance, heavy metals like Lead ( $Pb^{2+}$ ), Cadmium ( $Cd^{2+}$ ), or Chromium ( $Cr^{3+}$ ) are more likely to remain in their cationic form at lower pH levels (Liu *et al.*, 2019), making them more easily adsorbed onto biochar with negatively charged surfaces. At the same time, organic compounds with functional groups, such as  $-COOH$  or  $-NH_2$ , may ionize depending on the pH (Ni *et al.*, 2011). At low pH,  $-COOH$  may remain protonated, reducing their ability to complex with cations like heavy metals (Navarro *et al.*, 2005). Furthermore, these groups become deprotonated ( $-COO^-$ ) at high pH, enhancing their ability to bind to cations through complexation (Bala *et al.*, 2007).

Furthermore, BPA can ionize at higher pH, experiences greater adsorption at low pH, where it remains neutral, allowing for strong hydrophobic interactions with the biochar (Ahmed *et al.*, 2018). As pH increases, BPA becomes negatively charged, leading to electrostatic repulsion

from the similarly charged biochar surface (Ahmed *et al.*, 2018). Conversely, progesterone remains neutral across a wide pH range and is primarily adsorbed via hydrophobic and  $\pi$ - $\pi$  interactions, with higher adsorption at low to moderate pH (3-11) (Georgin *et al.*, 2024). Therefore, mixing different feedstocks for biochar preparation makes the balance of pH to maximize the adsorption efficiency for diverse pollutants.

### **2.5.7 Reusability and Environmental Sustainability**

The number of cycles a biochar can undergo before its adsorption capacity diminishes is a crucial factor for its practical applications. The reusability of biochar refers to its ability to maintain high adsorption capacity over multiple adsorption-desorption cycles (Munagapati *et al.*, 2020; Kayranli *et al.*, 2024). The reusability of biochar through adsorption-desorption cycles varies significantly depending on its application, feedstock, modification and target pollutants. Regeneration techniques can be desorption through chemical regeneration (Alsawy *et al.*, 2022). This involves the use of organic solvents (ethanol and methanol) or chemical reagents, depending on the nature of the pollutant to be desorbed (Li *et al.*, 2021; Gkika *et al.*, 2022).

Reusability is crucial in determining the long-term effectiveness and economic feasibility of biochar as an adsorbent (Hu *et al.*, 2022; Kayranli *et al.*, 2024). Different biochars are seen to regenerate in more than three cycles. For instance, algae-based materials maintained 60–80% of their initial BPA adsorption capacity after 4–6 cycles using ethanol as a regenerant (Liu *et al.*, 2017). Additionally, activated carbons synthesized from agricultural waste were found to regenerate five times after being used to remove BPA from wastewater (Zafar *et al.*, 2022). For the heavy metals, biochar can also be regenerated, with ~50% Pb(II) desorption from wheat straw biochar after 5 cycles; however, chemisorption limits full recovery (Alsawy *et al.*, 2022). While the use of fungal-based appeared to perform 85% at 3 cycle for removal of progesterone (Abdelwahab *et al.*, 2025).

The adsorption sites' stability can influence biochar's reusability, for biochar to be reusable, the active adsorption sites on its surface must remain intact after each cycle (Hu *et al.*, 2022). Furthermore, desorption efficiency is vital to reusability since it ensures that the pores and active sites are cleared, making them available for new adsorbate molecules in the next cycle (Gkika *et al.*, 2022). Incomplete desorption, where residues of the adsorbate remain in the pores, can block adsorption sites and reduce the total available surface area (Toncón-Leal *et al.*, 2021; Kayranli *et al.*, 2024), decreasing the biochar's capacity in future cycles. For

example, ammonium adsorption declined by 30% after 3 cycles due to pore blockage but remained viable for agricultural reuse (Rezaee *et al.*, 2021). Therefore, for biochar to be effectively used as a biosorbent, it should have high efficiency and be sustainable for environmental advantages.

## 2.6 Literature-based Novelty Statement and Research Gap

Recent investigations into the use of biochar for the remediation of EDCs have made significant progress; however, several persistent limitations hinder a comprehensive understanding and the scalable application of this technology. A major shortcoming in current literature is the predominant use of biochar derived from single feedstocks, such as wood, coconut shells or agricultural residues, in isolation (Wang *et al.*, 2021; Carnimeo *et al.*, 2023). This approach neglects the potential benefits of hybrid biochars synthesized from mixed feedstocks such as BP and MP, which are abundant and rich in complementary organic constituents. Hybrid biochars exhibit enhanced physicochemical properties, including increased surface area, improved pore distribution and diverse functional groups, which collectively contribute to superior adsorption capacity and selectivity toward EDCs.

Furthermore, most studies rely heavily on batch adsorption experiments conducted in synthetic wastewater systems (Kayranli *et al.*, 2024; Aricov & Leontieş, 2025). While these controlled environments offer reproducibility, they fail to replicate the complex chemical and biological matrices found in real municipal wastewater. This thesis addresses this gap by evaluating biochar performance in actual municipal wastewater, thereby providing more ecologically valid insights into its remediation potential.

Another gap lies in the limited investigation of competitive adsorption removal under co-existing EDCs. In real-world scenarios, compounds such as BPA and progesterone often co-occur, interacting with each other and with common co-occurring substances, including heavy metals and pharmaceuticals (Lalik *et al.*, 2025; Sundaram *et al.*, 2025). This study systematically investigates these competitive interactions, revealing the adsorption behaviour, selectivity and synergistic effects within multi-contaminant systems.

Moreover, the sustainability of biochar application hinges on its reusability and regeneration potential, which remain underreported in existing research. This thesis contributes novel data by assessing the recyclability of hybrid biochar across multiple adsorption-desorption cycles, using environmentally benign regeneration methods. The findings offer critical insights into the long-term feasibility and cost-effectiveness of biochar-based remediation strategies. By

addressing these interconnected gaps feedstock diversity, realistic wastewater matrices, competitive adsorption removal, and material reusability this study advances the scientific foundation for using biochar in complex environmental surroundings and supports its integration into circular economy frameworks for wastewater treatment.

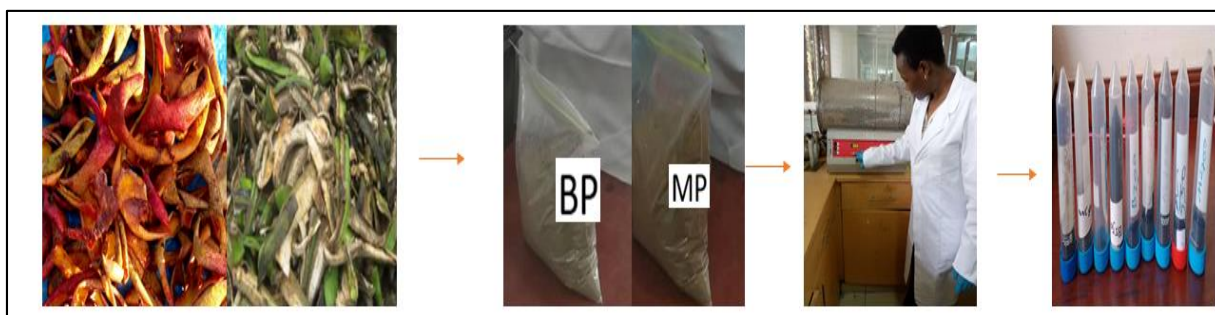
## CHAPTER THREE

### MATERIALS AND METHODS

#### 3.1 Experimental and Quality Assurance

##### 3.1.1 Biosorbent Preparation

Fruit waste samples were collected from a local market in Tengeru and Kilombero in Arusha, Tanzania and transported to the Nelson Mandela African Institution of Science and Technology (NM-AIST) laboratory in Tanzania for further processing. The samples were sorted to separate banana and mango peels from other wastes. The sorted samples were washed with tap water and rinsed with distilled water to remove solid and soluble particles. Then, the samples were cut into small pieces, sun-dried for two days and oven-dried at 105°C for 24 hours. Subsequently, the dried samples were ground into a powder and sieved through a 40-mesh sieve for further processing (Fig. 3).



**Figure 3: Materials preparation**

Then, for BPB and MPB preparation, 30 g of powdered peels were placed into a crucible in a tube furnace with nitrogen gas flow ( $2 \text{ Lmin}^{-1}$ ) to provide an oxygen-free environment at a specified temperature. For hybrid biochar (HB), 10 g of powdered peels, after mixing MP and BP powder in 1: 1.5 ratio, respectively, were used. Prior to selection of best combination ratio of biochar, characterization of BPB and MPB was done and its results was used as a criteria setting the ratios including 1:1, 2:1 and 1:1.5, respectively. All samples were pyrolysed at 300, 550, 700 and 800°C for 2 hours, with heating rates of 9, 11, 12 and 14°C  $\text{min}^{-1}$ , respectively. This is at different temperatures, where the rate of chemical reactions (e.g., bond breaking, volatile release) varied significantly. After the pyrolysis process, the biochar obtained was cooled, sieved using 80–180 mesh sieve and stored tightly in plastic containers at room temperature until the adsorption process.

### 3.1.2 Analytical Reagents

Analytical-grade chemicals and reagents were used during this experimental study. Thermo Fisher Scientific supplied a progesterone standard (98%) and a 2,2-Bis(4-hydroxyphenyl) propane standard (98%). Sodium hydroxide pellets (NaOH) (98%) and Hydrochloric acid (HCL) (37%) were supplied by Loba Chemie PVT. The LTD was used for pH regulation. Sigma-Aldrich provided High-Performance Liquid Chromatography (HPLC-grade) methanol (99.90%) for EDCs preparation. Solid-phase extraction (SPE) C18 cartridges (500 mg, 6 mL) obtained from J & K Scientific (Beijing, China) were used for hormone pre-concentration and matrix clean-up. The elution solvents, n-heptane (99%) and acetone (99.8%), as well as the GF/C-GDSO210-4045 membrane filters used for removing suspended debris and particulates, were supplied by Fisher Scientific (Waltham, MA, USA). Furthermore, deionised water was used as the primary solvent throughout the experimental procedures.

### 3.1.3 Equipment and Apparatus

Adsorption experiments were conducted using an orbital incubator (Model SI600C), ensuring uniform agitation and temperature control. Hormone quantification was performed via High-Performance Liquid Chromatography (HPLC) using an Agilent 1200 series system (Santa Clara, CA, USA) at the African Technical Research Centre (ATRC) laboratory.

Biochar synthesis was carried out using a split-hinge tube furnace (200°C split-hinge) for pyrolysis. Physical parameters of wastewater were measured using a Multiparameter probe (Model HI98494, Germany), and Total Suspended Solid TSS and Total Solid (TS) were measured through galvanometric methods. While chemical parameters, including Nitrate ( $\text{NO}_3^-$ ), Nitrite ( $\text{NO}_2^-$ ) and Phosphate ( $\text{PO}_4^{3-}$ ), were quantified using a Unico 2800 UV/VIS spectrophotometer. Potassium ( $\text{K}^+$ ) and Calcium ( $\text{Ca}^{2+}$ ) concentrations were determined using a flame atomic absorption spectrophotometer (PerkinElmer® FL 6500™ & FL 8500™). For organic load assessment, Chemical Oxygen Demand (COD) was measured using the (HI-839800 25 Vial) thermo-reactor and Biological Oxygen Demand (BOD) was analysed using the OxiTop Box 140601 reactor. All chemical analyses were conducted at the Tanzania Agricultural Research Institution (TARI), Uyoile laboratory.

Laboratory procedures were supported at the NM-AIST Laboratory. Adsorbents were ground using a mortar and pestle and sieved to obtain uniform particle sizes. Drying was achieved using laboratory ovens, while desiccators ensured a moisture-free storage environment. Beam

balances provided precise mass measurements and spatulas facilitated the transfer of solid samples. Standard laboratory containers, including test tubes, petri dishes and reagent bottles, were used for sample handling, incubation and observation. All equipment and reagents were selected based on their suitability for high-precision environmental analysis, ensuring the reliability and reproducibility of experimental outcomes.

### 3.2 Batch Adsorptions Experiment

Batch adsorption experiments were performed to evaluate the efficiency and optimal conditions of the prepared biochar for progesterone and BPA removal. The biochar which demonstrated high removal of EDCs, was subsequently characterised and further investigated through additional experiments. During the experiment, the biochar of banana and mango made under 300, 550, 700 and 800°C pyrolysis temperatures with different weights (0.10- 0.60 g) were mixed with 50 mL of a progesterone and BPA solution with a concentration of 12.50, 25 and 50 mg L<sup>-1</sup> independently in Erlenmeyer flasks. The progesterone solution was prepared in a 20:80 v/v mixture of methanol and water, while a 50:50 v/v mixture of methanol and water was used for BPA.

The experimental test was conducted under constant agitation at 140 rpm in an orbital incubator for 10, 30, 45, 60 and 120 minutes at an adsorption temperature of 25°C. The experiment was conducted at a pH range of 2–10, regulated by 0.20 M HCl and 0.20 M NaOH for progesterone and 0.20 M NH<sub>4</sub>OH for BPA. Then each sample was filtered by the Whatman filter 125 mmØ, and the filtrates were stored in plastic bottles at 5°C until HPLC hormone quantification. The adsorption efficiency ( $A_e$ ) of the biochar was determined by using Equation 1, while the adsorption capacity at a specific time ( $q_t$ ) was analysed using Equation 2 and the adsorption capacity at equilibrium ( $q_e$ ) was determined using Equation 3 (Vieira *et al.*, 2022; Wang *et al.*, 2023). The adsorption efficiency of the biosorbent was expressed in Equation 1.

$$A_e = \frac{C_o - C_e}{C_o} \times 100 \quad 1$$

Adsorption capacity at a specific time expressed in Equation 2:

$$q_t = \frac{(C_o - C_t) \times V}{m} \quad 2$$

The adsorption capacity of biosorbent at equilibrium shown in Equation 3:

$$q_e = \frac{(C_o - C_e) \times V}{m} \quad 3$$

where  $A_e$  is adsorption efficiency,  $C_o$  is an initial concentration of progesterone and BPA ( $\text{mgL}^{-1}$ ),  $C_e$  is an equilibrium concentration of progesterone and BPA ( $\text{mgL}^{-1}$ ),  $q_t$  is adsorption capacity at a specific time ( $\text{mg g}^{-1}$ ),  $q_e$  is adsorption capacity of biosorbent at equilibrium ( $\text{mg g}^{-1}$ ),  $m$  is mass of biosorbent (g),  $V$  is the volume of progesterone and BPA (mL).

### 3.3 Biosorbent Physiochemical Parameters

Physiochemical parameters were analysed to determine the properties of biochar. These properties involved carbon yield, total ash, moisture, biosorbent pH, point of zero charges, volatile matter and fixed carbon. Furthermore, the background concentration of BPA and progesterone was analysed to see if the biosorbent had been contaminated before conducting adsorption experiment.

#### 3.3.1 Ash Content

To determine ash content, 10 g of dried banana, mango and mixed (banana and mango peels) were placed into the different weighed crucibles and introduced into a furnace subjected to  $600^\circ\text{C}$  with a heating rate of  $12^\circ\text{C min}^{-1}$  for 3 hours. Then, the heated samples were removed, cooled in a desiccator and re-weighed. Thus the ash content was determined using Equation 4 (Özyuğuran & Yaman, 2017).

$$A_c = \frac{w_3 - w_1}{w_2 - w_1} \quad (4)$$

Whereby,  $A_c$  is ash content,  $w_1$  is the weight of the crucible,  $w_2$  is the weight of the crucible with samples, and  $w_3$  is the weight of the crucible and samples after being subjected to  $600^\circ\text{C}$  temperature.

#### 3.3.2 Moisture Content

In the determination of moisture content, 2.20 g of sample was introduced into a crucible, weighed and placed into the oven set at  $115^\circ\text{C}$  for 10 minutes. Then, the sample was removed and reweighed again using an analytical balance at 10-minute intervals until a constant weight was achieved. The moisture content was calculated using Equation 5 (Novak *et al.*, 2012):

$$M_c = \frac{w_3 - w_1}{w_2 - w_1} \quad (5)$$

Whereby,  $M_c$  is the moisture content,  $w_1$  is the weight of the crucible,  $w_2$  is the weight of the sample before being subjected to the oven, and  $w_3$  is the weight of the sample after oven drying.

### 3.3.3 Volatile Matter Determination

For the volatile matter determination, 2.20 g of the dried sample was placed in a crucible and heated at 900°C in the furnace for 10 minutes. The sample was then re-weighted after heating. The weight loss and volatile matter were analysed using Equation 6 and 7, respectively (Zhang *et al.*, 2017):

$$W_l = \frac{m_i - m_f}{m_i} \quad (6)$$

$$V_m = W_l - M_c \quad (7)$$

Whereby,  $W_l$  is weight loss,  $m_i$  is the initial weight of the sample,  $m_f$  is the final weight of the sample, and  $M_c$  is the moisture content determined using Equation 5.

### 3.3.4 Carbon Yield and Fixed Carbon Analysis

To determine the carbon yield and fixed carbon contents, 3 g of biosorbent powder was introduced into the weighed crucible; the weight of the crucible and sample was measured and introduced into a tube furnace, then heated at 850°C, at the heating rate of 12°C min<sup>-1</sup> maintained at 10°C to allow carbonization. After the retention time, the furnace was turned off, and then the crucible with biochar was removed and cooled in a desiccator. Then, it was weighed to get the difference. The carbon yield was calculated using Equation 8 (Weber & Quicker, 2018):

$$C_Y = \frac{M_a}{M_b} \times 100 \quad (8)$$

Whereby,  $C_Y$  is carbon yield,  $M_a$  is the mass of carbon after being removed from the furnace, and  $M_b$  is the mass of biomass before furnace processes. Furthermore, fixed carbon was determined by Equation 9:

$$F_C = 100 - M_c + A_c + V_M \quad (9)$$

Whereby,  $M_c$  is the moisture content,  $A_c$  is the ash content and  $V_m$  is the volatile matter.

### 3.3.5 pH and pH of Point of Zero Charge ( $pH_{pzc}$ ) Determination

To determine the biosorbent pH, 0.50 g of B700, M700 and HB700 was dissolved separately in 50 mL of distilled water and stirred at 140 rpm in an orbital shaker for 24 hours at 25°C. The sample was measured via a pH meter. For the  $pH_{pzc}$ , 0.50 g of biochar was added to 50 mL of distilled water at different initial pH levels (2-12), and the pH was regulated using 0.20 M NaOH and 0.20 M HCl. The pH was measured again to get the difference. Then, the  $pH_{pzc}$  of the biosorbent was determined as the intersection of the plot between the change in pH. The  $pH_{pzc}$  was calculated using Equation 10 (Vieira *et al.*, 2022):

$$\Delta pH = pH_i - pH_f \quad (10)$$

Whereby,  $pH_i$  is the initial pH,  $pH_f$  is the final pH and  $\Delta pH$  is the change in pH.

### 3.3.6 Progesterone and Bisphenol-A Background Contamination Analysis

For the BPA and progesterone background determinations, 0.50 g of B700, M700 and HB700 were dissolved separately in 50 mL of 50:50 v/v water and methanol to prepare a suspension. The solution was agitated in an orbital incubator at 25°C, 140 rpm for 24 hours to allow potential leaching of any residual organic compounds from the biochar. The mixture was then filtered to remove solid particles. The filtered solution was analysed using HPLC to determine if any organic compounds, such as naturally occurring plant-derived phenolic or progesterone residual carbon compounds, leached from the biochar. This step ensures that biochar does not introduce interfering substances that could affect either progesterone or BPA concentration.

### 3.3.7 Characterisation of Biosorbent Materials

Material characterisation was performed at the University of the Witwatersrand laboratory in Johannesburg, South Africa. Scanning Electron Microscopy coupled with Energy Dispersive Spectroscopy (SEM/EDS) (Model JSM-IT200A, JEOL Ltd., Tokyo, Japan) was employed to examine surface morphology and elemental distribution. The Brunauer–Emmett–Teller (BET) (Nova 1200e series Quantachrome) method was used to determine specific surface area, while X-ray Diffraction (XRD) analysis assessed the crystallinity and mineralogical composition of the biochar samples.

Elemental analysis for Carbon (C), Nitrogen (N) and Hydrogen (H) was conducted using an organic elemental analyser (Model Flash 2000, Thermo Fisher Scientific, Bremen, Germany). Additionally, Fourier Transform Infrared Spectroscopy (FTIR) (Model IRTracer-100, Shimadzu Corporation, Kyoto, Japan) was utilised to identify surface functional groups, which was carried out at the NM-AIST laboratory.

### **3.3.8 Wastewater Characterisation**

The wastewater samples were collected from Terat Waste Stabilisation Ponds (WSP) found in Arusha city, Tanzania. Composite wastewater samples were collected in pre-cleaned amber bottles and analysed for physicochemical parameters before spiking, after spiking with BPA and progesterone and after biosorption treatment. Field parameters (temperature, pH, dissolved oxygen and electrical conductivity) were measured in situ using a calibrated Multiparameter probe. Then, water samples in glass bottles were adjusted to pH 3 with HCl and stored in a cool box packed with ice packs at 4°C to prevent microbial degradation before being transported to the TARI Uyole laboratory for chemical analysis.

At the lab, the samples were filtered through 0.45 µm membranes where required and analysed in triplicate. Total solids (TS) and total suspended solids (TSS) were determined gravimetrically. The COD was measured using a HI-839800 thermo-reactor, while BOD<sub>5</sub> was analysed using an OxiTop Box 140601 system. Nutrients (Nitrate, Nitrite and Phosphate) were quantified using a Unico 2800 UV–Vis spectrophotometer with standard colorimetric methods and Ca<sup>2+</sup> and K<sup>+</sup> were determined using flame atomic absorption spectrophotometry. All instruments were calibrated prior to analysis, and blanks, standards and duplicates were included for quality control. Results were reported as mean ± standard deviation of triplicate measurements, and removal efficiencies after adsorption were calculated based on concentration differences before and after treatment.

### **3.3.9 Solid Phase Extraction and High Performance Liquid Chromatography Analysis**

Before SPE, the samples were filtered twice using GF/C and a 0.45 µm membrane filter to remove suspended solids. After filtration, SPE was performed according to the protocol of Kasambala *et al.* (2019), followed by some modifications made in the lab. Water samples were thawed at room temperature before starting extraction. The SPE used C-18 cartridges (500 mg, 6 mL) with a vacuum manifold pump. The cartridges were conditioned with 2 × 3 mL of n-heptane, 3 mL of acetone and 3 mL of distilled water, adjusted to pH 3, to remove impurities.

Samples were loaded into the system, supported by the Manford pump at 6 mL min<sup>-1</sup> and trapped on a glass fibre filter.

The cartridges were washed with 1 mL of methanol at 2 mL min<sup>-1</sup> to remove hydrophobic substances. After extraction, cartridges were dried in air using a vacuum manifold for about 30 minutes, then eluted with a mixture of 10 mL heptane and acetone (70:30). The eluates were dried in air at 30°C, then reconstituted in the HPLC mobile phase prior to analysis, improving detection sensitivity and minimising matrix effects during chromatographic quantification.

### **3.4 Effect of Various Factors on Pollutant Adsorption**

#### **3.4.1 Effect of the Initial pH of the Solution**

To examine the effect of pH on pollutant adsorption, 12.50 mg L<sup>-1</sup> solutions were prepared at pH 2, 4, 6, 8 and 10. The pH was adjusted using 0.20 M NaOH and 0.20 M HCl for progesterone, while 0.20 M HCl and 0.20 M NH<sub>4</sub>OH were used for BPA pH adjustments. Each solution was mixed with 0.50 g of HB700 biochar, then agitated in an orbital incubator at 140 rpm and 25°C for 60 min for progesterone and 120 min for BPA. Following adsorption, the samples were filtered and analysed via HPLC. Then, adsorption efficiency was determined using Equation 1.

#### **3.4.2 Hybrid Biochar Dosage on Pollutant Adsorption**

To evaluate the impact of adsorbent dosage on progesterone and BPA removal, biochar at varying doses (0.10, 0.20, 0.30, 0.40, 0.50 and 0.60 g) was added to 50 mL of an aqueous solution containing 12.50 mg L<sup>-1</sup> of the target compounds, maintained at a pH of 8. The mixture was agitated at 140 rpm and 25°C for 60 min for progesterone and 120 min for BPA to facilitate adsorption. After reaching equilibrium, the samples were filtered and residual concentrations were quantified using HPLC. Then, adsorption efficiency was determined using Equation 1.

#### **3.4.3 Adsorbate Dosage on Pollutant Adsorption**

The effect of adsorbate dosage was investigated to assess its impact on adsorption efficiency. During the adsorption experiment, 0.50 g of HB was mixed with 300, 250, 200, 150, 100, 50, 25 and 12.50 mg L<sup>-1</sup> of progesterone and BPA solutions individually and shaken in an orbital incubator for 60 min for progesterone and 120 min for BPA at 25°C and 140 rpm. Then, the sample was filtered and stored at 5°C until HPLC analysis. Removal efficiency was calculated to evaluate the effect of adsorbate dosage using Equation 1.

### 3.5 Hybrid Biochar Modelling Analysis

#### 3.5.1 Kinetic Experiments

During the adsorption kinetics experiment, 0.50 g of HB was dissolved in 50 mL of a progesterone hormone and BPA solution (12.50 mg L<sup>-1</sup>) separately under optimal pH conditions. Subsequently, this mixture was introduced into an orbital incubator and stirred at 140 rpm for 720 min at a temperature of 25°C. At specific time intervals of 10, 20, 30, 45, 60, 120, 180, 240, 360 and 720 min, samples were withdrawn from each Erlenmeyer flask using a clinical syringe and filtered. Then the filtrates were analysed using HPLC analysis. The method used in this study was modified from Vieira *et al.* (2022) for progesterone and Lee *et al.* (2021) for BPA. The calculation was performed using Equation 1 for adsorption efficiency and Equation 11, proposed by Amusat *et al.* (2023) for pseudo-first-order kinetics. Moreover, Equation 12, proposed by Ho and McKay (1999) was used to calculate second order kinetics.

$$q_e = Q_m \frac{K_L \times C_e}{1 + K_L \times C_e} \quad (11)$$

$$q_e = K_F \times C_e^{1/n} \quad (12)$$

Whereby,  $q_e$  (mg g<sup>-1</sup>) is the adsorption capacity of biochar at equilibrium,  $Q_m$  (mg g<sup>-1</sup>) is the theoretical maximum adsorption capacity by fitting,  $C_e$  (mg L<sup>-1</sup>) is the equilibrium aqueous concentration,  $K_L$  (L mg<sup>-1</sup>) is the Langmuir constant,  $K_F$  [(mg g<sup>-1</sup>) (L mg<sup>-1</sup>)<sup>1/n</sup>] and  $1/n$  are Freundlich constants that represent the adsorption density and intensity, respectively.

#### 3.5.2 Adsorption Thermodynamic

Temperature plays a more significant role in adsorption processes. The adsorption capacity was analysed by taking 0.50 g of biosorbent and 12.5 mgL<sup>-1</sup> of adsorbate at pH 8 for 120 min for BPA and 60 min for progesterone at 298K, 308K, 318K and 328K experiment temperatures. The change of enthalpy ( $\Delta H^\circ$ , kJ mol<sup>-1</sup>, Gibbs free energy ( $\Delta G^\circ$ , kJ mol<sup>-1</sup>) and entropy ( $\Delta S^\circ$ , kJ mol<sup>-1</sup>·K<sup>-1</sup>) were considered as the basic thermodynamics properties and were calculated using Equations (13-16) (Vieira *et al.*, 2022). Thermodynamic constant expressed in Equation 13.

$$K_d = \frac{q_e}{C_e} \quad (13)$$

$$K_c = 1000 \times K_d \quad (14)$$

$$\Delta G^o = -RT \ln K_c \quad (15)$$

$$\ln K_c = \frac{\Delta S^o}{R} - \frac{\Delta H^o}{R} \times \frac{1}{T} \quad (16)$$

Whereby,  $K_d$  – distribution coefficient ( $Lg^{-1}$ ),  $K_c$  is the thermodynamic constant (dimensionless),  $R$  is the universal gas constant ( $8.314 Jmol^{-1} \cdot K^{-1}$ ),  $T$  is the temperature (K),  $q_e$  ( $mg g^{-1}$ ) is the amount adsorbed on solid at equilibrium while  $C_e$  is the concentration of progesterone and BPA at equilibrium ( $mg L^{-1}$ ).

### 3.5.3 Adsorption Isotherms

An adsorption isotherm was conducted to assess the efficiency of biochar in absorbing EDCs at a specific contaminant concentration. During the adsorption isotherm experiment, 0.50 g HB was mixed with 300, 250, 200, 150, 100, 50, 25 and 12.5  $mg L^{-1}$  of progesterone hormone solution and BPA separately and shaken in an orbital incubator for 60 min for progesterone and 20 min for BPA at 25°C and 140 rpm. The solution was regulated to pH 8 using 0.20 M NaOH and 0.20 HCl for progesterone and 0.20 HCL and 0.20 for  $NH_4OH$ . Then, the sample was filtered before HPLC analysis. The Freundlich and Langmuir models and non-dimension separation factor fit the isotherm adsorption curves using Equation 18-20 (Hoslett *et al.*, 2020; Farias *et al.*, 2023). While Dubinin-Radushkevich (D-R) and Temkin isotherm models were fit using Equation 21 and 22 (Chen, 2015; Lu & Na, 2022).

Langmuir adsorption isotherm model

$$q_e = q_m \frac{K_L \times C_e}{1 + K_L \times C_e} \quad (18)$$

Freundlich adsorption isotherm model

$$q_e = K_F \times C_e^{1/n} \quad (19)$$

Non-dimensional separation factor

$$R_L = \frac{1}{1 + K_L \times C_i} \quad (20)$$

$$\text{Dubinin-Radushkevich (D-R)} \quad q_e = q_s \exp^{K_{DR}\epsilon^2} \quad (21)$$

$$\text{Temkin} \quad q_e = \frac{RT}{b} \ln AC_e \quad (22)$$

Whereby,  $q_e$  ( $\text{mg g}^{-1}$ ) is the adsorption capacity of biochar at equilibrium,  $q_m$  ( $\text{mg g}^{-1}$ ) is the theoretical maximum adsorption capacity by fitting,  $C_e$  ( $\text{mg L}^{-1}$ ) is the equilibrium aqueous concentration,  $K_L$  ( $\text{L mg}^{-1}$ ) is the Langmuir constant,  $K_F$  [ $(\text{mg g}^{-1}) (\text{L mg}^{-1})^{1/n}$ ] and  $1/n$  are Freundlich constants that represent the adsorption density and intensity, respectively. The  $C_i$  ( $\text{mg L}^{-1}$ ) is the initial concentration of adsorbate, while  $R_L$  is the dimensionless constant for the separation factor. The  $K_{DR}$  is the D-R constant ( $\text{mol}^2/\text{kJ}^2$ ),  $q_s$  ( $\text{mg P g}^{-1}$ ) is a constant in the D-R isotherm model which are related to adsorption capacity,  $\epsilon$  is the Polanyi potential while  $b$  and  $a$  are Temkin's constant,  $T$  is the absolute temperature (K) and  $R$  is the gas constant ( $\text{J K}^{-1} \text{mol}^{-1}$ ).

### 3.6 Effect of Co-existence of Bisphenol-A and Progesterone in Wastewater

The co-existing BPA and progesterone effect in synthetic wastewater was investigated to determine how their co-occurrence affects adsorbent efficiency. To experiment, 50 mL of a solution containing 12.50 mg of BPA and progesterone was prepared in 1 L of deionized water. This solution was mixed with 0.50 g of adsorbent in a 250 mL conical flask and agitated at 140 rpm at 25°C using an orbital shaker for 60 min for progesterone and 120 min for BPA, respectively. The filtrate was then analysed via HPLC. For real wastewater, the same amount of each pollutant (12.50 mg) was spiked into 1 L, following the same procedures as for synthetic wastewater.

### 3.7 Regeneration and Reusability of Biochar Analysis

Biochar was regenerated by dissolving 12.50  $\text{mg L}^{-1}$  of EDCs with 0.50 g of biochar at pH 8, stirred for 60 and 120 min for progesterone and BPA at 25°C with 140 rpm. Then, the filtration was followed by determining pollutants from the liquid phase. Then, the progesterone and BPA remaining attached to the biochar were washed with the mixture of methanol and distilled water (70:30 v/v), then filtered and the filtrate was analysed for EDCs desorption. The remaining biosorbent was oven-dried at 110°C for 60 min until a constant weight was attained. Then, the dried usable biosorbent was introduced into 50 mL of a known concentration of EDCs under

all other conditions. Regeneration and reusability were tested seven times for each. Then, all the filtrate was analysed for BPA and progesterone remaining using HPLC and removal efficiency was calculated using Equation 1.

### **3.8 Analytical Quality Assurance**

#### **3.8.1 Quality Control Assurance**

To ensure the quality of the experiments and the data obtained, all apparatus was rinsed twice using distilled water before and after each experiment. Each experimental run was conducted in triplicate to ensure the reproducibility of the results. Before the experiment, B700, M700 and HB700 biochar were analysed for background pollutant contamination. To ensure accurate sample measurement, the analytical balance was calibrated frequently. Furthermore, for the accuracy of pH determination, the pH meter was calibrated after each experimental run. Protective gear was worn throughout to prevent contamination during experimental procedures, with proper labelling. To prevent decomposition of the adsorbate, the stock solution and filtrate after adsorption for progesterone were stored at 5°C and analysed within five days. A bisphenol A stock solution was prepared during the experiment section and its filtrate was analysed using HPLC daily.

#### **3.8.2 Instrumental Analysis**

High-performance liquid chromatography (HPLC) with a UV detector from Agilent Technologies 1200 Series instruments (Santa Clara, CA, USA) was used to quantify progesterone EDCs. The HPLC-UV was equipped with a C18 reverse column (3.50 × 150 mm and 3.50 × 4.60 × 150 mm) from Agilent Technologies, located in Santa Clara, CA, USA to detect progesterone and BPA. The mobile phase consisted of acetone and water (90:10 v/v), and the detector wavelengths were 244 nm for progesterone and 214 nm for BPA. Also, the retention times were 2.56 min and 2.50 min for progesterone and BPA, respectively. A column temperature of 40°C, an injection volume of 5.00 µm and a flow rate of 1 mm/min were used.

To verify the accuracy of the equipment and reagents, we measured samples against an external calibration curve using 12.5 mg L<sup>-1</sup> of progesterone prepared in an 80:20 (v/v) mixture of distilled water and methanol, after serial dilution to five distinct concentrations (0.27, 0.14, 0.07, 0.03 and 0.02 mg mL<sup>-1</sup>). For BPA, a serial dilution was performed with concentrations of 0.012, 0.006, 0.03, 0.002 and 0.001 mg mL<sup>-1</sup>. By conducting these validation procedures, it could be confidently assured of the precision and reliability of the analytical processes.

### **3.8.3 Statistical Quality Assurance**

One-way ANOVA, Tukey's post hoc test and an independent t-test using GenStat 12<sup>th</sup> edition software was used to evaluate removal efficiency and variation in adsorption capacity. A significant difference was established at a 95% confidence level, where the  $p$ -value  $<0.05$  was considered statistically significant. The study used the OriginPro 2024 package and Microsoft Excel for data organisation and figure manipulation.

## CHAPTER FOUR

### RESULTS AND DISCUSSION

#### 4.1 Overview

This Chapter presents and discusses the key findings of the study in alignment with the research objectives: To evaluate the effectiveness of banana and mango peels biochar in isolated components for the removal of progesterone and BPA; to investigate the efficiency of mango and banana peels in a hybrid form in removing progesterone and BPA from wastewater and lastly to test the competitive removal effect on co-existing BPA and progesterone in synthetic and municipal wastewater by hybrid biochar. The results obtained from the experimental analysis were interpreted to provide a deeper understanding of the adsorption behaviour and overall performance of the hybrid biosorbent. The data were organized and presented through tables, figures and graphs to enhance clarity and understanding. Furthermore, the discussion compares these findings with existing literature, highlighting both agreements and discrepancies, as well as the broader significance of the results. This thorough analysis supports the development of well-informed conclusions and practical recommendations.

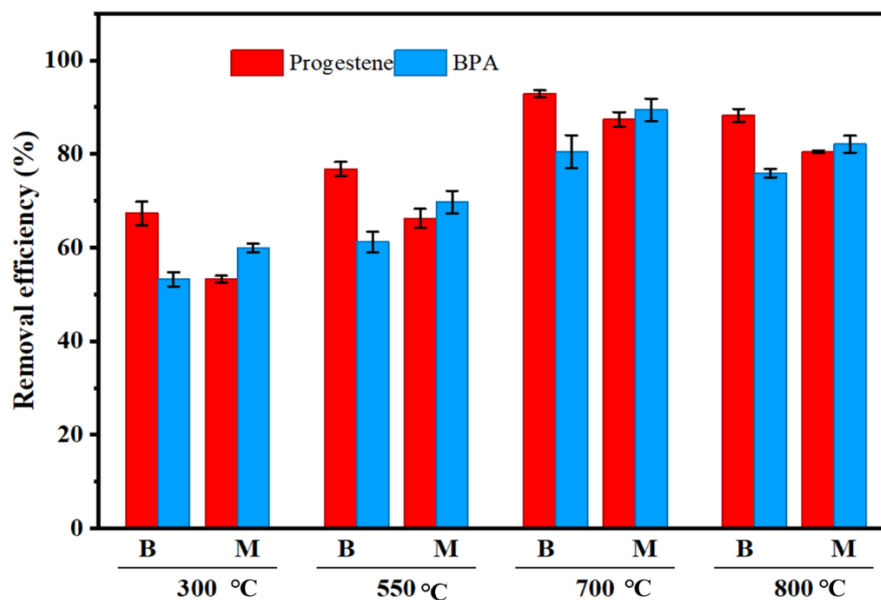
#### 4.2 Effectiveness of Banana and Mango Peels Biochar in Isolated Components for the Removal of Progesterone and Bisphenol A

##### 4.2.1 Batch Adsorption Experiment for Banana Peels Biochar and Mango Peels Biochar in Isolated Form

Adsorption experimental results demonstrated that biochar produced at a temperature of 700°C exhibited the highest efficiency in adsorption of progesterone and BPA from wastewater (Fig. 4). Subsequently, biochar produced at 800, 550 and 300°C was also effective in removing EDCs from wastewater. However, its effectiveness in removing progesterone and BPA was lower compared to the biochar produced at 700°C. This is because at lower pyrolysis temperatures (<400°C), the biochar has a lower surface area and poorly developed porosity, which hinders its efficiency to adsorb pollutants.

On the other hand, in medium pyrolysis temperatures (700°C), the biochar is characterized by higher carbon content, higher surface area, more developed micro- and mesoporous structure and optimal balance of surface functional groups, which make it vital for diverse hydrophobic pollutants adsorption, including EDCs (Muoghalu *et al.*, 2023). The biochar is more

hydrophobic at very high temperatures ( $\geq 800^{\circ}\text{C}$ ) due to the loss of oxygen and hydrogen atoms from functional groups. This increased hydrophobicity can hinder the adsorption of hydrophilic compounds, such as BPA or moderately hydrophobic compounds, like progesterone (Li *et al.*, 2023).



**Figure 4: Adsorption efficiency of BPB and MPB at different pyrolysis temperatures**

Additionally, the results showed that BPB and MPB exhibited higher removal efficiencies,  $92.80 \pm 0.78\%$  and  $87.93 \pm 8.70\%$  for progesterone; and  $80.50 \pm 2.20\%$  and  $90.40 \pm 2.40\%$ , for BPA. The results were obtained under optimal experimental conditions, including a biosorbent dose of 0.50 g, a contact time of 60 and 120 min for progesterone and BPA, respectively, a pH of 8 and a biosorbate dosage of  $12.50 \text{ mg g}^{-1}$ . This suggests that BPB may have better adsorption for progesterone, while MPB performs better for BPA removal. These results agree with other researchers. For instance, Achak *et al.* (2009) noted that the efficiency of BPB in adsorbing phenolic compounds is considerably greater than that found in this study.

In contrast, the efficiency of BPB and MPB in this study is higher than that reported for the shell of star apple, with 52% and 56% for BPA and progesterone, respectively, as studied by Amusat *et al.* (2023). However, this is lower than the removal efficiency of modified prickly pear, which showed 94% and 95% for BPA and progesterone, respectively (Amusat *et al.*, 2023). This can be attributed to aromaticity, a high initial pH, a high O: C ratio and a lower H: C ratio, which facilitate  $\pi$ - $\pi$  interactions, electrostatic complexation and functional group complexation between EDCs and the biosorbent surface. Therefore, optimized conditions create a synergistic environment for the adsorption of diverse pollutants. Thus, all experiments and characterizations utilize a biosorbent with a pyrolysis temperature of  $700^{\circ}\text{C}$ , a contact time

of 60 and 120 min for progesterone and BPA respectively, a pH of 8, a rotation per minute (rpm) of 140, a biosorbent dose of 0.50 g, a biosorbate dosage of 12.50 mgL<sup>-1</sup> and a temperature of 25°C, as these are optimal conditions for adsorbing the selected compound.

#### 4.2.2 Physicochemical Parameters of Banana Peels Biochar and Mango Peels Biochar

##### (i) Ash Content

Table 3 indicates the results for physicochemical parameters of BPB and MPB. The ash content of 7.80% and 8.60% in BPB and MPB, respectively, as observed in this study, suggests that the biochar contains a moderate concentration of inorganic elements, of which less than 10% are required for the adsorption of organic compounds.

**Table 3: Physicochemical parameters for BPB and MPB under 700°C pyrolysis temperature**

Parameters	BPB	MPB	% Required
Ash content%	7.80 ± 1.30	8.60 ± 0.21	< 10%
Moisture content%	1.50 ± 0.51	0.38 ± 0.83	< 10%
Carbon yield%	33.04 ± 0.80	23.92 ± 0.05	(20-35%)
Fixed carbon%	71.10±0.20	80.12±1.31	(60-90%)
Volatile matter%	19.19±0.02	10.91±0.12	< 20%
pH	10.50 ± 1.03	8.80 ± 1.52	Nil
pH <sub>pzc</sub>	7.20	10.90	Nil
Progesterone concentration	Nil	Nil	Nil
BPA concentration	Nil	Nil	Nil

Generally, the ash content of biochar can vary significantly depending on the feedstock used for its production (Taherymoosavi *et al.*, 2018). High ash content favour the adsorption of ions due to increased electrostatic sites, but could hinder the adsorption of organic compounds if it reduces the accessible carbon surfaces (Manzoor *et al.*, 2024). Nevertheless, the difference between MPB and BPB could be due to differences in either the chemical composition or moisture levels between mango and banana peels before pyrolysis.

##### (ii) Moisture Content

The total moisture content of 1.50 ± 0.51% and 0.38 ± 0.83% for BPB and MPB, respectively, was observed in this study. According to Chen *et al.* (2022), a high moisture content of more than 20% hinders the adsorption of hydrophobic organic compounds due to the increased polarity of the biochar surface. Furthermore, excessive moisture in biochar hinders the adsorption of pollutants by blocking pores and active sites available for adsorption (Ahmad *et al.*, 2020). Since the moisture content significantly influences the adsorption capacity of

biochar, it affects both the physical and chemical processes involved in the adsorption process. Therefore, the low moisture content from this study makes the BPB and MPB pronounced biosorbents for the removal of EDCs.

### **(iii) Volatile Matter**

The biosorbent volatile matter found in this study is less than 20% (Table 3). These are favourable for the adsorption of hydrophobic pollutants such as progesterone and BPA. Furthermore, the highest volatile matter observed by BPB indicates the presence of significant amounts of organic compounds that did not fully decompose (Ahmad *et al.*, 2020), potentially leading to enhanced reactivity and instability. High volatile matter content can increase the surface functionality of biochar due to the presence of various organic compounds containing functional groups, such as -OH, C-O and C=O (Ghidotti *et al.*, 2017) as evidenced in BPB by using FTIR characterization (Fig. 9a). These groups can enhance adsorption through hydrogen bonding, electrostatic interactions, and complexation. However, BPB is suspected to be more prone to degradation, potentially releasing adsorbed pollutants over time due to the presence of highly volatile matter.

### **(iv) Carbon Yield**

The carbon yields from this study showed in Table 3 are within the range with higher yields in banana peels biochar and lower yields in mango peels biochar. The carbon yield of BPB and MPB is a critical parameter that reflects the efficiency of converting organic materials into stable carbon forms. Generally, the carbon yield for agricultural feedstock biochar is 20-35% (Agu *et al.*, 2022). Higher carbon yields often enhance physical and chemical properties of biosorbent for pollutant adsorption. High carbon yield results into biochar with more surface areas and porosity due for the retention of more organic matter and thus fits for the adsorption of diverse pollutants (Dai *et al.*, 2019). This relatively high carbon yield suggests that banana peels are an efficient feedstock for producing stable, long-lasting, carbon-rich materials through pyrolysis. The high carbon yield in banana peels may be attributed to high lignin content (9.82 wt.%) compared to mango peels (4.42 wt%) (Orozco *et al.*, 2014).

### **(v) Fixed Carbon**

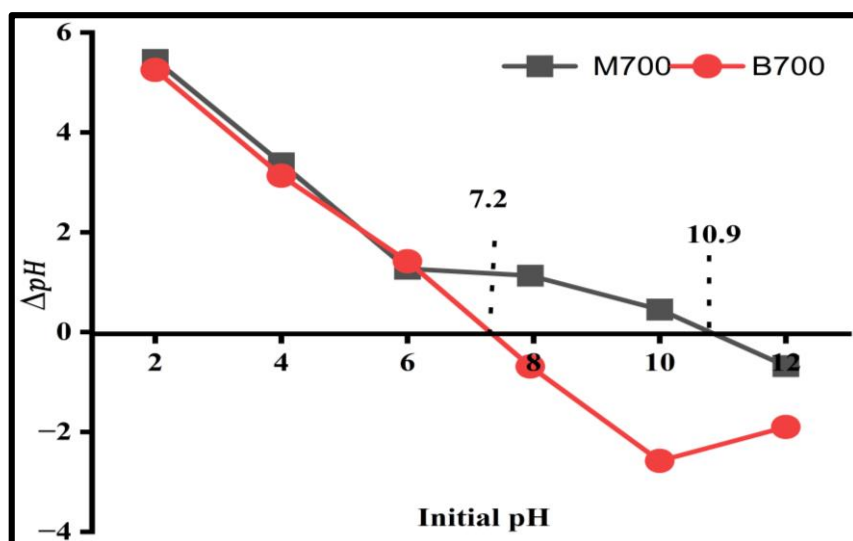
The fixed carbon of BPB and MPB is  $71.10 \pm 0.20\%$  and  $80.12 \pm 1.31\%$ , respectively, which are within the range (60-90%) (Agu *et al.*, 2022). The fixed carbon content is a crucial factor in determining the adsorption efficiency of biochar for organic pollutants, such as progesterone

and BPA. The higher fixed carbon content in both biochars of this study indicates greater thermal stability, a higher surface area and enhanced adsorption capacity (Gao *et al.*, 2024). In addition, the higher fixed carbon content in both biochars was aligned with better adsorption of progesterone and BPA, due to increased surface area and porosity.

**(vi) pH and pH of Point of Zero Charge ( $\text{pH}_{\text{pzc}}$ )**

In this study, both biochars exhibited high pH levels (Table 3), with the highest pH level observed in BPB compared to MPB, indicating that basic functional groups predominate in BPB. The pH of biochar plays a fundamental role in influencing the surface charge and ionization of the functional groups of the biosorbent surface and pollutants. However, progesterone, with a  $\text{p}K_{\text{a}}$  of  $\sim 13.00$ , is a neutral molecule at environmental pH (De Graaff & Grimard, 2018), so its adsorption is likely driven by hydrophobic interactions rather than electrostatic attraction. Likewise, BPA of  $\text{p}K_{\text{a}} \sim 9.60$  at pH 8.90 exists in both neutral and negatively charged forms (Qi *et al.*, 2023). The extreme alkalinity of BPB may cause significant electrostatic repulsion with BPA while favouring a neutral condition in progesterone.

Furthermore, the analysis of  $\text{pH}_{\text{pzc}}$  was conducted to determine whether the adsorbent surface is positively or negatively charged under various pH conditions. The study found that the  $\text{pH}_{\text{pzc}}$  values for BPB and MPB were 7.20 and 10.90, respectively (Fig. 5).



**Figure 5: The pH of point zero charges ( $\text{pH}_{\text{pzc}}$ ) for BPB and MPB**

In the case of MPB, where by the  $\text{pH}_{\text{pzc}} > \text{pH}$ , the biosorbent surface was positively charged, facilitating the adsorption of negatively charged species, such as bisphenolate ions. Bisphenol A, which exists as negatively and positively charged species around a pH of 8.01 (Wang *et al.*,

2019), is expected to exhibit high adsorption efficiency, as shown in Fig. 3. Conversely, progesterone, being a neutral compound at higher pH levels, does not benefit from enhanced adsorption through electrostatic attraction on the positively charged surface of MPB, leading to a lower removal efficiency compared to BPA. For BPB, where  $\text{pH} > \text{pH}_{\text{pzc}}$ , the biosorbent surface becomes negatively charged. At higher pH levels, BPA, a weakly acidic compound that can exist in an anionic form, experiences electrostatic repulsion from the negatively charged surface, reducing its adsorption capacity. However, other mechanisms, such as hydrophobic interactions and  $\pi$ - $\pi$  electron donor-acceptor (EDA) interactions, may contribute to BPA adsorption (Wang *et al.*, 2019; Wang & Zhang, 2020).

#### **(vii) Progesterone and Bisphenol A Background Contamination**

Table 3 indicates that PBA and progesterone contaminants were absent in BPB and MPB. The absence of background contamination of BPA and progesterone in biosorbent provides insight into the usefulness of these in adsorption. According to Kubesa (2017), lack of EDCs contamination on biosorbent may be due to EDCs thermal degradation under high pyrolysis temperatures. Likewise, BPA and progesterone decompose into more minor volatile compounds or gases ( $\text{CO}_2$ ,  $\text{CO}$ ,  $\text{CH}_4$ ) due to the cracking of aromatic rings and oxidation of functional groups during pyrolysis (Kubesa, 2017). Furthermore, these BP and MP mainly contain cellulose, hemicellulose, lignin and minor organic acids, and they do not naturally produce BPA and progesterone (Grassi *et al.*, 2012). Additionally, before pyrolysis, banana and mango peels are often washed to remove dust, pesticides and potential external contaminants. Therefore, this step ensures that no BPA or progesterone contamination is introduced from environmental sources.

### **4.2.3 Characterization of Banana Peels Biochar and Mango Peels Biochar**

#### **(i) Elemental Composition**

The carbon, nitrogen, and hydrogen contents of biochar were analysed using an organic elemental analyser to provide insights into the potential functional groups on the biochar surface that can interact with pollutants. The results showed that the carbon content of both types of biochar increased with increasing pyrolysis temperature (Table 4). The highest carbon content was observed in MPB, indicating successful carbonization, which provides a larger surface area and more active sites for pollutants to bind through various mechanisms, including  $\pi$ - $\pi$  interactions, hydrogen bonding, and electrostatic attraction (Luo *et al.*, 2022).

Additionally, the entire biochar is characterized by reduced elemental N, H and O values after the calcination process (Table 4). This is due to dehydration and nitrogen volatilization, which remove H, O and N, thereby increasing the relative carbon content. Other studies also observed the same (Chatterjee *et al.*, 2020; De Oliveira-Paiva *et al.*, 2024). In contrast, other biochar behaves differently in terms of carbon content, such as biochar from sewage sludge (Regkouzas & Diamadopoulou, 2019).

**Table 4: Elemental and atomic ratio analysis for biosorbent before and after pyrolysis**

Biochar (g)	Elemental analysis				Atomic ratio			
	% N	% C	% H	% O	H/C	N/C	O/C	(N+O)/C
BP	1.45	35.71	6.17	57.67	0.15	0.04	1.32	1.46
MP	0.99	31.53	4.19	63.28	0.13	0.03	2.01	2.04
B <sub>700</sub>	1.17	55.45	1.56	34.02	0.01	0.02	0.61	1.17
M <sub>700</sub>	0.74	74.12	1.47	15.07	0.01	0.01	0.21	0.74

**(ii) Atomic Ratio of Element in Bisphenol-A and Mango Peels Biochar**

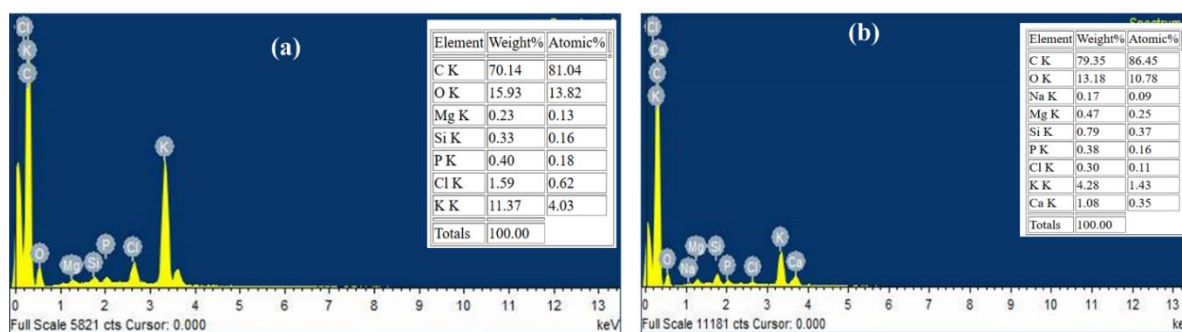
Atomic ratios of elements in biosorbent provide insight into the degree of carbonization, surface chemistry and adsorption mechanisms for organic pollutants. The N/C ratio indicates the availability of nitrogen-containing functional groups in the biochar. The relatively higher ratio in BPB exhibits a more pronounced presence of nitrogen-containing functional groups, which potentially increases its polarity and enhances the adsorption of polar compounds, such as BPA, through hydrogen bonding (Vieira *et al.*, 2022). However, the relatively small ratio in MPB indicates fewer nitrogen-containing functional groups and a less polar surface, which is effective for adsorbing non-polar compounds such as progesterone through hydrophobic interactions (Carnimeo *et al.*, 2023). However, this finding does not necessarily support that BPB has higher affinity for progesterone (neutral) than BPA (charged) because this is not the only factor that can explain the higher adsorption efficiency observed in this study.

Furthermore, H/C ratio is an important parameter that reflects the structural characteristics of biochar, influencing its adsorption properties. The lowest H/C ratio in BPB700 and MPB700 indicates a higher degree of carbonization. This leads to increased aromaticity, which improves the adsorption of hydrophobic compounds such as BPA through hydrophobic and  $\pi$ - $\pi$  stacking interactions (Wang & Zhang, 2020). Additionally, the lower O/C ratio in MPB indicates fewer oxygen-containing groups, resulting in a less polar and more hydrophobic surface. This characteristic is beneficial for adsorbing non-polar compounds, such as BPA, through hydrophobic interactions, which is also reflected in the adsorption efficiency trend (Fig. 4).

On the other hand, there is a high oxygen-carbon ratio (O/C) in BPB meaning it is more aromatic and has more oxygen-containing functional groups than MPB. Through hydrogen bonds and  $\pi$ - $\pi$  interactions, these groups can interact with the hydroxyl and carbonyl groups of progesterone and BPA, thereby facilitating enhanced adsorption efficiency (Ahmed *et al.*, 2023). The higher (N+O) C confirmed in BPB, which contributes to the biochar's adsorption capacity for pollutants (Cukierman *et al.*, 2019).

### (iii) Energy Dispersive X-Ray Spectroscopy Results

Figure 6 shows the EDS results to confirm the elemental composition of the BPB and MPB. It was found that the elemental compositions of C, O and K in all biosorbent were higher compared to other elements.



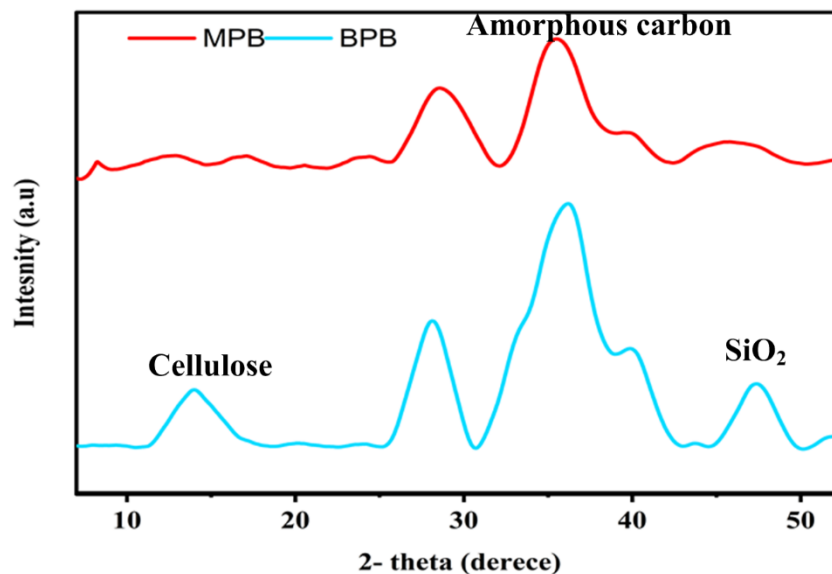
**Figure 6: Energy dispersive x-ray spectroscopy images for elementary composition for (a) BPB and (b) MPB focused on specific distinct areas**

Variations in the elemental compositions of biosorbents indicate variations in the organic nature of bananas and mangoes. On the other hand, elements such as Potassium (K), Calcium (Ca), Magnesium (Mg), Chlorine (Cl) and Silicon (Si) were observed in BPB and MPB. These elements contribute to improved structural stability, increased surface area, enhanced ion exchange capacity and favourable pH conditions for effective pollutants adsorption processes (Buss *et al.*, 2022). Furthermore, these elements enhance the adsorption capacity by improving the surface area, electrostatic attraction and cation exchange (Amusat *et al.*, 2023).

### (iv) X-ray Diffraction Analysis Results

X-ray diffraction provides insight into the crystalline and amorphous nature of the materials. Results show that MPB and BPB are amorphous due to the presence of broad scattering humps instead of sharp crystalline peaks (Bates, 2010). Amorphous biochar typically has a high surface area due to its irregular and non-crystalline structure, which provides many active sites for the adsorption of contaminants. Results for particular peaks observed at  $14^\circ$ ,  $27^\circ$ ,  $32^\circ$ ,  $35^\circ$

and 47° (Fig. 7) provide insight into the biochar's amorphous nature and mineralogical crystallinity composition (Areti *et al.*, 2024).

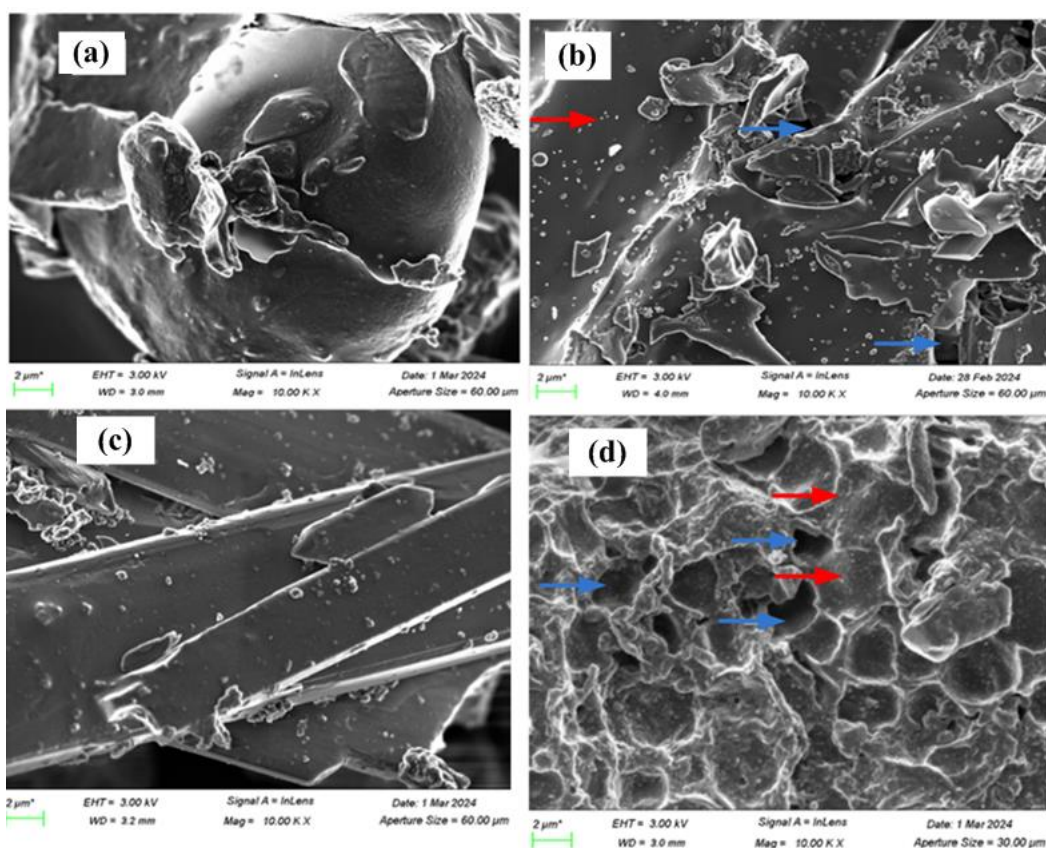


**Figure 7:** The XRD spectra for the BPB and MPB calcinated at 700°C

For instance, the broad peak at 35° is characteristic of amorphous carbon, indicating disordered carbon structures formed during pyrolysis. A broad peak indicates that the materials lack a well-ordered crystalline structure, which is common in biomass-derived biochar. Moreover, the 47° peak in BPB often corresponds to common minerals, such as silicate minerals (SiO<sub>2</sub>, quartz) and other minerals which may contribute to the structural stability of the biochar (Li *et al.*, 2019; Pokkiladathu *et al.*, 2022). Furthermore, the minerals present in the biochar may facilitate specific chemical interactions with progesterone, such as hydrogen bonding or Van der Waals forces. These interactions can enhance the binding affinity of BPA and progesterone to the biochar, improving removal efficiency. Likewise, an abnormal peak at 14° appeared in BPB, implying the availability of cellulose or hemicellulose due to incomplete pyrolysis.

#### (v) Scanning Electron Microscopy

The SEM micrographs were used to identify were used to describe the surface morphology of the biochar. From SEM, the biochar showed an irregular, porous, and rough surface structure with well-developed cavities and interconnected pores best in biochar than in powder (Fig. 8a, 8b, 8c, & 8d). This morphology is highly favourable for BPA and progesterone adsorption because it provides a large accessible surface area, more active sites and enhanced mass transfer of the molecules into the pore network.

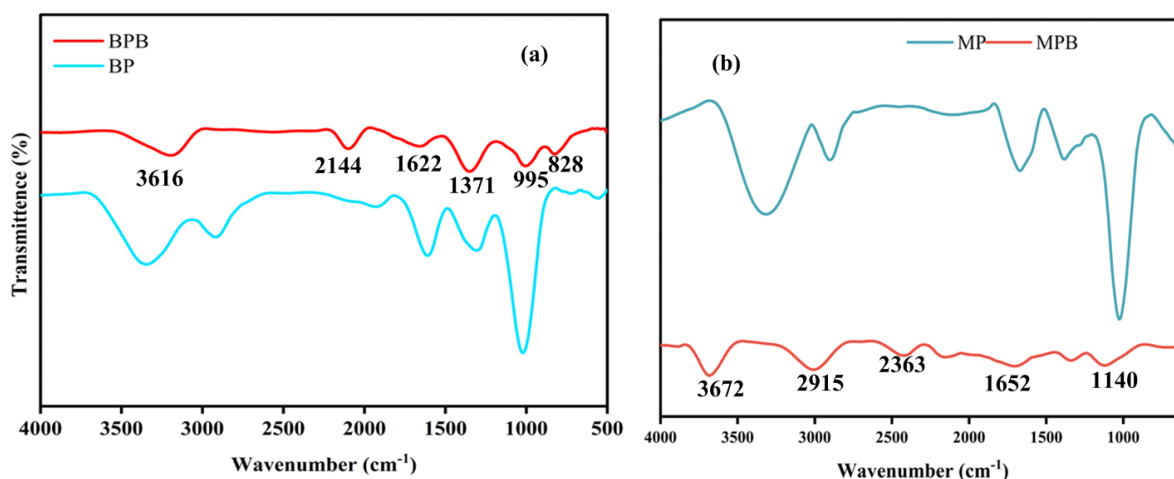


**Figure 8:** Scanning electron micrographs of: (a) Banana peels, (b) Banana peels-derived biochar, (c) Mango peels, and (d) Mango peels biochar. The solid blue arrow shows the pore space, and the red arrow shows the surface area

The rough texture and porous structure also facilitate hydrophobic interactions and pore-filling mechanisms, which are important for the effective adsorption of both BPA and progesterone. Moreover, rough surfaces are associated with higher surface areas, as they increase the complexity of the adsorption sites available for pollutants, as confirmed by BET analysis (Table 5). The MPB smoother surface areas can still aid in the adsorption of BPA, as it is partially hydrophobic and thus prefers to bind more effectively to non-polar, smooth surfaces rather than rough ones.

#### (vi) Fourier Transform Infrared Spectroscopy Functional Group Results

The FTIR results from MPB and BPB revealed the functional groups on its surface (Fig. 9 a & 9b), which can significantly impact its adsorption processes for removing organic pollutants. The BP and BPB indicate the variations of function groups before and after pyrolysis. For BP, pre- and post-calcination FTIR results at 700°C showed the disappearance of stretching vibrations of O–H at 3318  $\text{cm}^{-1}$ , indicating the breakdown of the bond during the high-temperature calcination process. The BPB had a broad, intense band between 2144 and 2050  $\text{cm}^{-1}$  that was weaker in BP.



**Figure 9: The FTIR results for (a) BPB and (b) MPB with various functional groups with more pronounced peaks in BPB compared to MPB**

Broad bands suggested that C=O groups from aldehydes, ketones or carboxylic acids might be present, indicating the existence of a carbonyl group in BPB due to biochar oxidation during calcination (Shah *et al.*, 2023). Vibrations at 2111 and 828  $\text{cm}^{-1}$  suggested the presence of C-H bonds in alkane or aromatic compounds, which could have originated from lipids, waxes or lignin in banana peels.

Additionally, vibrations at 995–1371  $\text{cm}^{-1}$  may indicate the presence of C-O groups, most likely from ether or ester groups formed during the oxidation of biochar (Shenwari *et al.*, 2019). The wavenumber ranges of 1571 to 1622  $\text{cm}^{-1}$  indicated the stretching vibration of C=C bonds, a characteristic of aromatic compounds. This suggests that BPB contains aromatic structures, possibly from lignin or other aromatic compounds found in banana peels (Shenwari *et al.*, 2019). The biochar's aromatic structures create favourable conditions for pollutant adsorption by providing sites for chemical bonding and physical interactions.

On the other hand, MP exhibits O-H stretching vibrations at 3400  $\text{cm}^{-1}$ , indicating the presence of hydroxyl groups due to water molecules, phenols, or carboxylic acid compounds. As the temperature increases, the O-H bond breaks down, resulting in a decrease in the number of hydroxyl groups and the band shifts to 3745  $\text{cm}^{-1}$  in BPB (Fig. 9b). The peak at approximately 2915  $\text{cm}^{-1}$  indicates the presence of -CH<sub>2</sub> groups, likely due to the presence of biopolymers (Zhang *et al.*, 2020a). The peak at 2363  $\text{cm}^{-1}$  indicates C≡N stretching vibrations, which may indicate the presence of isocyanates or nitriles. The wavenumber 2154  $\text{cm}^{-1}$  corresponds to the C≡C stretching vibration of alkynes, which could indicate the presence of alkynes in the biochar. The stretching at 1652  $\text{cm}^{-1}$  indicates the presence of C=C stretching vibrations, which may suggest the presence of aromatic rings or alkenes. The C-O stretching vibrations at 1292

and 1140  $\text{cm}^{-1}$  correspond to the C-O stretching vibrations of aromatic ethers and aliphatic ethers.

Therefore, Biochar has functional groups such as C=C, C≡C and C-H, making it aromatic. This means that progesterone and PBA pollutants can interact with the biochar surface by  $\pi$ - $\pi$  interactions (Peiris *et al.*, 2020; Amusat *et al.*, 2023). Also, H-bonding can happen between the hydroxyl and phenolic groups of progesterone and the hydroxyl, carbonyl and carboxyl groups of biochar (Zhang *et al.*, 2022). These various interactions significantly enhance the adsorption of heavy metals and organic pollutants onto the biochar surface.

#### (vii) Brunauer–Emmett–Teller for Pore Volume and Surface Area Characteristics

Adsorption experiments with  $\text{N}_2$  revealed the porous structure of BPB and MPB, as determined by the BET method, in terms of surface area and pore volume (Table 5). Results shows that the high surface area (564  $\text{m}^2 \text{g}^{-1}$  and 481  $\text{m}^2 \text{g}^{-1}$ ) and pore volume (0.62 vs. 0.63  $\text{mL g}^{-1}$ ) of MPB and BPB, respectively, can provide more adsorption sites, increase the diffusion rate of progesterone and BPA into the biochar and result in a higher adsorption capacity.

**Table 5:** The microstructure properties for the BPB and MPB show the specific surface area, pore volume, and pore radius with variation in pyrolysis temperature

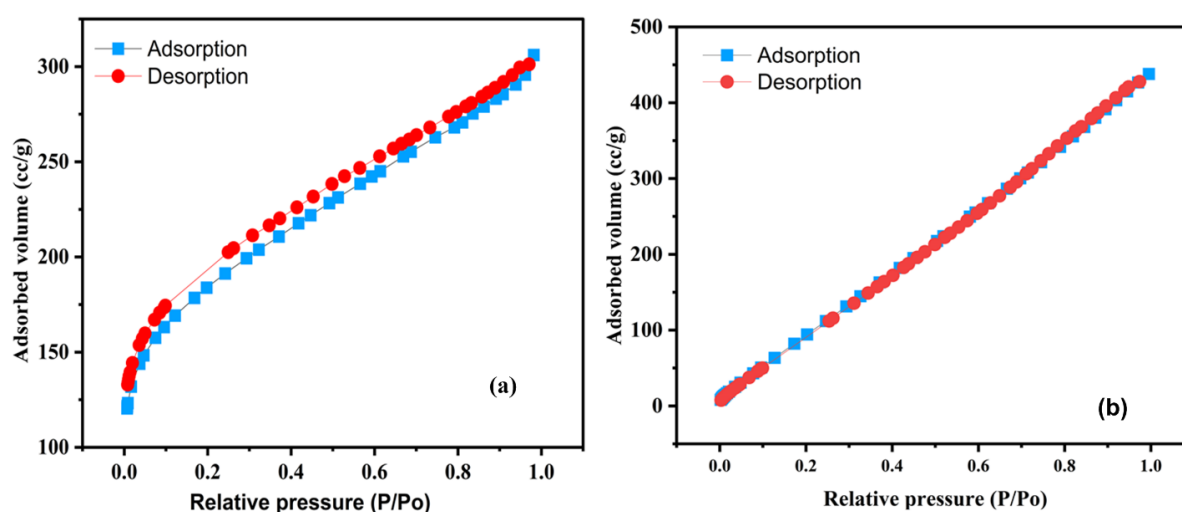
Biochar	Microstructure properties of MPB and BPB		
	BET surface ( $\text{m}^2 \text{g}^{-1}$ )	Pore volume ( $\text{mL g}^{-1}$ )	Pore radius ( $\text{Å}$ )
BP	397	0.45	12.82
BPB	481	0.63	15.12
MP	138	0.31	11.26
MPB	564	0.62	15.18

Even though MPB has a large surface area, it is suspected to effectively adsorb both small and large molecules of pollutants, such as BPA (0.9 nm) and progesterone (1.1 nm), compared to BPB. The surface areas reported in this study are higher than 238  $\text{m}^2 \text{g}^{-1}$  for banana peels at 650°C (Patel *et al.*, 2021) and 5.74  $\text{m}^2 \text{g}^{-1}$  for mango peels biochar at a pyrolysis temperature of 700°C (Zhang *et al.*, 2020). Furthermore, it is slightly lower than found in fungiculture for EDCs adsorption (Vieira *et al.*, 2022). On the other hand, both biochars exhibited similar pore volumes, indicating that they possess comparable capacities for storing adsorbates. Therefore, BPB and MPB's high surface area, pore volume, and moderate pore radius make them suitable candidates for various applications, including wastewater treatment, soil remediation and

nutrient retention in agricultural uses (Singh *et al.*, 2020; Choudhury *et al.*, 2022; Ding *et al.*, 2023).

Furthermore, adsorption-desorption isotherm curves are drawn to provide insights into the porosity, surface characteristics, and adsorption behaviour of the materials. The adsorption-desorption curve for mango peels biochar, following a Type IV isotherm (Fig. 10a), typically represents mesoporous materials with capillary condensation occurring in the pores. Monolayer adsorption dominates at low pressures, followed by multilayer adsorption. A plateau is then formed, indicating the filling of mesopores through capillary condensation at higher pressures is observed in other feedstock biosorbent (Azhagapillai *et al.*, 2023). On the other hand, biochar derived from banana peels often exhibits adsorption characteristics that align with a type II isotherm, characterised by the absence of a hysteresis loop (Fig. 10 b).

This suggests that this type of isotherm typically represents multilayer adsorption on non-porous or macro-porous materials with weak adsorbent-adsorbate interactions. Molecules stack in layers and desorb easily when the pressure decreases. Biochar can be reused multiple times as an adsorbent desorb entirely; therefore, it is suitable for non-persistent pollutants (Buttersack, 2022). This type of adsorption begins with the formation of a monolayer at low pressures, followed by multilayer adsorption as the pressure increases (Ren *et al.*, 2022). This structure is typical of biochar, featuring mesoporous and macroporous networks that result from incomplete pore filling during desorption (Chen *et al.*, 2022). This provides ample adsorption sites for target pollutants, such as progesterone and BPA.



**Figure 10:** The isotherm curves for adsorption-desorption of (a) MPB and (b)BPB

Therefore, this objective demonstrates that biochar derived from isolated banana and mango peels exhibits slightly higher adsorption efficiency and favourable physical characteristics,

such as a well-developed pore structure and surface functional groups, which significantly contribute to its performance in removing progesterone and BPA. These findings highlight the potential for co-pyrolysis of BP and MP to maximize removal efficiency without the need for chemical modification. Therefore, this makes agro-waste-derived biochars a cost-effective, eco-friendly, and sustainable alternative for mitigating EDCs in wastewater treatment applications.

### 4.3 Efficiency of Mango and Banana Peels in A Hybrid Form in Removing Progesterone and Bisphenol A

To address objective two, three mixing ratios of BPB and MPB were tested: 1:1, 2:1 and 1:1.5, respectively. Among these, the 1:1.5 ratio demonstrated the highest efficiency in removing both BPA and progesterone. The mixing ratio was determined based on material characterization results: The BET analysis was used to identify the biochar with the highest specific surface area and pore properties, while FTIR confirmed the availability of key surface functional groups and CHNOS analysis provided the elemental carbon content. Together, these parameters guided the selection of the optimal mixing ratio. All adsorption experiments were conducted under optimal conditions previously established for BPB and MPB.

#### 4.3.1 Hybrid Biochar for Progesterone and Bisphenol-A Adsorption Efficiency Results

Generally, the blended biochar made from banana and mango peels is effective in removing EDCs from water under optimal conditions. Results from this study (Fig. 11) indicate that increasing the pyrolysis temperature of biochar enhanced its adsorption efficiency towards EDCs pollutants.

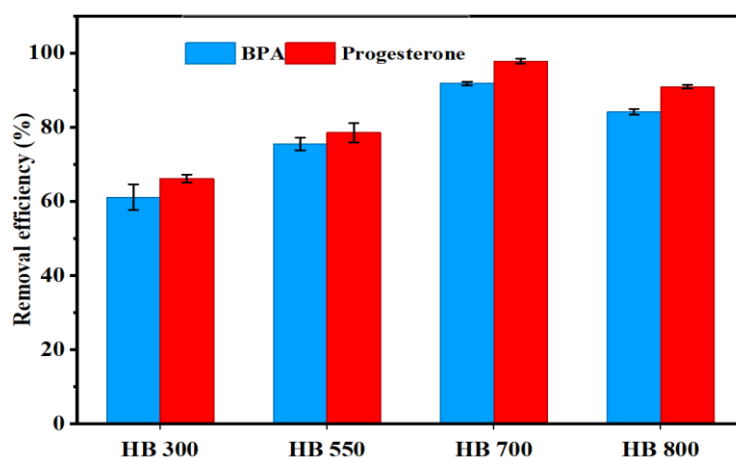


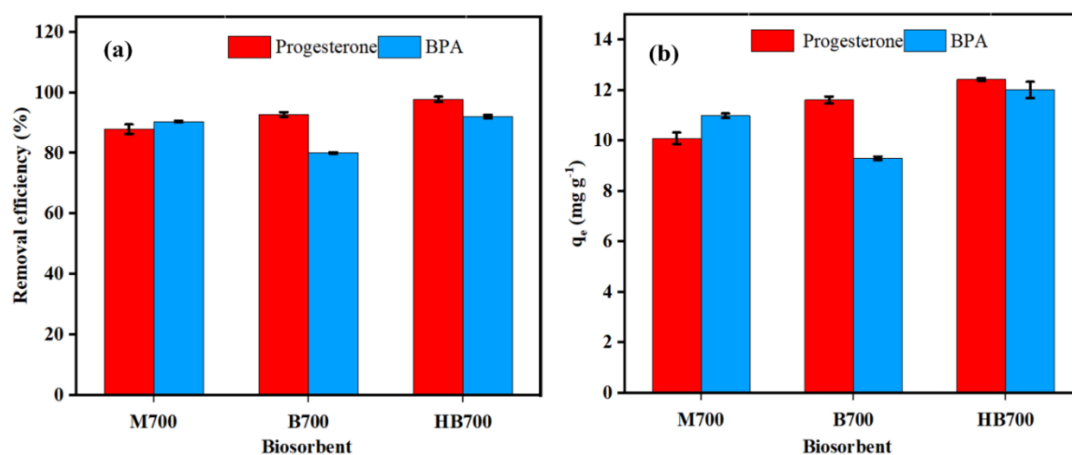
Figure 11: Removal efficiency for hybrid biochar at various pyrolysis temperatures

The highest adsorption efficiencies of  $97.80 \pm 0.02\%$  and  $91.80 \pm 0.50\%$  for progesterone and BPA, respectively, were achieved with biochar pyrolyzed at  $700^\circ\text{C}$ . At the same time, the lowest were obtained at  $300^\circ\text{C}$ . The highest adsorption efficiency at  $700^\circ\text{C}$  was due to the optimal combination of high porosity, retained functional groups and enhanced  $\pi$ - $\pi$  interactions. Lower temperatures ( $300^\circ\text{C}$ ,  $550^\circ\text{C}$ ) result in poor pore development, while over-carbonisation at  $800^\circ\text{C}$  reduces the number of active sites, making  $700^\circ\text{C}$  the optimal choice for adsorbing progesterone and BPA. In the HB system, the removal efficiency exhibited a statistically significant difference, with  $p$ -values of 0.018 for progesterone and  $<0.01$  for BPA. The Tukey confidence interval test indicates no significant difference in removal efficiency between HB300 and HB550, as well as between HB700 and HB800. This suggests that a medium pyrolysis temperature, with balanced functional groups and the development of a pore structure, provides better adsorption efficiency for BPA and progesterone.

The HB removal efficiency is higher than  $92.80 \pm 0.78\%$ ,  $80.50 \pm 2.20\%$  for BPB,  $87.90 \pm 1.56\%$  and  $90.40 \pm 2.40\%$  for MPB for progesterone and BPA removal, respectively. The higher adsorption efficiency of progesterone and BPA in HB (compared to single-peel biochars) is attributed to the synergistic effects between the two biomaterials. Banana peel biochar contributes oxygenated functional groups, such as O-H, C=O, and C-O (Fig. 9a), facilitating hydrogen bonding with progesterone. In contrast, MPB is rich in aromatic carbon (Fig. 9b), thereby contributing to the  $\pi$ - $\pi$  interaction with the benzene ring of BPA. The high adsorption efficiency of HB for both progesterone and BPA is due to its balanced porosity, synergistic functional groups, optimised hydrophobicity and multiple adsorption mechanisms. This makes it a superior adsorbent to single source biochars, ensuring effective removal of both pollutants.

The adsorption efficiency and capacity of HB and that from isolated banana and mango peels are significantly different ( $p = 0.05$ ) (Fig. 12a & 12b). The difference observed between HB and MPB may be due to the combination of banana and mango peels, which could create a more diverse surface with varying functional groups and porosities, potentially enhancing the adsorption capacity. The similarity in adsorption efficiency between BPB and HB indicates that variations in adsorption efficiency may be due to random fluctuations rather than an actual effect. Additionally, there is a possible overlap in adsorption mechanisms, such as pore distribution, functional groups, or solution conditions, which may balance the adsorption of both contaminants (Kazak, 2021).

The HB adsorption efficiency observed in this study was especially higher compared with single-feedstock biochar for removing EDCs from other studies (Esmaeeli *et al.*, 2017; Vieira *et al.*, 2022; Amusat *et al.*, 2023). It also exceeded the performance of co-pyrolyzed mixed biochar for EDC removal (Guo *et al.*, 2022), blended sucrose and mud for heavy metal removal (Kazak, 2021) and co-blended wood and plastic waste for removing phosphates and turbidity (Omoniyi & Salami, 2018).



**Figure 12:** The graph for (a) MPB, BPB and HB removal efficiency (b) adsorption capacity under 700°C pyrolysis temperature

Consequently, a hybrid biosorbent prepared at 700°C was utilised in all experimental analyses and characterisations throughout this study.

#### 4.3.2 Physiochemical Characterization of Hybrid Biochar

Table 6 presents the characterization results of the hybrid of banana and mango peels at 700°C, indicating promising properties for removing EDCs from wastewater. The HB's low ash content (2.70%) makes it well-suited for adsorbing organic pollutants, such as pesticides, herbicides and EDCs, from water and wastewater compared with single feedstocks (Johnson *et al.*, 2000; Ashesh *et al.*, 2022).

**Table 6:** Physiochemical properties of hybrid biochar

Parameters	HB	BPB	MPB	% Required
Ash content%	2.70 ± 0.15	7.80 ± 1.30	8.60 ± 0.21	< 10%
Moisture content%	0.60 ± 1.81	1.50 ± 0.51	0.38 ± 0.83	< 10%
Carbon yield%	28.05 ± 1.08	33.04 ± 0.80	23.92 ± 0.05	(20-35%)
Fixed carbon%	84.21 ± 0.91	71.10 ± 0.20	80.12 ± 1.31	(60-90%)
Volatile matter%	12.76 ± 2.10	19.19 ± 0.02	10.91 ± 0.12	< 20%
pH	8.90 ± 0.09	10.50 ± 1.03	8.80 ± 1.52	Nil
pH <sub>pzc</sub>	6.90	7.20	10.90	Nil
Progesterone concentration	Nil	Nil	Nil	Nil
BPA concentration	Nil	Nil	Nil	Nil

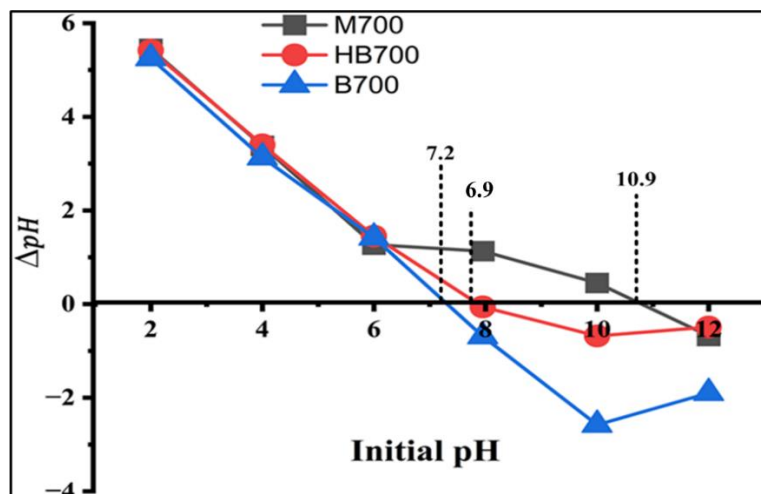
It was revealed that the HB ash content is lower than that of the singular component, which may indicate that mixing peels during biochar preparation could lead to more efficient combustion during pyrolysis, thereby reducing residual mineral matter. Moreover, synergistic effects between compounds from different peels could enhance the volatilization or decomposition processes. Therefore, HB is favourable for the adsorption of pollutants over BPB and MPB as higher ash contents which can reduce the surface area available for adsorption by filling pores with mineral residues, potentially decreasing overall porosity (Omoniyi & Salami, 2018).

The highest fixed carbon in HB (84.21%) and intermediate carbon yield (28.05%) between MPB and BPB (Table 6), result provide better properties for the adsorption efficiency because high carbon content provides more active sites for pollutant adsorption (Yang *et al.*, 2023). Furthermore, higher fixed carbon reduces oxygen-containing functional groups, making the biochar more hydrophobic, which enhances  $\pi$ - $\pi$  interactions and van der Waals forces (Ahmad & Danish, 2018), thereby improving the adsorption of nonpolar organic pollutants such as BPA and progesterone. A low total moisture content in HB is necessary, as excess moisture can hinder adsorption by blocking pores and active sites that are readily available for pollutant adsorption (Majumder & Das, 2022).

Furthermore, the intermediate volatile matter observed in HB indicates that the co-pyrolysis process during preparation resulted in a biochar with properties between those of the individual feedstocks. Therefore, HB provides a balance between surface functionality and stability for the effective adsorption of a broader range of pollutants.

Additionally, a pH of  $8.90 \pm 0.09$ , which is intermediate between MPB and BPB, suggests that both biochars have a basic surface charge. This can promote the adsorption of acidic and neutral EDCs through hydrogen bonding and  $\pi$ - $\pi$  interactions with the negatively charged surface functional groups. These results are in agreement with the study by Ahmad and Danish (2018) who indicated that pH affects the solubility and interaction of pollutants and biochar since, at higher pH, the competition between pollutants and  $H^+$  becomes low, best for the neutral and acid pollutants adsorption. This balance may be advantageous, as it reduces excessive electrostatic repulsion while retaining hydrophobic and  $\pi$ - $\pi$  interactions, which are crucial for BPA and progesterone adsorption. Therefore, HB might provide an optimal surface for progesterone removal compared to BPB, where excessive alkalinity could hinder adsorption.

For the  $pH_{pzc}$ , the HB surface charge appeared to be negative; however, at equilibrium, BPB is more negative, as the pH of 10.90 is greater than the pH of 8.99 of HB (Fig.13). A high negative surface charge for BPB makes a strong electrostatic repulsion of deprotonated BPA (for  $pH > 9.60$ ).



**Figure 13:** Hybrid biochar surface charge with comparison to solely biochar

In contrast, lower surface charge density (due to  $pH_{pzc}$  being closer to the pH of HB) reduces electrostatic repulsion, with BPA. Then, this is more favourable for the adsorption of aromatic BPA and progesterone due to hydrophobic interactions,  $\pi$ - $\pi$  stacking and Van der Waals forces (Xiao *et al.*, 2020). Therefore, this suggests that blending banana and mango peels during biochar production can improve their adsorption properties for a broader range of organic contaminants.

Furthermore, HB atomic ratio results demonstrated a balanced composition of elemental components and atomic ratios (Table 7), integrating advantageous properties from both banana and mango peels biochar. Although it contains more oxygen than mango peels biochar alone, HB effectively combines beneficial traits from both sources, potentially enhancing adsorption efficiency.

**Table 7:** The comparison of elemental and atomic ratios of hybrid biochar and banana and mango peels biochar

Biochar (g)	Elemental analysis				Atomic ratio			
	% N	% C	% H	% O	H/C	N/C	O/C	(N+O)/C
BPB	1.17	55.45	1.56	34.02	0.01	0.02	0.61	1.17
MPB	0.74	74.12	1.47	15.07	0.01	0.01	0.21	0.74
HB	0.44	56.02	0.55	30.99	0.02	0.01	0.55	0.56

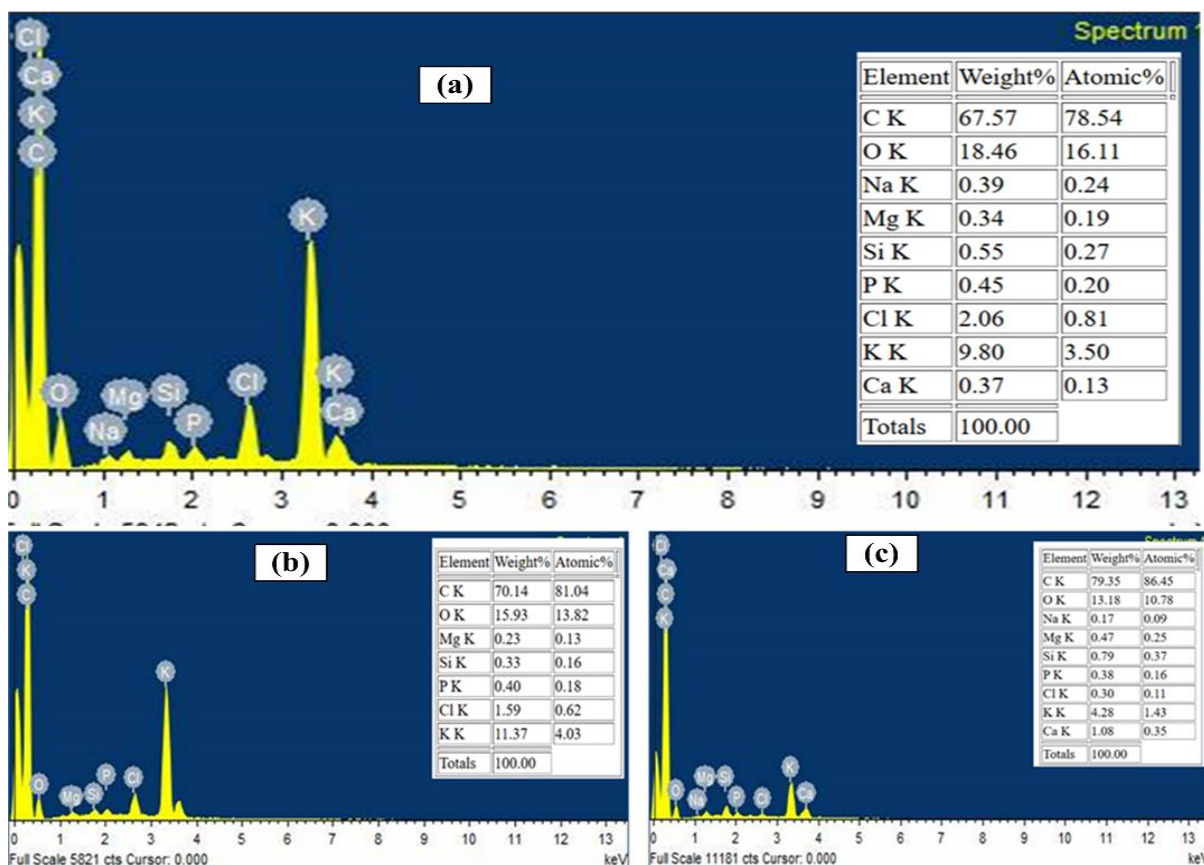
Regarding the O/C ratio, HB occupies an intermediate position between BPB and MPB, ensuring a balance in oxygenated functional groups that is crucial for capturing both hydrophobic and hydrophilic pollutants. Bisphenol A, characterized by its strong hydrophobic nature with some hydrogen bonding capability, contrasts with progesterone, which remains hydrophobic due to its steroid core but incorporates polarity from ketone regions (Cashin *et al.*, 2018). Despite a decrease in hydrophilicity, HB retains oxygen-containing functional groups, which support hydrophobic interactions with BPA through  $\pi$ - $\pi$  stacking and van der Waals forces while also preserving its hydrogen bonding capacity for progesterone.

Additionally, the H/C ratio serves as an indicator of biochar stability, with lower values (<0.60) generally indicating more stable and aromatic biochar (Xiao *et al.*, 2016), which is essential for pollutant adsorption. While HB exhibits a slightly higher H/C ratio than single feedstocks, this may improve its adsorption potential for both BPA and progesterone by facilitating hydrogen bonding interactions.

The (O+N)/C ratio represents the presence of oxygen- and nitrogen-containing functional groups relative to carbon, with a higher ratio indicating greater hydrophilicity due to the presence of polar functional groups, such as hydroxyl, carboxyl and amine groups (Chen *et al.*, 2015). Banana peels biochar shows the highest ratio, favouring hydrogen bonding and dipole interactions with polar compounds such as progesterone. Conversely, MPB, with the lowest ratio, remains predominantly hydrophobic, making it more suitable for  $\pi$ - $\pi$  interactions and van der Waals forces with BPA. The HB, by maintaining an intermediate ratio, achieves a balanced hydrophilicity and hydrophobicity, enabling effective adsorption of both BPA and progesterone through  $\pi$ - $\pi$  interactions and hydrogen bonding, making it the most versatile biosorbent for these pollutants.

### **4.3.3 Energy Dispersive X-Ray Spectroscopy Hybrid Biochar Analysis Results**

The EDS results (Fig. 14) confirm the presence of elements in the HB biosorbent. The concentrations of inorganic minerals observed in HB significantly influence the porosity, surface chemistry, and adsorption capacity of adsorbents.



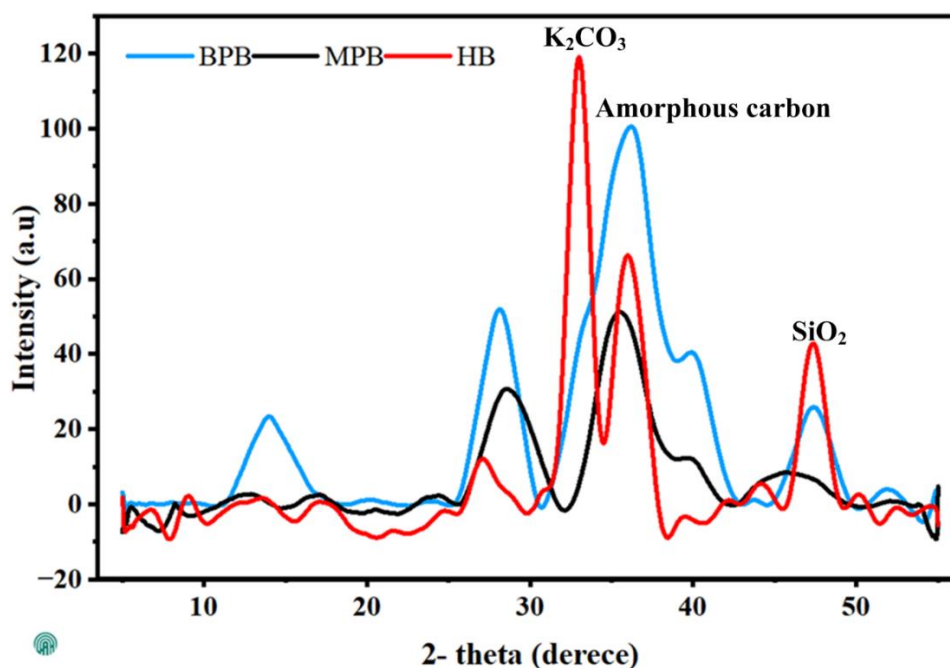
**Figure 14:** The EDS results for (a) HB with moderate content of C, O, K, and Ca compared to (b) BPB and (c) MPB

Silicon enhances mechanical stability but may limit adsorption by reducing the number of active functional groups. At the same time, Ca introduces basic sites that improve metal adsorption and affect the binding of heavy metals and nutrients (Bhatla *et al.*, 2018). Potassium can enhance electrostatic interactions with negatively charged adsorbates, though excessive K may compete with target molecules and hinder adsorption. Similarly, having more K can help plants retain essential cations, such as Ca, Mg and Ammonium (NH<sub>4</sub>OH), that plants need to grow (Coletto *et al.*, 2023).

Adsorbents like HB, BPB and MPB exhibited distinct behaviours based on their mineral composition: The HB balances Si, K and Ca (Fig. 16a), optimising properties for progesterone uptake while maintaining moderate BPA removal. The BPB excelled in progesterone adsorption but is less effective for BPA due to high K content and lack of Ca (Fig. 16b). The MPB performed best for BPA, moderately for progesterone and gained stability from Si and Ca (Fig. 16c). Therefore, this makes mineral-enriched HB a promising material for environmental remediation applications, particularly in wastewater treatment and soil amendment.

#### 4.3.4 Hybrid Biochar Crystallinity and Porosity Analysis Results

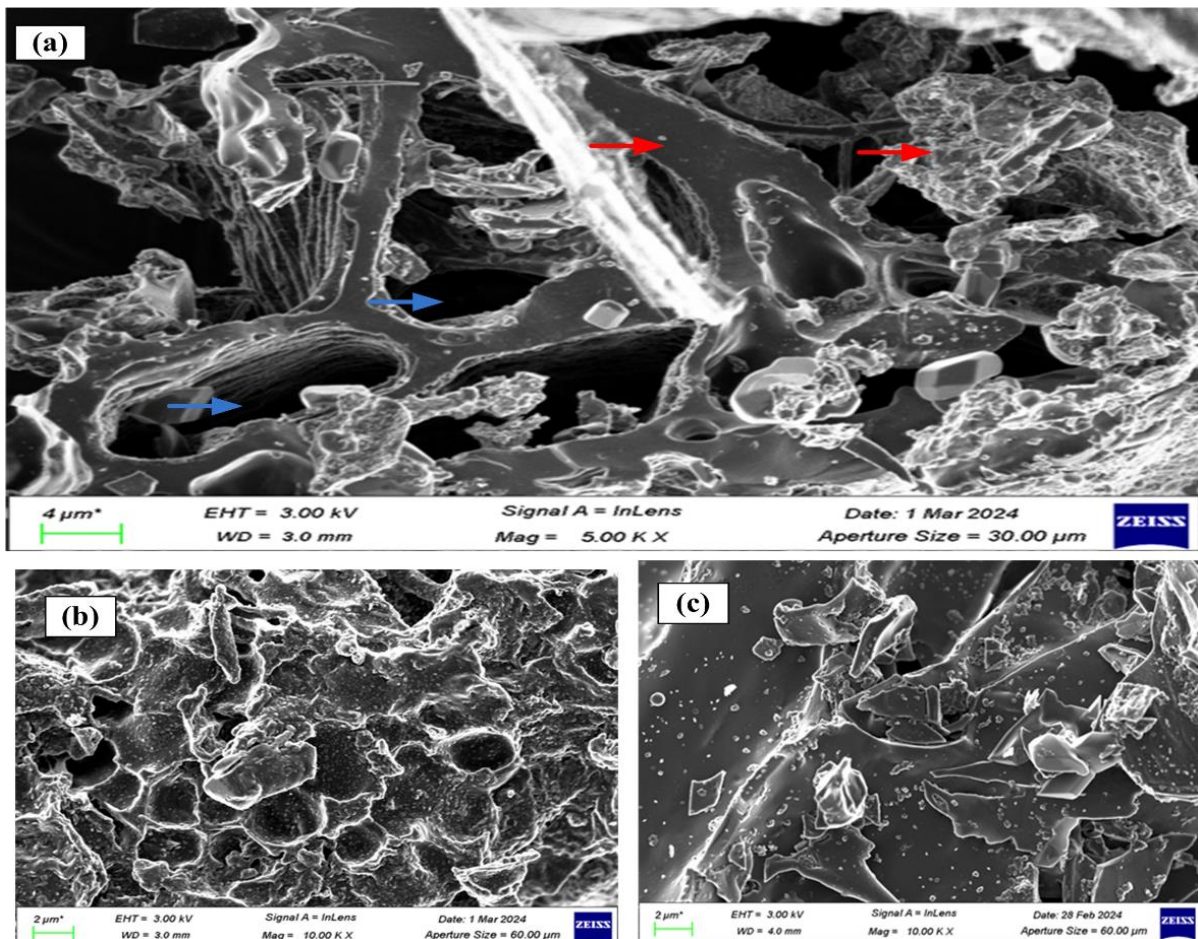
The X-ray diffraction image revealed that HB appeared to be amorphous, with some crystallinity due to the presence of inorganic minerals. On the other hand, peaks around  $32^\circ$  in HB (Fig. 15) could indicate the presence of potassium carbonate ( $K_2CO_3$ ) or other carbonate phases, which are common in plant-derived biochar (Kumar *et al.*, 2021). Like BPB, the HB also peaks at  $47^\circ$ , corresponded to a crystalline mineral phase, such as silica ( $SiO_2$ ) or potassium-related compounds. Banana peels are known to have a high potassium content, as confirmed by EDS characterization of BPB and HB. The available minerals may interact with progesterone and BPA through ion exchange or complexation, potentially increasing the overall adsorption to the biosorbent. Therefore, the co-pyrolysis of feedstocks yields biochar with hybrid characteristics, enabling the effective adsorption of diverse pollutants.



**Figure 15:** The X-Ray diffraction image for the hybrid biochar's crystallinity in comparison to BPB and MPB

#### 4.3.5 The Scanning Electron Microscopy Imaging Hybrid Biochar Results

The SEM image reveals a high development of porous properties on the surface of HB compared to MPB and BPB (Fig. 16a, 16b and 16c) providing a large surface area for pollutant diffusion. A rough and irregular surface is typically associated with higher surface areas, as it increases the complexity of the adsorption sites available for pollutants, as confirmed by BET results (Table 8). This feature represents the combined effect of both BPB with a rough surface and MPB with a smooth surface.



**Figure 16: The SEM morphological variation for (a) HB (b) BPB (c) BPB various pores**

Furthermore, the numerous and heterogeneous pores indicate that the biochar can adsorb a diverse range of micropollutants, which is advantageous for wastewater from various sources (Tong *et al.*, 2019). The SEM analysis often shows the formation of mineral nodules on the biochar surface. These nodules can contain minerals such as K, P, and C, supported by EDS analysis, which may enhance the biochar's adsorption properties by providing additional reactive sites. The presence of these minerals can also influence the interaction between the biochar and specific pollutants, potentially improving removal efficiency.

#### 4.3.6 The Brunauer-Emmett-Teller Hybrid Biochar Surface Area Analysis Results

The BET analysis of HB unveiled crucial characteristics that enhance its adsorbent capacity. The maximum BET results for HB demonstrate a surface area of  $652 \text{ m}^2 \text{ g}^{-1}$ , a total pore volume of  $0.75 \text{ cm}^3 \text{ g}^{-1}$ , and an average pore radius of  $15.20 \text{ \AA}$ , highlighting its potential for removing various organic pollutants, such as BPA and progesterone. The total pore volume and average pore radius reflect a combination of micro- and mesoporous structures, enabling biochar to

accommodate a diverse range of pore sizes and pollutant types, thereby increasing its usefulness in environmental applications.

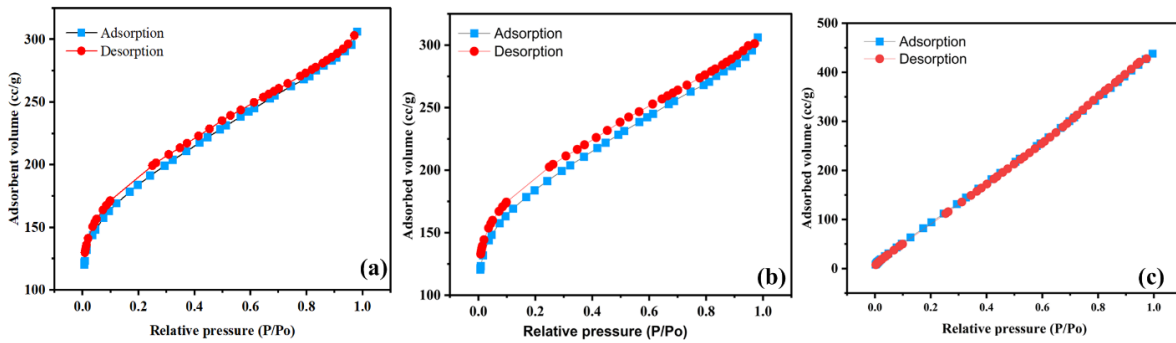
Compared to solely biosorbent, HB has the highest surface area (Table 8), which facilitates more active sites for adsorption. When compared to other co-pyrolysis biosorbent, HB's surface area exceeds that of mixed skin, pitch, leaves and polyethylene treated with Potassium hydroxide (KOH), which achieved  $581 \text{ m}^2 \text{ g}^{-1}$  for Lead (Pb (II) removal (Fan *et al.*, 2020) and the co-pyrolysis of corn stalk and rice waste, which attained  $490 \text{ m}^2 \text{ g}^{-1}$  (Wang *et al.*, 2020). However, HB's surface area is smaller than that achieved using the co-pyrolysis of peanut shells and vinasse under HCl modification, with a surface area of  $1200 \text{ m}^2 \text{ g}^{-1}$  (Arslanoğlu *et al.*, 2023) and those from mixed cyanobacteria with plastic waste, at  $1461 \text{ m}^2 \text{ g}^{-1}$  (Cao *et al.*, 2019). Therefore, the larger surface area of HB without any chemical modifications makes it valuable for adsorbing diverse micropollutants from wastewater.

**Table 8: Microstructural variations in hybrid biochar with comparison to banana and mango peels biochar**

Biochar	BET surface area ( $\text{m}^2/\text{g}$ )	Pore volume ( $\text{cc/g}$ )	Average pore diameter ( $\text{Å}$ )
BPB	481	0.62	15.01
MPB	564	0.63	15.30
HB	652	0.75	15.20

Furthermore, like MPB, the isotherm curve of HB shows a hysteresis loop, indicating that it likely corresponds to a mesoporous material. This is consistent with Type IV isotherms in the IUPAC classification (Rahman *et al.*, 2019). The adsorption curve (blue squares) and desorption curve (red circles) are distinct, with a gap between them (Fig. 17a and 17b), which reflects capillary condensation in mesopores. Furthermore, the hysteresis loop indicates that the material exhibits a combination of microporous and mesoporous structures (Toncón-Leal *et al.*, 2021), as confirmed by its surface area and pore volume (Table 8). These pores are favourable for trapping both larger and smaller organic pollutants.

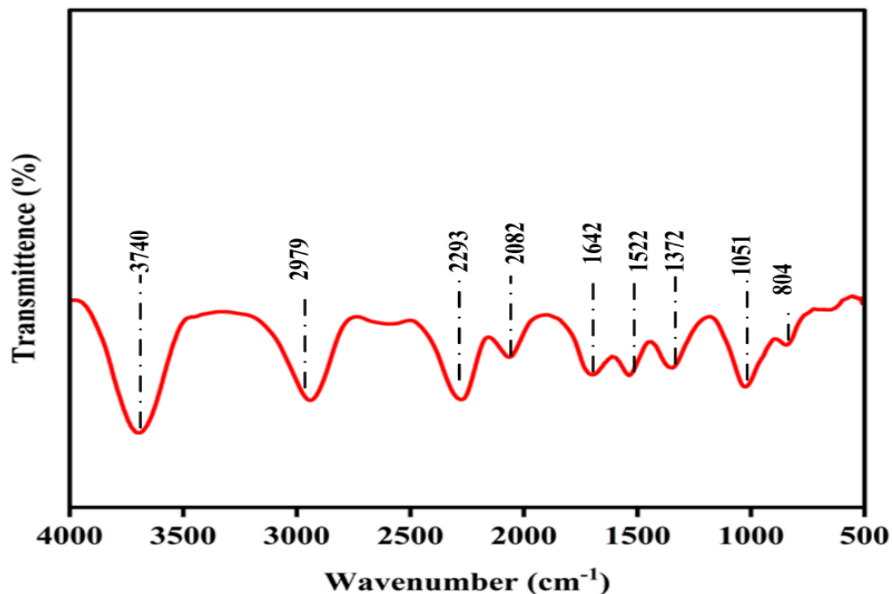
Furthermore, pores spaced far apart in the desorption path, especially in MPB, highlight structural heterogeneity, often leading to delayed desorption, which is advantageous for pollutant removal applications (Thommes *et al.*, 2015). This type of material behaviour has also been observed in many agricultural feedstock materials (Mandal *et al.*, 2017; Buttersack, 2022; Zuhara *et al.*, 2024).



**Figure 17: Adsorption–desorption isotherm with type IV curve for HB (a) and MPB (b) with type II in BPB (c)**

#### 4.3.7 Fourier Transform Infrared Spectroscopy Analysis for Hybrid Biochar

The FTIR results reveal that HB has a high degree of functional group pronouncement, which contributes to its effectiveness in removing EDCs (Fig. 18).



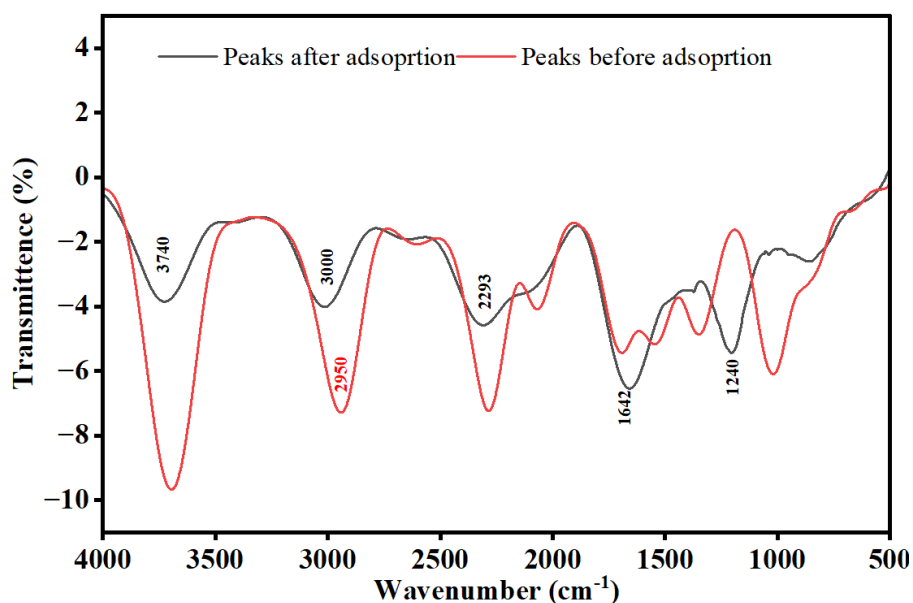
**Figure 18: The FTIR spectra for hybrid biochar revealed various spectra of the presence functional groups**

The peak  $804\text{ cm}^{-1}$  may be related to C-H out-of-plane bending vibrations, signifying the presence of aromatic compounds (Ferahtia, 2021), which can enhance the structural stability of biochar for pollutant adsorption and other applications. At  $1051\text{ cm}^{-1}$ , this peak can be assigned to the stretching vibration of the C-OH group in alcoholic groups and carboxylic acids, which is attributed to the mango peels content (Manjuladevi & Kalaiselvan, 2022). The peak at  $1372\text{ cm}^{-1}$  may be assigned to the symmetrical vibration of C-O groups, which are attributed to the decomposition of ethers, esters and phenols in both banana and mango peels (Guibaud *et al.*, 2003; Kamsonlian *et al.*, 2011).

Spectra at  $1522\text{ cm}^{-1}$  probably represent the C–N stretch of aromatic amines from mango peels (Ferahtia, 2021). The peak at  $1642\text{ cm}^{-1}$  is likely due to C=C stretching vibrations in aromatic rings or alkene of banana and mango peels (Zhang *et al.*, 2020; John *et al.*, 2022). The peaks at  $2082\text{ cm}^{-1}$  indicate C≡N stretching vibrations, possibly indicating isocyanates or nitriles of the mango peels (Zhang *et al.*, 2020). This functional group can enhance the interaction of the adsorbent with several pollutants, making them useful in adsorption applications. The  $2293\text{ cm}^{-1}$  peak may correspond to C≡C stretching vibrations, indicating the presence of alkynes, which can influence the chemical reactivity of biochar. The peak at  $2979\text{ cm}^{-1}$  is often associated with the stretching vibrations of C–H bonds in aliphatic hydrocarbons, suggesting that organic material contributes to its hydrophobic characteristics.

Moreover, the peaks at  $3655\text{ cm}^{-1}$  and  $3740\text{ cm}^{-1}$  indicate O–H stretching vibrations, commonly found in hydroxyl groups due to the decomposition of phenolic compounds in both banana and mango (Kamsonlian *et al.*, 2011; Zhang *et al.*, 2020). The O–H group can enhance hydrogen bonding with the carbonyl oxygen of the progesterone molecule and the O–H groups of BPA, facilitating its adsorption onto the biochar surface. Therefore, the combination of carbonyl, hydroxyl, nitrile and aliphatic structures in HB can enhance the biochar's hydrophilicity and its ability to interact with progesterone through mechanisms such as hydrogen bonding, electrostatic interactions, complexation and pore filling. However, the presence of C≡C and C=C facilitates  $\pi$ - $\pi$  interactions with the benzene rings of BPA, resulting in an effective adsorption efficiency.

On the other side, upon adsorption of progesterone and BPA, the FTIR spectrum of the biochar typically exhibits several modifications (Fig.19). The broad O–H stretching band around  $3740\text{ cm}^{-1}$  decreases in intensity, indicating hydrogen bonding between the adsorbent's hydroxyl or carboxyl groups and the phenolic –OH groups of BPA or the C=O group of progesterone (Lazim *et al.*, 2015; Mahgoub *et al.*, 2025). New peaks appear in around  $1200\text{ cm}^{-1}$ , attributed to the aryl ether (C–O–C) group in BPA (Lazim *et al.*, 2015). In addition, intensified peaks near  $1642\text{ cm}^{-1}$  may indicate  $\pi$ - $\pi$  interactions between the aromatic rings of BPA and the carbonaceous structure of biochar, or interactions with the C=C bonds of progesterone (Sirach & Dave, 2023). Peaks at  $2950\text{ cm}^{-1}$  shifted to  $3000\text{ cm}^{-1}$ , indicating the presence of aliphatic and aromatic C–H stretching from both adsorbates. Therefore, band shifting after progesterone and BPA adsorption confirm their interaction with biosorbent during adsorption processes.



**Figure 19:** The FTIR spectra variations before and after BPA and progesterone adsorption

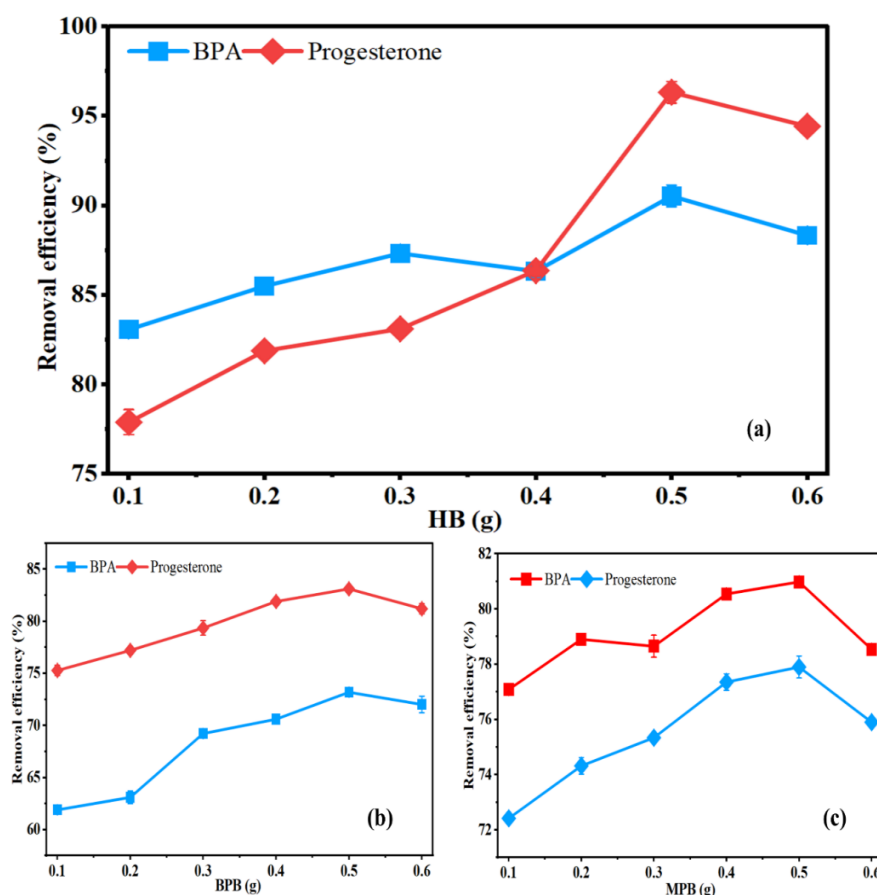
#### 4.3.8 Effect of Individual Factors on Hybrid Biochar Adsorption

Understanding the influence of individual operational factors is crucial in optimising the adsorption performance of hybrid biochar. This section examines how key parameters such as pH, contact time, adsorbent dosage, initial contaminant concentration and temperature affect the efficiency of EDCs removal by the hybrid biochar derived from banana and mango peels. By analysing the independent effects of each factor under controlled conditions, understanding can be gained on the adsorption behaviour and mechanisms involved, thereby supporting the development of more efficient wastewater treatment strategies using agro-waste-based adsorbents.

##### (i) Effect of Adsorbent Dosage

The amount of adsorbent used is crucial because it tells how well it can remove pollutants from water at a particular starting concentration. Figure 20a indicates the increase in removal efficiency as the biochar concentration increases. The progesterone and BPA equilibrium uptake was achieved using an adsorbent dosage between 0.10 and 0.60 g, reducing the adsorbate concentration from 12.50 to 0.11 mg L<sup>-1</sup> for BPA at a high adsorbent concentration. This trend suggests that higher amounts of biochar provide more available adsorption sites for pollutants. With more active sites available, the probability of adsorbate molecules finding and binding to a site increases, resulting in a higher removal efficiency.

Furthermore, the increased biochar content enables a more extensive interaction between the pollutants and the functional groups on the surface, resulting in a higher diffusion rate into the biochar pores (Farias *et al.*, 2023). However, the trend decreases at very high biochar concentrations (0.60 g) due to overcrowding, which reduces the accessibility of active sites by the adsorbate. Furthermore, at too high adsorbent concentrations, some of the surfaces or surface groups may not be fully saturated. This can cause adsorbent particles to adhere to one another, reducing the available surface area for adsorption. The removal efficiency of HB adsorption is slightly higher compared to MPB and BPB when applied in the sole form (Fig. 20b and 20c). This suggests that mixing feedstocks with different properties yields a biosorbent with enhanced properties, resulting in a higher removal efficiency of micropollutants. Furthermore, the results from this study concur with those of Prasad *et al.* (2008) and Zhang *et al.* (2020).



**Figure 20: The removal efficiency of (a) HB (b) BPB (c) MPB with the variation of adsorbent weight**

**(ii) Effect of Initial pH**

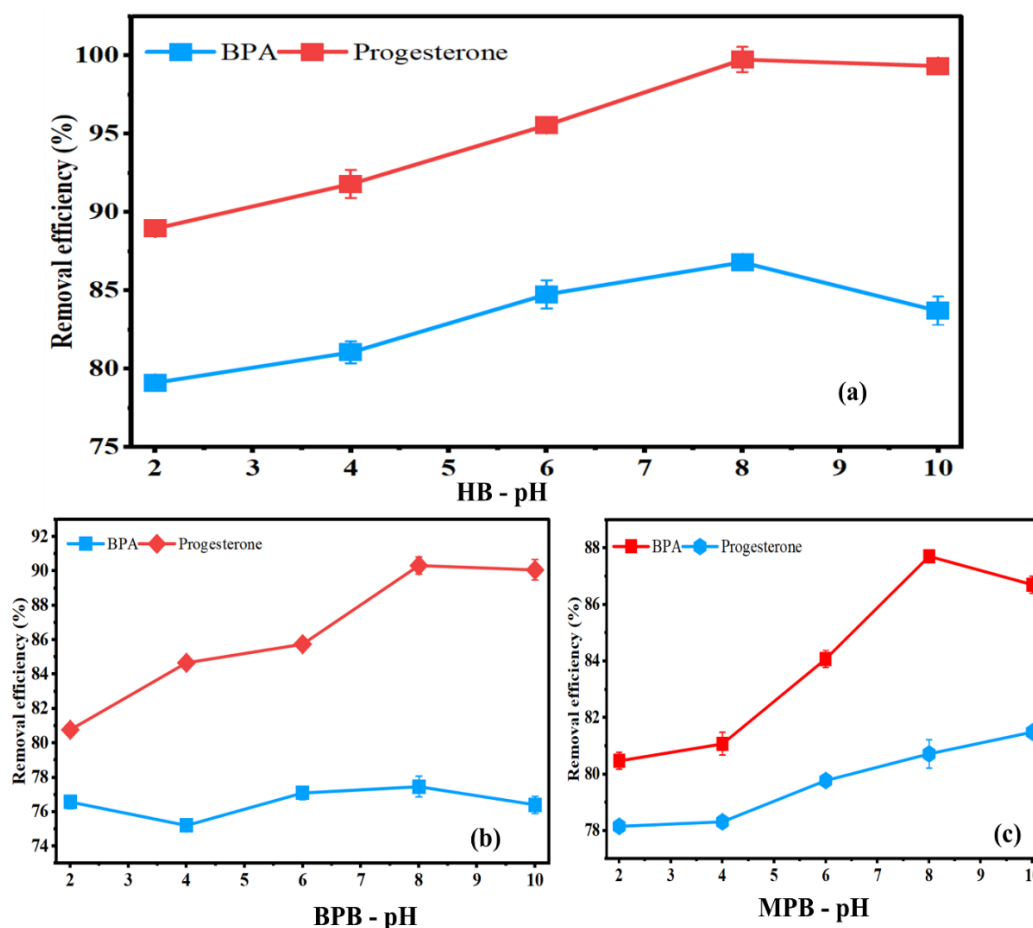
Figure 21a, 21b and 21c show that as the pH increased from 2 to 10, the adsorption efficiency increased for both BPB, MPB and HB. The HB was observed to have the highest removal

efficiency compared to individual biosorbents. The HB adsorption efficiency reaches a maximum of 96.70% and 88.81% for progesterone and BPA, respectively, at pH 8. For progesterone, this may be because at a pH level of 8, the concentration of other cations, such as  $H^+$ , decreased, reducing competition and allowing for more progesterone adsorption (Gardner & James, 2023). Therefore, progesterone can adhere to the surface of biochar through hydrophobic and  $\pi$ - $\pi$  interactions with aromatic rings present in the biochar (Gigli, 2025). Furthermore, at higher pH levels (10),  $OH^-$  ions from protonated biochar functional groups may compete with progesterone for active sites on the biochar (Georgin *et al.*, 2024), thereby reducing the available adsorption sites and, consequently, decreasing the adsorption capacity.

Likewise, the adsorption capacity of BPA is highest at a pH of 7.80 and decreases as the pH increases. This trend can likely be attributed to BPA having a  $pK_a$  of  $\sim 9.60$ , indicating that it exists in different forms depending on the pH (Amusat *et al.*, 2023). As the pH is less than 9.60, the BPA remains neutral. Therefore, at pH 7.80, BPA remains neutral primarily, allowing it to interact strongly with biochar's hydrophobic surface and aromatic rings via  $\pi$ - $\pi$  interactions between BPA's benzene rings and biochar's carbon-rich surface, as well as hydrogen bonding between BPA's hydroxyl groups ( $-OH$ ) and biochar's functional groups (Amusat *et al.*, 2023). As pH increases, BPA starts deprotonating with  $^-OH$  groups, loses  $H^+$  and becomes negatively charged, which reduces its adsorption capacity due to electrostatic repulsion.

Additionally, the low adsorption at higher pH values is due to electrostatic repulsion and competition with  $OH^-$  ions for adsorption sites. Therefore, the stability of the adsorption efficiency at pH 8 for different pollutants suggests that the HB is effective in removing EDCs under basic conditions, making it suitable for various wastewater treatment systems, as wastewater pH ranges between 6.50 and 8.50 (Mishra & Mahanty, 2012; Miruka, 2016).

The effect of pH from this study differs from those previously reported, as it shows a higher adsorption capacity for progesterone and BPA at an acidic medium (pH 2-4) (Amusat *et al.*, 2023a, 2023b). Furthermore, these results are related to other studies (Careghini *et al.*, 2015; Ma *et al.*, 2019; Vieira *et al.*, 2022). These variations in adsorption behaviour of a pollutant can vary significantly with pH, depending on the adsorbent's surface chemistry, functional groups, charge, and interaction mechanisms (Qiu *et al.*, 2022). Nevertheless, this material exhibits a high adsorption efficiency in slightly neutral to basic media, which is an advantage in wastewater applications.

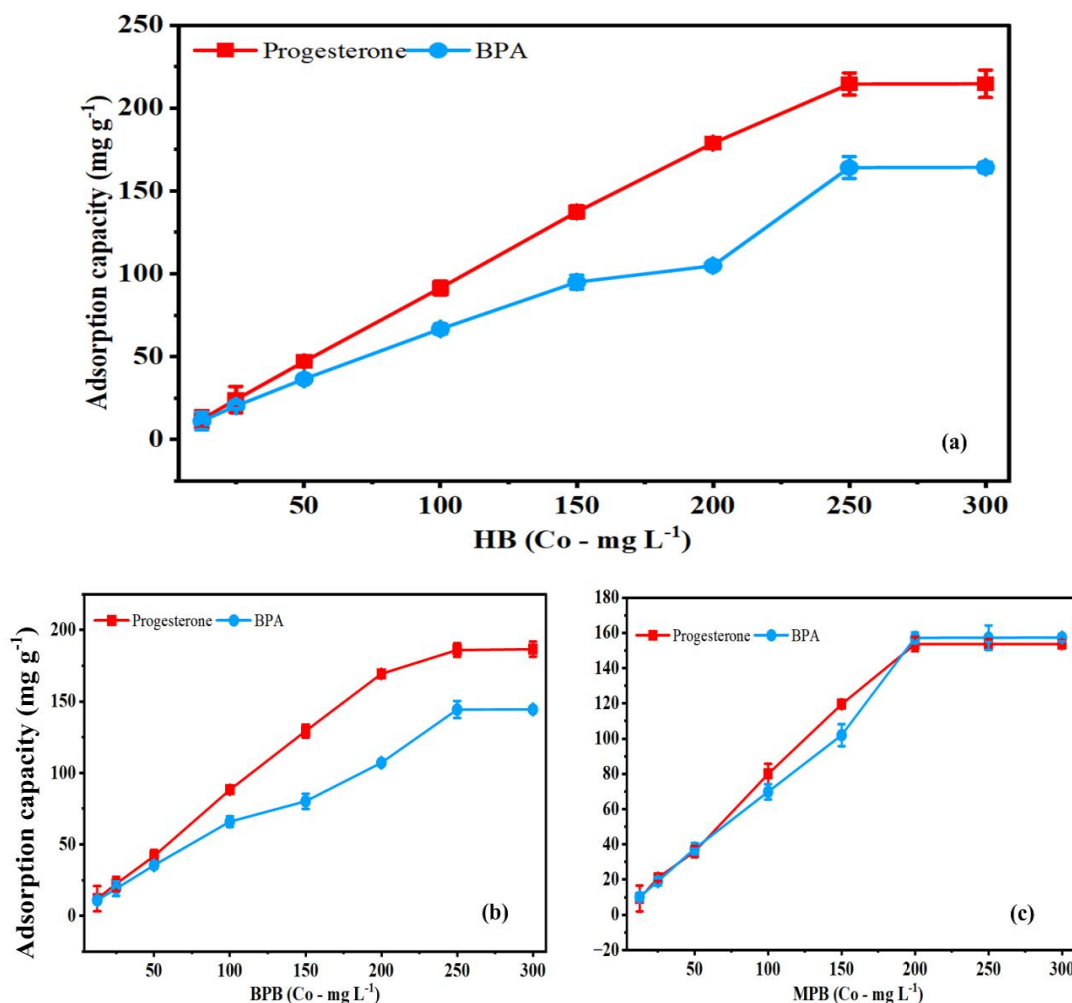


**Figure 21: Variations in BPA and progesterone adsorption efficiency for: (a) HB, (b) BPB, and (c) MPB along a range of pH**

### (iii) Effect of Initial Concentration of Adsorbate Dosage

The effect of the initial concentration of adsorbate was analysed, using different adsorbate concentrations at optimal conditions. Figure 22a, 22b and 22c show that biochar's equilibrium adsorption capacity for the bio-sorbate increased with higher initial concentrations for all adsorbents. Higher initial concentrations of the adsorbate result in faster diffusion into the pores of the biochar, allowing it to reach equilibrium more rapidly (Yu *et al.*, 2021). This rapid diffusion contributes to the overall adsorption capacity of biochar at varying initial concentrations.

However, there is a point at which pores and binding sites become saturated, resulting in a plateau in the adsorption capacity despite further increases in the adsorbate concentration. This suggests that the adsorbent has a limited capacity for adsorbing the pollutant and as the concentration increases, the adsorbent becomes saturated (Ambaye *et al.*, 2021; Tee *et al.*, 2022).



**Figure 22: The effect of the initial concentration of progesterone and BPA on the: (a) HB, (b) BPB, and (c) MPB**

Progesterone generally exhibits a higher hydrophobicity and possesses functional groups that may interact more strongly with the surface chemistry of biochar, such as  $\pi$ - $\pi$  interactions and hydrogen bonding. This can enhance its adsorption relative to BPA, which, while also hydrophobic, may have less favorable interactions with the available functional groups on the biochar. In addition, HB exhibits higher maximum equilibrium adsorption capacities of  $214.58 \pm 6.70 \text{ mg g}^{-1}$  for progesterone and  $164.20 \pm 1.20 \text{ mg g}^{-1}$  for BPA, indicating significant differences in the interaction of these two EDCs with the biochar surface. The HB  $q_{max}$  for the effect of adsorbate dosage is slightly higher than those from BPB ( $186.40 \pm 5.10$  and  $144.40 \pm 1.90 \text{ mg g}^{-1}$ ) and MPB ( $153.12 \pm 4.90$  and  $157.23 \pm 7.10 \text{ mg g}^{-1}$ ) for progesterone and BPA, respectively. This shows that mixing feedstock enhance the capacity of the biosorbent in accommodating the pollutants. On the other hand, a decrease in the removal efficiency of BPA and progesterone was observed with increasing adsorbate concentration in this study (Table 9).

**Table 9: Effect of initial concentration of BPA and progesterone on adsorption efficiency of HB, BPB and MPB**

<b>C<sub>o</sub> (mg L<sup>-1</sup>)</b>	<b>HB</b>		<b>BPB</b>		<b>MPB</b>	
	<b>Progesterone (%)</b>	<b>BPA (%)</b>	<b>Progesterone%</b>	<b>BPA%</b>	<b>Progesterone%</b>	<b>BPA%</b>
12.5	97.4 ± 4.71	89.20 ± 5.20	92.58±7.23	80.99±3.09	87.23±3.91	89.91±9.21
25	96.4 ± 6.32	81.10 ± 4.92	90.75±4.12	75.98±4.92	73.15±2.09	75.97±5.22
50	94.00 ± 1.54	73.00 ± 6.36	83.74±8.32	75.07±5.34	69.13±1.92	70.79±8.12
100	91.5 ± 3.92	70.80 ± 4.51	85.73±0.89	72.80±9.10	67.23±2.90	65.91±0.29
150	91.9 ± 2.79	68.90 ± 2.90	78.48±2.19	69.97±2.98	65.291±7.34	64.97±5.21
200	89.4 ± 2.83	63.20 ± 1.91	73.89±0.78	63.90±5.89	63.314±1.93	62.92±9.30
250	87.10 ± 5.31	65.64 ± 3.05	68.48±2.90	59.89±3.90	59.32±0.52	59.83±3.23
300	78.47 ± 2.80	54.70 ± 2.13	65.89±1.10	48.43±1.75	57.92±6.23	52.98±1.63

At lower initial concentrations, the number of available adsorption sites on the adsorbent surface is much greater than the number of adsorbate molecules. This allows for a high proportion of the contaminant to be captured, resulting in high removal efficiency. As the initial concentration increases, the number of adsorbate molecules becomes significantly larger relative to the available adsorption sites (Mahmoudian *et al.*, 2023), resulting in rapid saturation of the adsorbent surface. Once saturation occurs, additional adsorbate molecules remain in the solution and no further removal can occur, reducing the percentage of adsorbate removed even as the absolute amount adsorbed increases.

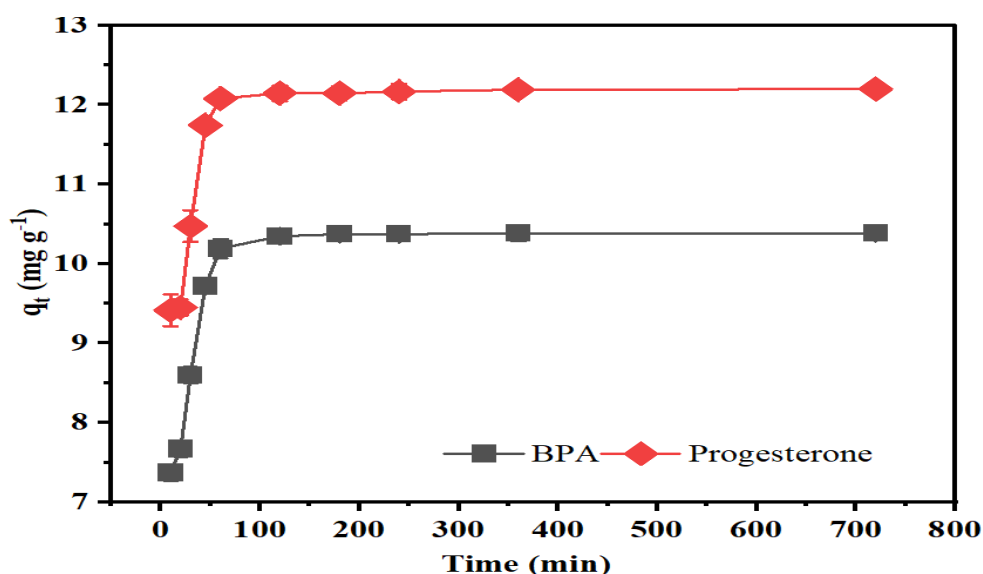
Furthermore, at higher concentrations, adsorbate molecules compete more intensely for the limited number of active sites. This competition further reduces the probability that each molecule will be adsorbed, lowering the overall removal efficiency. Furthermore, the HB removal efficiency appeared higher to both BPA and progesterone than those from BPB and MPB even at higher concentration. This indicates that co-pyrolysis of different feedstocks provides best biochar for the removal of EDCs.

This trend can also be observed in BPA adsorption studies, where it was found that increasing the initial BPA concentration from 10 mg L<sup>-1</sup> to 50 mg L<sup>-1</sup> resulted in a decrease in percentage removal, despite an increase in the total amount of BPA adsorbed per gram of adsorbent (Batra & Datta, 2022). Furthermore, similar trends are observed for hormone removal, specifically for estrogens and progesterone, where the removal efficiency increases with increasing adsorbent dosage but decreases with higher initial concentrations due to surface saturation (Zarghi *et al.*, 2019; Vieira *et al.*, 2022). This is a direct consequence of the fixed number of adsorption sites on the adsorbent.

#### **(iv) Effect of Contact Time**

The effect of contact time show that the adsorption rate is high at the beginning of the adsorption process, within 10 minutes, because the biochar surface is actively adsorbing the pollutant (Fig. 23). As the time progressed, the adsorption rate decreased as the available adsorption sites on the biochar surface became saturated. In general, the adsorption of EDCs in this study took place in two phases: the rapid phase, which ended around 30 min and the slow phase, which reached adsorption equilibrium with 12.07 mg g<sup>-1</sup> and 10.34 mg g<sup>-1</sup> of progesterone and BPA, respectively, within 60 min and 120 min. These showed that longer contact times provide more time for the adsorbate to diffuse into the adsorbent's pores and internal surfaces (Li *et al.*, 2022). The equilibrium reached differs with varying adsorption

capacities, likely because progesterone is a steroid hormone with a relatively compact and rigid structure, which may enable it to interact more quickly with active sites on the biochar surface (Amusat *et al.*, 2023).



**Figure 23:** Effect of contact time for the progesterone and BPA adsorption capacity

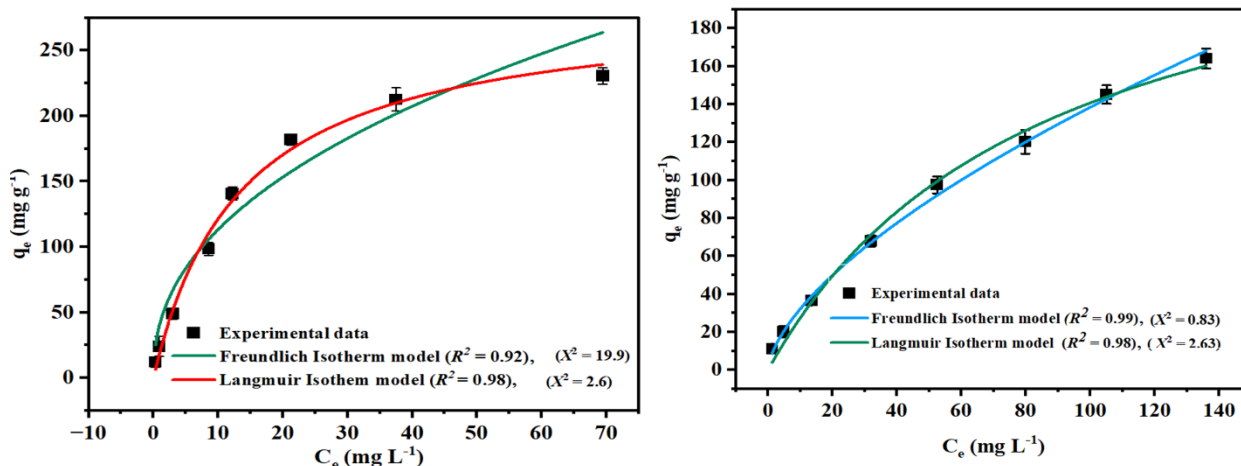
In contrast, BPA, being an aromatic compound with hydroxyl groups, may experience slower diffusion into the biochar pores due to steric hindrance (Xing *et al.*, 2023). This adsorption trend has also been observed by Aricov and Leontieş (2025) in the adsorption of BPA, Vieira *et al.* (2022) in the removal of steroid hormones and Nadew *et al.* (2023) in the removal of blue dye. Therefore, longer contact times provide more time for the adsorbate to diffuse into the adsorbent's pores and internal surfaces (Li *et al.*, 2022).

#### 4.3.9 Adsorption Isotherm Models

##### (i) Freundlich and Langmuir Isotherm Models

Adsorption isotherm analysis is used to study the equilibrium relationships between adsorbate and adsorbent at room temperature. Moreover, Fig. 24a and b show that adsorption of progesterone is better described by the Langmuir model, as evidenced by a higher correlation coefficient ( $R^2 = 0.98$ ) and a lower Chi-square value ( $\chi^2 = 2.60$ ), compared to the Freundlich model ( $R^2 = 0.92$ ,  $\chi^2 = 19.90$ ). Therefore, progesterone adsorption probably occurs in a monolayer with a homogeneous surface and uniform binding sites. This study resembles other studies that have used polymers for the removal of BPA and progesterone (Cáceres *et al.*, 2018) and the sorption of EDCs in polyamide microplastics (Dias *et al.*, 2023).

In contrast, for BPA, both models provide a good overall fit to the experimental data; however, the Freundlich model offers a slightly better fit, supported by its lower  $\chi^2$  value (0.83). Hence, the BPA adsorption process likely occurs on a heterogeneous surface with possible multilayer adsorption behaviour.



**Figure 24: Freundlich and Langmuir model for concentration variation of (a) progesterone, (b) BPA**

Furthermore, the Freundlich isotherm parameters for progesterone and BPA were 0.36 and 0.63, respectively, which fell within the range of 0 to 1. This suggests that the adsorption conditions for these EDCs were favourable, indicating a significant degree of surface heterogeneity and strong interactions between the adsorbate and the adsorbent. Furthermore, the higher  $K_F$  and  $K_L$  values (Table 10) indicate that adsorption occurs rapidly with a high affinity between the adsorbent and adsorbate. The greater difference in values between progesterone and BPA suggests that HB can hold more progesterone than BPA solute at equilibrium. This may be because progesterone is more hydrophobic and, therefore, expected to be more retained through  $\pi$ - $\pi$  stacking and hydrogen bonding, while BPA can be affected by steric hindrance facilitated by the benzene ring (Vieira *et al.*, 2022; Lekene *et al.*, 2023).

On the other hand, the Langmuir model's high maximal adsorption capacity ( $q_{max}$ ) (Table 10) suggests that the adsorbent can retain a significant amount of adsorbate under equilibrium conditions. Furthermore, the higher  $q_{max}$  suggests that treatment systems can be designed to effectively remove EDCs and other pollutants from wastewater, thereby contributing to more sustainable environmental management practices. The  $q_{max}$  from this study was higher than those from a single feedstock of banana and mango peels biochar from other studies (Annadurai *et al.*, 2002; Zhao *et al.*, 2013; Liu *et al.*, 2020a). However, it is also higher compared to Vieira *et al.* (2022), who use fungiculture waste biochar to remove EDCs, including progesterone. This is because using mixed feedstocks can improve biochar quality and increase oxygen-

containing functional groups for high  $\pi$ - $\pi$  interactions with aromatic groups of pollutants (Hoslett *et al.*, 2020).

**Table 10: Variations of parameters in different isotherm models for hybrid biochar**

Isotherm models	Parameters	EDCs	
		Progesterone	BPA
Freundlich	KF (mg g <sup>-1</sup> )	53.40	7.45
	n	2.70	1.58
	1/n	0.36	0.63
	R <sup>2</sup>	0.92	0.99
Chi-Square	$\chi^2$	19.90	0.83
Langmuir	q <sub>max</sub> (mg g <sup>-1</sup> )	307	260
	K <sub>L</sub> (L mg <sup>-1</sup> )	0.02	0.01
	R <sub>L</sub>	0.14	0.25
	R <sup>2</sup>	0.98	0.98
Chi-Square	$\chi^2$	2.60	3.18
Dubinin-Radushkevich (D-R)	K <sub>DR</sub> (mol <sup>2</sup> k <sup>-1</sup> J <sup>2</sup> )	0.210	1.16
	q <sub>max</sub> (mg g <sup>-1</sup> )	115.20	74.21
	$\mathcal{E}$ (kJ mol <sup>-1</sup> )	1.52	0.651
	R <sup>2</sup>	0.64	0.49
Temkin	A (L g <sup>-1</sup> )	1.95	2.22
	b (kJ mol <sup>-1</sup> )	55.67	81.95
	B	44.50	30.23
	R <sup>2</sup>	0.91	0.92

Similarly, mixed feedstock biochar also provides biochar with a heterogeneous surface morphology featuring pores of varying sizes, which effectively removes diverse micropollutants from wastewater (Tong *et al.*, 2019).

However, the separation factor ( $R_L$ ) should lie within the range ( $0 < R_L < 1$ ), where ( $R_L = 0$ ) indicates irreversible adsorption, ( $R_L = 1$ ) reflects a linear process, and ( $R_L > 1$ ) signifies unfavourable conditions. The  $R_L$  values in this study are 0.14 and 0.25 for progesterone and BPA, respectively. The  $R_L$  value for progesterone, being closer to 0, indicates highly favourable and near-irreversible adsorption due to strong adsorbate-adsorbent interactions. While the ( $R_L$ ) value for BPA is less favourable than that of progesterone, it still falls within the favourable range, suggesting comparatively weaker binding. Overall, the small ( $R_L$ ) values favourably indicate that the combination of diverse feedstocks exhibits a strong affinity for adsorbate removal, thereby creating a synergistic effect and enhancing the adsorption capacity of the materials.

### (ii) Dubinin-Radushkevich (D-R) isotherm model

The Dubinin-Radushkevich (D-R) isotherm model helps assess the nature and capacity of adsorption, particularly distinguishing between physical and chemical adsorption mechanisms. Table 10 indicates that both progesterone and BPA show low mean adsorption energies of  $1.52 \text{ kJ mol}^{-1}$  and  $0.65 \text{ kJ mol}^{-1}$ , respectively, which are well below the  $8 \text{ kJ mol}^{-1}$  threshold, confirming that physisorption (i.e., adsorption through weak van der Waals forces) dominates for both compounds.

Progesterone exhibits a significantly higher maximum adsorption capacity ( $q_{\text{max}} = 115.20 \text{ mg g}^{-1}$ ) than BPA, indicating a stronger affinity or better compatibility with the adsorbent's surface. The higher D-R constant for BPA ( $K_{\text{DR}} = 1.16 \text{ mol}^2 \text{ k}^{-1} \text{ J}^2$ ) corresponds to a steeper drop-off in adsorption with a lower adsorption energy and weaker binding. The lower adsorption energy (E) for both indicates weaker interactions with BPA, whose even lower E suggests an even weaker interaction with the adsorbent surface than progesterone. Both compounds exhibit an  $E < 8 \text{ kJ mol}^{-1}$  (physical adsorption), consistent with D-R studies on pharmaceuticals (N'diaye & Kankou, 2020) and organics (Bopda *et al.*, 2019).

The model's  $R^2$  values indicate only a moderate to poor fit to the experimental data (Fig. 23), especially in the case of BPA. The  $R^2$  values here are lower than those for D-R fits for heavy metals like lead ( $R^2 = 0.99$ ) and chromium ( $R^2 = 0.93$ ), suggesting that the D-R model is less suitable for organic compounds like these. This implies that the D-R model may not fully capture the complexity of the adsorption process and that alternative isotherm models like Langmuir or Freundlich offer more accurate descriptions (Table 10).

### (iii) Temkin Isotherm Model

The Temkin isotherm results reveal that both progesterone and BPA adsorption processes are well-described by this model, as indicated by high  $R^2$  values (Fig. 25), showing consistent interaction patterns with the adsorbent. Bisphenol A exhibits a slightly higher binding constant A value ( $2.22 \text{ L g}^{-1}$ ) than progesterone ( $1.95 \text{ L g}^{-1}$ ), suggesting that BPA binds slightly more strongly to the adsorbent surface at low concentrations. The binding affinity from this model does not align with that of the D-R, Freundlich, and Langmuir models, indicating that progesterone has a higher affinity for the adsorbent than BPA. The heat of adsorption  $b$  is higher for BPA (Table 10), indicating that the adsorption of BPA is more endothermic, involving more energy per mole.

However, progesterone has a higher Temkin constant B ( $44.50 \text{ J mol}^{-1}$  vs.  $30.23 \text{ J mol}^{-1}$ ), reflecting stronger interaction effects and potentially more uniform energy distribution across the adsorption surface. Overall, the Temkin model effectively captures the influence of adsorbate–adsorbent interactions for both compounds, suggesting that while BPA binds more energetically, progesterone may interact more uniformly across the adsorbent surface.

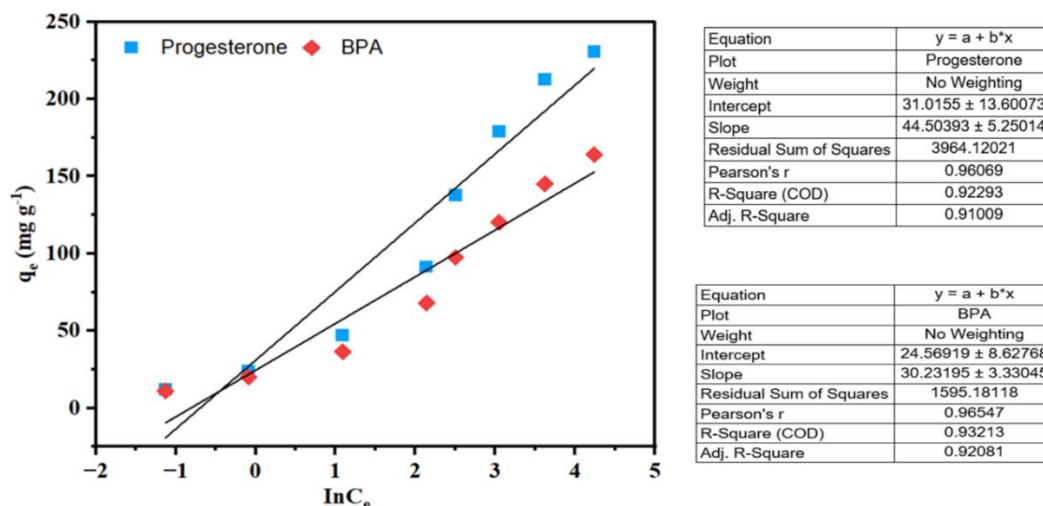


Figure 25: Temkin fitting isotherm model with best fit of BPA in hybrid biochar

#### 4.3.10 Kinetics Modelling

##### (i) Pseudo-First-Order and Pseudo-Second-Order Models

The non-linear model equations were used to determine the pseudo-first-order and pseudo-second-order kinetic parameters. Figure 26a and 26b show that the pseudo-second-order model provides a significantly better fit for both EDCs. It yields a high  $R^2$  value of 0.96 for progesterone, indicating strong linearity between experimental and predicted data.

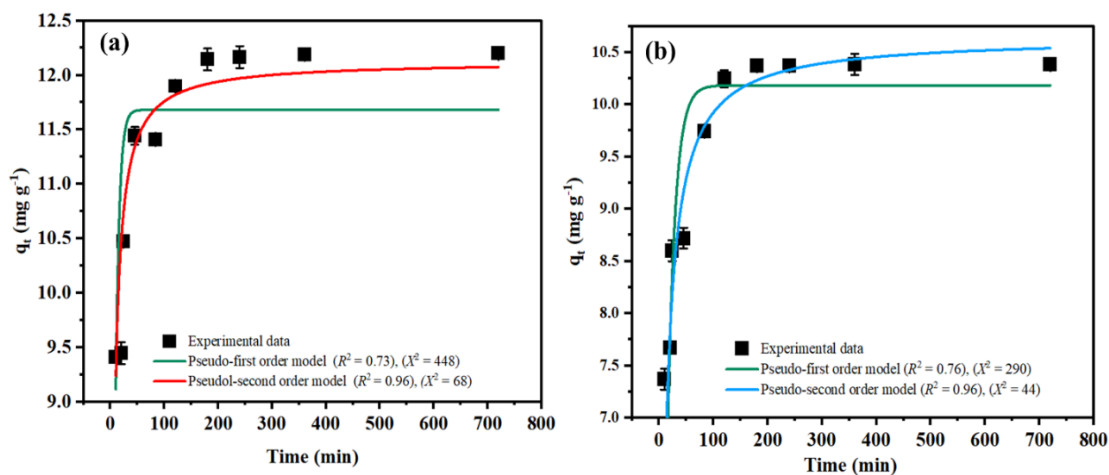


Figure 26: The graph for (a) Progesterone (b) BPA pseudo-first order and pseudo-second order kinetics models

The calculated  $q_e$  12.10 mg g<sup>-1</sup>, which is very close to the experimental value, and the lower  $\chi^2$ . For BPA, a similar pattern is observed (Fig. 27b); the pseudo-second-order model provides a significantly better fit, with an  $R^2$  value of 0.96, a calculated  $q_e$  of 10.60 mg g<sup>-1</sup> and a lower  $\chi^2$  value of 44. This remarkable reduction in error and improved alignment of data suggests that the adsorption of progesterone and BPA is better described by the pseudo-second-order kinetics with chemical bonding as the dominant mechanism. This suggests that a slow rate and adsorption chemisorption are the steps that determine the rate at which adsorbate and the functional groups in the biochar form chemical bonds (Dinçer, 2021; Hernández-Abreu *et al.*, 2021).

Chemisorption involves strong interactions, including covalent bonding, ionic attraction, surface complexation and ligand exchange, which are characterized by strong bonding and greater specificity for adsorption (Blesa *et al.*, 2000; Králik, 2014). Furthermore, chemisorption is an irreversible process, making it difficult for pollutants to desorb in the treated environment (Hernández-Abreu *et al.*, 2021). Therefore, chemisorption plays a key role in the adsorption of heavy metals, reactive gases and functionalised organics. This chemisorption trend has also been observed in the adsorption of organic pollutants and EDCs (Patel *et al.*, 2019; Vieira *et al.*, 2022). The same adsorption kinetics trend was also observed by Qi *et al.* (2023) using *Typha orientalis* as a wetland plant for BPA adsorption and by Hernández-Abreu *et al.* (2021) using lignin-based activated carbon.

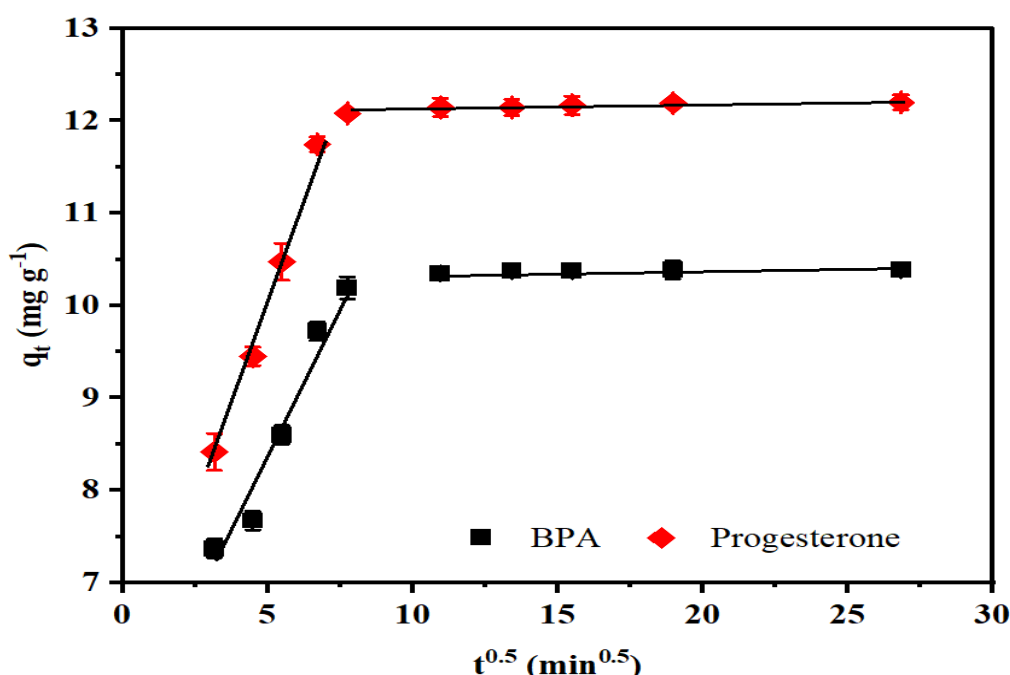
**Table 11: Pseudo-first and second-order parameters for hybrid biochar for adsorption of BPA and progesterone**

<b>Adsorbates</b>	<b>Models</b>	<b>Expe. <math>q_e</math></b>	<b>Calc. <math>q_e</math></b>	<b><math>k_1/k_2</math></b>	<b><math>R^2</math></b>	<b><math>\chi^2</math></b>	<b>Implication</b>	<b>Dominant mechanisms</b>
Progesterone	Pseudo-first order	12.20	11.60	0.15	0.73	448	Moderate rate, poor model fit	Physisorption (unlikely)
	Pseudo-second order	12.20	12.10	0.03	0.96	68.30	Slow rate, excellent model fit	Chemisorption (dominant)
BPA	Pseudo-first order	10.40	10.10	0.07	0.76	290	Moderate rate, poor model fit	Physisorption (unlikely)
	Pseudo-second order	10.40	11.60	0.01	0.96	44.50	Slow rate, excellent model fit	Chemisorption (Dominant)

## (ii) Intraparticle Diffusion Kinetics Model

The intraparticle diffusion model is a kinetic model used to describe the adsorption process where the rate-controlling step is the diffusion of adsorbate molecules inside the pores of the adsorbent particles. If a plot of solute adsorbed vs square root of the half time provides a straight line passing through the origin, the intraparticle diffusion is the rate-limiting step; if not, the rate-limiting step is affected by both film diffusion and intraparticle diffusion (Ofomaja *et al.*, 2020). Weber Jr and Morris (1963) propose an equation  $q_t = k_{id}t^{0.5} + C$ , where  $q_t$  is the adsorption capacity ( $\text{mg g}^{-1}$ ),  $t$  is the adsorption time (min),  $k_{id}$  is the rate constant ( $\text{mg g}^{-1} \text{min}^{0.5}$ ) and constant  $C$  ( $\text{mg g}^{-1}$ ) represents the boundary layer thickness.

In this study, intraparticle diffusion is not the only rate-limiting step, as the intercept for both pollutants does not pass through the origin (Fig. 27). The plot exhibits two distinct steps, with the first contributed by the effect of the boundary layer. At the same time, the second is attributed to intraparticle diffusion. Thus, the rate-limiting steps are both intraparticle diffusion and film diffusion.



**Figure 27: Intraparticle diffusion of progesterone and PBA into hybrid biochar**

Furthermore, the higher boundary layer effect for BPA ( $8.18 \text{ mg g}^{-1}$ ) compared to progesterone ( $7.25 \text{ mg g}^{-1}$ ) (Table 12) suggests that BPA experiences greater resistance from the external boundary layer than progesterone. Additionally, intraparticle diffusion analysis reveals that progesterone compounds exhibit nearly higher pore diffusion rates behaviour, with  $k_{id}$  of  $0.21$  vs  $0.11 \text{ mg g}^{-1} \cdot \text{min}^{0.5}$  of BPA. This implies that while progesterone adsorption is primarily

controlled by intraparticle diffusion, BPA adsorption is more strongly influenced by film diffusion.

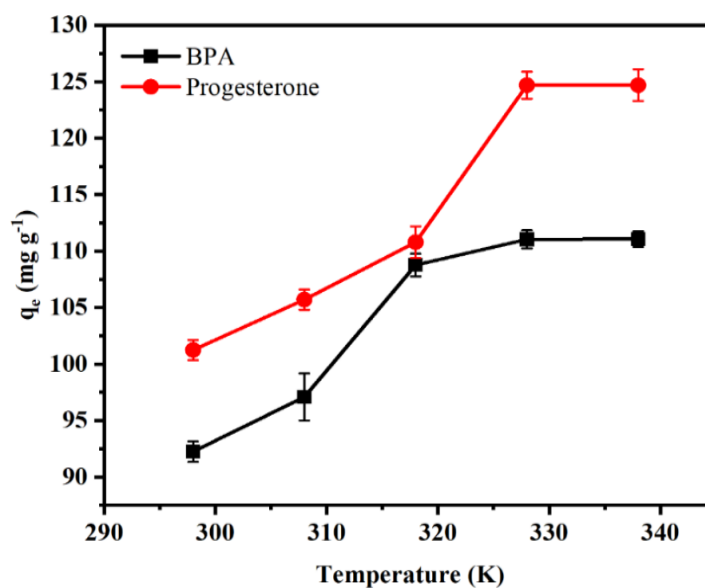
**Table 12: Intraparticle diffusion parameters for progesterone and BPA in hybrid biochar**

EDCs	$k_{id}(\text{mgg}^{-1} \text{min}^{0.5})$	$R^2$	C (mg g <sup>-1</sup> )	Implication	Dominant mechanisms
Progesterone	0.20	0.47	7.25	Moderate rate, poor fit	Intraparticle diffusion
BPA	0.11	0.40	8.18	Poor rate, poor fitting	Film diffusion

Overall, progesterone shows slightly more favourable adsorption kinetics. At the same time, BPA suffers from external mass transfer resistance, hence facilitating other interactions by other means, i.e, increasing temperature or stirring rate to reduce the boundary layer and increase the intraparticle diffusion rate (Ofomaja *et al.*, 2020; Wang & Guo, 2022), is important. Generally, the intraparticle model did not predict well the progesterone and BPA adsorption into the HB, as  $R^2$  values are very low (0.40 - 0.47), respectively. The same trend has been observed in the adsorption of acid dyes onto *S. marginatum* and red dye onto *S. platensis* (Daneshvar *et al.*, 2012; Dotto & Pinto, 2012).

#### 4.3.11 Adsorption Thermodynamics for hybrid Biochar

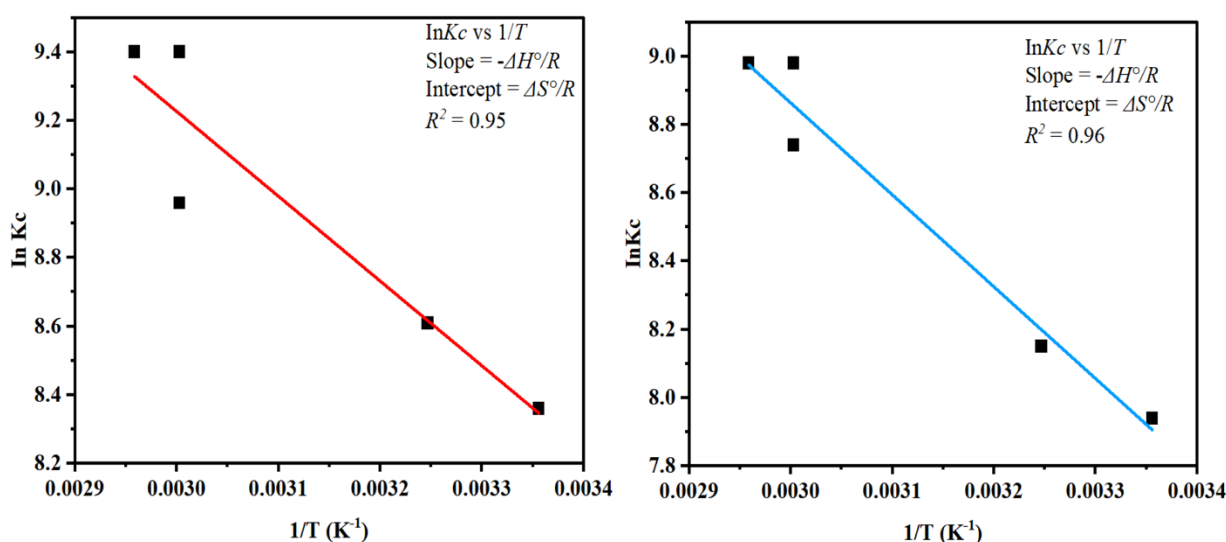
Thermodynamics analysis shows that as the temperature increases (298, 308, 318, 328 K), the adsorption capacity ( $q_e$ ) also increases (Fig. 28).



**Figure 28: Effect of temperature on the adsorption capacity of HB for BPA and progesterone**

This is because higher temperatures enhance molecular motion and diffusion, facilitating better interactions between adsorbates and adsorbents, particularly in porous materials (Yetgin & Amlani, 2024). Furthermore, as temperature increases, desorption can also increase (Liao *et al.*, 2024); therefore, the rates of adsorption and desorption become balanced and the net removal remains constant. This trend is also observed in the adsorption of methyl orange by magnetic activated carbon and in the removal of anionic and cationic dyes by banana peels biochar (Sevim *et al.*, 2021; Tolkou *et al.*, 2024).

Furthermore, thermodynamic properties provide insights into the feasibility, spontaneity and nature of interactions between adsorbates and adsorbents. The HB plot shows a relative correlation of 0.95 and 0.96 (Fig. 29) with the  $\Delta H^\circ$  and  $\Delta S^\circ$  determined by plotting  $\ln K_c$  vs  $1/T$ ,  $\Delta H^\circ = 23.59$  and  $22.33$  kJ mol<sup>-1</sup> for BPA and progesterone, respectively.



**Figure 29: Henry linear fitting graph for (a) progesterone (b) BPA on determination of thermodynamics parameters**

This indicates endothermic adsorption, where adsorption capacity increases with temperature (Üzek *et al.*, 2022). Furthermore, these magnitudes fall within the intermediate range between typical physisorption (<20 kJ/mol) and chemisorption (>40 kJ mol<sup>-1</sup>). These suggest that the adsorption might involve strong physisorption or weak chemisorption (Safaei-Farouji *et al.*, 2025). This range is often referred to as the transition zone, where both mechanisms may be involved (Du *et al.*, 2025). These mechanisms are facilitated by hydrogen bonding,  $\pi$ - $\pi$  stacking, partial electron transfer, electrostatic interactions, pore filling, complex formations, and induced dipole-dipole interactions. Furthermore, the entropies of  $\Delta S^\circ = 149.36$  and  $140.35$  J mol<sup>-1</sup>K<sup>-1</sup> were determined; the positive values indicate increased disorder during adsorption, suggesting that the interaction between the adsorbate and adsorbent leads to a more disordered

system. This could be due to the release of water molecules or other solvent interactions as adsorbates bind to the biochar surface. Moreover, the determined Gibbs free energy is negative at all measured temperatures (Table 13), suggesting that the adsorption process is spontaneous throughout.

**Table 13: Thermodynamic properties variations for progesterone and BPA**

Temperature (K)	$\Delta G^\circ$ (kJmol <sup>-1</sup> )		$\Delta H^\circ$ (kJmol <sup>-1</sup> )	$\Delta S^\circ$ (kJmol <sup>-1</sup> K <sup>-1</sup> )
	BPA	Progesterone		
298	-20.71	-19.67	23.59 <sup>a</sup> , 22.33 <sup>b</sup>	149.36 <sup>a</sup> , 140.35 <sup>b</sup>
308	-22.04	-20.86		
318	-23.68	-23.29		
328	-25.63	-24.48		
338	-26.42	-25.23		

a=progesterone, b=BPA

Similar thermodynamic properties have been reported in other studies on the adsorption of organic endocrine disruptors, where physical forces govern retention rather than chemical bonding (Valenzuela-Calahorro *et al.*, 2004b; Húmpola *et al.*, 2013; Munagapati *et al.*, 2020; Tolkou *et al.*, 2024). Therefore, thermodynamic parameters from this study suggest that progesterone and BPA adsorption onto the HB is a spontaneous, endothermic process dominated by intermediate mechanisms with moderate adsorption energy. This suggests that biochar derived from agricultural waste can effectively remove contaminants from water under favourable thermodynamic conditions.

In conclusion, the hybrid biochar derived from a combination of mango and banana peels exhibited the highest removal efficiency for both progesterone and BPA compared to the biochar produced from the individual peels. This enhanced performance is attributed to the synergistic effect resulting from the integration of the distinct physicochemical properties of each peels type, leading to an improved surface area, pore structure, and an abundance of functional groups favorable for adsorption. The results highlight the effectiveness of hybrid biochar as a superior, low-cost and environmentally friendly adsorbent, offering a promising solution for removing EDCs in wastewater treatment processes.

#### **4.4 To Evaluate the Competitive Adsorption Behaviour of Co-Existing Progesterone and Bisphenol A in Synthetic and Real Wastewater Matrices using the Optimized Hybrid Biochar**

In real wastewater systems, multiple contaminants often coexist and may compete for available adsorption sites, potentially affecting the overall removal performance of an adsorbent. This

section evaluates the competitive removal behaviour of BPA and progesterone when present simultaneously in aqueous solution. The interaction between the two EDCs is critical to understanding the practical applicability of the hybrid biochar in complex wastewater matrices. By assessing removal efficiency under co-contaminant conditions, insights can be drawn into adsorption selectivity, potential inhibitory effects, and the overall strength of biochar in multi-pollutant environments.

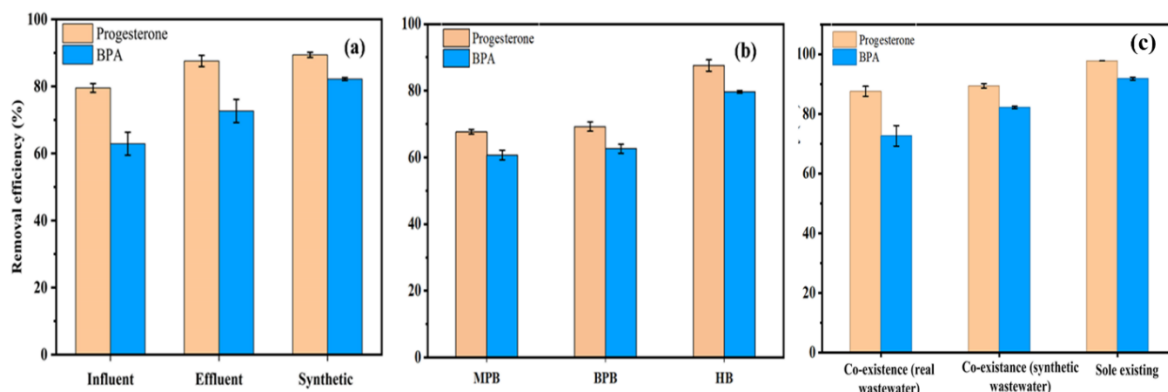
#### **4.4.1 Removal Efficiency of Hybrid Biochar in Wastewater**

Results from this study show that the removal efficiency of HB for progesterone and BPA in complex matrices and synthetic wastewater demonstrates strong performance, especially for progesterone. The removal efficiency was evaluated in various water matrices, including influent, effluent and synthetic wastewater, to assess its performance under different environmental conditions. In synthetic wastewater, which represents a controlled environment with minimal interference, biochar exhibited the highest removal efficiencies, 89.40% for progesterone and 81.20% for BPA, which was slightly lower compared to those observed when EDCs were applied in their sole form (Fig. 31c). This indicates that HB has a strong adsorption efficiency when not hindered by competing substances or background organic matter, which presumably compete for the accessible active sites on the surface of the HB (Tang *et al.*, 2022; Kamaraj *et al.*, 2024).

In contrast, the influent removal was  $79.50 \pm 1.31\%$  for progesterone and  $62.90 \pm 3.41\%$  for BPA, while the effluent exhibited improved performance with  $87.60 \pm 1.68\%$  and  $72.70 \pm 3.40\%$  for progesterone and BPA, respectively (Fig. 31a). The comparison of removal efficiency between real wastewater and synthetic wastewater showed a statistically significant difference, with a *p*-value of 0.04 and 0.041 for BPA and progesterone respectively. This suggests that the matrix of real wastewater, which typically contains a more complex mixture of organic and inorganic constituents, may affect the adsorption performance of the biochar. Therefore, settling prior to the effluent stage may enhance adsorption, possibly due to reduced interference (Rathi & Kumar, 2021; Lin *et al.*, 2022).

The HB demonstrated a significantly higher removal efficiency compared to biochar derived from banana or mango peels alone, with a *p*-value of 0.049 (Fig. 31b), indicating statistical significance at the 5% level. Combining banana and mango peels likely enhances the overall surface chemistry and structure of the biochar, leading to synergistic effects that improve

adsorption. Each peel type may contribute different functional groups and structural characteristics that complement each other.



**Figure 30:** The comparison on the HB efficiency in: (a) Influent, effluent and synthetic wastewater, (b) Solely and hybrid biosorbent forms, and (c) EDCs co-existence in real, synthetic and when applied individually

Furthermore, progesterone consistently showed higher removal than BPA across all matrices, likely due to its stronger affinity for the biochar surface through mechanisms such as electrostatic attraction and hydrogen bonding (Gao *et al.*, 2023). Moreover, higher removal efficiency in progesterone may be facilitated by the compacted structure, which simplifies the diffusion rate toward the active sites. The removal rates of this study are consistent with those of other studies using modified biochar (Amusat *et al.*, 2023b; Kamaraj *et al.*, 2024) and are slightly higher than those obtained by Ngeno *et al.* (2023). Highlighting HB suitability as a low-cost, efficient adsorbent for EDCs in water treatment since it is even better in the absence of chemical modifications.

#### 4.4.2 Effect of Physicochemical Parameters of Wastewater on Endocrine Disrupting Compounds Removal Efficiency

Several physicochemical parameters influence the adsorption efficiency of progesterone and BPA onto biochar in wastewater. A slightly alkaline pH of 7.60 has a slight effect on progesterone adsorption but may reduce BPA uptake due to its partial ionization and resulting electrostatic repulsion from the biochar surface (Almakhathi *et al.*, 2022). The moderate temperature of 21.50°C may slightly slow down adsorption kinetics but does not significantly hinder performance. High electrical conductivity (918  $\mu\text{Scm}^{-1}$ ), total solids (60.80  $\text{mg L}^{-1}$ ), total suspended solids (78.30  $\text{mg L}^{-1}$ ), and turbidity (180.70 NTU) indicate a complex matrix with a high load of competing ions and particulates, which can block adsorption sites, shield pollutants and reduce overall removal efficiency. Additionally, an unusually high dissolved oxygen level of 15.70  $\text{mg L}^{-1}$  may lead to the oxidative degradation of BPA and alter the biochar

surface by increasing polarity, potentially decreasing progesterone adsorption due to reduced hydrophobic interactions (Ojogoro *et al.*, 2017).

#### **4.4.3 Wastewater Physicochemical Parameters and Endocrine Disrupting Compounds Removal Challenges**

The presence of organic matter, nutrients, and inorganic ions in real wastewater significantly influences the adsorption of progesterone and BPA onto biochar. As shown in Table 14, the elevated COD and BOD values indicate a high organic load, which competes with the target pollutants for available adsorption sites and may cause pore blockage, thereby reducing adsorption efficiency (Godiya & Park, 2022). Nutrients such as  $\text{PO}_4^{3-}$ ,  $\text{NO}_3^-$  and nitrite  $\text{NO}_2^-$  introduce additional anions that can interfere with BPA adsorption by competing for active sites and altering surface charge and electrostatic interactions (Dinger, 2021). Multivalent cations, particularly  $\text{Ca}^{2+}$ , may enhance BPA adsorption through bridging interactions with negatively charged functional groups on the biochar surface, but they can also precipitate with phosphates or carbonates, fouling the sorbent surface and ultimately reducing adsorption performance (Hadoudi *et al.*, 2021).

In contrast, monovalent ions such as  $\text{K}^+$  mainly increase ionic strength and have a comparatively minor influence on the adsorption of both BPA and progesterone due to their weaker complexation ability (Ifelebuegu & Onwugbuta, 2016). Overall, these matrix components reduce adsorption efficiency in real wastewater, with BPA being more affected due to its higher polarity and ionisability, while progesterone is less affected due to its more hydrophobic nature.

Furthermore, the physicochemical characteristics of the real wastewater were determined before spiking, after spiking with BPA and progesterone and after biosorption treatment. The addition of the target pollutants resulted in negligible changes in bulk water quality parameters, including temperature, pH, conductivity, dissolved oxygen, total solids, total suspended solids,  $\text{BOD}_5$  and major ions. However, significant reductions in COD, BOD, nutrients ( $\text{NO}_3^-$ ,  $\text{NO}_2^-$ ,  $\text{PO}_4^{3-}$ ), conductivity and selected cations ( $\text{Ca}^{2+}$  and  $\text{K}^+$ ) were observed after adsorption, indicating that the biochar not only removed the endocrine-disrupting compounds but also improved the overall wastewater quality. These findings are consistent with observations reported in related studies (Walanda *et al.*, 2022; Bhuvanendran *et al.*, 2024).

**Table 14: Wastewater physiochemical parameters in raw wastewater, after spiking and after EDCs adsorption using EDCs**

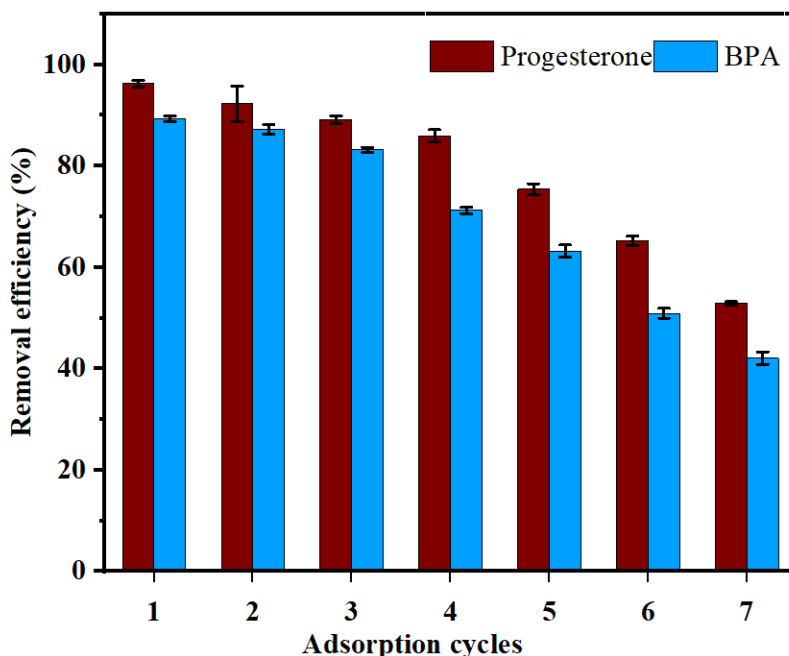
Parameters	Raw Wastewater	Spiked BPA & Progesterone	After Adsorption	SI unit
pH	7.60 ± 0.01	7.62 ± 0.09	7.61 ± 0.02	
Temperature	21.50 ± 0.50	21.51 ± 0.20	21.60 ± 0.06	°C
EC	918 ± 1.01	923 ± 2.21	886 ± 0.91	Sm cm <sup>-1</sup>
TS	60.80 ± 0.65	70.90 ± 0.11	59.90 ± 0.18	mg L <sup>-1</sup>
TSS	78.30 ± 0.90	83.50 ± 0.21	80.09 ± 0.35	mg L <sup>-1</sup>
Turbidity	180.70 ± 2.10	181.80 ± 3.10	173.90 ± 1.99	mg L <sup>-1</sup>
DO	15.70 ± 0.03	14.90 ± 0.01	13.70 ± 0.40	mg L <sup>-1</sup>
COD	45.50 ± 0.02	60.20 ± 0.04	49.19 ± 0.31	mg L <sup>-1</sup>
BOD	23.60 ± 0.01	26.60 ± 0.35	19.23 ± 0.91	mg L <sup>-1</sup>
Phosphate (PO <sub>4</sub> <sup>3-</sup> )	6.80 ± 0.03	6.90 ± 0.01	3.90 ± 0.05	mg L <sup>-1</sup>
Nitrate (NO <sub>3</sub> <sup>-</sup> )	19.00 ± 0.10	19.20 ± 0.04	17.21 ± 0.91	mg L <sup>-1</sup>
Nitrite (NO <sub>2</sub> <sup>-</sup> )	30.50 ± 0.09	30.70 ± 0.01	23.91 ± 0.23	mg L <sup>-1</sup>
Calcium (Ca <sup>2+</sup> )	50.70 ± 0.18	51.81 ± 0.20	39.82 ± 0.42	mg L <sup>-1</sup>
Potassium (K <sup>+</sup> )	9.20 ± 0.02	10.41 ± 0.07	7.92 ± 0.01	mg L <sup>-1</sup>

Therefore, the competitive removal efficiency study revealed that HB exhibited higher removal efficiencies for both BPA and progesterone in synthetic wastewater compared to real wastewater. This difference is likely due to the presence of various competing substances and background impurities in real wastewater, which can hinder adsorption by occupying active sites or interfering with the adsorption mechanism. In addition, notable changes in physicochemical parameters such as reductions in COD, BOD, nutrients, electrical conductivity and selected cations were observed after treatment, indicating that the adsorption process not only removed the target EDCs but also contributed to improving overall wastewater quality. Nevertheless, the HB maintained a commendable removal performance under both conditions, confirming its potential as a robust and efficient adsorbent for treating complex wastewater containing multiple EDCs.

#### **4.5 To Assess the Regeneration and Reusability of the Developed Hybrid Biochar Materials for Sustainable Wastewater Treatment Applications**

Assessing the regeneration and reusability of the developed hybrid biochar is a critical step toward determining its practicality for long-term, sustainable wastewater treatment. Beyond achieving high initial adsorption performance, an effective adsorbent must maintain its efficiency over multiple use cycles while minimising material consumption, operational costs and secondary waste generation. This objective, therefore, focuses on evaluating the hybrid biochar's ability to retain adsorption capacity after successive adsorption–desorption cycles, providing insight into its durability, stability and potential for repeated use in real wastewater treatment systems.

The regeneration and reusability performance of the hybrid biochar demonstrates its potential for long-term application in wastewater treatment. In this study, the biochar maintained high adsorption efficiency even after more than four consecutive adsorption–desorption cycles, indicating strong structural and chemical stability (Fig. 31).



**Figure 31:** The adsorption efficiency cycles for hybrid biochar on the progesterone and BPA removal

Furthermore, the high initial adsorption efficiency suggests that the biochar is highly effective at capturing progesterone and BPA from aqueous solutions. As the cycles progress, efficiencies decrease gradually, possibly because some of the biochar's active sites become saturated or blocked by adsorbed pollutants, resulting in reduced efficiency (Barquilha & Braga, 2021). Furthermore, efficiency decreases are facilitated by the gradual loss of active functional groups on the surface, and incomplete desorption of previously adsorbed BPA and progesterone molecules during the regeneration process.

Despite this minor decline, the sustained performance over multiple cycles suggests that the pore structure and surface chemistry of the hybrid biochar remain largely intact. The persistence of key functional groups, such as hydroxyl, carboxyl and aromatic structures, likely contributes to repeated adsorption via hydrogen bonding,  $\pi$ - $\pi$  interactions and hydrophobic interactions. Moreover, the co-pyrolysis of banana and mango peels may have created a more robust carbon matrix with improved resistance to physical and chemical degradation during regeneration.

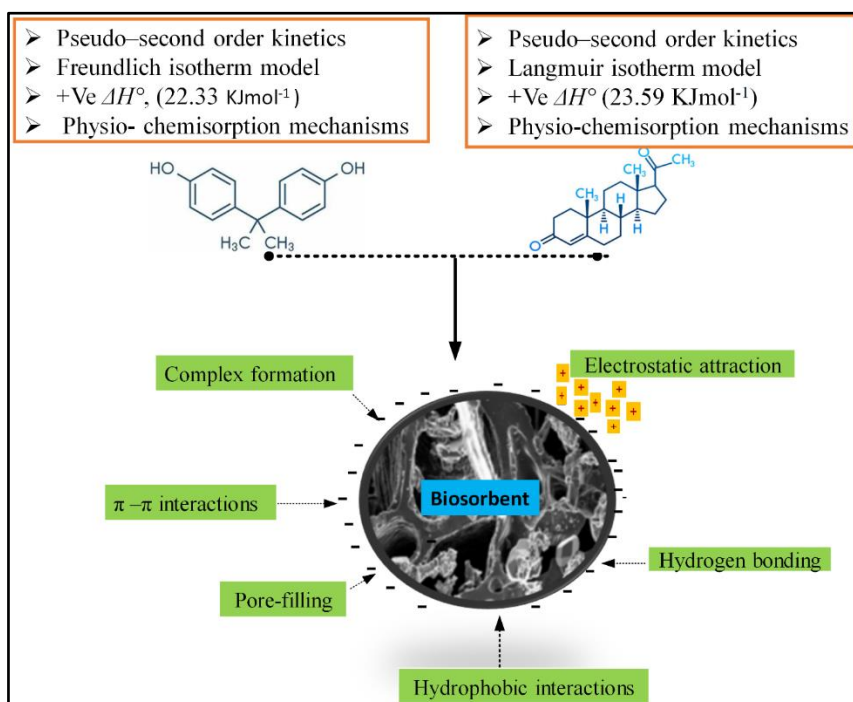
The biochar's ability to retain significant adsorption capacity across multiple cycles highlights its economic and environmental advantages, as it reduces the need for frequent replacement and minimises waste generation. This confirms that the hybrid biochar is not only effective in removing EDCs compounds but is also suitable for sustainable, large-scale wastewater treatment applications where repeated use of the adsorbent is essential. Results from this study is higher than those studied by Liu *et al.* (2017) with algae-based materials, which maintained 60% at 6 cycle for BPA removal. While the use of fungal-based appeared to perform 85% at 3 cycle for removal of progesterone (Abdelwahab *et al.*, 2025).

In this study, to degrade the adsorbed endocrine-disrupting compounds (EDCs), the spent biosorbent was regenerated by heating at 110°C for one hour which enabled degradation and desorption of the contaminants and restored its adsorption performance for subsequent cycles. After repeated use, the spent biochar can be safely managed through stabilization or incorporation into construction materials to prevent secondary pollution. This approach supports circular wastewater management by reducing waste, enabling material reuse, and minimizing environmental risks.

#### **4.6 Hybrid Biochar on Progesterone and Bisphenol-A Major Removal Mechanisms**

Hybrid biochar can effectively remove progesterone using various mechanisms facilitated by its physical and chemical properties (Fig. 32). Hybrid biochar contains various functional groups, such as C=O, O-H, C≡N, C-H, C≡C and C=C, which create active sites on the biochar surface for hormone binding and influence the various adsorption mechanisms of progesterone and BPA. Furthermore, availability of different inorganic elements such as K, Ca, Mg, Na, Si and P evidenced in Fig. 14a on the biochar surface provides additional removal mechanisms for organic contaminants.

Electrostatic attraction mechanisms play a vital role in progesterone adsorption processes facilitated by accessed environmental conditions. At basic conditions, the C-O group of the HB surface deprotonates, creating a negative charge surface that favoring adsorption of neutral or positively polarized regions of progesterone, leading to electrostatic attraction (Sippola, 2024). Also, the availability of a hydrogen atom in the hydroxyl group and the polarity of the carbonyl group in progesterone cause the O-H groups found on the hybrid biochar surface to form hydrogen bonds with the carbonyl oxygen of the progesterone and O-H groups of BPA (Vieira *et al.*, 2022).



**Figure 32: Major mechanisms applied for hybrid biochar on progesterone and BPA removal**

Likewise, HB large surface area and availability of mesoporous structure make the pore-filling mechanisms possible for the physical adsorption of progesterone and BPA molecules. During this, the porous structure of the biochar combines with the C-O and O-H groups of progesterone and BPA by van der Waals forces (Gao *et al.*, 2023), enhancing the entrapment of pollutants into the biochar pores. Moreover, the presence of metal ions on the biochar surface at the basic medium causes progesterone and BPA to form a surface complexation with metal ions or carbonates on the biochar surface, forming a stable complex that enhances the hormone's retention on the biochar surface, leading to improved removal rates.

Furthermore, progesterone is a hydrophobic molecule; thus, its removal can be enhanced through hydrophobic interaction mechanisms with the graphene structure of biochar. The biochar's aromatic rings and hydrophobic surfaces provide favourable conditions for such interactions, accompanied by the diffusion of pollutants into pores and non-carbonized materials. Therefore, understanding these mechanisms is critical for optimizing biochar production and application strategies in environmental remediation efforts targeting endocrine disruptors. Likewise,  $\pi$ - $\pi$  interactions with aromatic structures in biochar and the aromatic rings of BPA and progesterone via  $\pi$ - $\pi$  stacking (Michenzi *et al.*, 2024).

Therefore, removal of BPA and progesterone by HB is governed by a combination of chemisorption and physisorption mechanisms facilitated by both organic functional groups and inorganic elements. The functional groups enable  $\pi$ - $\pi$  electron donor-acceptor interactions,

hydrogen bonding and hydrophobic interactions, while the inorganic components contribute through metal–ligand complexation and electrostatic attraction, resulting in a multifunctional and synergistic adsorption system highly effective for the removal of diverse contaminants.

## CHAPTER FIVE

### CONCLUSION AND RECOMMENDATIONS

#### 5.1 Conclusion

Biochar derived from isolated banana and mango peels exhibits slightly higher adsorption efficiency and favourable physical characteristics, including a well-developed pore structure and surface functional groups, which significantly contribute to its performance in removing progesterone and BPA. Therefore, this makes banana and mango agro-waste-derived biochar cost-effective, eco-friendly and sustainable alternatives for mitigating EDCs in wastewater treatment applications.

The HB exhibited the highest removal efficiency for both progesterone and BPA compared to the biochar produced from the individual peels. This enhanced performance is attributed to the synergistic effect resulting from the integration of the distinct physicochemical properties of each peels type, leading to an improved surface area, pore structure and balanced functional groups that are favourable for adsorption. The results emphasize the effectiveness of hybrid biochar as a superior and environmentally friendly adsorbent, offering a promising solution for removing EDCs in wastewater treatment processes.

Characterization results revealed that hybridization significantly increased the surface area and porosity of the resulting biochar, providing more accessible adsorption sites. Additionally, the hybrid biochar possessed a higher density of functional groups, including hydroxyl, carboxyl and aromatic rings. These features promote stronger interactions with EDC molecules through hydrogen bonding, electrostatic interactions and  $\pi$ - $\pi$  electron exchange, thereby enhancing adsorption capacity.

Adsorption kinetics for both BPA and progesterone were best described by the pseudo-second-order model, indicating that chemisorption governs the removal process. This suggests that adsorption is primarily controlled by the availability of active sites and involves valence forces through electron sharing or exchange, rather than simple physical attraction. Thermodynamic analysis further supported these findings, with negative  $\Delta G^\circ$  values confirming the spontaneity of the process and positive  $\Delta H^\circ$  values indicating its endothermic nature. The intermediate adsorption energy values suggest a dual mechanism dominated by strong physisorption and weak chemisorption collectively referred to as physico-chemisorption.

The competitive removal efficiency study revealed that the HB exhibited higher removal efficiency for both BPA and progesterone in synthetic wastewater compared to real wastewater. This difference is likely due to the presence of various competing substances and background impurities in real wastewater, which can hinder adsorption by occupying active sites or interfering with the adsorption mechanism. Nevertheless, the HB maintained a commendable removal performance under both conditions, confirming its potential as a robust and efficient adsorbent for treating complex wastewater containing multiple endocrine-disrupting compounds.

Particularly, the HB retained high adsorption efficiency over more than four consecutive adsorptions–desorption cycles, indicating excellent reusability and operational stability. This durability enhances its practical applicability in real-world wastewater treatment systems, reducing operational costs and environmental impact.

Therefore, the use of mixed agricultural residues to produce HB not only improves adsorptive performance but also exemplifies a scalable, low-cost, and environmentally sustainable approach to water purification. The study not only adds scientific value to the field of environmental remediation but also supports the broader goal of promoting circular economy strategies by transforming agro-waste into high-value materials. Furthermore, this study aligns with global sustainability goals by promoting pollution reduction, fostering green innovation, and advancing access to clean water through resource-efficient and nature-based solutions.

## **5.2 Recommendations**

Based on the findings of this study, the following recommendations are proposed to guide future research and practical applications regarding the use of hybrid biochar for EDCs remediation:

- (i) To increase removal efficiency, particularly toward a target of 100% EDC elimination, future studies should employ chemical and thermal activation methods. Techniques such as metal oxide impregnation, steam activation, or acid/base treatments should be explored to improve the pore structure, surface area, and availability of functional groups on the biochar.
- (ii) While this study focused on BPA and progesterone, future work should include a broader spectrum of EDCs and emerging pollutants, including pharmaceutical residues,

pesticides, plasticizers and industrial chemicals, to fully evaluate biochar's versatility and capacity for multi-contaminant removal.

- (iii) Since the current study was conducted under controlled laboratory settings, future research should scale up to full-scale field experiments. Such studies should assess the real-world performance of biochar under actual wastewater treatment scenarios, comparing it with existing technologies and considering operational variability.
- (iv) Though mechanisms such as electrostatic attraction, hydrophobic interactions, and hydrogen bonding are known contributors, their relative contributions and dynamics remain poorly defined. Further research should aim to quantitatively unravel these mechanisms under varying environmental conditions and pollutant types, thereby refining adsorption models and biochar design.
- (v) More research is needed to evaluate the environmental implications of long-term biochar usage, especially concerning secondary pollution risks, its interaction with natural organic matter and potential effects on aquatic organisms and ecosystems. This will ensure that the use of biochar is both practical and environmentally responsible.
- (vi) Furthermore, more study is required to add knowledge on the regulatory frameworks governing the use of biochar in environmental applications, particularly concerning its use for contaminant removal. This can provide biochar technology's widespread adoption.
- (vii) Future studies should investigate the regeneration performance of HB over more than four adsorptions–desorption cycles to better determine its long-term reusability and stability. While the current results demonstrate that HB retains a considerable portion of its adsorption capacity within the first four cycles, extended cycling is necessary to evaluate potential structural degradation, loss of active sites and changes in surface chemistry that may occur under repeated regeneration.
- (viii) In addition, a comprehensive cost analysis should be conducted to evaluate the economic feasibility of using HB, including costs related to raw material collection, biochar production, activation, regeneration and disposal, in comparison with conventional adsorbents. This will provide a clearer understanding of HB's practical applicability for large-scale wastewater treatment.

- (ix) Future studies should include an Environmental Impact Assessment (EIA) of HB across its entire life cycle, considering emissions, energy use, leaching risks, and the fate of adsorbed pollutants to ensure its environmental safety and sustainability during large-scale application.

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## RESEARCH OUTPUTS

### (i) Publications

Kasambala, H. R., Rwiza, M. J., Mpumi, N., Mwema, M. F., & Njau, K. K. (2025). Biochars derived from banana and mango peels in isolated systems revealed high removal efficiency of endocrine-disrupting compounds from water. *Biomass Conversion and Biorefinery*, *15*(9), 13575-13588.

Kasambala, H. R., Rwiza, M. J., Mpumi, N., Mwema, M. F., & Njau, K. N. (2025). Agro-Waste-Derived Hybrid Biochar Exhibits High-Efficiency Removal of Endocrine-Disrupting Compounds. *Water, Air, & Soil Pollution*, *236*(5), 323.

Kasambala, H. R., Rwiza, M. J., Mpumi, N., Mwema, M. F., Machunda, R., Mtei, K., & Njau, K. N. (2024). A comprehensive review on the distribution of per-and poly-fluoroalkyl substances in the environment across Sub-Saharan Africa revealed significant variation in their concentrations. *Environmental Challenges*, *16*, 100975.

### (ii) Poster presentation