FUDMA Journal of Sciences (FJS)
ISSN online: 2616-1370
ISSN print: 2645 - 2944
Vol. 9 No. 2, February, 2025, pp 13 - 22
DOI: https://doi.org/10.33003/fjs-2025-0902-3037

ONE-POT BIOSYNTHESIS AND STRUCTURAL ELUCIDATION OF IRON-BIOCHAR NANOCOMPOSITES FROM WASTE COCONUT SHELLS

[1]Eche, P. I., *[1,2]Shaibu S. E., [1]Uwah, E. I. and [1]Moses, E. A.

[1]Department of Chemistry, University of Uyo, Uyo, Nigeria
[2]International Center for Energy and Environmental Sustainability Research (ICEESR)

*Corresponding authors’ email: shaibusolomon@uniuyo.edu.ng

ABSTRACT

This study explores the eco-friendly one-pot biosynthesis and structural characterization of iron-biochar nanocomposites (IBN) derived from waste coconut shells using *Allamanda cathartica* extract as a reducing agent. The synthesis process leveraged the phytochemical abundance of the plant extract and the porous structure of coconut biochar, resulting in a material with enhanced properties for environmental applications. Characterization techniques such as BET, XRD, SEM, HRTEM, EDX, and FTIR confirmed the successful integration of nanoscale iron into the biochar matrix. The BET analysis revealed a surface area of 34.035 m^2/g, a pore size of 102.2356 Å**,** and a pore volume of 0.110914 cm^3/g, indicating high adsorption potential. XRD patterns confirmed crystalline iron oxide phases, while SEM and TEM images revealed highly porous structures with uniformly dispersed iron nanoparticles. The FTIR identified OH, C-O and aromatic C=C stretching, and EDX confirmed the elemental composition, including iron, oxygen, silicon, aluminum, and carbon, ensuring structural stability and pollutant binding efficiency. Additionally, the phytochemical analysis of *Allamanda cathartica* extract identified alkaloids, flavonoids, and steroids, supporting its role as a green reducing agent. The study highlights the structural and functional advantages of IBN over unmodified biochar, emphasizing its increased surface area, porosity, and pollutant removal efficiency. Furthermore, the scalability and sustainability of the synthesis process underscore its potential for large-scale environmental applications, aligning with circular economy principles.

Keywords: Iron-biochar nanocomposite, *Allamanda cathartica*, Coconut-derived biochar, One-pot biosynthesis

INTRODUCTION

The pursuit of sustainable and environmentally friendly materials has sparked considerable interest in repurposing biomass waste to develop advanced functional materials. Biochar, a carbon-rich material produced via the pyrolysis of biomass, has gained recognition as a promising candidate for a wide range of environmental applications due to its abundance, cost-effectiveness, and versatility (Zhu *et al.,* 2019). This material is produced by heating organic biomass in the absence of oxygen, which results in a porous structure with a high surface area and a range of functional groups, making biochar an excellent candidate for pollutant adsorption, water filtration, and soil enhancement.

A key area of interest in biochar-based materials is the synthesis of iron-biochar nanocomposites from waste coconut shells. These shells, abundant in tropical regions as byproducts of coconut processing, present a valuable and renewable resource for biochar production. The incorporation of iron into biochar enhances its physicochemical properties, particularly through the formation of iron nanoparticles. Iron, known for its catalytic and magnetic properties, imparts additional functionalities to biochar, such as increased adsorption capacities, improved reactivity, and enhanced catalytic activities (Ahmad *et al.,* 2022; Essien *et al.,* 2018; Perveen *et al*., 2022; Shaibu *et al*., 2021). This combination makes iron-biochar nanocomposites highly effective for a range of environmental applications, particularly in water and wastewater treatment, soil decontamination, and air purification, where their high surface area and surface reactivity allow for efficient pollutant removal and environmental remediation (Ahmed *et al.*, 2022; Obinna and Ebere, 2019; da Silva *et al.*, 2024).

Among various preparation methods, one-pot biosynthesis approaches have gained significant attention due to their simplicity, efficiency, and ability to simultaneously functionalize biochar with iron nanoparticles. These techniques often involve combining a biochar precursor (e.g., coconut shells) with an iron precursor in a single-step reaction. This method significantly reduces synthesis time and complexity, while maintaining the ability to modify the surface characteristics of biochar, such as its porosity, surface charge, and reactivity (Shaibu *et al.,* 2014; Liu *et al.,* 2019). Furthermore, one-pot synthesis can help control the size, distribution, and dispersion of iron nanoparticles on the biochar surface, which is critical in determining the material's performance in pollutant adsorption and catalysis.

According to Ying et al. (2021), optimizing the one-pot biosynthesis method further could enhance the performance of iron-biochar nanocomposites. Key parameters such as pyrolysis temperature, reaction time, and the concentration of the iron precursor have been shown to play significant roles in shaping the material's structure and functionality. For instance, higher pyrolysis temperatures can increase the surface area of biochar, while adjusting the iron precursor concentration can regulate the formation of iron nanoparticles, leading to enhanced adsorption and catalytic activities (De Lima *et al.,* 2021). By fine-tuning these synthesis conditions, researchers can tailor iron-biochar nanocomposites to meet specific performance requirements, opening up new avenues for their use in diverse environmental applications.

Moreover, considering the cost-effectiveness and sustainability of production, one-pot biosynthesis offers a highly practical route for large-scale production of iron-biochar nanocomposites. The utilization of waste coconut shells in the production process not only helps reduce agricultural waste but also provides a sustainable, low-cost feedstock for biochar. This aligns with the growing demand for environmentally friendly technologies that minimize waste and promote the recycling of natural resources (da Silva *et al.,* 2024; Nunes *et al.,* 2020; Ramya *et al.,* 2022). In addition, the reduced environmental footprint of one-pot synthesis, with fewer reagents and energy-intensive steps, makes it a more sustainable option compared to traditional methods, further supporting its commercial viability (da Silva *et al., 2*024; Shaibu *et al., 2*022; Zhao *et al., 2*019).

This study focuses on the one-pot biosynthesis of iron-biochar nanocomposites from waste coconut shells, aiming to establish an eco-friendly and sustainable method for nanocomposite production.

MATERIALS AND METHODS

Collection of Samples and Preparation of Biochar

Waste coconut shells were collected from Akpanadem market in Uyo, Akwa Ibom State, Nigeria. The coconut shells were air-dried for a 7 days before being crushed and ground into fine particles with a diameter of less than 0.18 mm. The powder was then filled to the top of a ceramic saucepan and covered with a lid. Pyrolysis was performed in an oxygen-limited environment using a muffle furnace (KSW 12-11, Shanghai Laboratory Equipment Company Limited, China) at an initial temperature of 100 °C for one hour. This was followed by maintaining a constant temperature of 300 °C for six hours, with a gradual increase of 20 °C per minute. The biochar was then allowed to cool to room temperature, crushed, and sieved through a 0.18 mm mesh screen. The resulting biochar was washed with 0.1 M HCl and cleaned with deionized water until the pH of the liquid phase became neutral, as outlined by Zhu *et al*. (2019) and Adebayo *et al.,* *2*019.

Extract preparation and phytochemical Analysis of *Allamanda cathartica* leaves

Fresh leaves of *Allamanda cathartica* were sourced from the Akpanadem market located in Uyo Local Government Area (LGA) of Akwa Ibom state, Nigeria. The collected plant material was transported to the chemistry laboratory at the University of Uyo, Uyo, Nigeria. The leaves samples were thoroughly washed with distilled water, cut into small pieces, and air-dried. Subsequently, the dried leaves were finely ground and subjected to maceration by immersing 60 g of the powdered material in water for 48 hours with slight heating at 50 °C for 20 minutes. The extract was filtered and used for the synthesis of IBN. Additionally, extraction was done in methanol following previously stated preliminary steps, eventually, the solvent was removed using a rotary evaporator, yielding a crude extract. This extract was then utilized for phytochemical analysis, as described by Reethu and Dhabal (2024) and Enin *et al., 2*023, to assess the presence of various bioactive compounds.

Preparation of impregnated coconut Iron Nanocomposite

The synthesis of IBN was carried out using the eco-friendly solvent impregnation method detailed by Karunaratne *et al*. (2022). The process involved combining $Fe(NO_3)_3$ with coconut biochar particles. A mixture containing 3.6 g of $Fe(NO_3)_3\cdot9H_2O$ and 10 g of coconut biochar was prepared in a beaker, and stirred for 2 hours to ensure sufficient contact between the iron solution and the biochar. Subsequently, 100 mL of *Allamanda cathartica* extract was added to the slurry and slowly heated to about 50 °C with constant stirring for 2 hours. The eventual solution was filtered, dried and pulverized to get the IBN.

Characterization of IBN

Different techniques were employed to characterize the prepared the IBN. Fourier Transform Infrared (FTIR) spectra were obtained using a Bruker Platinum ATR Tensor 27 spectrometer in single mode at 4 cm^{-1} resolution, with a range of 4000-300 cm^{-1} and an average of 32 scans per sample. X-ray diffraction (XRD) was utilized for identifying unknown crystalline materials (e.g., minerals and inorganic compounds), characterizing crystalline materials, measuring sample purity, and determining unit cell dimensions. A Bruker multifunctional Powder x-ray diffractometer D8-Advance was employed using Cu-Kα radiation (λ= 0.1506 nm). The analysis was performed using locked coupled mode scanning at 40 kV, 30 mA, and a scanning speed of 5°/min. The size, shape, and surface morphology of the catalysts were assessed using a TESCAN Vega 3 LMH Scanning Electron Microscope (SEM) with a working distance of 15 mm and a voltage of 20 kV. Image acquisition was carried out at various magnifications (10, 20, 50, and 100 µm) with the samples mounted on a carbon tape-fast sample holder. High-Resolution Transmission Electron Microscopy (HRTEM) and Energy Dispersive X-ray Spectroscopy (EDS) were employed to determine the composition and distribution of the elements.

RESULTS AND DISCUSSION

Phytochemical Analysis of *Allamanda cathartica* Extract

The phytochemical analysis of *Allamanda cathartica* extract revealed the presence of several bioactive compounds in the plant as presented in Table 1.

Table 1: Preliminary Phytochemical Analysis of *Allamanda cathartica*

Phytochemical	Test	Result
Alkaloids	Iodine Test	+
Flavonoids	Alkaline Reagent Test	+
Steroids	Chloroform Test	+
Saponins	Frothing Test	-
Tannins	Ferric Chloride Test	-
Glycosides	Keller-Killani Test	-

The results indicated the presence of alkaloids in the plant, which aligns with the findings of Shaikh and Patil (2020). Alkaloids are nitrogenous compounds with potential pharmacological activities, including antimicrobial and anti-inflammatory effects (Enin *et al., 2*021 & Pandey and Kumar, 2024). The result also revealed the presence of flavonoids in the plant. Flavonoids are polyphenolic compounds known for their antioxidant, anti-inflammatory, and anticancer activities (Yuan *et al., 2*024). The presence of steroids in the plant was also revealed from the results, including anti-inflammatory and immunomodulatory effects (Shaikh and Patil, 2020). As shown in Table 1, it was observed that saponins were absent in *Allamanda cathartica*. Saponins are known for their anti-inflammatory and antimicrobial properties (Saravanakumar *et al.,* 2020).

The result also revealed the absence of tannins in the plant, although tannins have both antioxidant and antibacterial properties. From the results, glycosides were also in

Allamanda cathartica. This finding was not in agreement with Fraga-Corral *et al.*,(2021), where glycosides constituted 3% of *P. abies*, with essential oils accounting for 2% and flavone glycosides also being present. Glycosides are known for their anti-inflammatory and analgesic properties (Fraga-Corral *et al.*, 2021; Khan *et al.*, 2020). According to Dokhe *et al.* (2023), the presence of alkaloids, flavonoids, and steroids, in the plant can be linked to numerous health benefits, implying that *Allamanda cathartica* may have potential as a medicinal plant. Further investigation into these compounds' properties could lead to new therapeutic and medicinal applications.

Brunauer–Emmett–Teller (BET) surface area

The BET analysis of the iron-biochar nanocomposite (IBN) synthesized from waste coconut shells provided critical insights into its surface area, pore size, and pore volume—parameters pivotal for evaluating its adsorption capabilities as shown in Figure 3.1. The BET-specific surface area of the IBN was determined to be 34.035 m²/g, with a pore size of 102.2356 Å and a pore volume of 0.110914 cm³/g. A higher specific surface area enhances the adsorption capacity of materials by increasing the number of available active sites (Qu *et al.*, 2021). This is particularly beneficial for applications such as pollutant removal, where adsorption is the key mechanism. The measured surface area indicates that the incorporation of iron nanoparticles effectively improved the biochar's structural properties, likely by increasing porosity and creating additional microporous regions. The pore size of 102.2356 Å classifies the material as mesoporous, which is advantageous for adsorbing medium-sized molecules, such as organic pollutants and heavy metals, commonly found in environmental remediation contexts (Shaibu *et al.*, 2014). The pore volume of 0.110914 cm³/g further supports the material's capacity to accommodate a substantial amount of adsorbate molecules. These findings suggest that the one-pot biosynthesis method enhanced the textural properties of the coconut shell-derived biochar by introducing nanoscale iron, which not only augmented the surface area but also optimized pore distribution. This improvement is significant in adsorption-driven applications, as it ensures efficient diffusion of pollutants into the pores while providing ample active sites for binding (Ekman *et al.*, 2023). In addition, the BET analysis demonstrated that the IBN possesses a well-engineered porous structure, making it a promising material for adsorption-based environmental applications, such as water purification and pollutant remediation.

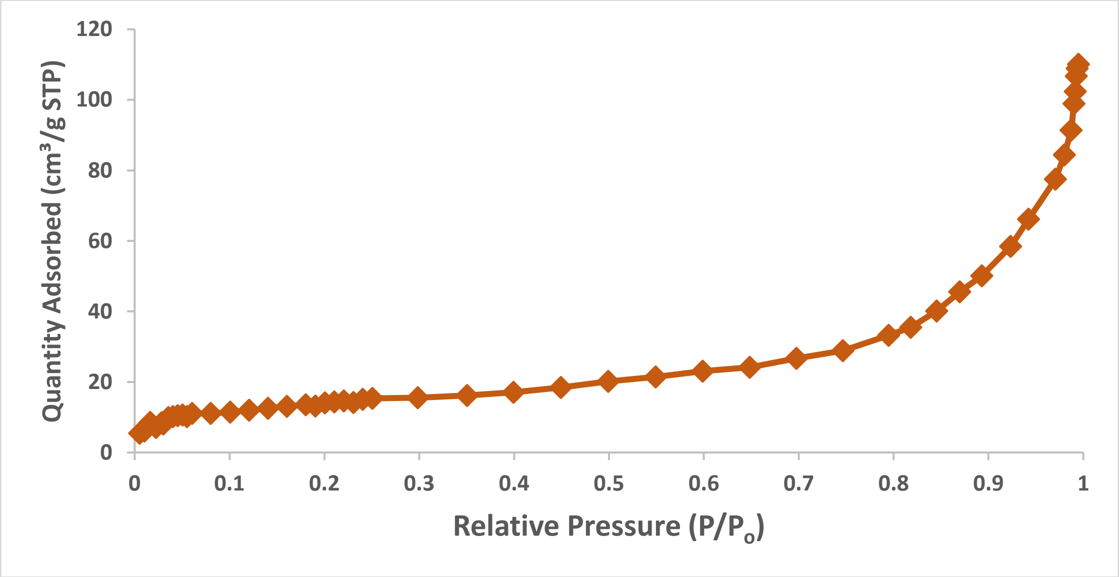

Figure 1: BET of IBN

Transmission electron microscopy (TEM)

The TEM analysis of the iron-biochar nanocomposite (IBN) provided detailed insights into its morphological characteristics, particularly in the nanoscale integration of iron particles within the biochar matrix. The TEM images revealed a highly porous structure with uniformly dispersed iron nanoparticles, indicating effective synthesis via the one-pot biosynthesis method as shown in Figure 3.2. The distinct morphology of the IBN, compared to the precursor coconut biochar, showcased the nanoscale incorporation of iron, which enhances its functional properties for adsorption and catalytic applications. The observed distribution of iron nanoparticles contributes to the increased surface area and active sites, critical for efficient pollutant removal. These nanoscale iron particles are instrumental in enhancing the magnetic properties of the composite, facilitating easy separation and recovery from aqueous solutions—a feature extensively reported in similar studies. For instance, Vishnu et al. (2021) observed similar nanoscale dispersion of iron in biochar composites, which significantly improved adsorption efficiency and magnetic recoverability. Similarly, Ahmad et al. (2022) demonstrated that nanoscale integration of metals in biochar enhanced surface reactivity and pollutant binding capacity. Furthermore, the porous architecture observed in the TEM images aligns with findings by Pang et al. (2021), who reported that biochar composites with well-defined pores and embedded nanoparticles exhibited superior adsorption and catalytic behaviors. The flake-like and microporous structures identified in this study provide pathways for pollutant diffusion and adsorption, aligning with prior research on biochar's enhanced pollutant-binding capabilities (Hussain *et al.*, 2023). In addition, the TEM results confirm the successful integration of nanoscale iron particles within the biochar matrix, enhancing its structural and functional properties for environmental applications. These findings corroborate previous studies, highlighting the potential of nano-engineered biochars in pollutant remediation through nanoscale modifications.

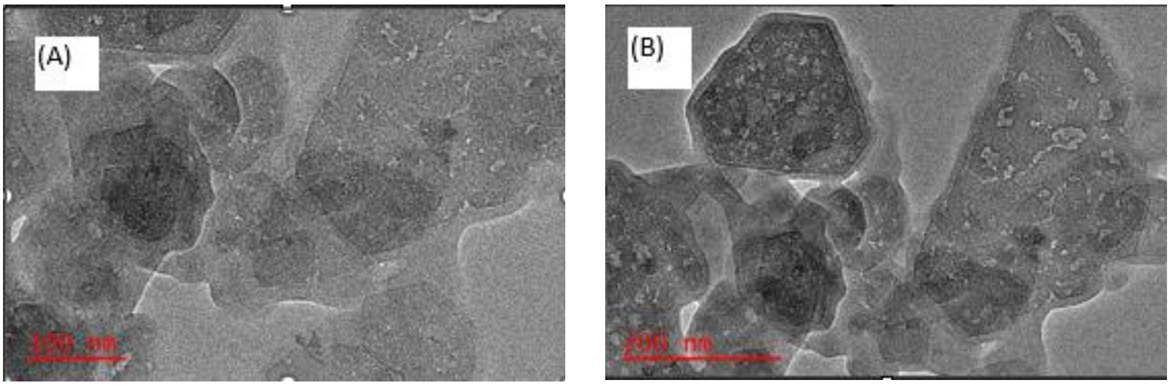

Figure 2: Transmission electron microscopy (TEM) of (a) coconut shell biochar and (b) IBN

Energy-dispersive X-ray spectroscopy (EDX)

The EDX analysis of the iron-biochar nanocomposite (IBN) provided vital information on the elemental composition and distribution within the synthesized material. The analysis revealed the presence of key elements such as oxygen, silicon, aluminum, carbon, and iron as shown in Figure 3. The incorporation of iron within the biochar matrix was particularly significant, confirming the successful synthesis of the nanocomposite through the one-pot biosynthesis method. The presence of carbon and oxygen as predominant elements is characteristic of biochar, derived from biomass pyrolysis, which is widely recognized for its high surface area and functional group diversity (Zhao et al., 2019). These elements are crucial for the adsorption capabilities of the material, as they provide active sites for pollutant binding, a key feature in biochar-based environmental applications (Li et al., 2019). The detection of silicon and aluminum suggests the inclusion of trace elements from the coconut shell precursor, which may enhance the composite's structural stability and contribute to additional adsorption mechanisms (Hoslett et al., 2019). Most notably, the incorporation of iron imparts magnetic properties to the IBN, enabling easy separation and recovery during environmental remediation processes, a feature extensively studied in recent biochar research (Pang et al., 2021). Similar studies have reported that iron-modified biochars exhibit superior adsorption efficiency for heavy metals and organic pollutants, primarily due to their increased reactivity, functionalized surfaces, and enhanced pore structures (Vishnu et al., 2021; Liu et al., 2019). The EDX analysis also highlighted variations in the elemental composition between raw coconut biochar and IBN, further supporting the successful impregnation of iron nanoparticles. This outcome aligns with recent studies demonstrating that iron incorporation improves biochar's pollutant removal capacity, enhances catalytic activity, and broadens its applicability for water and wastewater treatment (Shaibu et al., 2022; Ahmed et al., 2022). Additionally, recent advancements in biochar research suggest that modifying biochar with metals like iron, manganese, and copper can create multifunctional composites with synergistic adsorption and degradation capabilities, making them suitable for more complex environmental challenges (Komkiene & Baltrenaite, 2016; Yallappa et al., 2017).

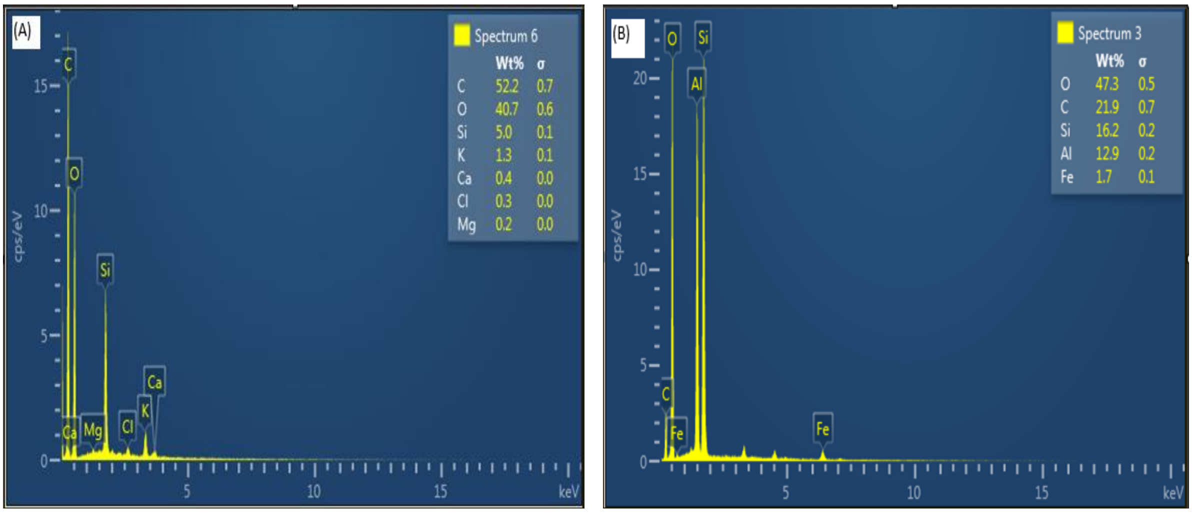

Figure 3: Energy-dispersive X-ray spectroscopy (EDX) analysis of (a) coconut shell biochar (b) IBN

Scanning electron microscopy (SEM)

The Scanning Electron Microscopy (SEM) analysis of the iron-biochar nanocomposite (IBN) revealed critical details about its surface morphology and structural characteristics, highlighting the effects of iron nanoparticle incorporation. SEM images showcased distinct surface features, including increased porosity and a flake-like structure, compared to the raw coconut biochar (Figure 4). The porous structure observed in IBN indicates a significant transformation during synthesis, attributed to the integration of iron nanoparticles. The presence of irregular micropores and macropores in the composite is particularly important for adsorption processes, providing ample active sites for pollutant binding and enhancing diffusion. These findings are in line with earlier research on biochar-based materials, where metal-doped biochars demonstrated enhanced porosity and adsorption capacity due to their modified structural properties (Ahmad and Haseeb, 2015; Simon et al., 2023; Kumar and Saha, 2022). The highly porous nature of IBN results from the release of volatile compounds during pyrolysis, a phenomenon commonly observed in iron-modified biochars, where metal impregnation influences carbonization dynamics and pore evolution (Komkiene & Baltrenaite, 2016; Zhang et al., 2020). The observed surface roughness and heterogeneity introduced by iron incorporation further increased the material's surface area and active binding sites, a feature that has been extensively documented in metal-enhanced biochars synthesized for pollutant removal (Li et al., 2018; Pang et al., 2021). These findings are in strong agreement with the reports by Vishnu et al. (2021) that biochar composites with iron doping exhibited greater textural complexity and higher adsorption efficiencies for heavy metals and organic contaminants, owing to the synergistic effect of biochar's porous structure and iron's catalytic properties. Similarly, submission by Shu et al. (2020) demonstrated that iron-loaded biochar composites facilitate magnetic separation and reusability, providing an advantage in practical applications which further highlights the importance of the current study. The SEM findings further validate the successful synthesis of a structurally optimized composite, reinforcing the established notion that iron incorporation enhances biochar's adsorption efficiency through increased porosity and functionalization. These results contribute to the broader field of biochar research, where metal-enhanced biochars are increasingly recognized for their potential in environmental remediation, water purification, and soil detoxification (Hussain et al., 2023; Shaibu et al., 2017). Thus, the current study aligns with recent advancements in engineered biochar materials, demonstrating that iron-modified biochar from coconut shells can serve as an efficient, scalable, and sustainable solution for pollutant removal.

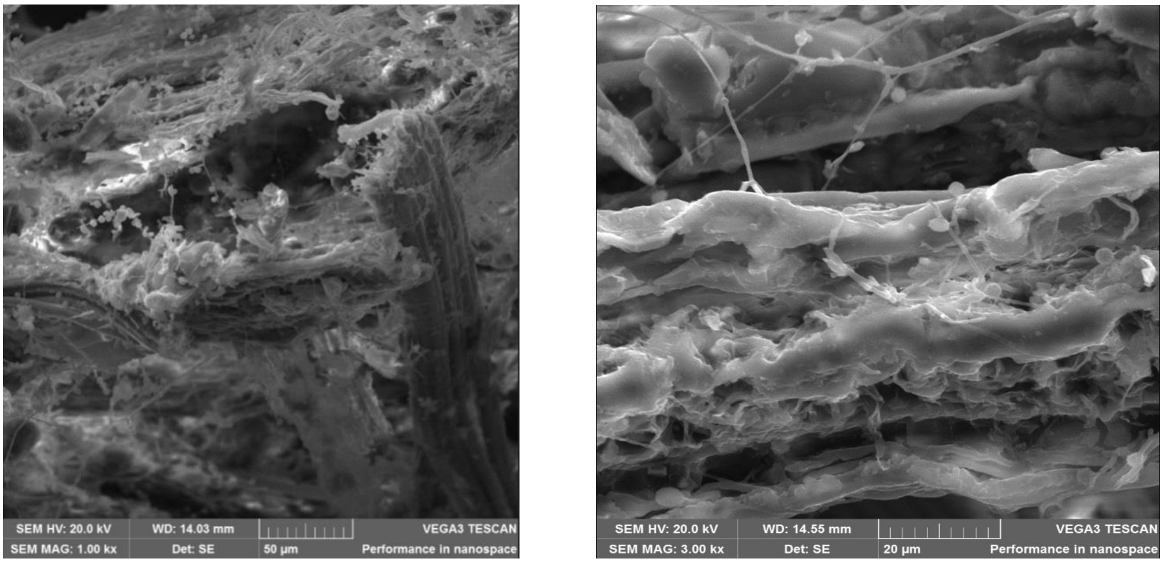

Figure 4: SEM images of (a) raw coconut shells biochar and (b) SEM image of iron-modified coconut shells biochar

Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR analysis of the iron-biochar nanocomposite (IBN) elucidated the functional groups present and their roles in enhancing the material's adsorption capabilities (Regmi *et al., 2*012; Shu *et al., 2*020; Usman *et al., 2*021; Zhao *et al., 2*019). The FTIR spectra of the IBN exhibited several characteristic peaks corresponding to various functional groups critical for pollutant interaction and adsorption. Key peaks included O-H stretching at 3543.01 cm^{-1} and N-H stretching at 3411.45 cm^{-1}, indicative of hydroxyl and amine groups, respectively. These groups are known for their hydrophilic nature and ability to form hydrogen bonds, facilitating the adsorption of polar molecules. The C-H stretching bands observed at 3041.20 cm^{-1} and 2921.98 cm^{-1} signify the presence of aliphatic hydrocarbons, which contribute to the hydrophobic interactions of the material. The peaks at 1677.19 cm^{-1} and 1631.35 cm^{-1} corresponded to C=O and C=C stretching vibrations, indicative of carbonyl and aromatic groups. These groups play a vital role in chelating metal ions and adsorbing organic pollutants, as corroborated by Zhao *et al.,*(2019), reporting similar adsorption mechanisms in biochar materials. Additionally, the presence of C-O stretching at 1149.63 cm^{-1} and C-H bending at 662.89 cm^{-1} highlighted the diverse chemical functionality of the IBN as shown in Table 2. These functional groups enhance the composite's reactivity by providing active sites for pollutant binding, supporting its potential in environmental applications. The integration of iron nanoparticles may also have contributed to modifications in the chemical environment, as evidenced by slight shifts in peak positions compared to unmodified biochar. In addition, the FTIR analysis confirmed the presence of functional groups essential for adsorption processes, validating the material's suitability for pollutant removal. These findings align with previous studies by Regmi et al. (2012), emphasizing the critical role of surface functional groups in the performance of biochar-based adsorbents. The FTIR analysis confirmed the presence of essential functional groups, validating IBN's adsorption potential for pollutant removal. Future research should explore enhancing iron loading efficiency, optimizing functionalization techniques, and assessing long-term stability in real-world applications. Investigating regeneration efficiency will provide insights into its reusability for sustainable applications. Additionally, exploring alternative metal dopants and hybrid biochar composites could further improve adsorption performance. The integration of IBN into advanced filtration systems and its application in industrial wastewater treatment could enhance scalability. Expanding studies on IBN's interaction with emerging contaminants will further solidify its role in sustainable environmental remediation strategies.

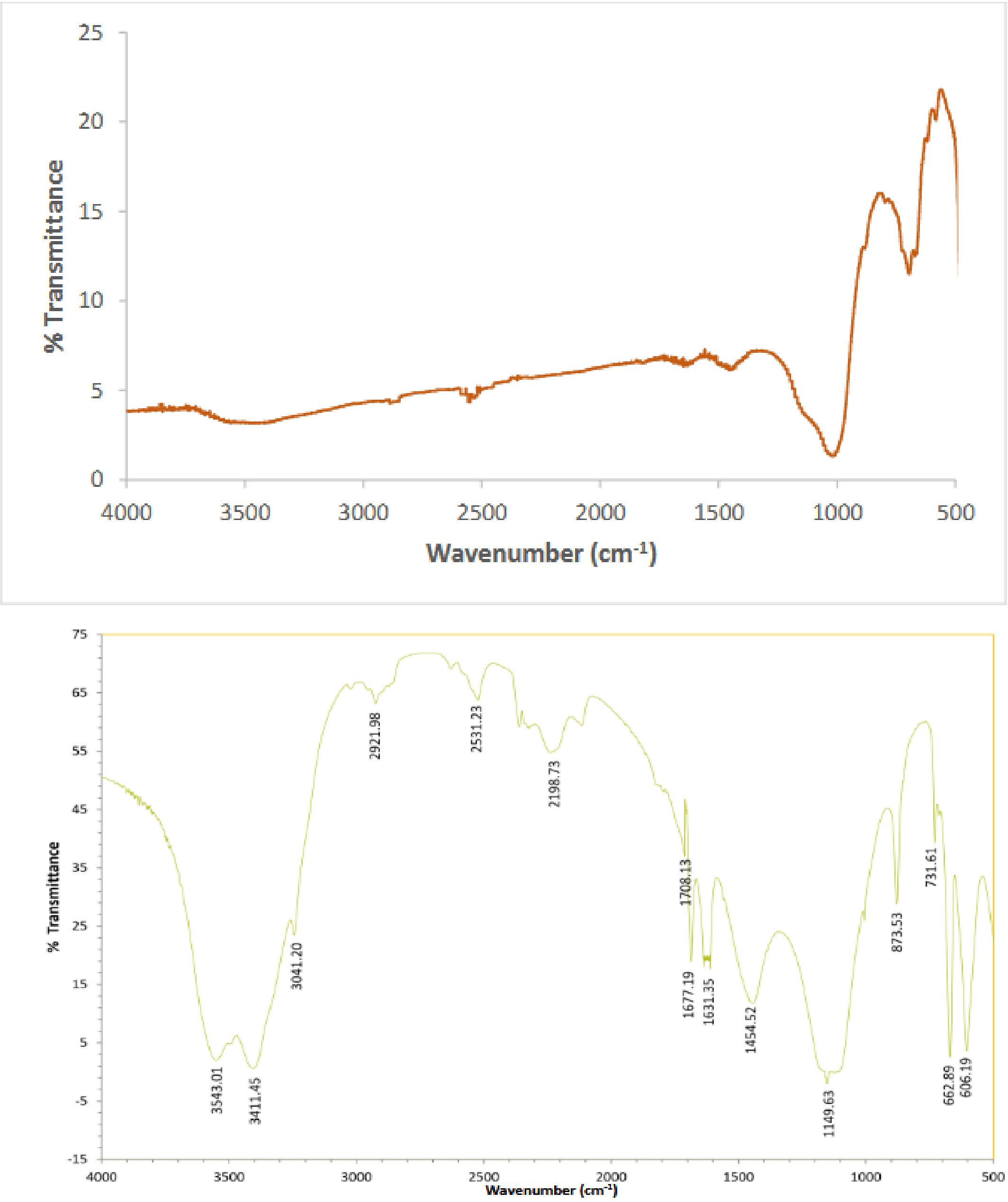

Figure 5: Fourier-transform infrared spectroscopy (FTIR) analysis of iron coconut shell (a) coconut shell biochar and (b) FTIR coconut shell iron biochar

Table 2: FTIR Analysis of Coconut Biochar and Iron-Biochar Nanocomposite (IBN)

Wavenumber (cm⁻¹)	Functional Group	Coconut Biochar	IBN
3543.01	O-H stretching	Present	Present
3411.45	N-H stretching	Absent	Present
3041.20	C-H stretching	Present	Present
2921.98	C-H stretching	Present	Present
2531.23	C=C stretching	Absent	Present
2198.73	C=C stretching	Absent	Present
1677.19	C=O stretching	Present	Present
1631.35	C=C stretching	Present	Present
1454.52	C-H bending	Present	Present
1149.63	C-O stretching	Present	Present
662.89	C-H bending	Absent	Present
606.19	C-Br stretching	Absent	Present

X-ray diffraction analysis (XRD)

The XRD analysis of the IBN revealed critical information about its crystalline structure and mineral composition. The XRD patterns for both the raw coconut biochar and the IBN exhibited distinct differences, underscoring the structural changes induced by the incorporation of iron nanoparticles during synthesis. The coconut biochar showed its highest peak at 24.5, indicative of an amorphous carbon structure typical of biochar derived from biomass. In contrast, the IBN displayed prominent peaks at 100, 15.90, and 250, with the highest intensity at 250 as shown in Figure 6. These peaks correspond to specific crystalline planes of iron oxides, confirming the successful integration of iron nanoparticles within the biochar matrix. The presence of these crystalline phases suggests improved structural organization compared to the amorphous nature of the raw biochar (Janu *et al.,* 2021). These findings align with studies such as those by Pang *et al.,*(2021) reporting similar transformations in biochar materials upon the incorporation of metal nanoparticles. The XRD patterns of the IBN highlight the material's enhanced structural integrity and increased functionality, which are crucial for applications like adsorption and catalysis (Janu *et al., 2*021; S. Li *et al., 2*019). Furthermore, the differences in peak intensities and positions between the biochar and the IBN indicate alterations in the interatomic spacing and crystallinity due to the nano-engineering process. These modifications likely enhance the material physicochemical properties, such as surface reactivity and pollutant-binding capabilities (Yallappa *et al., 2*017). Additionally, the XRD analysis confirms the successful synthesis of the IBN with well-defined crystalline phases of iron. These structural enhancements contribute to the composite's improved adsorption and catalytic properties, validating its potential for environmental applications such as pollutant removal and water treatment. The results are consistent with prior research emphasizing the role of crystallinity in biochar functionality (Hoslett *et al., 2*019; Pang *et al., 2*021; Thamilselvi & Radha, 2017; Yallappa *et al., 2*017).The XRD analysis confirmed the successful integration of iron nanoparticles, enhancing the crystallinity and structural integrity of the IBN. Future research should explore optimizing iron loading, controlling crystal growth mechanisms, and assessing the long-term stability of crystalline phases under real-world conditions. Investigating alternative metal dopants and their effects on crystallinity could further refine the material's adsorption and catalytic efficiency. Additionally, integrating IBN into multifunctional nanocomposites for applications such as advanced oxidation processes and selective pollutant removal may improve performance. Exploring its electronic and magnetic properties could expand its use in industrial catalysis, water purification, and environmental remediation.

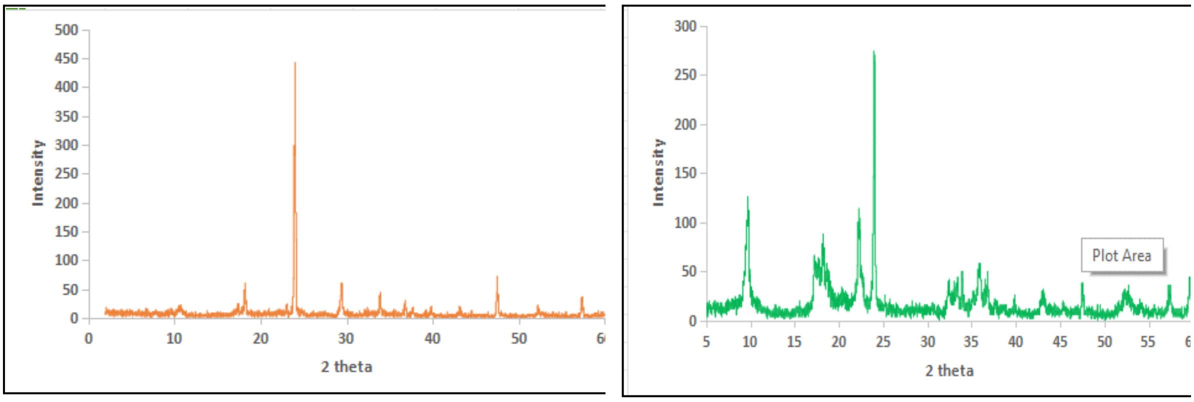

Figure 6: XRD image showing the composition of (a) coconut shells biochar and (b) iron-modified XRD coconut shells biochar

CONCLUSION

The one-pot biosynthesis of iron-biochar nanocomposites (IBN) from waste coconut shells using *Allamanda cathartica* extract successfully demonstrated an eco-friendly and efficient approach to synthesizing advanced functional materials. Comprehensive characterization revealed significant improvements in structural, morphological, and chemical properties, including enhanced surface area, porosity, and active sites for adsorption. The integration of iron nanoparticles not only improved the composite's pollutant removal capabilities but also imparted magnetic properties for easier recovery and reuse. This study establishes IBN as a promising candidate for environmental applications such as water purification and soil remediation. The sustainable approach of utilizing biomass waste and natural reducing agents aligns with global efforts toward circular economy and environmental conservation.

REFERENCES

Adebayo, G. B., Balogun, B. B., Shaibu, S. E., Jamiu, W., Etim, E. U., Oleh, F., Efiong N. E. & Ogboo, B. S. (2019). Comparative study on the adsorption capacity and kinetics of xylene onto rice husk and cassava peel activated carbon. *International Journal of Current Research*, *11*, 8282-8290.

Ahmad, K. S., Yaqoob, S., & Gul, M. M. (2022). Dynamic green synthesis of iron oxide and manganese oxide nanoparticles and their cogent antimicrobial, environmental, and electrical applications. *Reviews in Inorganic Chemistry*, *42*(3), 239–263. https://doi.org/10.1515/revic-2021-0033

Ahmad, R., & Haseeb, S. (2015). Absorptive removal of Pb2+, Cu2+ and Ni2+ from the aqueous solution by using groundnut husk modified with Guar Gum (GG): Kinetic and thermodynamic studies. *Groundwater for Sustainable Development*, *1*(1–2), 41–49. https://doi.org/10.1016/j.gsd.2015.11.001

Ahmed, S. F., Mofijur, M., Rafa, N., Chowdhury, A. T., Chowdhury, S., Nahrin, M., Islam, A. B. M. S., & Ong, H. C. (2022). Green approaches in synthesizing nanomaterials for environmental nanobioremediation: Technological advancements, applications, benefits and challenges. *Environmental Research*, *204*, 111967. https://doi.org/10.1016/j.envres.2021.111967

Altıkat, A., Alma, M. H., Altıkat, A., Bilgili, M. E., & Altıkat, S. (2024). A Comprehensive Study of Biochar Yield and

Quality Concerning Pyrolysis Conditions: A Multifaceted Approach. *Sustainability (Switzerland)*, *16*(2). https://doi.org/10.3390/su16020937

Beniah Obinna, I., & Christian Ebere, E. (2019). A review: Water pollution by heavy metal and organic pollutants: Brief review of sources, effects and progress on remediation with aquatic plants. *Analytical Methods in Environmental Chemistry Journal*, *2*(3), 5–38. https://doi.org/10.24200/amecj.v2.i03.66

da Silva, N. E. P., Bezerra, L. C. A., Araújo, R. F., Moura, T. A., Vieira, L. H. S., Alves, S. B. S., Fregolente, L. G., Ferreira, O. P., & Avelino, F. (2024). Coconut shell-based biochars produced by an innovative thermochemical process for obtaining improved lignocellulose-based adsorbents. *International Journal of Biological Macromolecules*, 133685.

De Lima, H. H. C., Llop, M. E. G., Dos Santos Maniezzo, R., Moisés, M. P., Janeiro, V., Arroyo, P. A., Guilherme, M. R., & Rinaldi, A. W. (2021). Enhanced removal of bisphenol A using pine-fruit shell-derived hydrochars: Adsorption mechanisms and reusability. *Journal of Hazardous Materials*, *416*, 126167. https://doi.org/10.1016/j.jhazmat.2021.126167

Dokhe, P., Girme, S., Barawant, M. M., & Abdi, G. (2023). Biochemical, Phytochemical Screening and Pharmacological Potential of Allamanda blanchetii (Purple Allamanda). *INTERNATIONAL JOURNAL OF PLANT AND ENVIRONMENT*, *9*(01), 89–92. https://doi.org/10.18811/ijpen.v9i01.15

Ekman, S., Dos Reis, G. S., Laisné, E., Thivet, J., Grimm, A., Lima, E. C., Naushad, Mu., & Dotto, G. L. (2023). Synthesis, Characterization, and Adsorption Properties of Nitrogen-Doped Nanoporous Biochar: Efficient Removal of Reactive Orange 16 Dye and Colorful Effluents. *Nanomaterials*, *13*(14), 2045. https://doi.org/10.3390/nano13142045

Enin, G. N., Shaibu, S. E., Ujah, G. A., Ibu, R. O., & Inangha, P. G. (2021). Phytochemical and Nutritive Composition of Uvariachamae P. Beauv. Leaves, Stem Bark and Root Bark. *ChemSearch Journal*, *12*(1), 9-14.

Enin, G. N., Antia, B. S., Shaibu, S. E., & Nyakno, I. (2023). Comparison of the chemical composition, nutritional values, total phenolics and flavonoids content of the ripe and unripe Solanum nigrum Linn. Fruits from Nigeria. *World Journal of Pharmacy and Pharmaceutical Sciences*, *12*(8), 1-18.

Essien, E. A., Kavaz, D., & Solomon, M. M. (2018). Olive leaves extract mediated zero-valent iron nanoparticles: Synthesis, characterization, and assessment as adsorbent for nickel (II) ions in aqueous medium. *Chemical Engineering Communications*, *205*(11), 1568–1582. https://doi.org/10.1080/00986445.2018.1461089

Fraga-Corral, M., Otero, P., Cassani, L., Echave, J., Garcia-Oliveira, P., Carpena, M., Chamorro, F., Lourenço-Lopes, C., Prieto, M. A., & Simal-Gandara, J. (2021). Traditional Applications of Tannin Rich Extracts Supported by Scientific Data: Chemical Composition, Bioavailability and Bioaccessibility. *Foods*, *10*(2), 251. https://doi.org/10.3390/foods10020251

Hoslett, J., Ghazal, H., Ahmad, D., & Jouhara, H. (2019). Removal of copper ions from aqueous solution using low temperature biochar derived from the pyrolysis of municipal solid waste. *Science of the Total Environment*, *673*, 777–789. https://doi.org/10.1016/j.scitotenv.2019.04.085

Hussain, T., Akhter, N., Nadeem, R., Rashid, U., Noreen, S., Anjum, S., Ullah, S., Hussain, H. R., Ashfaq, A., Perveen, S., A. Alharthi, F., & Kazerooni, E. A. (2023). Biogenic synthesis of date stones biochar-based zirconium oxide nanocomposite for the removal of hexavalent chromium from aqueous solution. *Applied Nanoscience (Switzerland)*, *13*(9), 6053–6066. https://doi.org/10.1007/s13204-022-02599-z

Janu, R., Mrlik, V., Ribitsch, D., Hofman, J., Sedláček, P., Bielská, L., & Soja, G. (2021). Biochar surface functional groups as affected by biomass feedstock, biochar composition and pyrolysis temperature. *Carbon Resources Conversion*, *4*(November 2020), 36–46. https://doi.org/10.1016/j.crcon.2021.01.003

Khan, H., Pervaiz, A., Intagliata, S., Das, N., Nagulapalli Venkata, K. C., Atanasov, A. G., Najda, A., Nabavi, S. M., Wang, D., Pittalà, V., & Bishayee, A. (2020). The analgesic potential of glycosides derived from medicinal plants. *DARU Journal of Pharmaceutical Sciences*, *28*(1), 387–401. https://doi.org/10.1007/s40199-019-00319-7

Komkiene, J., & Baltrenaite, E. (2016). Biochar as adsorbent for removal of heavy metal ions [Cadmium(II), Copper(II), Lead(II), Zinc(II)] from aqueous phase. *International Journal of Environmental Science and Technology*, *13*(2), 471–482. https://doi.org/10.1007/s13762-015-0873-3

Kumar, S., & Saha, A. (2022). Utilization of coconut shell biomass residue to develop sustainable biocomposites and characterize the physical, mechanical, thermal, and water absorption properties. *Biomass Conversion and Biorefinery*, *September 2022*. https://doi.org/10.1007/s13399-022-03293-4

Li, J., Xu, C., Zhang, Y., Tang, X., Qi, W., & Wang, Q. (2018). Gravity-directed separation of both immiscible and emulsified oil/water mixtures utilizing coconut shell layer. *Journal of Colloid and Interface Science*, *511*, 233–242. https://doi.org/10.1016/j.jcis.2017.09.111

Li, S., Harris, S., Anandhi, A., & Chen, G. (2019). Predicting biochar properties and functions based on feedstock and pyrolysis temperature: A review and data syntheses. In *Journal of Cleaner Production*. Elsevier Ltd. https://doi.org/10.1016/j.jclepro.2019.01.106

Li, T., Fang, F., Yang, Y., Shen, W., Bao, W., Zhang, T., Ai, F., Ding, X., Xin, H., & Wang, X. (2019). Surface nano-engineered wheat straw for portable and adjustable water purification. *Science of the Total Environment*, *655*, 1028–1036. https://doi.org/10.1016/j.scitotenv.2018.11.206

Liu, Z., Yu, L., Zhou, L., & Zhou, Z. (2019). One-Pot Biosynthesis of L -Aspartate from Maleate via an Engineered Strain Containing a Dual-Enzyme System. *Applied and Environmental Microbiology*, *85*(19), e01327-19. https://doi.org/10.1128/AEM.01327-19

Nunes, L. A., Silva, M. L. S., Gerber, J. Z., & Kalid, R. D. A. (2020). Waste green coconut shells: Diagnosis of the disposal and applications for use in other products. *Journal of Cleaner*

Production, *255*, 120169. https://doi.org/10.1016/j.jclepro.2020.120169

Pandey, P. P., & Kumar, M. S. (2024). Exploring the therapeutic potential of steroidal alkaloids in managing Alzheimer's disease. *Steroids*, *209*, 109468. https://doi.org/10.1016/j.steroids.2024.109468

Pang, Y. L., Law, Z. X., Lim, S., Chan, Y. Y., Shuit, S. H., Chong, W. C., & Lai, C. W. (2021). Enhanced photocatalytic degradation of methyl orange by coconut shell–derived biochar composites under visible LED light irradiation. *Environmental Science and Pollution Research*, *28*(21), 27457–27473. https://doi.org/10.1007/s11356-020-12251-4

Perveen, S., Nadeem, R., Rehman, S. U., Afzal, N., Anjum, S., Noreen, S., Saeed, R., Amami, M., Al-Mijalli, S. H., & Iqbal, M. (2022). Green synthesis of iron (Fe) nanoparticles using Plumeria obtusa extract as a reducing and stabilizing agent: Antimicrobial, antioxidant and biocompatibility studies. *Arabian Journal of Chemistry*, *15*(5), 103764. https://doi.org/10.1016/j.arabjc.2022.103764

Qu, J., Wang, Y., Tian, X., Jiang, Z., Deng, F., Tao, Y., Jiang, Q., Wang, L., & Zhang, Y. (2021). KOH-activated porous biochar with high specific surface area for adsorptive removal of chromium (VI) and naphthalene from water: Affecting factors, mechanisms and reusability exploration. *Journal of Hazardous Materials*, *401*, 123292. https://doi.org/10.1016/j.jhazmat.2020.123292

Ramya, A., Dhevagi, P., & Rakesh, S. S. (2022). Nanomaterials for Wastewater Remediation: Resolving Huge Problems with Tiny Particles. In A. Krishnan, B. Ravindran, B. Balasubramanian, H. C. Swart, S. J. Panchu, & R. Prasad (Eds.), *Emerging Nanomaterials for Advanced Technologies* (pp. 601–620). Springer International Publishing. https://doi.org/10.1007/978-3-030-80371-1_21

Reethu Metilda Manukonda & Satapathy Dhabal. (2024). Phytochemical Analysis and Evaluation of in Vitro Antimitotic Activity of *Allamanda cathartica* Methanolic Extract: Research Article. *Journal of Pharma Insights and Research*, *2*(4), 146–154. https://doi.org/10.69613/92x5vg75

Regmi, P., Garcia Moscoso, J. L., Kumar, S., Cao, X., Mao, J., & Schafran, G. (2012). Removal of copper and cadmium from aqueous solution using switchgrass biochar produced via hydrothermal carbonization process. *Journal of Environmental Management*, *109*, 61–69. https://doi.org/10.1016/j.jenvman.2012.04.047

Shaibu, S. E., Adekola, F. A., Adegoke, H. I., & Ayanda, O. S. (2014). A comparative study of the adsorption of methylene blue onto synthesized nanoscale zero-valent iron-bamboo and manganese-bamboo composites. *Materials*, *7*(6), 4493-4507.

Shaibu, S. E., Adekola, F. A., Adegoke, H. I., & Abdus-Salam, N. (2015). Heavy metal speciation patterns of selected dumpsites in Ilorin Metropolis. *International Journal of Chemical, Material and Environmental Research*, *2*(1), 1-11.

Shaibu, S. E., Adekola, F. A., Adegoke, H. I., & Ayanda, O. S. (2017). Kinetic and Adsorption Studies of a Thiazine and Triarylmethane Dye onto Bamboo Impregnated Nanoscale Manganese. *Int J Nano Med & Eng*, *2*(9), 143-149.

Shaibu, S. E., Inam, E. J., Moses, E. A., Abraham, N. A., & Offiong, N. A. O. (2021). 11 Remediation of Organic Pollutants Using Biobased Nanomaterials.

Shaibu, S. E., Inam, E. J. & Moses, E. A. (2022). Biogenic silver-kaolinite nanocomposite for the sequestration of lead and cadmium in simulated produced water. *Journal of Material and Environmental Sustainability Research*, *1*(2), 13-25.

Simon, N., Shaibu, S. E., Fatunla, O. K., Johnson, A. S., & Ekpo, P. A. (2023). Natural clay mineral-periwinkle activated carbon composite: characterisation and structural insight. *World Journal of Applied Science and Technology*, *15*(1), 15-18.

Shaikh, J. R., & Patil, M. (2020). Qualitative tests for preliminary phytochemical screening: An overview. *International Journal of Chemical Studies*, *8*(2), 603–608. https://doi.org/10.22271/chemi.2020.v8.i2i.8834

Shu, Y., Ji, B., Cui, B., Shi, Y., Wang, J., Hu, M., Luo, S., & Guo, D. (2020). Almond shell-derived, biochar-supported, nano-zero-valent iron composite for aqueous hexavalent chromium removal: Performance and mechanisms. *Nanomaterials*, *10*(2). https://doi.org/10.3390/nano10020198

Sutrisno, Soenoko, R., Irawan, Y. S., & Widodo, T. D. (2021). Effect of limestone mass concentration on tensile strength and surface morphology of coconut fiber. *IOP Conference Series: Materials Science and Engineering*, *1034*(1), 012168. https://doi.org/10.1088/1757-899x/1034/1/012168

Thamilselvi, V., & Radha, K. V. (2017). Silver Nanoparticle loaded corncob adsorbent for effluent treatment. *Journal of Environmental Chemical Engineering*, *5*(2), 1843–1854. https://doi.org/10.1016/j.jece.2017.03.020

Usman, M. A., Momohjimoh, I., & Usman, A. O. (2021). Characterization of groundnut shell powder as a potential reinforcement for biocomposites. *Polymers from Renewable Resources*, *12*(3–4), 77–91. https://doi.org/10.1177/20412479211008761

Vishnu, D., Dhandapani, B., Ramakrishnan, S. R., Pandian, P. K., & Raguraman, T. (2021). Fabrication of surface-engineered superparamagnetic nanocomposites (Co/Fe/Mn) with biochar from groundnut waste residues for the elimination of copper and lead metal ions. *Journal of Nanostructure in Chemistry*, *11*(2), 215–228. https://doi.org/10.1007/s40097-020-00360-y

Yallappa, S., Deepthi, D. R., Yashaswini, S., Hamsanandini, R., Chandraprasad, M., Ashok Kumar, S., & Hegde, G. (2017). Natural biowaste of Groundnut shell derived nano carbons: Synthesis, characterization and itsin vitro antibacterial activity. *Nano-Structures and Nano-Objects*, *12*, 84–90. https://doi.org/10.1016/j.nanoso.2017.09.009

Ying, Z., Chen, X., Li, H., Liu, X., Zhang, C., Zhang, J., & Yi, G. (2021). Efficient Adsorption of Methylene Blue by Porous Biochar Derived from Soybean Dreg Using a One-Pot Synthesis Method. *Molecules*, *26*(3), 661. https://doi.org/10.3390/molecules26030661

Yuan, D., Guo, Y., Pu, F., Yang, C., Xiao, X., Du, H., He, J., & Lu, S. (2024). Opportunities and challenges in enhancing the bioavailability and bioactivity of dietary flavonoids: A novel delivery system perspective. *Food Chemistry*, *430*, 137115. https://doi.org/10.1016/j.foodchem.2023.137115

Zhang, W., Li, Y., Ma, X., Qian, Y., & Wang, Z. (2020). Simultaneous NO/CO2 removal performance of biochar/limestone in calcium looping process. *Fuel*, *262*(July), 116428. https://doi.org/10.1016/j.fuel.2019.116428

Zhao, J. J., Shen, X. J., Domene, X., Alcañiz, J. M., Liao, X., & Palet, C. (2019). Comparison of biochars derived from different types of feedstock and their potential for heavy metal removal in multiple-metal solutions. *Scientific Reports*, *9*(1), 1–12. https://doi.org/10.1038/s41598-019-46234-4

Zhu, L., Tong, L., Zhao, N., Li, J., & Lv, Y. (2019). Coupling interaction between porous biochar and nano zero valent iron/nano α-hydroxyl iron oxide improves the remediation efficiency of cadmium in aqueous solution. *Chemosphere*, *219*, 493–503. https://doi.org/10.1016/j.chemosphere.2018.12.013

Zhu, Y., Fan, W., Zhou, T., & Li, X. (2019). Removal of chelated heavy metals from aqueous solution: A review of current methods and mechanisms. *Science of the Total Environment*, *678*(37), 253–266. https://doi.org/10.1016/j.scitotenv.2019.04.416

Zhu, Z., Wu, Y., Hu, C., Zhang, L., Ding, H., Zhu, Y., Fan, Y., Deng, H., Zhou, X., & Tang, S. (2022). Elimination of zinc ions from aqueous solution by a hydroxylapatite-biochar composite material with the hierarchical porous microstructures of sugarcane waste. *Journal of Cleaner Production*, *362*, 132483.