Published by

British Journal of Contemporary Research

BEXFORD PUBLISHING

Optimization and Physicochemical Characterization of H_2SO_4-Activated Charcoal from Coconut Shells: Textural, Morphological, and Surface Chemical Properties

Original Research Article | Volume 1 | Issue 3 | 2026 | Article Number: 027

Accepted: 28 July 2026 | Published: 10 August 2026 | ISSN: 2979-8582 (Online)

Crossref : https://doi.org/10.67693/BJCR-LGDHZ3LY

Aisha Aliyu Gaya[1], Abdussamad Mukhtar Mohammed[2], Farhana Aziz[3]

[1]Department of Applied Chemistry, Federal Polytechnic/Department of Chemistry, Yobe State University, Damaturu, Nigeria,
[2]Department of Chemistry, Faculty of Science, Yobe State University Damaturu, Nigeria,
[3]Advanced Membrane Technology Research Centre (AMTEC), Universiti Teknologi Malaysia/School of Chemical and Energy Engineering, Faculty of Engineering, Universiti Teknologi, Malaysia

Correspondence: Abdussamad Mukhtar Mohammed, abdussamadgwani@gmail.com

Abstract

This study reports the synthesis and comprehensive characterization of activated charcoal derived from coconut shells via sulfuric acid chemical activation. The carbonization-activation protocol was optimized to develop a porous carbonaceous material, which was systematically evaluated using Fourier Transform Infrared (FTIR) spectroscopy, Brunaue Emmett Teller (BET) surface area analysis, and Scanning Electron Microscopy (SEM). FTIR analysis confirmed the successful introduction of oxygen-containing functional groups (carbonyl at 1705.01 cm^{-1}, aromatic C=C at 1594.37 cm^{-1}, phenolic/ether C–O at 1196.53 cm^{-1}) that enhance surface reactivity toward polar adsorbates. BET analysis revealed a predominantly mesoporous architecture with a surface area of 52.96 m^2/g, total pore volume of 0.0539 cm^3/g, and average pore diameter of 4.07 nm properties favourable for liquid-phase diffusion of organic molecules. SEM imaging corroborated a hierarchical morphology featuring microporous transport channels (>50 nm) and mesoporous active sites (2–50 nm), though minor particle agglomeration was observed at higher magnifications. While the surface area is lower than literature values for highly activated coconut carbons (500–1200 m^2/g), the combined textural and chemical properties confirm the material's potential as a sustainable adsorbent precursor. This work provides foundational characterization data to guide future activation optimization and scale-up of coconut shell-derived carbons for environmental and industrial applications.

Keywords: Coconut Shell, Activated Charcoal, Chemical Activation, FTIR, BET Analysis, SEM, Mesoporous Carbon, Biomass Valorisation

1. INTRODUCTION

The global generation of agricultural waste exceeds 6 billion tonnes annually, presenting both an environmental challenge and a resource opportunity [1]. Coconut shells, a byproduct of the extensive coconut production in tropical regions, are often discarded or burned, leading to significant environmental pollution. However, these shells hold potential for sustainable applications that can mitigate their negative impact[2]. Various studies have explored innovative ways to utilize coconut shells, transforming them into valuable resources such as biochar, charcoal, and composite materials [3], [4]. These applications not only reduce waste but also offer economic and environmental benefits Coconut shells, a byproduct of the ~60 million tonnes of coconuts produced yearly in tropical regions, are often discarded or burned, contributing to air and soil pollution. Valorising this waste into high-value materials aligns with circular economy principles and sustainable development goals[5].

Activated charcoal (activated carbon) is a critical material for adsorption-based applications including water purification, air filtration, and catalysis [6], [7]. Commercial production predominantly relies on non-renewable precursors like coal and wood, raising concerns about deforestation, carbon footprint, and cost [8]. Biomass-derived activated carbons offer a sustainable alternative, with coconut shells being particularly promising due to their high fixed carbon content (~45–50%), low ash content (<5%), and inherent structural porosity[9] (Khandaker, *et al*., 2025).

Chemical activation using agents such as H_2SO_4, KOH, or H_3PO_4 is widely employed to develop porosity and introduce surface functional groups [1]. H_2SO_4 activation promotes dehydration, carbonization, and pore formation at relatively moderate temperatures, while simultaneously introducing oxygen-containing groups that enhance adsorption of polar compounds [10]. However, the physicochemical properties of the resulting carbon are highly sensitive to activation parameters (agent concentration, temperature, time), necessitating systematic characterization to establish structure-property relationships [11]

While numerous studies report coconut shell-based carbons with surface areas >500 m^2/g under optimized conditions (Boudalia, et al., 2026), comprehensive multi-technique characterization linking synthesis protocols to textural, morphological, and surface chemical properties remains limited. Moreover, standardized benchmarks for H_2SO_4-activated coconut shell charcoal are lacking, hindering reproducibility and comparative analysis across studies.

This study therefore aims to:

i. Synthesize activated charcoal from coconut shells via H_2SO_4 chemical activation under controlled conditions;
ii. Characterize its physicochemical properties using FTIR, BET, and SEM; and
iii. Establish baseline benchmarks to guide future optimization efforts.The work contributes foundational data for developing sustainable, biomass-derived adsorbents for environmental remediation.
iv. The development of activated carbon materials shows a clear progression in how activation methods influence pore structure and surface characteristics, which in turn determine adsorption performance. This evolution becomes particularly evident when comparing different chemical and physical activation routes applied to various carbon precursors.

Our study, using H_2SO_4 chemical activation, produced a relatively low surface area (52.96 m^2/g) with a small pore volume (0.0539 cm^3/g) and an average pore diameter of 4.07 nm. This suggests a material dominated by limited mesoporous development, likely with restricted internal diffusion pathways compared to more aggressively activated carbons.

In contrast, early foundational work by [12] demonstrated that KOH activation can generate highly developed porous structures, achieving surface areas as high as 2451 m^2/g with significant micropore volume (predominantly <2 nm). This established KOH as one of the most effective activating agents for producing highly microporous carbon suitable for gas adsorption and small-molecule uptake. Similarly, Cazetta et al. (2011) reported an even higher surface area (2825 m^2/g) using NaOH chemical activation with a high impregnation ratio. Their material exhibited a strongly microporous structure, reinforcing the role of strong alkali activation in maximizing surface development and internal porosity.

Physical chemical hybrid approaches also show strong performance. [10] combined steam/CO_2 activation with microwave heating and achieved exceptionally high surface areas exceeding 2000 m^2/g, although detailed pore distribution data were not fully reported. This highlights how thermal–physical synergy can significantly enhance pore formation beyond conventional chemical routes. In contrast, acid-based activation tends to produce more mesoporous structures with lower surface areas. For example, [13] using H_3PO_4 activation obtained a moderate surface area (~891 m^2/g) but a highly mesoporous structure (2–50 nm range with ~62.85% mesopore fraction), making it particularly suitable for larger organic molecules such as dyes. A broader review by [14] confirms this general trend, reporting typical activated carbon surface areas between 500–1640 m^2/g with pore volumes of 0.2–0.5 cm^3/g and mesopore sizes around 2–5 nm depending on activation method. This establishes a benchmark range for conventional activated carbons.

More recent comparative studies further emphasize the strong dependence of porosity on activation chemistry. [15] showed that $ZnCl_2$ activation combined with response surface optimization can yield surface areas between 346–525 m^2/g with enlarged mesoporous structures, making them more favorable for dye adsorption. Meanwhile, [16] directly compared H_2SO_4 and NaOH activation, showing a stark difference in performance: H_2SO_4 produced only ~40 m^2/g, whereas NaOH activation increased surface area to ~345 m^2/g with a pore volume of 0.20 cm^3/g and a dominant microporous structure (~1.92 nm).

The literature clearly shows that alkali-based activations (KOH, NaOH) tend to produce highly microporous, high-surface-area carbons, while acid-based activations (H_3PO_4, H_2SO_4) generally yield lower surface areas but more mesoporous or structurally open frameworks. Physical or hybrid activation methods further extend this range, sometimes achieving ultra-high surface areas above 2000 m^2/g.

The modest surface area in this study likely reflects suboptimal activation conditions (agent concentration, temperature, or time), highlighting a key opportunity for future optimization. Nevertheless, the mesoporous architecture supports efficient mass transfer in liquid-phase systems, partially compensating for limited surface area [11].

2. MATERIALS AND METHODS

2.1. Materials

Coconut shells were sourced locally, washed thoroughly with deionized water, sun-dried for 72 h, and crushed to 1–2 mm particle size. Sulfuric acid (H_2SO_4, 98%, analytical grade) was used as the activating agent. All chemicals were used as received without further purification.

2.2. Synthesis of Activated Charcoal

The activated charcoal was prepared via chemical activation as follows:

- **Impregnation**: Crushed coconut shells were impregnated with 1:1 (w/w) H_2SO_4 solution and stirred for 2 h to ensure uniform coating.
- **Drying**: The impregnated biomass was dried at 105°C for 24 h to remove moisture.

- **Carbonization**: Dried material was carbonized in a muffle furnace at 400° for 30 m under N_2 atmosphere (flow rate: 100 mL/min) to prevent oxidation.
- **Washing**: The resulting char was washed repeatedly with hot deionized water until neutral pH (pH 6.5–7.5) to remove residual acid and soluble byproducts.
- **Final Processing**: Washed material was dried at 105°C for 6 h, ground, and sieved to <150 µm for characterization.

2.3. Characterization Techniques

- **FTIR Spectroscopy**: Surface functional groups were identified using a] spectrometer (PerkinElmer Spectrum Two) in the range 4000–400 cm^{-1} with 32 scans at 4 cm^{-1} resolution. Samples were prepared as KBr pellets (1:100 sample:KBr ratio).
- **BET Surface Area Analysis**: N_2 adsorption desorption isotherms at 77 K were measured using an analyzer (Micromeritics ASAP 2020). Samples were degassed at 150°C for 12 h prior to analysis. Surface area was calculated using the BET equation in the relative pressure range $P/P_0 = 0.05–0.30$. Pore size distribution and pore volume were determined via the BJH method applied to the desorption branch.
- **SEM Imaging**: Surface morphology was examined using a scanning electron microscope (JEOL JSM-IT200) at accelerating voltage 15 kV. Samples were sputter-coated with gold (~10 nm) to enhance conductivity. Images were captured at magnifications ×500, ×5,000, and ×10,000.

3. RESULTS AND DISCUSSION

3.1. FTIR Analysis: Surface Functional Groups

FTIR spectroscopy identified key functional groups on the activated charcoal surface (Fig. 1)

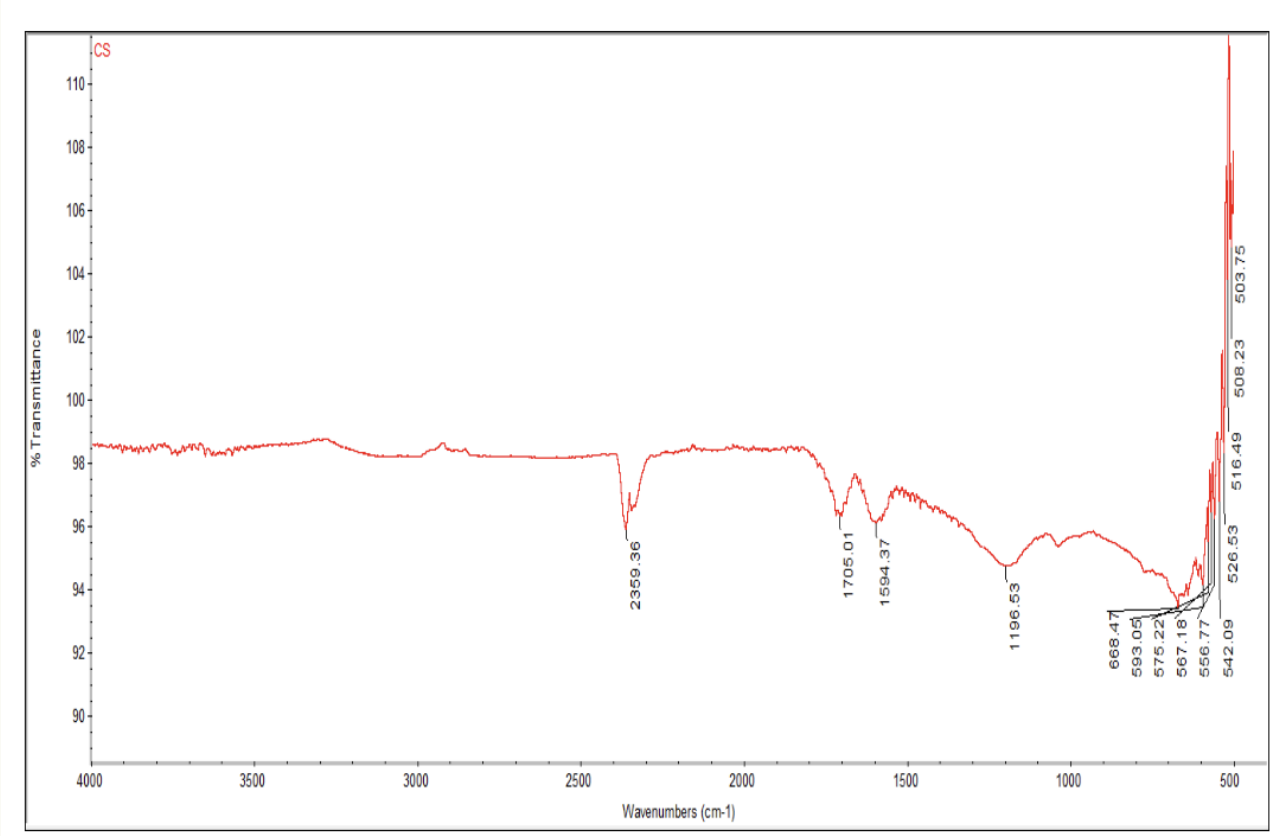

Figure 1 Fourier transform infrared (FTIR)

Table 2. The spectrum exhibited four characteristic absorption bands:

Wavenumber (cm^{-1})	Assignment	Bond Vibration	Role in Adsorption
2359.36	Atmospheric CO_2	O=C=O asymmetric stretch	Artifact; no chemical contribution
1705.01	Carbonyl (C=O)	Stretching (carboxylic acids, ketones, lactones)	Enables H-bonding and electrostatic interactions
1594.37	Aromatic C=C	Skeletal vibration of graphitic domains	Facilitates π–π stacking with aromatic adsorbates
1196.53	Phenolic/Ether (C–O–C, C–O)	C–O stretching	Enhances hydrophilicity and aqueous compatibility

The prominent carbonyl peak at 1705.01 cm^{-1} confirms successful oxidation during H_2SO_4 activation, introducing acidic functional groups that enhance affinity for basic/polar compounds [17]. The aromatic C=C band at 1594.37 cm^{-1} indicates preservation of graphitic structures during carbonization, which support π–π interactions with aromatic pollutants [16]. The C–O bands at 1196.53 cm^{-1} suggest the presence of phenolic and ether groups that improve wettability in aqueous systems [1]. Collectively, these functional groups create a chemically heterogeneous surface capable of multiple adsorption mechanisms, electrostatic attraction, hydrogen bonding, and π–π interactions making the material versatile for diverse applications.

3.2. BET Analysis: Textural Properties

N_2 adsorption desorption isotherms exhibited Type IV behaviour with H3 hysteresis, characteristic of mesoporous materials with slit-shaped pores [1].

Table 3: Key textural parameters are summarized

Parameter	**Value**	**Unit**
BET Surface Area	52.96	m^2/g
Langmuir Surface Area	83.28	m^2/g
Single-point Surface Area (P/P_0=0.53)	53.58	m^2/g
Total Pore Volume (adsorption)	0.0539	cm^3/g
Total Pore Volume (desorption)	0.0554	cm^3/g

The BET surface area of 52.96 m^2/g indicates a moderately developed porous structure. While lower than literature values for highly optimized coconut shell carbons (500–1200 m^2/g), the mesoporous dominance (average pore diameter: 4.07 nm) is advantageous for liquid-phase applications where diffusion of larger molecules (dyes, organic pollutants) is rate-limiting.

3.3. SEM Analysis: Surface Morphology

SEM imaging revealed a hierarchical pore structure across magnifications (Fig. 3a–c):

- **×500**: Irregular, fractured particles with macropores (>50 nm) and interparticle voids serving as primary transport channels.
- **×5,000**: Agglomerated clusters forming interconnected mesoporous networks; visible cavities between aggregates confirm mesopore presence.
- **×10,000**: Densely packed quasi-spherical fine particles with rough, highly irregular textures; micro/mesoporous features evident though micropores (<2 nm) remain below SEM resolution.

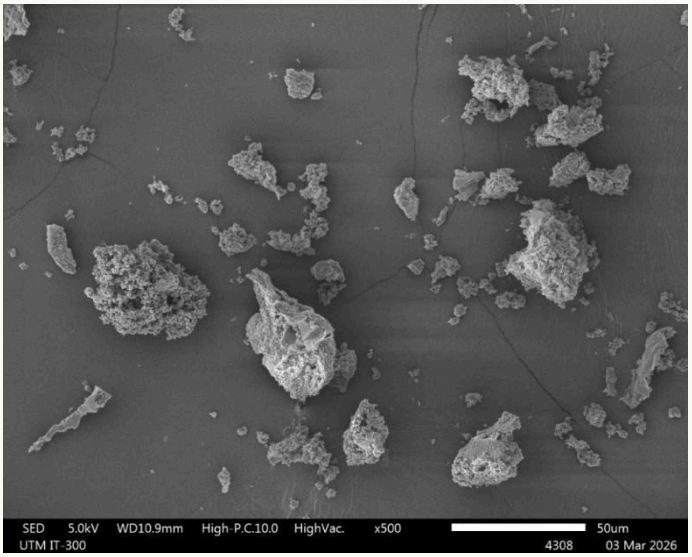

Figure 2a: SEM image at ×500

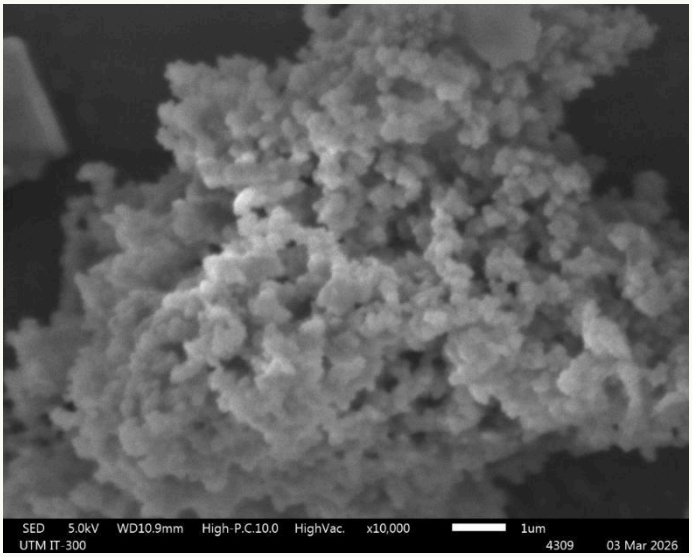

Figure 2b: SEM image at ×10,000

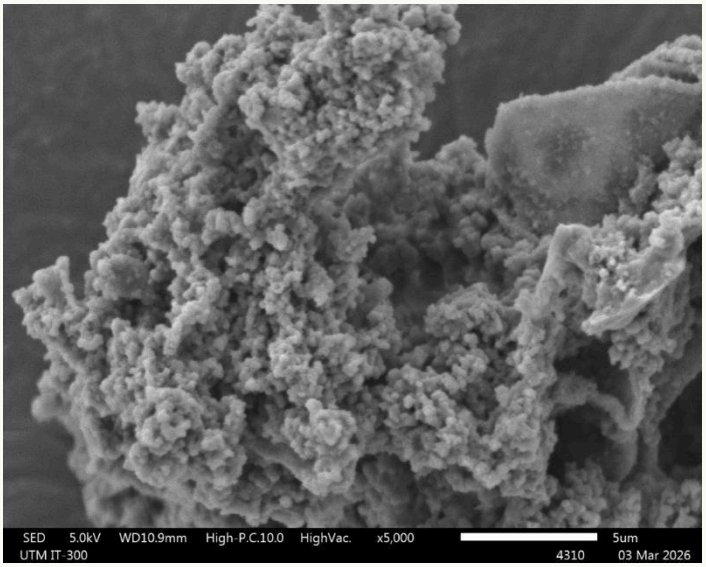

Figure 2c: SEM image at ×5,000

Particle agglomeration observed at higher magnifications may partially limit active site accessibility, suggesting that post-synthesis dispersion treatments could enhance performance [1], [9], [12], [18]. The hierarchical morphology macropores for transport, mesopores for adsorption corroborates BET findings and supports the material's suitability for liquid-phase applications.

3.4. Integrated Structure-Property Analysis

The combined characterization data reveal a material with:

- **Chemical functionality**: Oxygen-rich surface groups enabling multi-mechanism adsorption.
- **Textural suitability**: Mesoporous architecture favouring liquid-phase diffusion**.**
- **Morphological hierarchy**: Multi-scale porosity supporting mass transfer and site accessibility

While the surface area indicates optimization potential, the synergistic combination of properties confirms the technical viability of H_2SO_4-activated coconut shell charcoal as a sustainable adsorbent precursor.

4. CONCLUSION

This study successfully synthesized and comprehensively characterized activated charcoal from coconut shells via H_2SO_4 chemical activation. Key findings include:

1. **Surface Chemistry**: FTIR confirmed successful functionalization with carbonyl, aromatic, and phenolic/ether groups that enhance adsorption via electrostatic, H-bonding, and π–π interactions.
2. **Textural Properties**: BET analysis revealed a mesoporous structure (52.96 m^2/g surface area, 4.07 nm average pore diameter) suitable for liquid-phase applications, though lower than literature values indicating optimization potential.
3. **Morphology**: SEM imaging confirmed hierarchical porosity with macroporous transport channels and mesoporous active sites, albeit with minor particle agglomeration.
4. **Integrated Performance**: The combined chemical and textural properties support multi-mechanism adsorption, validating the material's potential for environmental remediation applications.

This work establishes foundational characterization benchmarks for H_2SO_4-activated coconut shell charcoal and provides a basis for future activation parameter optimization to enhance surface area and adsorption capacity.

5. RECOMMENDATIONS FOR FUTURE WORK

- Systematic screening of activation parameters (H_2SO_4 concentration, carbonization temperature/time) to maximize surface area and pore volume.
- Exploration of alternative activating agents (KOH, $ZnCl_2$, H_3PO_4) for comparative property development.
- Advanced characterization: XRD for crystallinity, TGA for thermal stability, Boehm titration for quantitative surface acidity.
- Scale-up studies: Pilot-scale production, energy balance analysis, and life-cycle assessment for techno-economic feasibility.

References

1. Khan MN, Sial TA, Ali A, Wahid F. Impact of agricultural wastes on environment and possible management strategies. Cham: Springer International Publishing; 2024.
2. Faizal ANM. Preparation of NaOH-activated coconut shell carbon for methylene blue adsorption. Desalination Water Treat. 2024. doi:10.1080/10406026.2024.2393604.
3. Abbas M, Trari M. Adsorption behavior of methylene blue onto activated coconut shell. J Environ Health Insights. 2024. doi:10.1177/15593258241290708.

4. Obeng GY, Amoah DY, Opoku R, Sekyere CK, Adjei EA, Mensah E. Coconut wastes as bioresource for sustainable energy: Quantifying wastes, calorific values and emissions in Ghana. Energies. 2020;13(9):2178.

5. Schroeder P, Anggraeni K, Weber U. The relevance of circular economy practices to the sustainable development goals. J Ind Ecol. 2019;23(1):77-95.

6. Wazir AH, Ullah I, Yaqoob K. Coconut biomass-derived activated carbon for dye removal from wastewater. Environ Eng Sci. 2023. doi:10.1089/ees.2022.0373.

7. Rammal J, et al. Comparative analysis of natural and synthetic materials for wastewater treatment: Plant powders, activated carbon, biochar, zeolite, and nanomaterials. J Chem Rev. 2025;7(4):566-590.

8. Mahapatra S, Kumar D, Singh B, Sachan PK. Biofuels and their sources of production: A review on cleaner sustainable alternatives against conventional fuel. Energy Nexus. 2021;4:100036.

9. Islam MA, Ahmed MJ, Khanday WA, Asif M, Hameed BH. Mesoporous activated coconut shell-derived hydrochar prepared via hydrothermal carbonization-NaOH activation for methylene blue adsorption. J Environ Manage. 2017;203:237-244.

10. Yang K, Peng J, Srinivasakannan C, Zhang L, Xia H, Duan X. Preparation of high surface area activated carbon from coconut shells using microwave heating. Bioresour Technol. 2010;101(15):6163-6169.

11. Boudalia S, et al. The valorization of agrifood byproducts and waste to advance the sustainable development goals: Current state and new perspectives. Sustainability. 2026;18(5):2165. doi:10.3390/su18052165.

12. Hu Z, Srinivasan MP. Preparation of high-surface-area activated carbons from coconut shell. Microporous Mesoporous Mater. 1999;27(1):11-18.

13. Wang X, et al. Optimization of mesoporous activated carbon from coconut shells by chemical activation with phosphoric acid. BioResources. 2013;8(4):6184-6195.

14. Ioannidou O, Zabaniotou A. Agricultural residues as precursors for activated carbon production—A review. Renew Sustain Energy Rev. 2007;11(9):1966-2005.

15. Tan IAW, Ahmad AL, Hameed BH. Adsorption of basic dye using activated carbon prepared from oil palm shell: Batch and fixed-bed studies. Desalination. 2008;225(1-3):13-28.

16. Saleem J, Moghal ZKB, Pradhan S, McKay G. High-performance activated carbon from coconut shells for dye removal: Study of isotherm and thermodynamics. RSC Adv. 2024;14(46):33797-33808.

17. Cazetta AL, et al. NaOH-activated carbon of high surface area produced from coconut shell: Kinetics and equilibrium studies of methylene blue adsorption. Chem Eng J. 2011;174(1):117-125.

18. Lua AC, Yang T. Characteristics of activated carbon prepared from pistachio-nut shell by zinc chloride activation under nitrogen and vacuum conditions. J Colloid Interface Sci. 2005;290(2):505-513.