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PRODUCTION AND CHARACTERIZATION OF ACTIVATED CARBON FROM COCONUT SHELL: EFFECT OF ACTIVATING AGENTS

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Abstract
The choice of chemical activating agents plays a vital role in shaping the morphology, porosity, and adsorption efficiency of biomass-derived activated carbon. In this work 1M solution of HCl, H_2SO_4, KOH, and NaOH, were used to activate the precursor for 6 hr, followed by carbonization at 500°C for 1 hr and their effects were investigated using morphological analysis, iodine number determination, and FTIR spectroscopy. Surface observations revealed that HCl produced irregular structures with mineral residues and limited porosity, whereas H_2SO_4 generated compact, layered frameworks that were stable yet less porous. By contrast, KOH produced highly porous structures with well-developed micro- and mesopores, identifying it as the most effective base activator. Iodine analysis indicated that acid-activated carbons exhibited the highest adsorption capacities, with H_2SO_4 (1795.00 mg/g) and HCl (1793.10 mg/g) outperforming KOH (1730.28 mg/g) and NaOH (1699.83 mg/g). The effectiveness order followed $H_2SO_4 \approx HCl > KOH > NaOH$, underscoring the stronger adsorption potential of acid treatments. In other word, H_2SO_4 promotes dehydration, sulfonation, and mesopore formation, while HCl primarily enhances porosity by leaching minerals. FTIR spectra supported these differences: KOH and NaOH exhibited similar oxygenated groups (C–O, O–H, aromatic C=C), H_2SO_4 introduced additional sulphate functionalities alongside stronger O–H and C=O peaks, and HCl increased acidic oxygen groups without sulfonation. In conclusion, KOH is effective for micropore development, but acid activators (H_2SO_4, HCl) deliver superior adsorption capacity through enhanced surface chemistry. These findings emphasize the need to tailor activating agents to the intended application by balancing pore structure and surface functionality.

1.0 INTRODUCTION

Activated carbon is a widely used adsorbent in water purification, gas separation, catalysis, and environmental remediation, owing to its high surface area, well-developed porosity, and abundance of surface functional groups. Its performance depends largely on the precursor material and activation method. Among biomass precursors, coconut shell is particularly

attractive due to its high lignin and carbon content, low ash content, mechanical strength, and availability in tropical regions (Chew et al., 2023; Baharum et al., 2020).

The production of activated carbon typically involves carbonization to remove volatile matter, followed by activation to develop porosity and surface chemistry. Activation may be achieved by physical means (e.g., steam or CO_2 at high temperature) or chemical methods (using acids, bases, or salts). Chemical activation is generally more effective, as it facilitates pore development at lower temperatures while simultaneously introducing surface functional groups (Chew et al., 2023; Ademiluyi and David-West., 2012).

The choice of activating agent significantly influences the final properties of activated carbon. Alkaline activators such as potassium hydroxide (KOH) and sodium hydroxide (NaOH) intercalate into the carbon matrix, promoting micropore formation through redox and etching reactions. KOH is particularly effective, often producing sponge-like carbons with exceptionally high surface areas, while NaOH yields similar but less pronounced effects (Samghouli *et al*., 2025). In contrast, acidic activators such as hydrochloric acid (HCl) and sulfuric acid (H_2SO_4) act mainly through demineralization and oxidation. HCl removes residual minerals and enlarges existing pores, whereas H_2SO_4 promotes dehydration and sulfonation, producing more stable structures enriched with acidic groups (S=O, carbonyl, carboxyl), which enhance polarity and adsorption of polar contaminants (Samghouli et al., 2025; Seo-Hyun Pak, 2016).

To evaluate these effects, a range of characterization techniques are employed, including scanning electron microscopy (SEM) for morphological analysis, iodine number or BET surface area measurements for adsorption capacity, Fourier-transform infrared spectroscopy (FTIR) for surface functional groups, and proximate/ultimate analysis for elemental composition and thermal stability.
By comparing different activating agents, researchers can tailor pore structure and surface chemistry to specific applications. While KOH remains the most effective for developing microporous carbons suited for gas adsorption, acid activators such as H_2SO_4 and HCl are advantageous in applications requiring enhanced surface acidity and polarity, including catalysis and wastewater treatment. Therefore, this study investigates the production and characterization of coconut-shell-derived activated carbon using HCl, H_2SO_4, KOH, and NaOH, with emphasis on their effects on morphology, porosity, surface chemistry, and adsorption performance.

2.0 MATERIALS AND METHODS

Coconut Shell, Beaker, Crusher and milling machine, Oven, Muffle Furnace, Sieve (0.28 mm), pH Meter, Glassware, Weighing Balance, Stop watch, Filter paper, Hydrochloric Acid (HCl), Sulphuric Acid (H_2SO_4), Potassium hydroxide (KOH), Sodium hydroxide (NaOH), distilled water.

2.1 Raw Materials Collection and Treatment:

Coconut shells were collected from local markets and roadside vendors in Bakin Dogo Market, Kaduna State, chosen for their availability, low cost, and high carbon content. The shells were manually broken to remove residual pulp and fibers, then thoroughly washed with clean tap water followed by distilled water to eliminate dirt, dust, and soluble impurities. The cleaned shells were sun-dried for 2-3 days and then oven-dried at 120°C for 1 hour to ensure complete removal of moisture. The dried shells were stored in airtight containers prior to particle size reduction and carbonization to prevent reabsorption of atmospheric moisture.

2.2 Crushing/Grinding Process:
The cooled coconut shell was subjected to mechanical size reduction to enhance its surface area and facilitate uniform chemical activation. The charred material was first manually broken into smaller fragments, then transferred to a high-speed mechanical grinder for fine grinding. The grinding process was carefully controlled to avoid excessive heat generation, which could alter the material's microstructure. The ground material was subsequently sieved using a standard mesh (0.28 mm) to obtain a uniform particle size distribution suitable for carbonization and activation. This size uniformity is essential to ensure consistent pore development during the activation process and to enhance adsorption efficiency during wastewater treatment.

2.3 Carbonization and Activation Process:
The coconut shell powder was treated with 1M solution of different activating agent (HCl, H_2SO_4, NaOH and KOH) for 6 hour at room temperature. The paste was washed with distilled water to a neutral pH, followed by oven drying overnight at 120^0C. The oven dried samples were subjected to carbonization in a muffle furnace under limited oxygen conditions at temperature of 500°C for 1 hour. This process facilitated the removal of volatile organic compounds and the formation of a rigid carbon skeleton. The resulting coconut shell activated carbon was allowed to cool in a desiccator to avoid moisture uptake and was then ground using a mechanical grinder. The ground material was sieved using a standard mesh (0.28 mm) and stored in air-tight labeled sample bottle for characterization.

2.4 Characterization of Produced Activated Carbon:

1. Scanning Electron Microscopy (SEM) Analysis
The morphological structure of the coconut shell activated carbon was examined using SEM (PhenomWorld Pro X). This technique provided visual evidence of the porosity and surface roughness, which are critical for adsorption.

2. Fourier Transform Infrared (FTIR) Spectroscopy:
FTIR spectrophotometer (Shimadzu 8400s) was used to identify the functional groups present on the surface of the activated carbon. Samples were prepared using the KBr pellet method and analyzed within the wavenumber range of 4000–400 cm^{-1}. The spectra obtained were interpreted to determine the presence of key functional groups such as hydroxyl (–OH), carbonyl (C=O), and carboxylic (–COOH) groups, which are responsible for adsorption. This analysis provided insight into the surface chemistry and potential adsorption mechanisms of the activated carbon derived from coconut shell.

3. Iodine value
Iodine value (IV) of each sample was determined by the titration of 0.1 M Sodium thiosulphate solution against 60.0 ml of the sample free liquor solution (prepared 0.5 g of each samples in 75.0 ml of 0.086 M iodine solution using starch indicator. The iodine value (IV) was calculated using the formula: $IV\ (mg/g) = (Y - X) * V * M * 12.69\ W$ Where Y = volume of thiosulphate for blank, X = Titer value, V = volume iodine solution used, W= Weight of sample. M = molarity of Sodium thiosulphate solution.

3.0 RESULTS AND DISCUSSION

3.1 SEM analysis

The morphological structure of the prepared activated carbon using HCl, H_2SO_4, NaOH and KOH as activating agents, is presented in figure 4a-d, respectively. SEM analysis revealed clear differences in morphology depending on the activating agent. HCl-treated carbon showed heterogeneous surfaces with residual minerals and limited pore formation, while H_2SO_4-treated carbon produced compact, layered structures with low porosity but high surface functionality. In contrast, NaOH-treated carbon exhibited moderate porosity with shallow, less connected pores, whereas KOH-treated carbon developed highly porous, sponge-like morphologies with micropore and mesopore networks. These morphological trends were consistent with iodine adsorption capacities: H_2SO_4 (1795.00 mg/g) and HCl (1793.10 mg/g) gave the highest values, reflecting their strong influence on surface functionalization. KOH (1730.28 mg/g**)** outperformed NaOH (1699.83 mg/g) in pore generation, though both bases showed lower iodine values than the acids. FTIR confirmed these distinctions, with acids introducing stronger O–H and C=O functionalities (and sulphate groups in H_2SO_4), while alkalis primarily generated carboxyl and phenolic groups associated with microporosity.

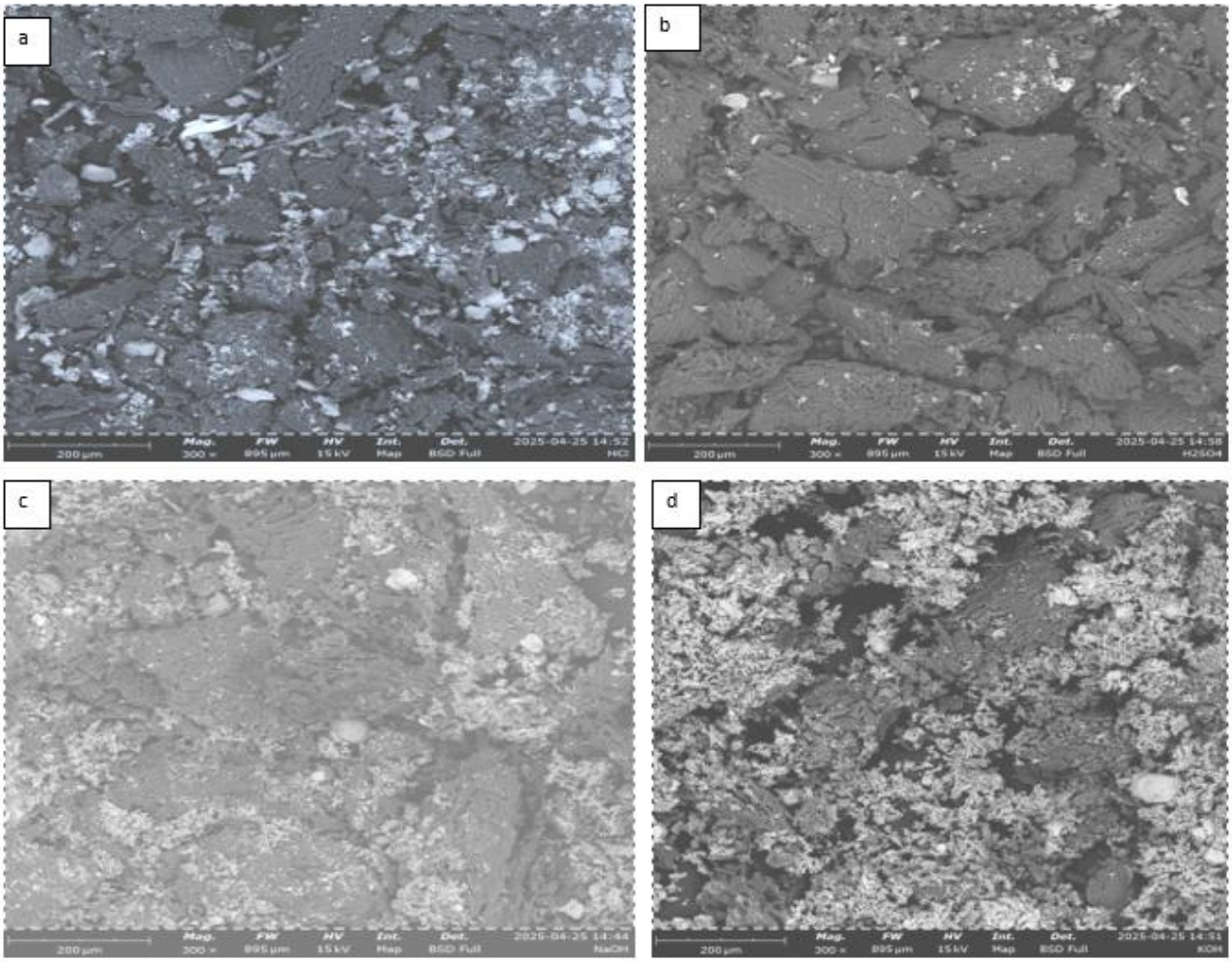

Figure 1. SEM Analysis of activated carbon using: a) HCl b) H_2SO_4 c) NaOH d) KOH

3.2 Iodine value analysis

The iodine values of the samples ranged between 1699.83 and 1795.00 mg/g as presented in Table 1. The highest adsorption capacity was obtained with H_2SO_4 (1795.00 mg/g), closely

followed by HCl (1793.10 mg/g). NaOH (1699.83 mg/g) showed the lowest value, indicating the weakest activation effect. KOH (1730.28 mg/g) performed better than NaOH but was less effective than the acids. In general, acid-activated carbons (H_2SO_4 and HCl) demonstrated greater adsorption potential compared to base-activated carbons (NaOH and KOH). H_2SO_4 and HCl showed nearly the same level of performance. The overall order of effectiveness was**:** $H_2SO_4 \approx HCl > KOH > NaOH$. Studies indicate that while H_2SO_4 and HCl can both serve as effective acid activators, their mechanisms differ: H_2SO_4 typically promotes dehydration/sulfonation and mesopore formation, whereas HCl primarily leaches minerals and enlarges pores (Chew et. al., 2023). Regarding bases, Research has shown that KOH outperforms NaOH in producing activated carbon with higher microporosity, BET surface area, and adsorption capacity (Togibasa *et. al*., 2021).

Table 1: IODINE VALUE DATA FOR FOUR CARBONIZED SAMPLES

S/N	SAMPLE ID	Blank Y (ml)	Sample X (ml)	Vol. Iodine (ml)	Thios. Conc. (Mol)	Const.	Mass (g)	Iodine Value IV = (y-x)*Vol.*Conc.*Const./Mass
1	NaOH	9.6	0.67	75	0.1	12.69	0.5	1699.8255
2	H_2SO_4	9.6	0.17	75	0.1	12.69	0.5	1795.0005
3	KOH	9.6	0.51	75	0.1	12.69	0.5	1730.2815
4	HCl	9.6	0.18	75	0.1	12.69	0.5	1793.097

3.3 FTIR Spectral Analysis of Activated Carbon

The presence of various functional groups on the surface of the samples was analysed using Fourier Transform Infrared (FTIR) spectrometer (Shimadzu 8400s) and the IR spectra of the activated carbon prepared from coconut shell using HCl, H_2SO_4, NaOH and KOH as activators are shown in figures 3, 4, 5 and 6, respectively. The resulting spectra revealed distinct functional group profiles, each reflecting the chemical influence of the activating agent on the carbon's surface properties. Activated carbon treated with HCl displayed peaks associated with hydroxyl (O–H) (3693.3 cm^{-1}) and aliphatic (C–H) (2974.8, 2850.6 cm^{-1}) groups, as well as carbonyl or aromatic (C=O/C=C) (1643.1 cm^{-1}) functionalities. These groups contribute to adsorption via hydrogen bonding and π–π interactions (Kumar et al., 2023). In comparison, H_2SO_4-treated carbon exhibited more intense O–H bands (3630.4–3749.7 cm^{-1}), along with sulfate-related S=O vibrations (1420.1 cm^{-1}) and out-of-plane aromatic bending mode (674.6 cm^{-1}), similar to what was reported by Ali *et. al*., 2020. The presence of these oxygen-rich groups enhances hydrophilicity and electrostatic attraction, particularly for organic pollutants, like cationic dye (Shojaei & Esmaeili, 2022). NaOH activation resulted in strong O–H (3049 cm^{-1}) and C–H (2881.2 cm^{-1}) stretching, along with carbonyl (C=O) (2642.70 cm^{-1}), and carboxyl-related (C–O) (1423.8 cm^{-1}) vibrations. These functionalities are known to facilitate adsorption through ion exchange and surface complexation (Kazempour & Bagheri-Mohagheghi, 2023). KOH-treated carbon showed similar C–H stretching (3000.6 cm^{-1}), with additional bands suggesting triple bond vibrations, and prominent C–O (1114.5 cm^{-1}) and N-H (868.7 cm^{-1}) features, which indicates a highly porous structure with basic surface sites. The variation in functional group intensity and diversity across samples directly affects their adsorption behaviour. Acid treatments tend to introduce more polar and oxygenated groups while base treatments favour carboxyl and phenolic groups (Shojaei & Esmaeili, 2022; Zhao et al., 2022). FTIR Peak Assignments of Activated Carbon Prepared with Different Activating Agents are presented in Table 2.

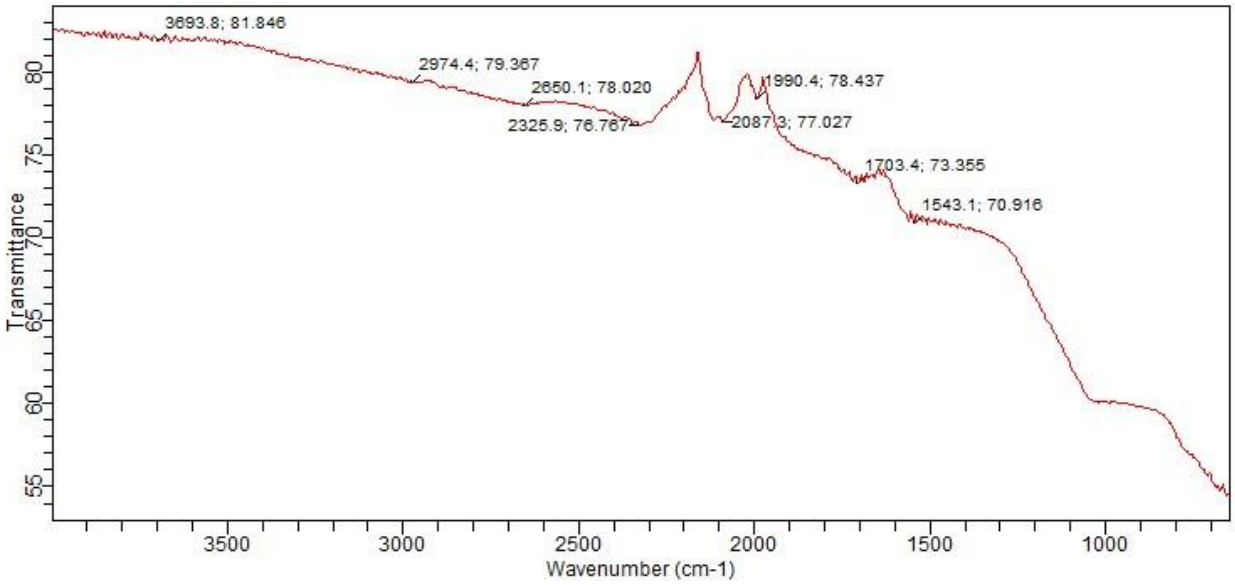

Figure 2. FTIR analysis using activated carbon prepared using HCl

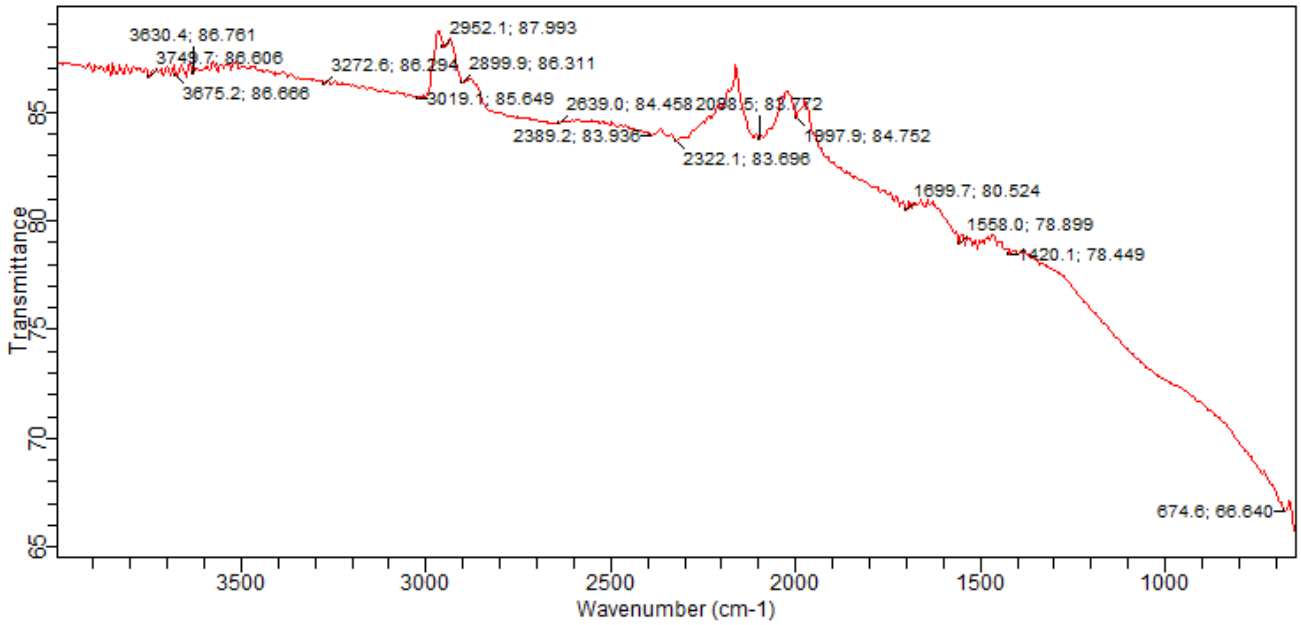

Figure 4.2. FTIR analysis using activated carbon prepared using H_2SO_4

4.0 CONCLUSIONS AND RECOMMENDATIONS

Activated carbon has been successfully prepared from coconut shell using HCl, H_2SO_4, NaOH and KOH as the activating agents. Surface analysis was done using SEM and FTIR spectroscopy, while the adsorption capacity was tested with iodine value analysis. Acid-activated carbons (H_2SO_4, HCl) showed the highest iodine values (1795 and 1793 mg/g), outperforming alkali-activated carbons (KOH: 1730 mg/g; NaOH: 1700 mg/g). H_2SO_4 promoted sulfonation and surface acidity, while HCl mainly enhanced oxygenation via demineralization. KOH produced the most porous sponge-like structure, ideal for micro porosity, whereas NaOH was the least effective. FTIR confirmed KOH and NaOH exhibited similar groups (C–O, O–H, aromatic C=C), H_2SO_4 introduced additional sulphate

functionalities alongside stronger O–H and C=O peaks, and HCl increased acidic oxygen groups without sulfonation, while bases favoured C–O functionalities. In conclusion, KOH is effective for micropore development, but acid activators (H_2SO_4, HCl) deliver superior adsorption capacity through enhanced surface chemistry. These findings emphasize the need to tailor activating agents to the intended application by balancing pore structure and surface functionality. The presence of functional groups such as hydroxyl, carboxyl, and aromatic groups enhances the material's performance in environmental applications.

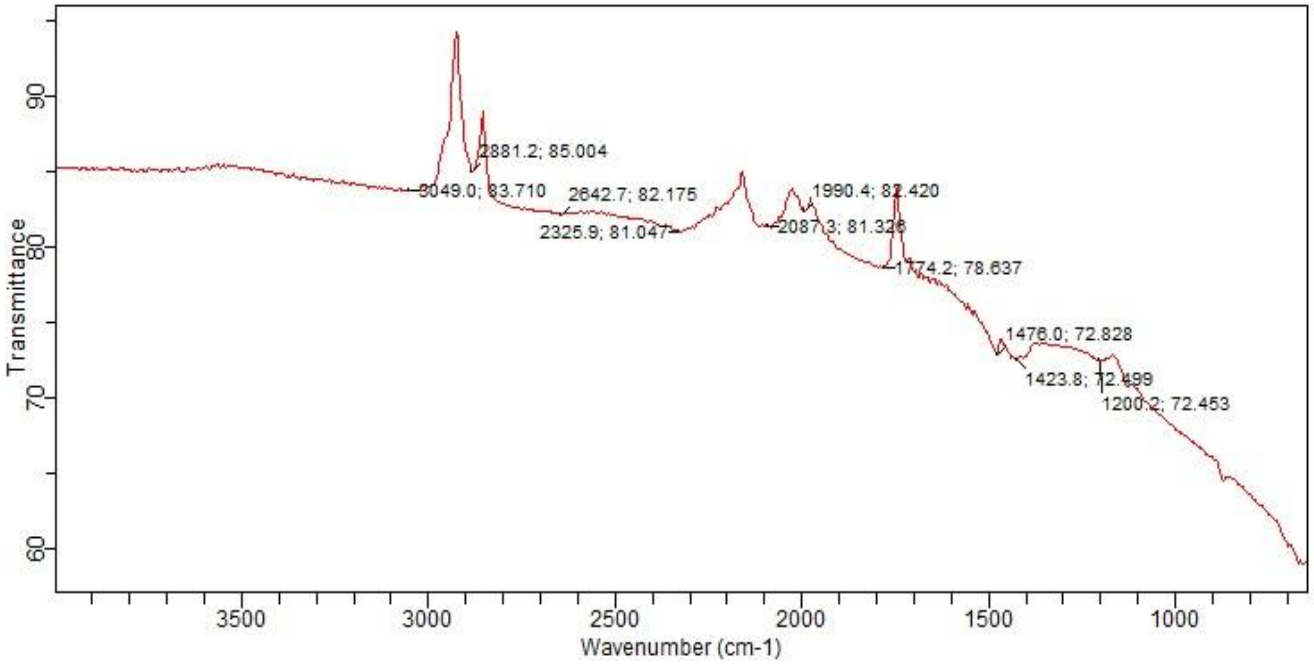

Figure 3. FTIR analysis using activated carbon prepared using NaOH

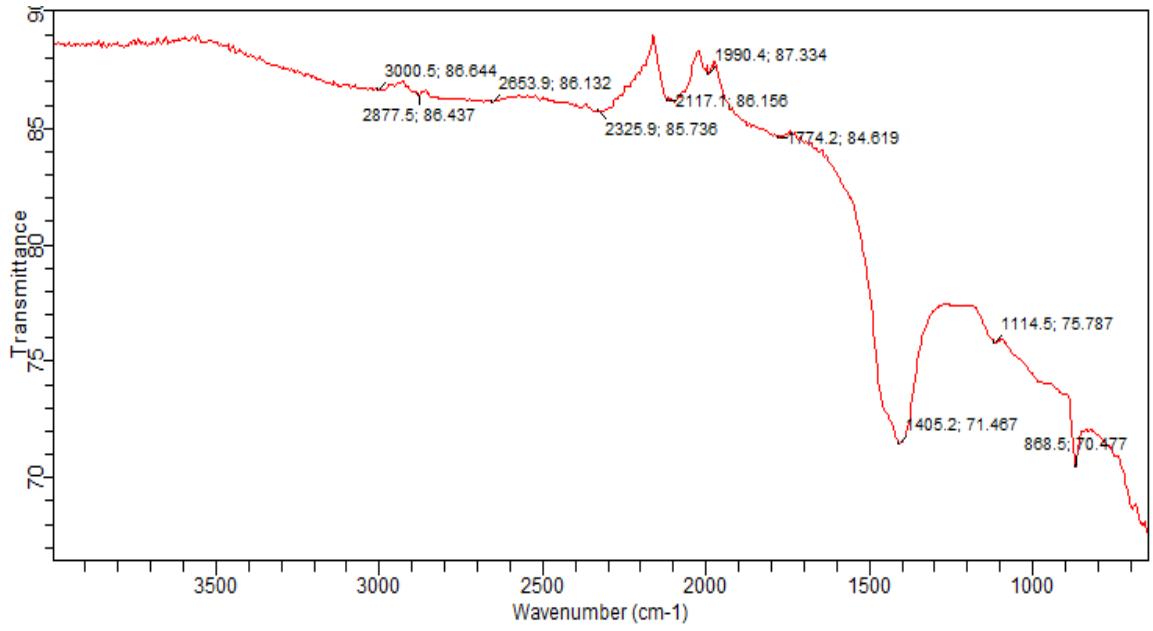

Figure 4. FTIR analysis using activated carbon prepared using KOH

Table 2. FTIR Peak Assignments of Activated Carbon Prepared with Different Activating Agents

Activating Agent	Wavenumber (cm^{-1})	Functional Group	Vibrational Mode	Reference
HCl	3693.3	O–H (hydroxyl)	Stretching	(Kumar et al., 2023)
	2974.8, 2850.6	C–H (alkyl)	Stretching	(Kumar et al., 2023)
	1643.1	C=O / C=C (carbonyl/aromatic)	Stretching	(Kumar et al., 2023)
H_2SO_4	3630.4–3749.7	O–H (hydroxyl)	Stretching	(Shojaei & Esmaeili, 2022)
	2952.1, 2899.9	C–H (alkyl)	Stretching	(Shojaei & Esmaeili, 2022)
	1699.7	C=O (carbonyl)	Stretching	(Shojaei & Esmaeili, 2022)
	1420.1	S=O (sulfate)	Stretching	(Shojaei & Esmaeili, 2022)
	674.6	Aromatic C–H	Out-of-plane bending	(Shojaei & Esmaeili, 2022)
NaOH	3049	O–H (hydroxyl)	Stretching	(Kazempour & Bagheri-Mohagheghi, 2023)
	2881.2	C–H (alkyl)	Stretching	(Kazempour & Bagheri-Mohagheghi, 2023)
	2642.7	C=O (carbonyl)	Stretching	(Kazempour & Bagheri-Mohagheghi, 2023)
	1423.8	C–O (phenolic/carboxylic)	Bending/Stretching	(Kazempour & Bagheri-Mohagheghi, 2023)
KOH	3000.6	C–H (alkyl)	Stretching	(Singh et al., 2021)
	1990.4	C≡C	Stretching	(Singh et al., 2021)
	1114.5	C–O (ether/phenolic)	Stretching	(Singh et al., 2021)
	868.7	N-H	Stretching	(Singh et al., 2021)

4.1 Recommendations

H_2SO_4 and HCl are best for water treatment (polar contaminants), while KOH is ideal for gas adsorption and energy storage. Future work should optimize activation conditions, explore acid–alkali sequential methods, and apply other characterization techniques for deeper characterization.

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Conflicts of Interest

The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results.

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