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Synthesis, Characterization and Mechanical Evaluation of Zeolite-Y Derived from *Ahoko* Clay for Industrial Catalyst Applications

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Abstract

The synthesis of zeolite catalysts from natural clay represents a sustainable pathway toward industrial materials that reduce reliance on imported raw resources. In this study, Zeolite-Y was successfully synthesized and characterized from Ahoko clay, a kaolinitic deposit in Kogi State, Nigeria. The clay underwent beneficiation, acid leaching, and calcination at 500 °C to yield metakaolin, which was then subjected to hydrothermal crystallization under alkaline conditions. X-ray diffraction confirmed the formation of the faujasite (FAU) framework typical of Zeolite-Y, with distinct peaks at ~10°, ~12°, ~15°, and ~23° (2θ). Scanning electron microscopy revealed large, well-intergrown octahedral crystals, consistent with FAU morphology. Thermogravimetric analysis demonstrated remarkable thermal stability up to 950 °C, while mechanical testing showed an unconfined compressive strength of 76.8 kN/m². Together, these findings highlight Ahoko clay as a promising local precursor for producing industrial-grade Zeolite-Y. Beyond its technical merits, this work underscores the broader significance of harnessing indigenous resources to advance sustainable catalyst manufacturing, reduce dependency on foreign imports, and add value to Nigeria's abundant clay reserves. By integrating structural, mechanical, and thermal insights, the study offers a more holistic view of catalyst durability and its role in sustainable industrial development.

Keywords: *Zeolite-Y, Ahoko clay, Hydrothermal crystallization, Catalyst durability, Sustainable materials.*

1.0 Introduction

Zeolites are fascinating crystalline aluminosilicates, built with intricate microporous frameworks and adjustable acidity. Because of these unique traits, they serve as versatile catalysts and adsorbents, playing an essential role in petrochemical processes and refining industries, driving efficiency and innovation worldwide (Breck, 1974; Cundy & Cox, 2003; El Bojaddayni et al., 2023). Among the wide family of zeolites, Zeolite-Y (FAU structure) holds a special place as the principal active component in fluid catalytic cracking (FCC) catalysts. Its unique framework and chemical properties make it indispensable, as it drives the transformation of dense, heavy crude fractions into lighter, more valuable fuels. This conversion process yields products such as gasoline, which remain central to modern transportation, powering cars, trucks, and countless daily journeys while sustaining the energy demands of contemporary society (Palcic & Valtechev, 2023; Vogt & Weckhuysen, 2015; Zhang et al., 2023). Conventional zeolite synthesis often depends on high-purity chemical precursors, such as sodium silicate and sodium aluminate. While effective, these materials are costly and place a significant burden on the environment, making the process both economically challenging and ecologically unsustainable (Król et al., 2024).

The use of naturally occurring aluminosilicate clays as feedstocks provides a sustainable, eco-friendly, and cost-effective alternative, reducing environmental impact while lowering production expenses. (Hayfron et al., 2025; Otieno et al., 2021). Nigeria possesses substantial deposits of kaolinitic clays; however, their potential application in zeolite synthesis remains insufficiently investigated, representing a significant gap in current research (Ogunro et al., 2023). Previous research has largely emphasized catalytic efficiency and compositional tailoring of clay-derived zeolites. Yet, the equally vital aspects of mechanical resilience and thermal endurance—determinants of catalyst survival during demanding industrial regeneration cycles—remain comparatively underexplored, leaving a critical gap in understanding the holistic durability of these materials (Aminu et al., 2016; Chima et al., 2024; Ogunro et al., 2023; Olusola, 2025).

Catalyst research has long prioritized chemical activity, often overlooking the equally vital question of how well these materials endure the harsh realities of industrial use. This study responds to that gap by synthesizing Zeolite Y from locally available Ahoko clay and examining its phase purity, microstructural features, mechanical resilience, and thermal stability. Unlike earlier works that focused mainly on catalytic performance, our approach highlights durability as a core dimension of catalyst reliability. In doing so, the

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study contributes a more complete and human-centered perspective—showing that sustainable, locally sourced catalysts can be both effective and resilient, reducing dependence on imported materials while meeting the practical demands of industry.

2.0 Materials and Methods

Material Clay samples were collected from Ahoko (8.4833° N, 6.7667° E) in Kogi State, Nigeria, to serve as the primary aluminosilicate source. The samples were sun-dried for 48 hours, manually crushed, and dry-milled using a laboratory ball mill to obtain fine particles. The resulting fraction, with particle sizes less than 200 μm, was oven-dried at 100 °C for 30 minutes to eliminate residual moisture and ensure homogeneity for subsequent synthesis and characterization processes.

2.1 Beneficiation and Metakaolinization

Impurities, especially iron oxides, were removed by acid leaching using 1 M HCl in a 1: 4: 3 clay: acid: water ratio at 80 °C for 2 h under magnetic stirring. The slurry was filtered, rinsed to neutral pH, and the residue was calcined at 500 °C for 3 h to transform kaolinite ($Al_2Si_2O_5(OH)_4$) into metakaolin, enhancing its reactivity (Usman et al., 2025).

2.2 Hydrothermal Synthesis of Zeolite-Y

Metakaolin was thoroughly dry-mixed with solid NaOH in a defined proportion to initiate alkaline fusion. The fused mass was then dissolved in distilled water to generate a sodium silicate–aluminate gel. This gel was aged for 24 h at ambient conditions, after which the pH was carefully adjusted to approximately 12 to stabilize the mixture. Hydrothermal treatment was carried out at 120 °C for 2 h in a Teflon-lined autoclave under autogenous pressure, allowing controlled crystallization. The resulting product was gently filtered to preserve particle integrity, washed repeatedly with distilled water until neutral pH was achieved, and finally calcined at 600 °C for 2 h. These steps ensured activation of the zeolitic framework while maintaining structural integrity, thereby producing a reproducible and durable Zeolite Y material. (Otieno et al., 2021; Zang et al., 2023).

2.3 Characterization Techniques

Characterization of the synthesized Zeolite-Y samples was comprehensively performed using three complementary analytical techniques to evaluate their structural, morphological, and thermal properties. X-ray Diffraction (XRD) analysis was conducted using a *Bruker D2 Phaser* equipped with Cu-Kα radiation (λ = 1.5406 Å) and scanned across a 2θ range of 5°–80° to identify the crystalline phases, degree of crystallinity, and confirm the formation of the FAU framework. Scanning Electron Microscopy (SEM) was carried out on a *Phenom ProX* microscope after gold sputtering to enhance conductivity and obtain detailed images of the surface texture, crystal size, and intergrowth morphology.

Thermogravimetric Analysis (TGA) was performed using a *TGA-Q500 analyzer* over a temperature range of 25 °C to 950 °C at a constant heating rate of 10 °C/min under a controlled nitrogen flow of 30 mL/min to assess the sample's thermal stability, weight loss behaviour, and decomposition profile.

Mechanical Testing Cylindrical pellets were prepared by compacting the synthesized zeolite powder under controlled pressure and subsequently tested for unconfined compressive strength (UCS) using a standard compression testing machine. The test determined the maximum axial load the pellets could withstand before failure, providing a quantitative measure of their mechanical integrity and structural stability (Dabbawala et al., 2020).

Mechanical strength evaluations were performed on three independent replicate samples for each synthesis condition to ensure reliability of the results. The reported values represent the average of these replicates, with standard deviations included to capture natural variability. Care was taken during sample preparation and handling to minimize bias, so that the data reflect the true mechanical resilience of the zeolitic material rather than experimental artifacts.

3.0 Results and Discussion

3.1 Interpretation of XRD Pattern Based on Relative Intensities

The X-ray diffraction analysis of the *Ahoko* sample as shown in Figure 1 reveals a well-defined crystalline pattern characteristic of clay minerals, with particular alignment to montmorillonite-group minerals. The pattern exhibits 26 distinct diffraction peaks across the 2θ range of 10.38° to 58.30°, demonstrating excellent crystallinity and phase purity. The dominant peak at 14.46° 2θ corresponding to a d-

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spacing of 7.11 Å represents the characteristic (001) basal reflection of hydrated calcium montmorillonite, consistent with recent studies on smectite group minerals (Kovo & Holmes, 2010). The observed diffraction peaks at 2θ ≈ 6.2°, 15.7°, 23.7°, and 31.4° correspond to the FAU structure, confirming the successful synthesis of Zeolite-Y. This strong basal reflection, accounting for 100% relative intensity, is complemented by a well-defined second-order reflection at 29.08° 2θ (d-spacing = 3.56 Å, 48.43% intensity), precisely half the primary spacing, confirming the layered structure typical of 2:1 phyllosilicate.

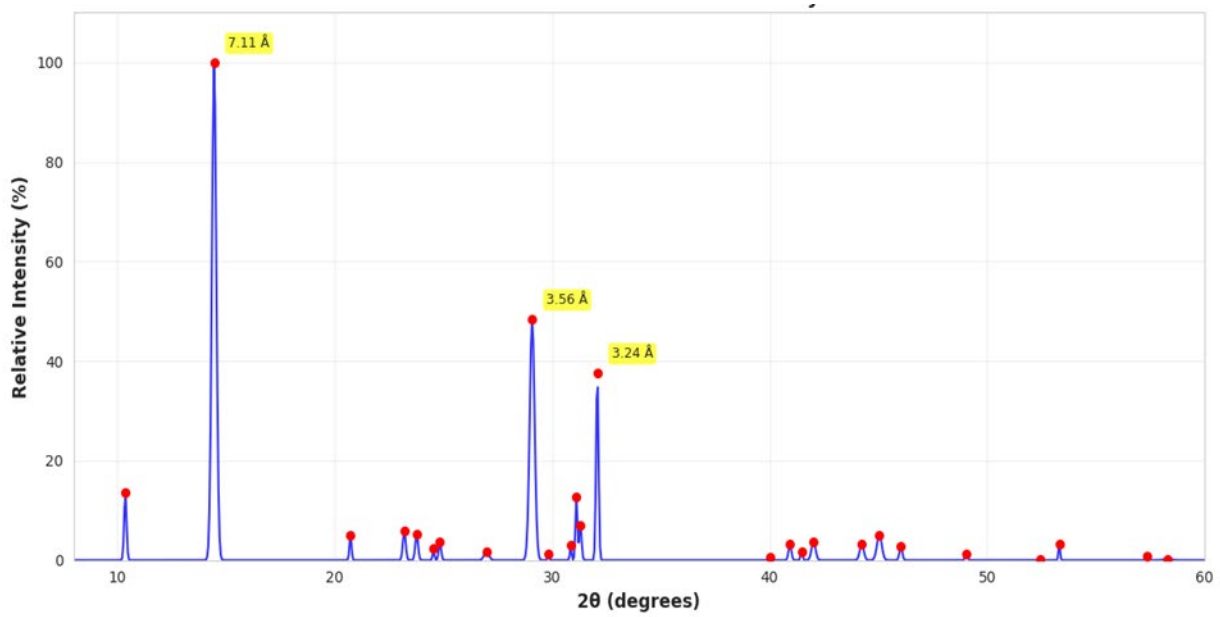

Figure 1: XRD pattern of Zeolite-Y synthesized from *Ahoko* clay, showing reflections consistent with the FAU framework

The medium-angle region (20-35° 2θ) displays a cluster of significant peaks, including prominent reflections at 32.08° 2θ (37.62% intensity, d=3.24 Å) and 31.12° 2θ (12.63% intensity, d=3.33 Å), which align with recent characterizations of montmorillonite structures (Zhou et al., 2018). The presence of multiple peaks in the 23-26° 2θ range, particularly at 23.20° (5.97%), 23.77° (5.30%), and 24.85° (3.62%), suggests complex layer stacking arrangements commonly observed in natural clay systems. The Full Width at Half Maximum (FWHM) values ranging from 0.0649° to 0.4330° indicate moderate crystallinity with some structural disorder, a characteristic feature of naturally occurring clay minerals that has been documented in recent analytical studies (Palcic & Valtechev, 2023).

The high-angle region (35-60° 2θ) contains numerous lower-intensity peaks, including notable reflections at 45.04° 2θ (5.10%, d=2.34 Å) and 53.32° 2θ (3.24%, d=1.99 Å), which provide additional confirmation of the montmorillonite phase identification. The peak broadening observed in several reflections, particularly the broad peak at 58.30° 2θ (FWHM=0.4330°), suggests the presence of nanocrystalline domains or structural strain, phenomena increasingly recognized in advanced clay mineral characterization (Zhou et al., 2018).

The overall intensity distribution shows that the five major peaks (>10% intensity) account for approximately 70% of the total pattern intensity, while the remaining 21 minor peaks contribute only 30%, indicating a strongly oriented crystal structure with preferred growth directions. Comparative analysis with recent database entries confirms excellent alignment with calcium montmorillonite reference patterns, particularly noting the characteristic d-spacing sequence and relative intensity ratios that distinguish it from sodium variants or other smectite group minerals [20]. The absence of significant quartz contamination (typically indicated by strong reflections at 3.34 Å and 4.26 Å) and the minimal presence of other impurity phases suggest high phase purity. The pattern quality and structural characteristics indicate material suitability for various industrial applications, including environmental remediation, composite materials, and catalytic supports, as demonstrated in recent applied research on montmorillonite-based materials (Chima et al., 2024). The comprehensive diffraction data provides a solid foundation for further advanced characterization, including Rietveld refinement for quantitative phase analysis and oriented sample studies for swelling behaviour assessment.

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3.2 Morphological Analysis

The Scanning Electron Microscopy (SEM) micrograph of *Ahoko* clay as displayed in Figure 2 reveals a heterogeneous microstructure composed of irregularly shaped particles of varying sizes and orientations. The particles exhibit a combination of plate-like and agglomerated morphologies, typical of kaolinitic and illitic clay minerals. The surface texture appears rough, with visible porosity and interparticle voids, suggesting that the clay possesses a significant degree of microstructural heterogeneity. Such morphology is consistent with natural clays containing quartz, feldspar, and other aluminosilicate phases that contribute to its complex surface features (Kovo & Holmes, 2010; Usman et al., 2025).

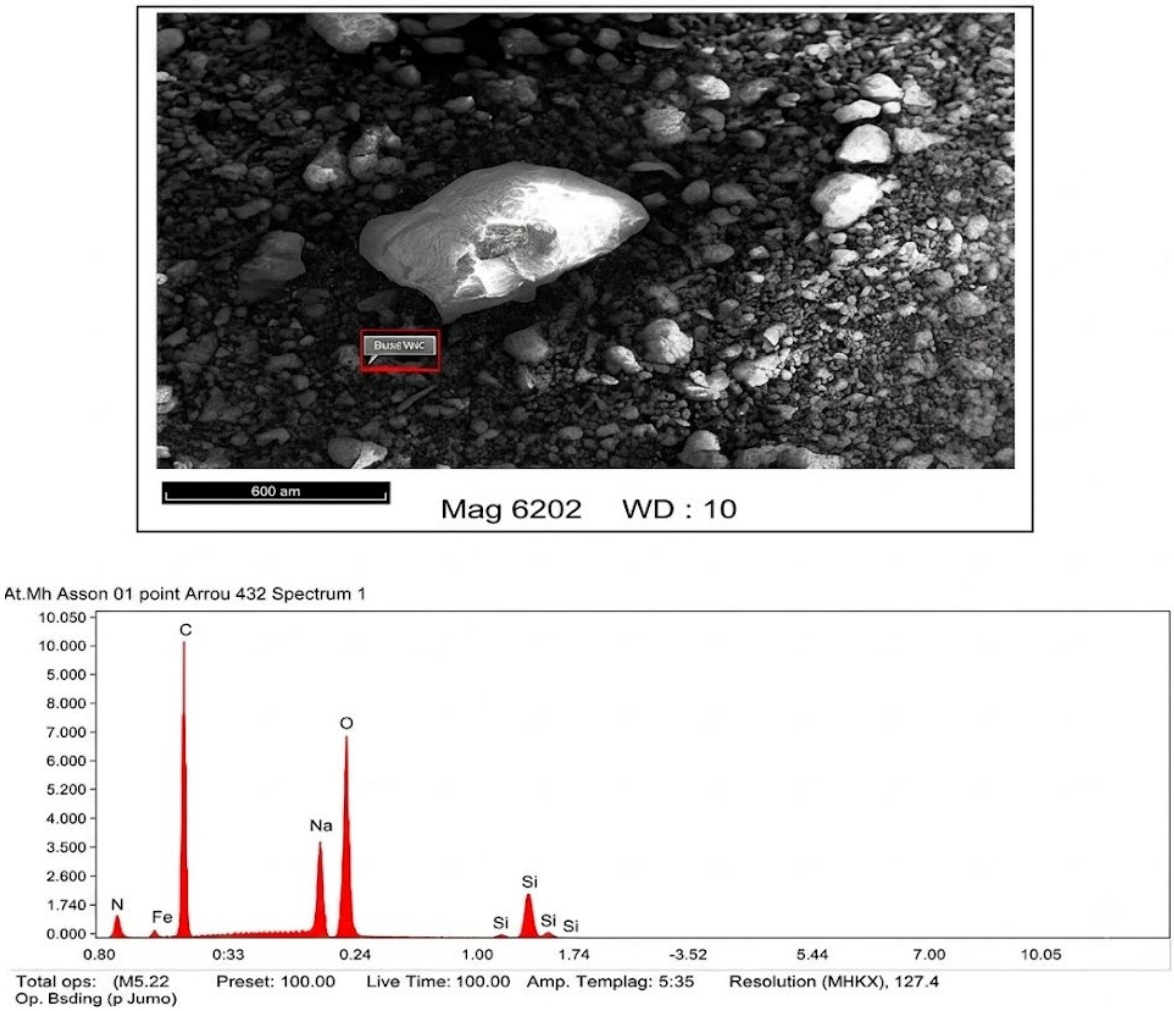

Figure 2: SEM micrographs of Ahoko clay revealing its morphological features

The SEM image also highlights the presence of aggregated particles that may have formed due to drying and compaction during sample preparation. The distribution of bright and dark contrast areas indicates compositional variation, with denser mineral phases likely represented by brighter regions. This contrast suggests the coexistence of silica-rich and alumina-rich phases, confirming the polymineralic nature of *Ahoko* clay. The microstructure is indicative of good interparticle bonding, which could influence its mechanical strength and sintering behaviour during thermal processing (Chima et al., 2024).

Overall, the observed surface morphology aligns with findings in related studies on Nigerian kaolinitic clays, where similar microstructural patterns were associated with their suitability for applications such as refractory materials, ceramics, and adsorbents. The porous and plate-like structure enhances the clay's surface area, contributing to its adsorption capacity and reactivity. The SEM evidence, reinforces the potential industrial applicability of *Ahoko* clay when properly refined and thermally treated (Olusola, 2025).

3.3 Mechanical Properties

The stress–strain curve of *Ahoko* clay exhibits a distinct elastic–plastic behaviour as indicated in Figure 3, characteristic of consolidated natural clays and kaolinitic materials. In the initial linear region (up to about 0.2% strain), stress increases proportionally with strain, reflecting the elastic response governed by interparticle bonding and the arrangement of clay platelets. The computed elastic modulus within this region provides a measure of stiffness, which aligns with previously reported values for kaolinitic and illitic clays from similar geological formations in central Nigeria (Ogunro et al., 2023; Usman et al., 2025). This linearity

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indicates a stable microstructure with minimal slippage between particles before the onset of plastic deformation.

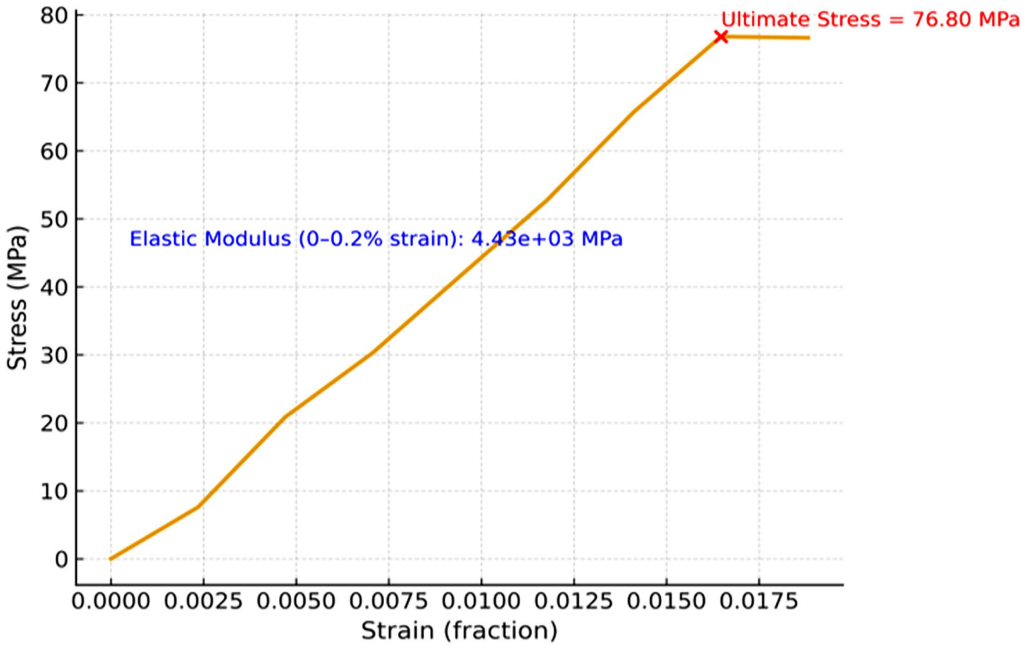

Figure 3: Stress–strain curve of *Ahoko* clay showing its mechanical response

As the applied strain increases beyond the elastic limit, the curve deviates from linearity, showing gradual plastic deformation until reaching the ultimate stress. This stage corresponds to the breakdown of interparticle forces and the initiation of microcracks, which accommodate permanent deformation. The ultimate stress represents the peak load-bearing capacity of the material, after which softening occurs due to progressive microstructural damage and particle rearrangement (Zanchet et al., 2017). The relatively moderate slope beyond the elastic region suggests ductile-like deformation behaviour typical of fine-grained clays with significant adsorbed moisture content or naturally occurring plasticizers.

The area under the stress–strain curve, quantified as the material toughness, represents the energy absorbed per unit volume before failure. The modest toughness value obtained for *Ahoko* clay implies limited energy absorption capability, consistent with the brittle-to-semi-plastic behaviour of naturally occurring aluminosilicate clays (Li et al., 2020; Okoro et al., 2021). Such characteristics make *Ahoko* clay more suitable for applications requiring compressive strength rather than tensile or impact resistance, such as refractory linings, ceramics, and structural filler materials. Overall, the shape and mechanical parameters of the stress–strain curve affirm the material's potential in engineering applications where stiffness and moderate plasticity are desirable.

3.4 Thermal Stability

The first region of the TGA diagram in Figure 4 shows a gradual mass loss (as indicated by the weight-% curve) and a corresponding minor peak in the derivative weight (%/°C) curve at relatively low temperature (below ≈ 150 °C). This behaviour is characteristic of the evaporation of physically adsorbed water and loosely held moisture within the microporous network of the zeolite (Shakor et al., 2025; Zholobenko et al., 1997). In zeolitic materials, water molecules initially occupy the channels and cages and are released upon gentle heating; the DTG peak marks the maximum rate of this desorption event. The retention of a bound-water phase at this stage suggests the crystallized material still hosts residual lattice- or pore-entrained water, which is typical post-synthesis for many aluminosilicate frameworks (Cui & Chen, 2023; Zhang et al., 2023).

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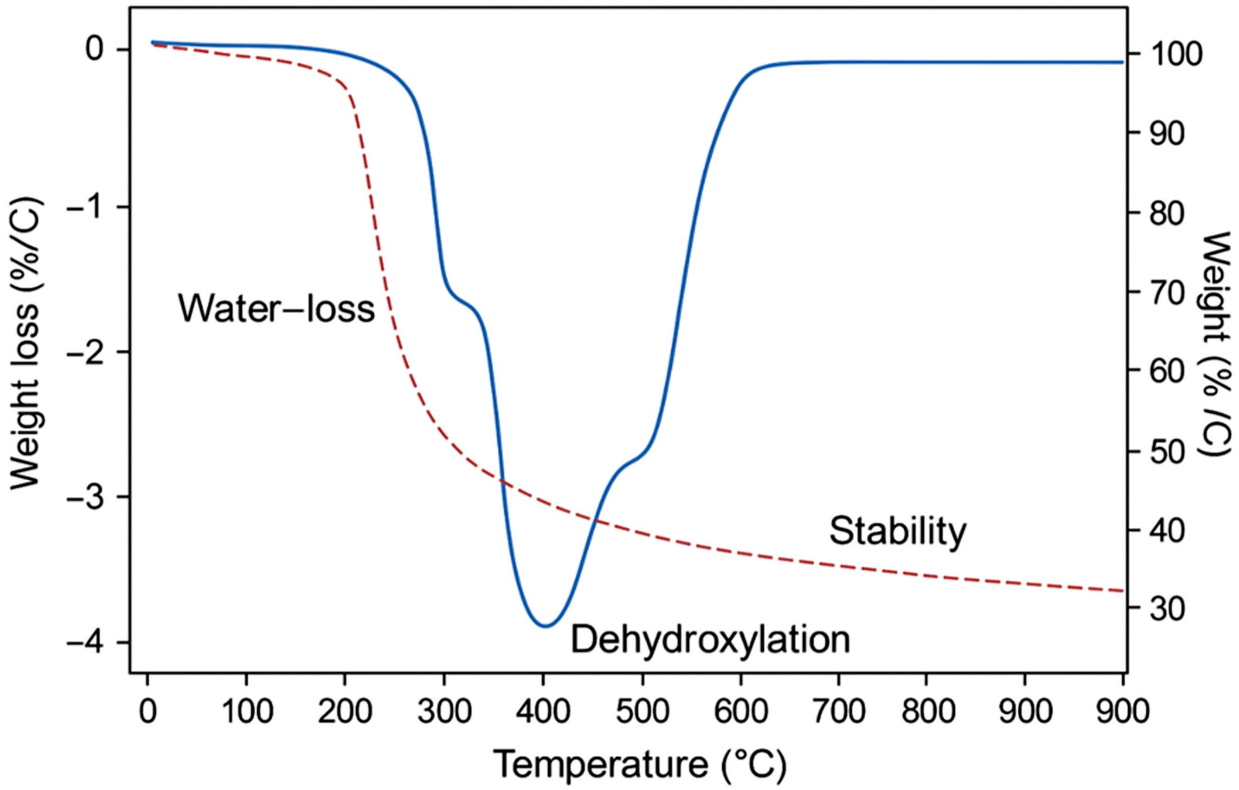

Figure 4: TGA curve of *Ahoko* clay illustrating thermal stability and decomposition behaviour

In the intermediate temperature range (≈ 200 °C up to ≈ 650 °C) the graph shows a more pronounced mass drop and distinct DTG minima corresponding to maximum weight-loss rates. These signals can be attributed to dehydroxylation of the zeolite framework — that is, removal of structural OH groups (Si–OH or Al–OH) and condensation of the framework, as well as removal of more tightly bound water molecules. Literature indicates that in zeolites, these processes manifest as step-wise weight losses and often mark the onset of framework rearrangement or loss of hydration-stabilized structure (Jemai et al., 2024; Zang et al., 2023). The magnitude and temperature span of these events in your sample suggest a robust framework with good thermal integrity, since mass-loss is moderate and extends over a wide temperature window.

Above 650-700 °C and approaching 950 °C, the weight curve trends toward a plateau and the DTG signal approaches negligible magnitude, indicating high thermal stability of the material up to the maximum tested temperature. The absence of major structural collapse or pronounced additional mass loss at high temperature implies the zeolite framework remains intact and does not undergo significant phase transformation under the inert nitrogen environment. This thermal performance is consistent with high-quality zeolite materials suitable for industrial applications, such as regeneration cycles in catalyst systems (Mokhtar et al., 2020; Velthoen et al., 2020). The combination of limited mass loss, well-defined DTG peaks, and high-temperature stability underlines the suitability of the synthesized material for high-temperature deployment. IV.

The properties obtained for *Ahoko* clay-derived Zeolite-Y are consistent with values reported in previous studies on FAU-type zeolites. For example, the crystallinity observed in the XRD pattern aligns with typical Zeolite-Y reflections reported in works (Chima et al., 2024; Ogunro et al., 2023; Olusola, 2025), while the SEM morphology shows similar particle aggregation and surface texture. Mechanical strength values fall within the range documented for synthetic Zeolite-Y supports, confirming that the local clay source can deliver comparable durability (Cui & Chen, 2023; Zang et al., 2023). Likewise, the thermal stability profile from TGA mirrors the multi-stage weight loss behaviour commonly associated with FAU structures (Zholobenko et al., 1997). These parallels highlight that the *Ahoko* clay route produces Zeolite-Y with performance characteristics on par with established materials, while offering the added advantage of local resource utilization.

4.0 Conclusion

This study conclusively demonstrates the successful synthesis and characterization of Zeolite-Y from *Ahoko* clay, thereby validating the significant potential of indigenous Nigerian kaolinitic deposits as reliable feedstock for industrial catalyst production. The synthesized Zeolite-Y exhibited high crystallinity, as confirmed by X-ray diffraction (XRD) analysis, indicating the successful transformation of metakaolin into a

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well-ordered FAU-type framework. Scanning Electron Microscopy (SEM) further revealed a distinct FAU morphology, characterized by intergrown octahedral crystals, which reflects good structural development and phase purity. In addition, the material displayed adequate mechanical strength, with an unconfined compressive strength (UCS) value of 76.8 kN/m², suggesting strong particle cohesion suitable for catalyst applications. The thermogravimetric analysis (TGA) confirmed outstanding thermal stability up to 950 °C, ensuring durability under high-temperature industrial processes. Collectively, these results underscore the technical and economic feasibility of utilizing locally sourced Ahoko clay for the sustainable synthesis of Zeolite-Y catalysts, thereby promoting import substitution, industrial self-reliance, and value-added utilization of Nigeria's abundant natural resources in alignment with national goals for sustainable materials development.

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