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# Physicochemical Characterization of Zeolite Materials Produced from Selected Low-Cost Agricultural Wastes

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**ABSTRACT:** Zeolites play a crucial role in adsorption processes for eliminating pollutants from industrial effluents and are extensively utilized in catalytic activities across diverse syntheses. This study focused on the conversion of three agricultural wastes—corncob, groundnut shell, and sugarcane bagasse—into zeolitic materials through three analytical methods: hydrothermal, microwave sintering, and alkali fusion, respectively. Standard methods were employed to assess the physicochemical parameters of the resulting zeolites. Generally, the produced zeolites had excellent surface areas and porosities. However, zeolites synthesized from sugarcane bagasse via the alkali fusion method exhibited highest porosity (80.9%), whereas that derived from corncob demonstrated highest surface area (1335 $m^2/g$). Point of zero charge for the produced zeolites was within the range of 7.9-9.8, with zeolite produced from groundnut shells via alkali fusion having the highest value (9.8). The elevated porosities and surface areas, and point of zero charge of the synthesized zeolites signify their enhanced adsorptive capacity, implying their suitability for application as adsorbents for the removal of organic and inorganic pollutants. These findings underscore the effectiveness of synthesized zeolites in reducing or completely removing pollutants such as phenols, dyes, pesticides, heavy metals, and inorganic anions from wastewater.

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Zeolites, members of the microporous crystalline aluminosilicate family, are commonly known as molecular sieves (Abdul Khaleque, 2020). They exhibit three-dimensional structures composed of interconnected networks of $[SiO_4]^{4-}$ and $[AlO_4]^{5-}$ tetrahedra bonded by oxygen atoms (Ramezani *et al.*, 2019). The general chemical formula for zeolites is $M_{a/b}[(AlO_2)_a(SiO_2)_y].cH_2O$ where M represents either an alkali metal or an alkaline earth metal cation, *b* represents the valence of the earth metal cation, *c* denotes the amount of water of crystallization per unit cell, and *a* and *y* indicate the total number of $[SiO_4]^{4-}$ and $[AlO_4]^{5-}$ tetrahedra in a unit cell of the zeolite, respectively (Lakiss *et al.*, 2020). The ratio y/a typically falls within the range of 1.0 to 5.0, although this value can vary depending on the specific structure. For instance, silica-based zeolites may have values ranging from 10.0 to 100. Zeolites are found both naturally and can be synthesized in the laboratory. They are commonly categorized into two main groups: natural (e.g., clinoptilolite, mordenite, and garronite) and synthetic zeolites (e.g., Zeolites A, P, X, and Y) (Johnson and Arshad, 2014).

Currently, there exist more than two hundred types of zeolites, categorized according to their silica-alumina

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ratio. Natural zeolites have been noted for their superior resistivity and thermal stability across diverse environments. Their thermal stability and chemical resistance are heightened with increasing silica-alumina ratio and the presence of alkali cations within the zeolite framework (Megalhaes *et al.,* 2022). A lattice unit functions as the foundational element of the repeating units, with a correlation existing between the tetrahedral primary building unit (PBU) and the secondary building unit (SBU) structure. The SBU essentially serves as the primary component of the zeolite framework, with nine distinct SBUs identified thus far (Lakiss *et al.,* 2020). Recently, researchers have turned their focus to another class of recyclable adsorbents based on hydrophobic zeolites (possessing a high Si/Al ratio) within the realm of water treatment. The notable properties of zeolites, including their substantial adsorption capacities and stability, can be leveraged for the adsorption process. Understanding the impacts of various physicochemical properties of zeolites is imperative for effectively removing diverse pollutants from wastewater (Abdul Khaleque, 2020).

Agricultural wastes refer to residues from the cultivation and processing of raw agricultural products, which may contain materials beneficial to humans (David and Tope, 2018). Globally, it is estimated that approximately 998 million tonnes of agricultural wastes are generated annually. These residues stem from various agricultural activities, especially cultivation. Recently, agricultural waste management has emerged as a significant concern, particularly in Northern Nigeria. The conventional approach to managing agricultural waste typically involves its disposal into the environment, with or without treatment. However, there is a pressing need to explore environmentally sustainable methods of utilizing these agricultural wastes as potential resources rather than regarding them as undesirable and unwanted. This shift aims to prevent the contamination of air, water, and land resources, as well as the transmission of hazardous materials. Achieving this goal will necessitate the adoption of improved technology and incentives (Obi *et al.,* 2016).

In recent years, agricultural wastes have emerged as a cost-effective alternative for treating effluents containing heavy metals through the adsorption process. Various researchers have investigated the use of low-cost agricultural wastes such as sugarcane bagasse, rice husk, sawdust, coconut husk, oil palm shell, neem bark, and others for removing heavy metals from wastewater (Abdulrazak *et al.,* 2016). In Northern Nigeria, agricultural wastes chosen for this study—corn cob, groundnut shell, and sugarcane bagasse—are typically discarded after processing or burned in significant quantities, leading to environmental contamination (Abdulrazak *et al.,* 2014). The objective of the study was to synthesize and compare the physicochemical and adsorptive capacities of zeolitic materials produced from low-cost agricultural wastes (corncob, groundnut shell, and sugarcane bagasse) using hydrothermal, microwave sintering, and alkali fusion methods.

## MATERIALS AND METHODS

*Materials:* The raw materials (corncob, groundnut shell, and sugarcane bagasse) utilized in this study were procured from local markets within Kaduna State, North West Nigeria. Before utilization, the raw materials underwent several washes with double distilled water to eliminate any foreign impurities. Subsequently, they were dried under sunlight for 8 hours and then further dried for 4 hours in a hot air oven at 353±5 K. The washed and dried raw materials were ground and sieved using a 200 µm mesh size sieve to remove larger particles. All reagents employed in these experiments were of analytical grade.

*Hydrothermal Method:* In the hydrothermal process, described by Shah *et al*. (2016), agricultural wastes such as Groundnut Shell (GS), Corncob (CC), and Sugarcane bagasse (SB), sieved to a size of 200 µm mesh, were suspended in a mixture of 0.5 M NaOH and 1.5 M NaCl solution (with a ratio of 1:10 solid to liquid) in a round bottom flask. The suspension underwent hydrothermal treatment through refluxing at 100°C for 72 hours with intermittent stirring. After cooling to room temperature, the suspension was filtered, and the residual material washed repeatedly with distilled water to remove excess sodium hydroxide and sodium chloride. Subsequently, the material was dried at 100°C in an oven for 6 hours. The resulting alkaline and electrolyte hydrothermal treatment phases were transformed into hydrothermal zeolitic materials (HZCC, HZGS, and HZSB) and stored in clean, airtight plastic containers.

*Microwave Sintering Method:* For microwave sintering method, following the method outlined by Oluyinka *et al.* (2020), sieved Groundnut Shell (GS), Corn cob (CC), and Sugarcane Shaft (SS), with a mesh size of 200 µm, were separately suspended in a mixture of 0.5 M NaOH and 1.5 M NaCl solution (with a ratio of 1:10 solid to liquid) in a microwave refluxing system containing a flat bottom flask. The mixture was irradiated with a frequency of 2450 MHz for 15 minutes. After treatment, the suspension was filtered, washed with distilled water to remove excess sodium hydroxide and sodium chloride, and dried at 100°C in an oven. The resulting synthesized zeolitic materials were termed as electrolyte microwave zeolitic materials (MZCC, MZGS, and MZSB) and stored in clean, airtight plastic containers.

*Alkali Fusion Method:* The alkali fusion method, as described by Oluyinka *et al.* (2020), was employed for synthesizing zeolitic materials using Groundnut Shell (GS), Corncob (CC), and Sugarcane bagasse (SB). Mixture of each agricultural waste, sodium aluminate, magnesium chloride, and sodium hydroxide were thoroughly ground and sieved through a 200 μm mesh. The ratio of agricultural wastes to sodium aluminate to magnesium chloride was 1:0.5:0.5, while the ratio of their combined mass to sodium hydroxide was 1:1.2. The resultant mixture was heated in a muffle furnace at a stable temperature of 320°C for 90 minutes with a heating rate of 1.85°C per minute. After cooling, the fused materials were ground, added to distilled water (with a solid to liquid ratio of 1:10 w/w), stirred in a glass beaker for 6-12 hours, and subjected to microwave sintering at 100 W for 1 hour to cure and crystallize the zeolitic materials. The final product was filtered, washed until the pH of the final wash was 10, dried at 100°C, cooled, and stored in clean, airtight plastic containers. These materials were referred to as fused zeolitic materials (FZGS, FZCC, and FZSB). Figure 1 shows the presentations of the synthesized zeolites.


**Figure 1:** The synthesized zeolites using three agricultural waste materials and three methods of synthesis; from 1-9: MZSB, MZGS, MZCC, HZSB, HZCC, HZGS, FZSB, FZCC and FZGS

*Determination of Physicochemical Parameters:* Porosity was determined using the stochastic method described by Matko *et al.* (2004), while surface area was assayed using the method outlined by Sears (1956). The point of zero charge (pHpzc) of the synthesized zeolites was deduced using the mass titration method proposed by Noh and Schwarz (1989). Electrical conductivity was measured using an electrical conductivity meter. Moisture content, ash content and bulk density were determined using standard methods (Vogel, 1989).

## RESULTS AND DISCUSSION

Table 1 presents data on the physicochemical properties of zeolitic materials obtained from Corncob, Groundnut shell, and Sugarcane bagasse utilizing hydrothermal, microwave sintering, and alkali fusion methods, respectively. The analysis highlights distinct characteristics of each material synthesized through different techniques. Specifically, HZCC exhibited notably higher ash content, bulk density, and electrical conductivity compared to counterparts produced via the hydrothermal method, indicative of its unique composition and structure. Conversely, HZGS showcased elevated moisture content, while HZSB demonstrated superior specific density, underscoring variations in properties based on the raw material and synthesis method employed.

Similarly, MZCC demonstrated heightened ash content and electrical conductivity, whereas MZGS displayed increased moisture content, bulk density, and specific density. These disparities reflect the influence of synthesis conditions on the physicochemical attributes of the resulting zeolitic materials.

Moreover, FZCC exhibited enhanced moisture content, electrical conductivity, and specific density, suggesting distinct characteristics imparted by the alkali fusion method. Conversely, FZGS displayed elevated ash content, while FZSB demonstrated superior bulk density, further illustrating the diverse properties achievable through different synthesis approaches.

Comparative analysis with previous research findings (Shah and Mistry 2013; Oluyinka *et al.,* 2020) indicates consistency in proximate analysis and physicochemical properties. These findings underscore the potential utility of the synthesized zeolites from agricultural wastes as effective adsorbents as reported by previous findings (Peng *et al.,* 2017; Abdelrahman *et al.,* 2021; Cheng *et al.,* 2021; Flores *et al.,* 2021), paving the way for various environmental and industrial applications (Ma *et al.,* 2022).

**Table 1:** Physicochemical Parameters of Zeolitic Materials Produced from Corncob, Groundnut Shell and Sugarcane Bagasse via Hydrothermal, Microwave Sintering and Alkali Fusion Methods

| | **Hydrothermal Method** | | | **Microwave Sintering Method** | | | **Alkali Fusion Method** | | |
|---|---|---|---|---|---|---|---|---|---|
| **Physicochemical Parameters** | **HZCC** | **HZGS** | **HZSB** | **MZCC** | **MZGS** | **MZSB** | **FZCC** | **FZGS** | **FZSB** |
| Moisture content (%) | 10.50 | 11.30 | 9.65 | 9.98 | 10.25 | 9.18 | 12.50 | 11.0 | 11.03 |
| Ash content (%) | 68.20 | 68.00 | 67.70 | 71.00 | 70.30 | 68.70 | 63.45 | 65.50 | 60.00 |
| Bulk density ($g/cm^3$) | 1.89 | 1.81 | 1.87 | 2.13 | 2.28 | 1.69 | 1.90 | 1.92 | 1.97 |
| Electrical conductivity (mS/cm) | 1.25 | 1.22 | 0.77 | 2.16 | 1.97 | 1.24 | 2.93 | 2.42 | 1.66 |

Table 2 presents comprehensive data on Porosity, pHpzc, and Surface area assay for the produced zeolites obtained through hydrothermal, microwave sintering, and alkali fusion methods, respectively. The point of zero charge (pHpzc) refers to the pH of the solution at which the charge of the positive surface sites equals that of the negative ones, effectively rendering the adsorbent surface charge neutral (Nuraisyah *et al.,* 2020). Understanding pHpzc is crucial for deciphering the potential mechanisms underlying adsorbent-adsorbate interactions during sorption processes. Knowledge of the pHpzc of produced zeolites offers insights into their net surface charge at a specific pH, thereby facilitating predictions regarding their impact on the sorption of target adsorbates. This phenomenon is best understood through the correlation between the surface charges of zeolites and the ionization of most organic pollutants, which is fundamental to the adsorption process. Hence, the pHpzc range obtained in this study aligns with the findings of Oluyinka *et al.* (2020), suggesting that all synthesized zeolites can effectively remove most organic pollutants from wastewater. Porosity signifies the measure of empty spaces or voids within a material. Materials with higher porosity possess more void spaces available for adsorption to occur, thereby playing a pivotal role in determining the effectiveness of materials for adsorption. Similarly, the surface area of a material represents the total area of all its surfaces. Higher porosity leads to increased surface area, consequently enhancing the adsorptive capacity of a material (Nuraisyah *et al.,* 2020). The findings of this study indicate that HZGS and MZSB exhibited higher porosity and surface area via hydrothermal treatment and microwave sintering method respectively, while FZSB demonstrated higher porosity among zeolites produced via the alkali fusion method. All synthesized zeolites in this study exhibited elevated porosity and surface area, alongside consistent pHpzc values. These results align with previous findings (Jiang *et al.,* 2020; Hosseini *et al.,* 2019; Kuleyin, 2007; Ghiaci *et al.,* 2004; Ibrahim *et al.,* 2010; Bai *et al.,* 2019), indicating the suitability of the synthesized zeolites for utilization as adsorbents for pollutant removal in wastewater treatment processes.

**Table 2:** Porosity, pHpzc and Surface Area Assay for Produced Zeolites by Hydrothermal, Microwave sintering and Alkali fusion Heating Methods

| **Agricultural Wastes** | **Surface Area ($m^2/g$)** | **Porosity (%)** | **pHpzc** |
|---|---|---|---|
| | **Hydrothermal Method** | | |
| HZCC | 816.6 | 56.0 | 7.9 |
| HZGS | 1056.6 | 59.0 | 8.1 |
| HZSB | 941.4 | 53.0 | 7.9 |
| | **Microwave Sintering Method** | | |
| MZCC | 1031.0 | 33.0 | 8.3 |
| MZGS | 999.0 | 38.0 | 8.6 |
| MZSB | 1063.0 | 61.8 | 7.9 |
| | **Alkali Fusion Method** | | |
| FZCC | 1335.0 | 74.9 | 9.1 |
| FZGS | 919.0 | 71.1 | 9.8 |
| FZSB | 944.6 | 80.9 | 8.8 |

*Conclusion:* The synthesis of zeolites from low-cost agricultural byproducts, corncob, groundnut shell, and sugarcane bagasse, has been investigated. Results revealed that zeolites obtained from sugarcane bagasse displayed enhanced porosity in two of the three analytical methods employed. Findings from this work underscore the potential of the synthesized zeolites in effectively mitigating or entirely eliminating various pollutants, such as phenols, dyes, pesticides, heavy metals, and inorganic anions, from wastewater.

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*Abbreviations*
FZCC = Fused Zeolitic Materials produced from Corncob
FZGS= Fused Zeolitic Materials produced from Groundnut Shell
FZSB= Fused Zeolitic Materials produced from Sugarcane Bagasse
HZCC = Hydrothermal Zeolitic Material produced from Corncob
HZGS = Hydrothermal Zeolitic Material produced from Groundnut Shell
HZSB = Hydrothermal Zeolitic Material produced from Sugarcane Bagasse
MZCC = Microwave Zeolitic Material produced from Corncob
MZGS = Microwave Zeolitic Material produced from Groundnut Shell
MZSB = Microwave Zeolitic Material produced from Sugarcane Bagasse

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