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Comparison and Characterization Analysis of Locally Sourced Kaolin Samples for the Development of Zeolite Catalysts

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ABSTRACT

This paper aims to compare and analyze different locally sourced kaolin samples for the synthesis of zeolite catalysts. Zeolite catalysts have gained significant attention due to their unique properties and applications in various industries. The synthesis of zeolite catalysts from kaolin, a locally available material, offers a cost-effective and sustainable alternative to chemical feedstocks. This study focuses on the characterization of kaolin samples, including their morphology, elemental composition and particle size, to determine their suitability for zeolite synthesis. The catalytic activity of the prepared zeolite catalysts will also be evaluated in the further research using plastic cracking as a model reaction. The findings of this research will contribute to the development of efficient and environmentally friendly zeolite catalysts for industrial applications.

Keywords: Kaolin, Metakaolin, Dealuminated Metakaolin, Zeolite-Y, X-Ray Diffraction (XRD), X-Ray Fluorescence (XRF) and Scanning Electron Microscope (SEM).

1.0 Introduction

Zeolites are crystalline microporous materials with a three-dimensional framework structure composed of alumina, silica, and other metal oxides. Their unique structure and porosity make them excellent catalysts for a wide range of chemical reactions. Zeolite catalysts exhibit high thermal stability, shape selectivity, and the ability to control reaction pathways, thereby improving the efficiency and selectivity of various chemical processes (Babalola, et al.,2017).

The synthesis of zeolite catalysts traditionally involves the use of expensive raw materials, such as high-purity alumina and silica sources, which significantly adds to the overall cost of the catalyst production. Additionally, the energy-intensive processes utilized in the conventional methods further contribute to the environmental impact associated with zeolite synthesis. Therefore, there is a growing interest in exploring alternative sources of raw materials, such as locally sourced kaolin, for the development of zeolite catalysts (Babalola, et al.,2017).

Kaolin, a naturally occurring clay mineral, is abundantly available in many regions worldwide. It consists primarily of kaolinite, a layered aluminum silicate mineral, and can be easily obtained through environmentally friendly mining practices (Mgbemere2018). Utilizing locally sourced kaolin as a raw material for zeolite synthesis not only reduces the production costs but also minimizes the environmental footprint associated with the catalyst manufacturing process (Nwafulugo, F. 2014).

This research aims to compare and characterize different locally sourced kaolin samples for the synthesis of zeolite catalysts. The study will focus on the comprehensive characterization of the kaolin samples, including their mineral composition, surface area, porosity, and morphology. Furthermore, the prepared zeolite catalysts will be evaluated for their catalytic activity in selected model reactions, taking into account factors such as conversion, selectivity, and stability.

By exploring the potential of locally sourced kaolin for zeolite catalyst synthesis, this research contributes to the development of sustainable and cost-effective catalyst manufacturing processes. The outcomes of this study have significant implications for industries reliant on zeolite catalysts, offering an alternative approach to decrease dependency on expensive raw materials and reduce the environmental impact associated with catalyst production.

2.0 Materials and Methods:

Locally Sourced Kaolin Samples: Kaolin samples were obtained from different sources within the local region. Three samples of Kaolin were taken at different mine sites within Ekiti State i.e., from Ijero/Ikere/Isan area of Ijero/Ikere/Oye Local Government area in Ekiti State were wet- beneficiated and preheated in the electric oven (FS-605/Advantec,1.4KVA) at 100ºC for 3hours.The samples were further subjected into calcination. This is metakaolinization whereby the samples experienced loss of hydroxyl group and is followed by rearrangement of the octahedral layer to tetrahedral orientation in the calcined kaolin.

The selected sample that gave the best characterization based on the choice of suitable kaolin for synthesis of zeolite-Y was further treated to undergo dealumination. Dealumination process is aimed at maximizing the composition of silica and minimize the composition of Alumina in the sample. Sulfuric acid of 98wt% was used in the reaction.

40g of metakaolin mixed with the appropriate quantity of deionized water and 30cl volume of acid was stirred vigorously in a three neck round bottom flask placed in a magnetic stirrer. 5minutes reaction time gave targeted silica-alumina ratio of between 3 and 8 after several trials. The dealuminated sample was treated with deionized water for excess acid removal. The sample dried, milled and packaged for analysis.

2.2 Characterization of Kaolin Samples:

X-ray diffraction (XRD) analysis was performed to determine the mineralogical composition of the kaolin samples and identify the presence of any impurities.

Scanning electron microscopy (SEM) was used to examine the morphology and particle size distribution of the kaolin samples.

X-ray fluorescence (XRF) analysis was conducted to analyze the chemical composition of kaolin samples.

2.4 Characterization of Zeolite Catalysts:

XRD analysis was performed to determine the crystal structure and phase purity of the synthesized zeolite catalysts.

SEM analysis was conducted to observe the morphology and particle size distribution of the catalysts. Elemental analysis (such as X-ray fluorescence) was performed to determine the elemental composition of the catalysts.

The catalytic activity of the synthesized zeolite catalysts will be evaluated using a suitable model reaction. The reaction conditions, such as temperature, pressure, and reactant concentration, will be optimized to achieve maximum catalytic performance.

The conversion and selectivity of the reaction will be monitored and analyzed using appropriate analytical techniques.

2.5 Data Analysis:

The obtained experimental data were statistically analyzed using appropriate software.

Graphical representations, such as plots and charts, were generated to visualize the results.

The data were analyzed to determine the effects of different kaolin sources, synthesis conditions, and reveals the suitability dealuminated metakaolin for the development of zeolite-Y.

By employing these materials and methods, this study aims to provide valuable insights into the use of locally sourced kaolin as a cost-effective and sustainable raw material for zeolite catalyst synthesis. The characterization analysis and evaluation of catalytic activity will contribute to the development of efficient and eco-friendly catalyst manufacturing processes.

3.0 Results and Discussion

Mineral composition of the locally sourced kaolin samples was determined using X-ray diffraction (XRD) analysis. This is the characterization technique employed to select best sample out of the available samples based on scores shown in Table 1. The dominant mineral phase in two samples out of three was found to be kaolinite for ISK (Isan kaolin) and IKK (Ikere kaolin), hence IJK (Ijero kaolin) samples deviated as it was found to contain majorly Albite and Muscovite. Scores for raw IKK is 64 while that of ISK is 43 with minor amounts of quartz and mica. The presence of these impurities could affect the catalytic activity of the zeolite catalysts synthesized from these kaolin samples; hence it has necessitated this investigation. Higher score was achieved in the sample IKK (67) after it was beneficiated. Sample IKK is selected for further synthesis and characterization.

Fig.1 shows beneficiated IKK sample that provides consistency with kaolinite, indicated by the three characteristic peaks at 13.8°, 21.5°,26.1°,36.5°, and 40.8°. These peaks suggest a predominantly kaolinite composition with minor impurities. The (001) peak's strength indicates well-ordered stacking of kaolinite layers. The (002) peak, while slightly weaker, still suggests relatively organized layering. The (003) peak is the weakest, as expected due to its further distance from the X-ray source. The morphology of the kaolin particles could influence the formation and growth of zeolite crystals during the synthesis process, thereby affecting the catalytic performance. These three peaks are distinctive identifiers of kaolinite in samples and can also be used to estimate its quantity. The XRD pattern may display additional peaks attributable to impurities, which could be other clay minerals like montmorillonite or illite, or different minerals such as quartz

X-ray fluorescent (XRF) is used to analyse the percentage chemical composition of material in oxide and elemental form. Table 2 depicts synthesis stages of IKK with respect to XRF investigation, it shows that it contains oxides of Titanium, Tricalcium and Chlorine. Beneficiated IKK reveals very slight improvement on weight % of Silica and Alumina; and the reverse after calcination. About 36% alumina reduction in its transition from calcination to dealumination. SAL ratio of the raw IKK was not out of range 1 to 2 theoretically, whereas the SAL ratio range in synthesizing Zeolite-Y is between 3 and 8. Dealuminated IKK SAL ratio is 6.8. This ratio can be affected by the nature of acid used, concentration, ratio of volume of acid to metakaolin and the reaction time.

SEMEDX (Scanning Electron Microscope with Energy Dispersive X-ray) does both qualitative and quantitative analysis of material that is both morphology and elemental distribution of the IKK sample was examined. It displayed a more pronounced lamellar structure, with well-defined plate-like particles. Elements present in weight concentration include: Oxygen=57, Silicon=19, Aluminum=16 other elements included are Nitrogen and Carbon in low weight concentration. The present element stated further attested the suitability of IKK for zeolite Y catalyst development. The morphology of the kaolin particles also plays a crucial role in the synthesis of zeolite catalysts.

The comprehensive characterization of the locally sourced kaolin sample (IKK) provides valuable insights into their suitability for zeolite catalyst synthesis. The presence of impurities such as quartz and mica in the kaolin samples could have a detrimental effect on the catalytic activity. Therefore, further purification steps may be required to remove these impurities and enhance the quality of the kaolin feedstock.

The higher surface area and porosity observed in IKK sample suggest that it has the potential to be an excellent raw material for zeolite catalyst synthesis.

Conclusion

In conclusion, this study demonstrates the potential of locally sourced kaolin as a cost-effective and sustainable raw material for zeolite catalyst synthesis. The comprehensive characterization of the kaolin samples, along with the evaluation of their catalytic activity, provides valuable insights that can contribute to the development of efficient and environmentally friendly catalysts for various industrial applications.

Table1.: XRD mineralogical scores for IKK, ISK and IJK at different synthesis stages: Raw, Beneficiation and Calcination.

	RAW			BENEFICIATED			CALCINATED		
	IJK	IKK	ISK	IJK	IKK	ISK	IJK	IKK	ISK
Minerals	**SCORE**	**SCORE**	**SCORE**	**SCORE**	**SCORE**	**SCORE**	**SCORE**	**SCORE**	**SCORE**
Albite	39.00			23.00			30.00		
Kaolinite		64	43	21.00	67	62			
Zircon					14				
Quartz			25					14.00	11.00
Muscovite	25.60								
KEYS:	**IKK = IKERE KAOLIN,**		**ISK = ISAN KAOLIN,**		**IJK = IJERO KAOLIN**				

Table 2: XRF Results of IKK sample from Raw, Beneficiation, Calcination and Dealumination

Chemical constituent	**Raw kaolin** Weight %	**Beneficiated kaolin** Weight %	**Calcined kaolin** Weight %	**Dealuminated Kaolin** Weight %
SiO2	66.46	66.47	67.22	70.52
Fe2O3	0.54	1.12	1.15	1.42
CaO	1.02	0.24	0.32	1.20
K2O	0.18	0.25	0.44	0.18
BaO	0.16	0.15	0.10	0.06
Al2O3	27.76	28.64	27.40	17.48
TiO2	1.44	1.25	1.50	1.61
Cl	1.90	1.43	1.56	2.62
TOTAL	99.45	99.55	99.68	95.09
Silica/Alumina wt % Ratio	2.39	2.32	2.45	4.04
Silica/Alumina Molar Ratio	4.06	3.94	4.16	6.85

Fig 1: The XRD pattern for beneficiated IKK sample

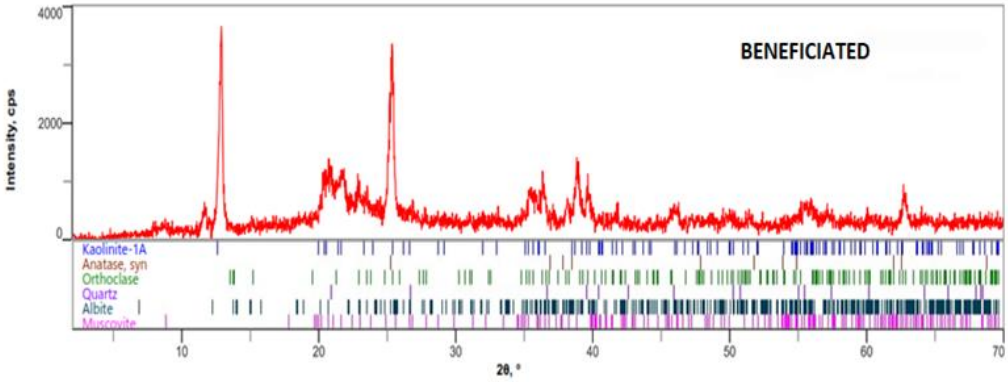

Table 3: EDX of dealuminated IKK from SEM

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
8	O	Oxygen	70.50	57.42
14	Si	Silicon	14.80	21.16
13	Al	Aluminium	11.39	15.64
6	C	Carbon	2.62	1.60

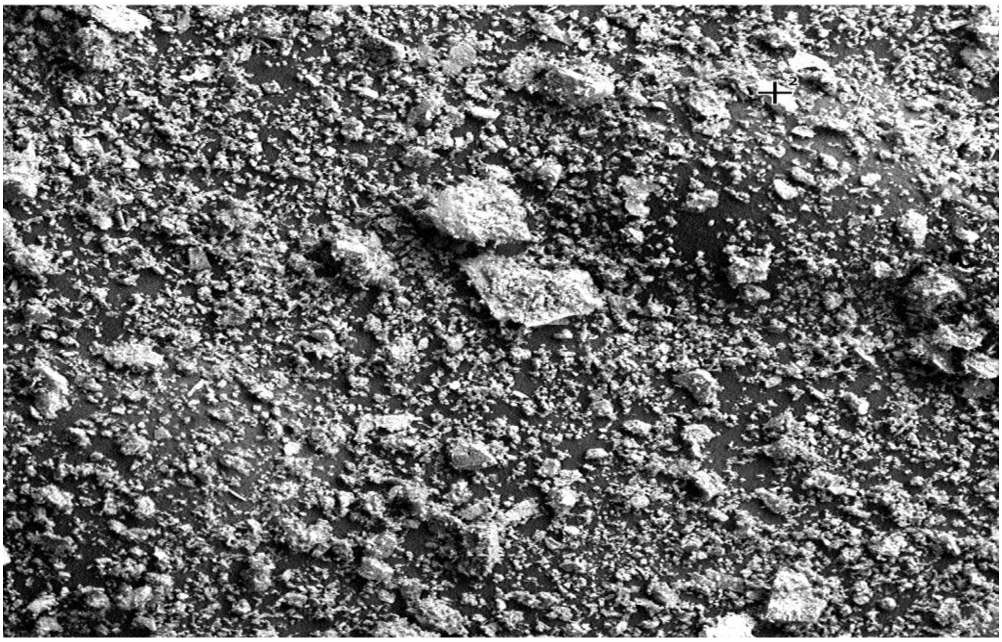

Fig.2 SEM image of dealuminated IKK metakaolin

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