Comparative Analysis of Zeolite Y From Nigerian Clay and Standard Grade

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***Abstract*— Zeolite Y catalyst with silica/alumina molar ratio of 4.70 was synthesized from Elefun (Nigeria) clay under hydrothermal treatment of calcined kaolin with aqueous NaOH at atmospheric pressure. This paper described the preparation of zeolite Y catalyst from metakaolin of quality Elefun kaolin by ageing at 34oC for 7days, and then crystallized at 100oC for 24 hours. The synthesized zeolite NaY was modified by exchanging with NH4Cl to obtain its hydrogen form with silica/alumina ratio of 3.18. Both developed and standard zeolite Y catalyst were then characterized by a variety of physicochemical methods, including XRD, XRF spectroscope. The morphologies were examined using SEM. Similar results were obtained, thus confirming the synthesis of zeolite Y.**

Keywords: Crystallization, hydrothermal, Zeolite Y, XRF, XRD, FT-IR, SEM

I. INTRODUCTION

The rising world energy demand which calls for the processing of heavy petroleum feedstock has increased the importance of developing new catalyst systems. Heavy feeds have residue contents of about 40% and require further processing in order to find a market, through catalytic process using zeolite catalyst.

Nigeria, with a huge amount of oil reserves estimated recently to be about 37.2 billion barrels and the 13th word producer of petroleum, with four refineries across the nation and a total processing capacity of 450,000 barrels per day consumes approximately 1600 tonnes of zeolite catalyst per day. The catalysts are imported annually over 500,000 tonnes, at a cost of about 5 billion naira [4]. Nigeria has a good potential to develop zeolite for its local use.

Zeolite are crystalline, micro porous, allumino silicate materials with a three dimensional fully crosslinked open framework structures that form uniformly sized pores of molecular dimensions. The materials have huge industrial, scientific and academic interest in the areas of ion exchange, petroleum refining and separation. The multifunctional activities of zeolite material in many industrial applications is due to their inherent properties such as uniform pore size catalytic activity, mobile cation and hydrophilicity/hydrophobicity [1].

II. PURPOSE

The goal of this research is to develop zeolite Y catalyst from Nigeria clay, characterize and compare with it with standard zeolite Y catalyst..

III. METHODOLOGY

Zeolite Y catalyst was prepared from Elefun kaolinite clay as shown in the flow chart (Figure 1).The process involves the bounding of sodium hydroxide with dealuminated kaolinite clay in a ratio of 2.5:1 by weight and molar composition of $6SiO_2$: Al_2O_3: 9Na2O: $24H_2O$ [4]. The metakaolin had been prepared from the beneficiation of the raw kaolin, followed by calcination at 850°C for 6 hours and partial deacidification using H_2SO_4 of 98% purity. The obtained gel was aged at 34°C for 7 days and hydrothermally crystallizes at 100°C for 24 hours. The NaY so obtained was modified to its hydrogen form HY by iron exchange with ammonium chloride solution as specified in Figure 1 and then calcined at 450°C for 4 hours.

Characterization and Analysis

The synthesized catalysts were characterized as follows: Chemical composition was recorded using x-ray fluorescence (XRF) in a Bruker-AXS S4 explorer apparatus fused with $Li_2B_4O_7$ at 1150°C. The x-ray diffraction (XRD) pattern of powdered catalyst was obtained on an Empyrean DT 674 diffractometer by panalytical. The radiation employed was CuKa with a wavelength of 1.5418Å. All samples were scanned from 5° to 40° 2Θ. The morphology was obtained using a pro x phenom desktop scanning electron microscope (SEM) at accelerating voltage of 15kV and working distance of 10.0mm. And the performance test was investigated using a bench scale fixed bed reactor at a temperature range of 420°C to 520°C and contact time of 5 to 60 seconds. The same procedure was repeated using standard zeolite Y catalyst.

IV. RESULTS AND DISCUSSION

Kaolin clay available in Elefun Local Government area of Ogun state was used as Raw material for Zeolite Y catalyst preparation. Table 2 shows the Chemical analysis of the various stages of the synthesized material. The compositional analysis conducted on the raw, beneficiated, calcined and final product from Elefun kaolin using X-Ray Fluorescence (XRF) is as shown in Table 2. The results show that Elefun kaolin is rich in oxides of potassium, iron, titanium, and magnesium. It also indicates the effect of beneficiation on the treated raw kaolin as value of SiO2 reduced from 53.80% to 51.60% due to remover of free silica (quartz) from raw kaolin. Table 2 also indicates that Elefun kaolin is ferric in nature due to its high content of iron oxide as compared with that of potassium. Similarly, the white colour of the raw and beneficiated Elefun Kaolin can be attributed to their significant contents of TiO2.

Pure raw kaolinite clay is expected to have silica/alumina ratio of between1 to 2 [2,6]. Table 2 shows that the SiO2/AlO2 ratio of 1.45 and 1.47 for the raw and beneficiated kaolin respectively are within theoretical value.

X- Ray diffraction was used to study the crystalline and framework structure of Zeolites [3]. Figure 2 (a) represents the X-ray diffraction pattern data prepared catalyst with 4.70 Si/Al ratios. This pattern was compared with X-ray data of standard Zeolite Y (Figure 2(b). Table 1 showed that the lattice spacing of prepared catalyst sample gave similar lattice spacing with standard zeolite Y. This means that the prepared catalyst has approximately the same crystal structure as the imported standard type Zeolite Y.

Table 1 showed that the synthesized zeolite Y has D spacing of 2.87 at angle 2 theta degrees of 31.10 which is approximately the same with the standard industrial zeolite Y with D spacing of 2.88 at angle 2 theta of 30.94 degree. The hexagonal ordering of SEM images of the synthesized zeolite Y and standard zeolite Y catalyst as shown in Figure 3(a) and 3(b) respectively signifies a typical zeolite Y. This illustrate that synthesized zeolite Y crystal has the same shape and size as the standard zeolite Y. However, the zeolite coverage was different due to the presence of quartz (free silica) in the synthesized zeolite Y catalyst. The SEM images of the synthesized zeolites Y demonstrated that by increasing the Na2O and Al2O3 ratio, the Si/Al ratio decreases [4]. Whilst the change in the water ratio had no effect on Si/Al ratio [7].The chemical composition of the catalysts as presented in Table 2 showed that the impurities were minimal.

V. CONCLUSION

The comparison of the X-ray diffraction, Lattice spacing and SEM results for prepared zeolite Y catalyst with that of Standard zeolite Y shows that the developed catalyst is approximately the same with standard

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Table 1. Comparison of lattice spacing, between Synthesized Zeolite Y and standard zeolite Y Catalyst

Zeolite Y catalyst prepared from Elefun kaolin		Standard Zeolite Y catalyst	
Angle (2Theta) deg.	d, spacing (Å)	Angle (2Theta)deg.	d, spacing (Å)
12.48	7.09	12.47	7.09
20.18	4.40	19.80	4.48
21.11	4.21	21.76	4.08
26.77	3.33	26.74	3.33
28.11	3.12	28.21	3.16
29.36	3.04	29.55	3.02
31.10	2.87	30.94	2.88
32.13	2.78	32.26	2.77
33.40	2.65	33.49	2.67
35.69	2.51	35.95	2.49
38.52	2.39	38.16	2.35
40.97	2.20	39.27	2.29

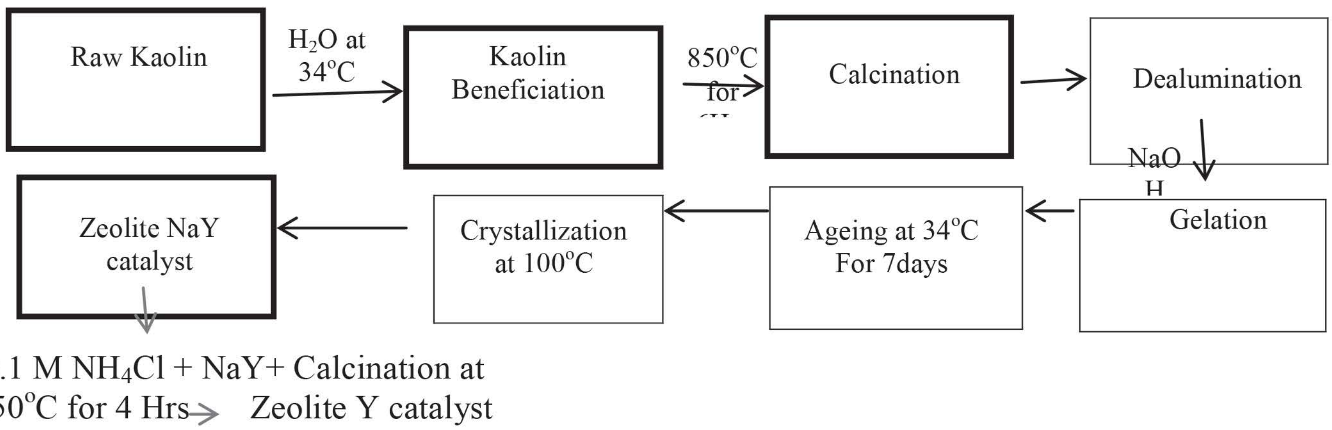


Fig. 1. Experimental protocol of Zeolite Y Synthesis

Table 2. XRF results of the Raw, Beneficiated, Calcined and Synthesized zeolite Y from Elefun kaolin

Chemical constituent	Raw kaolin	Beneficiated kaolin	Calcined kaolin	Synthesized Zeolite *Na*Y	Zeolite Y
	Weight %	Weight %	Weight %	Weight%	Weight %
SiO_2	53.80	51.60	51.5	51.100	58.5
Al_2O_3	37.01	35.00	36.900	18.500	31.30
K_2O	0.63	0.54	0.615	0.522	0.98
TiO_2	4.76	5.10	4.520	5.99	5.04
V_2O_5	0.21	0.22	0.510	0.210	0.02
Fe_2O_3	2.57	2.79	1.480	1.920	1.42
SO_3	-	-	-	5.100	0.55
CaO	0.30	0.35	0.035	0.43	0.17
MnO	-	-	-	0.03	0.02
CuO	-	-	-	-	0.01
L.O.I *	0.73	4.40	4.41	16.2	1.99
Total	99.27	95.60	95.59	83.80	98.01
Silica/ Alumina	1.45	1.47	2.373	4.700	3.63

***Loss on Ignition**

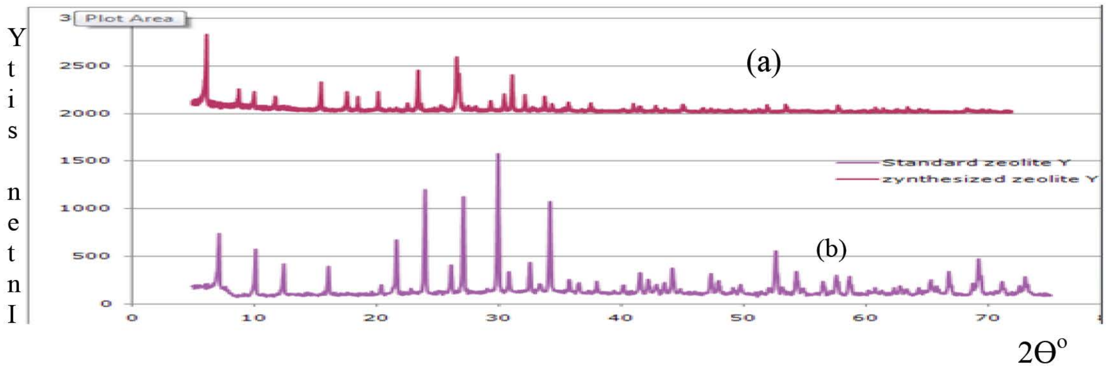
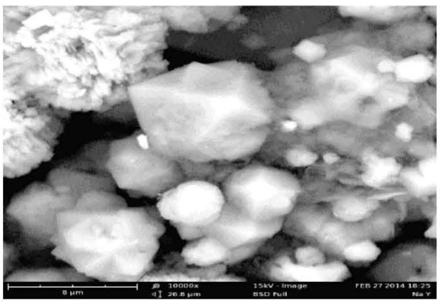
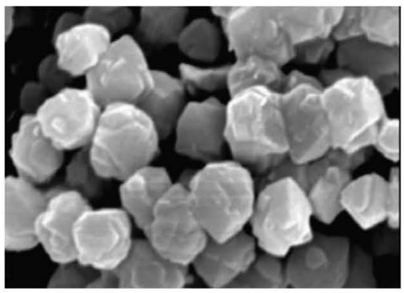


Fig. 2. Powder XRD pattern of (a) synthesized zeolite Y and (b) standard zeolite Y catalyst



(a)



(b)

Fig. 3. SEM Images of (a) Synthesized zeolite Y (b) Standard zeolite Y