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HYDROTHERMAL SYNTHESIS OF ZEOLITE Y FROM OKPELLA KAOLIN CLAY

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ABSTRACT

This study entails the hydrothermal synthesis of Zeolite Y catalyst from locally sourced kaolin clay obtained from Okpella in Edo state, Nigeria. The experimental procedure was carried out following established standard procedures for zeolite synthesis. Calcination of the beneficiated kaolin was done at 750^oC using an electric furnace followed by dealumination of the metakaolin using H_2SO_4. The synthesis gel was formed by reacting Dealuminated metakaolin with sodium hydroxide pellets according to the ratio of 2.5:1 by weight and molar composition of $6SiO_2 : Al_2O_3 : 9Na_2O : 24H_2O$. The Gel obtained was aged for 7 days at room temperature and then hydrothermally crystallized at 100^0C for 24 hours. Characterization of the synthesized zeolite was carried out using X-ray diffraction and field emission scanning electron microscope. The results obtained showed that Zeolite Y with SiO_2/Al_2O_3 molar ratio of 3.46 was successfully synthesized from locally sourced kaolin clay.

Keywords: Kaolin clay, Zeolite Y, Characterisation, XRD Analysis.

INTRODUCTION

Zeolites are materials made up of microporous aluminosilicate crystals that are used as ion exchangers in the detergent industry, in radioactive waste storage, in the treatment of liquid waste, as separators in purification, environmental treatment, in the catalytic cracking of petroleum and in refining petrochemical, coal and fine chemical industries [1]. They lose water when heated to form a porous structure with large surface areas. The properties that enables them to perform all these functions in several industrial applications are their uniform pore size distribution and shape, the mobility of their cations to act as catalysts and their hydrophilic and/or hydrophobic nature to some solutes [2]. Primarily, zeolites are built from [SiO $[AlO_4]^{5-}$ tetrahedral which are infinitely extended in a three dimensional network that is linked together by a shared oxygen atom. There are more than 200 types of zeolites and they can be identified by their Silicon to Aluminium (Si/Al) ratio present in the atomic structure of the zeolite[3]. The ratio can vary from 1 in type A zeolites to infinity in silicalite, which is an aluminium free crystalline silica modified zeolite[4]. When the Si/Al ratio of zeolites increase, both their thermal stability and acid resistance also increases. This current research seeks to synthesize zeolite Y from okpella kaolin clay using the novel method of Dealumination.

MATERIALS AND METHODS

Materials

The kaolin clay used in this research work was sourced from Okpella village, Edo State Nigeria, from the "Freedom Group Mining Company". Sodium hydroxide pellets and concentrated sulfuric acid each of 98% purity were obtained from Sigma-Aldrich, Lobal Chemie, and used in this work for zeolite Y synthesis.

Experimental Procedure

Beneficiation and Size Reduction of Raw Okpella Kaolin

Beneficiation was done to remove impurities like organic matter and other particles. To achieve this the clay was washed with distilled water to remove impurities and later sun dried. No other chemical or physical treatment was done prior to this. The dried clay was then crushed to fine powder and sieved via a 75micron sieve and packaged for analysis.

Calcination of Beneficiated Okpella kaolin

The beneficiated kaolin was calcined at 750^oC using a programmable electric furnace, the temperature was increased at a steady rate of 10^oC min^{-1} up to the calcination temperature. The metakaolin so obtained was put in a dessicator for controlled cooling.

Dealumination of Metakaolin

150g of metakaolin was mixed with 10ml of 10M sulphuric acid in a conical flask, followed by vigorous stirring to obtain mixed slurry. The resulting slurry was then left to react in a water bath under manual stirring for 10 hrs at a temperature of 90^oC. At the end of the reaction period, the samples were washed with deionized water mixed with 10M of $BaCl_2$. This was done to remove every trace of acid that was still left in the sample. The samples were dried at 70^0C in an electric oven. After which they were stored.

Synthesis of the Gel

The Dealuminated metakaolin was reacted with sodium hydroxide pellets according to the ratio of 2.5:1 by weight and molar composition of $6SiO_2 : Al_2O_3 : 9Na_2O : 24H_2O$ [5]. The Gel obtained was aged for 7 days at room temperature and then hydrothermally crystallized at 100^0C for 24 hours.

Transforming the Synthesis Gel (Zeolite NaY) to its Hydrogen Form

The synthesized gel (i.e zeolite NaY) was further mixed with a 0.1M NH_4Cl using a ratio of 100ml of solution to 10g of zeolite NaY at room temperature and stirred vigorously using a magnetic stirrer for ten minutes. This was done twice to obtain optimum ammonium exchange. The products of this operation was rinsed with deionized water until the solid was free of chloride, after with it was dried at room temperature for 24 hours. Lastly, it was placed in the oven set at 350^0C for 4 hours, in order to strip off ammonia and leave the zeolite Y in its hydrogen form.

Characterization of the Synthesized Zeolite

To determine the crystal structure and relative crystallinity of the synthesized zeolite, the X-ray diffraction (XRD) patterns were recorded on an X-ray diffraction machine (Cubic 3 Cement PAN analytical) using Cu-ka radiation with a wavelength of 1.540598. The morphology of the synthesized zeolite was analyzed using field emission scanning electron microscopy (Model FESEM: JSM-7100FA JEOL USA, Inc.). The IR spectra were recorded within the range of 4000 to 400 cm^{-1}.

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RESULTS AND DISCUSSION

XRD Analysis

Table 1.0 shows that SiO_2/Al_2O_3 ratio of raw Okpella clay is 1.482, and it is within the theoretical value because pure raw kaolinite clay is expected to have silica/Alumina ratio of 1 to 2 [6, 7]. This is essential given the fact that faujasites zeolites comprising of zeolite X and Y have a high silica to alumina ratio. Hence the starting clay material for development of such zeolites have to be commensurate to meet this need. The SiO_2/Al_2O_3 ratio of the synthesized material is 3.51.

Table 1: XRD Analysis of Okpella Kaolin Clay

Raw Okpella Kaolin Clay	
Composition	**Percentage (%)Oxides (Weight %) Content**
SiO_2	51.847
Al_2O_3	34.962
K_2O	10.516
TiO_2	0.000
SnO_2	1.521
Rb_2O	0.788
Fe_2O_3	0.123
P_2O_5	0.0025
ZnO	0.122
NiO	0.1185
Zeolite HY	
SiO_2	54.89
Al_2O_3	15.60
K_2O	11.516
TiO_2	0.000
SnO_2	2.534
Rb_2O	1.234
Fe_2O_3	0.5856
P_2O_5	0.022
ZnO	0.5856
NiO	0.2185
NaO	12.8143

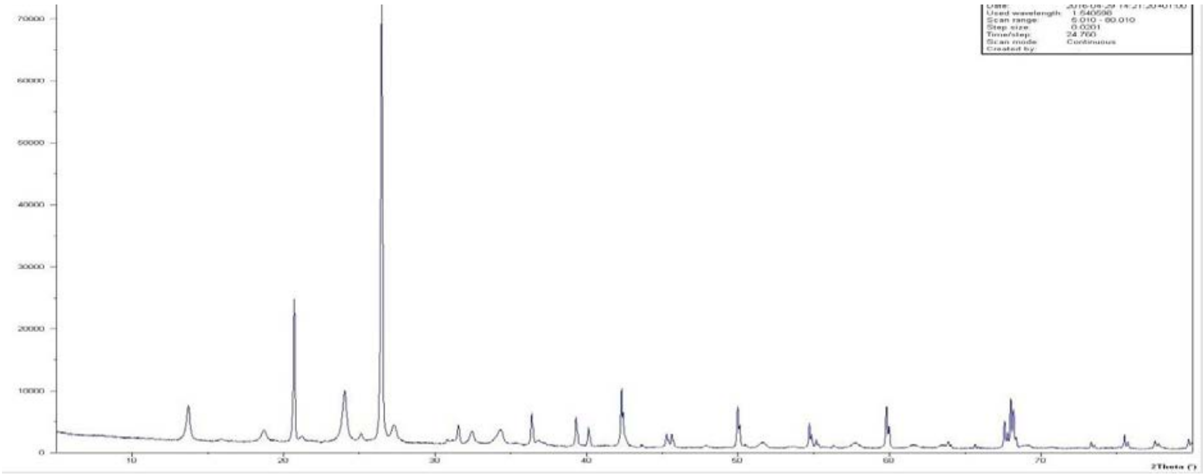


Figure 1: XRD Pattern of the Zeolite NaY

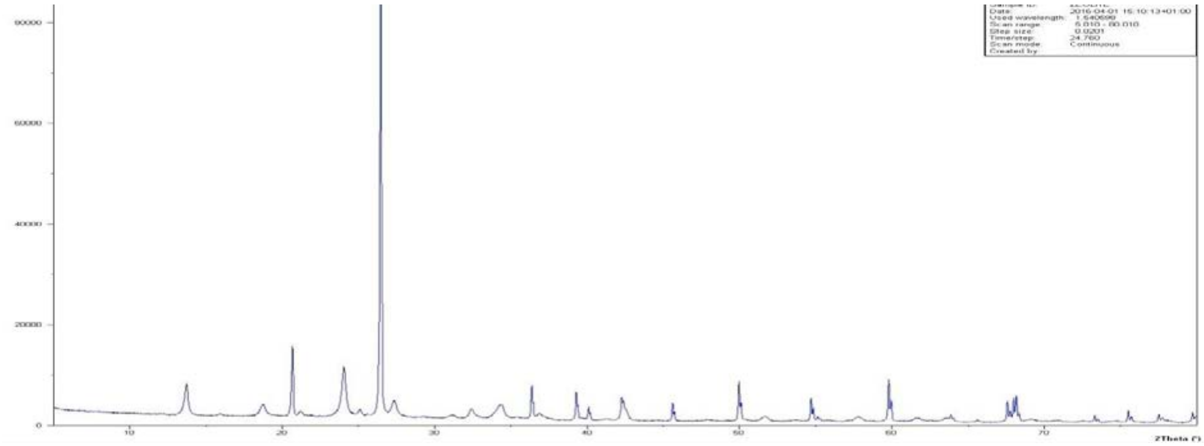


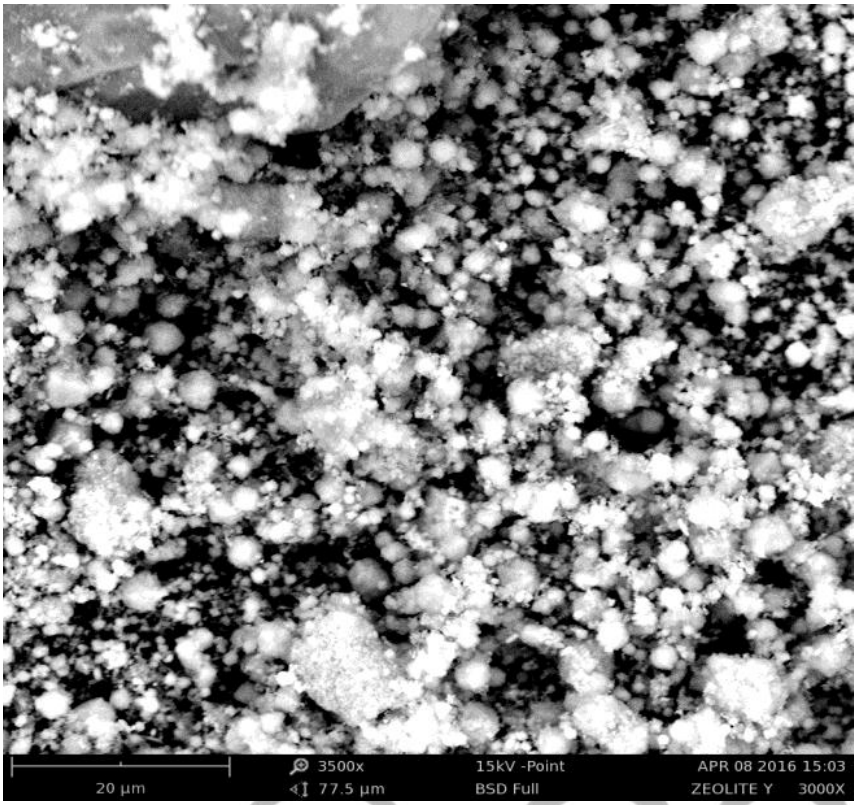
Figure 2: XRD Pattern of the Zeolite HY

The samples analyzed have similar amounts of Aluminum oxide and silicon oxide. The oxygen peak in relation to the metal is constant for the samples. Aside Oxygen, Silicon and Aluminum, there is no prominent peak in representing any other metal. For all the samples the main elements present by percentage mass are Oxygen, Silicon and Aluminum. These results are consistent with typical elemental content of zeolite Y.The Zeolite peaks could be observed in both XRD patterns at Bragg's angles of 14, 19, 21, 24, 26.8, 31.9, 34.9, 39.9, 49.8, 51°. The intensities of the peaks at Bragg's angles of 14, 19, 21, 24, 26.8, 31.9, 34.9, 49.8 and 51° corresponding to 980, 589, 2500, 500, 8000, 500, 500, 980 and 510 counts, respectively and characteristic of zeolite Y [8].

FESEM IMAGES

Below are the FESEM Images at (a) 3500 (b) 5000

(a)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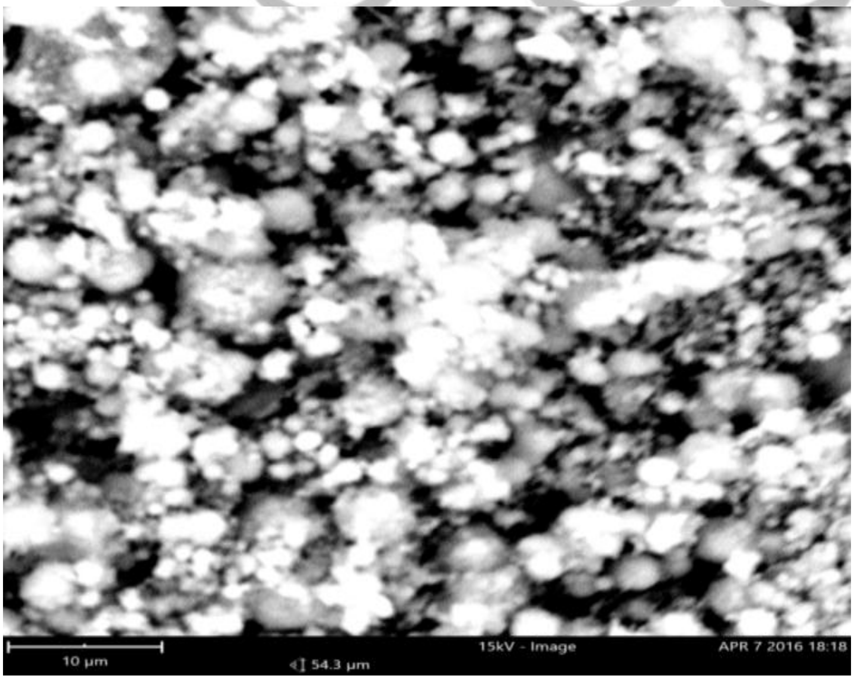
3500x
77.5 µm
15kV -Point
BSD Full
APR 08 2016 15:03
ZEOLITE Y 3000X

(b)


10 µm
54.3 µm
15kV - Image
APR 7 2016 18:18

CONCLUSION

Zeolite Y was successfully synthesized from okpella kaolin clay from Edo state, Nigeria. The Dealuminated metakaolin was treated with sodium hydroxide and aged for 7 days. The Zeolite NaY was then modified to its hydrogen form by ion exchange with NH_4Cl to give a SiO_2/Al_2O_3 molar ratio of 3.51. The ion exchange decreased the peak intensities of zeolite HY. The XRD analysis pattern is similar to nearly crystalline of zeolite Y type, indicating the formation of zeolite HY.

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