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SYNTHESIS OF ZEOLITE Y USING A FUSION TEMPERATURE OF 600°C

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ABSTRACT

Sandstone is an abundant raw material. In the event of synthesizing zeolite Y (Faujasite) from the afore-mentioned raw material, an X-Ray Fluorescence (XRF) elemental analysis was carried out revealing SiO_2 (Silica) of 87.5%, Al_2O_3 (Alumina) of about 6.8% as well as Na_2O of 0.08%.Hydrothermal synthesis of zeolite was adopted and it was realized

Introduction

Zeolites are an assortment of hydrated aluminum-silicates made from alkali or alkaline earth metals (sodium, potassium,magnesium, calcium). Zeolites have channels filled with water and exchangeable cations and a three-dimensional crystalline structure made of tetrahedral silica or alumina anions that are tightly bound at all corners. The isomorphous replacement of Al3+ with Si4+ creates a deficit of positive charge in the structure, making it ideal for ion exchange. Exchangeable cations like Na+, Ca2+, K+, and Mg2+, which are monovalent and divalent, counterbalance this. These cations are situated on particular places in the framework channels and coordinated with the predetermined quantity of water molecules (Peri et al., 2004; Cakicioglu-Ozkan and Ulku, 2005). The primary

that the siliceous part of the material depolymerizes over a reasonable exposure to high temperature and excess Alumina (Al_2O_3). The synthesis of zeolite Y (Faujasite)with limited Al_2O_3 for 6h at 80˚C yielded a siliceous material (quartz) of 1452.4 intensity on the XRD pattern whereas synthesis of same form of zeolite from the same material with excess Al_2O_3 (Alumina) at same temperature of 80˚C for a contact/reaction time of 3h, 6h and 9h yielded intensities of 854.5, 764.7 and 720.9 respectively as well as formation of various zeolite phases in contrast to the resistivity of the siliceous material (quartz) experienced in the synthesis done with limited Al_2O_3 (Alumina) being figures 2,3 and 4 with set reaction time of 3,6 and 9 hours respectively. It was observed that the tendency of formation of the zeolite Y material increases against the decreasing and depolymerizing quartz (siliceous material) with increased reaction time. The plot of Faujisite intensity against reaction time shows a rise in the formation ability of the zeolite Y with increase reaction time due to the presence of excess Al_2O_3 which helps in weakening the bonds of the quartz thereby depolymerizing it to form more of the required material rather than the unwanted. While for the Quartz intensity plotted against reaction time, it implies the effect of Al_2O_3 on the siliceous component of the material over sufficient reaction time.

Keywords: Synthesis, Zeolite Y, Fusion Temperature, XRF, XRD.

effect of this type of structure may be seen in the reversibility of the hydration and cation exchange processes, which protect the original network. As a result, they exhibit unique qualities (such as adsorbtion-desorption capacity, ion exchange capacity, and catalytic characteristics), which give this category of minerals significant and varied usage options. Natural and manufactured zeolites can be employed as ionic or molecular filters because of their unique

tectosilicate-type crystal structure, which has pores that are bigger than the ions (or molecules) that flow through them.

Without a doubt, the significance of zeolite synthesis, which has applications in the following areas, cannot be overstated

The exchange of ions of the same charge between an insoluble solid and a solution in contact with it is known as ion-exchange and is utilized in purification and separation operations.

• Adsorption: This is the process of retaining molecules of a gas, liquid, or solute as a thin coating on surfaces inside or outside the material.

• Catalysis: This is when a substance known as a catalyst participates in a chemical reaction, increasing the rate of the reaction.

MATERIALS AND METHOD

MATERIALS AND EQUIPMENT

MATERIALS

1- Sandstone sourced from the river-side of Kogi-river in Zaria Metropolis
2-Al_2O_3
3-NaOH pellet(s)
4-Distilled water
5-Whatman filter-paper(s)
6-Water
7-Sample bags

EQUIPMENT

1- Buchner-funnel
2- Filtration set-up
3- Weighing analytical balance
4-High temperature furnace
5-Aluminium pot
6-Kerosene stove / Water-bath
7-Ceramic crucible
8-Air-tight jar(s)
9-Laboratory thermometer
10-Measuring cylinder
11- Blender

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12-Laboratory drying oven

Figure 1: A muffled furnace

Figure 2: Filtration vacuum pump

METHODOLOGY

Sandstone Collection: The sandstone material was sourced from the riverside somewhere within Zaria metropolis of Kaduna state, Nigeria.

Beneficiation: The beneficiation was simply carried out using water to get rid of the unwanted particles mixed with the desired sand material. The beneficiation was done in a basin of which the sandstone was poured, followed by addition of water and thorough hand-washing. After which the washed sandstone was spread in the open to dry its accompanied moisture content at room temperature.

Zeolite Y synthesis procedure

Particle size reduction: The beneficiated sandstone was crushed using a milling machine in one of the geotechnical laboratories situated in Geology department of Ahmadu Bello University, Zaria.

XRF Analysis: The X-ray Fluorescent Spectrometry (XRF) analysis was conducted in the multi-user laboratory situated in department of chemistry of Ahmadu Bello University, Zaria in order to ascertain the elemental composition of the raw material (sandstone).

Fusion: The crushed and analysed sandstone was weighed to the required proportion or ratio needed for fusion with Al_2O_3 and NaOH. The

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requirements for each component needed to be fused was calculated using the stoichiometry method of analysis as shown in the appendix A of this work and calcined as well as fused for 12 hours at a fusion temperature of 600˚C.

Ageing: For the pre-ageing**,** 43.5g of sandstone, 83.4g of NaOHand 17.75g of Al_2O_3 were individually weighed, blended and allowed to stand for a period of 144 hours (6 days) at room temperature. Also, the post-ageing was done after the alkali fusion where the fused sample was allowed to stand for a period of twenty-four (24) hours at normal room temperature.

Synthesis: After the post-ageing, the following were carried out:-

Agitation and Shaking: The four evenly divided and weighed samples were charged into an air-tight jar each and 87ml of water was added into each jar. The solution(s) or mixtures were thoroughly shaken for each hourthroughoutthe twenty-four hours (24) of the day.

Heating: The well shaken samples were then introduced to a water bath and heated at a monitored temperature of 80˚C for 3, 6 and 9 hours respectively.

Filtration: The synthesized zeolite was allowed to cool off for some time and filtered using a vacuum filter set-up. The sample jars were washed/rinse with distilled water to get all the lingering samples off the jar walls. The cake(s) that formed on the filter-paper were removed and the collected filtrate was discarded accordingly as it's unwanted.

Drying: The gotten cakes (synthesized zeolite) from the filtration step were charged into a laboratory oven and dried at a temperature of 60˚C overnight.

Characterization Techniques

XRF Analysis: The X-ray Fluorescence Spectometry (XRF) analysis was conducted

XRD Analysis: The dried synthesized product was characterized using the XRD analysis to explore the chemical components available in the zeolite synthesized. More so, this gives the crystallographic structural status and phases of the entire component in a material. Besides, X-ray Powder

diffraction (XRD) is an efficient analytical technique used to identify and characterize unknown crystalline material.

RESULTS AND DISCUSSION

Elemental Analysis Result

Table 1 : XRF analysis result

Element	Concentration (wt%)
Na_2O	0.082
MgO	0.194
Al_2O_3	6.787
SiO_2	87.305
P_2O_5	0.373
SO_3	0.531
Cl	0.042
CaO	2.318
K_2O	0.177
TiO_2	0.202
CrO_3	0.004
Mn_2O_3	0.022
Fe_2O_3	1.957
ZnO	0.00
SrO	0.007

X-ray Powder Diffraction (XRD): This gives the crystallographic structural status and phases of the entire component in amaterial. X-ray Powder diffraction (XRD) is an efficient analytical technique used to identify and characterize unknown crystalline material.

Zeolite Synthesized at 80°C for 3 hours respectively**.**

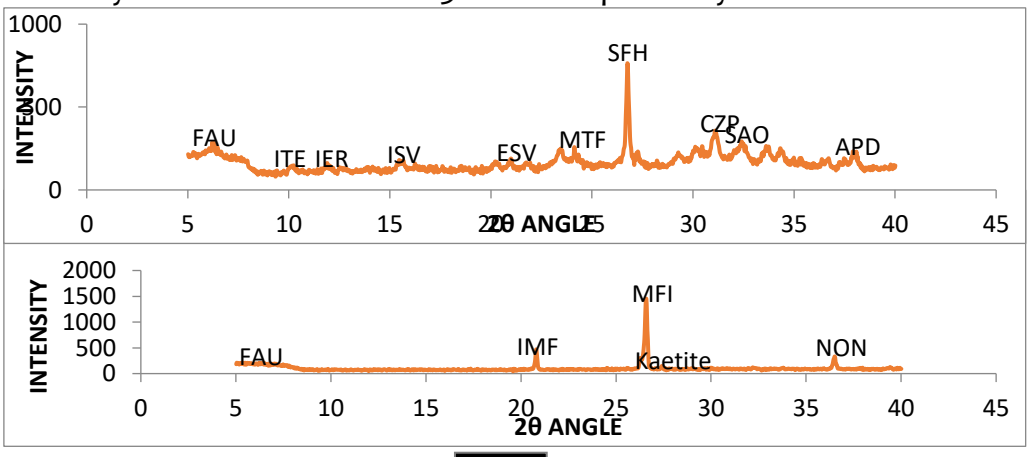

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Fig 3 : XRD Pattern for Zeolite Control Synthesized at 80˚C for 3 hours and XRD Pattern for

Table 2: Peak name (s) and Refined Chemical Composition (s) of Zeolite Y Control-point Synthesized at 60˚C for 6 hours

Code	Component	Chemical Composition
IMF	Calcined IM-5	$[Si_{228}O_{576}]$
MFI	ZSM-5 Calcined	$[Si_{96}O_{192}]$
Kaetite	-	$[Si_{12}O_{24}]$
IHW	ITQ-32	$[Si_{112}O_{224}]$
MTW	ZSM-12 Calcined	$[Si_{56}O_{112}]$
NON	2-aminopentane Nonasil	$[C_{36}][Si_{88}O_{176}]$
SZR	SUZ-4 hydrated	$[K_{3.256}][Si_{72}O_{144}]$
APD	AlPO-D	$[Al_{16}P_{16}O_{64}]$

TABLE 3: Peak name(s) and Refined Chemical Composition (s) of Sandstone-based ZeolitControl synthesized at 60˚C for 3 hours

Code	Component	Chemical Composition
GON	GUS-1	$[Si_{32}O_{64}]$
ITE	ITQ-3	$[Si_{64}O_{128}]$
IFR	ITQ-4, Calcined	$[Si_{32}O_{64}]$
ISV	ITQ-7,Siliceous,Calcined	$[Si_{64}O_{128}]$
SAV	Magnesium STA-7	$[Mg_{4.8}Al_{19.2}P_{24}O_{96}]$
ESV	ERS-7 framework	$[Si_{48}O_{96}]$
MTF	MCM-35	$[Si_{44}O_{88}]$
FAU	Faujasite	$[Al_{56}O_{22.4}Si_{175.7}][Al_{16.3}O_{384}]$
SFH	SSZ-53	$[Si_{64}O_{128}]$
OSI	UiO-6,Calcined	$[Al_{16}P_{16}O_{64}]$
CZP	Chiral Sodium Zincophosphate $P6_122$	$[Na_{2.04}O_{4.08}Zn_{12}P_{12}O_{48}]$
SAO	STA-1 Magnesium Aluminophosphate	$[P_{28}Al_{28}O_{112}]$
UFI	UZM-5	$[K_{5.204}Si_{36}Al_8O_{128}]$

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APD	AlPO-D	$[Al_{16}P_{16}O_{64}]$

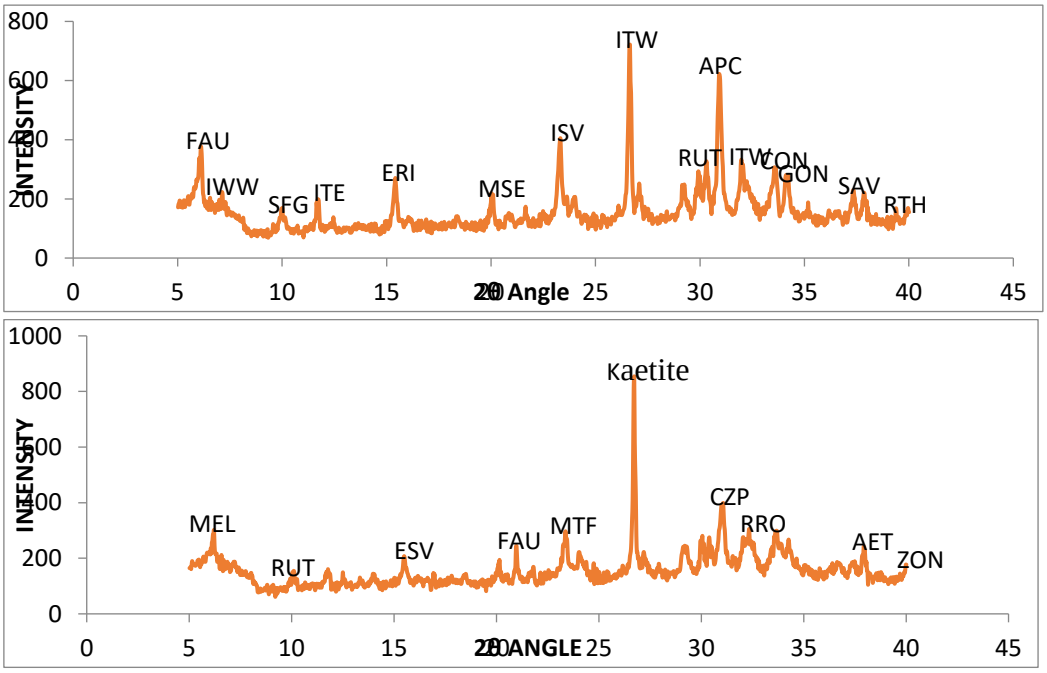

Figure 4: XRD Pattern for Zeolite synthesizedat 80˚C for 6 hours and 9 hours respectively

TABLE 4: Peak name (s) and Refined Chemical composition (s) of Zeolite Y synthesized at 60˚C for 6 hours

Code	Component	Chemical Composition
MEL	(ZSM-11,Calcined)	$[Si_{96}O_{192}]$
RUT	RUB-1 o,SiO_2 framework	$[Si_{36}O_{72}]$
MFS	(ZM-57,SiO_2 framework)	$[Si_{36}O_{72}]$
MFI	Tetrapropyl ammonium ZSM-5	$[Si_{96}O_{192}]$
ESV	ERS-7 framework	$[Si_{48}O_{96}]$
SFE	(SSZ-48,Calcined)	$[Si_{14}O_{28}]$
FAU	(Na-X,Dehydrated)	$[Na_{92.9}][Si_{103.68}Al_{88.32}O_{384}]$
IWW	ITQ-22	$[Si_{112}O_{224}]$
MTF	MCM-35	$[Si_{44}O_{88}]$
UFI	UZM-5	$[K_{5.204}][Si_{56}Al_8O_{128}]$
Kaetite	-	$[Si_{12}O_{24}]$
SAV	(Magnesium STA-7)	$[Mg_{4.8}Al_{19.2}P_{24}O_{96}]$

Tridynite		$[Si_{48}O_{96}]$
CZP	Chiral Sodium Zincophosphate	$[Na_{2.04}O_{4.08}][Zn_{12}P_{12}O_{48}]$
RRO	RUB-41	$[Si_{18}O_{36}]$
EZI	-	$[Al_{24}P_{24}O_{96}]$
ZON	Tetramethylammonium ZAPO-M1	$[Si_{112}O_{224}]$

TABLE 5: Peak name (s) and Refined Chemical composition (s) of Zeolite Y synthesized at 60˚C for 9 hours

Code	Component	Chemical Composition
FAU	Faujasite	$[Na_{40.32}(H_2O)_{171.84}][Si_{103.68}Al_{88.32}O_{384}]$
IWW	ITQ-22	$[Si_{112}O_{224}]$
SFG	SSZ-58	$[Si_{74}O_{148}]$
CDO	MCM-65,Calcined	$[Si_{36}O_{72}]$
ITE	ITQ-3,Calcined	$[Si_{64}O_{128}]$
ERI	Erionite	$[K_2Na_{1.86}Ca_{1.3}Mg_{0.7}(H_2O)_{6.12}][Si_{27}Al_9O_{72}]$
MSE	MCM-68	$[Si_{112}O_{224}]$
OSI	UiO-6, Calcined	$[Al_{16}P_{16}O_{64}]$
ISV	ITQ-7,Siliceous, Calcined	$[Si_{64}O_{128}]$
ITW	ITQ-12	$[Si_{24}O_{48}]$
RUT	RUB-10,SiO_2 Framework	$[Si_{36}O_{72}]$
CON	CIT-1	$[Si_{56}O_{112}]$
GON	GUS-1	$[Si_{32}O_{64}]$
APC	AlPO-C, Hydrated	$[(H_2O)_{16}O_8][Al_{16}P_{16}O_{64}]$
ITW	ITQ-12	$[Si_{24}O_{48}]$
CON	CIT-1	$[Si_{56}O_{112}]$
EZT	EMM-3	$[Al_{24}P_{24}O_{96}]$
LAU	Laumonite	$[Ca_4(H_2O)_{18}][Si_{16}Al_8O_{48}]$
SAV	Magnesium STA-7	$[Mg_{4.8}Al_{19.2}P_{24}O_{96}]$

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CDO	UZM-25,calcined	$[Si_{36}O_{72}]$
RTH	RUB-13	$[Si_{36}O_{64}]$

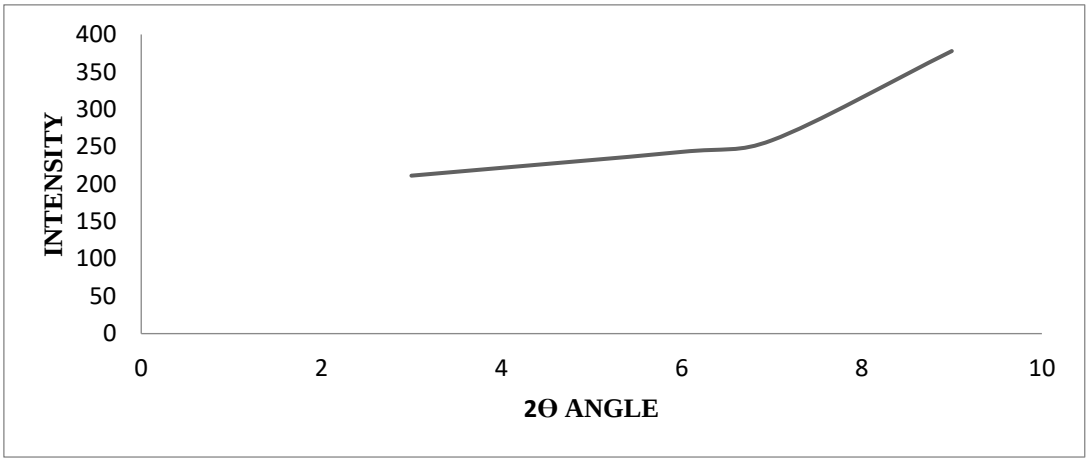

Figure 5: Plot of Faujisite zeolite formation against Reaction time

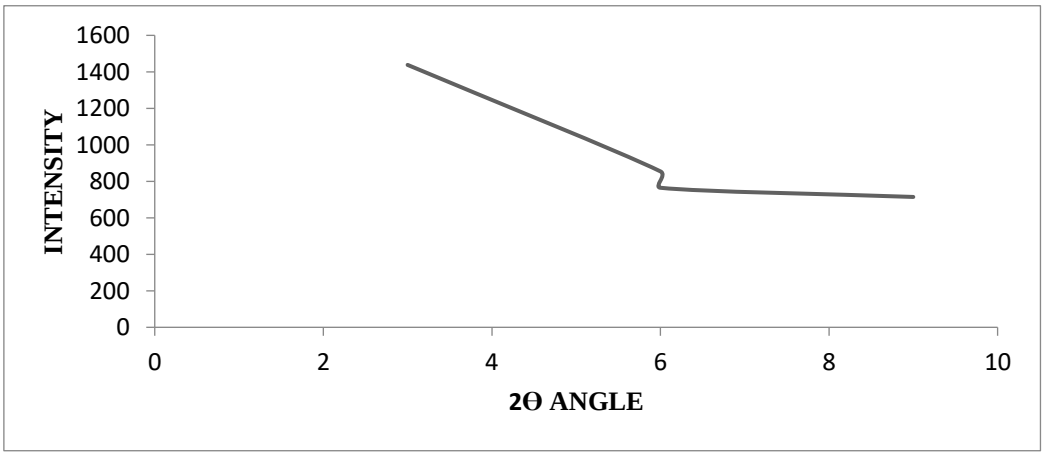

Figure 6: Plot of Quartz depolymerisation against Reaction time

RESULTS AND DISCUSSION

The results gotten from the synthesis of zeolite using alkali fusion at a fusion temperature of 600˚C are as presented in the tables above.

Table 1 is shows the XRF analysis result of the beneficiated sandstone material showing the presence of Na_2O,MgO,Al_2O_3,P_2O_5,SO_3,Cletc in trace amount relative to the major compositions of concern i.e $SiO_2$and Al_2O_3which are in weight percentage (wt%) of 87.305 wt% and 6.787 wt% , respectively.

The XRD results are shown in figures 4, 5 and 6 respectively as peaks which have corresponding tables attached to them in order to create easy understanding of what the peaks represent.

Figure 3 shows the XRD pattern of the zeolite control synthesized in the absence of excess Al_2O_3at 80˚C for 6 hours which indicates a zeolite Y intensity of 469.1 at a 2 Theta angle of 20.8 against the corresponding quartz of 1438.1 intensity and 2 Theta angle of 26.57.

Figures 4, 5 and 6 represent the XRD patterns of zeolite Y synthesized with excess Al_2O_3 at 3,6, and 9 hours respectively to balance the required SiO_2 to Al_2O_3 ratio for the formation of the zeolite material in view which in turn enhances the depolymerisation of the siliceous material (Quartz) present in the sandstone.

CONCLUSION

From the results generated so far, it can be established that the introduction of excess alumina (Al_2O_3) coupled with high fusion temperature has a positive impact on quartz deterioration, depolymerisation and breaking of the strong bonds lying between the quartz crystals relative to less alumina (Al_2O_3) content thereby giving way or room for the binding, fusing and coalescing of the SiO_2 and Al_2O_3 crystals to form ''Aluminosilicate'' which is termed ''Zeolite''.

RECOMMENDATION

More research should be conducted in this area in order to ascertain the exact amount of alumina (Al_2O_3) coupled with high fusion temperature needed for quartz deterioration, depolymerisation and breaking of the strong bonds lying between the quartz crystals relative to less alumina (Al_2O_3) content thereby giving way or room for the binding, fusing and coalescing of the SiO_2 and Al_2O_3 crystals to form ''Aluminosilicate'' which is termed ''Zeolite''

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