



DESIGN AND FABRICATION OF PILOT SCALE ZEOLITE Y GELATION UNIT OF 3.0 KG METAKAOLIN PER BATCH CAPACITY

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Abstract - A pilot-size gelation unit to convert 3.0kg of metakaolin (with already ascertained silica: alumina ratio) per batch was designed, fabricated and test ran. The metakaolin was prepared from locally sourced Udung Uko kaolinite clay in Akwa Ibom State, Nigeria. Sodium Hydroxide was used during the reaction and NaAlO_2 and Na_2SiO_3 was obtained as a solid product. The gelation unit comprises of a vertical vessel, on it was mounted an electric motor, relieve valve, heating element, isolation valves, sight glass and pressure indicator. The Control panel had a temperature indicator/controller, timer, switches, contactors etc. Mole ratios of the components in the reaction mixture were $\text{Na}_2\text{O} : \text{Al}_2\text{O}_3 : \text{SiO}_2 : \text{H}_2\text{O} = 3.76 : 1 : 2.81 : 112.67$. The material of construction selected for the gelation reactor was stainless steel type 316 to withstand the alkaline environment of the concentrated sodium hydroxide at the reaction temperature $>140^\circ\text{C}$. Power requirement of the reactor stirrer motor was 111W and the reactor was 18 liters, the pressure relief valve range was 0 - 10 bars and temperature gauge range was 0 - 300°C . Rockwool of calculated thickness of 6.143cm was used as lagging material to minimize heat loss to the surrounding. The equipment was test ran and the resulting gel was aged and analyzed using X-ray fluorescence (XRF) and X-ray diffraction (XRD) machines and gel was found to be Zeolite Y.

Keywords: Design, Fabrication, Gelation, Metakaolin, Zeolite Y

1 Introduction and Review of Related Literature

Metakaolin (MK) is anhydrous calcined form of kaolin (Clay), calcination of kaolin is usually done at high temperature at an extended number of hours in a furnace; Egwu *et al.* (2022). Many previous research on metakaolin gelation; Akinruli *et al.* (2021), Krisnandi *et al.* (2018), Maciver *et al.* (2020) Mgbemere *et al.* (2018), Omisanya *et al.* (2012) and Yusuf *et al.* (2019); were done at the laboratory scale with no consideration for the design and fabrication of the gel reactor.

Gels may be produced using a copolymerization or condensation-polymerization method. Hydrothermal production of Zeolites typically entails combining metakaolin in a very alkaline atmosphere and stirring to create a uniform gel (Biljana *et al.*, 2010). Before crystallization, the

resultant gel is aged in a sealed autoclave at a predetermined temperature for a defined period, Biljana *et al.* (2010).

The three main reactants of this synthesis are alkali, mineralizer, and water. The framework's major construction unit(s) are silica and alumina, with alumina also contributing to the framework's residual charge, Kotek *et al.* (2012).

The counter ion that generates the ion exchanging property is the alkali cation, which functions as a guest molecule and neutralizes any remaining charge in the framework, Rahier *et al.*, (2000). Water serves as a solvent and guest molecule, while hydroxyl radicals (OH-) provide an environment favorable for nucleation and crystal formation. When crystallization of certain Zeolites is desired, other elements known as structural directing agents (SDAs) or templates are used. Organic

templates and inorganic templates are the two basic categories here, Rahier *et al.* (2000).

In Zeolite synthesis, organic/inorganic molecules serve as templates for new structures to form during gelation or nucleation by arranging oxide tetrahedra in a certain geometric topology around themselves, Manabhanjan *et al.* (2015).

The template, or SDA, plays a crucial role in Zeolite crystallization since it determines the final structure. Cationic inorganic templates are essential to the Zeolite crystallization process, Idowu & Sanya, (2015). However, the use of an organic template has greatly increased the variety of produced Zeolite structures; Babalola *et al.* (2017).

In most cases, we choose our templates according to factors like:

- i) Solubility of the solution
- ii) Stability in synthesis conditions,
- iii) steric compatibility
- iv) feasible framework stabilization
- v) How easily the templates can be removed without damaging the Zeolite structure or having a harmful effect on the environment. Babalola, (2015)

The template, whether organic and inorganic, basically performs four functions

- a) Influence the gelation and/or nucleation process
- b) Lower the chemical potential of the lattice formed
- c) Improvement of the stability
- d) Control of the formation of a particular topology through its size and shape.

Templates used in Zeolite synthesis include cations such as Lithium (Li^+), Sodium (Na^+), Potassium (K^+),

Calcium (Ca^{2+}) and Barium (Ba^{2+})

Arikan *et al.* (2009).

This work is therefore aimed at designing and fabricating a gelation reactor for Zeolite Y synthesis.

2 Methodology

2.1 Design

Aspen Plus version 11 was used to simulate the design process with known parameters (from laboratory results). A series of computations

and concepts of basic engineering were performed.

A 3.0-kilogram batch of dealuminated metakaolin (DMK) with a known silica and alumina ratio was used. This was initially calcined to Metakoalin (MK).

2.1.2 Gelation reactor:

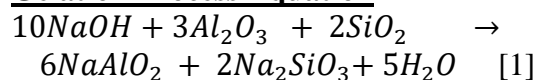
The model chosen for gel formation was a batch reactor: The result of the DMK from X-ray fluorescence (XRF) analysis was used in determining the mass balance around the reactor and thus the volume of the reactor. The XRF results of the DMK and MK used in the design are shown in Table 1 below.

Table 1: XRF results of the DMK/MK

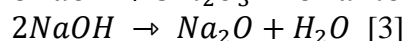
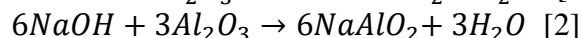
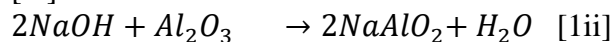
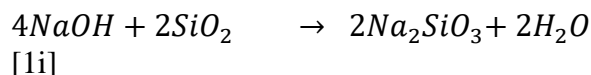
Chemical Composition	DMK Mole %	MK Mole %
SiO_2	51.1	51.5
Al_2O_3	18.5	36.9
SO_3	5.1	5.1
K_2O	0.522	0.615

Source: Babalola, (2015)

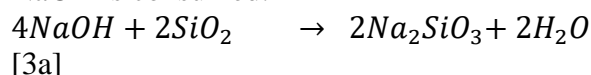
Gelation Process Equation



From the Equation 1, the following equation are attained.



The moles of NaOH left will further react with the unreacted SiO_2 from the equation Hence the reaction is expected to continue till all NaOH is consumed.



Determining the Material balance around the reactor

Figure 1 and Table 2 shows the material balance around the reactor.

Aspen Plus version 11 simulation results with XRF result were used and compared to obtain the data in Table 2.



Figure 1: Material Balance around the reactor.

Table 2: Material Balance for Gelation

Components	DMK/MK (g)	NAOH (g)	GEL(g)
H_2O	556.22	9795.60	11076.56
$NaOH$	0.00	2069.20	0.00
$Al_2O_3(s)$	492.38	0.00	0.00
$SiO_2(s)$	1360.03	0.00	75.60
Na_2SiO_3	0.00	0.00	2614.20
$NaAlO_2$	0.00	0.00	507.10
K_2O	13.90	0.00	13.90
SO_3	135.74	0.00	135.75
Others	103.23	0.00	103.23
Sub-Total	2661.50	11864.80	14526.3
Total	14526.3		14526.3

Determination of volume of Gelation Reactor

Data gotten from Table 2 were worked on to generate data for Table 3 which was used to calculate the volume for the reactor.

Table 3: Determination of volume of Gelation Reactor

Components	Density (ρ)	Mass (kg)	Fractional Composition (x)	$\rho_i x_i$
$NaOH$	2.13	2069.20	0.67	1.43
Al_2O_3	3.95	492.38	0.20	0.79
SiO_2	2.65	1435.63	0.13	0.35
Others		10529.09		
Total		14526.3	1.00	2.56

Volume of Reactor (V):

$$V = \frac{Mass}{Density} = \frac{14526.3}{2.56} = 5674.33 \text{ cm}^3$$

A safety factor of 3 to give room for gases, water vapour, pressure build up was chosen.

The actual Volume will be $3 * 5674.33 \text{ cm}^3 = 17023.078 \text{ cm}^3 \cong 18000 \text{ cm}^3$

Mole ratios of the components in the reaction mixture were

$$Na_2O : Al_2O_3 : SiO_2 : H_2O = 3.76 : 1 : 2.81 : 112.67$$

Height(h) and Diameter (d) of cylindrical reactor:

$$V = Ah = \pi r^2 h = \frac{\pi d^2 h}{4}$$

$$h = 2d \quad (\text{Perry and Green, 2008})$$

Thus,

$$V = \frac{\pi d^2 h}{4} = \frac{\pi d^2 (2d)}{4} = \frac{\pi d^3}{2} = \frac{3.142 d^3}{2}$$

$$d^3 = \frac{2V}{\pi} = \frac{2 * 18000}{3.142} = 11457.67 \text{ cm}$$

$$d = \sqrt[3]{(11457.67)}$$

$$d = 22.54 \text{ cm} \cong 23 \text{ cm}$$

$$h = 2d = 2 * 23 = 46 \text{ cm}$$

Determining Time (t) for the reaction

From Fogler, (2006) (Page 150, 2nd order in batch reactor), Time (t) for the gelation reaction can be calculated from:

$$t = \frac{X_A}{K(1-X_A)C_{A0}} \quad [4]$$

Where:

K is rate constant as calculated using Bawa., (2017) parameters

X Fractional Conversion (90%)

C_{A0} is initial concentration of Na
0.9

$$t = \frac{0.056 * (1 - 0.9) * 0.0533711}{0.056 * (1 - 0.9) * 0.0533711} = 3011sec = 50.18mins$$

$t \cong 50mins$

Thus, time for gelation is approximately fifty minutes

Determination of insulation material thickness (t_{rw})

The following evaluation was performed to determine the optimal thickness of rock-wool (rw) insulation around the reactor to reduce heat transfer to the surroundings:

$$q = \frac{m_{total} C_{pav} \Delta T}{t} \quad [5]$$

Where

m_{total} = Total mass of reacting system (Subtotal of streams 1 and 2)

Amkpa J A, and Nur A B., (2016).

C_{pav} = average specific heat capacity

ΔT = maximum temperature allowed to be lost through the insulation material.

t = reaction time (50.18 minutes)

$$q = \frac{m_{total} C_{pav} \Delta T}{t} = \frac{14.5263 * 4200 * 2}{50.18 * 60} = 40.53W$$

$$q = \frac{2\pi L \Delta T_{Overall}}{\ln \frac{r_2}{r_1} + \frac{\ln \frac{r_3}{r_2}}{k_{rw}} + \frac{\ln \frac{r_4}{r_3}}{k_a}} \quad [6]$$

Where

q is the energy flow per unit time;

k_a is the thermal conductivity of aluminum;

k_s is the thermal conductivity of stainless steel;

k_{rw} is the thermal conductivity of rock wool;

L is the height of the cylinder/reactor;

r_1 is the inner radius of the stainless steel (0.150m- diameter was calculated as 30cm);

r_2 is the inner radius of the rock wool (0.152m);

r_3 is the inner radius of the aluminium casing; (0.152 + t_{rw})

r_4 is the outer radius of the aluminum casing;(0.154 + t_{rw})

$k_s = 16.0958W/mK$

$k_{rw} = 0.0415W/mK$

$k_a = 273W/mK$

$$40.53 = \frac{2 * 3.142 * 0.6 * 90}{\ln \left(\frac{0.152}{0.150} \right) + \frac{\ln \left(\frac{0.152 + t_{rw}}{0.152} \right)}{0.0415} + \frac{\ln \left(\frac{0.154 + t_{rw}}{0.152 + t_{rw}} \right)}{273}}$$

$$40.53 = \frac{339.336}{0.0008229 + \frac{\ln \left(\frac{0.152 + t_{rw}}{0.152} \right)}{0.0415} + \frac{\ln \left(\frac{0.154 + t_{rw}}{0.152 + t_{rw}} \right)}{273}}$$

$$t_{rw} = 0.06143m = 6.143cm$$

Determination of the power requirement of the motor for metakaolin slurry mixing:

$$N_p = f(Re, Fr)$$

Where:

N_p =Power Number

Re= Reynolds number

Fr= Froude Number

The Above equation then becomes:

$$\frac{P}{\rho N^3 D_T^5} = f\left(\frac{\rho N D_T^2}{\mu}, \frac{N^2 D_T}{g}\right) \quad [7]$$

For un-baffled tanks, at higher Reynolds number, a vortex f and the Froude number Fr has a significant effect on the power number.

The general equation is then modified

$$P = N_p \rho N^3 D_T^5 \quad \text{Mc Cabe et al, (1993)}$$

Where:

P is Rotor Power (Watt)

N_p is power number

ρ is density of slurry (kg/m³=2.56 kg/m³)

N^3 is Rotor agitation speed (rev/sec)

D_T^5 is Rotor/impeller diameter (meter)

To compensate for transmission losses, frictional losses in the gear box and reduced efficiency of the electric motor, a safety factor of 25% was selected.

$$P = N_p * \rho * N^3 * D_T^5$$

$$P = 1.27 * 2560 * 5.8^3 * 0.1695^5 = 89W$$

$$P_{actual} = 1.25 * 89 = 111W$$

2.2 Material Selection

The gelation process took place in a reactor built of type 316 stainless steel. Improvisational elements were also considered. Outlet Pipes and fittings were mostly constructed of plastic material because they were easily available and corrosion resistant. Stainless steel was also used for certain of the top fittings, notably those that would be subjected to high temperatures, pressure and corrosion.

2.3 Fabrication and assembling

The fabrication of the gelation unit followed the completion of the simulation of the unit, design, and material selection. It was made separately and then assembled together. A pressure gauge, level gauge, pressure relief

valve, and temperature gauge were all part of the reactor's built-in equipment.

Fabricated Reactor

Figure 2 below shows the Fabricated gelation Unit

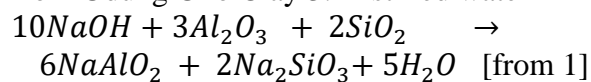


Figure 2: Fabricated Reactor

2.4 Test Running

The materials used for the reaction in other to test run the gelation unit include:

1. Concentrated sodium hydroxide, 98 wt%
2. Dealuminated Metakaolin (3.0 kg), prepared from Udung Uko Clay
3. Distilled water



Distilled water was added to the gel reactor at the specified level. Once the motor was running, the 3.0 kg of metakaolin required to react with the sodium hydroxide was added to the gel reactor. Reaction started instantly when

reactor homogeneity was achieved and slurry (35% solid) with timing was complete. The gel was collected from the bottom valve of the reactor. The alkali in the metakaolin was then aged and profiled (as shown in Figure 3).

3. Discussion of Results and Findings

Tables 4a and 4b below show the XRF analysis result and the breakdown of the gelation unit reactor design specifications respectively. Figure 3 shows the X-ray diffraction (XRD) Pattern for gelled/9 days aged sample where Zeolite Y (faujasite) was formed while Table 5 is the financial cost of the design and Fabrication.

Table 4a: Results of X-ray fluorescence (XRF) Analysis of Raw, Beneficiated, Calcined, Gelation/Aged Udung Uko kaolin

Chemical Constituent	Raw Clay (Mole %)	Beneficiated (Mole %)	Calcined (Mole %)	Crystallized after 9 days Aging (Mole %)
SiO ₂	84.64	72.90	70.77	68.44
Al ₂ O ₃	7.38	10.91	13.19	17.05
CaO	0.76	4.66	7.94	0.916
SO ₃	0.65	1.69	0.00	1.67
K ₂ O	1.24	1.56	1.60	1.42
Fe ₂ O ₃	0.57	2.05	1.68	2.52
TiO ₂	1.77	3.35	2.86	4.84
Cl	2.95	2.84	1.91	3.10
LOI	0.37	1.02	0.36	5.60
SiO ₂ /Al ₂ O ₃	11.45	6.68	5.36	4.01

Table 4b: Design specifications

S/N	Design parameters	Specifications
1	Volume of gel reactor,	18 liters
2	Height of reactor	46cm
3	Internal diameter of reactor	23cm
4	Thickness of stainless steel	2mm
5	Speed of gear impeller	6.5 rev/sec (100RPM)
6	Density of slurry	2.56g/cm ³
7	Viscosity of slurry	1.73 x 10 ⁻² Pa.S
8	Reynolds number of impellers,	NRe 4983
9	Power requirement for the stirrer motor.	111W
10	Insulating material	Rock wool
11	Thickness of insulating material	6.143cm

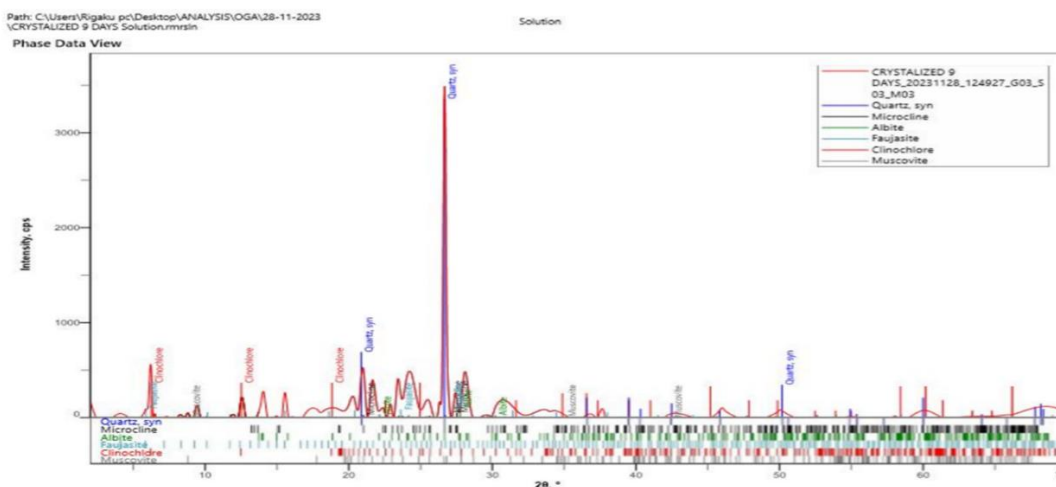


Figure 3: XRD Pattern for 9 days Aged sample

TABLE 5: BILL OF ENGINEERING MEASUREMENT AND EVALUATION (BEME) FOR THE DESIGN AND FABRICATION OF A PILOT SCALE GELATION UNIT OF 3.0 KG METAKAOLIN PER BATCH CAPACITY

	DESCRIPTION	MATERIAL AND SIZES	QUANTITY	RATE (#)	COST (#)
1	STAINLESS STEEL PLATE (GRADE 316)	2MM*1000*950	1	70,000	70,000
2	SCREWS/CONTROL PANEL		1	7,500	7,500
3	ELECTRIC MOTOR WITH REDUCTION GEAR (100RPM ELECTRIC MOTOR)	DC 24V	1	30,000	30,000
4	ELECTRIC CABLE	1.5MM*3 CORES	3 YARDS	2000	6,000
5	PLUG/SWITCHES	15 AMPS/100AMPS			12,000
7	ELECTRIC HEAT RESISTANT CABLE	4MM (SINGLE CORE)	10YARDS	800	8000
6	DIGITAL THERMOMETER	RexC100	1	18,000	18,000
7	THERMOCOUPLE		1	5,000	5,000
8	CONTACTORS	SCHNEIDER Telemecanique	1 1	16,000 8,000	16,000 8,000
9	CIRCUIT (RESISTOR, DIODES, CAPACITORS) FOR THE DC MOTOR STEP DOWN TRANSFORMER (220-26,2000mA)		1 1	4,500 3,500	4,500 3,500
10	BOLTS, WASHES, GASKET, TUBING, CLIPS, FOIL TAPE, 75 MICRONS STAINLESS STEEL WIRE MESH, AND NUTS	STEEL M10*1.25			10,000
11	WELDED ELECTRODE	GAUGE 12	3 PACKETS	22,000	66,000
12	CERAMIC CONNECTOR		1	2,000	2,000
13	BEARINGS (6203)		2	1000	2000
14	STAINLESS STEEL ROUND BAR/ SHAFT (GRADE 304)	20MM*50	1	9,000	9,000
15	MILD STEEL SQUARE BAR	3MM*50*50	1	26,000	26,000
16	PVC OUTLET VALVES AND PIPES WITH SEIVES	4 INCHES		20000	20,000
17	STAINLESS STEEL FILLER ROD		0.5	24,000	12000
18	TIMER RELAY	PAULGRAND	2	6,000	12,000
19	RELIEVE VALVE	CDI STL MO 140PSI	1	12,000	12,000
20	PRESSURE INDICATOR	BOURDON 316SS	1	24,000	24,000
21	TEMPERATURE INDICATOR	WSS 311W	1	26,000	26,000
22	2.5KW HEATING ELEMENT (NICHROME WIRE)	3.5 YARDS	3.5	6,200	21,700
23	INSULATION MATERIALS (ROCK WOOL)		0.5 BAG	36,000	18,000
25	WORKMANSHIP/LABOUR				30,000
26	TRANSPORTATION AND MISCELLANEOUS				15,000
27	GRAND TOTAL				494,200

4. Conclusion and Recommendations

Gelation unit which comprised of gel reactor and its accessories was successfully designed, fabricated and test-ran. Gel is a chemical intermediate for Zeolite Y production, the materials selected for the fabrication of the gelation unit were stainless steel Type 316, rock wool, plastic pipes, plastic valves, temperature gauge, heating element, sight glass, pressure relief valve etc. The gel formed was profiled and found to be Zeolite Y. It is anticipated that this work will promote large scale industrial production which is a shift from the usual laboratory gel synthesis for Zeolite production and hence will engender socio-economic development of nations with abundant kaolin resource such as Nigeria.

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