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IMPROVING ZEOLITE Y SYNTHESIS: EXPLORING THE EFFECTS OF STARTING MATERIAL, REACTION CONDITIONS AND METHODS TO ENHANCE PERFORMANCE

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Abstract: Zeolite Y, a crystalline aluminosilicate with a highly ordered nanoporous structure, is a versatile material widely utilized in various industrial applications due to its remarkable catalytic, adsorbent and ion-exchange properties. This material plays a crucial role in the petrochemical industry, where it functions as a catalyst in the processes, helping to enhance the production of chemicals. Additionally, Zeolite Y is highly effective in gas separation technologies, where it selectively adsorbs certain gases, making it indispensable in natural gas processing and air purification. Its ion-exchange capacity also makes it a valuable tool in water treatment, where it aids in the removal of undesirable ions, thus improving water quality. The broad applicability of Zeolite Y extends to sectors such as environmental management, energy production and industrial manufacturing. To improve its use, simulation tools like Aspen Plus are employed to model the synthesis processes, providing insights into reaction conditions and material behaviours. This modelling technique helps identify optimal condition for maximizing efficiency and reducing energy consumption. This work emphasized the significance of improving energy to enhance Zeolite Y performance in specific processes. In this work, 205,280.84KJ/Hr of energy was saved during Zeolite Y synthesis; the starting material (clay) was sourced locally from Udung Uko in Akwa Ibom State of Nigeria.The method used ultimately ensured materials, manpower and energy were maximized to reduce production cost, reduce equipment downtime, improve compliance to industrial and government regulations, boost productivity, improve product quality, meet some sustainable development goals and reduce synthesis error.

Keywords: Application, Processes, Simulation, Synthesis Zeolite Y

1. Introduction

Zeolite is a mineral known for its crystalline structure and microporous nature. The unique properties of Zeolites, such as high surface area, excellent adsorption capabilities, and catalytic activity, make them versatile in various applications [1], [2].

Zeolite Y (a specific type of Zeolite), has found use as a catalyst in different chemical reactions. Its shape selectivity and other properties contribute to its effectiveness in promoting specific chemical transformations. This makes Zeolite Y valuable in industries where precise control over reaction pathways is crucial, enhancing the efficiency of processes. It has found applications in catalyzing reactions such as cracking, isomerization, and alkylation [1]. Zeolite Y's high surface area and well-defined pore structure make it effective for adsorption applications. It has been used for adsorption of gases, liquids, and pollutants from various environment [3]. Zeolite Y is employed in ion exchange processes due to its ability to selectively exchange ions. It has been used in water softening, heavy metal removal and as a component in detergents [4]. Zeolite Y is used as a builder in detergents, contributing to the removal of calcium and magnesium ions in hard water. This application enhances the overall performance of detergents [5]. Zeolite Y has been incorporated into membranes for gas and liquid separations. These membranes exhibit high selectivity based on molecular size and shape, making them valuable for various separation processes [2], [6].

2. Materials and Method

Aspen Plus version 11 was used to simulate the design process with known parameters (from laboratory results-some of which are shown in Table 1). A series of computations and concepts of basic engineering were performed.

3.0 kilograms batch of raw kaolinite clay (kaolin) from Udung Uko in Akwa Ibom State was used for the experiment/simulation.

The reaction set and simulation strategy during the processes are presented below:

(A) The reaction set:

(i) Calcination

There are changes that occur when kaolin is heated in the furnace. The reaction series include the formation of highly disordered metakaolin formed at about 550-900^0C as shown in the Equation 1:

$$2Al_2Si_2O_5(OH)_{4(s)} \xrightarrow{Heat\ (550-900^0C)} Al_2Si_2O_{7(s)} + 4H_2O \quad (1)$$

Kaolin → Metakaolin + steam

Further heating to about 925^0C, a defected alumina silica structure called spinel is formed

$$2Al_2Si_2O_{7(s)} \xrightarrow{Heat(925^0C)} Si_3Al_4O_{12(s)} + SiO_{2(s)} \quad (2)$$

Metakaolin → Spinel + Crystobalite

then at 1050ºC, spinel will become mullite with more SiO_2 formed thus

$$3Si_3Al_4O_{12(s)} \xrightarrow{Heat\ (1050^0C)} 2Si_2Al_6O_{13(s)} + 5SiO_{2(s)} \quad (3)$$

Spinel → Mullite + Crystobalite

(ii) Dealumination

Dealumination reaction is given as shown in Equation 4:

$$Al_2Si_2O_{7(s)} + H_2SO_{4(l)} \rightarrow Al_2(SO_4)_3.3H_2O + 2SiO_{2(s)} \quad (4)$$

Metakaolin + Acid → Alum + Silica

This can be summarized thus,

$$Al_2O_{3(s)} + 3H_2SO_{4(l)} \rightarrow Al_2(SO_4)_{3(l)} + 3H_2O$$

The benefit of leached alum in the production of alumina was studied by Kenneth and Nwankwo, [7]. The leached alum from dealumination of the Udung Uko clay (Akwa Ibom State, Nigeria) can be used in the production of aluminum at the proposed Aluminum Smelter

Company of Nigeria (ALSCON), Ibekwe in Ikot Abasi as cited in both Vanguard Newspaper, Nov 16,2019 and Business Day Newspaper July 29,2019

(iii) Gelation Method

Na_2O:SiO_2 was taken to be 0.6

And

$H_2O : Na_2O$ was taken to be 30,

$$2NaOH \rightarrow Na_2O + H_2O \quad (5)$$

That is

$$Na_2O + H_2O \rightarrow 2NaOH$$

Sodium aluminate salts ($6NaAlO_2 + 2NaSiO_3$) were formed from alkaline fusion as shown in the Equation 6:

$$10NaOH_{(aq)} + Al_2O_{3(s)} + 2SiO_{2(s)} \rightarrow 6NaAlO_2 + 2Na_2SiO_3 + 5H_2O \quad (6)$$

Sodium dealuminated Calcined Kaolin Sodium Aluminate salts

More Sodium fuses with more of the silica, enhancing more gel(Na_2SiO_3) formation/crystal growth as shown in the Equation 7:

$$2NaOH_{(aq)} + SiO_{2(s)} \rightarrow Na_2SiO_3 + H_2O \quad (7)$$

(B) The Simulation Strategy:

The simulation was carried out with Aspen Plus software to construct the detailed process flowsheets, simulate the chemical reactions and analyzed the thermodynamics and kinetics of the Zeolite formation. This simulation software facilitates the exploration of different reaction conditions and reaction condition.

The simulation set up and components were selected as either solid or non-conventional property. 31 component ID were imputed. The temperature, pressure and flow rate of materials were captured in the developed process flow diagram and the results (input, cost, stream) of the individual units were evaluated. Other considerations to the simulation were property type (thermodynamics), method (common filter), solubility (3), base method (solids), phase (vapour-liquid) and calculation option (STEAM-TA). The design inspired energy utilization in the synthesis of the Zeolite.

3. Results and Discussion

3.1 Improvement deployed for Synthesis

3.0 kilograms of Clay from Udung Uko (Akwa Ibom State Nigeria) was sourced and used for the synthesis. The raw data used for the simulation was from the X-Ray Fluorescence (XRF) analysis of the Raw, Beneficiated, Calcined, Udung Uko kaolin shown in Table 1. The molar ratio of SiO_2/Al_2O_3 in the starting material (clay) can be adjusted to influence the framework structure of Zeolite Y [8].

Synthesis temperature and reaction time was controlled to influence the crystallization process and crystal size of Zeolite Y in accordance with what was reported in [9].

The concentration of Sodium Hydroxide (NaOH) to control the alkalinity of the reaction mixture was also adjusted in the simulation as highlighted in [10]. Organic templates or structure-directing agents incorporated into the mix was able to control the crystal size and shape of the formation in line with the procedure reported in [11]. This also affected Seeding techniques to induce nucleation and promote the formation of Zeolite Y crystals [12]. Post-synthesis treatments such as ion exchange, calcination or steaming to modify the properties of Zeolite Y was also applied according to the work of Corma and Rey [13]. Other results and discussion of the simulation are as shown in i,ii,iii

Table 1: Results of X-ray fluorescence (XRF) Analysis of Raw, Beneficiated, Calcined, Udung Uko kaolin.

	ENERGY IN (kJ/hr)		ENERGY OUT (kJ/hr)	
	DRY-K1	DUTY	CALC-MK	STEAM
Enthalpy flows	-42315.34	3575.17	-34068.39	-4671.77
Total	-38740.16		-38740.16	

Table 2: Energy balance of the Furnace Unit

Chemical Constituent	Raw Clay Mole %	Beneficiated Mole %	Calcined Mole %
SiO_2	84.64	72.90	70.77
Al_2O_3	7.38	10.91	13.19
CaO	0.76	4.66	7.94
SO_3	0.65	1.69	0.00
K_2O	1.24	1.56	1.60
Fe_2O_3	0.57	2.05	1.68
TiO_2	1.77	3.35	2.86
Cl	2.95	2.84	1.91
LOI*	0.37	1.02	0.36
SiO_2/Al_2O_3	11.45	6.68	5.36

LOI*: Loss on ignition

3.2 Improvement of synthesis Equipment

(i) Improving the Furnace

The exhaust of the furnace (Steam) was not wasted but utilized in the heat exchanger to heat up water(W) and air (A) to generate warm water (W1) and hot air (A1) used in other processes. Energy recovered was -38740.16KJ/Hr as shown in Table 2.

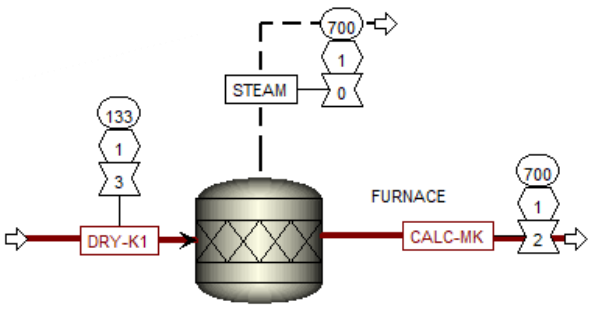

Figure 1: Improved Furnace Unit

In the furnace, the reaction set that took place when Dry kaolin (Dry K1) was converted to Calcined metakaolin (Calc-MK):

$Al_2Si_2O_5(OH_4)_{(s)}\ (Kaolin) \rightarrow Al_2Si_2O_{7(s)}\ (Metakaolin) + 2H_2O_{(g)}$ (8)

$Al_2Si_2O_{7(s)}\ (Metakaolin) \rightarrow Al_2O_{3_{(s)}} + 2SiO_{2(s)}$

$3Al_2O_{3_{(s)}} + 2SiO_{2(s)} \rightarrow Si_2Al_6O_{13(s)}\ (Mullite)$

(ii) Improving the multiple Tube Heat Exchanger (MTHE)

The inlets of the MTHE generated the warm water (W1) and hot air (A1), these outlet streams are used in the beneficiation and drying Units respectively.

Energy recovered in the MTHE unit was 122916.85KJ/Hr.

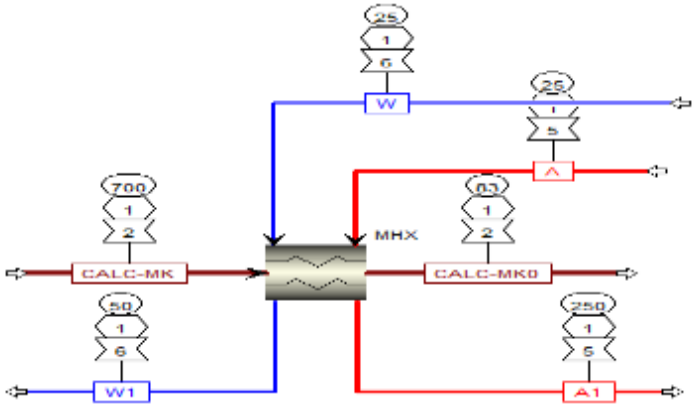

Figure 2: Improved multiple Tube Heat Exchanger Unit

Table 3: Energy balance of the multiple Tube Heat Exchanger Unit

	ENERGY IN (kJ/hr)			ENERGY OUT (kJ/hr)		
	A	CALC-MK	W	A1	CALC-MK0	W1
Enthalpy flows	0.00	-34068.39	-88848.46	1176.59	-35829.18	-88264.26
Total	-122916.85			-122916.85		

(iii) Improving the Dryer

The hot (steam) exhaust of the furnace (800°C) which was used to heat up the air via the heat exchanger (A2-250°C) was channeled through the wet kaolin (Wet-K) to dry it. What then leaves the drying Unit was dry kaolin (Dry -K) as shown in Figure 3.

Energy recovered in the dryer was 43623.83KJ/Hr as shown in Table 4.

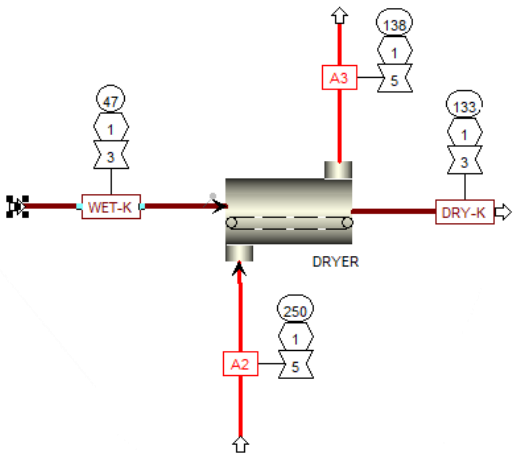

Figure 3: Improved Drying Unit

Table 4: Energy balance of the drying Unit

	ENERGY IN (kJ/hr)		ENERGY OUT (kJ/hr)	
	A2	WET-K	A3	DRY-K
Enthalpy flows	1176.60	-44800.43	-1303.37	-42315.34
Total	-43623.83		-43623.83	

Table 5: Production Plant flowsheet Stream Name and Description

S/N	STREAM NAME	DESCRIPTION
1	A2	Air Circulation
2	A3	
3	CALC-MK	Metakaolin obtained after calcination
4	CALC-MK0	
5	DRY-K/K1	Dry Kaolin
6	W	Water Circulation
7	W1	

4. Conclusion

5.

The objectives of this research have been largely met. The sequence of the synthesis involved data gathering, simulation of the synthesis process using laboratory results, literature data to design then improvement of the process and process equipment. The very first step was collection of raw kaolin from Udung Uko, subjecting it to beneficiation to confirm its suitability for Zeolite Y synthesis. The simulated furnace converted kaolin to metakaolin which ensured the refined kaolin was in a reactive metastable phase. This amorphous metakaolin was obtained at a temperature of 800℃ and a residence time of 4 hours. The percentage of the impurities in the metakaolin was reduced through beneficiation, the silica/alumina molar ratio reduced from 11.45 to 6.68.

In conclusion, it can be said that at least 205,280.84KJ/Hr. of energy was saved during Zeolite Y synthesis and that starting materials (Clay) can be sourced locally (in this case, from Udung Uko in Akwa Ibom State of Nigeria. Also, materials, manpower and energy were maximized to reduce production cost, reduce equipment downtime, improve compliance to industrial and government regulations, boost productivity, improve product quality, meet some sustainable development goals and reduce synthesis error.

Continued exploration of this approach/strategies (using natural Clay and not synthetic chemicals) not only contributes to advancements in materials science but also offers sustainable solutions to address contemporary challenges in the fields of catalysis, material performance and environmental remediation.

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