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CONVERTING CLAY TO ZEOLITE Y CATALYST: AN ECO-FRIENDLY AND SUSTAINABLE SYNTHESIS METHOD

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Abstract: In this study, we explored an eco-friendly way to the synthesis of Zeolite Y, a versatile and widely-used Zeolitic material, from locally sourced kaolinite clay. Zeolites are crystalline aluminosilicate materials known for their distinctive porous structures and unique catalytic properties, which make them highly valuable in various industrial applications. The focus of our research was to investigate clay as an alternative raw material for Zeolite Y synthesis, offering a more sustainable solution compared to traditional methods that often rely on energy-intensive and environmentally harmful raw materials. The synthesis process involved extracting and purifying kaolinite clay minerals, followed by the hydrothermal activation method to produce Zeolite Y. This technique was designed to minimize environmental impact while maintaining the material's efficiency. To ensure the synthesized Zeolite Y met quality standards, we used several analytical techniques, including X-Ray Fluorescence (XRF) to determine elemental composition, X-Ray Diffraction (XRD) to analyse crystal structure, Scanning Electron Microscopy (SEM) to observe the material's morphology, and the Brunauer–Emmett–Teller (BET) method to assess its specific surface area. The results revealed that the properties of the Zeolite Y synthesized from clay were comparable to those of commercially available imported materials, demonstrating that the eco-friendly synthesis method can yield a product of similar quality and performance. This strategy not only provides a sustainable alternative to conventional synthesis methods but also promotes the use of locally sourced materials, contributing to both economic and environmental benefits.

Keywords: Aluminosilicate, Clay, Eco-friendly, Synthesis, Zeolite Y.

1.0 Introduction

Zeolites are well-known for their diverse applications in catalysis, adsorption, and ion exchange due to their unique porous structures and acidic sites. Traditional methods for synthesizing Zeolite Y typically involve the use of sodium aluminate and sodium silicate, which are derived from non-renewable resources and involve high-energy processes [1]. Kaolin clay group, which is represented chemically as $Al_2O_3SiO_2.2H_2O$, is an important material used in the synthesis of Zeolite. Kaolin which is composed of 90 - 95% kaolinite, is a two layered alumino-silicate material having chemical composition similar to that of Zeolite, thus, clay can be used as precursor for Zeolite nanomaterial synthesis [2]. According to Yadav and Garg [3], Zeolite is an

important industrial catalyst which could be employed in industrial processes such as fluid catalytic cracking, ion exchange, catalysis, sorption etc. Zeolites could be obtained as naturally occurring mineral or synthesized in the laboratory, the use of Zeolite for other industrial purposes such as the removal of sulphur, oxygenate, nitrogenate, and aromatics are well documented in literature. [1]. Zeolite is used for refinery operations for cracking, agriculture (as manure), aquaculture, healthcare application, environmental protection, ion exchangers in laundry detergents in which calcium and magnesium in water are exchanged for sodium present in the Zeolite [4]. Another industrial application of Zeolite is in the area of catalysis. For instance, over 99% of high-octane gasoline production in the world is dependent on Zeolite and this is largely due to its intrinsic properties. Okoye [5] described catalytic cracking using Zeolite as the most important operation that is used to obtain high-grade petroleum products from lower grade high molecular feed during refining operations. Its advantages over thermal cracking are in both the yield and product quality. Catalytic cracking using Zeolite also produces fewer hydrocarbon gases [6]. Due to its importance, over 500,000 tons of Zeolite nanomaterials are imported annually, at about 5 billion-naira [7]. Having seen the importance of Zeolite, this study investigates the use of clay as a sustainable and readily available source for synthesizing Zeolite Y, exploring the potential of eco-friendly chemistry principles in Zeolite production.

2.0 Materials and Method

The experimental procedures involved the extraction and purification of clay minerals, followed by the synthesis of Zeolite Y through hydrothermal activation methods. Various characterization techniques such as XRF, XRD, SEM and BET were deployed. Materials: Udung Uko clay (from Akwa Ibom State, Nigeria), Water, NaOH,

i) Method of eco-friendly Solvents: Water based synthesis was used as it is a more sustainable option compared to organic solvents. Use of environmentally friendly solvents or solvent-free synthesis methods to reduce the environmental impact is required for eco-friendly Approach for Sustainable Catalyst Production [8].

ii) Sustainable Precursors: Clay is a natural source of silica and alumina and chosen as a sustainable and readily available raw materials as precursors for Zeolite Y synthesis. [9].

iii) Energy-Efficient Techniques: Optimize reaction conditions to reduce energy consumption was deployed, Heat generated from the furnace was used to carry out drying. or other energy-efficient techniques for Zeolite synthesis [10].

iv) Recyclable Templates: We used recyclable or biodegradable templates (e.g NaOH), in the synthesis process. Organic templates are often used but choosing those that can be easily recycled or decomposed is more environmentally friendly [11].

Sodium aluminate salts ($6NaAlO_2 + 2NaSiO_3$) were formed from alkaline fusion as shown in the Equation 1:

$$10NaOH_{(aq)} + Al_2O_{3(s)} + 2SiO_{2(s)} \rightarrow 6NaAlO_2 + 2Na_2SiO_3 + 5H_2O \quad (1)$$

Sodium dealuminated Calcined Kaolin Sodium Aluminate salts

v) Waste Minimization: We implemented methods to minimize waste generation during the synthesis process. This included efficient recovery and recycling of by-products [12]. Leached alum from the dealumination operation can be very beneficial, the benefit of leached alum in the production of alumina was studied by Kenneth and Nwankwo, [13].

Dealumination reaction to form alum is given as shown in Equation 2:

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

Metakaolin Acid Alum Silica

3.0 Results and Discussion

The results demonstrate the successful synthesis of Zeolite Y from clay, highlighting the influence of synthesis parameters on the Zeolite's crystallinity, morphology, particle size and surface area. The eco-friendly approach using clay as a precursor showcases the potential for sustainable Zeolite production with reduced environmental impact [14]. The synthesized Zeolite Y from clay holds promise for applications in catalysis, including the catalytic conversion of biomass, oil refining processes, and environmental remediation. Its sustainable production pathway aligns with the growing demand for eco-friendly and renewable technologies [15].

a) X-Ray Fluorescence (XRF) Analysis

The compositional analysis of raw, beneficiated, calcined and aged Udung Uko kaolin was conducted using XRF with result presented in Table 1. The data reveals contaminant including potassium, iron, titanium and calcium oxide [10], [16]. Beneficiation reduced SiO_2 from 84.64 % to 72.90% indicating removal of free silica (quartz) [17]. Table 1 shows the beneficiated kaolin ferric nature, attributed to iron oxide presence, surpassing potassium levels. The significant TiO_2 content contributes to the white colour of the raw and beneficiated kaolin [18], [19]. The SiO_2/Al_2O_3 ratio for raw and beneficiated kaolin (11.45 and 6.68 respectively) falls within theoretical limits. Comparison of beneficiated and calcined Udung Uko kaolin reveals differences in the chemical composition. Notably, SO_3 decreased from 1.699% to 0.00% and Iron, Titanium and chloride values also reduced, likely due to calcination-induced removal of impurities e,g Illite, SO_3, albite and orthoclase [20].

Dealumination using the conventional method significantly altered the Udung Uko clay composition. Fe_2O_3 decreased from 1.68 to 1.20 % while Alumina increase slightly from 13.19 to 13.62 %, potentially due to factors like reaction time, acid quality or analysis error [21].

CaO and TiO_2 reduced from 7.94 to 0.55 % and 1.91 to 1.42 % respectively. This finding aligns with Belver et al [14]. The SiO_2/Al_2O_3 ratio increased from 5.36 to 5.83 % accompanied by a silica content rise. Dealumination of Udung Uko metakaolin reduced impurities, modifying the chemical composition and structure of the starting material.

b) X-Ray Diffraction (XRD) Analysis

XRD patterns of commercial Zeolite Y and synthesized Zeolite exhibit characteristic peaks at $2\theta = 7.5$, indicating similarities in their crystal structures [18]. Minor discrepancies are attributed to impurities such as Fe^{2+}, Ti^{2+} and Ca^{2+} [17]. Aging (curing) effect on TiO_2 and SiO_2 content were observed; the reduction in SiO_2 content led to increased TiO_2 content due to partial depolymerization of silica or solubility of occluded silica grains in the heterogeneous skeleton, achieving equilibrium [15]. Prior to aging, the Raw Udung Uko Clay underwent Beneficiation, Calcination and Dealumination as evident in XRD Pattern (Figures 1,2,3 and 4). This process increased the silicate anion concentration in solution. XRD patterns of synthesized Zeolite from Udung Uko kaolin at varying aging times (Figures 5a and 5b) closely match commercial Zeolite Y (Figure 5c).

However, crystallinity decreased with prolonged aging, indicating significant influence on Zeolite synthesis from metakaolin. The coordinated state of phases in the XRD spectra, (Figures 5a and 5b) reveals that 7 days aging yielded high crystallinity with a prominent Zeolite NaY peak. In contrast, 9 days aging showed decreased intensity due to quartz formation suppressing the crystallinity, we also see that the sample aged for 9 days compared favorably to the market commercial grade Zeolite Y and also that aged for 7 days compared favorably to the market commercial grade Zeolite Y both with reference to lattice spacing and 2 theta (Table 3 and Figure 7).

Prolonged aging (greater that 9 days), is predicted to reduce of K_2O content and potentially enhance crystallinity. This aligns with Babalola's assertion [1] that extended aging improves Zeolite yield and quality consistent with BET result.

c) Scanning Electron Microscopy (SEM) result of the developed Zeolite Y

The SEM images of synthesized Zeolite Y (Figures 6a and 6b) and commercial Zeolite Y (Figure 6c) reveals similarities in morphology. Figures 6a and 6b show minimal impurities (quartz), consistent with XRD findings [1,4]. The images exhibit spherical ordering, comparable to literature reports on Zeolite Y [17]. The 9 days aged Zeolite crystal exhibit similar shape and size to commercial Zeolite Y, featuring uniform, spherical morphology with particle sizes approximately 1µm [1]. This corroborates Babalola et al [18] SEM images of commercial Zeolite Y. Increasing SiO_2 and Al_2O_3 ratios in synthesized Zeolite (7 day and 9 day aged), decreases SiO_2/Al_2O_3 ratios from 5.83 to 4.77 to 4.01 respectively this observation aligns with expectation for Zeolite Y synthesis.

d) Brunauer–Emmett–Teller (BET) Surface area result of synthesized Zeolite Y

The synthesized Zeolite Y exhibited varying surface properties with aging time. After 7 days, the Zeolite had BET surface area of 199.139 square meters/gram, pore volume of 0.181 cubic centimeters/gram and average pore diameter of 28 Å. In contrast, the 9-day aged Zeolite displayed improved surface characteristics: BET surface area of 262.381 square meters per gram, pore volume of 0.227 cubic centimeters/gram and its average pore diameter as 26.4 Å. This 32% increase in surface area from 7 to 9 days indicates that aging enhances Zeolite material surface properties [1]. Commercial Zeolite Y, meanwhile, boasts higher surface area 640-696 square meters/gram, pore volume (0.325-0.447 cubic centimeters/gram) and smaller average pore diameter (1.99-2.71 Å). [16]. Tables 2a and 2b summarize the BET surface area results

Table 1: Results of X-Ray Fluorescence (XRF) Analysis of Raw, Beneficiated, Calcined, Dealuminated, Aged Udung Uko kaolin

Chemical Constituent	Raw Clay (Mole %)	Beneficiated (Mole %)	Calcined (Mole %)	Dealumination (Mole %)	Crystallized after 7 days Aging (Mole %)	Crystallized after 9 days Aging (Mole %)
SiO_2	84.64	72.90	70.77	79.55	71.43	68.44
Al_2O_3	7.38	10.91	13.19	13.62	14.95	17.05
CaO	0.76	4.66	7.94	0.55	0.81	0.916
SO_3	0.65	1.69	0.00	0.00	0.70	1.67
K_2O	1.24	1.56	1.60	1.490	1.54	1.42
Fe_2O_3	0.57	2.05	1.68	1.20	2.80	2.52
TiO_2	1.77	3.35	2.86	2.14	3.97	4.84
Cl	2.95	2.84	1.91	1.42	3.75	3.10
LOI*	0.37	1.02	0.36	2.11	1.03	5.60
SiO_2/Al_2O_3	11.45	6.68	5.36	5.83	4.77	4.01

LOI*: Loss on ignition

XRD Results:

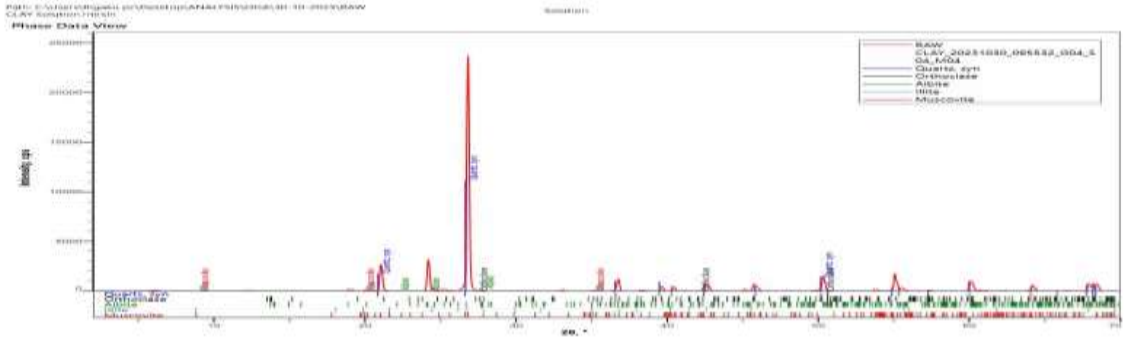


Figure 1: XRD Pattern for Raw Udung Uko Clay

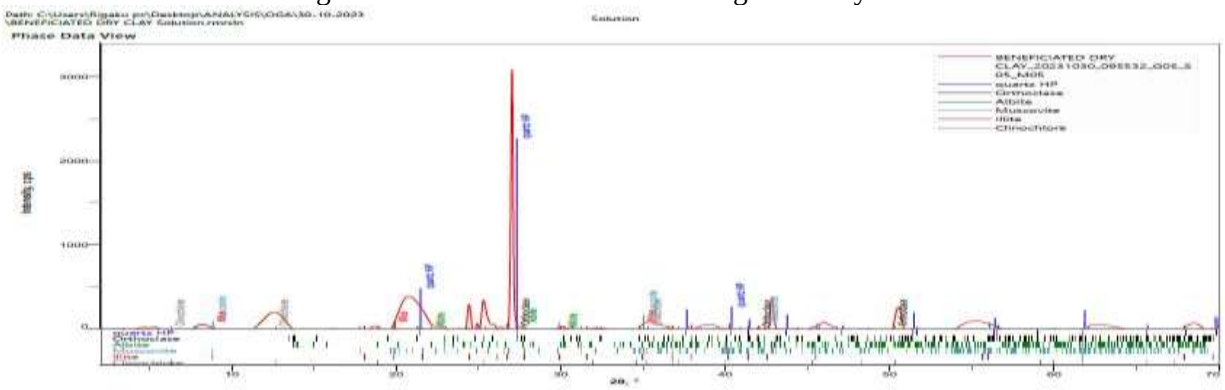


Figure 2: XRD Pattern for Beneficiated Udung Uko Clay

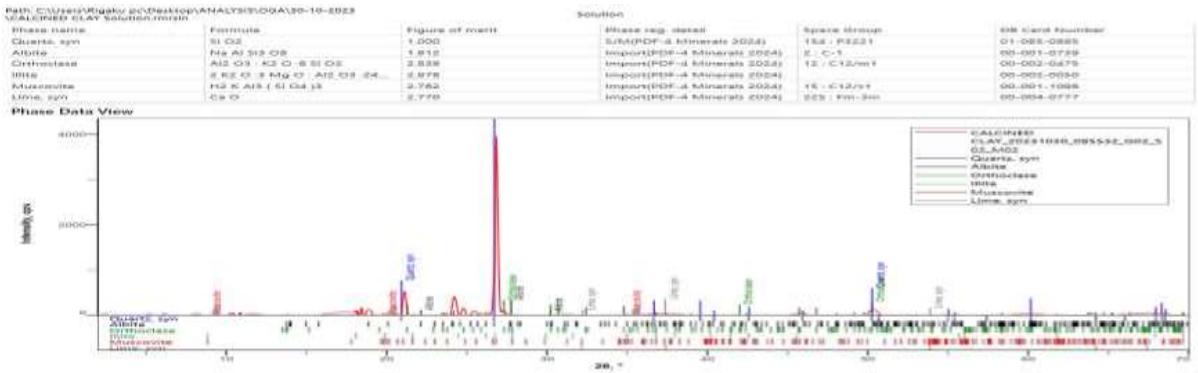


Figure 3: XRD Pattern for Calcined Udung Uko Clay

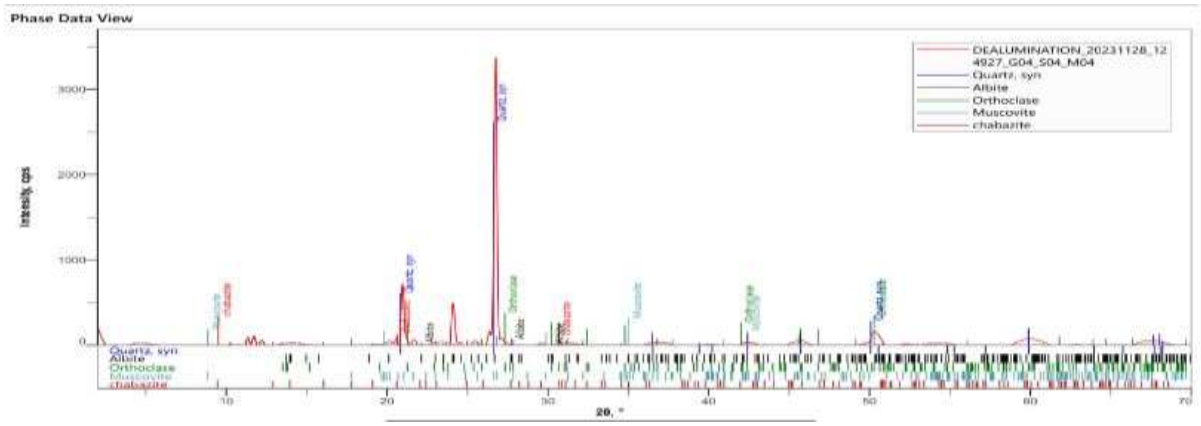


Figure 4: XRD Pattern for Dealuminated Udung Uko Clay

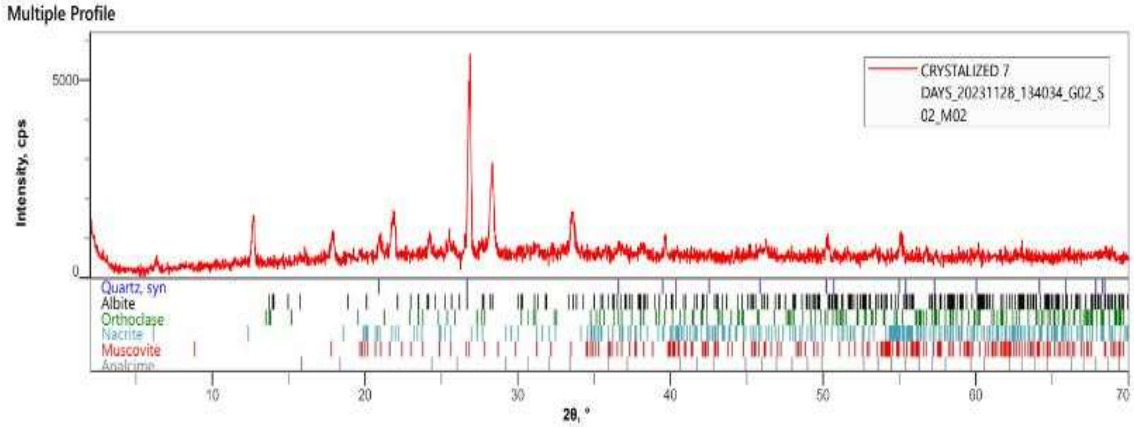


Figure 5(a): XRD Pattern for 7 days Aged sample

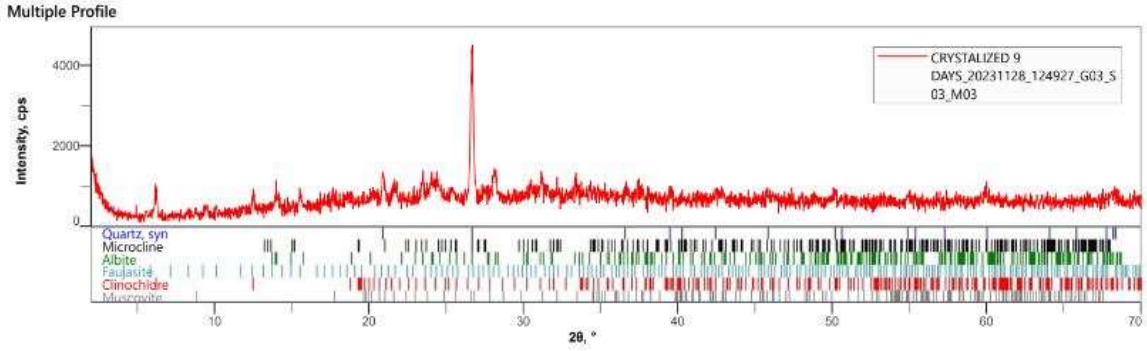


Figure 5(b): XRD Pattern for 9 days Aged sample

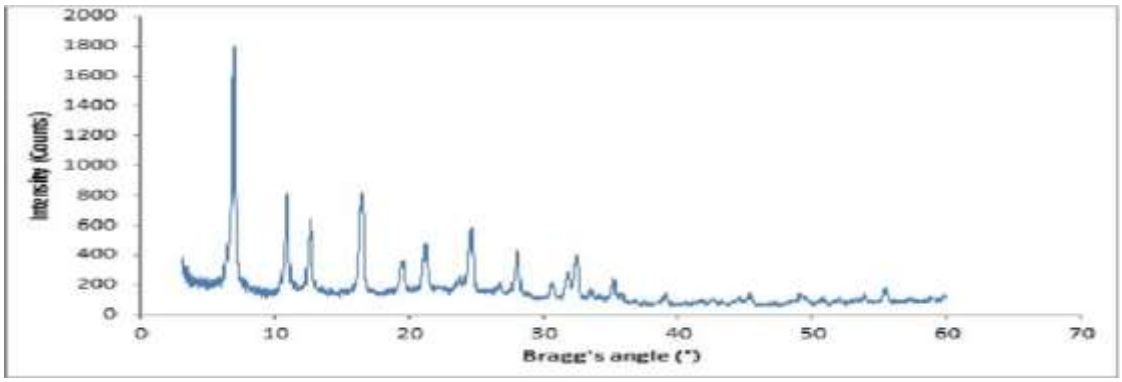
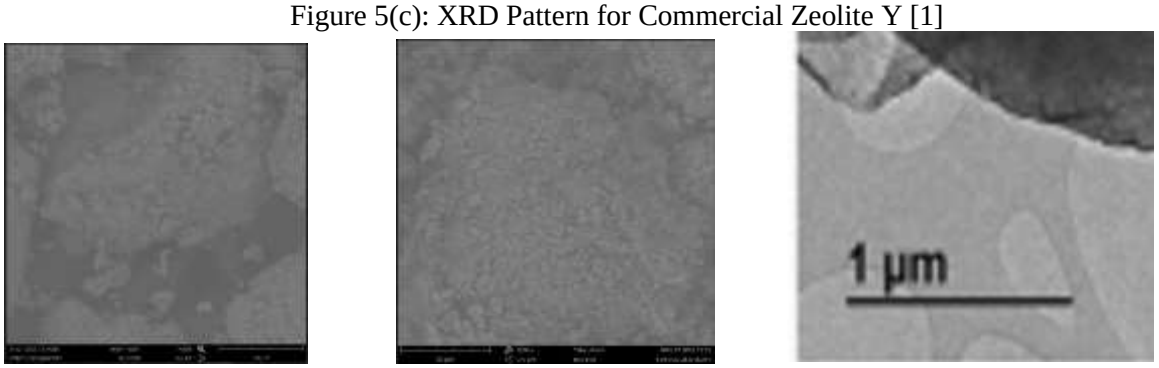


Figure 5(c): XRD Pattern for Commercial Zeolite Y [1]



(a) 7 days of Aged Sample (b) 9 days of Aged Sample (c) Commercial Zeolite Y [1]

Figure 6 SEM Images of (a)7 days Aged Sample (b) 9 days Aged Sample and (c) Commercial Zeolite Y

BET Surface area result of synthesized Zeolite Y

The BET surface result is presented in the tables below

Table 2a: BET Surface area result for Zeolite Y after 7 days of aging

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Analysis

Operator:	Abdulrahman Abdulkareem	**Date:**	2023/12/07
Sample ID:	7days	**Filename:**	7days.qps
Sample Desc:		**Comment:**	
Sample weight:	0.2 g	**Sample Volume:**	1 cc
Outgas Time:	3.0 hrs	**OutgasTemp:**	250.0 C
Analysis gas:	Nitrogen	**Bath Temp:**	273.0 K
Press. Tolerance:	0.100/0.100 (ads/des)	**Equil time:**	60/60 sec (ads/des)
Analysis Time:	72.8 min	**End of run:**	2023/12/07 11:31:09
Cell ID:	1		

Report

Operator:	Abdulrahman Abdulkareem	**Date:**	2023/12/07
Equil timeout:	240/240 sec (ads/des)		
Instrument:	Nova Station A		

Area-Volume Summary

Data Reduction Parameters Data

Thermal Transpiration: on **Eff. mol. diameter (D):** 3.54 Å **Eff. cell stem diam. (d):** 4.0000 mm

t-Method	**Calc. method:** de Boer
BJH/DH method	**Moving pt. avg.:** off
DR method	**Affinity coefficient (ß):** 0.3300
HK method	**Tabulated data interval:** 1
SF method	**Tabulated data interval:** 1
DFT method	**Calc. Model:** N2 at 77 K on carbon (slit pore, NLDFT equilibrium model)
	Rel. press. range: 0.0000 - 1.0000 **Moving pt. avg:** off
Adsorbate	**Nitrogen** **Temperature** 77.350K
	Molec. Wt.: 28.013 **Cross Section:** 16.200 A² **Liquid Density:** 0.808 g/cc
	Critical Temp.: 126.200 K **Critical Press.:** 33.500 atm **SuperCritic. K.:** 1.000
Adsorbent	**Carbon**
	DR. Exp (n): 2.000

Surface Area Data

SinglePoint BET	1.405e+02 m²/g
MultiPoint BET	1.991e+02 m²/g
Langmuir surface area	5.035e+02 m²/g
BJH method cumulative adsorption surface area	2.425e+02 m²/g
DH method cumulative adsorption surface area	2.578e+02 m²/g
t-method external surface area	1.991e+02 m²/g
DR method micropore area	2.414e+02 m²/g
DFT cumulative surface area	5.695e+01 m²/g

Pore Volume Data

BJH method cumulative adsorption pore volume	1.187e-01 cc/g
DH method cumulative adsorption pore volume	1.213e-01 cc/g
DR method micropore volume	8.578e-02 cc/g
HK method micropore volume	3.952e-02 cc/g
SF method micropore volume	1.146e-02 cc/g
DFT method cumulative pore volume	6.624e-02 cc/g

Pore Size Data

BJH method adsorption pore Diameter (Mode Dv(d))	2.446e+00 nm
DH method adsorption pore Diameter (Mode Dv(d))	2.124e+00 nm
DR method micropore Pore width	5.867e+00 nm
DA method pore Diameter (Mode)	2.800e+00 nm
HK method pore Diameter (Mode)	3.675e-01 nm
SF method pore Diameter (Mode)	4.523e-01 nm
DFT pore Diameter (Mode)	2.647e+00 nm

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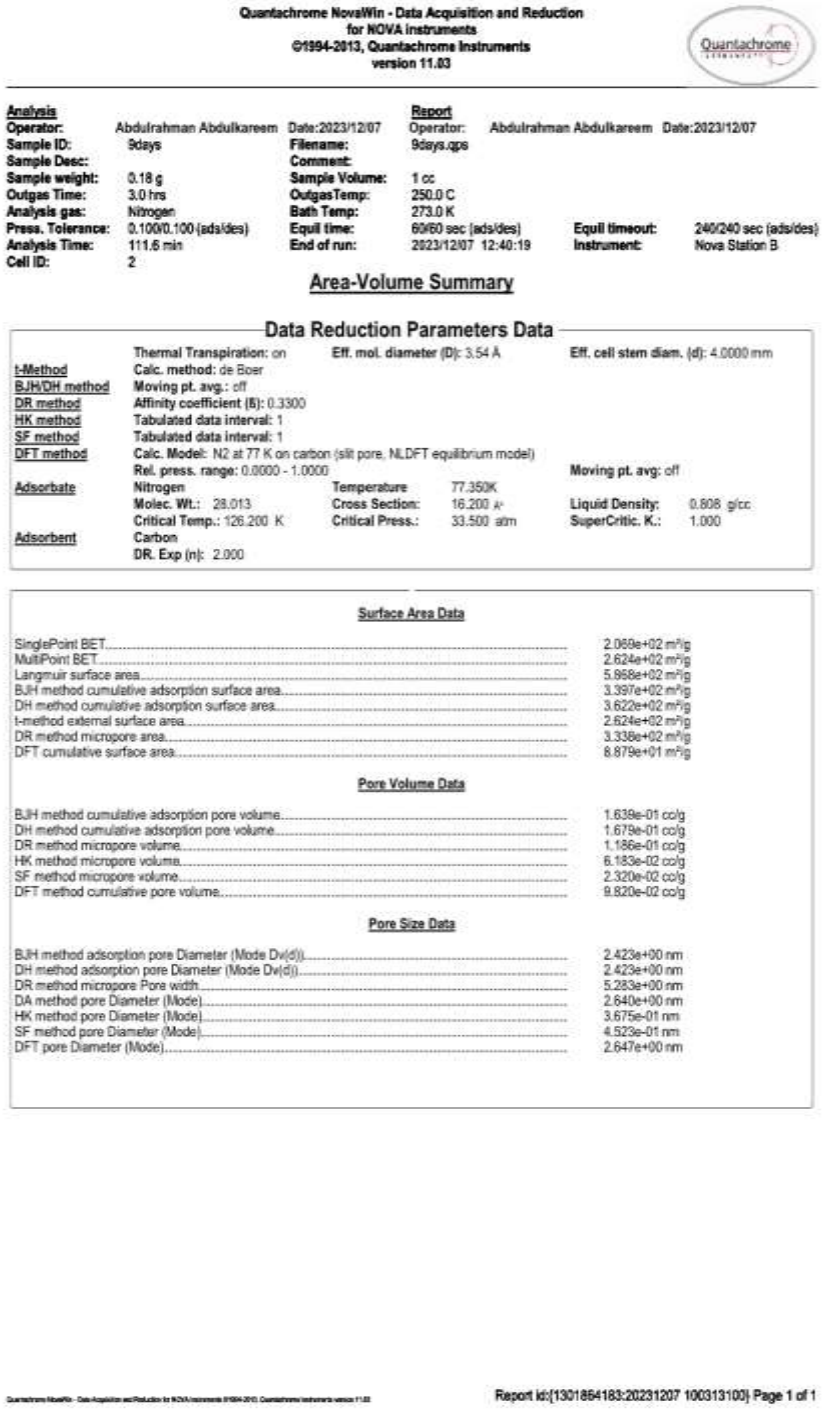
Table 2b: BET Surface area result for Zeolite Y after 9 days of aging



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Analysis				Report			
Operator:	Abdulrahman Abdulkareem	Date:2023/12/07		Operator:	Abdulrahman Abdulkareem	Date:2023/12/07	
Sample ID:	9days	Filename:	9days.qps				
Sample Desc:		Comment:					
Sample weight:	0.18 g	Sample Volume:	1 cc				
Outgas Time:	3.0 hrs	OutgasTemp:	250.0 C				
Analysis gas:	Nitrogen	Bath Temp:	273.0 K				
Press. Tolerance:	0.100/0.100 (ads/des)	Equil time:	60/60 sec (ads/des)	Equil timeout:	240/240 sec (ads/des)		
Analysis Time:	111.6 min	End of run:	2023/12/07 12:40:19	Instrument:	Nova Station B		
Cell ID:	2						

Area-Volume Summary

Data Reduction Parameters Data

	Thermal Transpiration: on	Eff. mol. diameter (D): 3.54 Å		Eff. cell stem diam. (d): 4.0000 mm
t-Method	Calc. method: de Boer			
BJH/DH method	Moving pt. avg.: off			
DR method	Affinity coefficient (ß): 0.3300			
HK method	Tabulated data interval: 1			
SF method	Tabulated data interval: 1			
DFT method	Calc. Model: N2 at 77 K on carbon (slit pore, NLDFT equilibrium model)			
	Rel. press. range: 0.0000 - 1.0000			Moving pt. avg: off
Adsorbate	Nitrogen	Temperature	77.350K	
	Molec. Wt.: 28.013	Cross Section:	16.200 Å²	Liquid Density: 0.808 g/cc
	Critical Temp.: 126.200 K	Critical Press.:	33.500 atm	SuperCritic. K.: 1.000
Adsorbent	Carbon			
	DR. Exp (n): 2.000			

Surface Area Data

SinglePoint BET	2.069e+02 m²/g
MultiPoint BET	2.624e+02 m²/g
Langmuir surface area	5.868e+02 m²/g
BJH method cumulative adsorption surface area	3.397e+02 m²/g
DH method cumulative adsorption surface area	3.622e+02 m²/g
t-method external surface area	2.624e+02 m²/g
DR method micropore area	3.338e+02 m²/g
DFT cumulative surface area	8.879e+01 m²/g

Pore Volume Data

BJH method cumulative adsorption pore volume	1.639e-01 cc/g
DH method cumulative adsorption pore volume	1.679e-01 cc/g
DR method micropore volume	1.186e-01 cc/g
HK method micropore volume	6.183e-02 cc/g
SF method micropore volume	2.320e-02 cc/g
DFT method cumulative pore volume	9.820e-02 cc/g

Pore Size Data

BJH method adsorption pore Diameter (Mode Dv(d))	2.423e+00 nm
DH method adsorption pore Diameter (Mode Dv(d))	2.423e+00 nm
DR method micropore Pore width	5.283e+00 nm
DA method pore Diameter (Mode)	2.640e+00 nm
HK method pore Diameter (Mode)	3.675e-01 nm
SF method pore Diameter (Mode)	4.523e-01 nm
DFT pore Diameter (Mode)	2.647e+00 nm

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Table 3: Comparison of lattice spacing, between the developed Zeolite and Commercial Zeolite Y. The XRD data for commercial Zeolite Y: characteristic peaks at 2θ = 7.5 [1]

7 days aged Sample		9 days aged Sample		Commercial Zeolite Y	
Angle (2Theta) deg	d, spacing (Å)	Angle (2Theta) deg	d, spacing (Å)	Angle (2Theta) deg	d, spacing (Å)
12.67	6.97	6.18	14.274	12.47	7.09
17.87	4.96	14.09	6.280	19.80	4.48
20.93	4.24	20.89	4.25	21.76	4.08
21.86	4.06	21.69	4.09	26.74	3.33
24.23	3.67	24.42	3.64	28.21	3.16
26.84	3.32	25.46	3.50	29.55	3.02
28.32	3.15	26.63	3.34	30.94	2.88
33.54	2.67	28.17	3.16	32.26	2.77
39.64	2.27	31.09	2.87	33.49	2.67
50.20	1.82	68.13	1.38	35.95	2.49
55.04	1.67			38.16	2.35

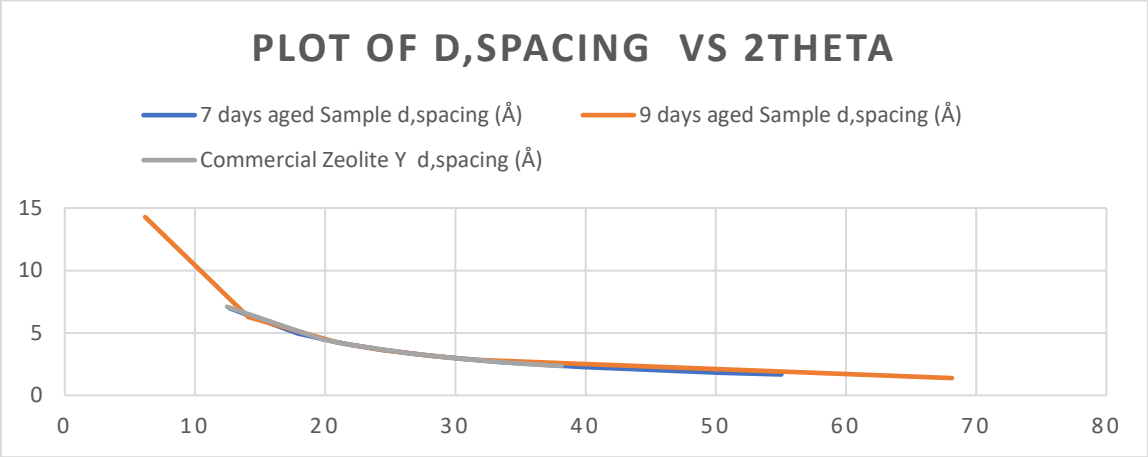

Figure 7: d spacing Vs 2 theta for aged and commercial Zeolite Y

4.0 Conclusion

This study presents a viable and environmentally conscious approach to Zeolite Y synthesis using clay as a precursor. The results demonstrate the feasibility of producing a sustainable catalyst with potential applications in various industries. The eco-friendly synthesis approach contributes to the ongoing efforts in developing eco-friendly materials for a more sustainable future that provide insights into the eco-friendly synthesis of Zeolite Y from clay.

The environmentally friendly synthesis method not only enhances the sustainability profile of Zeolite Y production but also broadens its applications as an eco-friendly catalyst.

By adopting this sustainable approach, researchers and industries can contribute to the development of eco-friendly catalysts that aligns with the sustainable development goals (SDG).

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