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Assessment of the Residue Characteristics of Edda Clay and Mbaduku Clay after Acid Leaching

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ABSTRACT: The assessment of the residue of Edda clay and Mbaduku clay was carried out. The XRD of the clays showed both clays to contain high kaolin content of 58% and 52%Theratio of silica to alumina of both clays was also determined and found to2.3 and 1.98. Hydrothermal processing was applied in achieving this. The process involved the reaction of calcined clay sample with varying concentrations of H_2SO_4 at temperature of about 89°C with additions of sodium carbonate to the mixture. The clay sample was maintained for 30 minutes at this temperature. This produced a clear solution above the residue. After filtration, the residue was then dried in an oven at 600^{0}C, weighed and in every case there showed a reduction in weight of the starting clay material.

I. INTRODUCTION

Kaolin has been described byMeoret al (2006) as a soft, white plastic clay mineral that consists chiefly of kaolinite. Widely known as a hydrated aluminum silicate $Al_2Si_2O_5(OH)_4$. It isformed by the alteration of feldspar and muscovite. It is an important industrial mineral, with various uses in many industrial applications. Nigeria for example has abundant deposits of kaolin dispersed in different states. However, the use locally is mostly limited to use in the ceramic and paint industry. In order to refine this kaoline, large investments is needed for the exploration of the mineral and its purification. Alumina which can be obtained from kaolin has been rarely extracted from these minerals across Nigeria. This

Kaolin is also useful raw material in the synthesis of Zeolites, since the synthesized zeolites have better properties compared to the naturally occurring ones. Golubev et al. (2021). The hydrothermal process used in leaching clays using alkalis, yields an aluminate gel which is a precursor for zeolite synthesis. Golubev et al in their work further state that the leaching process decreases the Si-Al ratio thus favoring the zeolitization process.

The zeolite synthesis procedure follows a two-step process as discussed by Kovo and Holmes (2010) and Benharrats, et al (2018).Kowo and Holmes further asserts that zeolite synthesis can be largely affected variables such as temperature, pressure, batch composition, Si/Al ratio, alkalinity, inorganic cations, aging time, stirring rate, organic templates, water content, and seeding. Furthermore, Tantawy and Alomari (2019) has detailed the process where alumina is first leached out from a kaolinite mineral and the which is often an alumino-silicate gel in the second stage is crystallized when mixed with a structural directing agent under a controlled temperature schedule in an autoclave. Furthermore, the type of acid used affects the composition of the final residue. Meoret al (2006),further stated that Al-Si ratio is critical to the outcome of the synthesis procedure as either a higher or lower value creates an excess of either alumina or Silica which greatly affects crystallinity and poresize formed.The type of clay used also affects the properties of the synthesized Zeolite, this is because clays with low alumina content results in zeolites of poor properties.

Temuujin, (2006) The final mineralogical and chemical composition of an acid-leached clay depends on many factors, including the composition of the starting clay, the acid concentration and the duration and temperature of the leaching process.

This research focuses of utilizing high kaolinite clays from Edda and Mbaduku clay in the processing of alumina residue as a precursor material for zeolite synthesis using variable concentration of sulphuric and hydrochloric acids and caustic soda as an additive.

II. MATERIALS & METHOD

Nguzu Edda clay used in this study was obtained from Court areain Edda local government area of Ebonyi state. Another sample used in the study was Mbaduku kaolin obtained at Mba-Adigam in Mbaduku, Vandeikya local Government area of Benue state. The samples were both dried atmospherically and crushed using an edge mill and sieved to obtain particles below 75 µm in size. This size was chosen due to its high surface area and its reaction with acids will be more effective.Calcining was done at 900^{0}C for 5 hours in a gas-firedkiln. Different dilute concentrations of hydrochloric acid and sulphuric acids were prepared containing 2, 5, 8 and 10 molar concentrations.

250ml of a 2M sulfuric acid (H_2SO_4) solution was mixed with 250ml of 2M hydrochloric acid (HCl) acid inside the flaskand then, 60g of the calcined sample mixed with 6g of sodium hydroxide pellets was then dissolves inside the acid mixture. The flack was fitted with a thermometer and a reflux condenser. The mixture was heated with an electric hot plate with magnetic stirrer to a temperature of 87^{0}Cthen maintained at that temperature range for 45 min while stirring.

After 45 minutes, the mixture was cooled to room temperature and filtered to remove the leach gelatinousresidue.The weight of the residue was determined. The experiment was repeated with different concentrations of acids while the weight of sodium hydroxide pellets was kept constant.The residue was the driedin an oven at 120°C for 8 hours.

s/no	Amount of clay	Concentration in M	Amount or residue after leatching (g)
1	60	10	28
2	60	8	31
3	60	5	34
4	60	2	39

Table 1 showing the amount of Mbaduku clay reacted with varying concentrations of acid.

s/no	Amount of clay	Molar Concentration	Amount or residue after leatching

1	60	10	29
2	60	8	31
3	60	5	33
4	60	2	36

Table 2 showing the amount of Edda clay reacted with varying concentrations of acid.

s/no	Amount of clay	Concentration in M	Amount or residue after leatching
1	60	10	28
2	60	8	30
3	60	5	31.7
4	60	2	33.6

Table 3 showing the amount of Nsu clay reacted with varying concentrations of acid.

s/no	Amount of clay	Concentration in M	Temperature of reaction	Amount or residue after leatching
1	50	10	65^{0}C	33
2	50	8	65^{0}C	40
3	50	5	65^{0}C	47
4	50	2	65^{0}C	44

Table 4 showing the amount of Mbaduku clay reacted with varying concentrations of acid at 65^{0}C.

s/no	Amount of clay	Concentration in M	Temperature of reaction	Amount or residue after leatching
1	50	10	65^{0}C	28
2	50	8	65^{0}C	31
3	50	5	65^{0}C	34
4	50	2	65^{0}C	38

Table 5 showing the amount of Edda clay reacted with varying concentrations of acid at 65^{0}C.

III.

IV. RESULT AND DISCUSSION

Analysis of kaolin samples were undertaken using X-Ray fluorescence, Phillips PW 1400 brand, using sodium bromide as the flux material. The x-ray diffraction patterns of the sets of samples were also obtained.

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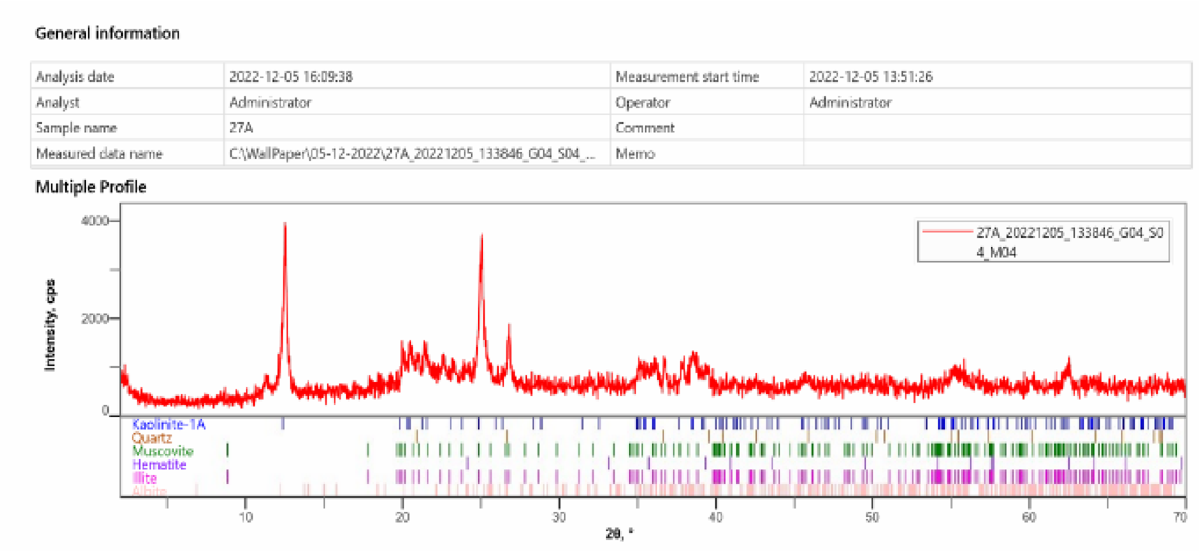

Fig. 1 X-ray diffractogram of Edda clay

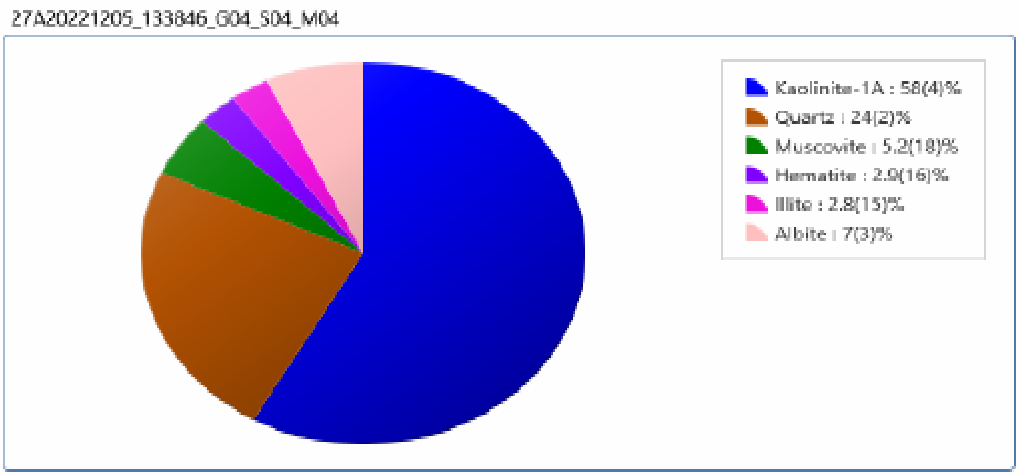

Fig. 2 mineralogy chart of Edda clay

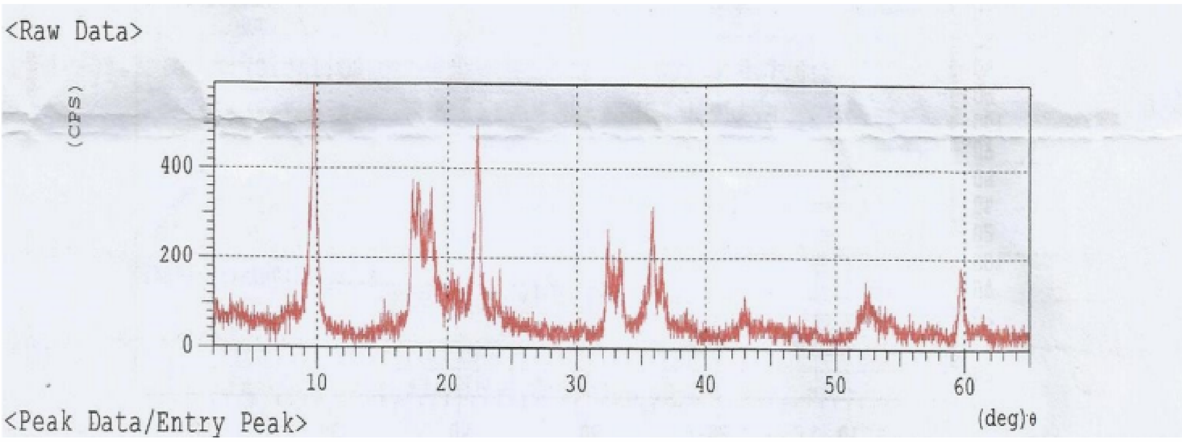

Fig. 3 Xraydiffractogram of Vandeikya clay

	Composition (%)	
Oxide	Mbaduku clay	Edda clay
SiO_2	56.53	53.32
$A1_20_3$	28.05	26.56
Fe_20_3	5.32	3.32
MgO	4.04	3.04
LOI	5.10	6.70

Table 6. Chemical Analysis of Edda and vandeikya clay raw clay

The characteristic white residue of the reaction between the 2 different clays with the acids was weighed after calcining was recorded at between 33 and 44 foe Mbaduku clay and 28 and 38 for Edda clay. The chemical analysis of the original kaolin showing 28.05 wt % of$A1_2O_3$; compared to SiO_2 (55.53 wt %).

The chemical characteristics of the dried residue for both showed a reduction of the alumina content to as much as between 72% and 76% for Mbaduku clay and Edda clay respectively.

V. CONCLUSION

This work studied the hydrothermal processing of rick kaolin clays from Mbaduku clay and Edda clay with a view of synthesizing a silica rich residue as a precursor material in the synthesis of Zeolites.

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