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CHARACTERISATION OF BANA KAOLIN AS ZEOLITE-SODALITE PRECURSOR

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ABSTRACT

This research aimed to assess the physicochemical properties of Bana/Niger Kaolin for its application as a zeolite-sodalite precursor. First, X-Ray Fluorescence, X-Ray Diffraction and thermogravimetric and differential thermal techniques were used to carry out the characterization of Bana Kaolin. To prepare metakaolin, the kaolinitic clay was subjected to calcination at 650°C for 3 hours. The gel resulted from alkaline treatment with 3 mol/L sodium hydroxide conducted at 50°C for one hour on the calcined product. Following this, the obtained gel underwent crystallisation at 110°C for 4 hours. To remove alkali, the resulting products were washed using distilled water. After drying, the resulting zeolite (sodalite) was characterized. It was found that the major phases in the raw clay sample are kaolinite and quartz, with minor elements found through chemical analysis. The high proportions of Silicon (52.17%) and Aluminum (32.8%) correspond to the major peaks of quartz and kaolinite found in mineralogical analysis results. The resulting synthesis product consisted of a mixture of sodalite and quartz.

Keywords: Kaolin; Physicochemical Properties; Zeolite; Sodalite; Synthesizing.

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INTRODUCTION

The global kaolin production was approximately 47.59 million tons in 2025 and is expected to reach 58.49 million tons by 2030. The growing demand for kaolin in industries such as paper, ceramics and zeolite requires the identification of new kaolin deposits with suitable properties.[1,2] Zeolites are versatile materials with a wide range of applications, including environmental protection, enhancing petrochemical products, and petroleum refining.[3] Zeolite, in various forms, including types A, Y, P, cancrinite and sodalite, can be synthesized using either chemical precursors or natural kaolin.[4–6] Compared to other zeolites, sodalite exhibits high stability. Sodalite zeolite is a highly crystalline, porous aluminosilicate that is used as an efficient catalyst and adsorbent.[3] The synthesis of sodalite can involve various chemical precursors[1,4,5,7] The synthesis process involving chemical precursors yields pure products, but it is expensive, highlighting the need for affordable materials available in large quantities, like kaolin. Kaolin is a clay mineral in which kaolinite is the predominant mineral phase. The presence of different minerals in assorted proportions may influence the properties of this clay.[8] The characterization of each raw kaolinic clay is therefore essential for a better understanding of its properties and for its use. Kaolin, which has not been previously characterized or exploited for industrial purposes, has been discovered in the Bana commune of Niger. The characterization and the application of this kaolin in sodalite synthesis are necessary to determine its qualities and potential uses. If the deposit is found to be of high quality, it will attract investment and stimulate economic growth for the country. The present work aims to develop an easy and cheap method for synthesizing sodalite zeolite using natural kaolin from Bana village. First of all, the elemental, the mineralogical compositions and the thermal behaviour of this kaolin were determined. The resulting zeolite structure and morphology were also examined.

EXPERIMENTAL

Sampling, preparation and Characterisation of Bana kaolin

The studied clay material was collected from Bana village near Dallol Foga (11°59'05"N,3°32'25"E) in Niger. To avoid contamination, the samples were stored in labelled bags. Samples were also thoroughly documented (GPS coordinates, photos (Fig.-1)). To remove adsorbed water, the collected material was sun-dried for 24 hours. Then, the dried material was crushed and passed through a mesh to obtain particles with an average diameter of 50 microns. The physicochemical properties of Bana kaolin, the resulting metakaolin and synthesized sodalite were investigated using several instrumental techniques: The chemical composition of this kaolin, including major and minor elements were determined using the X-ray fluorescence (PanAlytical serie axios Phillips). The thermal stability and mass loss of the raw kaolin were assessed using the Thermogravimetric Analysis. The mineralogical and crystal structure of Bana kaolin, the resulting metakaolin and synthesized sodalite were investigated using the Rigaku Miniflex X-Ray Diffraction. The Goniomer is of Braggs Brentan geometry with a 150 mm radius, and has a measurement range 2 theta of 2° to 145°. The x-ray source is a copper anode (λ cu kα=1.5418 Å).

Fig.-1: Samples Collected from Bana kaolin Deposit

Purification of Bana Kaolin

The process of purifying kaolin may differ according to the quality of the material and its proposed application. The kaolin from Bana has been purified according to the following steps. First, free silica was removed by sieving up to 50 µm from the ground clay. The 2-µmm particle size phases were obtained by siphoning the first phase using demineralized water and 1M sodium chloride solution (NaCl). The goal of this treatment is to eliminate the free silica by enhancing the density of water. According to Archimedes' principle, raising the water density aids in keeping clay particles suspended. The non-decanted portion of 2 liters tube was recovered and then dried in an oven.

Synthesis of Sodalite-Zeolite

Based on the thermogravimetric and differential thermal analyses (TGA-DTA) (Fig.-3), metakaolin was prepared by calcining 20g of kaolin at 650°C for 3h. XRD analysis was used to determine the mineralogical composition of the calcined product. The product obtained is heated at 80°C using demineralized water for 3h. A mechanism for sodalite synthesis was proposed using metakaolin and NaOH via the hydrothermal reaction method. The gel resulted from alkaline treatment with 3 mol/L NaOH conducted at 50°C for one hour on the calcined product. Following this, the obtained gel underwent crystallization at 90°C for 3 hours. After reaction, the products were filtered and washed with distilled water to eliminate excess alkali. After drying, the sodalite mineralogical composition and morphology were determined using XRD and Scanning Electron Microscopy (SEM).

RESULTS AND DISCUSSION

Bana Kaolin Oxide Percentage

The oxide percentage of the Bana kaolin is summarized in Table-1. The raw material is composed mainly of silica and alumina oxides. The calculated silica and alumina oxide percentage ratio of 1.59 for the kaolin sample is near the commercial kaolin ratio (1.69).[8] This ratio corresponds to the white kaolinitic clay level with high silica oxide.[10] The analyzed kaolin exhibits high values of SiO_2 and Al_2O_3 that align well with the standards for zeolite. Besides silica and alumina, other minor elements such as hematite

Fe_2O_3, calcium CaO, titanium TiO_2 oxides and various other trace elements are present in Bana kaolin at varying levels. These are manganese oxide, zirconium dioxide, vanadium pentoxide and sulfate trioxide, which are present in very low concentrations. These oxides are commonly associated with kaolinitic clay.[1,11] The results also showed that this kaolin contains chlorine and sodium, chemical compounds that are part of the composition of sodalite. Chlorine and sodium are crucial components required for the synthesis of sodalite zeolite from kaolin. These components are very important to the stability of sodalite structure and influence its physicochemical properties.[12] In addition to chlorine and sodium, this kaolinitic material also contains TiO_2 and Fe_2O_3. These oxides significantly influenced the formation and development of crystalline phases during thermal treatment.[13] Many studies revealed that even an iron ion concentration of just 60 ppm yields significant sodalite catalytic performance.[14]

Table-1: Oxide Composition of the Studied Clay

Oxide	Al2O3	SiO_2	V_2O_5	Fe_2O_3	MnO	CaO	SO_3	TiO_2	Cl	ZrO_2	Na_2O	SiO_2/ Al_2O_3
Percentage (%)	32.8	52.17	0.30	4.79	0.23	1.77	0.53	5.54	0.85	0.22	0.14	1.59

Mineralogical analysis of Bana Kaolin

The crystalline phases in the raw were investigated using X-Ray Diffraction (XRD) analysis. As shown from Fig.-2, the kaolinitic clay from the Bana village phase has a triclinic structure with unit-cell parameters of a, b and c, respectively, 5.1490 (Å), 8.9335 (Å) and 7.3844 (Å), α= 91.9300°, β= 105.0420°, γ= 89.7910° and density = 2.62 g/cm^3. The kaolin sample, as shown in the XRD pattern (Fig.-2), predominantly features the kaolinite phase, with major peaks appearing at 2θ angles of 12.36, 19.91, 20.37, 24.89, 34.88, 35.95, 38.46, 39.23, and 45.52. The obtained values are in good agreement with the literature values.[14] As indicated by XRD analysis, Bana kaolin has its major peaks associated with kaolinite(k) and quartz (q). The high proportion of Fe_2O_3 observed in the chemical characterization can be explained by its isomorphic substitution within the clay mineral structure, which was not detected with XRD.[15] The XRD pattern also shows a high amount of free silica observed in the oxide's composition of Bana kaolin (Table-1).

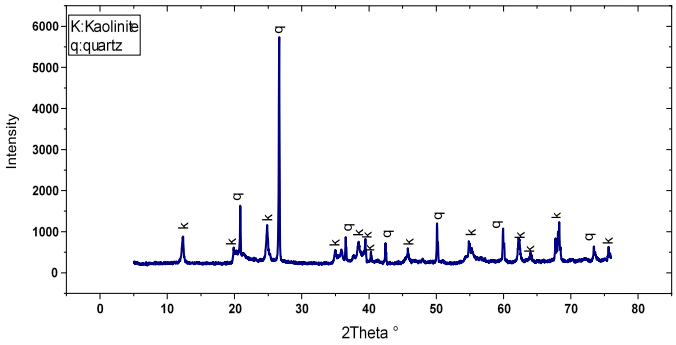

Fig.-2: Mineralogical Diagrams of the Raw Clay

Thermogravimetric and Differential Thermal Analyses (TGA/DTA)

During the preparation of zeolite, kaolin is treated by calcination to produce metakaolin, which is an amorphous aluminosilicate material. Among the numerous methods available for analyzing the dehydroxylation of kaolin, thermal analysis techniques are still significant tools.[16] During the kaolinite transformation to metakaolin, the thermal analysis helps to understand the effect of temperature on the kaolin structure and properties. The TGA curves obtained for the kaolin sample heated from 30 to 950 °C are shown in Fig.-3. Thermal characterization of Kaolinic clay samples showed two endothermic transformations identified as water loss and mass loss related to dehydroxylation of kaolinite.[17] The first endothermic mass loss between 30 and 300 °C corresponds to the evaporation of water. Mass loss related to dehydroxylation of the Kaolinite begins around 350 °C. This phenomenon is well-known; it is linked to the loss of hydroxyl groups from kaolinite during the formation of metakaolin. The breakdown of kaolinite into metakaolin is still complete around 650-700 °C. Beyond 400 °C, the mass of the sample

continues to decrease until 680 °C. The weight loss percentage linked to the dihydroxylation highlights the good phyllosilicate content of this material.

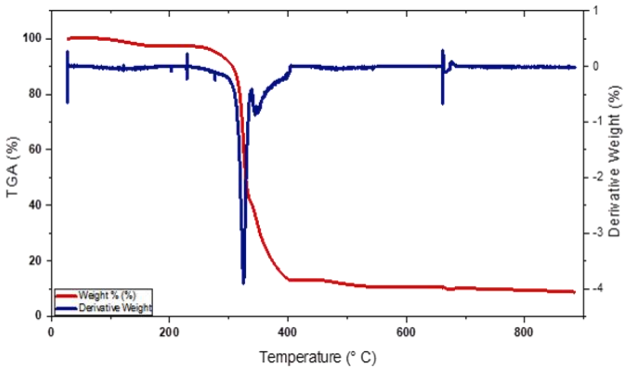

Fig.-3: Thermal Analyses for Bana Kaolin

X-ray diffraction Analysis of the Metakaolin and the Synthesised Sodalite

The mineralogical analysis results of Bana kaolin, the synthesized metakaolin and sodalite zeolite are shown in the diagram of Fig.-4. The composition of Bana kaolin includes kaolinite and quartz. When subjected to purification and calcination, these peaks disappeared, leaving behind a uniform amorphous band of metakaolin detectable by XRD. The peaks of anatase and quartz are still detected, since much higher temperatures than 650 ºC would be necessary to destroy the structure of these minerals.[18] The main peaks of sodalite are located at 2θ =14.00°, 24.43°, 31.79°, 34.86°, and 43.01°(Fig.-4). These peaks are in good agreement with the literature values.[4]

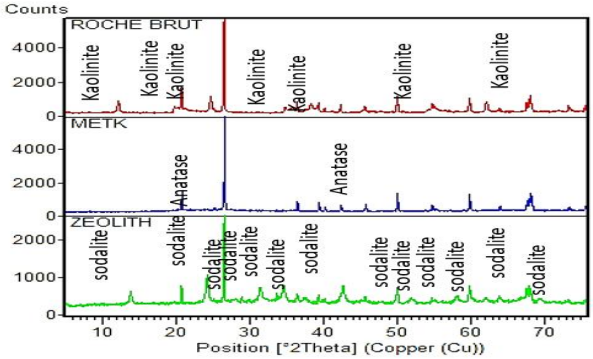

Fig.-4: Mineralogical Analysis of the Metakaolin and the Synthesized Sodalite

Zeolite Morphology

The morphology images of the synthesised sodalite are shown in the Fig.-5. The synthesis Product, from metakaolin calcined at 650 °C, has a rounded format, as“wool skeins”, characteristics of polycrystalline clusters of sodalite.[14,19]

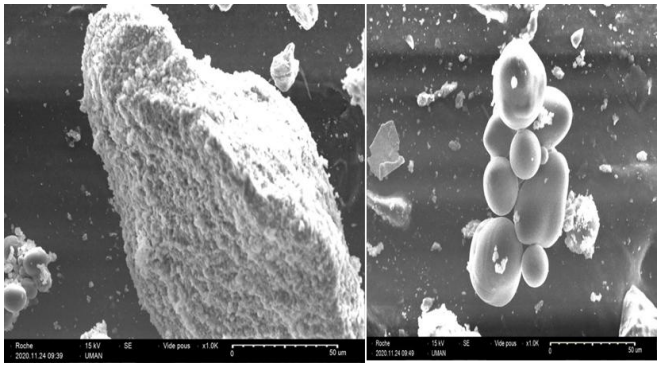

Fig.-5: SEM Images of the Synthesised Zeolite

CONCLUSION

Zeolite sodalite was synthesized using natural material not yet used in industry (kaolinic clay from Bana village). This clay from Bana village was characterized to determine its oxide, mineralogical compositions and thermal behavior. The obtained results correspond to a kaolinitic clay rich in aluminium and silica (52.17 of SiO_2 and 32.8 of Al_2O_3). In addition to these major oxides, this kaolinite also contains minor oxides favorable to the synthesis of sodalite zeolite. These include chlorine, sodium, iron and titanium. The XRD analyses showed that quartz and kaolinite were the crystalline phases detected. Based on its mineral and chemical composition and the synthesized product, this clay can be used as a kaolin source with applications in zeolite synthesis. Research will be conducted to suggest industrial uses for this kaolin in the manufacture of various types of zeolites (A, Y, P).

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CONFLICT OF INTERESTS

No conflict of interest is declared by the authors.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-9: Industry, Innovation, and Infrastructure.* All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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