

## COMPARATIVE STUDY OF ACTIVATED KAOLIN FROM AHOKO CLAY AND LEMU CLAY

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<https://doi.org/10.62050/faic2024.bop24.008>

### ABSTRACT

A major issue facing the globe today is climate change and global warming, which are caused by the use of fossil fuels in transportation and other industries that generate large amounts of CO<sub>2</sub> into the atmosphere. These CO<sub>2</sub> gases is the major greenhouse gas which causes damages to health and environment. In this work, Ahoko kaolin and Lemu kaolin were activated and compared as an adsorbent for CO<sub>2</sub> adsorption from the atmosphere. Both clays were activated using acid in order to improve the adsorbent. The BET analysis was carried out to determine the surface area, pore volume and pore size of both samples before and after activation. The result shows that the kaolin from Ahoko has a larger surface area with 82% increment of the activated samples compared to the raw samples. Also the activated Lemu kaolin increases by 132% compared to raw Lemu kaolin. The SEM analysis was also done before and after the activation of both samples to identify and interpret the surface morphology of the adsorbent (Ahoko kaolin and Lemu Kaolin). The FT-IR revealed that both samples have characteristic peaks associated with hydroxyl group, silicate group and oxides group. The research shows that the activated kaolin shows a better result compared to the raw kaolin from both samples. It also shows that activated kaolin from Lemu will likely capture CO<sub>2</sub> more, compared to activated kaolin from Ahoko.

**Key Words:** Clay, Activated Kaolin, Kaolinite, Climate Change, Greenhouse Gas.

### 1.0 Introduction

In some years back, there was a spike in air pollution due to an intensive use of fossil fuels in industry, transportation and building sector. These persistent increases are the main contributor to acid rain, climate change and global warming. The environment and human health suffer as a result of these effect. According to studies, using fossil fuels produces greenhouse gas emissions which are the cause of climate change and global warming. (Hileman, 2018). Most greenhouse gasses released into the atmosphere in significant quantities are assumed to be carbon dioxide (CO<sub>2</sub>) (Akpassi, 2021).

How to capture CO<sub>2</sub> reasonably and efficiently to achieve carbon neutrality is an urgent question. There are several ways which are; reducing the use of fossil fuels, switching to non-carbon emitting-resources i.e. renewable energy and CO<sub>2</sub> capture and sequestration. CO<sub>2</sub>



capture is the most effective way to lower atmospheric CO<sub>2</sub> emissions and is discussed in this report. Solid adsorption is favored above other technologies for CO<sub>2</sub> capture due to low energy requirements, low capital and operating cost, together with limited secondary waste generation (Rashidi *et al.*, 2013). One of the most common natural materials is clay mineral, which can be found for a reasonable price and has excellent mechanical and chemical stability. One type of clay mineral is kaolinite, which is made up of 1:1 ratios of octahedral hydroxide sheets and tetrahedral silicate sheets (Neeraj and Chandra, 2021).

Due to its inactive nature and low specific surface area, natural kaolinite has been shown to have a far lower adsorption capacity than activated carbon, a typical adsorbent (Qian *et al.*, 2019) Thus, for kaolinite to be suitable for practical usage, its adsorption qualities need to be improved. One of the many activation techniques is physical change, which can be done by heating or microwave treatment. The most popular physical modification is called a "thermal treatment," which modifies a substance's crystalline structure or chemical composition through temperature changes. Heating clay minerals to a high temperature can change their content and structure (Osuna *et al.*, 2023). Thermal treatment can significantly alter a number of physiochemical characteristics, including swelling, strength, particle size, catalytic activity, and mineralogy (Onal *et al.*, 2007).

Chemical modification is typically accomplished by changing surface functional groups, surface area, and structure by the use of acids, bases, and organic compounds. The best method for activating kaolinite is to treat it with acid and alkaline (Hussin *et al.*, 2011). Belver *et al.* (2002) conducted a systematic study on the chemical activation of kaolinite in both acidic and alkaline environments. The findings indicate that kaolinite treated with alkaline typically exhibits a relatively low surface area. On the other hand, it has been observed that acid treatment is advantageous for activating kaolinite to enhance its textural qualities, specifically pore volume and surface area for CO<sub>2</sub> uptake.

## 2.0 Materials and Methods

### 2.1 Materials

Kaolin from Ahoko in Kogi State and Lemu in Niger state, Hydrochloric acid, distilled water was gotten from Chemical Engineering Laboratory, Federal University of Technology, Minna, Niger state, Nigeria.

### 2.2 Sample preparation

The activated kaolin used in this work was prepared and made available. Ahoko clay was transported from Ahoko in Kogi State while Lemu clay was transported from Lemu in Niger State to the laboratory. Stones and other unwanted materials were removed from the clay samples. The clay samples were grounded in a ceramic mortar. Fifty grams (50 g) of the grounded clay sample was weighed into a 500 ml capacity beaker and water was added to the capacity of the beaker. The sample was stirred with a glass rod and the sand and other quartz materials was allowed to settle within 5 minutes. The suspended clay in the supernatant was decanted into other beaker. This will be allowed to settle for 24 hours (hrs). After 24hrs, the settled kaolin was obtained by decanting the supernatant. The kaolin sample was dried in an oven for 1hr at 105°C The dried kaolin sample was activated using 2 M solution of hydrochloric acid. One hundred milliliters (100 ml) of 2 M solution of hydrochloric acid was added to 50 g of kaolin in a beaker. The mixture was stirred for 1 hour and was left for digestion to take place for 24 hrs. The mixture was filtered and washed thoroughly with distilled water until the washed water become neutral to litmus paper. The activated kaolin was dried in an oven at 105°C for 1hour after which it was calcined at 450°C for 3 hours.



### 3.0 Results and Discussion

#### 3.1 Brunauer-Emmet –Teller (BET) Analysis

The BET analysis was carried on the raw and activated sample of both Ahoko and Lemu kaolin to identify the physical properties of the adsorbents which are the BET surface area, pore volume and pore size. Table 1 shows that the BET surface area for raw Ahoko kaolin increases from 491.0m<sup>2</sup>/g before activation to 893.3 m<sup>2</sup>/g after activation which shows 82 % increment, while the kaolin from Lemu increases from 422.8 m<sup>2</sup>/g before activation to 983.5 m<sup>2</sup>/g which shows 132 % increment. Similarly, the pore volume for Ahoko kaolin also increases by 81.95 % after activation while that of Lemu kaolin increases by 132 %. The pore size of kaolin from Ahoko also increased by 11 %.

**Table 1: BET results for the adsorbent**

Samples	BET Surface Area(m <sup>2</sup> /g)	Increment (%)	Pore volume (cc/g)	Pore size (nm)
Raw Ahoko	491.0		0.1745	5.839
Activated Ahoko	893.3	82	0.3175	6.503
Raw Lemu	422.8		0.1502	6.214
Activated Lemu	983.5	132	0.3495	3.866

The analysis result showed that the values for the two activated samples were higher after activation compared to the raw sample. This means that activation process enhances pore development and produces new pore, which therefore increased their BET surface area. Although the properties of both samples when activated improved but that of activated Lemu has higher properties than that of activated Ahoko except for the pore size. Since both were activated and calcined at the same temperature probably some molecules of the activating agent could not be completely removed thereby blocking and reducing the pore size of the activated Lemu.

#### 3.2 Scanning Electron Microscopy (SEM) Analysis

The SEM images are used to identify and interpret the surface morphology of the adsorbent before and after activation. From Figure 1 it can be observed that the activated form of Lemu clay shows more of flakes like shape as compared to the raw clay. This could be attributed to the heat treatment given to the clay and subsequent chemical activation which open up more pore in the clay particles. This means more pore will be available per m<sup>2</sup> of the sample for the attraction of the CO<sub>2</sub> particles from the surrounding. It could also be observed that the raw kaolin looks more condense in particles than the calcined and activated kaolin. The hollow and cylindrical looks of the activated kaolin is good for intra-particle diffusion during adsorption process. The white patches which are likely to depict the presence Alumina and silicate are more pronounced in the activated kaolin. This observation is supported with the result obtained in EDX analysis which shows the presence of Ca and Al in high percentage (Figure 1 c and d).

From Figure 2 it can be seen that the microstructure of the raw clay exhibits a close packing of particles with minimal void spaces. This dense arrangement is a characteristics of untreated kaolin clay whereas from Figure 2b, it can be seen that the activation has led to a significant alteration in the microstructure, evidenced by the formation of voids and an increased



interparticle distance. This creation of new pores suggests the removal of impurities and the development of a more reactive surface.

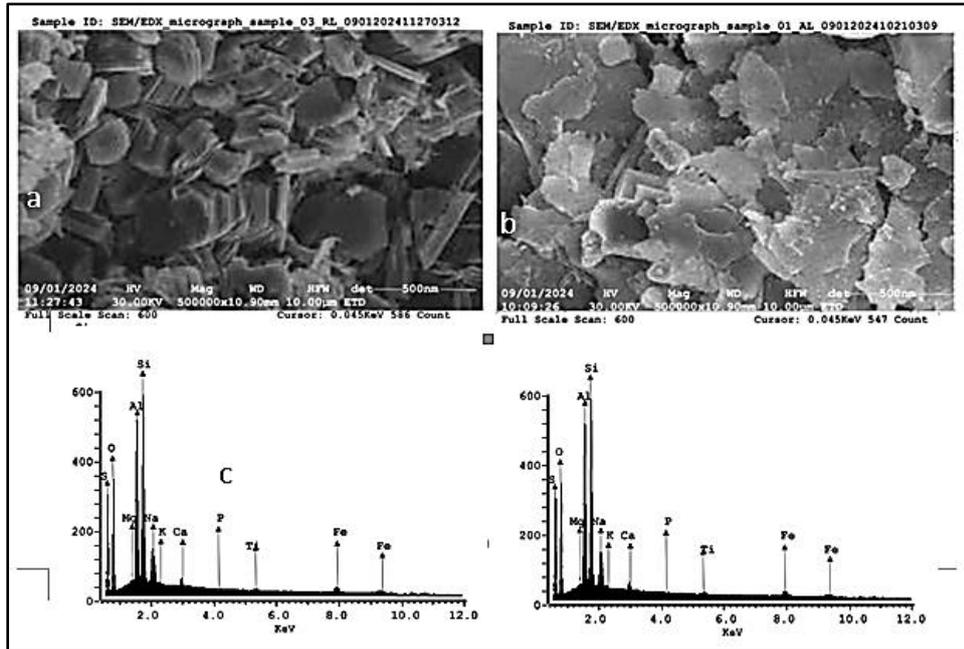


Figure 1: (a) Raw Lemu Kaolin Clay (b) Activated Lemu Kaolin clay

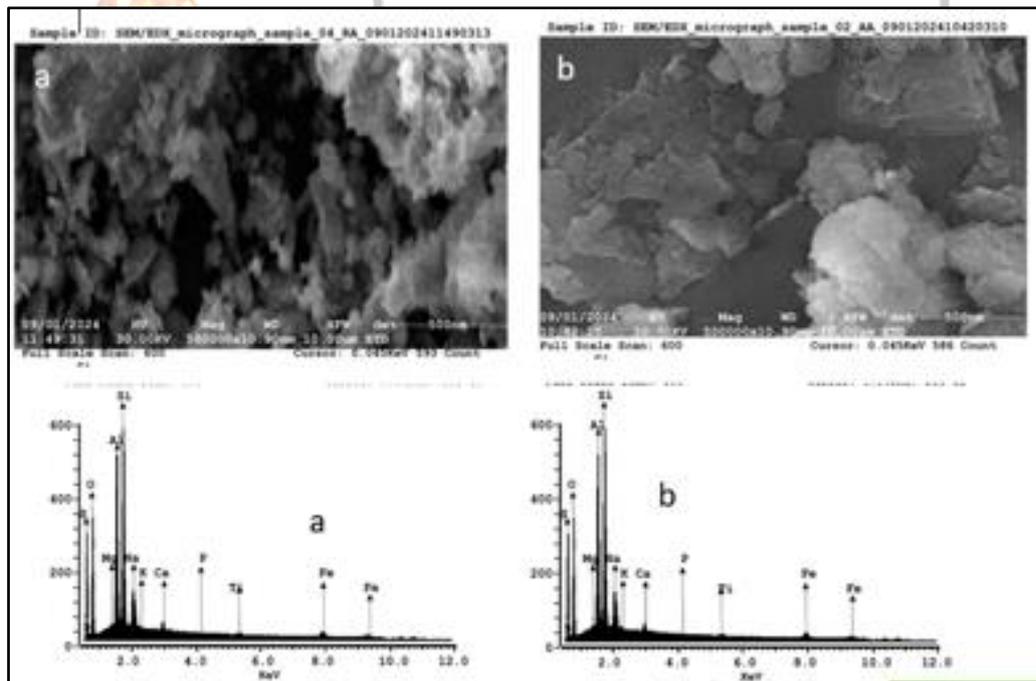


Figure 2: (a) Raw Ahoko Kaolin Clay (b) Activated Ahoko Kaolin Clay

Also, the broadening of the particle size distribution in the activated Ahoko clay as compared to the raw clay, the activation process has led to the generation of smaller particles, contributing to the increased surface area. The observed changes in the surface morphology and



microstructure suggest that the clay activation process has successfully modified the physical characteristics of the clay.

### 3.3 Fourier Transform InfraRed (FT-IR) Analysis

The FT-IR analysis of Raw Lemu kaolin and Activated Lemu kaolin reveal notable similarities in their compositions, indicating the mineralogical nature of Lemu kaolin. Both raw and activated Lemu kaolin exhibit characteristic peaks associated with hydroxyl groups, such as Al-OH stretching vibrations at peak  $3204.62\text{cm}^{-1}$  for raw Lemu and  $3104.25\text{cm}^{-1}$  for activated Lemu kaolin with transmittance of 48 % and 57.31% and O-H deformations which helps in the capture and retention of  $\text{CO}_2$  on the clay surface. Silicates features are obvious in both samples, with peak 2500.39 2399.05 and 1310.78 for raw Lemu kaolin with transmittance of 72 %, 76 % and 72 % and peak 2403.16 and 692.19 with transmittance of 70 % and 74 % corresponding to Si-O stretching and in plane Si-O stretching vibrations.

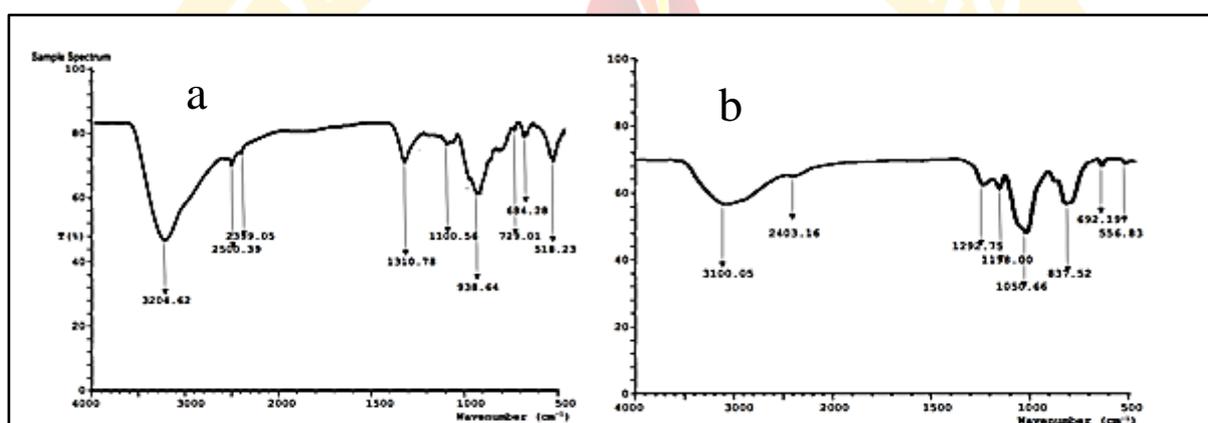


Figure 3: FTIR (a) Raw Lemu Kaolin Clay (b) Activated Lemu Kaolin

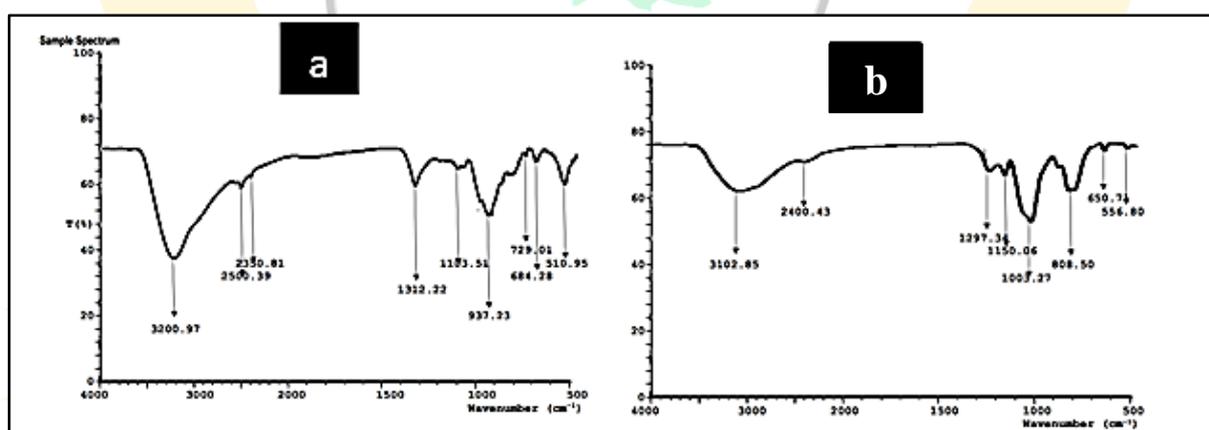


Figure 4: (a)Raw Ahoko Kaolin Clay (b)Activated Ahoko Kaolin Clay

This functional group can participate in chemical interaction with  $\text{CO}_2$  molecules during adsorption. Additionally, oxides, particularly iron and titanium are indicated by peaks  $518.23$  for raw Lemu and  $556.83\text{cm}^{-1}$  for activated Lemu kaolin related to Fe-O,  $\text{Fe}_2\text{O}_3$ , Ti-O and Si-O-Al stretching vibrations in both Raw Lemu and Activated Lemu. Which influence the crystal structure and properties of the Clay. However, distinct changes are observed in activated Lemu kaolin compared to Raw Lemu kaolin. Activated Lemu kaolin generally displays higher

transmittance values across peaks, suggesting a more pronounced activation or modification process. These variations in transmittance implies alteration in the molecular structure, conforming the efficacy of the activation process in transforming the raw Lemu kaolin into its activated form. The comparative FTIR analysis thus highlights both the commonalities and modification between raw Lemu kaolin and activated Lemu kaolin, offering valuable insights into the mineralogical changes induced by the activation treatment.

For Figure 4 The Fourier-Transform Infrared (FTIR) analysis of Raw Ahoko kaolin and Activated Ahoko kaolin provides valuable insights into the alterations induced by the activation process. In Raw Ahoko clay, the presence of hydroxyl groups is indicated by the Al-O-H stretching vibration peak at  $3200.97\text{ cm}^{-1}$  and the O-H deformation peak at  $937.23\text{ cm}^{-1}$ . Silicates features are identified through peaks associated with Si-O stretching and in-plane Si-O stretching vibration ( $2500.39, 2350.81, 1312.22\text{ cm}^{-1}$ ) along with Si-O planar stretching ( $1103.51\text{ cm}^{-1}$ ) and Si-O perpendicular vibrations ( $684.28\text{ cm}^{-1}$ ). Additionally, the presence of oxides is suggested by the peak  $510.95\text{ cm}^{-1}$ , corresponding to Fe-O,  $\text{Fe}_2\text{O}_3$ , Ti-O, and Si-O-Al stretching vibrations. In Activated Ahoko kaolin, the hydroxyl-related peak at  $3102.85\text{ cm}^{-1}$  and  $808.50\text{ cm}^{-1}$  indicates the changes in the hydroxyl content during activation. Silicate-related peaks ( $2400.43, 1297.34, 1150.06, 1003.27, 650.71, 556.80\text{ cm}^{-1}$ ) persist, suggesting the presence of silicates in both samples but with altered vibrational characteristics in Activated Ahoko. The Oxide-related peak at  $556.80\text{ cm}^{-1}$  is more pronounced in Activated Ahoko Kaolin, indicating an increase in oxide content during activation. These findings collectively signify that the activation process modifies the molecular structure of Ahoko kaolin, influencing the composition of hydroxyl groups, silicates and oxides.

#### 4.0 Conclusion

The study concludes that the activation process significantly enhances the adsorption properties of kaolin from both Ahoko and Lemu. Specifically, the activated kaolin from Lemu shows a higher increase in BET surface area and pore volume compared to the activated kaolin from Ahoko, indicating a better potential for  $\text{CO}_2$  capture. Acid activation significantly enhanced the adsorption properties of kaolin, with activated Lemu kaolin exhibiting a higher increase in BET surface area (132%) compared to activated Ahoko kaolin (82%). SEM analysis revealed improved porosity and morphological modifications that favor  $\text{CO}_2$  capture, while FT-IR confirmed structural changes in functional groups, supporting enhanced adsorption potential. Although both clays showed improved properties post-activation, Lemu kaolin emerged as the superior candidate for  $\text{CO}_2$  adsorption due to its greater surface area and pore volume. These findings highlight the potential of locally sourced kaolin as an efficient, cost-effective adsorbent for environmental applications. Overall, the research demonstrates that activated kaolin, particularly from Lemu, is more effective for  $\text{CO}_2$  adsorption than raw kaolin.

#### Conflict of Interest

The authors declare no competing interest.

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