EFFECT OF TEMPERATURE ON BASE- ACTIVATED CLAY FROM GEGU-EBGA REGION IN KOGI STATE

BY

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CERTIFICATION

This is to certify that this project research was carried out by **UMORU ABDULHAKEEM**, Matriculation number PSC2008014 in the Department of chemistry, Faculty of Physical sciences, University of Benin, Benin City, Nigeria.

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PROF. E.E.I. IRABOR (Head of Department)	DATE

DEDICATION

This project work is dedicated to God Almighty through whom the success of this work became a reality, Alhamdulillah.

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TABLE OF CONTENT

ABSTRACT

Natural clay minerals are abundant and versatile, offering a broad range of applications across various industries due to their unique physical and chemical properties. Their adsorption capacity and catalytic capabilities were enhanced through specific treatments. This study examined the effect of temperature on base-activated clay from the Gegu-Egba region, Kogi State, Nigeria. Clay minerals, valued for their high surface area and structural properties, were widely used in catalysis, adsorption, and refining. In this research, the Gegu-egba clay samples were treated with 30% NaOH and heated at 200°C (F3A) and 400°C (F3B) to assess structural and chemical changes. Characterization techniques, including X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), energy-dispersive X-ray spectroscopy (EDX), and Brunauer-Emmett-Teller (BET) analysis, were employed to evaluate their structural, morphological, and textural properties FTIR analysis indicates progressive dehydroxylation, with the disappearance of free hydroxyl groups and shifts in Si–O and Al–O–Si vibrations, suggesting kaolinite transformation into metakaolin. XRD results confirm a decline in kaolinite content (23% to 3.1%) and increased feldspar influence, supporting amorphization at 400°C. SEM-EDX analysis shows increased porosity and redistribution of elemental composition, notably a decrease in Si and Ti with a rise in Al content. BET surface area decreases from 249.577 m^2/g at 200°C to 214.149 m^2/g at 400°C, indicating structural densification. These findings emphasized the role of optimized thermal treatment in enhancing base-activated clay for industrial applications such as catalysis, adsorption and wastewater treatment.

CHAPTER ONE

1.0 INTRODUCTION AND LITERATURE REVIEW

1.1 INTRODUCTION

Clay minerals constitute a diverse class of naturally occurring hydrous aluminosilicates with layered structures, which account for their distinctive physical and chemical properties. These include high surface area, cation exchange capacity, and plasticity when mixed with water—features that make clays highly versatile materials for numerous applications. Clays have been widely utilized in ceramics, construction, and agriculture since time immemorial because they are widespread, low-cost, and simple to process. Recently, their applications in environmental cleanup, wastewater treatment, and catalysis have attracted growing attention, especially as industries look for sustainable and environmentally friendly options (Murray, 2007).

Probably the most intriguing aspect concerning clay minerals lies in their eventual chemical and thermal modifications, vastly improving their functionality. For instance, acid or base activation significantly raises their surface area and increases porosity; thus, a more effective functioning as adsorbents or catalysts in general chemical reactions will result (Adekola *et al.*, 2016). The same happens with thermal treatment, which can modify their structural and chemical properties, such as dehydroxylation and phase transformation, further enhancing their catalytic performance. Such modifications enable clays to be tailored for specific applications in the petrochemical industry, wastewater treatment, or as catalysts in organic synthesis. According to Adams & McCabe (2006), The increasing interest in clay minerals as catalysts is due to their natural abundance, low cost, and environmental compatibility. Unlike synthetic catalysts whose syntheses often involve expensive and toxic reagents, clays are an environmentally benign alternative and concur with the principles of green chemistry. Clay, which is found in soil is one of the most popular and well-known heterogeneous catalysts utilized in chemical reactions. Clay and clay modified catalysts have been widely used to catalyze various types of organic reactions such as esterification reaction, isomerization reactions, oxidation of alcohols, dehydrogenation, epoxidaton and several more. Due to its favorable properties such as low cost, thermal stability, selectivity, large surface area, ion exchange capacity, easily separated, as well as environmentally friendly, (kaur and kishore. 2012).

They are naturally occurring aluminosilicates having sheets structures. They are frequently utilized as components of carbonless copying papers as well as catalysts and catalyst support in alkylation, dimerization, and polymerization operations. A few clays, like bentonite, kaolin, ball clay, antopulgite, ball clay, and montrimolinite, have been known for a while, but there are not many examples of them that have been characterized using cutting-edge methods. (Venkatathri, 2006)

This work reports the effect of temperature on base-activated clay sourced from Gegu-Egba, Kogi State, Nigeria. The geological environment is abundant with deposits of clay minerals that are largely underutilized despite their potential. This research aims at optimizing such clays to achieve the desired catalytic properties by investigating thermal activation and looking for their application in catalysis, which, besides the advancement of scientific knowledge, may serve local economic interests.

In sum, these clay minerals are industrially fundamental as well as find much potential use in modern applications, especially catalysis. Their ability to be tailored with regard to chemical and heat treatment provides a key resource for sustainable technological development. Therefore, this work was done to reveal the hidden opportunities of Nigerian clay minerals, opening the way for the creation of innovative and environmentally friendly solutions in catalysis.

1.1.1 BACKGROUND STUDY

Kaolinitic clay, which is mainly made up of kaolinite ($Al_2Si_2O_5(OH)_4$), is a naturally occurring mineral that is widely available in the Gegu-Egba region of Kogi State, Nigeria (Olasupo & Sujatha, 2020). Kaolinite is a 1:1 layered aluminosilicate mineral, distinguished by its alternating tetrahedral silica (SiO_4) and octahedral alumina (AlO_6) sheets. This framework provides kaolinite with special properties, including low cation exchange capacity (CEC) and good thermal stability, rendering it an ideal material for various industrial applications (Bergaya et al., 2006). The Gegu-Egba kaolinitic clay, specifically, has been widely investigated for its potential applications in water treatment, paper coating, ceramics, and catalysis (Adekola et al., 2016).

Among the most important uses of kaolinitic clay is catalysis. Nonetheless, raw kaolin may need to be activated for its surface characteristics and reactivity to be increased for industrial application. Activation by base, wherein the clay is treated with basic solutions like sodium hydroxide (NaOH) or potassium hydroxide (KOH), is the typical procedure adopted. This not only generates novel active sites on the surface of the clay, but also augments its surface area and develops porosity, thus improving the catalytic activity (Adekola *et al.*, 2016). For instance, base-activated kaolinitic clay has been effectively used in chemical reactions like hydrocarbon cracking, esterification, and transesterification (Adams & McCabe, 2006).

Thermal treatment is the other important factor that affects the catalytic behavior of kaolinitic clay. Upon heating to high temperature, kaolinite undergoes structural modifications like dehydroxylation and phase changes, which could enhance its catalytic activity greatly (Murray, 2007). For instance, heating kaolinite above 500°C converts it into metakaolin, a highly reactive amorphous phase with enhanced catalytic properties (Bergaya *et al.*, 2006). Metakaolin is particularly useful in industrial processes such as biodiesel production and the synthesis of fine chemicals.

The combination of base activation and thermal treatment offers a promising approach to optimizing the catalytic performance of kaolinitic clay. By precisely adjusting the parameters of activation, for example, the concentration of alkaline solution and thermal treatment temperature, it is possible to make the clay's properties fit special industrial demands. For instance, research has established that thermally treated base-activated kaolinitic clay at optimum temperatures has enhanced catalytic activity for reactions like cracking of heavy crude oil and esterification of fatty acid (Adekola *et al.*, 2016).

The availability of kaolinitic clay in the Gegu-Egba area has great potential for economic growth in Nigeria. By utilizing this natural resource and applying advanced activation techniques, the local industries will be capable of reducing their reliance on imported catalysts and manufacturing value-added products. This could create employment opportunities, technological advancement, and environmentally friendly development within the region. However, further studies need to be carried out in order to tap into the complete potential of Gegu-Egba kaolinitic clay properties and its activation processes. For example, upcoming research can be directed towards studying the influence of various alkaline solutions, activation times, and heat treatment protocols on the catalytic activity of clay. Besides, it is necessary to take into consideration the environmental impacts of such activation protocols so that they are aligned with green approach philosophy.

In summary, Gegu-Egba region Kogi State, Nigeria kaolinitic clay is a valuable natural material with high potential for industrial use, especially in catalysis. By employing base activation and thermal treatment, the surface characteristics and catalytic behavior of the clay can be improved so that it is amenable to a variety of chemical processes. Optimization of these activation procedures is one area in which great potential exists for the utilization of this vast resource to spur industrial development and economic growth in Nigeria. Future research should focus on refining these methods and exploring new applications to fully unlock the potential of Gegu-Egba kaolinitic clay in the global market.

1.1.2 JUSTIFICATION OF STUDY

Thus, research on temperature's effect on clay treated with alkaline solutions will be significant, since heating greatly alters the physical structure, chemical behavior, and catalytic ability of clays. Naturally occurring water, aluminum, and silicon minerals, clays consist of a layered structure similar to microscopic building blocks. It is these layers which provide a vast surface area allowing clay to interface with other materials. Upon heating, clays lose water molecules and hydroxyl (OH) groups that were entrapped between their layers; new spaces and active sites are therefore created on their surfaces. According to Bergaya *al et.* (2006), this feature improves the ability of clays to catalyze chemical reactions, such as heavy oil cracking into fuels or plant oil trans-esterification into biodiesel. Knowing how temperature influences these properties, industries are in a position to design more affordable, more efficient clay-based catalysts-substances that accelerate reactions without being consumed-to replace costly or polluting alternatives.

Beyond industrial applications, this work bears direct relevance to agriculture, particularly in areas like Nigeria where soils are rich in clays but usually quite underproductive. Clay's natural ability to retain and release nutrients-soil fertility-is defined by its cation exchange capacity or CEC. Heating and treating clay with alkaline solutions can make it more absorbent, acting almost like a sponge, thereby retaining potassium, calcium, and magnesium-nutrients that are important for crops (Murray, 2007). Heat-treated clay could release nutrients to plants slowly and help reduce the amount of synthetic fertilizers required. This work might unlock low-cost methods in many regions of the world to improve soil productivity and increase food output with minimal damage to farming sustainability.

The other major driving force is the environmental and economic value of clay as a catalyst. Traditional catalysts used in industries often rely on rare metals, such as platinum, or synthetic chemicals, which are costly, scarce, and environmentally damaging. On the other hand, clay is abundant, inexpensive, and nontoxic, an environmentally friendly alternative (Adekola *et al.*, 2016). For example, biodiesel production already utilizes clay catalysts, presenting a greener and cheaper method compared to conventional approaches. By optimizing how clay is heated and treated with alkaline solutions, this study is aimed at widening its use in industries, a step toward attaining global goals on the adoption of sustainable technologies.

Other environmental benefits of clay extend even further. Whereas synthetic catalysts can contaminate soil and water when they are discarded, clay is biodegradable and harmless.

Besides, clay-based catalysts have the potential to make industrial processes more energy-efficient. For example, during oil refining, modified clays help in the conversion of heavy crude oil into gasoline and diesel with less energy use, directly contributing to a reduction of greenhouse gas emissions. This efficiency contributes to world-wide efforts against climate change, while lowering production costs.

This also bridges gaps in materials science: What can be realized from the study of heat-treated clays might lead to new uses other than catalysis and agriculture, such as filtering out contaminants from wastewater and hardening sustainable construction materials. In itself, the versatility makes it a prime tool for emerging solutions in environmental cleanup, renewable energy, and advanced manufacturing.

1.1.3 SCOPE OF WORK

The scope of work entails collecting clay samples from Gegu-Egba location and activating them using 30% NaOH. Subsequently, the study will investigate the influence of temperature within the range of 200°C to 400°C on the properties of the Base-activated Gegu-Egba clay. The modified clay will be characterized using techniques such as Brunauer-Emmett-Teller (BET) analysis for surface area measurement, Fourier Transform Infrared Spectroscopy (FTIR) for functional group analysis, and Infrared Spectroscopy (IR) for structural analysis. The results will be used to draw conclusions about the suitability of the clay as a catalyst under different temperature conditions.

1.1.4 AIM AND OBJECTIVES

The aim of this work is to investigate the effect of temperature (ranging from 200°C to 400°C) on the properties of acid-activated Gegu-Egba clay, collected from Gegu-Egba region of Kogi state and activated using 30% NaOH, aiming to understand the temperature-dependent behaviour of the clay and optimize its industrial applications. To address the following aim, the objectives will be;

- To collect representative clay samples from an Gegu-Egba location and conduct preliminary characterization to determine mineralogical composition, particle size distribution, and physical properties, and to analyze impurities, organic matter, and moisture content.
- To prepare clay samples for base activation by drying and grinding to a uniform particle size, to activate the clay using 30% NaOH according to established protocols, and to monitor activation parameters to ensure consistency.
- To design experiments to subject acid-activated Gegu-Egba clay samples to controlled temperature variations within the range of 200°C to 400°C, to implement temperature-controlled conditions in the laboratory setup, and to conduct experiments using appropriate equipment.
- To perform comprehensive analysis of physical properties such as surface area, pore size distribution, and morphological characteristics using techniques like BET surface area analysis and scanning electron microscopy (SEM), and to utilize analytical methods such as X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR) to assess chemical composition and structural changes.

To collect data on changes in physical and chemical properties of acid-activated Gegu-Egba clay samples resulting from temperature variations, to analyze experimental data to identify trends, correlations, and dependencies between temperature and clay properties, and to interpret findings to elucidate the temperature-dependent behaviour of the clay and its implications for industrial applications.

1.2 LITERATURE REVIEW

1.2.1 CLAY MINERALS

Clay minerals are naturally occurring hydrous aluminosilicates, composed of layered sheets of silicon-oxygen tetrahedra and aluminum/magnesium-oxygen-hydroxyl octahedra. They generally have particle sizes <2 µm and are formed by the weathering of rocks, such as feldspar and volcanic ash (Bergaya & Lagaly, 2013). Their layered structure and high surface area enable unique properties such as cation exchange and adsorption. Common types include kaolinite (1:1 layers), montmorillonite (2:1 layers), and illite (2:1 layers with potassium ions).

These minerals have crucial applications in industry, such as catalysts and ceramics, and in the environment, such as nutrient retention in soils. Their reactivity and structure can be modified via thermal or chemical treatments, making them adaptable for uses in catalysis, pollution remediation, and agriculture.

1.2.1.1 PROPERTIES OF CLAY

Clay minerals, as a group of hydrous aluminosilicates, are characterized by their layered atomic structures and particle sizes usually less than 2 micrometers (Bergaya & Lagaly, 2013). They develop organically from the weathering of parent rocks, such as feldspar and volcanic ash, with their properties closely related to their structure. For example, kaolinite, montmorillonite, and illite—some of the most common clays—vary in layer arrangements (1:1 or 2:1 silicate-to-alumina layers) and interlayer cations, which determine their reactivity and uses (Murray, 2007). Below, we discuss their characteristic properties and practical utility.

Surface Area and Reactivity

One of the most significant properties of clay minerals is their large surface area, which is a result of their layered structure and small particle size. Smectite-group clays, i.e., montmorillonite, have surface areas of over 800 m^2/g upon exfoliation, as their interlayer spaces open up to receive water or organic molecules (Sposito, 2008). It is an important property in catalysis, where clays are used as substrates for the immobilization of reactive sites. Acid-activated montmorillonite, for instance, is used extensively in esterification reactions because of its porous nature and Brønsted acidity (Adams & McCabe, 2006). Likewise, kaolinite's modest surface area (~20 m^2/g) suits ceramics applications, where porosity controlled by firing prevents cracking (Guggenheim & Martin, 1995).

Cation Exchange Capacity (CEC)

Clay minerals have an inherent capacity for cation adsorption and exchange—a property measured as cation exchange capacity (CEC). This is due to substitutions in their crystal lattice, e.g., magnesium for aluminum in octahedral layers, that produce a permanent negative charge (Brady & Weil, 2008). Smectites such as montmorillonite possess high CEC values (80–150 meq/100 g) that allow them to hold nutrients such as potassium (K^+) and ammonium (NH_4^+) in soils. Kaolinite, with a low CEC (3–15 meq/100 g), has limited nutrient-holding capacity but is easier to use in paper coatings where ionic interference is not wanted (Murray, 2007). Farmers in regions of clay soils, such as Nigeria, have a tendency to incorporate organic amendments into soil in order to enhance CEC, thereby enhancing crop production in regions of nutrient deficiency (Adekola *et al*., 2016).

Plasticity and Swelling Behavior

Clay minerals exhibit plasticity—the ability to be deformed without fracturing—when combined with water, as water molecules form hydrogen bonds with their charged surfaces. Bentonite, rich in smectite, possesses high plasticity and is utilized in drilling muds for borehole stabilization by creating a viscous, flexible film (Padulosi *et al*., 2023). Swelling, another important characteristic of smectites, occurs when water penetrates interlayer spaces, increasing the clay's volume significantly. This property is advantageous in applications like landfill liners to inhibit leachate seepage; however, excessive swelling can destabilize building foundations in clay-rich soils (Reddy *et al*., 2020).

Thermal and Chemical Modifications

Heating clays modifies their structure and reactivity. For example, heating kaolinite to 550°C eliminates hydroxyl groups, forming metakaolin ($Al_2Si_2O_7$), a product utilized to improve the durability of concrete:

$$Al_2Si_2O_5(OH)_4 \rightarrow Al_2Si_2O_7 + 2H_2O\uparrow$$

Likewise, acid or alkali treatment of clays can enhance porosity or create active sites. Acid-activated bentonite, for instance, is utilized in petroleum refining to crack heavy hydrocarbons into lighter fuels (Adams & McCabe, 2006).

Environmental and Industrial Relevance

The versatility of clay minerals forms the basis of their application in varied areas. In wastewater treatment, montmorillonite's large CEC enables it to adsorb heavy metals such as lead (Pb^{2+}) and cadmium (Cd^{2+}), whereas organoclays—modified with surfactants—adsorb organic pollutants such as pesticides (Sposito, 2008). In agriculture, clay-humus complexes enhance the structure of soil and water retention, especially in dry climates. Also relying on clays are industrial applications: kaolinite is a significant component in porcelain, and the fibrous nature of palygorskite renders it the ideal substance for cat litter due to its high absorbency (Murray, 2007).

The ongoing exploration of clay minerals reflects their potential to address modern challenges. Researchers are investigating their role in carbon capture, where their high surface area and CEC could trap CO_2 emissions, and in drug delivery systems, where their layered structures might encapsulate pharmaceuticals (Bergaya & Lagaly, 2013). As industries prioritize sustainability, clays offer a renewable, low-cost alternative to synthetic materials, bridging the gap between ecological responsibility and technological advancement.

1.2.1.2 CLASSIFICATION OF CLAY MINERALS

1. Kaolinite Group:

Kaolinite is a dioctahedral clay mineral characterized by its one-to-one layer structure composed of silica (SiO_4) tetrahedral sheets and alumina (AlO_6) octahedral sheets linked by oxygen atoms. Kaolinite exhibits properties such as low shrinkage upon firing, white color, high plasticity, and excellent thermal stability (Olaremu, 2021). Due to its unique properties, kaolinite finds extensive use in the ceramics industry for manufacturing porcelain, tiles, sanitaryware, and refractory materials. It is also used in paper coatings, paints, rubber compounds, and pharmaceutical formulations.

2. Montmorillonite Group:

Montmorillonite belongs to the smectite group of clay minerals and is characterized by its three-layer structure comprising two tetrahedral silica sheets sandwiching an octahedral alumina sheet. Montmorillonite exhibits high cation exchange capacity (CEC), swelling behaviour in the presence of water, and colloidal properties (Park *et al.*, 2016). Montmorillonite is widely used in the drilling industry as a key component of drilling fluids or muds, where it helps provide viscosity, lubrication, and suspension properties. It is also utilized in cat litter, geosynthetic clay liners (GCLs), and as a sealant in civil engineering applications.

3. Illite Group:

Illite is a non-expanding, dioctahedral clay mineral characterized by its potassium (K) ions within the interlayer spaces. Illite possesses a 2:1 layer structure similar to montmorillonite but lacks significant swelling characteristics. It is often found associated with mica minerals. Illite is utilized in drilling muds for its stability and resistance to swelling (Singh, 2022). It is also used in construction materials, ceramics, and as a filler in paper, plastics, and rubber.

4. Other Clay Minerals:

- Smectite: Smectite minerals such as hectorite and saponite exhibit similar properties to montmorillonite and are used in cosmetics, pharmaceuticals, and as rheological modifiers in various industrial applications.
- Chlorite: Chlorite is a phyllosilicate mineral with a 2:1 layer structure like illite but contains magnesium (Mg) and iron (Fe) in its octahedral layers. It finds applications in soil stabilization, oil drilling, and as a filler in plastics and rubber.

1.2.1.3 STRUCTURE AND CHEMICAL COMPOSITION OF CLAY MIENRALS

Clay minerals are layered hydrous aluminosilicates with a repeating structure of tetrahedral and octahedral sheets. They are the basic units making up all clay structures, and different mineral groups are defined by varying stackings of these sheets (Bergaya *et al*., 2006). The key to describing their peculiar physical and chemical behaviors is understanding this atomic structure.

Tetrahedral and Octahedral Sheets

The tetrahedral sheet consists of silicon (Si) atoms coordinated to four oxygen atoms in a hexagonal arrangement of SiO_4 tetrahedra. In contrast, the octahedral sheet consists of aluminum (Al) or magnesium (Mg) atoms coordinated to six oxygen or hydroxyl (OH^-) groups in an octahedral arrangement. The sheets are bonded together by shared oxygen atoms to form discrete layer types (Sposito, 2008). For example:

1:1 layer clays (i.e., kaolinite) consist of a single tetrahedral sheet attached to a single octahedral sheet through hydrogen bonds.

2:1 layer clays (i.e., montmorillonite) consist of an octahedral sheet between two tetrahedral sheets, bonded by weak van der Waals forces or interlayer cations.

The general chemical formula for 2:1 clays such as montmorillonite is

$(Na,Ca)_{0.3}(Al,Mg)_2Si4O_{10}(OH)2\cdot nH_2O$

, where interlayer cations (Na^+, Ca^{2+}) compensate for the negative charge from substitutions, e.g., Mg^{2+} for Al^{3+} in the octahedral layer (Murray, 2007).

Layer Stacking and Bonding

In kaolinite, classified as a 1:1 clay mineral, the structure comprises alternating tetrahedral and octahedral sheets that create inflexible layers interconnected through robust hydrogen bonds between the hydroxyl groups present in the octahedral sheet and the oxygen atoms of the tetrahedral sheet. This dense arrangement restricts expansion, resulting in a low cation exchange capacity (CEC) while providing substantial mechanical stability, thereby rendering kaolinite particularly suitable for ceramic applications.

Smectites like montmorillonite, on the other hand, possess 2:1 layers with loosely held interlayer cations (e.g., Na^+, Ca^{2+}) and water molecules. The layers can expand when hydrated and are larger in surface area and CEC. Sodium montmorillonite, for instance, swells greatly in water, a property that is taken advantage of in drilling muds to lubricate boreholes (Bergaya *et al*., 2006).

Chemical Diversity

The chemical character of clays varies based on their tetrahedral or octahedral sheet substitutions. For instance: Illite, a 2:1 clay, contains potassium (K^+) ions tightly fixed between layers due to isomorphic substitution of Al^{3+} for Si^{4+} in tetrahedral sites. This stabilizes the structure, preventing swelling (Sposito, 2008).

Chlorite includes an additional hydroxide sheet (Mg-Al-OH) between its 2:1 layers, enhancing thermal stability for uses in foundry molds (Murray, 2007).

Implications of Structure

The layered structure and chemical substitutions govern clays' industrial and environmental roles. For example: Smectites' interlayers, being expandable, allow them to adsorb organic pollutants in wastewater treatment (Brady & Weil, 2008).

The non-expandable nature of kaolinite makes it chemically inert and, therefore, usable as a binder for pharmaceutical tablets.

1.2.1.4 APPLICATIONS OF CLAY MINERALS

Clay minerals are versatile materials with diverse applications across industries, owing to their unique structural, chemical, and physical properties. Below is a categorized overview of their key uses:

1. Construction and Ceramics

Clay minerals are foundational in construction and ceramics due to their plasticity, binding strength, and thermal stability. Kaolinite, for instance, is prized in ceramics for its high plasticity and low shrinkage during firing, making it ideal for producing porcelain, tiles, and refractory materials (Finlay *et al.*, 2020). Similarly, bentonite and illite are critical in manufacturing bricks and cement, where their ability to harden at high temperatures ensures durability (Singh, 2022).

2. Agriculture

In agriculture, clay minerals enhance soil health by improving water retention, nutrient availability, and structural stability. Their high cation exchange capacity (CEC) allows soils to retain essential nutrients like potassium and ammonium, reducing fertilizer leaching and boosting crop yields. Smectite clays, such as montmorillonite, are particularly effective in arid regions for maintaining soil moisture (Sverdrup *et al.*, 2019).

3. Environmental Remediation

Clay minerals are vital in addressing environmental pollution. Their large surface area and adsorption capacity enable them to trap heavy metals (e.g., lead, cadmium) and organic contaminants (e.g., pesticides) in soil and groundwater. Bentonite-based barriers, for example, are used in landfills to prevent toxic leachate migration (Sverdrup *et al.*, 2019).

4. Petroleum and Drilling

In the petroleum industry, smectite clays like montmorillonite are key components of drilling fluids. These fluids stabilize boreholes by forming a protective mud cake, lubricating drill bits, and transporting rock cuttings to the surface. Their rheological properties prevent well collapse and fluid loss in porous formations (Ramos *et al*., 2024).

5. Pharmaceuticals and Cosmetics

The adsorptive and rheological properties of clay minerals are exploited in pharmaceuticals and cosmetics. Kaolinite and bentonite serve as excipients in drug delivery systems, while their absorbent qualities make them effective in antiperspirants and facial masks. Their inert nature ensures compatibility with sensitive formulations (Yu *et al*., 2022).

1.2.2 SIGNIFICANCE OF CLAY MINERALOGY IN INDUSTRIAL AND ENVIRONMENTAL USES

Clay mineralogy is of great importance in both improving industrial operations and solving environmental issues due to the special physicochemical properties of clay minerals. These naturally occurring aluminosilicates have layered configurations, high surface reactivity, and adjustable adsorption capacities, which render them essential in various industries. Here, we discuss their key roles in streamlining industrial processes, driving innovation, and ensuring sustainability.

Industrial Process Optimization

Clay mineralogy guides raw material selection across industries like ceramics, construction, and cosmetics. Through mineralogical composition analysis, industries can optimize parameters such as firing temperatures or chemical treatment to improve product performance.

For example, the low shrinkage of kaolinite upon firing provides structural integrity for porcelain production, whereas smectite-rich clays provide plasticity for brick production (Olaremu, 2021). Likewise, in catalysis, high surface area and cation exchange capacity (CEC) clays are designed to improve reaction efficiency. For instance, acid-activated montmorillonite improves biodiesel production through the optimization of transesterification kinetics (Yu *et al.*, 2022). This precision reduces defects, energy consumption, and enhances cost-effectiveness.

Technological Innovation

Clay mineralogy advances have accelerated the creation of advanced materials. Clays' layered nature and nanoscale size allow them to be incorporated into electronics, aerospace, and biomedical nanocomposites. For example, polymer-clay nanocomposites have excellent mechanical strength and thermal stability, and thus are suitable for the production of lightweight auto parts (Zhou *et al.*, 2021). In medicine, halloysite nanotubes—a tubular clay mineral—can be used as drug carriers thanks to their high loading capacity and controlled release (Zhou *et al.*, 2021). These developments are a result of knowing structure-property relations so that researchers can tailor-design materials for specific functionalities.

Environmental Remediation

Clay minerals play a central role in the remediation of soil and water pollution. Their high CEC and adsorption capacity facilitate the immobilization of heavy metals (e.g., Pb^{2+}, Cd^{2+}) and organic pollutants (e.g., pesticides) in polluted soils. Bentonite barriers, for instance, are used to prevent leachate migration in landfills by swelling to close porous substrates (Padilla-Ortega *et al.*, 2020). In wastewater treatment, kaolinite and montmorillonite efficiently eliminate dyes and nutrients via ion exchange and surface complexation. Designing clay mineralogy for target pollutants—like applying surfactant-modified clays for hydrophobic contaminants—improves remediation efficiency at lower operational costs.

Sustainability and Resource Efficiency

Clay mineralogy facilitates sustainable practices through resource optimization and minimization of waste. The characterization of clay deposits allows industries to target high-quality reserves, preventing overexploitation. For instance, recycling ceramic wastes to produce secondary raw materials alleviates landfill pressures and preserves natural kaolinite deposits (Peng *et al*., 2020). Moreover, green technologies based on clays, like photocatalytic clays for air cleaning or biochar-clay composite for carbon capture, are supportive of worldwide decarbonization efforts. Such uses highlight clays' contribution to the shift towards circular economies and low-carbon industries.

1.2.3 CATALYSIS

Catalysis is the increase in the rate of a chemical reaction by the addition of a catalyst that is not chemically changed by the reaction. Catalysts lower the activation energy required for reactions so that they can proceed more efficiently and quickly. This is a key concept in industry, where catalysts help to produce valuable chemicals, fuels and pharmaceuticals (Ertl *et al*., 2008). Catalysis is involved in about 90% of industrial chemical processes and is therefore crucial for modern industry (Ertl *et al*., 2008).

Types of Catalysis

Catalysis is usually categorized as homogeneous catalysis and heterogeneous catalysis.

Homogeneous Catalysis

In homogeneous catalysis the catalyst and reactants are in the same phase (usually liquid). Homogeneous catalysis occurs in organic synthesis such as polymerization, hydroformylation, and esterification. An example is sulfuric acid (H2SO4) in biodiesel esterification, where it acts as a proton donor to promote the reaction (Sheldon, 2017).

Heterogeneous Catalysis

In heterogeneous catalysis, the catalysts are in a different phase from the reactants. This type is very widely applied in industrial applications, particularly in energy and environmental processes. For example, solid catalysts such as zeolites are used in fluid catalytic cracking (FCC), a process in petroleum refining to break large hydrocarbon molecules into smaller valuable fractions, such as gasoline (Bartholomew & Hecker, 2019). Metal catalysts, such as platinum and palladium, are used in catalytic converters to reduce harmful vehicle emissions by converting toxic gases such as carbon monoxide (CO) into less toxic substances (Twigg, 2011).

1.2.4 CLAY CATALYSIS

Clay minerals have received much attention as catalysts because of their favorable characteristics for catalyzing a wide range of chemical reactions. Their abundance, low cost, controllable surface chemistry, and benign environmental profile make them promising alternatives to conventional catalysts, as pointed out by Munnik *et al.* (2015). Clay catalysis is crucial in many reactions, including organic synthesis, petrochemical processes, environmental remediation, and biomass conversion. Mechanisms in clay catalysis and optimizing their catalytic performance are important research areas with great potential for industrial applications, as pointed out by Liu and Zhang (2014).

Another important and attractive feature of the clay minerals as catalysts is their extremely high surface area and porosity that provides a large number of active sites for catalytic reactions. The layered structure of clay minerals (e. g., montmorillonite and kaolinite) allows guest molecules to intercalate into the interlayer space, thus allowing reactions to take place in confined spaces (Nor & Al-Amiery, 2024). In addition, hydroxyl groups (-OH) and exchangeable cations on the surface of the clay mineral interact with reactant molecules, stabilize intermediates, and promote catalytic transformations.

Clay minerals have been known to exhibit catalytic activity via different mechanisms, depending on the type of reaction and clay mineral nature. According to Khan *et al.* (2023), acidic or basic sites on clay surfaces are critical for catalysis. Acidic sites connected to aluminum or silicon centers facilitate proton transfer and substrate activation in acid-catalyzed reactions such as esterification, alkylation, and hydrocarbon cracking. Basic sites with hydroxyl groups or exchangeable cations promote nucleophilic reactions and carbon-carbon or carbon-heteroatom bonds in base-catalyzed reactions, according to Khan *et al*. (2023).

Clay minerals are widely employed as catalysts in organic synthesis because of mild reaction conditions, high selectivity and recyclability. Park *et al*. (2016) reported the application of montmorillonite clay in the synthesis of fine chemicals, pharmaceuticals and biofuels through Friedel-Crafts acylation, aldol condensation and transesterification. Kaolinite-based catalysts are employed in the production of biodiesel from vegetable oils by catalyzing triglyceride transesterification with methanol to form fatty acid methyl esters (FAMEs).

In addition to their well-known role in organic synthesis, clay minerals are also attractive catalysts for a range of petrochemical processes, i.e. the conversion of crude oil and natural gas into value-added products. For instance, bentonite has been widely studied for its catalytic activity in hydrocracking, hydroisomerization and Fischer-Tropsch synthesis, which are key steps in petroleum refining and gas-to-liquids (GTL) processes (Yassin *et al.,* 2022; see also Reddy *et al*., 2025). The catalytic activity of clay minerals in these processes can lead to higher yields of desired products, reduced energy consumption and lower environmental impact compared to conventional catalysts.

Furthermore, clay minerals are also very suitable for environmental catalysis, for example the degradation of organic pollutants and the removal of heavy metals from wastewater. The photocatalytic degradation of organic pollutants over clay-based catalysts under UV or visible light irradiation has been investigated as a promising sustainable water purification method (Peng *et al*., 2020). Furthermore, catalytic redox reactions with functionalized clay minerals have been investigated for the removal of toxic heavy metals by adsorption followed by reduction to less toxic forms.

In the case of biomass conversion, acidic clay catalysts have been employed for the conversion of biomass-derived feedstocks into biofuels and platform chemicals. Hydrolysis and dehydration of biomass-derived carbohydrates with acidic clay catalysts give furans such as 5-hydroxymethylfurfural (HMF), a precursor for biofuels and bioplastics (Peng *et al*., 2020). Clay-supported metal catalysts have been investigated for hydrogenation and deoxygenation of biomass-derived compounds to produce hydrocarbons for transportation fuels (Zhao *et al*., 2020).

1.2.5 BASE ACTIVATION OF CLAY MINERALS

Base activation of clay minerals is a chemical modification procedure where clays are subjected to alkaline solutions in order to improve their surface properties. It is a widely applied technique to increase the adsorption capacity, surface area and porosity of clays and to tailor their surface characteristics for catalytic applications, wastewater treatment and other industrial applications (Adekola *et al.,* 2016; Yahia *et al*., 2019). The activation process alters the clay structure by forming new active sites that increase its reactivity and efficiency in different applications.

1.2.6 CHARACTERIZATION TECHNIQUES

Characterization methods are critical for evaluating base-activated clays. They provide information on structure, composition, surface area, porosity, and catalytic activity. These methods help researchers understand base activation-induced changes and optimize clays for specific applications. Below are some commonly used characterization techniques:

X-ray Diffraction (XRD)

XRD can be employed to determine the crystalline structure and phase composition of the clay minerals before and after base activation. The crystalline structure of the clay mineral may be changed by the dissolution of some cations and the formation of new phases after base activation. Changes in peak positions, changes in peak intensities and new peaks in XRD patterns indicate structural changes from base treatment. For example, in a study on the synthesis of zeolite A from kaolinite clay, XRD confirmed the formation of zeolite after base activation (Abdullahi *et al.,* 2023).

Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy can be used to detect functional groups on base-activated clays. Base activation introduces or changes these groups on the clay surface, which affects surface chemistry and catalytic properties. FTIR spectra detect characteristic absorption bands for functional groups on clays, providing qualitative information on chemical changes from base treatment. For example, Abdullahi *et al.* (2023) confirmed Si–O, Si–Al, Al–O, and metal–oxygen bonds via FTIR, which indicated successful clay structure modification.

Brunauer–Emmett–Teller (BET) Surface Area Analysis

BET analysis is applied to calculate the specific surface area and pore size distribution of base-activated clays. Surface area increases by the formation of mesopores and micropores during base activation which also enhances adsorption capacity and catalytic activity. The quantitative information on surface area, pore volume and pore size distribution from BET analysis are very important for evaluation of base activation effectiveness. Abdullahi *et al.* (2023) reported that surface area and pore volume increased after base activation and the samples were more porous.

Scanning Electron Microscopy (SEM)

SEM is used to image the morphology, particle size and surface characteristics of micro- and nanostructured base-activated clays. SEM images show pore formation and surface texture changes during base activation. SEM images are used to characterize clay particle distribution, morphology and agglomeration. Abdullahi *et al*. (2023) observed cubic morphology with larger, monodispersed and partially spherical particles and concluded that base activation changed clay morphology.

These characterization techniques provide useful information about base activation effects in clays and facilitate their application in different industrial processes.

CHAPTER TWO

2.0 MATERIALS AND METHOD

2.1 MATERIALS AND REAGENTS

- Clay
- Pulverized
- Distilled water
- Distilled deionized water
- Sodium Hydroxide

2.1.1 APPARATUS

- Mortal
- Pestle
- Crucibles
- Beakers
- Volumetric flasks
- Conical flasks
- Measuring Cylinder
- Wash bottle
- Analytical balance
- Glass rod
- Funnel

- Filter paper
- Graduated Cylinder
- Petri dish
- Spatula

2.1.2 EQUIPMENTS

- Electric grinding machine
- Electric orbital shaker
- Fume Chamber
- Drying Oven
- Muffle furnace

2.2 METHODOLOGY

2.2.1 SAMPLE PREPARATION

The Gegu-Egba clay sample was crushed using ceramic mortar and pestle and finally pulverized mechanically in a ceramic plate.

2.2.2 PRETREATMENT OF CLAY

The clay sample was heated at 105-110°C in an oven to remove moisture Upon cooling the already heated clay was kept in a desiccator.

2.2.3 PREPARATION OF NAOH STOCK SOLUTION

30g of NaOH pellet was weighed with an analytical weighing balance and dissolve with distilled deionized water to 100ml mark, in a volumetric flask, it was stirred with a glass rod to dissolve the sodium hydroxide (NaOH) completely

2.2.4 BASE TREATMENT

10_g of the Gegu-Egba clay sample was mixed with the 30%(W/v) NaOH (100ml) and pour into a round bottom flask, covered and agitated the clay and thoroughly using a mechanical shaker for 24hrs.

2.2.5 WASHING AND FILTRATION

After 24hrs agitation, the clay sample was filtered with filter paper, and was wash with distilled deionized water to neutrality.

2.2.6 DRYING AND HEATING

After filtration, the base activated clay was put in crucible and dried in an oven at 105-110°C, thereafter, it was heated to 200°C & 400°C separately in a muffle furnace for three hours (3hrs) then allowed to cool in a desiccator. Store the treated clay in a dry, clean container with a tight-fitting lid to prevent contamination.

The activated base clay samples were subjected to the following analysis; X-ray Diffractometer (XRD), Fourier Transform Infrared (FT-IR) Spectroscopy, Brunauer-Emmet-Teller surface area analysis, and Scanning Electron Microscopy-Energy Dispersive X-ray, Scanning Electron Microscope.

CHAPTER THREE

3.0 RESULT AND DISCUSSION

3.1 RESULT

The Gegu - egba modified with 30%NaOH at 200°c and 400°c clay samples were characterized using FT-IR, SEM-EDX, BET and XRD analytical techniques.

Table 3.1: Results for FTIR analysis of 30% NaOH treated clay at 200°c

Wavenumber (cm^{-1})	Functional Group	Possible Assignment
3623.0	–OH Stretching	Free hydroxyl groups (Kaolinite)
3693.8	–OH Stretching	Structural hydroxyl (Clay minerals)
1807.8	C=O Stretching (Weak)	Possible carbonyl impurity
1114.5	Si–O Stretching	Quartz, silicates
752.9	Si–O Bending	Quartz or feldspar
689.6	Al–O–Si Stretching	Clay minerals (Kaolinite, Muscovite)
913.2	Al–OH Bending	Kaolinite
1028.7	Si–O–Si Stretching	Quartz, silicates

Figure 3.1: FTIR analysis of 30% NaOH treated clay at 200°c

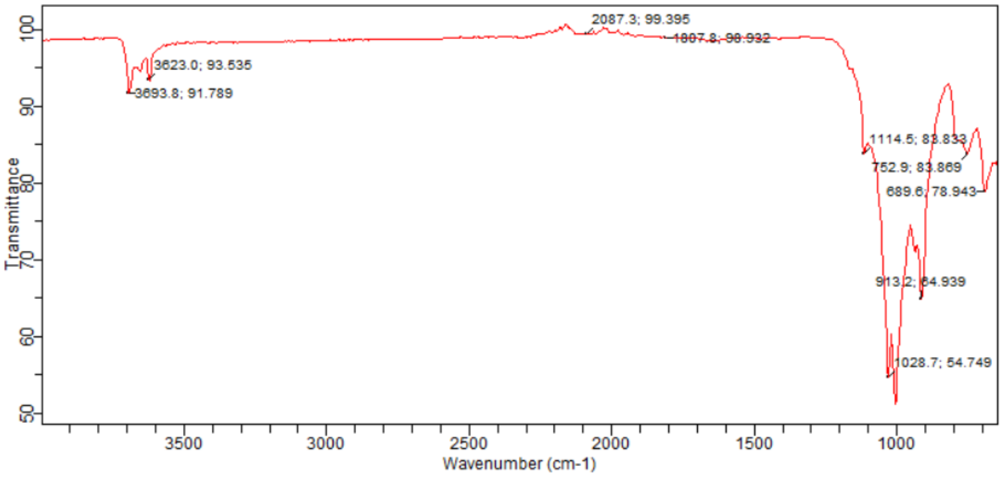
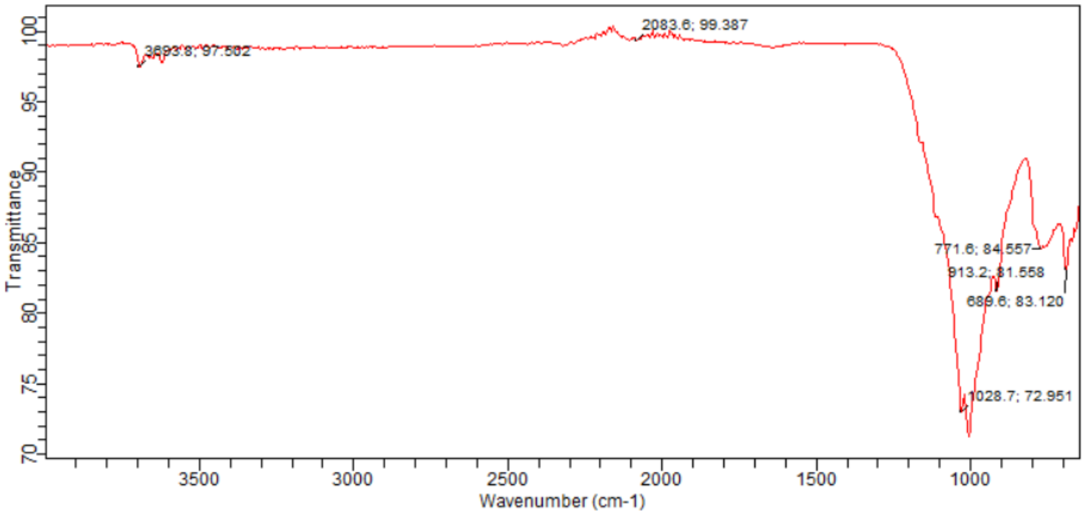


Table 3.2: Results for FTIR analysis of 30% NaOH treated clay at 400°c

Wavenumber (cm⁻¹)	Functional Group	Possible Assignment
3693.8	–OH Stretching	Structural hydroxyl (Kaolinite, Clay minerals)
1028.7	Si–O–Si Stretching	Quartz, silicates
913.2	Al–OH Bending	Kaolinite
889.6	Al–O–Si Stretching	Clay minerals (Kaolinite, Muscovite)
771.8	Si–O Bending	Quartz or feldspar

Figure 3.2: FTIR analysis of 30% NaOH treated clay at 400°c



The FTIR analysis of 30% NaOH-treated clay at **200°C and 400°C** reveals key structural transformations. At **200°C**, the presence of **free hydroxyl (-OH) stretching at 3623 cm⁻¹** suggests adsorbed water, while **structural hydroxyl (-OH) at 3693.8 cm⁻¹** indicates intact kaolinite. However, at **400°C**, the disappearance of free hydroxyl groups signifies **dehydration and early dehydroxylation**, a precursor to **metakaolin formation**. The **Si–O stretching at 1114.5 cm⁻¹ in the 200°C sample disappears at 400°C**, suggesting alterations in the **silicate framework**, while the **Si–O–Si stretching at 1028.7 cm⁻¹ remains**, indicating quartz stability. The **shift in Si–O bending from 752.9 cm⁻¹ to 771.8 cm⁻¹** suggests **structural realignment** in quartz and feldspar. Additionally, the **weak C=O peak at 1807.8 cm⁻¹ at 200°C disappears at 400°C**, implying **thermal decomposition of carbonyl impurities**.

The **Al–O–Si stretching shifts from 689.6 cm⁻¹ to 889.6 cm⁻¹**, suggesting possible **restructuring of clay minerals, including muscovite and feldspar**, under heat and NaOH influence. The **Al–OH bending peak at 913.2 cm⁻¹**, characteristic of **kaolinite**, remains in both spectra but may weaken at higher temperatures, indicating progressive dehydroxylation**.** These changes align with the transformation of **kaolinite to metakaolin**, which enhances its **pozzolanic reactivity and adsorption capacity**. The overall reduction in functional groups at **400°C** highlights the **thermal stability of quartz and feldspar**, while the clay matrix undergoes gradual **structural modifications**, making it more reactive for applications like **geopolymer synthesis and industrial catalysis**.

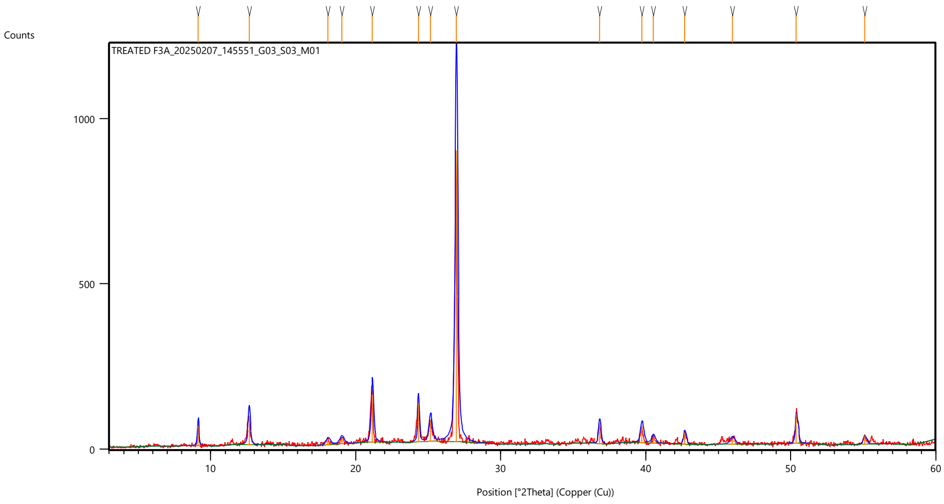


Figure 3.3: XRD analysis of 30% NaOH treated clay at 200°c

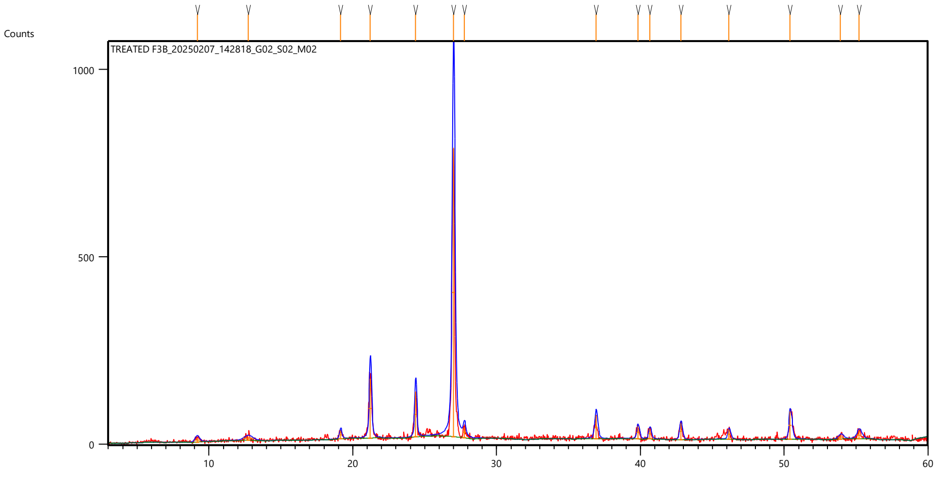


Figure 3.4: XRD analysis of 30% NaOH treated clay at 400°c

Table 3.3: X-Ray Diffraction (XRD) Analysis of 30% NaOH-Treated Clay Samples at Different Temperatures

Parameter	F3A (30% NaOH treated clay at 200^{o}c)	F3B(30% NaOH treated clay at 400^{o}c)	Interpretation
Quartz	67 ± 5	59 ± 3	Quartz is the dominant mineral in both samples, suggesting a high silicate content.
Kaolinite-1A	23 ± 3	3.1 ± 17	Higher in F3A, indicating more clay content, potentially affecting plasticity and adsorption properties.
Orthoclase	1.7 ± 12	33 ± 3	Much higher in F3B, suggesting stronger feldspar influence, possibly affecting thermal stability.
Albite	2 ± 3	1.5 ± 11	Low albite levels in both, indicating minimal sodium feldspar presence.
Muscovite	0.3 ± 15	2.3 ± 10	Slightly more in F3B, implying higher mica content, which may affect mechanical properties.
Clinochlore	6 ± 4	1 ± 3	Found in both, but significantly higher in F3A, affecting chemical reactivity and thermal behavior.

The XRD analysis of 30% NaOH-treated clay at 200°C and 400°C reveals significant changes in crystallinity due to thermal treatment. At 200°C (F3A), the sample retains a higher kaolinite content (23%), indicating that the clay structure is still largely intact. This corresponds with the strong structural hydroxyl (-OH) FTIR peaks, suggesting limited dehydroxylation. The dominant quartz phase (67%) remains stable, as quartz is highly crystalline and does not decompose under these conditions. The presence of clinochlore (6%) further supports the crystallinity of F3A, as this mineral contributes to chemical reactivity. However, the low orthoclase content (1.7%) suggests minimal feldspar transformation at this temperature. These findings align with the FTIR results, where strong Si–O and Al–O–Si peaks confirm the presence of an organized crystalline structure with limited disruption.

At 400°C (F3B), kaolinite content drastically reduces to 3.1%, indicating significant dehydroxylation and amorphization, a key step toward metakaolin formation. This explains the disappearance of free hydroxyl (-OH) groups in the FTIR spectrum, as kaolinite begins to lose its crystallinity. The increase in orthoclase (33%) suggests feldspar restructuring, enhancing the sample's thermal stability. The reduction in clinochlore (from 6% to 1%) further confirms mineral breakdown, leading to structural reorganization. Additionally, the slight decrease in quartz content (59%) implies minor amorphization, though quartz remains largely stable. These changes in mineral phases reflect a progressive transition from a crystalline to a partially amorphous structure, making the material more reactive for applications like geopolymer synthesis and adsorption processes.

SEM - EDX

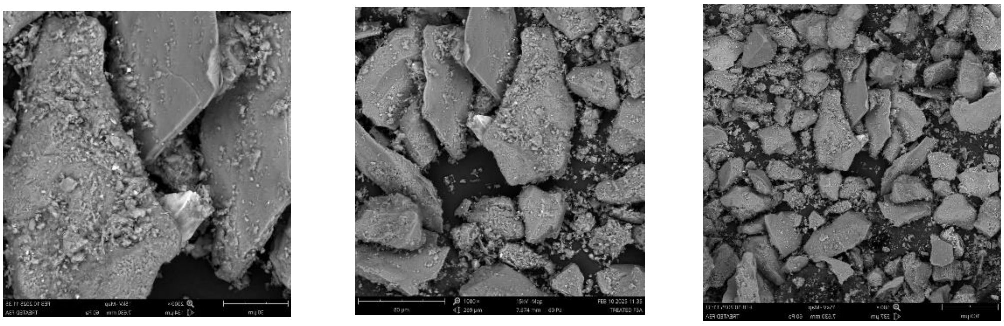


Figure 3.5: SEM image for 30% NaOH treated clay at 200^{o}c at 2000, 1000, 500 magnification.

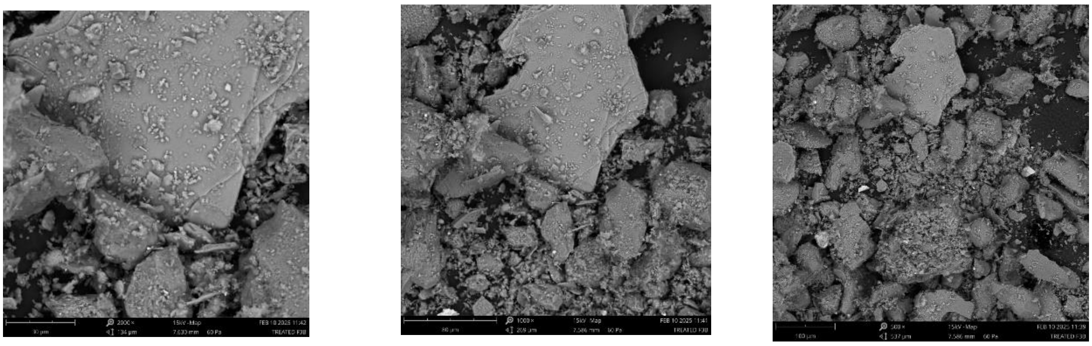


Figure 3.6: SEM image for 30% NaOH treated clay at 400^{o}c at 2000, 1000, 500 magnification

Table 3.4: Energy Dispersive X-ray Spectroscopy (EDX) Analysis of the elemental composition of 30% NaOH-Treated Clay Samples at Different Temperatures

Element Name	(F3A)weight composition for 30% NaOH treated clay at 200^{O}C	(F3B)weight composition for 30% NaOH treated clay at 400^{O}C
Silicon	75.33	73.41
Aluminium	19.19	20.77
Potassium	3.38	3.58
Titanium	0.61	0.16
Sulphur	0.11	0.10
Chlorine	0.09	0.06
Calcium	0.00	0.00
Magnesium	0.00	0.00
Manganese	0.00	0.00
Iron	0.00	0.00
Phosphorus	0.00	0.00

The SEM-EDX analysis of the 30% NaOH-treated clay samples at different temperatures reveals notable differences in elemental composition and surface morphology. The SEM images show that the sample treated at 200°C (F3A) retains a more defined and structured granular form, whereas the 400°C-treated sample (F3B) appears more fragmented, indicating thermal degradation and increased porosity. This transformation aligns with the EDX results, where F3B exhibits a slight reduction in silicon (Si) content, likely due to structural breakdown and loss of some silicate phases. The increase in aluminium (Al) content in F3B suggests a possible phase transformation where kaolinite partially converts into more thermally stable aluminium-rich phases, such as metakaolin.

Additionally, minor changes in potassium and sodium levels indicate that alkali elements may have redistributed due to thermal treatment, potentially affecting the material's reactivity and adsorption properties. The sharp reduction in titanium content in F3B suggests that the mineral phase containing titanium is more susceptible to high-temperature decomposition or leaching. The absence of iron, calcium, magnesium, and phosphorus in both samples suggests that these elements were either below detection limits or not significantly present in the raw clay. Overall, the SEM-EDX results demonstrate that increasing the treatment temperature alters the elemental distribution and morphology, which may influence the material's suitability for industrial applications such as adsorption, catalysis, or ceramics.

Table 3.5: BET (Brunauer–Emmett–Teller) Analysis of 30% NaOH-Treated Clay Samples at Different Temperatures

Characterization Results	**30% NaOH treated clay at 200^{O}C**	**30% NaOH treated clay at 400^{O}C**
Surface area by BET (m^2/g)	249.577	214.149
Surface area by BJH adsorption (m^2/g)	284.891	279.907
Pore volume (cc/g)	**0.139**	**0.137**
Pore diameter (nm)	**2.136**	**2.093**

At 200°C, the 30% NaOH-treated clay shows a BET surface area of 249.577 m^2/g, which is higher than the 214.149 m^2/g measured at 400°C, indicating that the material undergoes structural changes as the temperature increases. The reduction in surface area is likely due to the collapse or sintering of smaller micropores, a common effect at elevated temperatures. This decrease can result in a less accessible surface for adsorption, suggesting that the porosity is affected by the high temperature. However, the BJH adsorption surface area remains relatively stable, with a minor reduction from 284.891 m²/g at 200°C to 279.907 m²/g at 400°C. This suggests that mesoporous structures, which are less sensitive to temperature, are better preserved, indicating that these pores are still available for adsorption even after treatment at 400°C.

The pore volume decreases only slightly from 0.139 cc/g at 200°C to 0.137 cc/g at 400°C, reflecting minimal change in the overall porosity of the material despite the temperature increase. Similarly, the pore diameter shrinks from 2.136 nm to 2.093 nm, indicating a slight contraction in pore size as a result of higher-temperature treatment. This minor reduction in both pore volume and diameter suggests that while the overall porosity is maintained, there is some degree of densification or shrinkage in the structure. This could impact adsorption capacity, particularly for larger molecules, but the material likely retains sufficient mesoporosity for adsorption applications, especially for smaller molecules that can access the remaining open pores.

3.3 CONCLUSION

The characterization of 30% NaOH-treated clay at 200°C and 400°C using FTIR, XRD, SEM-EDX, and BET analyses reveals significant structural and compositional modifications due to thermal treatment. FTIR results indicate the progressive dehydroxylation of kaolinite at higher temperatures, leading to the transformation into metakaolin, which enhances its pozzolanic reactivity. XRD analysis confirms the reduction of kaolinite and clinochlore with increasing temperature, while quartz and feldspar remain stable, contributing to structural rigidity. SEM-EDX findings highlight changes in surface morphology and elemental composition, with increased porosity and slight reductions in silicon content, suggesting partial phase transformations.

BET analysis shows that the surface area and pore characteristics decrease slightly at 400°C, indicating minor densification and reduced microporosity, though mesoporous structures are largely retained. These results demonstrate that thermal treatment significantly influences the clay’s physicochemical properties, enhancing its potential for applications in geopolymer synthesis, catalysis, and adsorption. The study confirms that controlled NaOH modification and thermal activation can optimize clay properties for industrial and environmental applications.

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