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Research Article

Investigating the effect of acid concentration on the surface and structural properties of HCl-modified natural clay

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ABSTRACT

This study examined the changes in the structural and surface properties of Omialafara natural clay upon acid modification, using different concentrations of HCl solution. The changes in the natural clay before and after the acid treatments were studied by different instrumental techniques such as powder XRD, FTIR, BET, and SEM-EDX. The surface acidity of the clays was measured via pyridine-FTIR spectroscopy. The SEM images of the clays revealed different morphological features due to changes in the clay structures upon acid modifications. The specific surface area (SSA) of the clay increased at high concentrations of acid treatment, with the highest SSA value of 528 m^2/g obtained at 35% HCl. The high SSA value, which is an important parameter in adsorption studies, was caused by structural changes within the clay matrix upon acid activation. The Si/Al ratios were directly proportional to the SSA values for the modified clays. Measurement of the surface acidity revealed intense adsorption bands at 1580 cm^{-1} for Brønsted acid sites (BAS) and 1438 and 1483 cm^{-1} for Lewis acid sites (LAS) for the acid-modified clays at modest concentrations (15% and 25% HCl). At low (5%) and high (35%) acid concentrations, weak and low-intensity bands were observed for BAS and LAS. The surface acidities of the unmodified and modified clays showed an inverse proportionality to Si/Al ratios, revealing that high Al content may be required to maintain high acidity in the modified clays for catalysis. While the 35% HCl-modified clay with the highest SSA value (528 m^2/g), Si/Al ratio of 5.35, and low surface acidity may be efficient in adsorption studies, the 25% HCl-modified clay, which has a high SSA value of 450 m^2/g, Si/Al ratio of 3.81, and a very high surface acidity, may be a suitable catalyst for various acid-catalyzed reactions.

Keywords: Acid modification; Calcination; Unmodified natural clay; Pore volume; Surface properties; Specific surface area

Introduction

Natural clay minerals have gained a lot of academic and industrial research attention because they are the most abundant, non-toxic, and low-cost aluminosilicate materials with diverse applications [1]. Agriculture, pharmaceuticals, production (ceramics and insulating materials), the oil industry (mud drilling), etc. are just a few of the industries in which they are employed [2, 3]. However, most of the surface properties of these natural clays require fine-tuning to satisfy specific applications since the mineral compositions of these clays vary based on their origin and parent material [4].

Surface modification is one technique used to improve the physicochemical properties of materials, particularly natural clay minerals employed as catalysts in chemical synthesis and remediation studies [5, 6]. Some of these physicochemical properties include particle size, specific surface area (SSA), cation exchange capacity (CEC), porosity, plasticity, swelling and adsorptive properties, thermal stability, and chemical composition, which have a major influence on the properties of the clay and its application [7–9]. Employing clay minerals in their natural state without prior modification in adsorption studies, remediation, and catalytic studies has been reported to be less efficient [8, 10]. Therefore, modifications are tailored to improve these

properties for specific applications, and the products obtained are usually termed "designer clay minerals" [11].

The application for which clay minerals will be employed is a critical consideration when selecting the best techniques for altering the surface characteristics [12]. There are several methods employed in the modification of natural clay minerals. The accustomed methods used are thermal excitation and acid-base activation [5]. Other methods include interlayer ion exchange, intercalation methods, and surface grafting techniques. The designer clay minerals produced from these modifications have been reported to have enhanced adsorption capacity, improved catalytic sites (Lewis acid and Bronsted acid sites), and mechanical properties and were found to be efficient in applications such as catalysis, soil stabilization, water purification, and bone tissue engineering [13–16]. For example, Krupskaya *et al*. [17] investigated how inorganic acid (HCl and HNO_3) treatments affected the structural changes of montmorillonite clay obtained from deposits in Taganskoye, Kazakhstan, and Mukhortala, Russia, respectively. The modifications were performed under the acceptable condition for producing acid-modified clays for use as sorbents and catalysts in the chemical industries. They demonstrated that structural modifications occurred due to the partial substitutions of the interlayer cations with oxonium ions upon acid treatments, and this structural modification intensified with an increase in acid concentrations and reaction time. The changes were evident in the structural morphologies, increased SSA, pore volumes, and pore diameters of the modified clays [17]. Similarly, Soni *et al*. [18] investigated the effect of a series of organic and mineral acids on the structure and surface morphology of natural clay obtained from Rajasthan in India. They reported a significant alteration in the morphology and surface areas of the acid-modified clay, and the improvement of the surface properties was linked to the nature and strength of the acid employed in the treatment. These modifications had a significant impact on the catalytic efficiency of the clays to catalyze the degradation of Eriochrome Black T [18]. These highlight the importance of acid treatments in the production of designer clay minerals for specific applications.

Nigeria is rich in natural clay minerals, with an estimated clay deposit of approximately 700 million metric tons [19]. Numerous studies have been conducted on the various clay deposits located in different parts of the country. However, the plethora of literature on these natural clay deposits focuses on physicochemical properties, geotechnical properties, and mineralogical compositions [20–25], and there is a dearth of studies on their surface modifications [10, 26, 27] despite their relevance in various fields. For instance, Ajemba and Onukwuli [26] studied the effect of H_2SO_4 acid concentrations (1 M – 6 M) on the surface modification of Ukpor clay obtained from Nnewi local government area of Anambra state, Nigeria, in a bid to improve the adsorptive performance of the natural clay towards palm oil bleaching. They reported that 4 M H_2SO_4 acid-treated Ukpor clay with an improved SSA gave the best adsorptive capacity under the conditions employed.

The physicochemical, mineralogical, and geotechnical properties of the natural clay mineral obtained from Omialafara clay deposit in Ifon, Ose Local Government Area, Ondo State, Nigeria, have previously been reported [28–30]. For the first time, our group successfully modified the surface properties of this natural clay with different chemical treatments [31]. In our earlier studies, we employed very low concentrations (5% and 15%) of strong HCl acid and a weakly acidic NH_4Cl solution, respectively, in the modification of natural clay, and samples were calcined at 400 ^oC. Although there was evidence of morphological and surface modifications, there was no clear understanding of the effect of the acid concentrations on the observed changes, as properties such as SSA reduced significantly upon treatment with 5% of the respective acid concentration and were only higher than that of the parent clay when treated with 15% HCl [31]. In this study, a detailed investigation into the impact of HCl at higher concentrations on the structural and surface properties of the clay is described as part of our continued effort to improve the properties of Omilafara natural clay. The acid-modified clay materials were calcined at 400 ^oC and analyzed using various analytical techniques. including XRD, FTIR, BET, and SEM-EDX. The SSA and other porosity measurements were utilized to better understand the changes in the morphology and surface properties of the clay while making reference to the data obtained from our previous study. The surface acidity of the clays was measured via pyridine adsorption FTIR spectroscopy.

Materials and Methods

Materials

The natural clay samples used in this study were acquired from the Omialafara clay deposit in the Ifon Ose Local Government Area of Ondo State, which lies between. between latitudes N 6^o 54' 58'' and longitude E5^o 46' 20'' [29]. All chemicals used in this study were of analytical grade and were purchased from Pyrex Chemicals. They were utilized without any further purification. In this study, the 25% and 35% HCl concentrations employed for the modification of the clay samples were prepared from 37% concentrated HCl. This experiment employed deionized and distilled water as a solvent.

Pre-treatment of natural clay

The procedure for the pre-treatment of the natural clay was previously reported [31]. Briefly, the milled clay samples were sieved using a 2-micrometer sieve, and their particle size was further reduced to fine powdered form with the aid of a ceramic plate and mortar. The powdered samples obtained were then calcined in a muffle furnace at 400 ^oC for 3 hours.

HCl-modified natural clay

To better understand the effect of HCl acid concentration on the surface properties of Omilafara clay, the clay samples were subjected to the same reaction conditions (weight of sample, temperature, time, and calcination temperature) employed in our previous study for the treatment of the clay at lower (5% and

15%) concentrations of HCl [31]. This method was adopted to allow for data comparison. Into a round-bottom flask containing 200 ml of 25% and 35% HCl solutions, respectively, 5 g of the calcined clay sample was added. The resulting red coloured mixture was stirred mechanically for 24 hours and then left to settle for 30 minutes; thereafter, the clear top solution was decanted. Using water, the residue was washed repeatedly until there was no trace of chlorine in the filtrate upon the silver nitrate test. The washed samples were then air-dried overnight and calcined at 400 °C for 3 hours. For the basis of comparison, 5 g of the unmodified clay sample was calcined at 400 °C for 3 hours without any prior chemical modification, and the data obtained from the unmodified sample were on par with that of our previous study [31].

Characterization

The HCl-modified samples were characterized by Fourier transform infrared spectroscopy (FTIR) with spectra recorded on the Agilent Cary 630 FT-IR spectrometer, while powder XRD patterns were recorded on the Rigaku Miniflex 600 X-ray Diffractometer, and peaks were assigned using the table of key lines of X-ray diffraction of clay minerals compiled by Pei-Yan Chen in accordance with the Joint Committee on Powder Diffraction Standards (JCPDS) data card numbers [32]. The specific surface area, surface area, and pore volume of the samples were obtained from N_2 adsorption isotherms, which were recorded on a Quantachrome Bet Nova 4200e surface area analyzer. The JSM-7600F Schottky Field Emission Scanning Electron Microscope (SEM) provided information about the morphologies of the HCl-modified samples, and the elemental composition of the samples was determined using the SEM analyzer coupled with an energy dispersive X-ray analyzer (EDX).

Surface acidity measurement via pyridine adsorption experiment

The surface acidity of the unmodified and acid-modified clays (5%, 15%, 25%, and 35% HCl) was determined using pyridine adsorption FTIR spectroscopy as described by Reddy et al. [33]. In this procedure, 0.5 g of each clay sample (modified and unmodified) was first oven-dried at 100 °C for 1 hour prior to pyridine treatment. Approximately 0.1 mL (1 drop) of pyridine was allowed to come in contact with each sample and thereafter subjected to heat treatment at 100°C for approximately 1 h to eliminate physisorbed pyridine. After cooling, the adsorbed pyridine on the surface of the clays was analyzed using FTIR spectroscopy.

Results and Discussion

XRD Analysis

The serpentine clay group seen in the lizardite form is characterized by the reflection peaks at 2θ values of approximately 24°, 43°, and 60°, respectively, in the XRD for 25% and 35% HCl-modified clay (Figures 1a and b). The assignment of these peaks and the classification of the clay as serpentine are consistent with previous studies on Omialafara clay [29, 31] as well as those reported by Rivero Crespo *et al*. [34]. Furthermore, the presence of silica (quartz) in the clay mineral is identified by the basal reflection peaks at 2θ values of approximately 21.22°, 26.98°, and 50.36° respectively, for 25% HCl-modified and 21.32°, 27.02°, and 50.58°, respectively, for 35% HCl-modified. The data are on par with the diffraction patterns previously reported for the unmodified clay calcined at 400 °C [31]. The notable difference for the HCl-modified clay samples at different acid concentrations is the basal peak intensity at an approximate 2θ value of 27° for quartz that increased significantly with an increase in acid concentration (Figures 1a and b). It is important to state that in the mineralogical assay of Omialafara clay conducted by Irabor and Anaun [29], they detected the presence of the rare organic mineral evenkite, which typically has a chemical composition of $C_{23}H_{48}$ [35]. In their investigation, they attributed, in part, the high values for the volatile materials (loss on ignition) to the presence of evenkite. The reflection peaks for evenkite were not detected on the X-ray diffractogram, perhaps due to the structural distortions of the hydrocarbon mineral that may have occurred due to thermal (400 °C) and acid treatments.

FTIR analysis

Figures 1a and b show the FTIR of natural clay samples modified by 25% and 35% HCl. Both spectra showed a characteristic broad band between 3690 cm^{-1} and 3300 cm^{-1} for an O-H stretch, typical of clay minerals [36]. The hydroxyl functional groups are present in the silica-tetrahedral sheet and alumina-octahedral sheets and are usually linked together by water molecules [36]. The absorption bands at 1051 cm^{-1} and 1047 cm^{-1} are attributed to the Si-O bond for 25% and 35% HCl-modified clay samples, respectively (Figures 1a and 1b). The signal is typical of clay minerals due to the presence of silicate in the framework. Furthermore, the absorption bands at 775 and 693 cm^{-1}, respectively, are assigned to the Si-O symmetrical stretching and bending frequencies. These FTIR data are consistent with previous studies on clay minerals [8, 31]. Comparing the FITR spectra of the acid-treated clay with that of the untreated clay (Figure 2c) from a previous study [31], the notable difference is the flattening out of the O-H vibration between ~3690 and ~3300 cm^{-1} and the Al-OH band at 913 cm^{-1} for the acid-treated clay samples as shown in Figures 2a, b, and c. These changes are indicative of stronger Si-O bonds due to the removal of interfering species (loss of water or hydroxyl groups) and a possible dissolution of Al^{3+} from the octahedral sheets in the crystal lattice of the clay minerals [8].

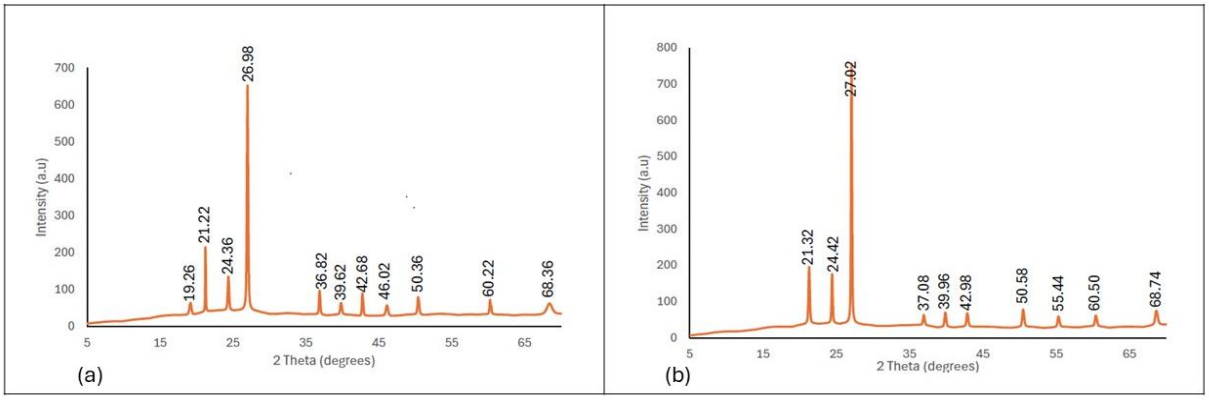


Figure 1. XRD diffractogram of: (a) 25% HCl-modified clay (b) 35% HCl-modified clay.

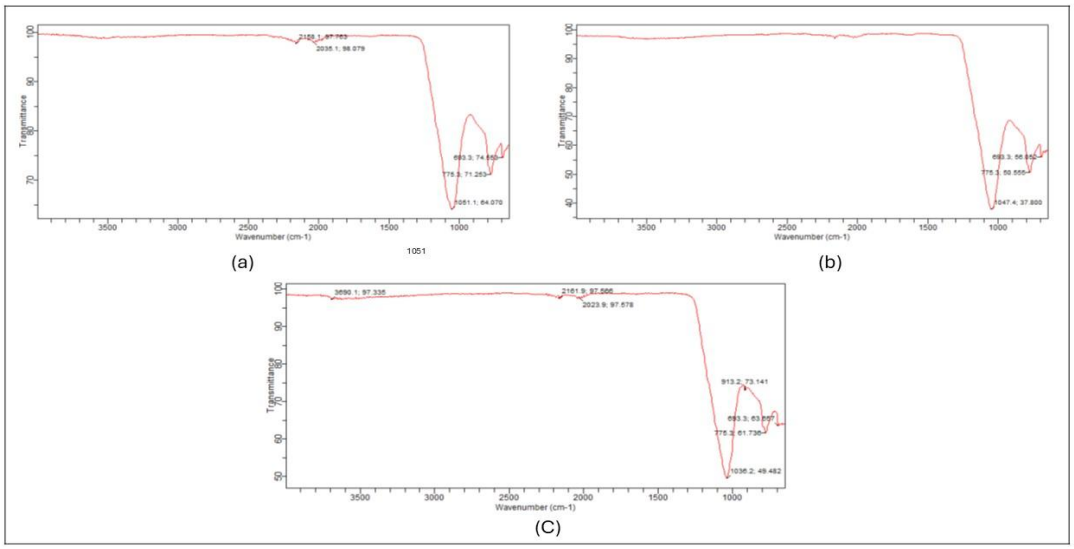


Figure 2. FTIR Spectra of: (a) 25% HCl-modified clay; (b) 35% HCl-modified clay; (c) unmodified clay adopted from Omoruyi et al., 2025

Table 1. Surface parameters of unmodified and HCl-modified natural clay calcined at 400 °C.

Sample	Specific Surface Area (m^2/g)	Pore volume (cm^3/g)	Pore Size (nm)	Ref
Untreated clay	345	0.198	2.132	Omoruyi *et al*. [31]
5% HCl-modified clay	285	0.172	2.138	Omoruyi *et al*.[31]
15% HCl-modified clay	359	0.181	2.100	Omoruyi *et al*.[31]
25% HCl-treated clay	450	0.235	2.104	This work
35% HCl-treated clay	528	0.264	2.125	This work

BET Analysis

The SSA of clay minerals is one way of measuring its adsorption capacity, and this property is intrinsically linked to the cation exchange capacity (CEC) of the clay [17]. The higher the SSA values, the higher the CEC of the clay minerals. The SSA for the HCl-modified Omialafara clay sample calcined at 400 °C was calculated using the Brunauer-Emmett-Teller (BET) method, while the pore volumes and pore sizes (diameter) were estimated using the Barret-Joyner-Halenda (BJH) method described in the literature [37]. The surface parameters of 25% and 35% HCl-modified clay samples were compared to those of unmodified clay as well as the 5% and 15% HCl-modified clay samples previously reported by our group [31]. Table 1 provides an overview of the surface characteristics of the unmodified and the HCl-modified clays. The treatment of the parent clay with 25% and 35% HCl, respectively, led to a significant increase in the SSA of clay samples relative to the unmodified clay, with an increase of approximately 30% and 50%, respectively (Table 1), an indication that structural changes occurred within the clay matrix. The increase in SSA upon acid treatment suggests a possible replacement of the interlayer cations (CEC) with

protons from the acid, leading to partial dissolution of the clay matrix. In addition, the possible leaching of structural Al^{3+} (dealumination) or Fe^{3+} or any other cation present within the octahedral sheets has also been attributed to an increase in porosity [8, 38]. Furthermore, structural changes may occur as a result of the protonation of hydroxyl groups within the octahedral sheets of the clay mineral, which may result in changes in Al^{3+} coordination [17]. A graphical representation of the effect of different HCl concentrations (0%, 5%, 15%, 25%, and 35%) on the SSA of Omialafara clay is shown in Figure 3. Except for 5% HCl-modified clay, which showed a decrease of 17% in comparison with the unmodified (0% HCl) clay [31], all other acid treatments led to a steady increase in the SSA with an increasing HCl concentration. The pore sizes of the unmodified and HCl-modified clay samples remain considerably constant at an average of 2.120 nm. However, the pore volumes changed upon HCl treatment following a similar trend to that of the SSA (Table 1). In a related study, Krupskaya *et al*. [17] stated that structural changes arising from the leaching of interlayer cations and changes in Al^{3+} coordination upon acid treatment lead to the generation of porous structures within the modified clay that do not necessarily have an impact on pore sizes but rather on the SSA and pore volumes as observed in this study. The extent of the structural changes that occur upon acid treatment was highlighted by Barakan and Aghazadeh [5] and is influenced by the nature of the acid, acid concentration, contact time, and temperature. The data from the BET analysis of Omialafara clay show that a correlation exists between the increase in SSA, pore volume, and the concentration of the acid employed in the modification of the natural clay.

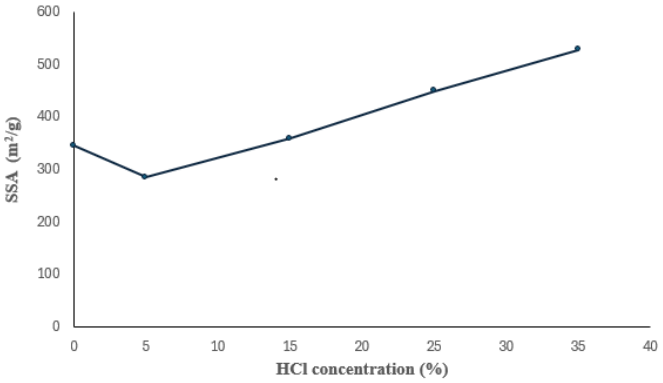


Figure 3. A plot of the acid concentrations against specific surface area (SSA).

SEM-EDX Analysis

Figure 4 displays the scanning electron microscopy (SEM) pictures of 25% and 35% HCl-modified natural clay samples, which provide information regarding their surface characteristics at 100 µm. The surface morphologies of the SEM micrographs of 25% and 35% HCl-modified natural clay calcined at 400°C differ from that previously published for the parent (unmodified) clay sample calcined at 400°C [31]. By comparison, the SEM micrograph for the 25% HCl-modified clay (Figure 3) shows the presence of an agglomerated surface with larger pore sizes relative to the SEM image for the unmodified clay. In contrast to the 25% HCl-modified clay and the unmodified clay samples, the micrograph of the 35% HCl-modified clay displays a rough, flake-like surface structure with larger pore sizes. Figure 5 provides a graphical summary of the pertinent elemental compositions of the acid-modified (5%, 15%, 25%, and 35% HCl) and unmodified (0% HCl) clay acquired from the energy-dispersive X-ray analyzer (EDX). Carbon (C), oxygen (O), aluminum (Al), and silicon (Si) were the most common elements found in large amounts (≥ 4%) for the HCl-modified (25% and 25%) clays, while elements like calcium (Ca), iron (Fe), and sodium (Na) were either found in small amounts (< 4%) or trace amounts (< 0.5%). The reduction of sodium from 3% for the unmodified clay to trace quantities for 25% HCl-modified clay, which was below detection limits for 35% HCl-treated clay, was one significant difference between the elemental composition of the HCl-modified (25% and 35%) clays and the unmodified clay (Figure 5). Additionally, there was a slight decrease in Fe from 3.4% for the unmodified clay to 3.0% for the 25% and 35% HCl-modified clay, respectively. These data support the evidence from the BET analysis that the structural alterations observed were caused by the leaching of structural and interlayer cations in the clay mineral's octahedral sheets. Furthermore, the data from 5% HCl and 15% HCl treatment of the clay [31] showed that upon treatment of the clay with 5% HCl, a significant amount (4%) of magnesium (Mg), previously not detected in the unmodified clay, perhaps due to the presence of impurities, was detected. There was a reduction in Mg content from 4% to 1.02% when the clay was treated with a higher acid concentration of 15% HCl (Figure 5). Notably, Mg was below the detection limit for 25 and 35% HCl-modified clays. The observed reduction in the composition of some elements, leading to the observed structural changes, is in concordance with the literature [8, 17, 8, 39].

A plot of the Si/Al ratios of the natural clay with the different concentrations of acid treatment is depicted in Figure 6. The Si/Al ratio of 5.58 for the untreated clay (0% HCl) decreased gradually with increasing acid concentration for the treated clays until 35% HCl treatment, where an increase was observed, resulting in an Si/Al ratio of 5.35. The data suggests that structural changes may have occurred from leaching of the interlayer cations of the clay with little or no dealumination occurring at lower concentrations of the acid, while at higher concentrations, dealumination may have contributed to the overall structural changes of the clay. Si/Al ratios have a direct impact on the SSA and surface acidity of the treated clay. Previous studies have shown that high Si/Al ratios for acid-treated aluminosilicate materials result from low Al content due to dissolution of Al, leading to an increase in SSA. In contrast, low Si/Al ratios indicate high Al content and low SSA [36, 40]. This trend was also observed in this study except for 25% HCl-modified clay, whose Si/Al ratio was lower than that of the unmodified sample but had a higher SSA value compared to the parent clay.

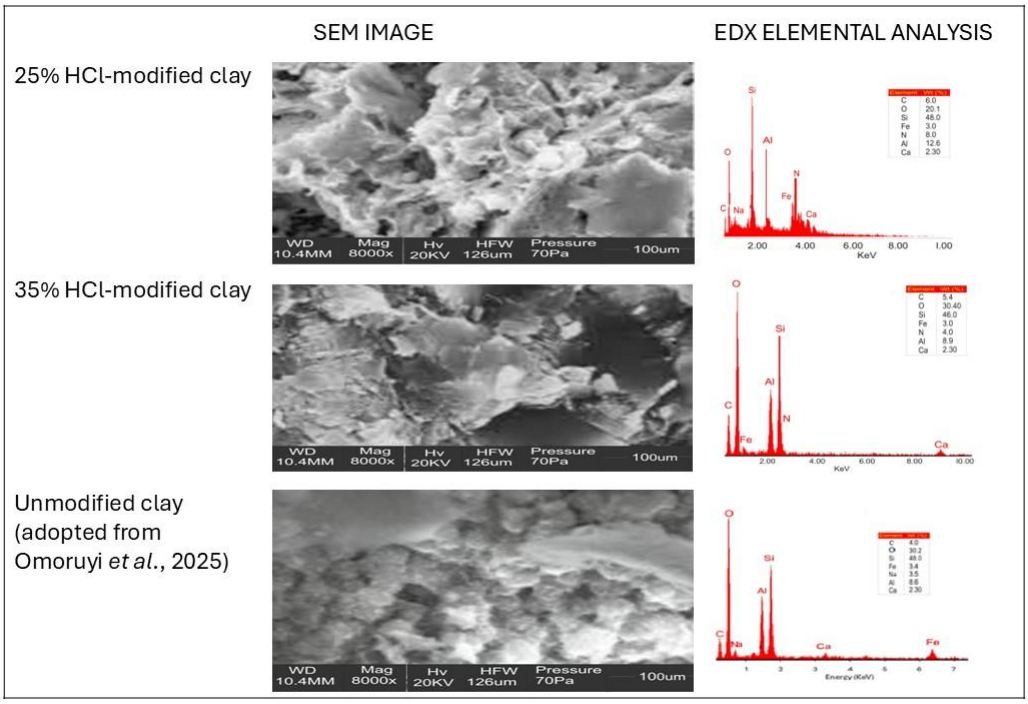


Figure 4. SEM-EDX of HCl-modified and unmodified natural clay

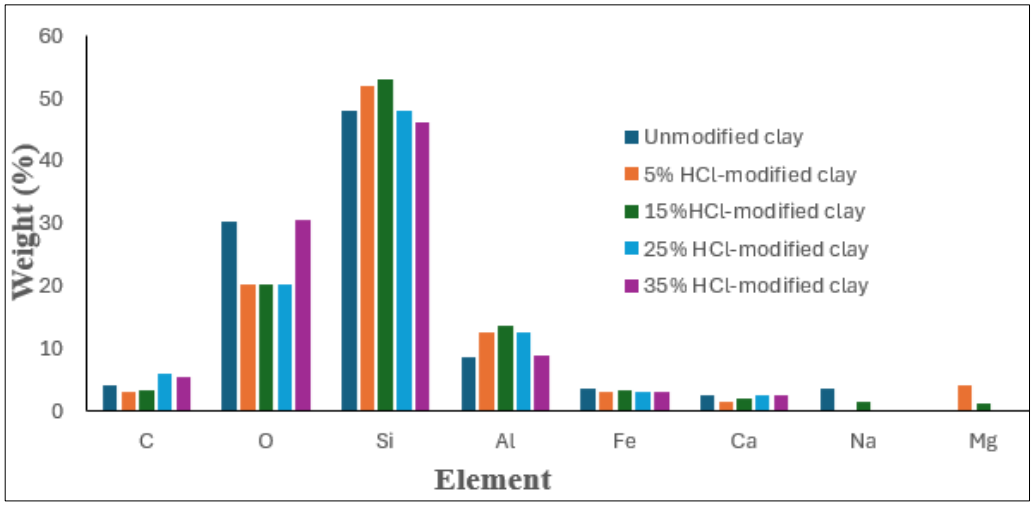


Figure 5. Selected elemental compositions of HCl-modified and unmodified natural clay from EDX analysis.

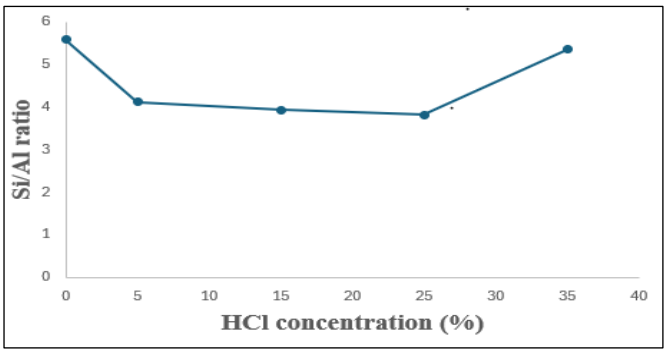


Figure 6. Plot of acid concentrations against Si/Al ratios

Surface acidity

The surface acidity of aluminosilicate materials is composed of Lewis acid sites (LAS) and Brønsted acid sites (BAS), which are easily differentiated by FTIR spectroscopy in the presence of a probe molecule. For the first time, the surface acidity of Omialafara clay (modified and unmodified) was investigated using pyridine as the probe molecule. Figure 7 shows the FTIR spectra obtained from the acidity measurement of the unmodified (0% HCl) and HCl-modified (5%, 15%, 25%, and 35%) clays. The band at 1580 cm^{-1} is attributed to the pyridinium ion ($C_5H_6N^+$), which results from the protonation of the pyridine probe molecule by the BAS of the clay minerals.

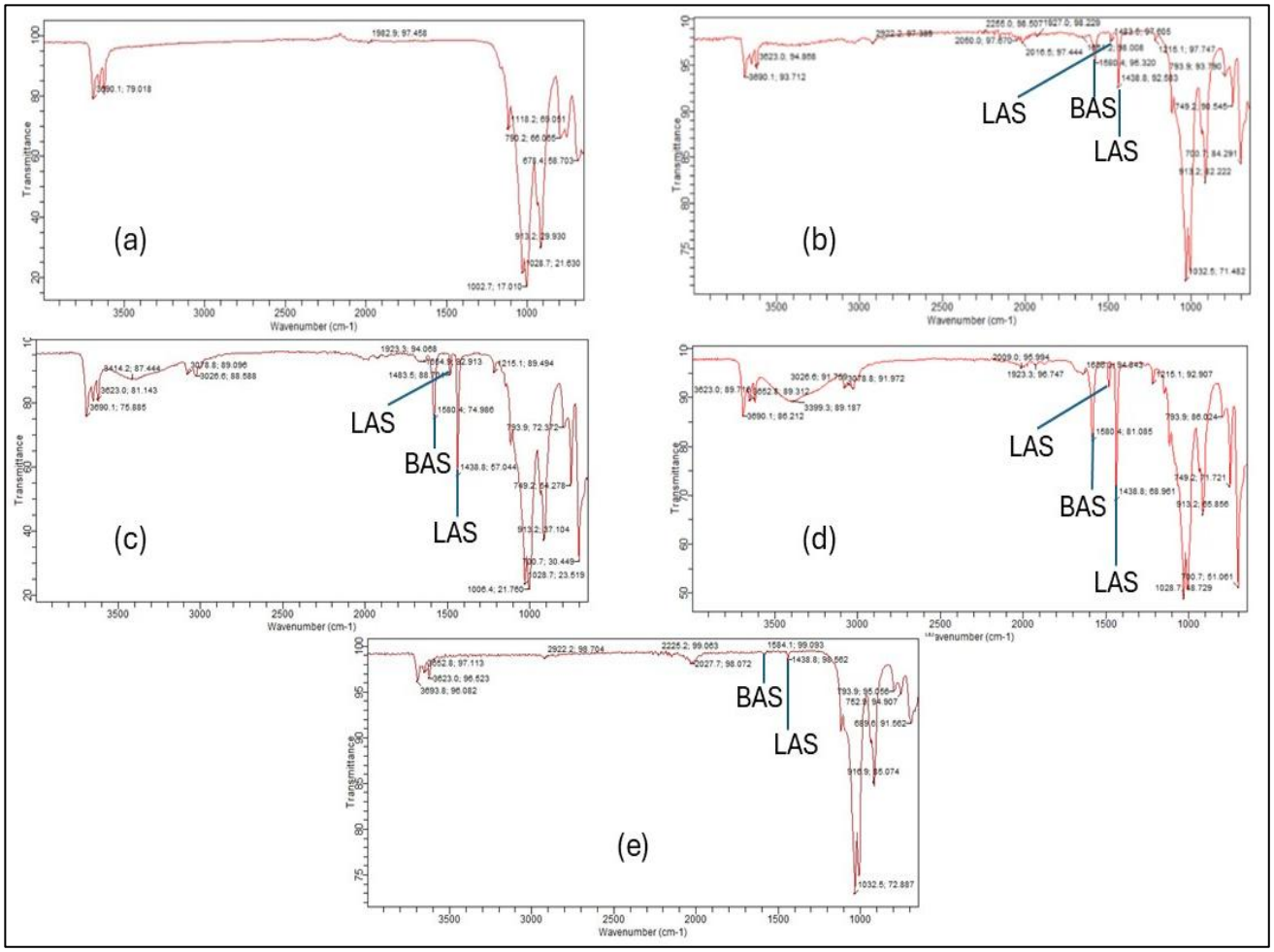


Figure 7. FTIR pyridine adsorption spectra of: (a) unmodified clay, (b) 5% HCl-modified clay, (c) 15% HCl-modified clay, (d) 25% HCl-modified clay, and (e) 35% HCl-modified clay, showing Lewis acid sites (LAS) and Brønsted acid sites (BAS).

The bands between 1438 and 1483 cm^{-1} correspond to LAS on the clay material as a result of pyridine (Lewis base) coordination [33]. It can be clearly seen that the predominant acid sites are the Lewis acids. The clay treated with 15% and 25% HCl, which had the lowest SI/Al ratios (3.93 and 3.81, respectively), was found to have strong adsorption peaks for BAS and LAS, respectively, indicating high acidity. The intensities of these peaks decreased significantly for 5% and 35% HCl-modified clay with elevated Si/Al ratios. The increased Brønsted and Lewis acid sites at low Si/Al ratios are attributed to the high Al content, which requires more protons (cations) to balance the charges, and the presence of coordinatively unsaturated cationic metal sites (Lewis acid sites) in the clay minerals [33]. The findings are in good agreement with the study on aluminosilicate materials conducted by Lara Pardo *et al.* [40], where the Si/Al ratios equally showed an indirect proportionality to surface acidity. It is important to state that the BAS and LAS were not detected in the unmodified clay with the highest Si/Al ratio of 5.58.

Conclusion

Natural clay obtained from Omialafara, Ondo State, Nigeria, was structurally modified with different concentrations of HCl and calcined at 400 °C. The structural changes that occurred in the natural clay before and after the acid treatments were thoroughly investigated by different instrumental techniques such as powder XRD, FTIR, BET, and SEM-EDX. The surface acidity of the clays was measured via pyridine adsorption FTIR spectroscopy. XRD analysis revealed that the clay mineralogical composition did not change significantly upon acid treatment. BET and SEM analysis revealed some structural changes for acid-modified clays in comparison to the unmodified clay. SSA, pore volumes, and Si/Al ratios increased with increasing acid concentration upon treatments. Treatment of the natural clay with a 35% HCl solution resulted in the highest SSA value of 528 m^2/g making it a potentially useful material for adsorption studies. The 25% HCl-modified clay with the lowest Si/Al of 3.81 displayed the most intense adsorption bands for BAS and LAS, indicating significant surface acidity, which is crucial for catalytic research. This study concludes that structural changes occurred upon acid modifications, and the changes varied

significantly with increasing acid concentrations. The data obtained from the surface acidity measurement revealed that the acid-modified clays exhibited better Brønsted and Lewis acidities compared to the unmodified clay. Additionally, the SSA, pore volumes, and surface acidity were dependent on the Si/Al ratios.

Author Contributions

UO -Conceptualization (lead), Methodology (lead), Writing – original draft (lead). UO and CAU- Final draft Both authors then critically reviewed the following versions. Both have read and approved the final version of the paper and its submission and have given consent for publication.

Declaration of Generative AI and AI-Assisted Technologies in Scientific Writing

Free AI grammar checker (Scibbr)- Used to check grammar, punctuation and syntax error.

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Data Availability Statement

All relevant data are available within the manuscript

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None

Conflict of Interest

The authors declare that they have no conflicts of interest.

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