

Effects of thermal and chemical modification on the physical properties of Ugandan Mutaka Kaolin

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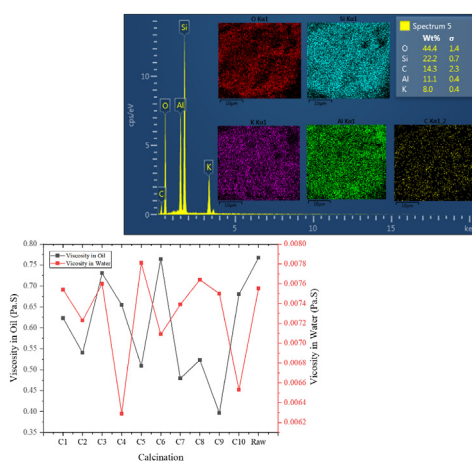
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HIGHLIGHTS

- Calcined kaolin exhibited lower specific gravity compared to TiO₂.
- Classified samples with pH ≤ 4.0 or ≥ 9.0 as corrosive.
- Optimal oil absorption seen at 800–900 °C due to favorable metakaolin properties.
- Chemical concentration affected kaolin dispersion, differentiating it from calcined kaolin.

GRAPHICAL ABSTRACT



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ABSTRACT

This study investigates the impact of thermal treatment at temperatures ranging from 600 °C to 1050 °C and chemical treatment using (COOH)₂·2H₂O and Al₂(MoO₄)₃ at concentrations of 0.01 M, 0.05 M, and 0.1 M. The modified kaolin samples' pH, oil adsorption capacity, refractive index, specific gravity, and viscosity were examined. Comprehensive analyses were performed to characterize the modified kaolin samples. The spectrum results revealed dealumination, with a corresponding increase in silicon content due to chemical treatment, while the aluminum content decreased compared to thermal treatment results. As observed with the calcined kaolin sample, a significant portion of the OH stretch groups vanished with disappearance stretches along the bands at 1229.6 and 1009.2 cm⁻¹, corresponding to Si–O stretching vibrations. The specific gravity of calcined kaolin was observed to be relatively lower than TiO₂. Furthermore, the obtained pH of 4.0 or lower, or a pH of 9.0 or higher, is classified as corrosive. The ideal temperature range for achieving optimal oil absorption lies within the 800 °C–900 °C range, where metakaolin properties favor effective oil uptake. The chemical concentration had a notable impact on the dispersion of kaolin powders, in contrast to calcined kaolin. At 800 °C, calcined kaolin attained an almost ideal refractive index for water-based paints, closely aligning with the refractive index of water.

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1. Introduction

Kaolin, a versatile clay mineral with widespread applications in various industries [1,2], has garnered significant attention due to its unique physico-chemical properties. Among the various varieties of kaolin, the Ugandan Mutaka kaolin stands out as a promising natural resource with considerable potential [3]. Its abundant availability and favorable mineral composition make it an attractive candidate for exploring novel modifications that can enhance its physical properties and widen its range of applications [4,5].

The physical properties of kaolin are strongly influenced by its crystal structure, particle size, surface area, and surface charge [6], which are, in turn, dictated by its geological origin and processing techniques. For decades, researchers and industries have been exploring ways to modify kaolin to tailor its properties for specific uses [7–9]. Among these modifications, thermal and chemical treatments have emerged as promising methods to induce desirable kaolin structure and property alterations. While kaolin pigments offer cost-effectiveness, their properties can be further enhanced through modification or treatment to meet specific application requirements [6]. However, impurities associated with raw kaolin often limit its utility in producing kaolin-based products [10]. Consequently, modification processes, such as chemical and thermal treatments, must be explored more to improve properties like viscosity, solid content, pH, sedimentation, oil adsorption capacity, refractive index, and specific gravity [11].

Additionally, few studies have focused on analyzing these properties of kaolin. Analyzing these properties can provide important insights into the physical properties of kaolin and its potential applications in paint, coating, and other industrial applications [1,2]. Among the literature, Chandrasekhar and Ramaswamy [12] conducted an analysis focusing on optical properties, pH, true and apparent densities, as well as the particle size distribution of calcined kaolin. Similarly, Cheng [13] conducted research that revealed how insufficient or over-calcination negatively impacts the oil absorption performance of calcined kaolin. However, Zewdie et al. [2] conducted a comprehensive investigation into the impact of acid concentration, reaction temperature, and contact time on iron extraction. Their findings revealed a direct correlation between oxalic acid concentration, leaching temperature, and contact time, leading to an increased rate of iron extraction. Additionally, the utilization of calcined kaolin in composites has been explored, with findings indicating improved mechanical properties compared to raw kaolin [14]. Moreover, in the production of calcined kaolin, the soluble Al_2O_3 content is a critical quality control criterion for certain specialty applications [15].

Despite the increasing interest in kaolin modification worldwide, a significant knowledge gap concerning the effects of thermal and chemical treatments remains, especially on Ugandan Mutaka kaolin's physical properties. This study aims to provide essential insights into the physical properties of Ugandan Mutaka Kaolin and the effects of thermal and chemical modification on these properties. The results of this investigation are poised to contribute significantly to the field, paving the way for advancements in kaolin utilization across various industries, particularly in paint and coating applications. The specific objectives of this study are as follows:

1. To investigate the effect of thermal and chemical modification on the pH, oil adsorption capacity, refractive index, specific gravity, and viscosity of Ugandan Mutaka Kaolin.
2. To compare the physical properties of Ugandan Mutaka Kaolin after thermal and chemical modification to identify the most effective method for modifying its properties.

2. Experimental

2.1. Materials

The kaolin minerals in this study are based in the southwestern region of Uganda, specifically from the Mutaka Kaolin deposit in the Mitoma district (which was cut off from the former Bushenyi District). The extraction of minerals and experimental analysis were conducted in Uganda's southwestern and central parts, respectively. The study involved the assistance of an expert in materials to help select kaolin mineral samples.

2.2. Treatment

2.2.1. Ammonium molybdate and oxalic acid

Three solutions with varying concentrations of oxalic acid and ammonium molybdate were prepared, each having concentrations of 0.01 M, 0.05 M, and 0.1 M, as shown in Table 1. These solutions were prepared based on the respective molecular weights of the two compounds. The mixture of each solution was carried out in a 500 ml Pyrex flask and stirred at 400 rpm for 20 min using a magnetic stirrer at 23 °C. After mixing the kaolin and solutions, the resulting mixture was transferred into Falcon tubes. The tubes were then centrifuged using a Megafuge 8 centrifuge at 4500 rpm for 10 min [2]. To remove unspent solutions and other impurities, the residue was washed repeatedly with distilled water. Then, the kaolin sample was dried at 100 °C for 24 h.

2.2.2. Calcination

The calcination process of kaolin was meticulously performed in a state-of-the-art muffle furnace, specifically the Carbolite Model CWFT315. This advanced furnace allowed precise temperature control, enabling the experiment to cover a wide range of temperatures, from 600 °C to 1050 °C, as shown in Table 1. The temperature intervals of 50 °C were carefully selected to capture crucial changes in the kaolin's properties at different calcination stages. The calcined and raw kaolin were then analyzed using Atomic Absorption Spectrometry (AAS) to get the chemical composition of kaolin samples before and after calcination (see Table 2).

2.3. Characterizations

2.3.1. Energy dispersive X-ray spectroscopy (EDX) analysis

The elemental compositions of the kaolin samples were thoroughly analyzed, and the obtained morphologies were mapped using EDX.

Table 1
Thermal and chemical treatments of kaolin.

Identification code	Type of treatment	Description
C1	Thermal treatment	600 °C
C2		650 °C
C3		700 °C
C4		750 °C
C5		800 °C
C6		850 °C
C7		900 °C
C8		950 °C
C9		1000 °C
C10	1050 °C	
AM1	Ammonium molybdate treatment	0.01 M
AM2		0.05 M
AM3		0.1 M
OA1	Oxalic acid treatment	0.01 M
OA2		0.05 M
OA3		0.1 M

Before analysis, a carbon-coating process using the Agar Turbo Carbon coater was employed to enhance material conductivity. Subsequently, the energy dispersion technique was applied to identify individual elements and discern any lateral variations in composition from specific selected regions.

2.3.2. Fourier transform infrared (FTIR) analysis

Functional group analysis of the samples was performed using a Shimadzu FTIR spectrometer model 4100. The FTIR analysis was conducted in the solid state, where small fragments of each silica sphere were powdered and deposited on the attenuated total reflectance system for data acquisition, as described in Refs. [16,17]. The analysis covered a wavenumber range from 4000 cm^{-1} –500 cm^{-1} .

2.4. Physical properties

2.4.1. Specific gravity

The ASTM D 1475–13 standard procedure was employed for determining the density of the kaolin sample in relation to the density of water. This method provides vital insights into the quality and consistency of kaolin, essential for its various industrial applications. Gravimetric analysis, using the analytical scale 9T202 (Adam Germany), was employed to measure the sample's density in this study. Approximately 5 g of the kaolin sample was accurately weighed and transferred into a falcon tube. The sample volume was then recorded after carefully packing it in the falcon tube until no further changes in volume occurred.

2.4.2. pH

Five grams of the sample was weighed in duplicate and placed into separate falcon tubes. Subsequently, freshly boiled 100 g of warm distilled water to eliminate carbon dioxide was added to each falcon tube. The mixture was allowed to soak for 20 min and was then vigorously centrifuged at 4500 rpm for approximately 5 min. After centrifugation, the solution was allowed to settle and cool to 25 °C. The clear liquid was carefully decanted, and its pH was promptly determined using a suitable indicator.

2.4.3. Oil absorption capacity

The samples, weighing 2 g each, were combined with 6 ml of vegetable oil in pre-weighed centrifuge tubes. The contents were stirred for 1 min using a thin brass wire to ensure proper sample dispersion in the oil. Following a 30-min holding period, the tubes were centrifuged at 3000 rpm for 25 min. After centrifugation, the separated oil was carefully decanted, and the tubes were inverted for a specific duration to allow the oil to drain before being weighed, as described in ASTM D281-12 methods.

2.4.4. Viscosity

Kaolin dispersions in water were prepared using ball milling with alumina balls. Viscosity and rheological behavior were evaluated using a Viscometer (BROOKFIELD, USA) at a controlled temperature of 26 °C, as described in Ref. [18]. All measurements were conducted with a temperature controller to maintain consistency.

2.4.5. Refractive index

The refractive index is crucial in differentiating between a material functioning as a pigment or a filler. This distinction is often evident from

the optical characteristics of the substance when integrated into a coating or paint material. The method described in Field [18] was employed to ascertain the refractive index in this research.

3. Results and discussion

3.1. Characterization

3.1.1. EDX analysis

Fig. 1(a)–(c) shows the EDS analysis results, elemental compositions in the kaolin sample after undergoing treatments with thermal treatment, and ammonium molybdate and oxalic acid. As depicted in Fig. 1(a)–(c), the EDS analysis of the kaolin samples revealed the presence of oxygen (O), aluminium (Al), silicon (Si), carbon (C), and potassium (K) elements. Well-formed stacks of kaolinite layers were observed in the samples, with the major elements being O, Al, and Si, accompanied by minor amounts of C and K. The spectrum results indicated de-alumination, with a corresponding increase in silicon content due to chemical treatment, while the aluminum content decreased compared to thermal treatment results. This finding was consistent with the research by Ref. [19]. Despite the similar morphologies exhibited by the kaolin samples, there were variations in the EDS results presented in Fig. 1(a)–(c). Additionally, a small amount of trace elements was recorded for the mapped area in EDX, possibly originating from impurities associated with dickite during its formation due to feldspar weathering, as observed by Ref. [20]. Understanding these compositional changes is crucial for optimizing kaolin as a paint component. Altered elemental ratios can influence properties like color, adhesion, and durability in paint formulations.

3.1.2. FTIR analysis

Fig. 2(a) and (b) depicts the FTIR spectra of the uncalculated kaolin sample and calcined kaolin samples. As observed with the calcined kaolin sample, a significant portion of the OH stretch groups vanished, as shown in Fig. 2(a). Additionally, disappearance stretches are evident in Fig. 2(a), along with the bands at 1229.6 and 1009.2 cm^{-1} , corresponding to Si–O stretching vibrations. These structural modifications can impact the interaction between kaolin and other paint components, influencing properties like viscosity, dispersion, and adhesion [21]. The FTIR results show that the OH stretching vibrations are observed in the wavenumber range of 3710 to 3550 cm^{-1} (see Fig. 2(b) and (c)). This alteration of the sample structure was due to the modified treatment. Notable peaks in Fig. 2(b) and (c) corresponding to the γ Si–O stretching vibrations of the sample are observed at 1103–1093 cm^{-1} , 980–976 cm^{-1} , and 867–846 cm^{-1} . These distinctive frequencies are characteristic of kaolinite's specific molecular vibrations. Furthermore, the spectrum of both samples displays a significant band at 605 cm^{-1} and 597 cm^{-1} , consistent with previous studies [2,22], suggesting the presence of quartz in the samples.

3.2. Properties of modified kaolin samples

3.2.1. Specific gravity

The specific gravity of kaolin, determined after calcination at 800 °C, was found to be 1.11, as illustrated in Fig. 3(a). This value is in proximity to both the specific gravities obtained at 600 °C, 700 °C, and 900 °C, as well as the specific gravity of water (1.0). The result shows that the specific gravity of calcined kaolin is notably lower than that of TiO_2 (4.26), the

Table 2

Chemical composition of raw and calcined kaolin.

Sample name	Fe (ppm)	Pb	Na	Ca	K	Hg	Al (%)	Si
Raw kaolin	226.48	50.56	458.41	765.26	365.32	14.96	21.15	22.99
Calcinated kaolin	210.10	46.15	1794.12	783.16	290.86	11.99	21.09	22.93

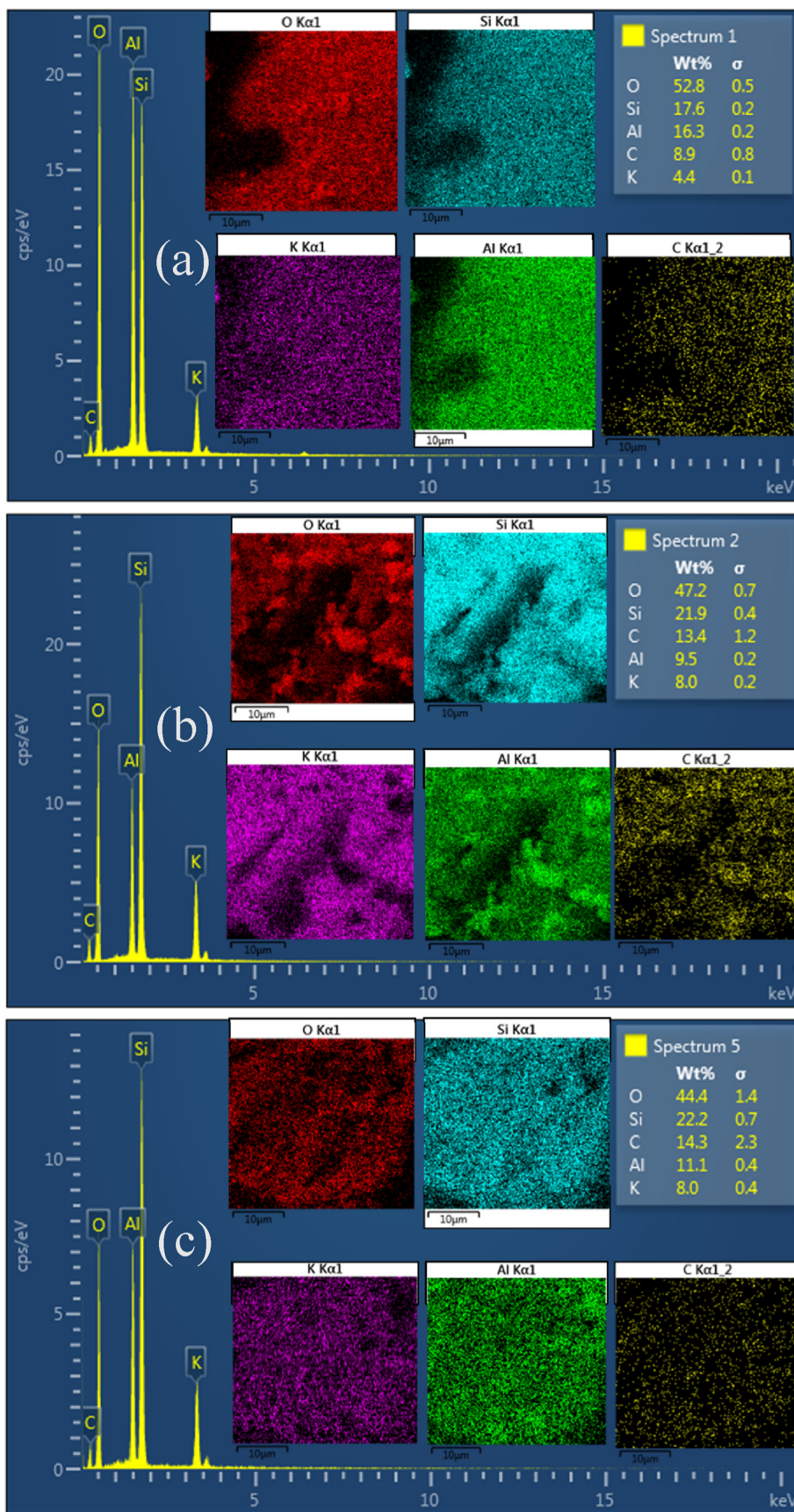


Fig. 1. EDX spectrum of kaolin samples treated with (a) temperature at 800 °C (b) aluminum molybdate (c) oxalic acid samples.

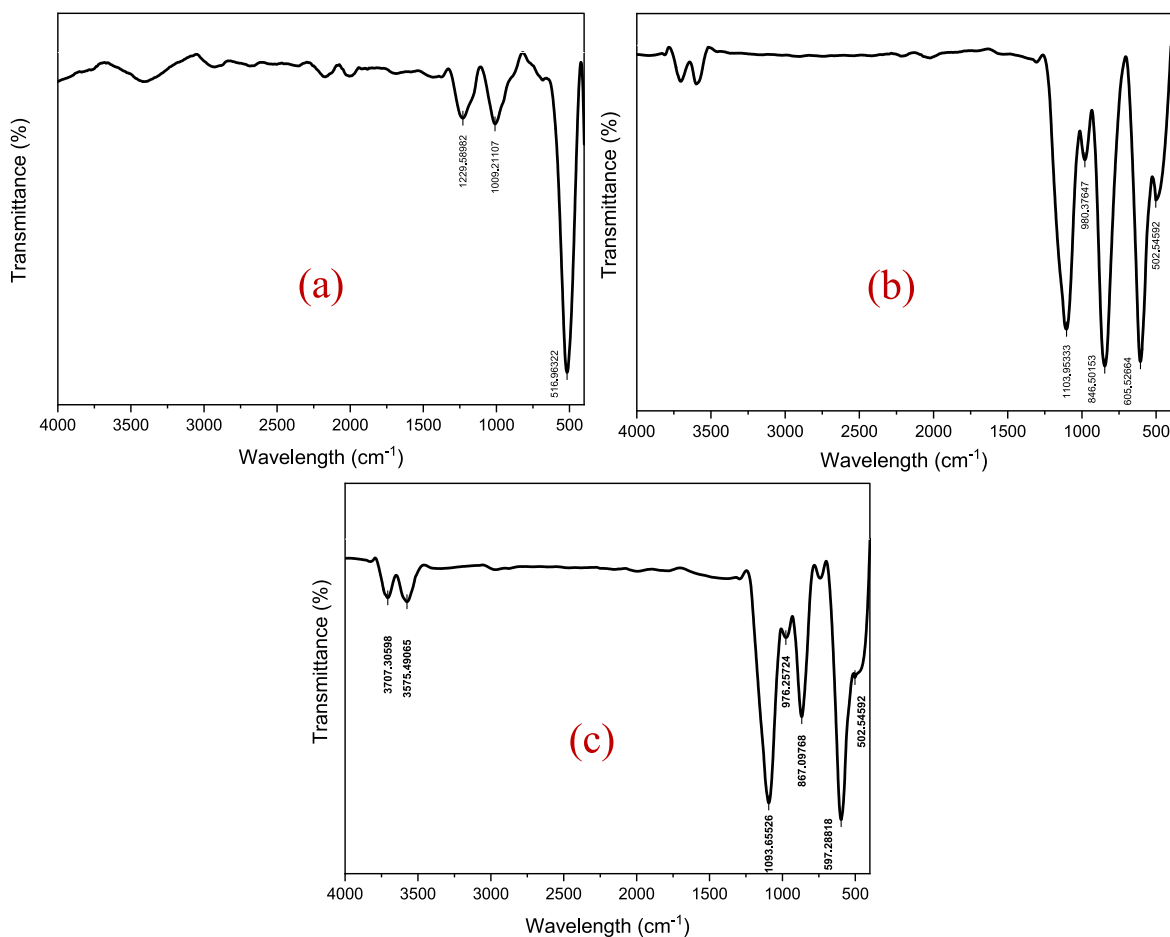


Fig. 2. FTIR results of kaolin from (a) calcination, (b) aluminum molybdate, and (c) oxalic acid samples.

primary pigment used in paint formulations. This characteristic is desirable in paint formulations, contributing to reduced settling tendencies and cost savings. Additionally, it was lower than the specific gravity values of extenders such as whiting (2.7), talc (2.65), and mica (2.8–2.85) [9]. The lower specific gravity of extenders presents a desirable characteristic in coatings formulations, leading to significant cost savings. Pigments with low specific gravity generally demonstrate reduced settling tendencies and exhibit high tinting strengths in paint fields [23].

As shown in Fig. 3(b), the specific gravity of chemically treated kaolin varies within the range of 1.43–1.69 for all concentrations 0.01 M, 0.05 M, and 0.1 M. This indicates that the filler’s density in water-based paint is significantly higher than that of calcined kaolin by more than 17%. A lower specific gravity in fillers results in reduced settling tendencies in paints and higher oil absorption capacity.

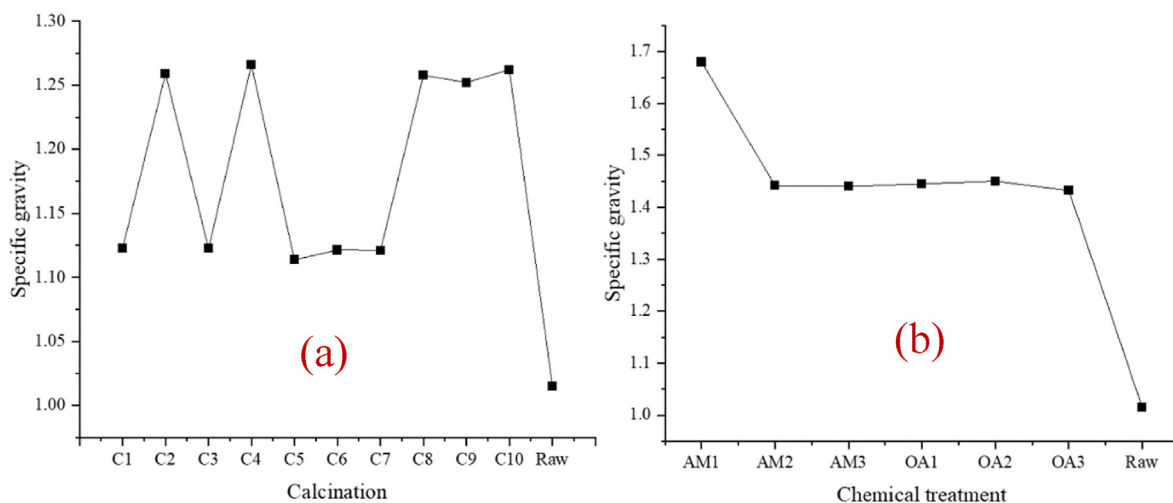


Fig. 3. Specific gravity of kaolin samples at different (a) temperatures (b) concentration.

3.2.2. pH

At higher temperature values, there was a significant decrease in pH values (see Fig. 4), corresponding to the increased specific gravity of calcined kaolin. This observation aligns with previous reports, where an increase in pH value has been shown to enhance the dispersion of kaolin powders by augmenting the negative charges on kaolin particles [24].

The pH of calcined kaolin undergoes a transition from alkaline to acidic as the temperature increases from 600 °C to 1050 °C, as displayed in Fig. 4(a). This change occurs due to increased reactivity during the transformation of kaolin from an amorphous substance to a crystalline metakaolin form, which exhibits higher reactivity up to 900 °C, beyond which mullite is formed. As a result, calcined kaolin at 1050 °C is unsuitable as a filler for paint due to its acidic nature, which could lead to surface corrosion upon application. For optimal performance and resistance to bacterial attack, the acceptable pH range for coating and paint filler lies between 7 and 9 [24]. To ensure compatibility with these requirements, the calcination temperature range of 600–850 °C with a pH range of 7.76–8.56 is recommended for the calcined kaolin used as a paint filler. This pH range offers a suitable environment for the filler's performance while avoiding adverse effects such as corrosion and bacterial susceptibility.

From Fig. 4(b), chemicals with a pH of 4.0 or lower or a pH of 9.0 or higher are classified as corrosive. Consequently, the pH values exhibited by the chemically treated fillers, with averages of 9.0 at respective concentrations of 0.01 M, 0.05 M, and 0.1 M for oxalic acid and ammonium molybdate, are not suitable for paint applications. To render them compatible with paints, additives would be necessary to reduce their corrosive nature. Thus, chemical treatment alone is insufficient in treating fillers if pH is the only parameter under consideration.

3.2.3. Oil absorption

From Fig. 5(a), the oil absorption behavior of kaolin follows a general trend of declining from 600 °C to 700 °C, during which the kaolin has not fully transformed into crystalline metakaolin. At 800 °C, the kaolin exists as metakaolin crystals that closely agglomerate, reducing oil absorption within its spaces. Beyond 800 °C, the oil absorption capacity starts to increase as further heating causes additional mass loss in the kaolin, creating more voids for oil absorption. However, heating beyond 900 °C results in fewer spaces available for oil absorption because the kaolin has transformed into mullite. As reported previously, insufficient and over-calcination can decrease the oil-absorption performance of calcined kaolin [13]. Therefore, the ideal temperature range for achieving optimal oil absorption lies within the 800 °C–900 °C range, where metakaolin properties favor effective oil uptake.

A low absorption of pigments indicates a reduced demand for the

product in paint formulations, leading to lower paint viscosity. A lower oil absorption can accommodate higher pigment loading and achieve denser packing. Conversely, pigments with higher oil absorption may decrease gloss, adhesion, corrosion resistance, and durability. Therefore, filler calcined at 800 °C would require the least amount of resin per unit during paint production, making it an ideal temperature for filler production. Fig. 5(b) shows that the oil absorption increases with an increase in chemical concentration. Although the oil absorption of chemically treated samples is much lower than that of calcined samples, the reduced oil absorption by an extender implies lower resin demand without compromising other coating properties [9].

3.2.4. Viscosity

The viscosity of kaolin at all calcination temperatures exhibits low values in water, with most of them being less than 0.01 Pa s, particularly at a minimum of 750 °C (see Fig. 6(a)). Based on these findings, to minimize the impact of calcination on kaolin's viscosity, C3 was chosen as the optimal viscosity level for further investigations. However, it is worth noting that inconsistent viscosity results were observed with calcined kaolin. This behavior can be attributed to the time-dependent structural transformation in the dispersion caused by shear perturbations [18].

The chemical concentration had a notable impact on the dispersion of kaolin powders, in contrast to calcined kaolin. As depicted in Fig. 6(b), the viscosity in the oil of kaolin treated with aluminum molybdate increased progressively with rising concentration, reaching approximately 0.85 Pa s. This finding aligns with a study by Ref. [25], where higher concentrations of silica particles formed a network, slightly elevating viscosity. On the other hand, the viscosity in water exhibited a gradual decrease with increasing concentration content, reaching below 0.005 Pa s, followed by a rapid increase.

3.2.5. Refractive index

As depicted in Fig. 7(a), most of the calcined kaolin samples displayed lower refractive indices in oil than in water. However, two specific samples, C7 and C9, stood out as reliable fillers with the most suitable refractive index in oil for paints. It is worth noting that certain extenders may lead to poor opacity in solvent-based paints due to their lower refractive indices [18] of particular significance is the remarkable finding that at 800 °C, calcined kaolin attains an almost ideal refractive index for water-based paints, hovering around 1.33, which closely aligns with the refractive index of water. This observation highlights the potential of the 800 °C-processed calcined kaolin as an optimal filler for water-based paint formulations, promising enhanced optical properties and improved reflectivity. Such alignment between the filler refractive index

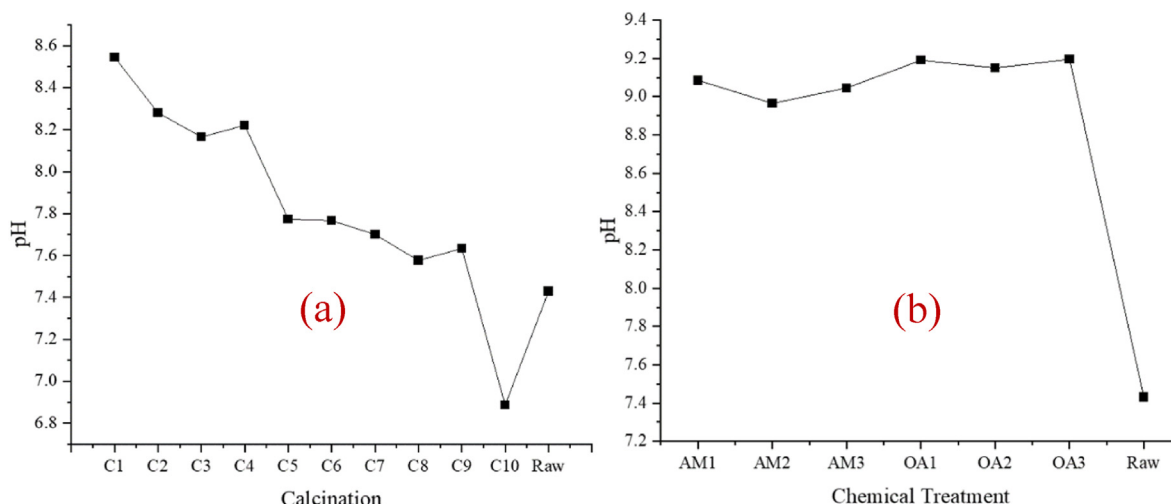


Fig. 4. pH values of kaolin samples treated with (a) temperature and (b) chemical concentration.

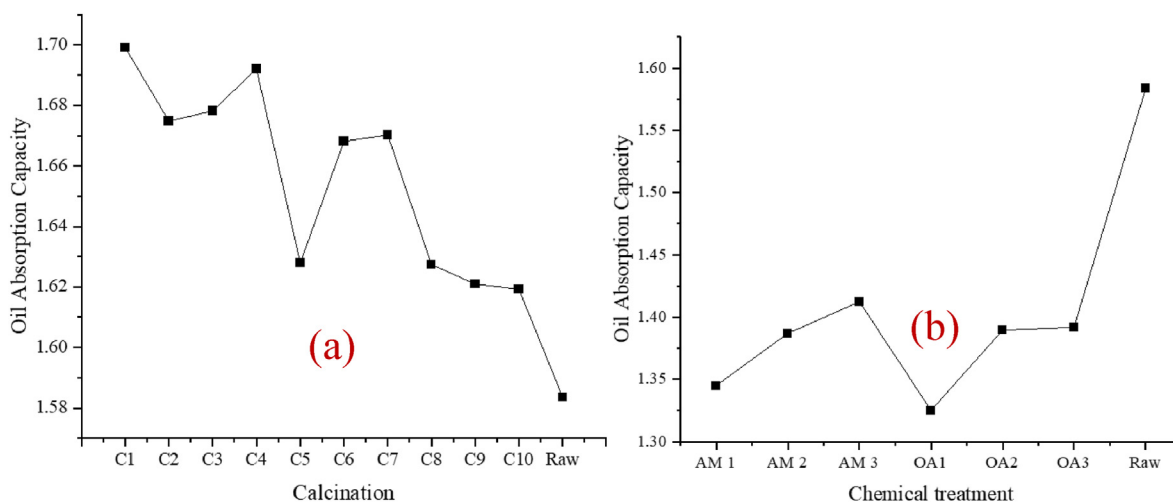


Fig. 5. Oil absorption capacity of kaolin samples treated with (a) temperature (b) chemical concentration.

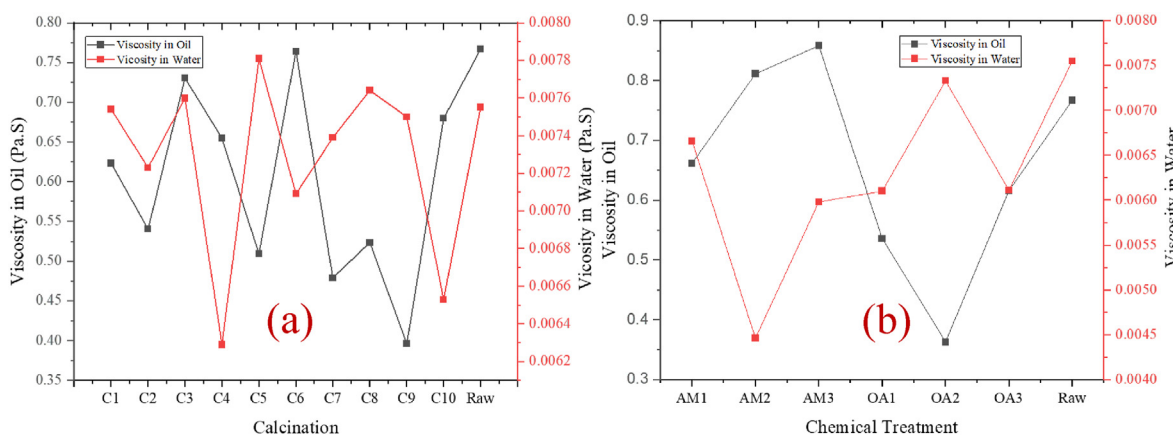


Fig. 6. Viscosity in oil/water of kaolin samples treated with (a) temperature and (b) chemical concentration.

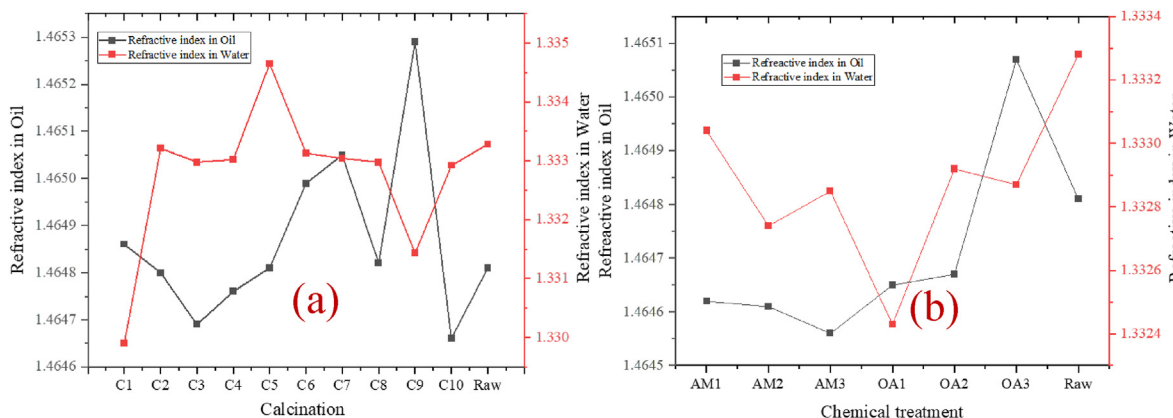


Fig. 7. The refractive index in oil/water of kaolin samples treated with (a) temperature, (b) chemical concentration.

and the paint medium is crucial in achieving a high degree of opacity in the cured paint film, enabling painters and manufacturers to create visually impressive coatings.

In chemical treatments (see Fig. 7(b)), it is fascinating to observe how the refractive index of kaolin, when submerged in water, closely approaches that of water itself. This intriguing characteristic renders kaolin an exemplary filler material with regard to optical properties in aqueous systems. Similarly, when dealing with oil-based mediums, the trend in

refractive index for calcined kaolin remains consistent despite various chemical treatments. This remarkable stability in refractive behavior further reinforces the suitability of kaolin as a filler material in both water and oil applications.

4. Conclusion

In this study, the effects of thermal and chemical modifications on

various properties of kaolin, including pH, oil adsorption capacity, refractive index, specific gravity, and viscosity, were thoroughly investigated. Additionally, comprehensive analyses were performed to characterize the modified kaolin samples. From the results, the following conclusions were obtained:

1. EDX analysis revealed trace elements in the mapped area, likely originating from impurities associated with dickite during its formation due to feldspar weathering. Calcination of the kaolin samples resulted in the disappearance of significant OH stretch groups, accompanied by Si–O stretching vibrations at 1229.6 and 1009.2 cm^{-1} .
2. The kaolin samples' specific gravity was lower than that of TiO_2 and other extenders reported in the literature. This might benefit paint applications, such as reduced settling tendencies and higher oil absorption capacity.
3. Based on the study's findings, a recommended calcination temperature ranges between 600 and 850 °C with a pH range of 7.76–8.56 is suggested for the calcined kaolin to be used as a paint filler, ensuring compatibility with the desired properties. Beyond 800 °C, the oil absorption capacity increases due to further mass loss in the kaolin, creating more voids for enhanced oil absorption.
4. The study also investigated viscosity in water at varying concentration content and observed a gradual decrease followed by a rapid increase, with values reaching below 0.005 Pa s. Among the samples, C7 and C9 emerged as reliable fillers with the most suitable refractive index in oil for paints.

CRediT authorship contribution statement

Andrew Kasumba Buyondo: Writing – review & editing, Writing – original draft, Methodology, Investigation, Funding acquisition, Data curation, Conceptualization. **Hillary Kasedde:** Validation, Supervision, Formal analysis, Data curation, Writing – original draft. **John Baptist Kirabira:** Writing – review & editing, Supervision, Funding acquisition, Formal analysis. **Ocident Bongomin:** Writing – review & editing, Software, Formal analysis.

Declaration of generative AI and AI-assisted technologies in the writing process

While preparing this work, the author(s) did not use English tools for assistance.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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