TIJER || ISSN 2349-9249 || © October 2025, Volume 12, Issue 10 || www.tijer.org

A Review on the Dehydroxylation Behavior of Kaolinite via Thermogravimetric Analysis

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Abstract—Thermogravimetric Analysis (TGA) is essential for understanding how kaolinite decomposes thermally, especially when it turns into metakaolin. This analytical method makes it easier to evaluate variables that have a big impact on kaolinite's thermal stability, like crystallinity, particle size, and chemical makeup. These metrics offer information on the material's structural evolution following heating and are closely linked to differences in the dehydroxylation temperature and weight loss patterns seen after thermal treatment. This review also emphasizes the effects of several treatment approaches meant to improve kaolinite's functional qualities. It has been claimed that methods such as organosilane functionalization, beneficiation, and the addition of additives like alumina significantly increase structural and thermal stability. These changes increase the material's resilience to heat and broaden its use in industrial settings.

The results provide important information for kaolinite optimization in cutting-edge applications, especially in the creation of environmentally friendly geopolymers and fire-resistant coatings. All things considered, the review advances a thorough comprehension of the thermal behavior and transformation mechanisms of kaolinite, facilitating its efficient application in high-performance, environmentally friendly material systems.

Keywords: Kaolinite, Metakaolin, Thermal Properties,Crystallinity,Geopolymer, Dehydroxylation.Thermogravimetric Analysis

1. INTRODUCTION

Kaolinite belongs to the 1:1 clay mineral group and is a soft, layered silicate clay mineral that occurs naturally. It is made up of alternating layers of alumina octahedra and silica tetrahedra, and its idealized chemical formula is $Al_2Si_2O_5(OH)_4$ [1]. Kaolinite is used extensively in many different sectors, including ceramics, paints, paper, rubber, plastics, medicines, and more recently, as a precursor in geopolymer and eco-concrete applications, because of its exceptional chemical stability, high whiteness, low abrasiveness, and abundant availability[2]. Because of its conversion into metakaolin, a highly reactive amorphous aluminosilicate phase that is essential to the development of sustainable binder and advanced ceramics, the behavior of kaolinite during heat treatment has been the focus of extensive research[3].

When heated, kaolinite goes through a well-defined dehydroxylation reaction that produces metakaolin, usually at temperatures between 450°C and 650°C. The octahedral layer's structural hydroxyl groups are lost during this process, which significantly alters the material's crystallinity and reactivity[4].

In geopolymerization and refractory applications, where phase purity and thermal history affect performance, the change is especially intriguing. One essential tool for describing this change is thermogravimetric analysis, or TGA [5]. Mass loss events including moisture release, dehydroxylation, and even higher-temperature phase transitions like mullite formation which are usually identified at temperatures close to 950°C and higher can be quantified with its assistance[4].

Additionally, the thermal behavior of kaolinite can be greatly impacted by elements like surface modifications, impurities, particle size, and crystallinity. Comparative investigations utilizing TGA and controlled rate thermal analysis, for instance, have shown that higher crystallinity is generally linked to higher dehydroxylation temperatures [6].

Similarly, the development of a pure metakaolin phase may be impeded by the presence of iron oxides and quartz, which are frequently found in kaolin that comes from natural sources [7]. Accordingly, calcination, magnetic separation, and acid leaching are often used beneficiation processes to improve the thermal and structural characteristics of raw kaolin[8] .

Furthermore, recent studies have concentrated on altering the kaolinite surface by the application of organosilane and further chemical treatments in order to increase its compatibility in polymer matrices[9]. These changes affect TGA's thermal degradation profile, which makes it a trustworthy indicator of composite system performance. Therefore, knowing how kaolinite behaves under heat stress via TGA helps with both industrial optimization of kaolin-based systems and scholarly research[10].

2. LITERATURE REVIEW

Thermogravimetric analysis (TGA) has emerged as an indispensable technique for evaluating the thermal decomposition and phase transformation of kaolinite, particularly during its conversion to metakaolin[4]. This transformation, driven by the dehydroxylation of structural hydroxyl groups, has been widely studied due to its implications in producing high-reactivity aluminosilicate materials for geopolymers, ceramics, and fire-resistant composites[11].

2.1. Thermal Behavior and Dehydroxylation

The average temperature range for kaolinite dehydroxylation is 450–650°C the exact temperature depends on crystallinity, particle shape, and impurity concentrations. The energy needed to break well-ordered hydroxyl bonds causes more crystalline kaolinite samples to show higher dehydroxylation temperatures[4]. Their controlled rate thermal analysis and TGA study revealed clear mass loss patterns, including a moisture loss phase below 200°C that was followed by the main dehydroxylation peak[12]. Similarly the significance of surface flaws and mineralogical purity, pointing out that kaolin with fewer imperfections typically shows stronger dehydroxylation peaks[13]. In accordance with the theoretical structural water content of kaolinite, the mass loss resulting from the removal of the hydroxyl group was measured at 11.86%[14].

2.2. Modeling and Kinetic Analysis

Kinetic models have been used by a number of investigations to comprehend the mechanism of kaolinite's heat degradation. By using is oconversional techniques the dehydroxylation has an activation energy that ranges from 246.6 to 281 kJ/mol and follows a third-order reaction model. 95% conversion happens at about 635°C, according to their TGA research, which emphasizes the importance of this temperature range for industrial calcination[15].

2.3. Beneficiation and Chemical Treatment's Effects

Impurities like iron oxides and quartz are frequently present in raw kaolin, which impair phase change and weaken metakaolin's qualities[13]. Beneficiation processes like calcination, magnetic separation, and acid leaching are commonly used to improve the thermal and structural properties of kaolinite[16]. The beneficiated Ethiopian kaolin was more pure, and TGA verified that the elimination of mineral impurities induced a shift in the dehydroxylation temperature. Chemical processes like organosilane functionalization have also drawn interest[17]. In their study of kaolinite treated with organosilanes discovered changed thermal degradation profiles, suggesting that surface modification can change thermal behavior. This directly affects kaolin-polymer composites, where heat stability and compatibility are crucial[18].

2.4. Composite Formation and Additives

The thermal breakdown of kaolinite-based systems has been discovered to be impacted by the addition of additives like alumina[19].The demonstrated that kaolin and alumina mixes enhanced the fire-retardant coatings' char shape and heat resistance[20]. TGA played a key role in determining the ideal compositions for thermal protection and measuring the weight loss[21].

Additionally, in a NETZSCH application pointed out that TGA detects higher temperature changes including mullite production above 950°C in addition to tracking moisture and hydroxyl loss. For ceramic applications that demand thermal resilience, these insights are essential[22].

2.5. Implications for Industry and the Environment

Understanding metakaolin's production through TGA is essential given its use in environmentally friendly applications and sustainable building[23]. Further highlighting the wide range of applications for materials derived from kaolin, research has connected the thermal properties of natural and modified clays to their effectiveness in adsorbing organic contaminants[24].

In conclusion, TGA is frequently mentioned in the literature as a key instrument for evaluating the thermal behavior of kaolinite. Metakaolin quality is determined by dehydroxylation kinetics, which are impacted by structural characteristics and chemical treatments. The application of TGA in kaolin optimization for high-performance and sustainable materials is still being refined by ongoing research[25].

3. OVERVIEW OF KAOLINITE (KAOLIN)

3.1. Availability and Production

Kaolinite is a clay mineral that occurs naturally and is widely distributed throughout the world. Countries including the United States (especially Georgia), Brazil, China, India, and Ethiopia are home to significant kaolinite resources[26]. It often forms when feldspar-rich rocks, like granite, deteriorate and eventually decompose chemically to produce clay that is rich in kaolin. Open-pit techniques are typically used in kaolinite mining[27]. It frequently goes through a number of beneficiation procedures after extraction to enhance its quality and appropriateness for industrial uses.

These procedures include acid leaching to improve purity, magnetic separation to get rid of iron-bearing minerals, washing to get rid of impurities, and screening to manage particle size[28]. High-grade kaolinite needed for specialist applications such as ceramics, medicines, and geopolymers is produced with the aid of such procedures [29].

3.2. Cost

A number of variables, including the material's purity, particle size, processing technique, and intended use, affect how much kaolinite-based materials cost. The price of minimally processed crude kaolin ranges from $30 to $80 per metric ton. However, the cost might increase to between $200 and $500 per ton when kaolin is processed and calcined further to create metakaolin, a highly reactive pozzolanic substance utilized in geopolymer and advanced building applications[30]. Furthermore, the cost of production rises when kaolinite undergoes chemical treatment or modification, such as organosilane functionalization, to improve its performance in coatings or composite materials [31] .

3.3. Physical Characteristics

Kaolinite has a smooth, chalky texture and is often white or off-white in color. Because of its tiny particle size typically less than 2 micrometers it appears smooth and powdery. It is light due to its comparatively low specific gravity of around 2.6 [32]. When combined with water, kaolinite's moderate fluidity makes it easy to form, which is a useful quality for molding and ceramic applications.

The mineral's layered crystal structure, which limits its shrink-swell behavior and adds to its structural stability, is defined by a 1:1 arrangement of silica and alumina sheets. It has a specific surface area of 10–20 m^2/g, which influences its adsorption and chemical reactivity[33].

3.4. Composition of Chemicals

The ideal chemical formula for kaolinite is $Al_2Si_2O_5(OH)_4$, which indicates that its main constituents are silica and alumina with hydroxyl groups. About 45–47% silicon dioxide (SiO_2) and 37–39% aluminum oxide (AlO_3) are typically present. Tiny amounts of titanium dioxide (TiO_2), usually less than 2%, and iron oxide (FeO_3), usually less than 1%, are present as impurities. The oxide of magnesium (MgO), calcium oxide (CaO), sodium oxide (NaO), and potassium oxide (K_2O) may also be present in trace amounts. These trace elements and impurities, including feldspar and quartz, might affect how well kaolinite performs and behaves during thermal transformation in industrial settings, especially where exact chemical stability is needed[34] .

3.5. Thermal Properties

When exposed to rising temperatures, kaolinite changes in a number of ways. Dehydroxylation, or the removal of structural water (OH) groups, is the first notable alteration that takes place between 450 and 650°C. This results in a mass loss of roughly 12 to 15%. Metakaolin, a highly reactive amorphous substance that begins to develop at about 550°C, is the result of this process. After being heated above 900°C, metakaolin changes into phases resembling spinel. At 950°C, mullite ($Al_6Si_2O_{13}$), a stable crystalline phase, finally forms. Products made from kaolinite are appropriate for high-temperature and fire-resistant applications because of these thermal changes, which greatly increase the material's strength and heat resistance. Chemical surface changes like organosilane coatings or the addition of stabilizing chemicals like alumina might further increase thermal stability[35].

3.6. Mechanical Characteristics

Because of its layered structure and tiny particle size, kaolinite has a relatively low mechanical strength when it is in its raw state. In its natural state, it is rarely employed for construction purposes. But when it is thermally processed into metakaolin and added to cutting-edge materials like high-performance cement or geopolymers, the resulting composites show noticeably better mechanical qualities. For example, metakaolin-based geopolymer concrete can have compressive strength values of 70–90 MPa, which is on par with or even higher than conventional Portland cement-based concrete. Although the mineral is not very hard, calcination makes it more rigid. Although the modulus of elasticity of raw kaolinite is poorly understood, calcined kaolinite materials show remarkable mechanical resilience and are appropriate for load-bearing construction structures[29].

3.6. Applications

Kaolinite distinct chemical and physical traits give it a broad range of industrial and commercial uses. Because of its great plasticity and firing properties, it is used as a basic raw material in ceramics to make porcelain, tiles, and sanitaryware. It enhances the brightness, opacity, and smoothness of paper by serving as a coating pigment and filler in the paper industry[36].

Kaolinite is used as an extender and white pigment in paints and coatings to save costs and improve durability. It serves as a filler material in the creation of plastics and rubber products, enhancing their tensile strength and rigidity[37]. Because of its fine, non-reactive, and absorbent qualities, it can be used as a mild abrasive and a carrier for active substances in cosmetics and pharmaceuticals. Kaolin-alumina compositions improve fire retardancy in intumescent and fire-resistant coatings. Metakaolin is a highly reactive pozzolan that enhances sustainability and performance in the manufacturing of geopolymer concrete and eco-cement Furthermore, surface-modified kaolinite is used for the adsorption of contaminants in membrane technologies and water purification systems[38] .

3.7. Research Performed Thus Far

Numerous investigations into the characteristics, thermal behavior, and uses of kaolinite have been carried out. In order to evaluate Ethiopian kaolin's industrial viability, looked into its beneficiation process, focusing on purifying methods[39]. In order to comprehend the transformation behavior of kaolinite under heat performed thermal analyses, such as thermogravimetric analysis (TGA) and controlled-rate thermal analysis (CRTA). The mechanical and structural characteristics of geopolymers made with metakaolin were investigated and demonstrating its use in eco-construction and examined the thermal resistance of fire-retardant coatings made from kaolin and alumina. To increase kaolinite's mechanical and thermal performance in composite material[40].

4. METHODLOGY

This review study uses a qualitative methodology to analyze and contrast results from previous research on the thermal behavior of kaolinite using thermogravimetric analysis (TGA). TGA is a method that, in a controlled environment, calculates a material's mass loss as a function of temperature or time. It is especially helpful for determining the phases of phase changes and thermal breakdown in minerals like kaolinite.

TGA analysis of kaolinite samples usually reveals a three-step mass loss pattern. The loss of physically adsorbed water is associated with the first stage, which takes place between 25°C and 150°C. In the second stage, known as dehydroxylation, which takes place between 400°C and 700°C, the structural hydroxyl groups are eliminated, resulting in the creation of amorphous metakaolin. Beyond 900°C, the third stage is linked to the transition into thermally stable crystalline forms called spinel and mullite [41].

In the examined research, kaolinite samples were heated in nitrogen or air atmospheres at rates usually between 5°C/min and 20°C/min. Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), and Differential Thermal Analysis (DTA) were frequently used in conjunction with TGA data to validate phase identification and structural alterations [4].

Additionally, the impact of beneficiation techniques (such as calcination, magnetic separation, and acid leaching) on the thermal stability and purity of kaolinite was investigated. These procedures affect the dehydroxylation's beginning and peak temperatures, suggesting changes in the crystal structure or the elimination of impurities. Some investigations used model-free techniques, such as the Kissinger-Akahira-Sunose (KAS) and Flynn-Wall-Ozawa (FWO) methods, to compute the activation energy for the dehydroxylation process. Through an analysis of these results, this paper seeks to provide an overview of the value of TGA as a diagnostic tool in maximizing the conversion of kaolinite into metakaolin for a range of industrial uses[42].

5. RESULTS

The most influential study among the evaluated works based on a thorough analysis of thermogravimetric analysis (TGA) investigations on kaolinite because of its comprehensive approach that integrates thermal characterisation, chemical treatment, and beneficiation. By using TGA in conjunction with XRD and FTIR investigations to validate phase change, this study provides a multifaceted understanding that demonstrates a direct relationship between thermal behavior and purification level. Their findings confirmed the appropriateness of post-beneficiation Ethiopian kaolin for sophisticated applications like the creation of ceramic membranes by demonstrating consistent dehydroxylation behavior with reduced impurity interference[8].

On the other hand, the study offered crucial kinetic modeling data; nevertheless, its industrial relevance is limited due to its lack of a strong focus on the impacts of contaminants or surface treatments[25]. In a similar offered a perceptive analysis of natural kaolin deposits and their TGA profile; nonetheless, it was devoid of comprehensive modeling of kinetic or structural transformation[17]. Although there was little comparison between untreated and treated samples emphasized the impact of organosilane functionalization on the structural stability and TGA pattern of kaolinite, indicating potential directions for polymer composites in the future.

Combining structural validation, material beneficiation, and application mapping produced encouraging results that showed the new material system's improved performance and usefulness [8].

6. CONCLUSION

This paper demonstrates that Thermogravimetric Analysis (TGA) is a vital tool for examining the thermal behavior of kaolinite, specifically its dehydroxylation, which turns it into metakaolin and usually takes place between 450°C and 800°C. Crystallinity, particle size, impurities, and surface treatments such as organosilane functionalization all have a substantial impact on the mass loss profile and dehydroxylation kinetics of kaolinite, as the reviewed research consistently demonstrate.

Raw kaolin with more impurities has uneven thermal behavior, whereas kaolin with more crystallinity typically shows a sharper and higher-temperature dehydroxylation peak. By purifying kaolinite and reducing the activation energy needed for dehydroxylation, beneficiation techniques such acid leaching, magnetic separation, and thermal calcination increase the material's acceptability for industrial uses. Additionally, chemical surface changes such organosilane treatment improve compatibility with polymer matrices and favorably shift thermal degradation profiles, demonstrating the usefulness of TGA in performance prediction for composite systems.

Overall, data from various geographical sources, including Malaysia,Ethiopia,Romania and others, is synthesized to support the conclusion that modifying kaolinite through purification and functionalization processes improves thermal stability and expands its potential applications, especially in geopolymers, fire-resistant coatings, and ceramic membranes[14-16,18].

7. FUTURE SCOPE

1. **Nanostructured Kaolinite Studies:** Investigating how nanoscale kaolinite behaves thermally and how it interacts with other nanomaterials, such as graphene oxide, to improve the properties of sophisticated composites[43].

2. **Using TGA with Real-Time FTIR**: This method provides a greater understanding of the kinetics of dehydroxylation by tracking developed gases and the breakdown of hydroxyl bonds[44].

3. **Broader Impurity Mapping:** In-depth impurity profiling and how it affects the energy required for dehydroxylation and the yield of geopolymers[45].

4. **Scale-Up Studies:** Pilot-scale testing of chemically modified and beneficiated kaolin in actual industrial processes, such as casting geopolymer concrete or 3D printing ceramics[46].

5. **Environmental Impact Assessment:** Life-cycle analysis comparing the energy, emissions, and recyclability of purified and functionalized kaolin versus typical kaolin usage[47].

6. **Machine Learning in TGA Interpretation:** Using experimental databases, AI and ML models are integrated to forecast phase changes and thermal behavior in modified kaolinites[48].

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