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Thermophysical Characterization, Thermogravimetric Analysis and Determination of the Specific Surface Area of a Clay-Diatomite Mixture Treated with Quicklime

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Abstract: This study aims to perform thermophysical characterization, thermogravimetric analysis, and specific surface area determination of a lime-stabilized mixture composed of previously studied Gaoui clay and Michemiré diatomite. Lime stabilization of clay is a widespread technique; the addition of diatomite significantly reduced the mixture's thermophysical properties due to its porous structure. The absolute density was determined using a helium pycnometer, and the specific surface area, air permeability test, was also determined using a Blaine permeabilimeter. Experimental results showed that the thermal conductivity and thermal effusivity of the clay decreased significantly with the addition of the other two materials. They decreased from 0.74 W/m·K to 0.338 W/m·K and from 985.5 $W/s^{1/2}/m^2/K$ to 519.6 $W/s^{1/2}/m^2/K$ respectively, for the 100% clay and 50% clay + 35% diatomite + 15% lime formulations, at a compaction pressure of 3 MPa. Thermogravimetric analysis showed that at low temperatures (0 to 150 °C), free water from the materials began to evaporate. From medium temperatures (400 to 600 °C) to high temperatures (1,000 °C), the results showed a mass loss of 6% for Gaoui clay, 2% for Michemiré diatomite, and 1.5% for lime. The results of the Blaine test and air permeability tests demonstrated that the specific surface area of 100% clay is high at 0.355 m^2/kg, followed by that of 100% diatomite at 0.305 m^2/kg and that of 100% lime at 0.273 m^2/kg.

Key words: Clay, diatomite, lime, thermophysical characterization, thermogravimetric analysis, Blaine fineness.

1. Introduction

Africa, with an estimated annual population growth rate of 5% over the last decade, has approximately 1.5 billion inhabitants, 50% of whom are expected to live in urban areas by 2050. This demographic dynamism is causing a housing crisis coupled with increasing energy demand in the building sector [1]. Among the sectors with the greatest impact, buildings stand out for their high energy consumption, representing 40% of the energy used and 25% of global CO_2 emissions. Earth is a building material that has ecological advantages, and its hygrothermal properties, which are very beneficial for housing construction, can contribute to reducing energy consumption and pollution [2]. The exploitation of natural resources has become crucial to meeting growing energy needs. These resources can significantly improve the thermal insulation of buildings, thereby reducing energy consumption and enhancing their energy efficiency. Their use can mitigate the environmental and economic costs associated with energy consumption, which is essential for achieving sustainable development goals and preserving the environment for future

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generations [3]. Indeed, clay, requiring no real processing, has established itself as the quintessential ecological building material with a very low carbon footprint and widespread availability. As for diatomite, it is also a material increasingly used as an additive or substitute in various applications thanks to its unique properties such as high porosity, low density, and high silica content. It has a very low operating cost, is readily available, and is often used as a mineral additive in the manufacture of pottery and lightweight bricks [4]. The objective of this work is to perform the thermophysical characterization and thermogravimetric analysis of the mixture of these two lime-treated materials for use in building construction, offering prospects for sustainable development.

2. Materials and Methods

This section details the experimental approaches and equipment used for the thermophysical characterization and thermogravimetric analysis of our samples. The aim is to provide a sufficiently precise description to allow for the reproducibility of the experiments and the understanding of the results obtained.

2.1 Clay

Clay is a very ancient building material, found all over the world. It is one of the first natural materials used by humans to build houses, particularly in rural areas. Easy to work with, it requires neither significant effort nor advanced technical knowledge. It forms from the decomposition of rocks through physical and then chemical weathering. Over time, clay undergoes constant transformation, which alters its properties depending on its environment [5]. It is a loose (unconsolidated) sediment composed of extremely fine particles, measuring less than 2 μm. Mineralogically, clay is characterized by a layered structure of tetrahedral and octahedral layers that are stacked and separated by interlayer spaces. This unique architecture gives clay a high sensitivity to water. It has a strong affinity for moisture; as its water content increases, the thickening of the adsorbed water films causes a significant increase in its total volume [6]. The clay chosen was from the Gaoui site, class A3, with a plasticity index of 35%. 2 mm sieve fragments were used to prepare our test specimens for thermal testing.

2.2 Diatomite

Diatomaceous earth is a soft, friable, fine-grained, weakly cemented, highly porous, and lightweight sedimentary siliceous rock. Its interesting characteristics in the construction sector include its low bulk density, low thermal conductivity, and large specific surface area [7]. Diatomite originates from a pale, light siliceous sedimentary rock composed mainly of fossilized skeletal remains of diatoms, a unicellular aquatic plant related to microscopic diatom algae, ranging in size from 0.75 to 1.500 μm, during the Tertiary and Quaternary periods [8]. According to Vizinet, J. and B. De Reviers [9], the use of diatomite is very ancient; historically, in 532 AD, it was used to make lightweight bricks during the construction of the Hagia Sophia Cathedral in Constantinople. Studies conducted by Kipsanai, J., et al. [10] show that diatomite is a material with properties such as low calcium content, a high glassy phase (80-90% of which are particles smaller than 45 μm), an unbaked material content of less than 5%, a high amorphous SiO_2 content, and the associated pozzolanic activity of diatomaceous earth, which are important aspects of its application in construction. The diatomite chosen is from the Michemiré locality, located at 13°49'29" North and 15°44'39" East. This variety is the most widely used in construction in the North and Northwest of the country and is white and very fine. The one-millimeter sieve fragments are collected for the preparation of our test specimens.

2.3 Quicklime

It is a whitish powder obtained by the thermal decomposition of limestone. Chemically, it is calcium oxide containing varying amounts of magnesium oxide

[11]. Soil stabilization is a method of soil improvement aimed at achieving permanent stability by modifying soil properties. This technique involves adding a hydraulic binder to the material. Quicklime is used in combination with clay and diatomite in varying proportions to determine the new properties of the mixture. Stabilization therefore relies on the reaction between the lime and the silico-aluminous components present in the clay and diatomite mixture [12].

2.4 Thermophysical Characterization

The FP2C measuring device is used for thermophysical testing with its two probes. The hot wire represents a material's ability to transfer heat under steady-state conditions, while the hot plate characterizes a material's ability to rapidly absorb or release heat during transient thermal contact. The probe is placed between two samples of the same size and formulation, thus sandwiching the probe between the samples.

Thermal conductivity is one of the most sought-after thermophysical properties for building materials. A hot-wire probe is used to determine it. The heat flux density, expressed in W/m², is given by the following equation:

$$\Phi = \frac{\lambda}{e}(T_i - T_e) \quad (1)$$

Thermal effusivity is a key parameter for understanding heat exchange between a material and its environment. To determine this value, we used a hot plate probe. It depends on the thermal conductivity, thermal inertia, and density of the material. It has important applications in many fields, particularly in building construction:

$$\mathrm{E} = \sqrt{\lambda \times \rho \times \mathrm{C}_P} \quad (2)$$

2.5 Disc Crusher

This machine is used to reduce the particle size of various materials by grinding them into a fine powder. It has been set to a rotation speed of 800 rpm and a grinding time of 40 seconds, after which the motor stops. Immediately after grinding, each powder of a specific material formulation is placed in a tube and labeled, meaning it is ready for testing with a helium pycnometer, thermogravimetric analysis, and Blaine fineness.

2.6 Absolute Density

The helium pycnometer is used to measure the density of materials with high precision. The supply circuit and various valves are controlled by a computer using the ACCUPyc software. Since the mass being measured is introduced into the pycnometer chamber, a waiting period of 20 to 30 minutes is required before the next test can begin. This time allows the gas to circulate through the volume occupied by the material. The operating principle of the helium pycnometer is based on Mariotte's Law (or Boyle's Law), which relates the pressure, volume, and temperature of a gas.

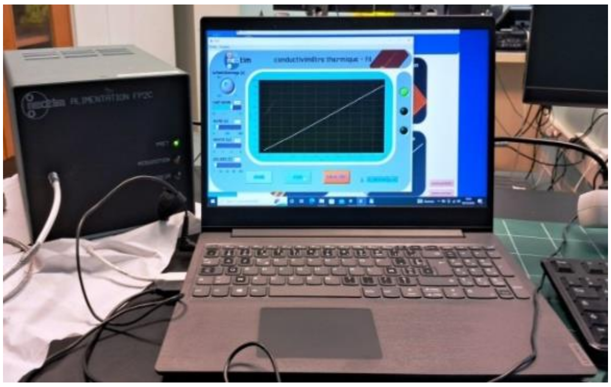

Fig. 1 FP2C thermal measuring device and computer.

Fig. 2 Disc crusher.

Fig. 3 Helium pycnometer.

2.7 Thermogravimetric Analysis

Thermogravimetric analysis (TGA) heats the material to 1,000 °C in 90 minutes. During this temperature increase, the organic elements within the material will calcine, and the temperature will then decrease to 25 °C within the same timeframe. TGA is a method for characterizing materials that directly measures mass changes as a function of temperature or time. It is based on the interaction between the sample, its mass, its thermal profile, and its gaseous environment. TGA is commonly used to determine material properties, evaluate reaction kinetics, or study gas adsorption on solids [13]. This measurement technique is frequently coupled with other analytical methods, such as differential scanning calorimetry (DSC), for simultaneous and complementary analysis.

2.8 Blaine's Finesse

The Blaine fineness test, also known as the air permeability test, is a standard method for evaluating the fineness of a powdered material by measuring its specific surface area. It is a crucial indicator of its reactive properties and performance in construction applications. The principle is to compact the sample in a cell and measure, using a stopwatch, the time it takes for a volume of air to pass through this bed of powder. The slower the airflow, the finer the material and the higher its specific surface area. The following equation allowed us to calculate the specific surface area of materials:

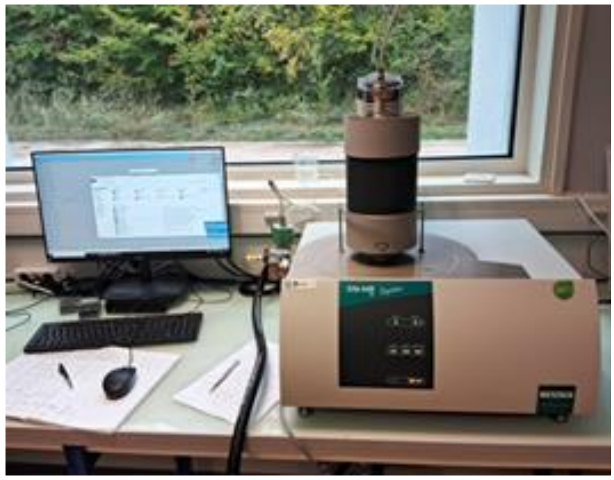

Fig. 4 NETZSCH STA 449 F3 Jupiter instrument for simultaneous measurement of TGA and DTA.

Fig. 5 Blaine permeabilimeter.

$$s = \frac{k \times \sqrt{e^3} \times \sqrt{t}}{\rho \times \sqrt{\eta} \times (1 - e)} \quad (3)$$

where k = 23.1 and | = 0.0001808 à 20 °C.

3. Results and Discussion

In this section, we examine the thermophysical specifications, thermogravimetry, and Blaine fineness of the study materials, as determined by the aforementioned experiments.

The histograms in the Fig. 6 are those of the absolute densities determined by the helium pycnometer of 100% materials, then the mixture.

The Fig. 6 shows that 100% lime has the highest absolute density, followed by 100% clay. 100% diatomite has the lowest absolute density in the group. Increasing the lime content consistently results in an increase in the absolute density of the mixture.

The Figs. 7 and 8 show the thermophysical results, namely thermal conductivity and thermal effusivity. The 10×10×2 cm^3 specimens, passing through a 2 mm sieve, were manufactured under a compaction pressure of 30 bar.

The graph shows that 100% clay has a very high thermal conductivity compared to the other formulations, and that of 100% diatomite is the lowest [10] found a thermal conductivity of diatomite of 0.171 W/mK, which is close to ours with a slight difference of 0.01 W/mK. The Fig. 7 shows the thermal effusivity

The histograms of thermal effusivity follow the same pattern as those of thermal conductivity. The work carried out by Abdallah et al. in 2023 on Faya diatomite yielded a value of 339 $W.s^{1/2}.m^{-2}.K^{-1}$ for thermal effusivity, which converges with ours. According to studies conducted by Balaska, A. et al. [14] due to its low thermal conductivity and relatively light density, the product can be used to demonstrate high insulation.

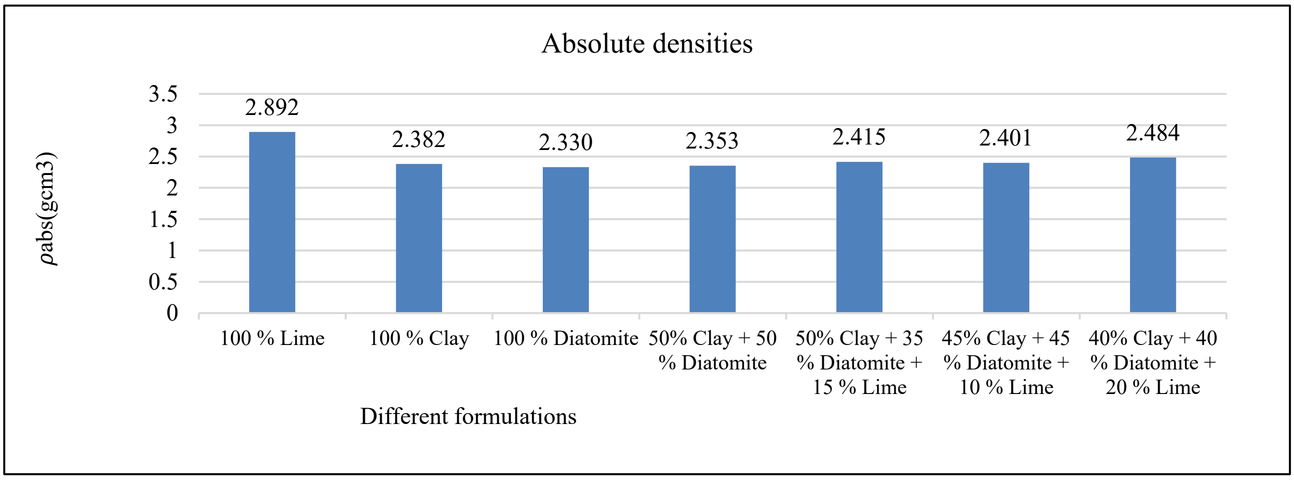

Fig. 6 Absolute density of the different formulations.

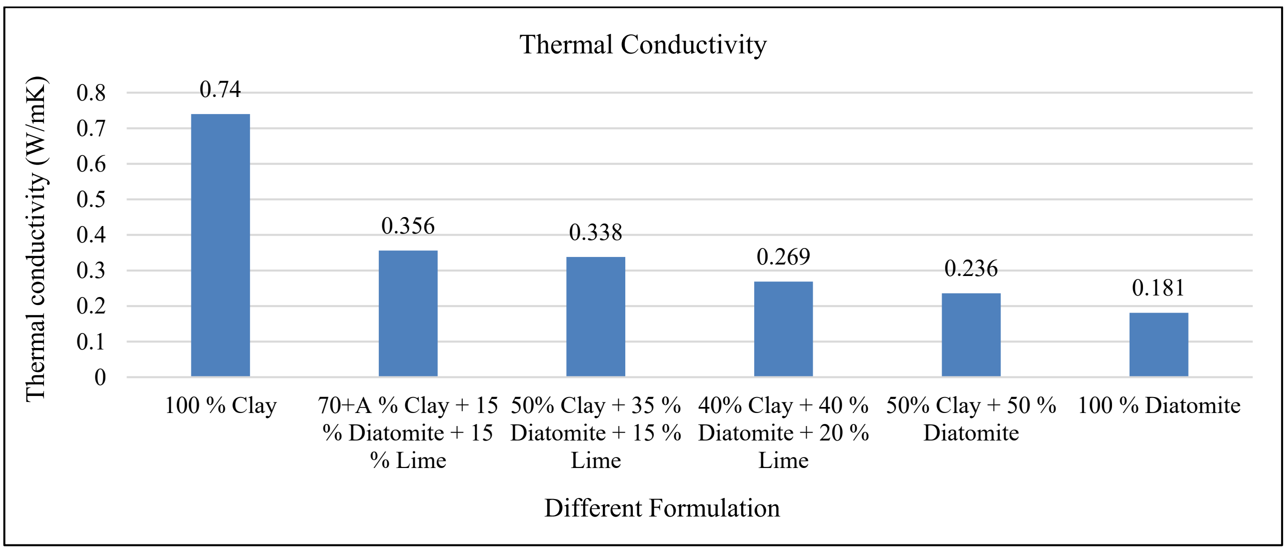

Fig. 7 Thermal conductivity graph.

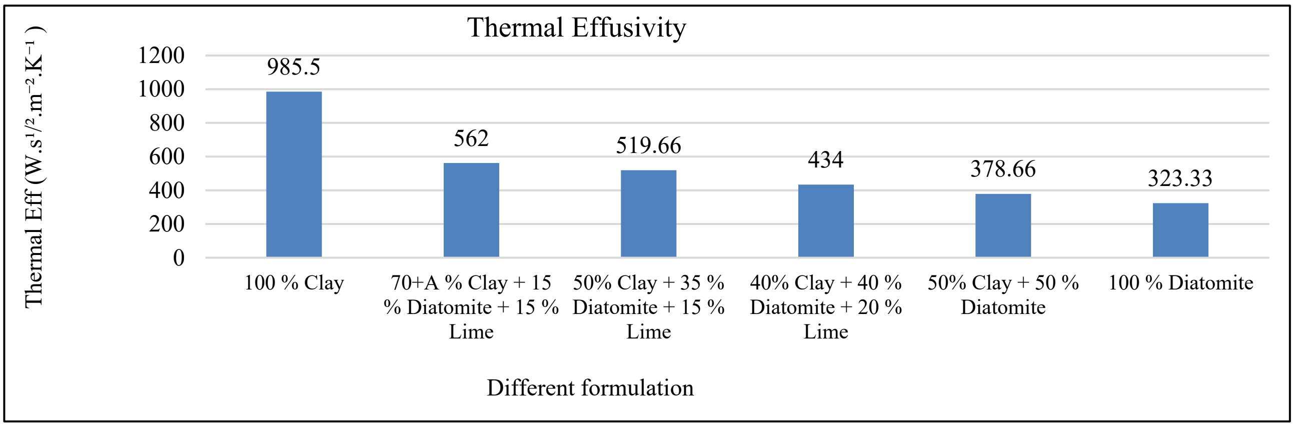

Fig. 8 Thermal effusivity graph.

The Figs. 9-12 will show the results of the thermogravimetric analysis, starting with the unmixed materials, i.e., clays, diatomite, and lime at 100%, followed by mixtures of the three with specific percentages. The phenomena are observed in three phases: low, medium, and high temperature. The TGA curves are black, representing the relative mass loss as a percentage, and the DTA or DSC curves are gray, representing the thermal signal, which indicates endothermic or exothermic heat changes. They occupy the ordinates and are both plotted as a function of temperature.

The interpretation of these curves allows us to determine the thermal stability, composition, and thermal phenomena of the materials under study.

At low temperatures (from 20 °C to 150 °C) an initial mass loss is observed. This generally corresponds to the loss of adsorbed or interstitial water, the relative mass decreasing from 100% to approximately 89 to 90%, representing a loss of approximately 10 to 11% for the TGA curve and for the DTA curve a major endothermic peak is visible, with a maximum between 100 °C and 150 °C;

At the average temperature (from 400 °C to 600 °C), often associated with the dehydroxylation of the clay, a second, more gradual mass loss is visible, the mass goes from 90% to about 84%, i.e., a loss of about 6%, indicating the TGA curve, on the other hand the DTA curve shows an endothermic peak with a minimum between 500 °C and 550 °C;

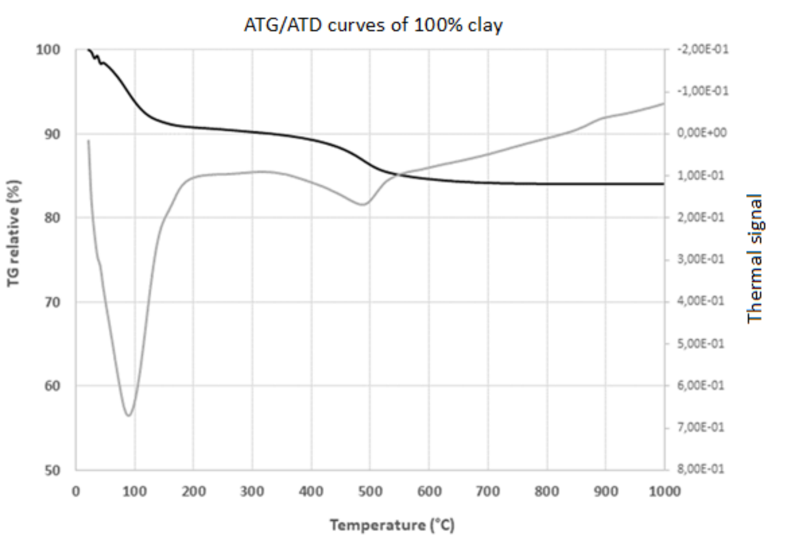

Fig. 9 TGA/DTA curves of Gaoui clay.

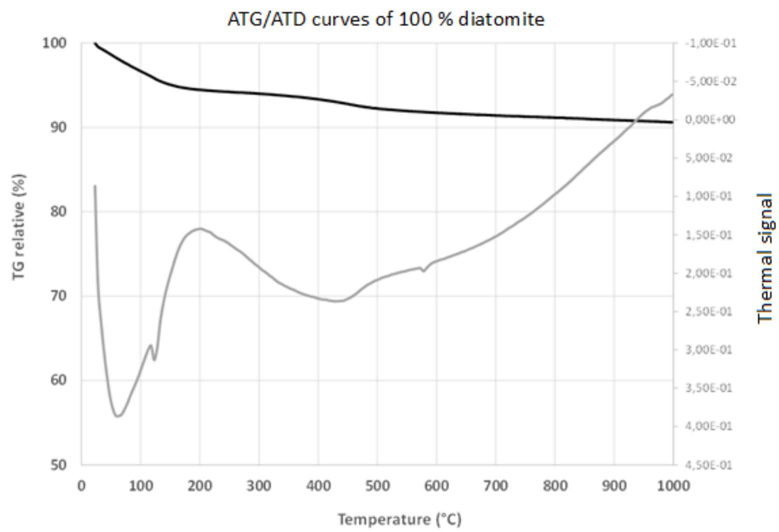

Fig. 10 ATG/ATD curves of Michemire diatomite.

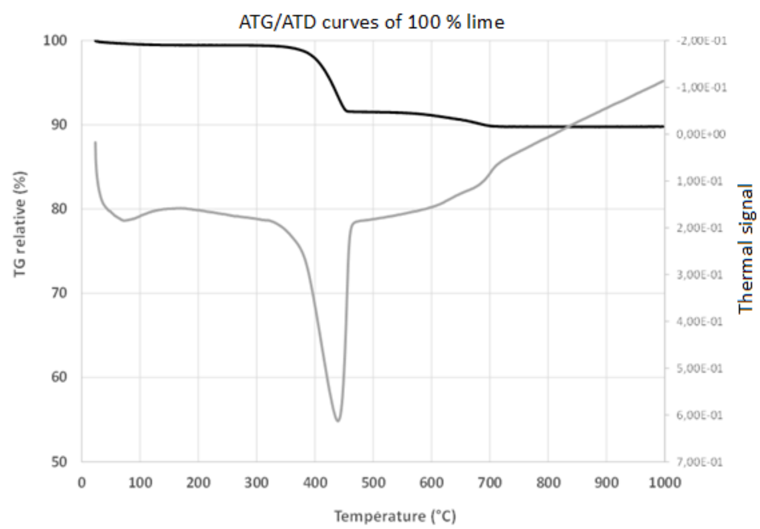

Fig. 11 TGA/TD curves of quicklime.

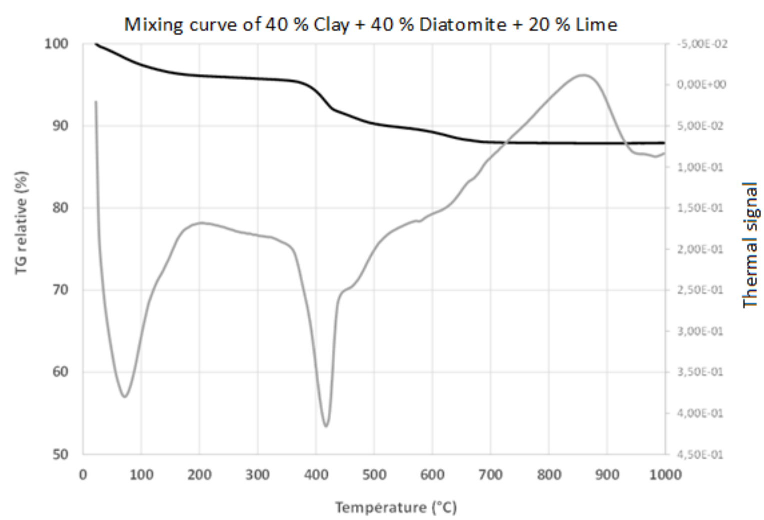

Fig. 12 TGA/DTA curves of the mixture of 40% Clay + 40% Diatomite + 20% lime.

At high temperatures, mass loss becomes very small or almost zero after approximately 700 °C, with the TGA curve reaching a stable plateau around 84%. In contrast, the DTA curve shows a slight drift towards the exothermic peak around 870 °C; this is the endothermic phenomenon followed by a change in slope towards the exothermic phase, but without a distinct sharp peak in the visible range. This minor mass loss at higher temperatures could indicate the decomposition of carbonates or other minerals [15]. The results obtained by Gourouza M. et al. [16] are in agreement with ours, at low temperature a mass loss of about 7.6% is obtained and is followed by another loss of 5.6% at medium temperature.

At low temperatures (25 °C to 200 °C), a mass loss is observed, generally attributed to the evaporation of adsorbed or interlamellar water present in the diatomite. The relative mass loss decreases from approximately 100% to 93%, representing a loss of 7% according to the TGA curve. The DTA curve shows an endothermic peak corresponding to the evaporation of free and weakly bound water, with a minimum around 60 °C. A second, smaller endothermic peak is visible between 120 °C and 150 °C. This process requires energy for the water to transition from the adsorbed to the gaseous state.

At an average temperature between 400 °C and 600 °C, we observe a slower, progressive, and continuous mass loss. The relative mass decreases from approximately 93% to approximately 91%, representing a mass loss of about 2%. This is the dehydroxilization of opal or amorphous material. The second, smaller peak at approximately 580 °C corresponds to the polymorphic transformation of quartz, which changes from α-quartz to β-quartz.

At high temperatures (above 600 °C), mass loss becomes very low or negligible, reaching a stable plateau around 91%. However, the ATD curve rises towards the zero axis and continues its upward drift, forming an exotherm as it approaches 1,000 °C, without a sharp peak. The same phenomena have been observed by Meradi H. et al. [17] for low temperatures. Combined TGA/DTA analyses confirm that the main mass loss is due to dehydration, potentially followed by the decomposition of organic matter or dehydroxylation; at high temperatures, it can be said that it is the structural reorganization of amorphous silica.

At low temperatures (approximately 25 °C to 300 °C), the ATG curve shows a very small mass loss, visibly very close to 100%. This phenomenon, characterized by the absence of significant mass loss, indicates that the lime does not contain much free water. Regarding the ATD curve, a slight endothermic peak, corresponding to the moisture content of the lime, is observed, which quickly subsides, followed by a flat plateau up to approximately 300 °C.

At medium temperatures (approximately 300 °C to 500 °C), a very significant and rapid mass loss occurs, starting around 350°C and ending around 500 °C. The mass decreases from approximately 100% to 91.5%, representing a loss of about 8.5%. Meanwhile, there is a very deep and sharp endothermic peak at the bottom of the ATD curve, which is the dominant phenomenon, with a minimum between 440 °C and 450 °C. This is the dehydroxylation of slaked lime, calcium hydroxide $Ca(OH)_2$

At high temperatures (above 600 °C), a slight further loss in relative mass is observed, beginning around 700 °C, decreasing from approximately 91.5% to approximately 90%, representing a loss of about 1.5% according to the TGA curve. Regarding the DTA curve, a broad endothermic shoulder is observed, although less pronounced than the initial peak. This phenomenon results from the decarbonation of small quantities of calcium carbonate ($CaCO_3$) present as an impurity or from the partial carbonation of the lime.

Studies carried out by Amira A. [18] show that calcium hydroxide is obtained between 400 °C and 520 °C and calcium carbonate is obtained between 650 °C and 890 °C, hence these temperature ranges correspond well with our results.

As with the previous curves, the TGA/DTA curves of the mixture are described as follows:

At low temperatures (25 °C to 100 °C), a mass loss is observed on the TGA curve. This slight initial mass loss is approximately 3 to 4%. The mass decreases from 100% to approximately 96 to 97%. As for the DTA curve, an endothermic peak is observed, which is often attributed to the release of moisture (or evaporation of adsorbed water) or other highly volatile components contained in the mixture.

At temperatures between 200 °C and 400 °C, we observe a second mass loss, which decreases from 96% to approximately 93%. This is due to the dehydration of a constituent or the volatilization of an organic component.

At an average temperature between 400 °C and 600 °C, there is a major mass loss, ranging from approximately 89% to 93%; this is the main thermal decomposition of the mixture’s components. It is an endothermic process with the first, larger peak at approximately 430 °C corresponding to the decomposition of lime, a second, smaller peak at 460 °C attributed to the disintegration of clay, and finally a third, smaller peak at 580 °C attributed to the disintegration of diatomite.

At high temperatures around 800 °C, a very slight mass loss of one percent (from approximately 89% to 88%) could be linked to the decomposition of carbonates. A positive exothermic peak is observed between 800 °C and 900 °C, coinciding with this loss. The mass remains constant at 88% after 900 °C, likely consisting of stable inorganic materials in the mixture that do not decompose up to 1,000 °C.

At low temperatures (0 to 100 °C), the TGA curve decreases, resulting in a mass loss of approximately 8%, with the initial mass dropping from 100% to about 92%. The DTA curve shows an endothermic peak (heat absorption), a phenomenon often linked to dehydration or generally attributed to the loss of free water.

At temperatures between 100 and 400 °C, the TGA curve is almost horizontal, indicating that the sample of this formulation is thermally stable within this temperature range, with a negligible mass loss of approximately 1%. The DTA curve shows a small peak at 130 °C, corresponding to the decomposition of diatomite. At an average temperature between 400 °C and 600 °C, there is a 4% mass loss, ranging from approximately 91% to 87%. This is the primary thermal decomposition of the mixture's components. It is an endothermic process with a peak at approximately 470 °C corresponding to the decomposition of clay, and a smaller peak at 580 °C attributed to the disintegration of quartz.

The Fig. 13 below shows the curves of the thermogravimetric and differential thermal analysis for the formulation 50% clay + 35% diatomite + 15% lime. The study of these graphical data reveals the mass changes resulting from dehydration and decomposition, as well as the variations in heat flow of the sample, in correlation with the temperature increase up to 1,000 °C.

At high temperatures (700 °C to 1,000 °C), the TGA curve flattens out, and the relative mass stabilizes around 87%. This phenomenon is likely attributed to the inorganic fraction or a stable carbon residue, indicating that there is no longer a significant decomposition reaction. The DTA curve shows an upward-pointing peak; this is an exothermic phenomenon often linked to crystallization.

The air permeability test, or Blaine fineness test, is the standard method for determining the specific surface area, or total area of particles per unit mass, generally expressed in cm^2/g or m^2/kg of a material. It is the primary physical parameter controlling reaction kinetics.

The specific surface areas of the materials studied are determined using the formula in equation (3). Specific surface areas of 0.355 m^2/kg were obtained for 100% clay, 0.305 m^2/kg for 100% diatomite, and 0.273 m^2/kg for 100% lime.

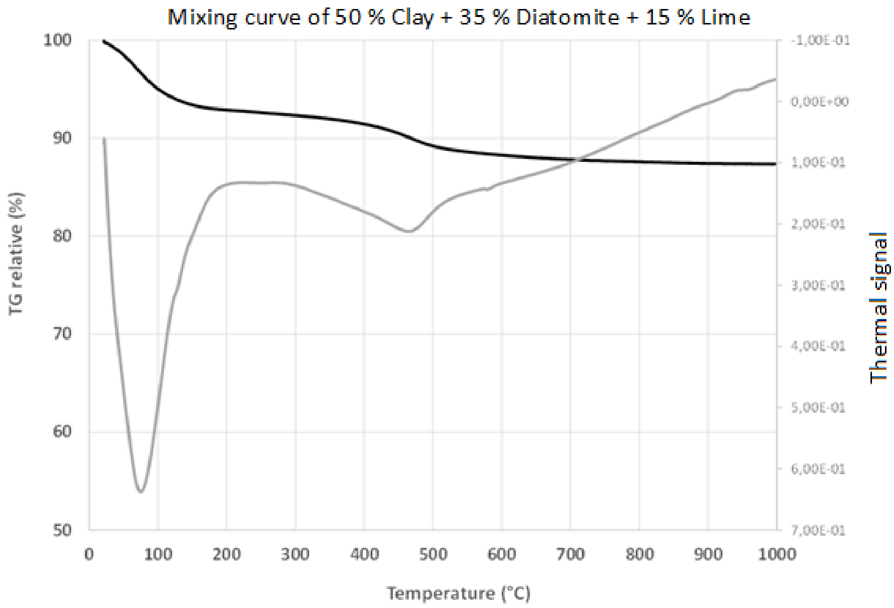

Fig. 13 TGA/DTA curves of the mixture of 50% Clay + 35% Diatomite + 15% lime.

4. Conclusion

The absolute density of the formulations is directly proportional to the lime content and inversely related to the diatomite content, confirming that lime is the densest component and diatomite the least dense among the three materials studied.

Diatomite has the lowest thermal conductivity. This is due to its low density and, consequently, its very high porosity, which traps air and acts as an excellent insulator, thus reducing heat transfer. Clay has the highest thermal conductivity; the addition of diatomite and lime to clay reduced its thermal conductivity compared to clay alone.

The interpretation of Thermogravimetric Analysis (TGA) and Differential Thermal Analysis (DTA) curves allowed us to determine the thermal stability, composition, and thermal phenomena of materials, such as decomposition and phase changes. Finally, the Blaine fineness, the main physical parameter controlling surface reaction kinetics, was determined. Clay had the highest value, followed by diatomite, and then lime.

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Conflicts of Interest

The authors declare no conflicts of interest.

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