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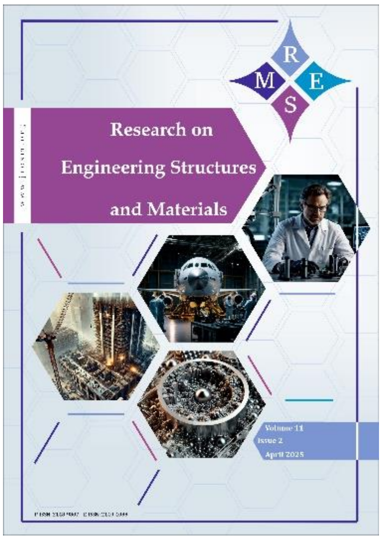

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Research Article

Physico-chemical characterization of fine gray clay with micrometric particle size

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Abstract

Clays are an interesting material used in several areas because of their abundance and natural ecological properties. Clay can be a building material either with rammed raw earth or fired clay bricks, also, it is used for the production of ceramics and pottery. In addition to that, absorbent clays are employed in soil remediation and water filtration, without forgetting the benefits of clays in health and wellness which allows them to be used in cosmetics, skincare, and hair products. In this study, thermophysical properties of a gray clay from the Gharb region of Morocco were investigated, to exploit its main assets and try to improve its weaknesses and limitations, so that this clay can be useful in different domains. Thus, this investigation will allow to control and to enhance this clay's properties to be used in different sectors in an optimal way. This clay is a very fine material, mainly composed of quartz, showing a thermal stability around 900°C and good mechanical strengths. In addition to that, this material has a thermal conductivity of 0.62 W $m^{-1}K^{-1}$ which proves that it is a good insulator.

1. Introduction

Clay-based materials are used in different domains. Pottery production depend mainly on clay composition and its source of extraction [1-3], also since unsafe drinking water alone can cause many different illnesses, one of which is diarrhea and The World Health Organization (WHO) estimated that approximately 9.1% of the global burden of disease and 6.3% of all deaths worldwide could be prevented by improving water, sanitation and hygiene[4], it is important to consider nano clays as a natural solution for water treatment because of its effectiveness against contaminants and bacteria[5].

In addition to that, clay is introduced as an ecological material to minimize the use of concrete and reduce the embodied energy of the construction sector. Since clay was the most used across centuries, it was the main subject of numerous studies. Souileh et al. [6] investigated shale clay from Settat-Khouribga for potential civil engineering use. El Azhari et al. examined and compared the thermal performances of three types of unfired clay bricks from three different regions of Morocco [7] And also studied the influence of unfired clay use on the energy efficiency of buildings in south Morocco [8]. A similar study by Fgaier et al. [9] investigated the thermal behavior of North France's unfired clay bricks and examined the indoor temperature inside a building under real-life conditions. Atbir et al. [10] investigated natural white and red clay coming from the middle Atlas of Morocco and characterized this material mechanically and thermally. They also enhanced their properties and texture by adding sheep wool as ecological animal fibers. Generally, clay is incorporated with ecological

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additives to enhance not only its thermal and acoustic properties but also its positive environmental impact, for example, grapevine shoots, psyllium seeds, rice husk, wood waste, granular cork [11-15]. However, mechanical strength is usually weakened, consequently, cement and lime can be added to restore them. El Wardi et al. [16] also enhanced Clayey soil from the Bensmim Region of Morocco with cork, which has caused a deterioration of mechanical properties. However, they created a sandwich material using cement mortar and plaster coating layers to improve thermal and mechanical properties, which has led to an ecological brick for construction. Moreover, Mountoumnjou et al. [17] proposed new low-cost ceramic microfiltration membranes composed of Kaolin, Bentonite, and limestone for bacteria removal, and Kassa et al. [18] conducted a series of cone index tests on clay treated with waste-based stabilizers to investigate the strength development based on their water absorption and retention capacities. This paper studies the thermophysical properties of local clay coming from the Gharb region of Morocco, with the perspective of identifying qualities to exploit and flaws to improve, therefore the various results of this study will provide insights into this clay's thermal insulation, ceramic manufacturing and sustainable construction with the aim to contribute to the development of optimized clay-based materials for various industrial applications, such as construction, water filtration and purification, pottery and production of ceramics.

2. Materials and Methods

2.1. Clay Material

The clay used for this study is gray clay taken from Khenichet, a small village in the Gharb region of Morocco (Figure 1). Knowing that many houses in this location were built using the rammed earth technique mixing this clayey soil with straw fibers.

Fig. 1. Khenichet - Sidi Kacem – Morocco

Fig. 2. Clay raw material

These houses prompted us to characterize this material and identify its thermal, mechanical, and chemical properties. This clay was collected in large quantities to realize different tests. The soil is rinsed with tap water and passed through a 100 µm sieve. Afterward, it is placed in a container and left in an oven at 105 °C until its mass becomes constant. The next step involves manually grinding the clay to reduce it to powder form ready for use (Figure 2).

Characterization Methods

2.2.1 X-Ray Diffraction

To determine the chemical composition of Khnichet's clay, a test of X-Ray Diffraction was performed. The PANalytical EMPYREAN diffractometer was used with a Copper (Cu) anode as a radiation source and wavelengths k- α1 (1,54060˚A) and K- α2 (1,54443˚A). It is a high-resolution test with a step size of 0.066 °2θ and a counting time per step of 113,2 seconds with a goniometer setup of a 240 mm radius and a divergence slit of 0.2177° width. The measurement was conducted in air at an ambient temperature of 25°C.

2.2.2 Particle Size Analysis

The particle size analysis was realized based on the sedimentometry method since the sieve analysis and the air separation method were inefficient and unable to differentiate between the small particles of this material. How- ever, the sedimentometry method can distinguish between particles with a diameter smaller than 80 µm. This test was performed following the specifications of the NF P94-057 standard. An 82,2g sample of Khenichet's clay was mixed with 440,2ml of distilled water, followed by the addition of 60 ml of a 5% sodium hexametaphosphate (SHMP) deflocculant solution. A mechanical stirrer mixes the whole mixture and the sedimentation speed determines the particle size.

2.2.3 Atterberg Limits

The Atterberg limits are conventional constants that define the threshold between:

- The transition of the soil from a liquid state to a plastic state (liquid limit: Wl)
- The transition of the soil from a plastic state to a solid state (plastic limit: Wp)

The value of these limits is the soil's water content at the considered transitional state, expressed as a percentage of the dry weight of the material. Therefore, the plasticity index Ip is defined, describing the range of plasticity being the difference between the two limits, Ip = Wl −Wp. The liquid limit is determined using the Casagrande device, while the plastic limit is identified using 3 mm diameter rolls.

2.2.4 Thermogravimetric Analysis and Differential Thermal Analysis

Differential Thermal Analysis (DTA) and Thermogravimetric Analysis (TGA) are the most commonly used techniques for studying the transformations that occur with energy consumption or release, whether or not associated with a loss of mass. DTA enables the identification of endothermic, exothermic, and invariant phenomena by measuring the temperature difference between the sample and a reference material over time or as a function of the sample's temperature, all under a controlled atmosphere. On the other hand, TGA permits tracking mass variations during heat treatment. It is useful for studying reactions involving volatilization, fixation of certain constituents, or interaction with a gas phase. The information obtained from TGA is mostly complementary to that of the DTA, and both tests were performed in N_2 atmosphere.

2.2.5 Mechanical Characterization

Compression strength is the most important characteristics of mechanical properties in the building sector. The uniaxial compression test for compression strength were conducted according to the standard EN 196-1 using a CONTROLS PILOT COMPACT-Line presented in Figure 3.

Fig. 3. Controls Pilot Compact-Line for compression strengths

2.2.6 Thermal Characterization

Thermal conductivity, thermal effusivity, and thermal capacity were analyzed for thermal characterization. The asymmetric hot plane method in steady-state was used to determine thermal conductivity [19], in fact, an experimental setup is constructed (Figure 4), consisting of a 40 Ω electric resistor powered by a 6V voltage, positioned beneath the sample, and on top of a 1 cm layer of polyethylene insulating foam. This assembly is placed between two 5 cm thick aluminum blocks. K-type thermocouples are strategically placed between the heating resistor and the insulating foam, between the foam and the lower aluminum block, and between the sample and the upper aluminum block. The thermocouple signals are captured using a data acquisition system and the temperature values in the steady-state permit a direct calculation of thermal conductivity.

Fig. 4. The asymmetric hot plane method in steady-state device

Fig. 5. The hot plane method in the transient regime

On the other hand, the hot plane method in the transient regime was for thermal effusivity and thermal capacity analysis (Figure 5) [19], where the electric resistor and the sample are positioned between two 5 cm thick polyurethane foam blocks, with the entire assembly placed between two 5 cm thick aluminum blocks.

A single thermocouple is situated between the resistor and the bottom layer of insulating foam. Temperature values were recorded using the data acquisition system, until stabilization is achieved after 45 minutes. During this transient phase, we analyze the change in temperature difference from the initial value in order to generate the experimental temperature thermogram. Subsequently, we determine the parameters that minimize the quadratic error between this experimental profile and the theoretical model of the hot plate method using a

MATLAB code of the Marquardt-Levenberg algorithm, which gives an estimated thermal effusivity and thermal capacity values.

2.3 Sample Preparation and Shrinkage Study

After getting the clay ready to use, mechanical and thermal samples are prepared by mixing it with water. This mixture appears to shrink more by increasing the water volume as shown in Figure 6. Making 256 cm^3 samples by varying the water-clay ratio from 0,3 to 0,8 and letting them dry for 20 days at ambient temperature has caused a considerable volume diminution. The ratio of 0,3 reduced the original volume by 23,5% and the 0,8 ratio has caused a diminution of 59,4% as illustrated in Figure 7.

Fig. 6. Illustration of clay shrinkage

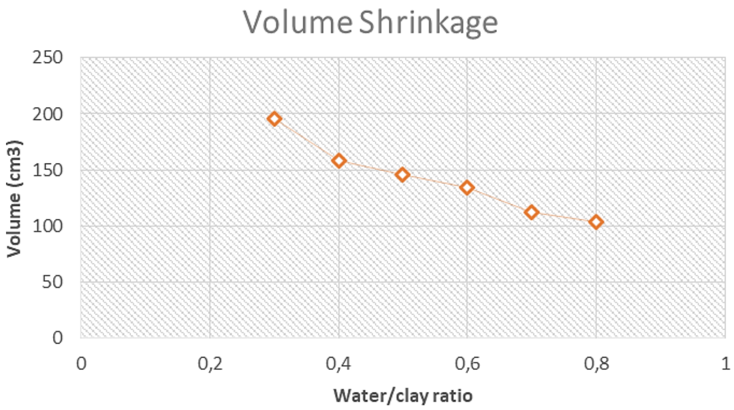

Fig. 7. Volume shrinkage as a function of water-clay ratio

The choice of water-clay ratio should minimize the shrinkage but also have a good texture and excellent workability, which leads us to the water-clay ratio of 0,5 since 0,3 and 0,4 didn't have a good texture and didn't take the shape of the molds. Therefore, 3 samples of 4×4×16 cm^3 for mechanical tests (Figure 8) and 3 samples of 10×10×3 cm^3 for thermal characterization (Figure 9) were made by mixing clay and water with a ratio of 0,5. Concerning the drying process, the samples were dried at ambient temperature for 10 days, and after unmolding the samples they were put in an oven at 40 °C for 24 hours. The mean density value of this material obtained from three samples is given in Table 1.

Fig. 8. Mechanical test samples

Fig. 9. Thermal test samples

Table 1. Density results

Sample	Clay	Relative error (%)
1	1,838	1,23
2	1,772	2,35
3	1,835	1,12
Mean Density Value ($g.cm^{-3}$)	1,815	

3. Results and Discussions

3.1 X-Ray Diffraction

The analysis of our material X-Ray diffraction pattern has given the results shown in Figure 10. There are four major spectra whose intensities are high and they present Quartz (SiO_2), Calcite ($Ca(CO_3)$), Muscovite ($KAl_2Si_3AlO_{10}(OH)_2$) and Kaolinite($Al_2Si_2O_5(OH)_4$). The percentages of these components according to XRD diffractograms are displayed in Table 2.

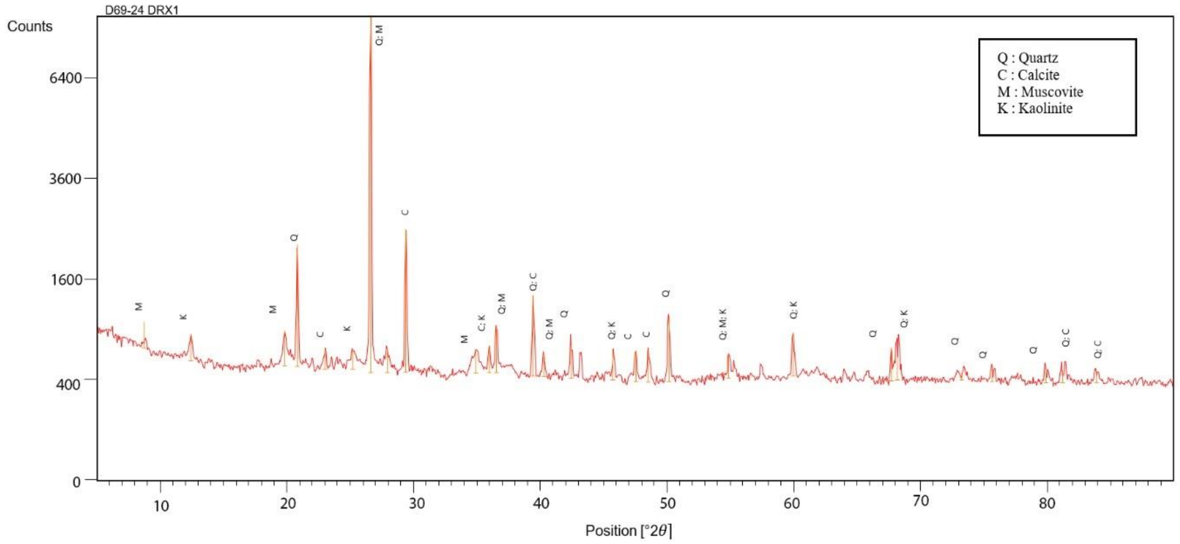

Fig. 10. X - Ray Diffraction pattern of Khenichet's clay

Table 2. Percentages of Khenichet's clay main components

Compound name	ICDD	Score	Scale factor	Chemical formula	Percentage (%)
Quartz	01-085-1054	76	0,986	SiO_2	74,936
Calcite	01-083-0578	57	0,300	$Ca(CO_3)$	17,1
Muscovite	00-007-0025	38	0,049	$KAl_2Si_3AlO_{10}(OH)_2$	1,862
Kaolinite	00-029-1488	31	0,025	$Al_2Si_2O_5(OH)_4$	0,775

This composition allows us to judge that this clay has a good compression strength since quartz is the most dominant component. Also, the high percentage of calcite proves a good resistance to high temperatures. Additionally, the low percentages of muscovite and kaolinite indicate that this clay should be enhanced with other materials to be used in pottery.

3.1 Particle Size Analysis

The Sedimentation method provided us with the granulometric curve shown in Figure 11. This curve, which resembles those of fine-grained materials, proves that this soil have micrometric-sized particle since more than 50% of the particle diameter does not exceed 20 µm and the biggest diameter of this material particle is 50 µm. Its fineness allows it to be a binder that can be mixed with other additives (palm seeds, hemp wool, bamboo fibers, wood waste...[14, 20, 21, 22] or a material useful for water filtration and purification at least from the big micrometric contaminants.

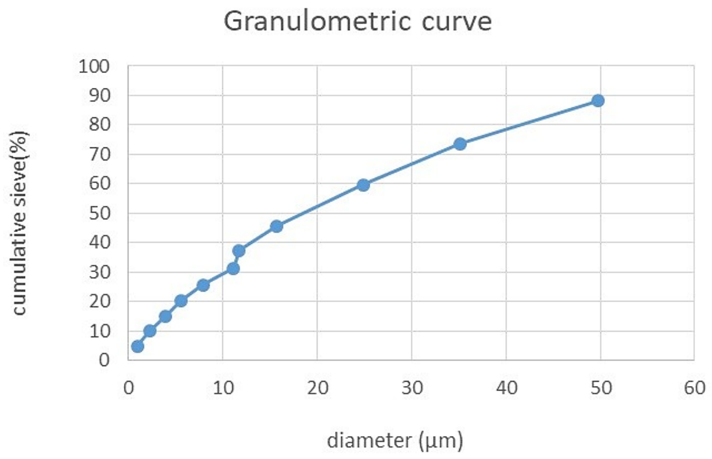

Fig. 11. Particle size analysis

3.2 Atterberg Limits

The plasticity analysis provided the Table 3 giving liquidity limit, plasticity limit, and plasticity index values. According to the Casagrande diagram (Figure 12), these values classify Khenichet's clay among materials of medium plasticity, which confirms that this clay should be enhanced by more kaolinite to be used in pottery.

Table 3. Atterberg limits results

Liquidity limit W_L (%)	Plasticity limit W_P (%)	Plasticity index I_P (%)
43.5	24.13	19.36

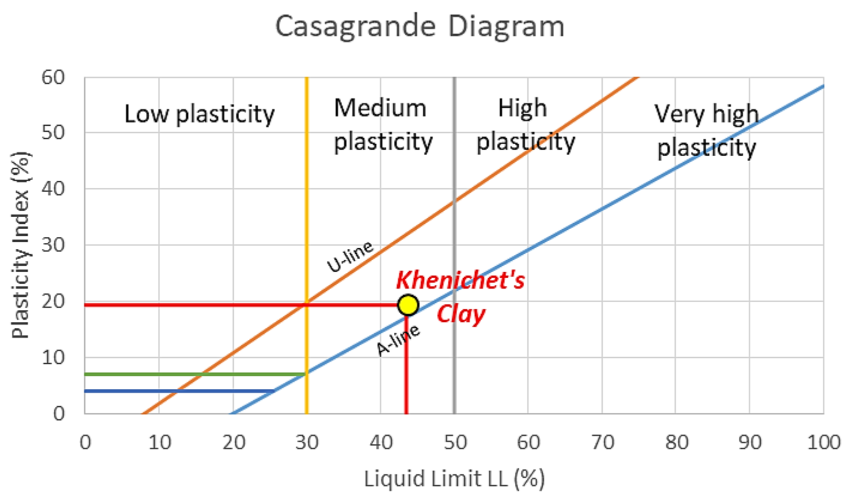

Fig. 12. Casagrande diagram for soil classification

3.3 Differential Thermal Analysis and Thermogravimetric Analysis

The behavior of Khenichet's clay after its exposure to temperature variation from ambient temperature to 1100°C is expressed in Figure 13.

The analysis of the analytical curves shows different zones:

- Zone 1: Around 100 °C, a first endothermic phenomenon is observed, coupled with a mass loss of approximately 1%, related to the release of physiosorbed water [23]

$$2\,FeOOH(s) \rightarrow Fe_2O_3(s) + H_2O\,(g) \quad (1)$$

- Zone 2: Between 300 °C and 480 °C, a second endothermic peak is observed, corresponding to the transformation of goethite into hematite, according to reaction (1). The associated mass loss is about 5%.
- Zone 3: Around 575 °C, an endothermic peak is observed, corresponding to the allotropic transformation of quartz (α → β).
- Zone 4: Between 700 °C and 850 °C, a strong endothermic transformation is recorded, associated with a mass loss of about 15%, which results from the decarbonation of calcite ($CaCO_3$) [24].

$$CaCO_3(s) \rightarrow CaO(s) + CO_2(g) \quad (1)$$

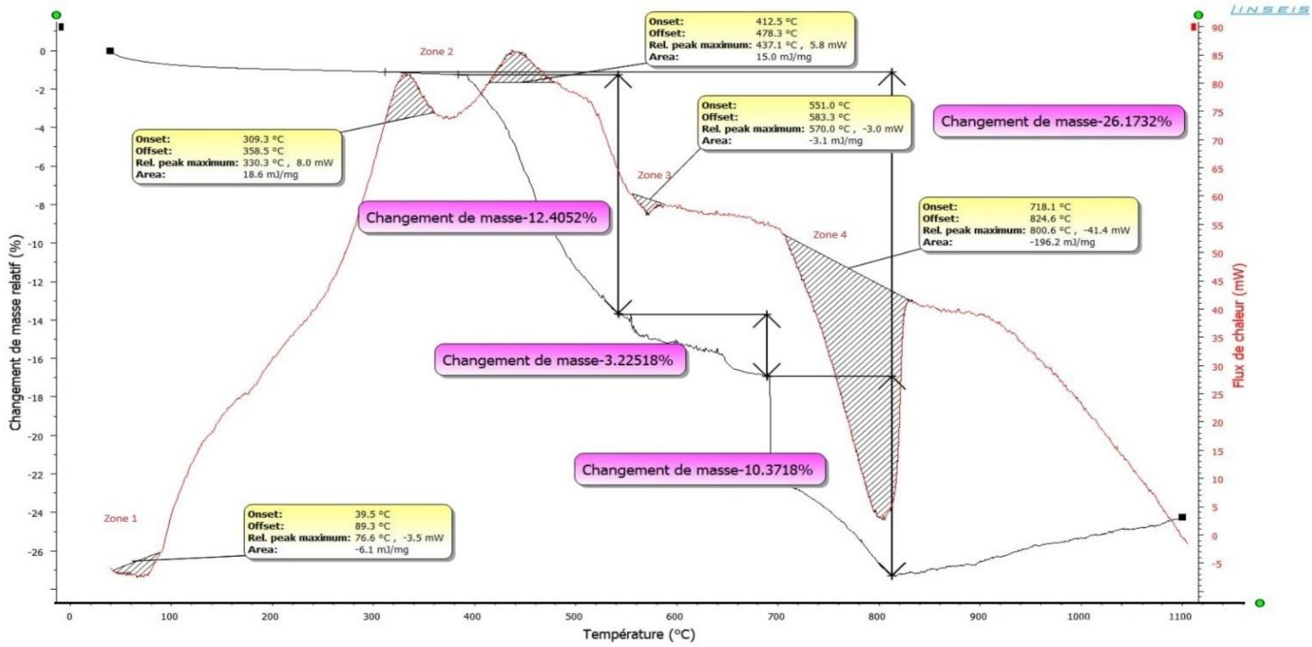

Fig. 13. DTA and TGA thermoanalytical curves

This analysis show that Khenichet's clay is losing a total mass of 26.17% before 850°C, but at 900°C this material become thermally stable, contrary to concrete material that attend stability between 1100°C and 1300°C.

3.4 Mechanical Characterization

Mean values of compression strength of Khenichet's clay are given in Table 4. The value of 4 MPa as compression strength is good since the average value of natural clay's compression strength falls within the 2 to 4.5 MPa range. The high percentage of quartz within this material explains this value since Michot et al. [25] showed that compression strength increases with the elevation of quartz percentage. Also, the fineness of this clayey soil reduces porosity and allows a good bonding between particles which strengthens the mechanical properties [11]. Atbir et al.[10] found a compression strength of 5,7 MPa and a flexural strength of 1,8 MPa for white clay and 4,4 MPa and 1,5 MPa respectively as compression and flexural strengths for red clay and these values were obtained after 90 days. Another work by Atbir et al. [26] investigated the compression behavior at 70 days of a clay material and found a value of 2,05 MPa. As for the clay used by Niyomukiza et al. [22], it has a compression strength of 3,3 MPa and this value was boosted to 4,4 MPa after the firing process. In line with this findings, agricultural wastes, as demonstrated by Kelani et al.[27], can improve the mechanical strength of concrete since the addition of sugar-cane bagasse ash, optimally (around 10%), has increased the compression strength by 12.6%.

Table 4. Khenichet's clay flexural and compression strength

Sample	Compression test			Relative Error (%)
	part 1	part 2	Mean value	Compression strength
1	4,18	4,13	4,155	4,8
2	3,58	3,62	3,6	9,2
3	4,72	3,55	4,135	4,3
Average Strength (MPa)			3,9633	

3.5 Thermal Characterization

This part presents the thermal conductivity, thermal diffusivity, and thermal capacity as three essential parameters in thermal characterization. Table 5 shows the results of thermal conductivity (λ). According to the results of table 5, this material is a moderate insulator with thermal conductivity of 0.62 $Wm^{-1}k^{-1}$ which is a good value falling within the typical range of building materials (0.1 to 2 $Wm^{-1}k^{-1}$)[28] and a better value than conventional concrete. This clay is mainly composed of quartz (75%) and Calcite (13%) with small quantities of Muscovite and Kaolinite, with this composition, it is natural for thermal conductivity to increase [25], in addition, the small particle size of this material decreases the porosity and increases the material's density. Therefore, the possibility of incorporating this clay with big particles of ecological additives will permit the creation of pores allowing more air space, which will cause the improvement of thermal properties.

Table 5. Thermal conductivity of Khenichet's clay samples

Material	Sample	Thermal conductivity (λ)	Sample mean λ value	Relative error	Material mean λ value
Clay	1_{clay}	0,66	0,63	0,01	0,62 $Wm^{-1}k^{-1}$
		0,66			
		0,57			
	2_{clay}	0,61	0,64	0,02	
		0,61			
		0,70			
	3_{clay}	0,60	0,61	0,01	
		0,65			
		0,60			

Generally, ecological additives improve the materials' thermal properties as proved in similar studies; thermal conductivity has decreased by 43% after the addition of granular cork to clay with a volume fraction of 45.6% [15], also the reduction attended 68% by filling the entire apparent volume with granular cork and the clay paste is added in such a way that the intergranular space between the cork granules is filled [16]. In addition to that, Atbir et al. [10] tried mixing red clay and white clay with sheep wool fibers which caused a reduction of thermal conductivity that reached 0.351 $Wm^{-1}k^{-1}$ for white clay and 0.382 $Wm^{-1}k^{-1}$ for red clay for 27g of fibers. Also, straw is very efficient as an ecological additive since it has reduced thermal conductivity from 0.504 $Wm^{-1}k^{-1}$ of a 100% clay sample to 0.263 $Wm^{-1}k^{-1}$ with only 4% of straw added [29]. There are a lot of studies in which a variety of ecological additives has been tested (wood waste, sisal fibers, palm seeds, bamboo fibers, hemp wool...[14, 30, 22, 21, 20]) always leading to the enhancement of thermal properties but with the difference that lies in the percentage of added ecological additives and the degree of thermal conductivity reduction, which indicates the insulating effectiveness of these additives.

As for the results of thermal effusivity, as a fitting parameter of the minimization of quadratic error between the theoretical and the experimental temperature thermograms of the hot plane method in the transient regime, they are presented in Table 6.

Table 6. Khenichet's clay Thermal effusivity (E)

Material	Sample	Thermal effusivity (E)	E sample average value	Relative error	E Material average value
Clay	1_{clay}	1155,79 1313,25 1202,18	1223,74	0,13	1081,32 $Wm^{-2}k^{-1}s^{1/2}$
	2_{clay}	953,19 825,84 1116,18	965,07	0,10	
	3_{clay}	1025,58 1156,50 983,42	1055,16	0,01	

The thermal effusivity of this material is equal to 1081.32 $Wm^{-2}K^{-1}s^{1/2}$, which is a normal value for clays that should be reduced by adding some additives. Atbir et al. [31] reduced thermal effusivity from 873 $Wm^{-2}K^{-1}s^{1/2}$ to 751 $Wm^{-2}K^{-1}s^{1/2}$just by adding 15 g of sheep wool fibers, and Laamrani et al. [32] reduced thermal effusivity by 49% when mixing clay with straw fibers going from 1001 to 510 $Wm^{-2}K^{-1}s^{1/2}$. Eventually thermal capacity ρc is estimated also using the method of the hot plane method in the transient regime and also obtained by minimizing the quadratic error between the theoretical and the experimental thermograms. Table 7 presents the results of this parameter and shows a mean value of 5516.44 $KJm^{-3}K^{-1}$.

Table 7. Thermal capacity results

Material	Sample	Thermal capacity (ρc)	ρc sample's mean value	Relative error	ρc material's mean value
Clay	1_{clay}	2591,68 3781,87 11331,71	5901,75	0,06	5516,44 $KJm^{-3}k^{-1}$
	2_{clay}	2779,69 5295,77 3077,05	3717,51	0,32	
	3_{clay}	2343,16 3580,08 14866,95	6930,07	0,25	

4. Conclusion

This work evaluated gray clay thermophysical properties sourced from the Gharb region of Morocco. Khenichet's clay is a fine material having particles not exceeding 50µm which exhibits considerable promise in purification and water potential. Also, the medium plasticity allows to take advantage of this clay in ceramic production. Additionally, it is composed mainly of Quartz and Calcite with fraction of Muscovite, and Kaolinite, therefore it should be mixed with other types of clay in order to have an enhanced structure in pottery and ceramics. However, the Quartz indicates a good compression strength of 4MPa and the Calcite caused the enhancement of thermal properties with a thermal conductivity of 0.6 $Wm^{-1}K^{-1}$, a thermal effusivity of 1081 $Wm^{-2}k^{-1}s^{1/2}$ and a thermal capacity of $KJm^{-3}K^{-1}$. Although, there is a possibility to improve mechanical properties using stabilization with cement or lime, the firing process at elevated temperatures-often exceeding 900°C- requiring a considerable amount of energy, or the compression techniques that involve high-pressure processing demanding mechanical energy and infrastructure that may not be readily accessible or sustainable, Hence, these energy-consuming transformations will increase the embodied energy of this material's production process, causing more emissions of greenhouse gases and more pollution, which will make this material lose its natural and ecological aspect . On the other hand, thermal properties indicate a great insulating potential and this material can be improved by incorporating this clay with ecological additives (seeds, fibers, wastes, porous materials...), which introduce air-filled voids to reduce thermal conductivity and improve the overall thermal performance. Finally, the characterization of this clay demonstrated the great potential of the studied clay and allows us to have the perspective of enhancing its properties and utilize it as a sustainable construction, thermally efficient material, and potentially in water filtration. Its local availability, fine particle size, and favorable thermomechanical properties make it a viable eco-material.

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