



Original Article

Physico-chemical characterization of clay raw materials from the Thicky quarry (Senegal) for the manufacture of earth bricks

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ABSTRACT

This study focuses on the physico-chemical characterization of the clayey raw materials of the Thicky quarry. We made a geotechnical, mineralogical, chemical and microscopic study of the laterite and the Thicky clay. Mineralogical analysis by X-ray diffraction (XRD) reveals that our clay consists mainly of quartz (Q), kaolinite (K), illite (I) and calcite (C) while laterite consists of quartz (Q), kaolinite (K), hematite (H) and calcite (C). The results of the chemical composition show the presence of SiO_2 , Al_2O_3 and Fe_2O_3 in large quantities and also K_2O , CaO , MgO , Na_2O , TiO_2 and P_2O_5 in small quantities in our materials. Infrared spectroscopy reveals the presence of relatively broad absorption bands located around 3500 cm^{-1} which are related to phyllosilicates such as kaolinite or illite. Microscopic observation of clay and laterite samples shows that the texture is relatively compact, formed of several superimposed planar sheets with turbostratic disorder. The results obtained show that these clayey raw materials are poor in organic matter and calcium carbonates so they can be used for the manufacture of earth bricks.

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1. INTRODUCTION

To understand a material, it is essential to characterize it, that is to say to determine its properties. The characterization of a material in a general way is the whole of the processes of identification which allow to evaluate the properties of the material.

The use of earth-based materials in the building industry requires a good knowledge of their physicochemical properties in order to best meet the severe constraints imposed in the construction sector.

Today, more than any other material, soil is the basis of human construction. Its popularity, availability, ease of extraction and processing, ensure its use as a building material for the future. The chemical nature, the physical form, the surface state of the different materials constituting this material give it particular properties that can be used in several industrial sectors. Thus, to properly use a material in construction, it is important to know its specific characteristics. Raw materials are valued according to their physical and chemical properties, in relation to the available industrial

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processes. Depending on the origin of the raw materials, a small variation in one of these characteristics can considerably limit the field of application [1]. In an industrial context, it is necessary to be interested in the size, shape, density, specific surface and structural characteristics while identifying the chemical and mineralogical compositions of the raw materials. The earth used in construction must meet the technological requirements in order to obtain good quality products. Thus the raw materials must be poor in organic matter and have a good plasticity to facilitate the manufacture of bricks which it ensures the cohesion in raw [2].

Numerous material characterization techniques exist and are essentially based on various basic physico-chemical principles.

These are characterizations:

- geotechnical (natural water content, organic matter content, Atterberg limits, methylene blue test) ;
- microscopic;
- chemical (Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES), X-ray fluorescence, atomic absorption spectroscopy);
- mineralogical (X-ray diffraction, infrared spectroscopy).

In our study, we are interested in clay and laterite which are materials used in Senegal in construction. The objective of this work is to determine the physico-chemical properties of clay and laterite from Thicky quarry (Senegal).

2. MATERIALS AND METHODS

2.1. Presentation of the Site

The soil of Senegal contains a large number of clay and laterite deposits [3, 4].

The study was conducted on clay soil and laterite collected from the Thicky quarry. Thicky is located in the Thies region, in western Senegal at 14°50' N, 17°06' W. The geographical map is shown in Figure 1.

2.2. Geotechnical Identification Tests

a) Natural water content (*W*)

The water content of the various samples was determined by the method of drying in the oven, following the experimental standard [XP CEN ISO/TS 17892-1, 2005] [1]. This involves determining the mass of free water eliminated by drying in an oven at a temperature of 105°C. The determination of the natural water content is based on the following formula:

$$W(\%) = \frac{m_h - m_s}{m_s} \times 100 \quad (1)$$

with

mh: mass of the soil sample in its natural state;

ms: mass of this soil sample after being oven dried at 105°C for 24 hours;

mh - ms = mass of water [2].

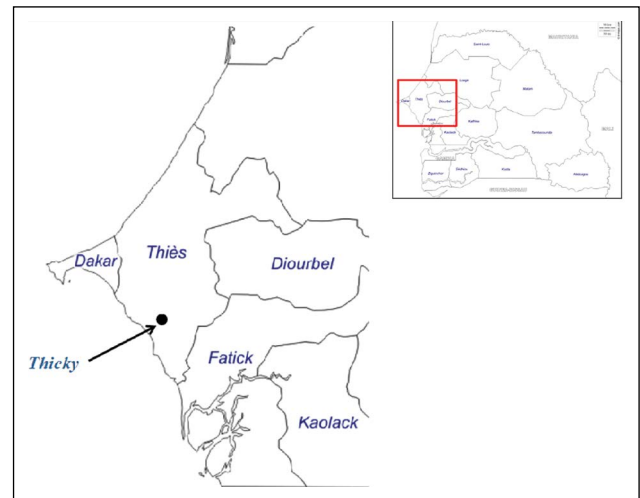


Figure 1. Position of Thicky in Thies region (Senegal).

b) Organic matter (MO) content

The organic matter (MO) content in the soil is equal to the ratio of the difference between the initial sample mass m and the sample mass after the reaction with hydrogen peroxide m' to the initial test mass m (100 grams) [2].

It was determined according to the french standard [NF P94-055, 1993] [5].

The organic matter (MO) content is calculated by the formula:

$$MO(\%) = \frac{m - m'}{m} \times 100 \quad (2)$$

c) Calcium carbonate ($CaCO_3$) content

The calcium carbonate ($CaCO_3$) content was determined according to the french standard [NFP94-048, 1996] [6], using the Bernard calcimeter.

The $CaCO_3$ content is calculated by the formula:

$$\%CaCO_3 = \frac{m_t V_b (\theta_t + 273)}{m V_t (\theta_b + 273)} \times 100 \quad (3)$$

with

V_b : volume of carbon dioxide (CO_2)

V_t : volume of CO_2 after reaction with excess HCl

m : mass of soil sample

m_t : mass of pure $CaCO_3$

θ_b and θ_t : the test room temperatures, respectively, during the material test and during the pure calcium carbonate test, expressed in degrees Celsius.

d) Particle size analysis

Particle size analysis presents the percentage distribution of solid particles according to their dimensions.

For our raw materials, the particle size analysis was done according to two techniques: the coarser fraction ($>80 \mu m$) was analyzed by wet sieving, and the finer fraction ($<80 \mu m$) by sedimentometry according to the standard [NF P 94-057, 1992] [7].

e) Atterberg limits

Atterberg limits are tests that allow to define indicators qualifying the plasticity of a soil, and more precisely to predict the behavior of soils during earthwork operations, in particular under the action of water content variations. The purpose of these tests is to determine the liquidity limit W_L and the plasticity limit W_p of the raw materials. The difference between the liquidity and plasticity limits gives the plasticity index I_p .

The liquidity limit (W_L) was measured by the method of the dish of Casagrande and the plasticity limit (W_p) by the method of the roller. These measures were realized according to the french standard [NF P94-051, 1993] [8].

f) Methylene blue test

This is to determine the necessary amount of this dye to cover the internal and external surfaces of all the clay particles present in the sample. The test consists in measuring by dosage the quantity of methylene blue which can be adsorbed by the material suspended in water. The methylene blue value (MBV) was determined on the total sample according to [NF P94-068, 1998] [9].

The specific surface of particles can be determined by the amount of adsorbed methylene blue (MB). Thus, the specific surface area (S_s) can be calculated from the following equation [10]:

$$S_s = \frac{m_{MB}}{m_s} \times \frac{A_v}{319.86} \times A_{MB} \quad (m^2 / g) \quad (4)$$

where

m_{MB} is the mass of the absorbed MB at the point of complete cation replacement, m_s is the mass of the soil specimen, A_v is Avogadro's number ($6.02 \times 10^{23} \text{ mol}^{-1}$) and A_{MB} is the area covered by one MB molecule (typically assumed to be 130 \AA^2).

g) Bulk density of raw materials

The bulk density of a material is the density of a cubic meter of the material taken in a heap, including both permeable and impermeable voids of the particle as well as the voids between particles. The sample to be tested is placed in a funnel closed by a metal plate. This plate is slid and the aggregates are collected in a cylinder of known volume (V). After removing the excess material, the mass (m) of the material is determined.

The bulk density is given by the following equation:

$$\rho_{app} = \frac{m}{V} \quad (5)$$

2.3. Mineralogical Analysis of Raw Materials

a) X-ray diffraction (XRD)

The mineralogical composition of the samples was determined by the X-ray diffraction technique. This technique allows the identification of the different crystallized mineral phases present in the sample. The general method consists in bombarding the sample with X-rays coming from a

metal anode and which are first collimated by a divergence slit in order to produce a sub-parallel beam.

X-ray diffraction (XRD) analysis was carried out using a diffractometer model X'PERT Pro MPD PANALYTICAL. The equipment uses a copper anode tube ($\lambda=1.789 \text{ \AA}$), a standard resolution goniometer containing the geometry for performing measurements, symmetrical and asymmetrical configurations, a minimum step size of 0.002° and an X-ray proportional counter.

b) Infrared spectroscopy (IR)

Infrared spectrometry is one of the most widely used tools for the characterization and identification of organic molecules. It is used to determine the functional groups present in the material. The method of infrared spectroscopy requires the use of an infrared-transparent medium such as potassium bromide (KBr). The method involves grinding a few milligrams (0.5 to 1 mg) of the sample in the presence of dry KBr powder in an agate mortar. The mixture is then compressed in a hydraulic press.

The infrared spectra of our raw materials are obtained thanks to a Nicolet 6700 FT-IR apparatus on a recording interval included between 400 cm^{-1} and 4000 cm^{-1} medium infrared range.

2.4. Chemical Analysis of Raw Materials

The elemental chemical analysis consists in measuring in atomic form the different chemical elements that constitute the sample. The elemental chemical analysis of our raw materials was obtained by ICP-AES (Atomic Emission Spectrometry in an inductively coupled plasma). Nitric (HNO_3) (2 ml) and hydrofluoric (HF) acids (6 ml) were used for wet solution in a volume of 100 ml. The results were obtained under a pressure of 60 bar with a step of 0.8 bar/second, at the temperature of 240°C , with a power of 900 W, a ramp of 20 min and a step of 30 min.

2.5. Microscopic Observations by SEM

Scanning Electron Microscope (SEM) allows to observe the surface topography of a sample by scanning its surface with an electron beam and collecting the image formed. For our samples, clay powder was deposited on a carbonaceous pellet. This pellet is then metallized by covering it with a thin layer of metal by sputtering platinum to make it conductive. Once metallized, the sample is introduced into the SEM chamber for analysis.

3. RESULTS AND DISCUSSION

3.1. Geotechnical identification tests

a) Natural water content (W)

The results of the natural water content of our samples are presented in Table 1.

Both samples have low natural water contents. However, the clay sample has a natural water content 4.78% higher than the laterite sample which has a water content of 1.50%.

Table 1. Natural water content of samples

Samples	Wet mass: m_h (g)	Dry mass: m_s (g)	Water content: W (%)
Clay	99.88	95.32	4.78
Laterite	100	98.52	1.50

Table 2. Organic matter content of samples

Samples	Initial test mass: m (g)	Mass after the reaction: m' (g)	Organic matter content MO (%)
Clay	100	95.09	4.91
Laterite	100	98.54	1.46

Table 3. Carbonate content and classification of the different samples

Samples	Carbonate content in %	Classification
Clay	5	Not marl
Laterite	1.25	Not marl

Variations in natural water content may be related to the amount of fine or clay elements contained in the analyzed samples.

b) Organic matter (MO) content

The results of the organic content of our samples are presented in Table 2.

The results show that Clay is weakly organic (OM = 4.91%) while Laterite is non-organic (OM = 1.46%) [11].

Therefore, their negative effect in the brick making process (stabilization or firing) is negligible.

c) Calcium carbonate ($CaCO_3$) content

The results of the calcium carbonate content of our samples are presented in Table 3.

Analysis of the results shows that both samples are classified as non-marl materials, since the calcium carbonate content does not exceed 10%.

d) Particle size analysis

Figure 2 show the results obtained. The results indicate that Thick clay is composed mainly clay (45%), silt (47%) and sand (8%). Laterite is composed clay (8%), silt (20%) and sand (72%) [12].

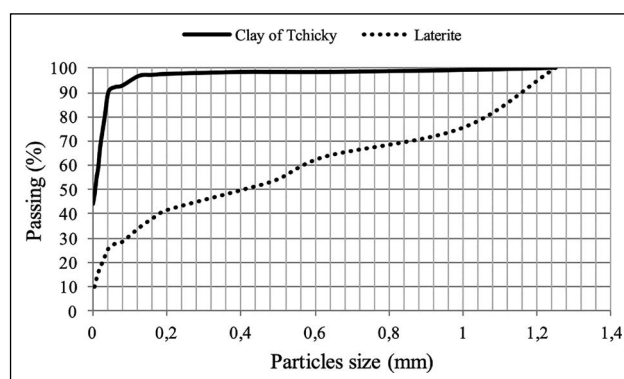
e) Atterberg limits

The liquidity limit of the clay is 41.8% and that of the laterite is 38%.

Plasticity limit of the clay is 24.3% while the plasticity limit of the laterite is 21.9%. Plasticity indices of clay and laterite are 17.5% and 16.1% respectively [12]. These results show that the Thick clay and laterite are moderately plastic [13].

f) Methylene blue test

The specific surface of the clay S_s (Clay) = $83.25 \text{ m}^2/\text{g}$ shows that our clay contains illite [10]. The specific surface of laterite S_s (Laterite) = $22.76 \text{ m}^2/\text{g}$ shows the presence of kaolinite [10].

**Figure 2.** Particle size curves of raw materials (clay and laterite) [12].

The particle size results could also confirm these obtained values of specific surface area because Thick clay contains more fine fraction ($<2 \mu\text{m}$) than laterite. The finer the clay the greater its specific surface area. Also, these specific surface area values suggest that both Thick clay and laterite may contain clay minerals such as illite and kaolinite.

g) Bulk density of raw materials

The bulk density of clay is 1297 kg/m^3 and that of laterite is 1366 kg/m^3 . Both materials have bulk densities of the same order of magnitude. But clay has a lower bulk density than laterite. So clay is less dense than laterite.

3.2. Mineralogical Analysis of Raw Materials

a) X-ray diffraction (XRD)

The diffractograms of the two raw samples are shown in Figure 3.

The results show that the Thick clay (TR1) and laterite (TR2) are essentially composed of quartz and clay.

To differentiate the clays, it is therefore necessary to perform the clay slide test.

Thus, we performed various treatments to modify the inter-sheet space: heating, acidification, replacement of the interfoliar cation. Two of these treatments were successively applied, namely saturation by a polyalcohol (Ethylene-Glycol: E.G) and heating to 500°C on the oriented preparations already analyzed under natural conditions.

Saturation by a polyalcohol aims at swelling the clay mineral sheets by introducing, in the interfoliar space, ethylene glycol molecules of big size.

Heating allows the characterization of clay minerals particularly sensitive to heat such as kaolinite and other hydrated minerals.

We can therefore recognize the clay by looking at the evolution of the diffraction peaks at low angles according to the treatment. The XRD results of the oriented aggregate slides are shown in Figure 4.

The diffractograms of the oriented aggregate slides show that the Thick clay (TR1) consists of kaolinite (7.13 \AA) and illite (10 \AA) while the laterite (TR2) consists of kaolinite (7.13 \AA).

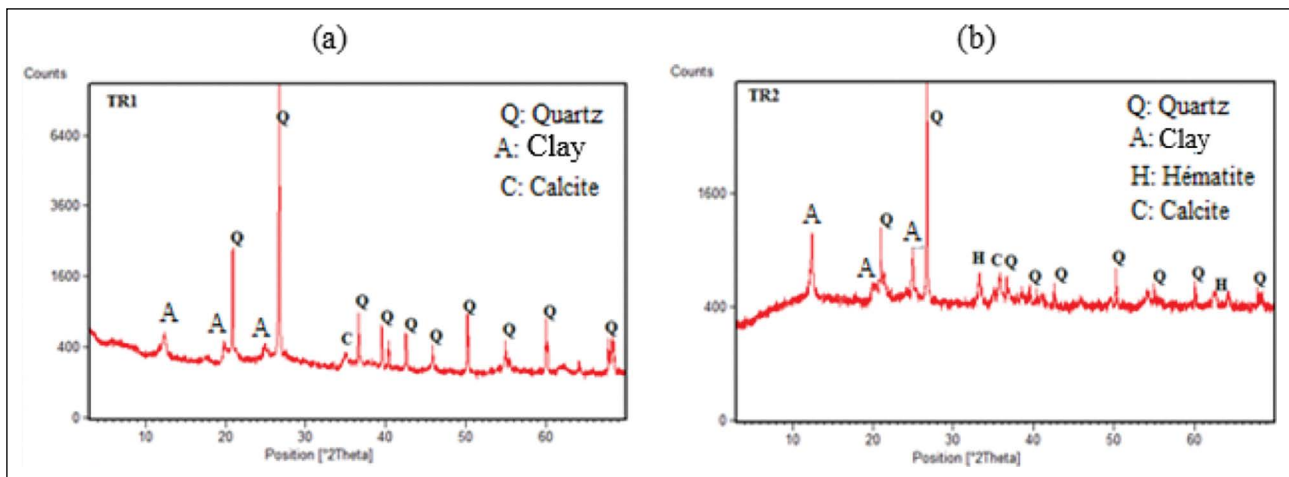


Figure 3. X-ray diffractograms of clay (a) and laterite (b) powders before using oriented aggregate slides.

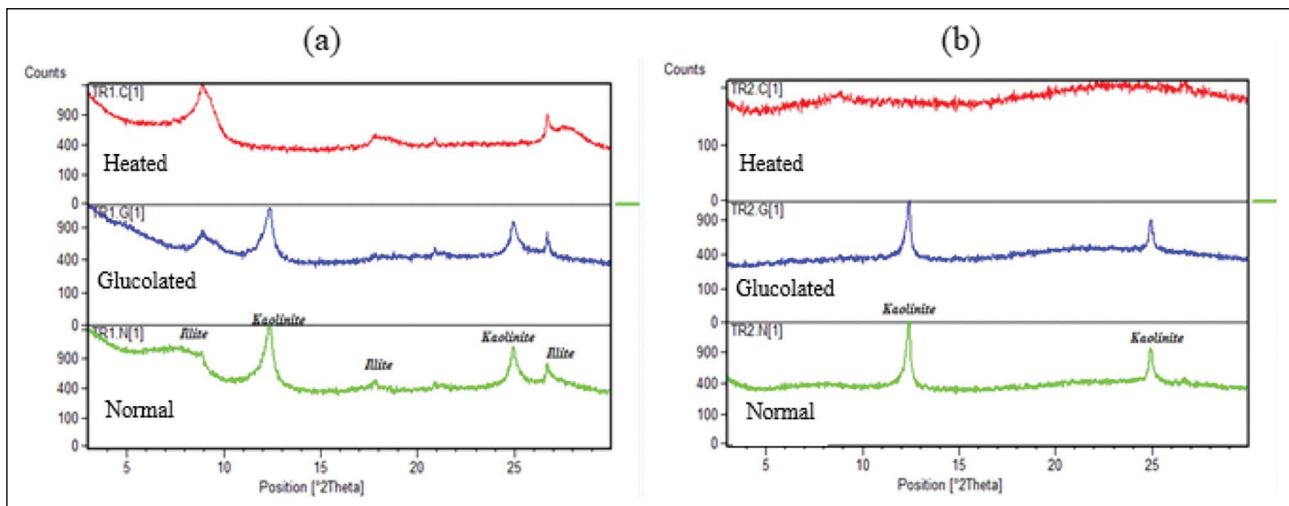


Figure 4. X-ray diffractograms of oriented aggregate slides of clay (a) and laterite (b).

Table 4. Chemical composition of raw materials (wt %)

Sample	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	MnO	MgO	CaO	Na ₂ O	K ₂ O	TiO ₂	P ₂ O ₅	PF
Laterite	23,00	22,77	33,67	<L.D.	0,13	0,16	0,03	0,11	1,32	1,63	16,14
Clay	74,04	12,16	4,12	<L.D.	0,69	0,19	0,16	1,24	1,04	0,10	7,05

Figure 5 shows the diffractograms of the two raw materials studied.

The XRD results show that the Thick clay consists mainly of quartz (Q), kaolinite (K), illite (I) and calcite (C) while the laterite consists of quartz (Q), kaolinite (K), hematite (H) and calcite.

b) Infrared spectroscopy (IR)

Within the framework of our study, the infrared spectra of clay and laterite presented on Figure 6 are obtained thanks to an apparatus of type Nicolet 6700 FT-IR on an interval of recording ranging between 400 cm⁻¹ and 4000 cm⁻¹ medium infrared domain.

From these spectra, two essential groups stand out: the OH and H₂O groups (bands of 1640 cm⁻¹ and 3456 cm⁻¹)

characterizing respectively deformation and elongation vibrations; and the Si-O group (broad, intense band centered around 1027 cm⁻¹) corresponding to the bond valence vibration in clay minerals.

The relatively broad absorption bands located around 3500 cm⁻¹ are related to phyllosilicates such as kaolinite or illite.

The absorption band around 1640 cm⁻¹ may be due to the presence of interfoliar water.

Overall, the similar patterns of the IR spectra confirm the information provided by the X-ray diffraction. However, elemental chemical analysis will help to further this information by providing the amount in oxide of each chemical element.

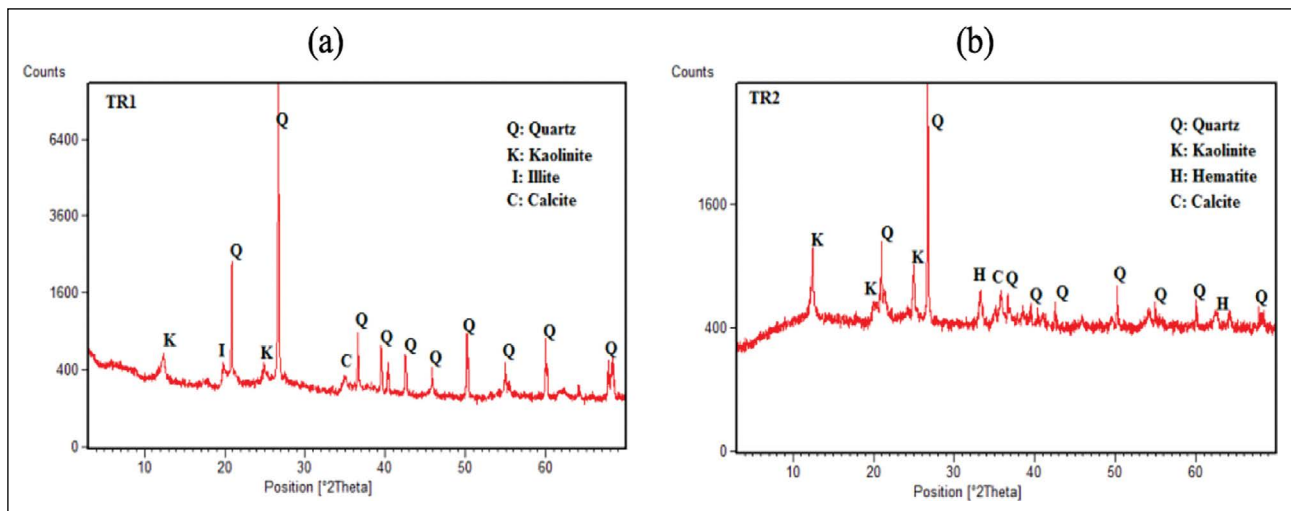


Figure 5. X-ray diffractograms of clay powders (a) and laterite (b).

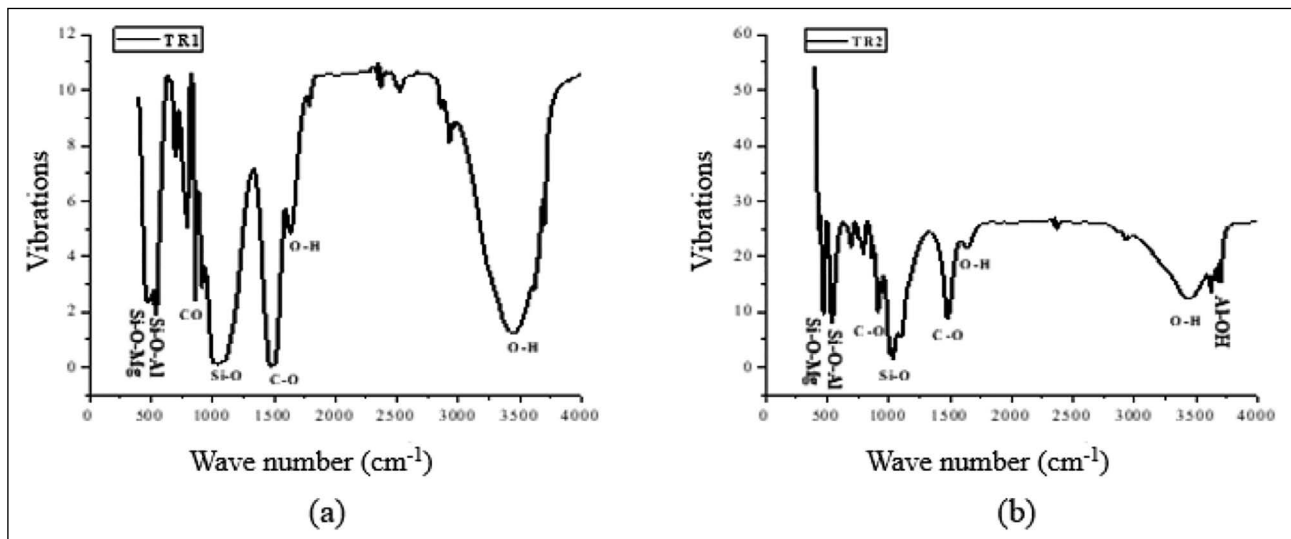


Figure 6. Infrared spectra IR of clay (a) and laterite (b).

3.3. Chemical Analysis of Samples

Chemical composition of samples was determined by Bodian et al. [12].

The results of the chemical composition of the samples are presented in Table 4.

The results showed that SiO₂, Al₂O₃ and Fe₂O₃ are the most abundant oxides in both samples while K₂O, CaO, MgO, Na₂O, TiO₂ and P₂O₅ are present only in small amounts.

The SiO₂/Al₂O₃ ratio of the clay sample shows that there is a large amount of kaolinite in the material. The results also indicate a low illite content in the clay and the presence of a large amount of quartz in the clay than in the laterite.

3.4. SEM Microscopic Observations

The microstructure of the samples was observed and analyzed using a JSM-5400 scanning electron microscope (SEM).

Figure 7 and Figure 8 show the microscopic images of the different samples.

Microscopic observation of clay and laterite samples shows that the texture is relatively compact, formed of several superimposed planar sheets with a turbostratic disorder.

We observe the presence of quartz in both samples and that the matrix of the laterite sample is denser than that of the clay. We also note the presence of some pores and microcracks.

Both samples have an open texture with no preferential arrangement of the laminae. The random orientation reveals voids in the structure of the samples, and subsequently the appearance of a network of discontinuities.

4. CONCLUSION

In this paper, we have determined the physico-chemical characteristics of the clayey raw materials from Thick quarry.

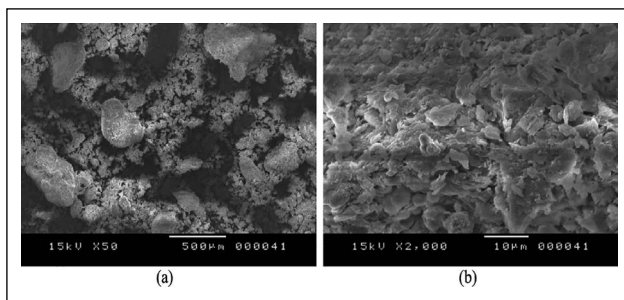


Figure 7. SEM images of clay.

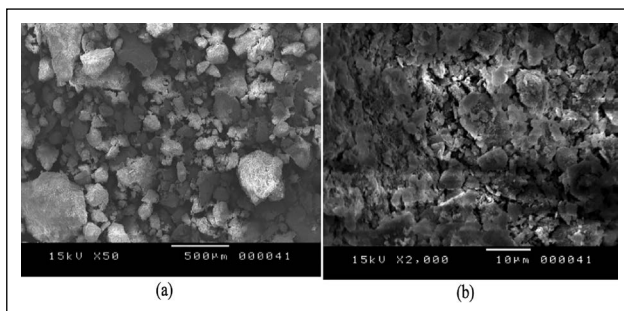


Figure 8. SEM images of laterite.

We determined the water content, organic content and calcium carbonate CaCO_3 content of the raw materials. The water content results show that both samples have low natural water contents.

The results of organic matter content show that the Clay is low organic while the Laterite is non-organic. Therefore, the negative effect of organic matter in the brick making process (stabilization or firing) is negligible.

The calcium carbonate content results show that both raw materials are classified as non-marl materials, since the calcium carbonate content does not exceed 10%. Both materials have bulk densities of the same order of magnitude.

Mineralogical analysis by X-ray diffraction (XRD) reveals that our clay consists mainly of quartz (Q), kaolinite (K), illite (I), and calcite (C) while laterite consists of quartz (Q), kaolinite (K), hematite (H), and calcite.

The results of the chemical composition show the presence of SiO_2 , Al_2O_3 and Fe_2O_3 in large quantities and also K_2O , CaO , MgO , Na_2O , TiO_2 and P_2O_5 in small quantities in our materials.

Infrared results reveal the presence of relatively broad absorption bands located around 3500 cm^{-1} which are related to phyllosilicates such as kaolinite or illite.

Microscopic observation of clay and laterite samples shows that the texture is relatively compact, formed by several superimposed planar sheets with turbostratic disorder.

These results of the physico-chemical characterization show us that our two raw materials can be used in construction because they present the characteristics sought in the processes of manufacture of the earth bricks.

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DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declare that they have no conflict of interest.

FINANCIAL DISCLOSURE

The author declared that this study has received no financial support.

PEER-REVIEW

Externally peer-reviewed.

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