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Characterization and Evaluation of Some Technological Properties of a Clay Soil Sample Collected at Loudima (Congo-Brazzaville)

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Abstract

This work aims to characterize clay soil taken from Loudima, one of the localities in the department of Bouenza in Congo Brazzaville. X-ray diffraction, infrared spectroscopy, MEB, X-ray chemical analysis and mechanical tests were performed. The results obtained show that the Loud soil sample is a mixture of Kaolinite (41.33%), quartz (16.03%) and iron oxide (2.93%). By chemical analysis, we noted that the SiO_2/Al_2O_3 ratio = 2.16, which indicates that this soil contains a significant content of quartz. The MEB showed the presence of scattered clay leaves in the form of sticks and some aggregates. The technological tests carried out with this soil have a linear shrinkage of 3.1% at 1150˚C., an absorption rate of 6.1% at 1150˚C., a compression strength of 25.5 MPa at 1150˚C., and a flexural strength of 4.7 MPa at 1150˚C. This gives the Loud sample the possibility to be used as a raw material for the manufacture of terracotta bricks.

Subject Areas

Fundamentals of Material Science

Keywords

Clay, Characterization, Technological Tests, Bricks

1. Introduction

The manufacture of building materials and pottery dates back to antiquity. Use clay in applications industrial is a tributary of knowledge of their properties physico-chemical and technology. Clay bricks have always been an integral part of building materials [1]. Due to their mechanical and thermal properties, these bricks are increasingly stressed [2]. The raw material used is clay. The importance of materials to base clay in the ceramics industry, results from their plastic properties and the property of the shaped product to keep it shaped after cooking. The complexity of the transformations that the product undergoes shaped, depends on the chemical and mineralogical composition of the raw material and the nature and quantity of impurities present.

In Congo Brazzaville, a large part of the clay deposits is not characterized nowadays despite their artisanal use as raw material for building materials.

The clay taken from Loudima is used by craftsmen to make cooked bricks. These bricks are made by hand, although some craftsmen use manual presses or wooden molds. Cooking is carried out using artisanal ovens with a variety of energy sources, so the cooking time is random; the products of such a cooking lack uniformity and have cracks in the absence of being recovered to break while presenting a certain fragility.

Such products discourage craftsmen and encourage them to abandon brick plants, as has been observed in Loutété, Makoua and many other localities in the country (Congo).

These concerns are well in line with the major project to characterize Congolese clay soils with a view to their upgrading by the Laboratory of Mineral and Applied Chemistry (LaCMA).

It is in this perspective that we have looked at the physicochemical characterization and thermomechanical behavior of the clay soil of Loudima in order to identify the causes that could justify the problems faced by the craftsmen of this locality, to formulate possible proposals to help improve the quality of the bricks to be produced.

2. Materials and Methods

2.1. Location of the Study Area

The sample was taken at Loudima in the department of BOUENZA. The map in **Figure 1** shows the location of the sampling site.

2.2. Experimental Techniques and Procedure

The characterization techniques used in this study are X-ray diffraction, infrared spectroscopy, scanning electron microscopy, chemical analysis and technological testing.

2.2.1. Geotechnical Characterization

The geotechnical study was carried out at the Office of Building and Public Works Control (BCBPT). The Atterberg limits (liquidity limit, plasticity limit and plasticity index) were measured according to NF P 94-051 [4]. The particle size analysis was carried out according to NF P 94-057 [5].

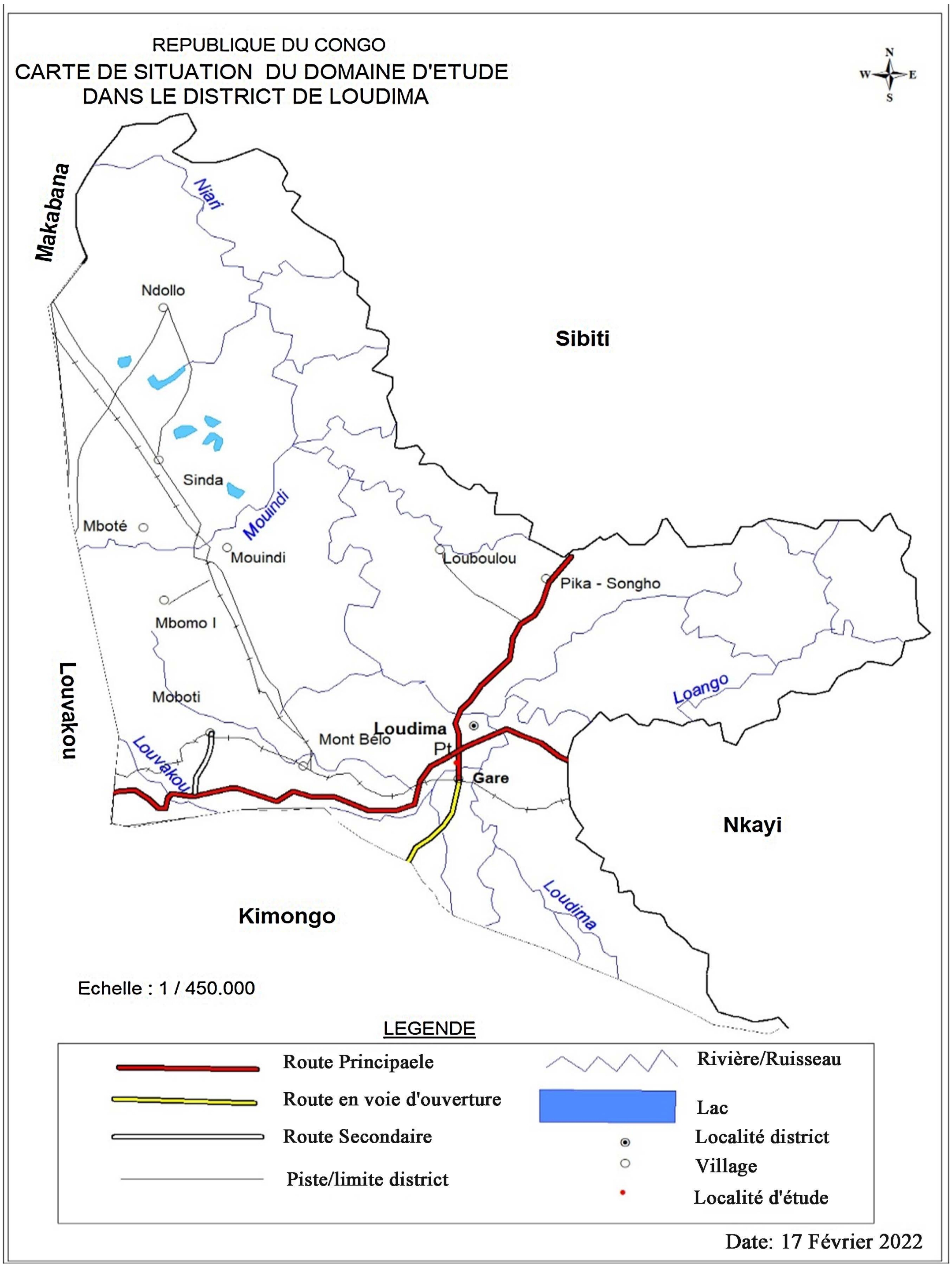

Figure 1. Location of the sampling site on the map of the Bouenza department [3] (Geographical Institute, February 2022).

2.2.2. X-Ray Diffraction (XRD)

The crystalline phases were identified by X-ray diffraction on powders with a Philips X-pert pro-MPD X-ray diffractometer with Cu-K radiation at 40 KV and 40 mA operating at an angle of 2˚ between 0˚ and 60˚. This study was conducted at the Ithemba Laboratory in Cape Town, South Africa.

2.2.3. Infrared Spectroscopy

This technique has proved to be a valuable tool known mainly for its sensitivity to detecting highly hydrated minerals with high thermal instability (Maglione 1975).

The FOURIER Transform Infrared Spectra (IRTF) was performed using a PERKIN ELMER SPECTRUM 100-FT-IR SPECTROMETER at the chemistry laboratory of the University of Western Cape in Cape Town, South Africa.

2.2.4. Scanning Electron Microscopy

The advantage of scanning electron microscope (SEM) observation is that it allows us to visualize surfaces, and to highlight details of great finesse especially to characterize the inter and intra-granular spaces (the first space between the grains and within the bonding phase, the second is in the constituent grains). The observation was carried out at the Ithemba laboratory in Cape Town, South Africa.

2.2.5. Chemical Analysis

The elemental chemical analysis of the Loud sample was performed using an optical emission spectrometer (OPEC-ICP) at the Science Service Laboratory (SC) in Cape Town, South Africa.

2.2.6. Technological Properties

1) Preparation and firing of test samples

The sample of clay soil is dried at 105˚C. in an oven. Grinding and sieving with a 34 (2 mm) module sieve are applied. After weighing a given mass and moistening taking into account the results of the Proctor test, briquettes are shaped with a mold of dimensions 4 cm × 4 cm × 16 cm.

The briquettes are dried in the open air for 28 days and then at different temperatures in an oven.

2) Determination of technological properties

The linear cooking shrinkage is measured using the sliding foot. Let Lo and L be the lengths of the briquettes before and after the heat treatment.

Linear shrinkage is given by the following relation:

$$R_L = \frac{L_0 - l}{L_0} \times 100 \tag{1}$$

The 3-point bending strength was measured according to EN 196-1 [6] [7].

The following formula is used to calculate:

$$\sigma_{RF} = \frac{3}{2} \times \frac{F_f \times L}{b \times d^2} \tag{2}$$

On the other hand, the absorption rate is carried out according to ASTM C 20-74 [8]. This water absorption is calculated by the following relation:

$$Abs = \frac{m_h - m_s}{m_h} \times 100 \tag{3}$$

On the other hand, the compressive strength is given by the following relation:

$$R_c = \frac{F_m}{s} \tag{4}$$

3. Results and Discussions

3.1. Geotechnical Properties

The results of the particle size distribution and the Atterberg limits of the Loud sample are given in Table 1.

Of the grain size, the clay soil of Loudima (Loud) consists mainly of 49% clay particles and 33% silt. There is 18% sand.

With a fine particle content of around 40%, as shown in Moutou *et al.* [9] [10] [11] and Wetshondo [12], Loud is a raw material in the manufacture of terra-cotta materials. (See Figure 2)

Using the texture triangle, Loud is revealed to be a clay-textured soil [13].

This texture indicates the presence of a clayey species of low plastic (Figure 3) type 1/1.

Table 1. Geotechnical properties of the Loud sample.

	Clay	Silt	Sand
Granulometry	49%	33%	18%
	Liquidity limit	Plasticity limit	Plasticity index
Atterberg limit	41%	20.3%	20.7%

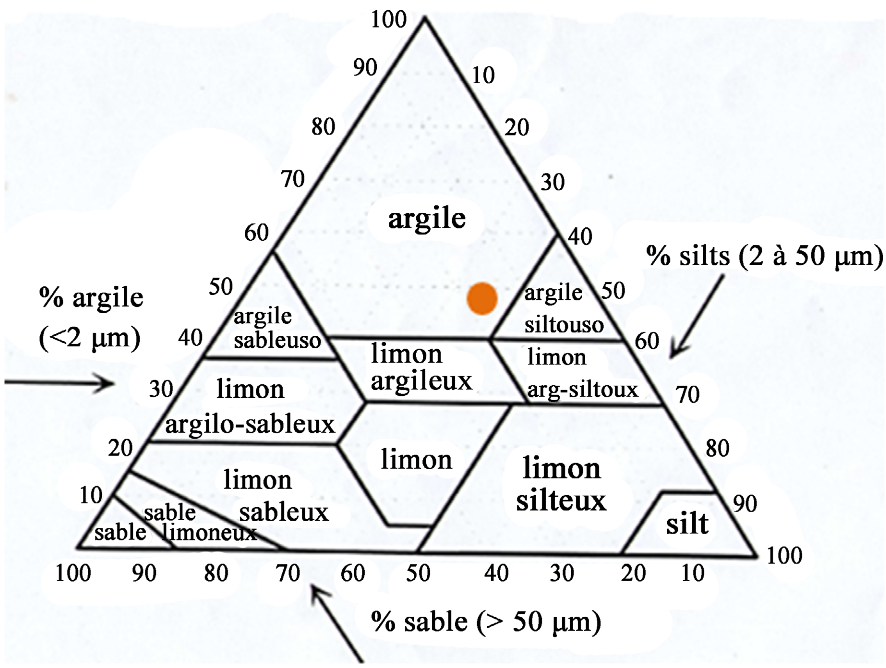

Figure 2. Loud sample position in texture triangle

With a clay texture, we can check the frequency of use of this soil after mixing with water. We thus use the Sheppard triangle shown in Figure 4.

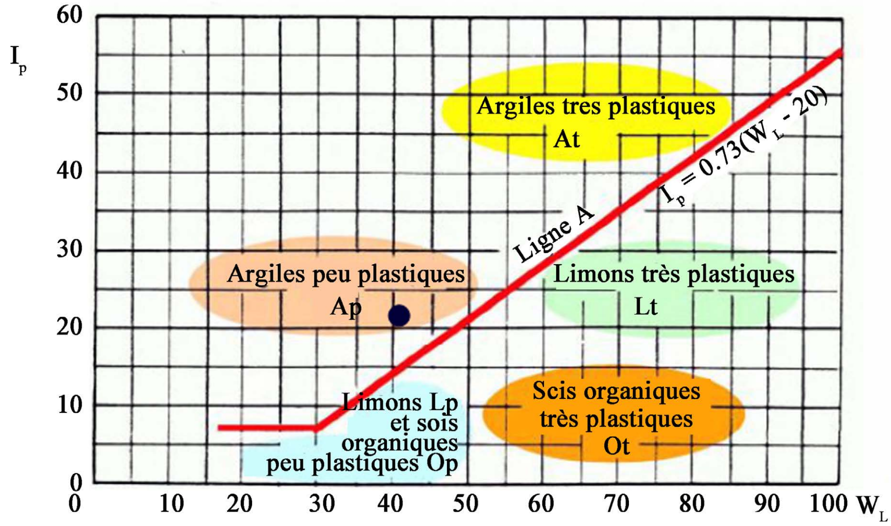

Figure 3. Loud's position in the Casagrande chart.

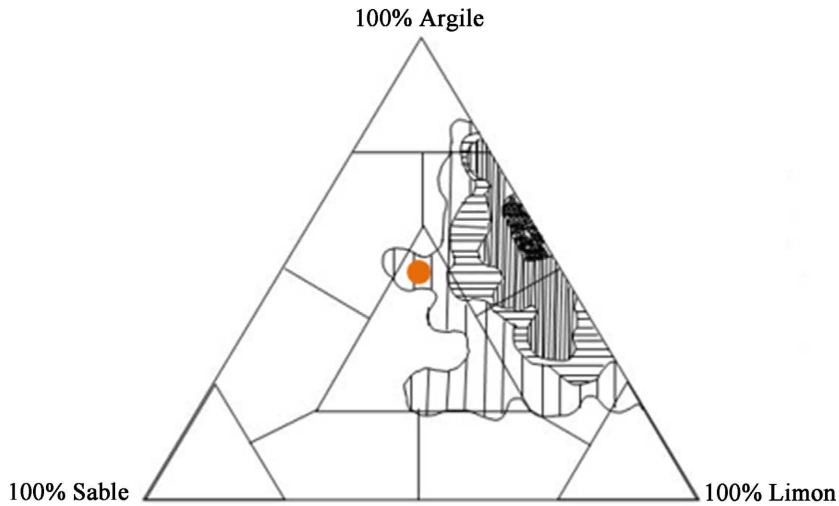

Figure 4. Loud's position in Shepard's triangle.

The Loud sample is soil with low frequency of use; it would have difficulties to adapt to a shape given to it. This low frequency can be attributed to the relatively average amount of fine particles. This tool is used to identify soils potentially useful for agriculture, so this result is in line with Dondi's predictions, which indicate instead a high frequency of use of clay raw materials for the manufacture of 'structural ceramics'.

A possible use for the Loud sample is identified by the Winkler triangle from the results of its particle size distribution. From **Figure 5** we can see that the Loud sample is suitable for terracotta products. In view of its position in this triangle (zone 1), it is estimated that Loud can be used as a base material for the manufacture of thin-walled bricks [14].

Being able to be used well in the making of bricks, the workability map allows us to know whether this clay soil is well adapted to take shape during molding in order to adapt to the shape of the piece to be made. **Figure 6** shows Loud's position

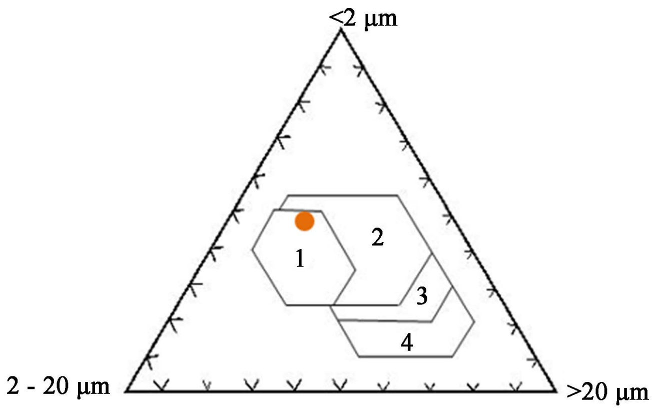

Figure 5. Loud sample position in Winkler diagram.

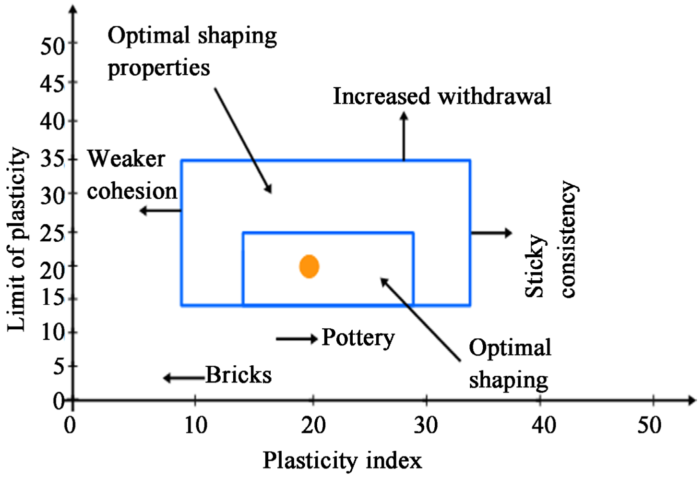

Figure 6. Positioning of the Loudima sample in the Bain and Higlyn workability map.

in the map of workability from the Atterberg boundary results.

It can be seen that the clay soil of Loudima has optimum shaping properties. Its use in brickmaking is also confirmed through this map. The clay soil of Loudima is well adapted to molding, it has a relatively medium sticky consistency and the cohesion of the grains is also relatively medium. The brick made with Loud does not have a significant shrinkage.

3.2. Mineralogical Characteristics

3.2.1. X-Ray Diffraction

The X-ray diffractogram is given in **Figure 7**.

Analysis of this diffractogram identified the following clay mineral species: Kaolinite (7.23 Å, 4.46 Å, 4.17 Å, 3.55 Å, 1.67 Å), quartz (4.25 Å, 3.34 Å, 2.45 Å, 2.28 Å, 2.24 Å, 2.13 Å, 2.13 Å, 1.82 Å, 1.67 Å) and hematite (3.55 Å, 2.49 Å).

The relatively high intensity of the quartz line indicates the significant presence of free silica. The good resolution of the quartz peaks reflects its good crystallinity.

In the X-ray diffractogram of ordered kaolinite, the interval between 19˚and 23˚usually has three distinct peaks, while the increase in disorder causes

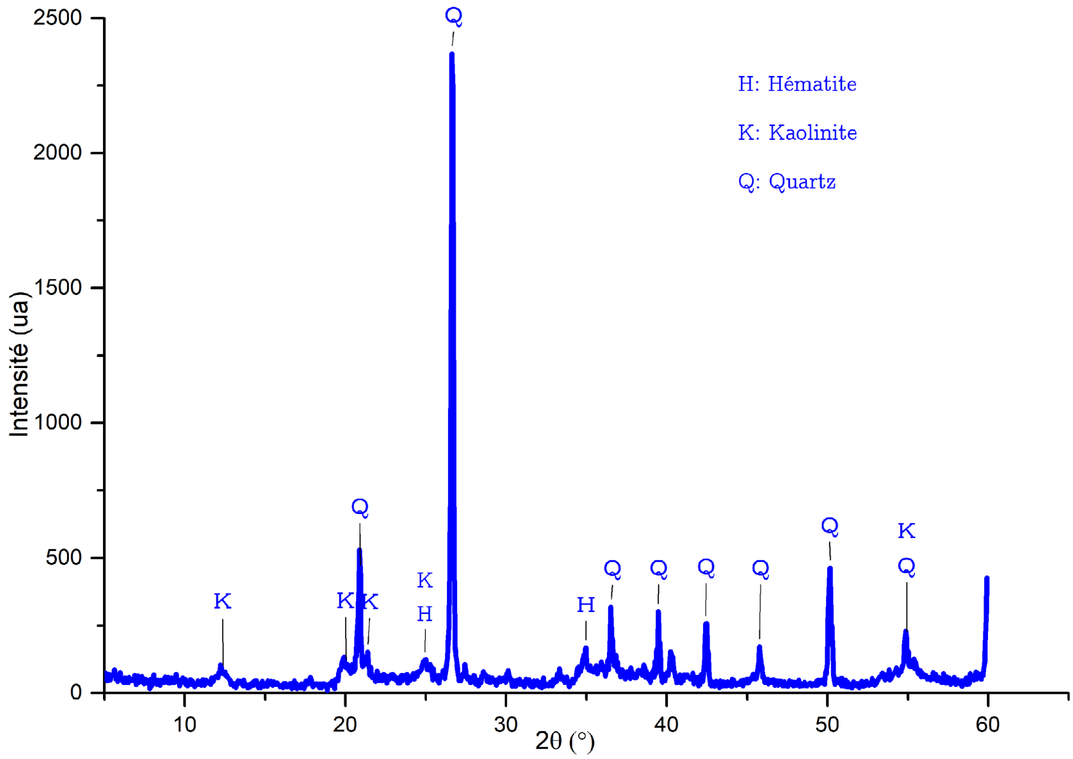

Figure 7. Loud X-ray diffractogram.

the sequence of reflections 02l, 11l in the interval 20˚ - 33˚ to become increasingly blurred until only a smooth diffraction band with the superimposed basal reflection 002 [15]. If we observe a peak in this interval, we can think that Loud's kaolinite is disordered.

Since the Loud sample through the DRX revealed the characteristic peaks of kaolinite associated with quartz and hematite, we can estimate that Loud contains kaolinite as the only clay mineral, which is useful for the manufacture of terracotta products.

3.2.2. Infrared Spectroscopy

Figure 8 shows the infrared (IR) spectroscopy of the Loudima clay soil.

Loud infrared spectroscopy confirms the presence of Kaolinite (whose X-ray diffraction peaks have been identified) in the bands 3692 and 3621 cm^{-1} assigned to OH stretch vibration modes [16] [17] [18].

The IR spectrum of kaolinite crystallizes with four bands linked to the four hydroxyl groups when the latter is crystallized, on the other hand, these bands are reduced to three by a low crystallization [17]. The presence of water is identified by the presence of the band at 3431 cm^{-1}.

The absence of the 3672 cm^{-1} band in our spectrum suggests that our sample has a disordered kaolinite.

The frequency range from 3700 cm^{-1} to 3100 cm^{-1} corresponds to the OH group stretching mode [16].

The presence of quartz in the sample is manifested by bands at 685 cm^{-1} (Si-O

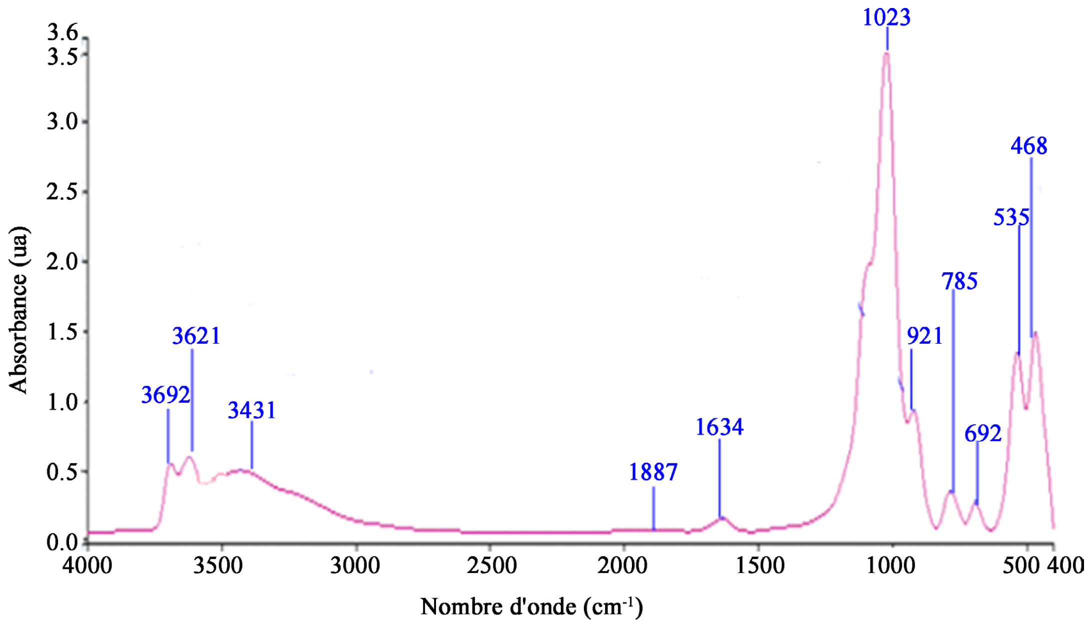

Figure 8. IR spectrum of the Loud sample.

elongation mode) and at 692, 1023 cm^{-1} (Si-O deformation mode).

At 1634 cm^{-1} we observe an absorption band attributed to the H_2O valence deformation vibrations of water.

These results are well in line with what the DRX revealed as species in Loud.

3.3. Chemical and Mineralogical Analysis

3.3.1. Chemical Analysis

Table 2. Chemical Properties of the Loud Sample

Oxide	SiO_2	Al_2O_3	Fe_2O_3	MgO	K_2O	TiO_2	CaO
Mass percentage	35.26	16.34	2.93	2.33	0.95	0.24	0.03

The major oxides are silicon and aluminum. Iron and magnesium are also important. (See **Table 2**)

As a result, the low aluminum oxide content does not give Loud the possibility to synthesize refractory materials. It is a silico-clay material [19].

The SiO_2/Al_2O_3 ratio is equal to 2.15. This high ratio indicates a relatively abundant presence of quartz in this clay soil.

According to Dondi, clays that give colored and clear cooking are distinguished by a content of Fe_2O_3 oxide around 3%. Thus, this assertion is well related to Loud's result.

The content of coloring oxide (Fe_2O_3+ TiO_2) is 3.17%. These levels of coloring oxide indicate that our soil will be used in the manufacture of red bricks [20]. Color is one of the considerable assets in the manufacture of bricks. These coloring oxides also contribute to improving vitrification at low temperatures.

The K_2O and CaO contents associated with kaolinite and illite for the manufacture of brick with the best hardness, the best mechanical resistance to compression and the appropriate porosity are respectively between 0.5% to 4% and 0.5% to 15%.

Low levels of alkali oxides (K_2O and CaO: 0.98) could adversely affect the consolidation process as they provide the fusible material needed for consolidation. The low CaO content (0.03%) would not favor the creation of porosity in Loud by releasing CO_2 during cooking.

Based on the results of the chemical analysis, we can say that the clay soil of Loudima can be used as raw material in the ceramic industry [12].

Compared with the chemical composition of the clay material used in the manufacture of bricks and tiles as presented by Osomba 2012 [12], Loud can be estimated as a clay raw material that can be used in the manufacture of bricks or tiles.

The resulting chemical composition allowed Loud to be placed in Augustinik's diagram, thus presaging his potential use for bricks.

In this diagram, Loud is located in the field of earthenware pottery. This does not give it the possibility to be used for making terracotta bricks. (See **Figure 9**)

Thus, in order to give it this possibility, it is obvious to envisage the addition of an additive having the effect of increasing the quantity of alkali metal and alkaline-earth metal oxides while increasing the quantity of silica in order to reduce the alumina/silica ratio.

3.3.2. Loud Mineral Balance

The quantitative mineral balance is obtained from the following formula [21] [22]:

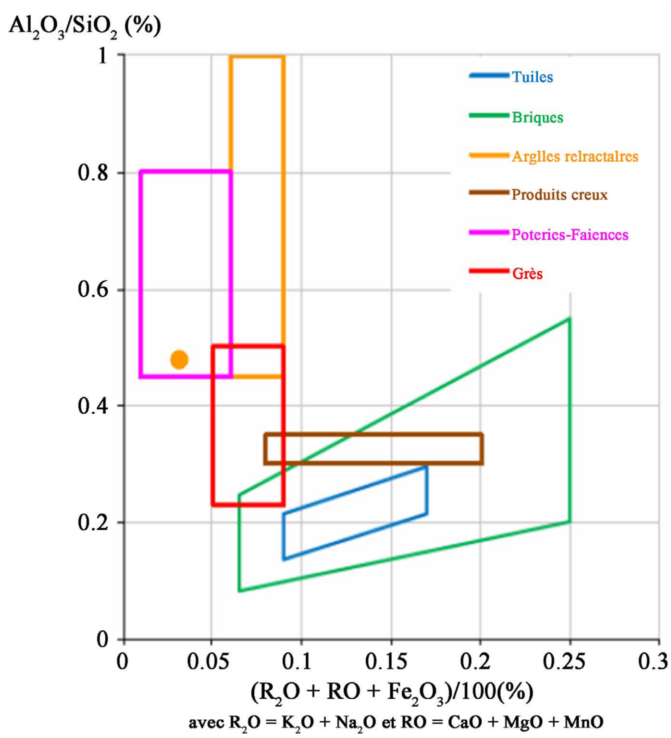

Figure 9. Location of Loud in the Augustinik diagram.

$$T (a) = \Sigma Mi Pa \quad (5)$$

where:

T (a) is the content (% oxide) of chemical element a;

Mi is the (%) mineral content i in the test material containing element a

Pa is the proportion of element a in mineral i (this proportion is deducted from the ideal formula (simplified formula) assigned to mineral i.

Calculations are based on simplified chemical formulae expressed as oxides. Mineral species are inferred from DRX results expressing their diffraction peaks.

For kaolinite, we have $2SiO_2$, Al_2O_3, $2H_2O$;

For quartz: SiO_2

For Hematite: Fe_2O_3

Thus:

$$\text{Kaolinite } \% = (\%Al_2O_3)/102 \times 258 \quad (6)$$

$$\% \text{ Quartz} = \% \text{ } SiO_2 - \% \text{ Kaolinite} \times (60 \times 2)/258 \quad (7)$$

With the molar mass of quartz (SiO_2) 60, the molar mass of Al_2O_3 102, molar mass of kaolinite 258 respectively in $g \cdot mol^{-1}$.

Once all the calculations have been made, the following results appear: 41.33% and 16.03% respectively representing the Kaolinite and quartz content in the sample.

We note that the clay mineral contained in Loud is kaolinite with quartz as the main impurity.

3.4. Scanning Electron Microscopy (SEM)

Scanning microscopy can be used to observe the texture of the clay sample and to characterize and characterize mineralogical assemblages.

The scanning electron microscopy image shows the presence of randomly scattered clay sheets in rolled form. The image obtained by scanning electron microscopy of the clay sample is represented in **Figure 10** below. Clay particles are formed as clusters of aggregates and wafers with irregular outlines.

It is a morphology encountered both for poorly crystallized kaolinites as observed by kanon. Quartz is in the form of small grains [15] [23].

3.5. Propriétés Technologiques

3.5.1 Linear Shrinkage on Firing

From this curve, we see an increase in linear shrinkage as a function of temperature. This can be explained by the reorganization of amorphous matter (meta kaolinite) resulting from the transformation of kaolinite under the effect of temperature.

We find from 850˚C to 1050˚C. that the removal of the briquette has a significant growth. This is consistent with the transition from the amorphous state of kaolinite to mullite. (See **Figure 11**)

From 1050˚C to 1150˚C., we observe a small variation in the shrinkage. The absence of a step would reflect the still incomplete state of consolidation. This

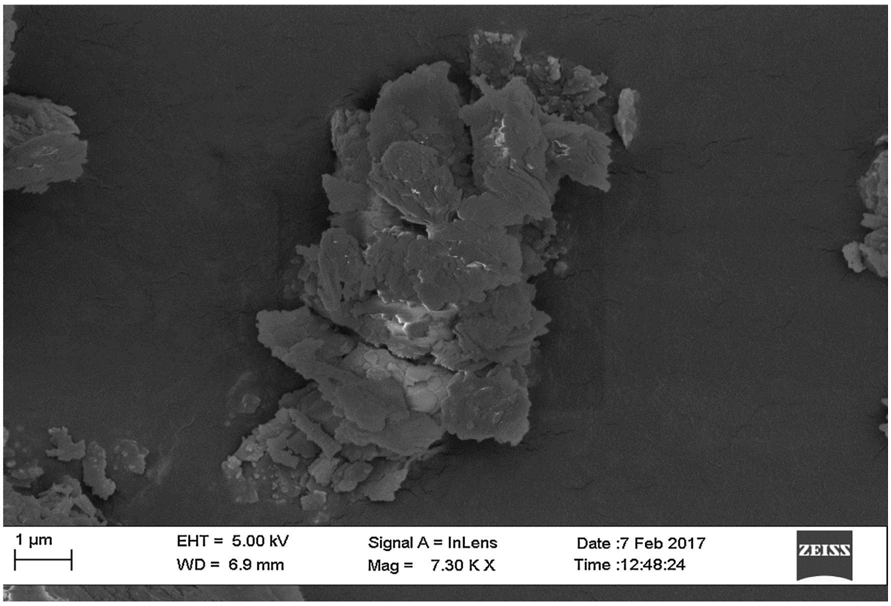

Figure 10. Loud scanning electron microscopy.

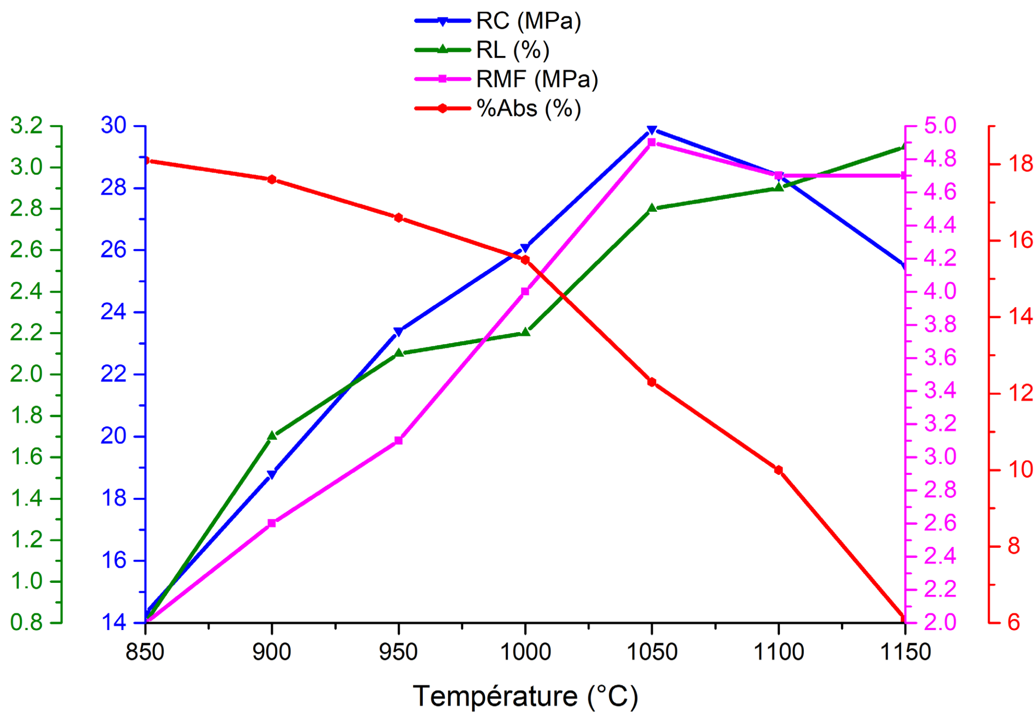

Figure 11. Evolution of the few technological properties.

small variation in shrinkage could well be explained by the presence of quartz, which could already tend to give cristobalite or tridymite, which could well hinder the evolution of shrinkage because of the space it occupies by magnification.

With regard to the withdrawal, Loud would be suitable for the manufacture of terracotta products.

Loud has a cooking shrinkage of less than 8% in accordance with French standard NF P13-301 [24].

3.5.2. Water Absorption Rate

The water absorption rate decreases with increasing cooking temperature. The decrease in water absorption may be related to the gradual disappearance of open porosity [8]. The liquid phase from Fe_2O_3 and K_2O caused by the increase in temperature favors the approximation of the solid particles. This would significantly reduce the rate of water absorption and by deduction the open porosity. At 1050˚C. there is a reduction in the absorption rate to 6% which is less than 15%, which is in accordance with the provisions of standard NF 13-301 [24]. This confirms the idea of using Loud to make bricks.

Low absorption would be one of the defenses of bricks against external aggressive agents.

3.5.3. Mechanical Compression Strength

Mechanical compressive strength increases with temperature.

This compression is progressive from 850˚C to 1050˚C, from this, we see a reduction in this compression up to 1150˚C. The presence of quartz derived from Loud' grain size and mineralogical balance may be responsible for this decrease after the reorganization of meta kaolinite to mullite, on the other hand, the low proportion of illite and oxides such as K_2O and CaO do not promote the consolidation of the brick by reducing the pores. We can therefore estimate that the sintering was not done well. Indeed, at 1100˚C., Baccour *et al.*, [25] and Moutou *et al.* [9] [10] [11] showed during sintering, cristobalite appears (quartz turns into cristobalite). This transformation is accompanied by a change in volume. And at that temperature, they also found that the conversion of quartz into cristobalite lowers mechanical strength.

Moreover, from 850˚C to 1150˚C., although the progression is not constant, it can nevertheless be noted the evolution of this resistance, this is explained by the fact that the molten clay particles during heating touch each other punctually and thus consolidate, conferring a high strength on the bricks because the compression strength of a fired brick can generally range from 10 to 20 MPa.

3.5.4. Mechanical Flexural Strength

Loud's mechanical flexural strength increases with temperature. The increase is gradual from 850˚C to 1050˚C. and then there is a decrease in the RMF.

From its particle size, the presence of sand in Loud is not negligible, which would have

The effect is to reduce the FMR. after any chemical transformations of kaolinite have been made.

This could also be explained with respect to linear shrinkage at the same temperature range.

Compared to current industry standards (RMFs range from 6.9 to 27.6 MPa) Loud does not have a good RMF (Bories, 2015) [26].

The standard NBN EN ISO 10545-3 (1997) [8] sets a minimum of 8 MPa for bricks intended for non-decorative masonry, again it is clear that Loud does not comply with this requirement.

The low values obtained can be justified by the low content of the alkali and alkaline-earth compounds revealed by the chemical composition.

4. Conclusions

This work aims at upgrading the clay soil collected at Loudima for use in ceramics. Geotechnical and mineralogical characterizations were carried out on the one hand and some technological tests were also carried out on the other.

From a geotechnical point of view, Loud's particle size showed the presence of 49% clay combined with 33% silt and 18% sand. Of the Atterberg limits, Loud has a plasticity of 20.7%. This gives it a clay texture and a plastic consistency. Loud has a frequency of use with optimal shaping properties whose utility has a tendency for baked bricks.

The fine particles identified in the geotechnical tests are confirmed by DRX and infrared, which show the presence of kaolinite at 41.33% as the dominant species. This is consistent with the results of the chemical analysis, which gives silica and alumina as the main oxides.

The morphology obtained by MEB shows that the kaolinite contained in Loud is poorly crystallized and that the quartz associated therewith is in the form of small grains.

From a technological point of view, Loud has a linear shrinkage at firing, the mechanical strengths in compression and bending increase throughout the rise in temperature. The water absorption rate decreases with temperature. It emerges from this that consolidation and densification do not exhibit a level of satisfaction because of the non-constancy of these properties. However, it should be noted that the Loud sample has the potential for making terracotta bricks with the possibility of improving these properties and these technological properties. It would also be obvious to assess thermal conductivity in order to assess the level of thermal comfort.

Finally, the problems justifying the poor quality of the bricks produced in Loudima are linked to the chemical and mineralogical composition of this clay soil. In fact, the levels of sand, alkaline and alkaline-earth oxides and also iron oxides are not in the quantity necessary to produce a fired brick with better compressive and bending strengths. The same is true for the water absorption capacity, which is not the capacity that would give these bricks the desired thermal comfort. This justifies the choice of making formulations with an additive to evaluate its contribution through geotechnical, mineralogical and geotechnical studies.

Conflicts of Interest

The authors declare no conflicts of interest.

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