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Characterization and Valuation of a Clay Soil Sampled in Londéla-Kayes in the Republic of Congo

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Abstract

In order to characterize and enhance clay collected in Londéla-kayes in the Republic of Congo, in this work, it was a question of proceeding to the mineralogical, physico-chemical, thermal and geotechnical characterization of this clay. Next, determine the technological properties of fired bricks. For this, various methods were used in particular: X-ray diffraction, infrared spectroscopy, gravimetric thermal analysis and differential thermal analysis, dilatometric analysis, scanning electron microscopy, specific surface and analysis chemical. It appears that, for the mineralogical characterization, kaolinite is the most abundant mineral of this clay. The results of dilatometric analysis have shown that this clay can be fired at low temperatures. The geotechnical characterization showed that it is plastic clay thus exhibiting a high shrinkage. The results of the technological properties of LON1 bricks have shown that this clay cannot be used in the manufacture of fired bricks. The geotechnical properties must be improved by adding additives in order to improve the technological properties of the fired bricks.

Keywords

Clay, Characterization, Valuation

1. Introduction

Clay refers to any natural material, composed of fine grains, which turns plastic in the presence of suitable water contents and hardens by drying or heating [1]. The latter is mainly used in the ceramic and pharmaceutical industries, in road construction, in economic housing and in the chemical industry [2]. In the chemical industry, they initiate certain reactions (cracking of mineral oils or polymerization of certain organic molecules) [3]. The structural determination of a clay, directs towards the use of it. In recent years, our laboratory, which favors applied research, has been interested in some applications of clay raw materials, in particular the field of ceramics and, above all, has worked to determine for many clayey sites in Congo Brazzaville the physico-chemical, thermal and mineralogical compositions as well as geotechnical properties [4] [5] [6] [7] [8]. In Londéla-kayes, clay soils are mainly used by rural populations for the manufacture of fired bricks. The fired brick manufacturing units are functional, but the products obtained are not of good quality. This could be due to the lack of mastery of the technology as well as the quality of the clays used. The resulting bricks break and do not withstand heavy rains. In order to support rural populations in this sector, our work consists of characterizing these clays, determining the technological properties of fired bricks in order to better understand their use. By characterizing and determining the technological properties of fired bricks from the clay sample at the study site, using modern techniques and equipment, this research may provide answers to certain questions of fundamental interest.

2. Material and Method

2.1. Location and Site of Clay Sampling

The sample was taken in Kayes Bakou in the village of Londéla-kayes district located in the southwest of the Republic of Congo near the border between the Republic of Congo and the Democratic Republic of Congo. Sampling took place during the dry season. The sample taken from this site was named LON1. The GPS coordinates of the sampling site are presented in **Table 1**.

The GPS coordinates of the study area allowed us to locate the LON1 sample collection site in **Figure 1**.

2.2. Experimental Study

The sample was dried at room temperature, crushed and then sieved at 2 mm. The sieve obtained was subjected to a series of physico-chemical and mineralogical analyzes.

2.2.1. X-Ray Diffraction

It was carried out in Abidjan in Ivory Coast at the crystallography laboratory. This analysis allowed us to know the mineralogical of LON1.

Table 1. GPS coordinates of the sampling.

Sample	Latitude	Longitude
LON1	04˚44'44.3"S	013˚31'01.1"E

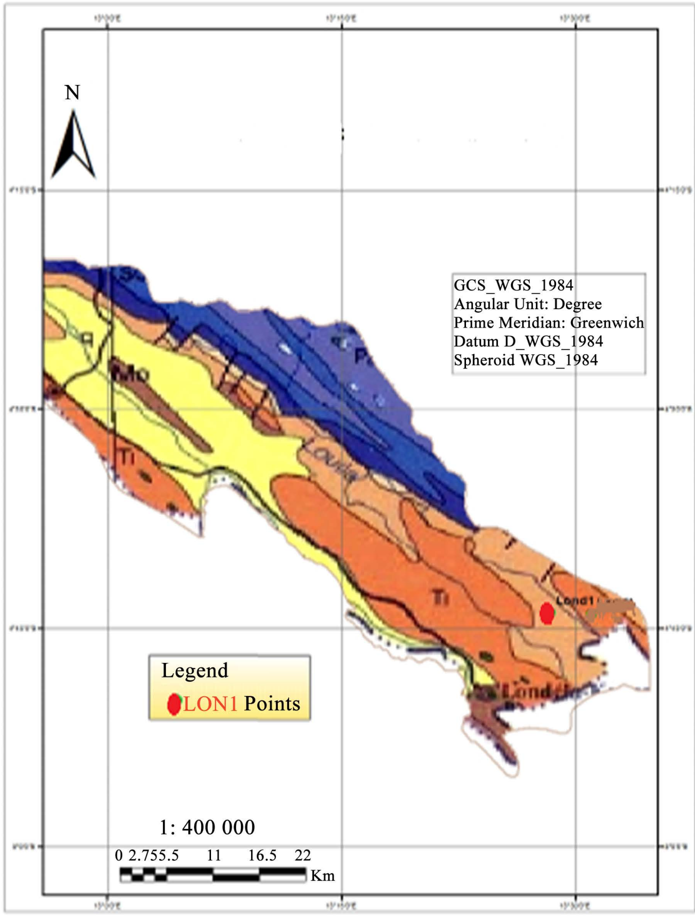

Figure 1. Location of LON1 sampling sites.

2.2.2. Infrared Spectroscopy

It was carried out in Cameroon at the Faculty of Science of the University of Yaoundé 1 in the Laboratory of Applied Analytical Chemistry. This technique was used for the determination of the functional groups present in LON1. Each link has characteristic vibrations that allow the types of links to be identified.

2.2.3. Gravimetric Thermal Analysis and Differential

Thermal analysis they were carried out in France at the University of Limoge at the Technical Center of ceramic Transfer. These analyzes were made in order to know the behavior of LON1 according to the temperature range going from 0˚C to 1200˚C.

2.2.4. Dilatometric Analysis

It was carried out in France at the University of Limoge at the Technical Center of Ceramic Transfer. It consists of monitoring the sintering ability of LON1 during a thermal cycle over the temperature range from 0˚C to 1400˚C.

2.2.5. Scanning Electron Microscopy

It was performed in China at Fudan University Shanghai in the Physics Applied to Materials Laboratory. This technique allows us to observe the surface morphology of LON1, then to determine the quality and quantity of the chemical elements present in this soil.

2.2.6. Chemical Analysis

The chemical analysis of the major elements was carried out at the Petrographic and Geological Research Center (PGRC) of Nancy in France by the method described by Carignan *et al.* [9].

2.2.7. Particle Size Analysis

It was carried out in Congo Brazzaville in the laboratory of the Building and Public Works Control Offices in the acronym BPWCO. This analysis was carried out to determine the distribution of particles in LON1 by following the recommendations of standards NF P 94-056 and NF P 94-057 [10].

2.2.8. Atterberg Limits

They were also carried out in Congo at the Building and Works Control Office in the acronym BWCO. This analysis was carried out to determine the liquidity limits (LL) and the plasticity limits (LP) of LON1 by following the recommendations of standard NF P 94-051 [11].

2.2.9. Specific Surface

It was carried out in France at the University of Limoge at the Technical Center of ceramic Transfer (TCC.T) by the BET method. This analysis was carried out to determine the total surface area available in LON1 according to an internal protocol inspired by standard NFISO 9277 [12].

2.3. Technological Properties

They were carried out in Congo at the Office for the Control of Buildings and Public Works. The technological properties of LON1 were determined on the test tube according to two steps:

- Production of the test tube: The clay soil samples were dried at 105˚C in an oven. Grinding and sieving with a 34 modulus (2 mm) sieve is applied. After weighing a given mass, briquettes are shaped with a mold measuring 4 cm × 4 cm × 16 cm. The smallbrickwas air dried for five days.
- Baking: the specimens were baked in an oven at different temperatures (850˚C, 900˚C, 950˚C, 1000˚C, 1050˚C, 1100˚C and 1150˚C).

2.3.1. Linear Shrinkage during Cooking

The determination of the shrinkage percentages during banking was made by studying the variation in the average length of the strokes recorded on the briquettes between drying and firing. It was determined by the following formula:

$$R(\%) = \frac{L_0 - L}{L_0} \times 100 \tag{1}$$

With:

L_0: Drying length.

L: Banking length.

R: linear shrinkage during banking.

2.3.2. Water Absorption

Water absorption was determined by immersing a baked brick specimen in water for 24 hours. It was determined by the following relation:

$$\text{Abs}(\%) = \frac{m_h - m_s}{m_s} \times 100 \quad (2)$$

With:

m_h: The wet mass of the test specimen after immersion.

m_s: The dry mass of the test tube.

Abs: Water absorption.

2.3.3. Mechanical Resistance

The determination of the mechanical resistance to bending and compression of fired bricks was carried out with BPWCO according to European standards NBN EN 196-1 and EN 772-1 respectively [13].

3. Results and Discussion

3.1. Diffractogram of the Crude Sample of LON1

Figure 2 illustrates the diffractogram of the raw sample of LON1.

Analysis of this diffractogram allowed us to identify reflections characteristic of phyllosilicates. The inter-reticular distance corresponding to d = 7.27 Å is characteristic of kaolinite with its harmonics at 4.27 Å; 2.38 Å [14]. The presence of kaolinite in LON1 can be explained by the climate encountered in the Republic of Congo, which is predominantly humid tropical favoring very extensive weathering of rocks. In fact, the formation of clays is linked to the physicochemical and biological alteration that affects the bedrock. In Congo Brazzaville, the clays are mainly made up of kaolinite [5] [6] [8]. The inter-reticular distance corresponding to d = 10.30 Å is characteristic of illite [14]. The octahedral layer in illite is made up of Iron or Magnesium; that of LON1 could contain iron

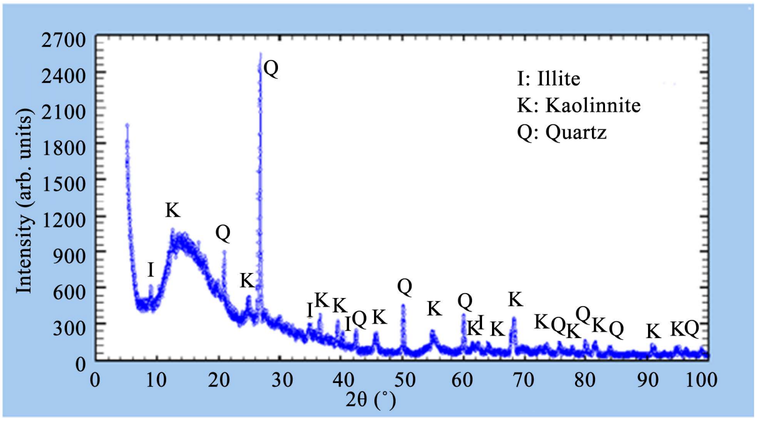

Figure 2. Diffractogram of the raw sample of LON1.

because the naked eye observation of LON1 shows a red coloration. As an associated mineral, characteristic reflections detected at 3.59 Å; 3.34 Å; 2.14 Å; 1.81 Å; 1.26 Å; 1.20 Å; 1.15 Å and 1.03 Å correspond to the peaks of quartz. Quartz is the most common mineral found in clay soils.

3.2. IR Spectrum of Raw Sample LON1

Figure 3 illustrates the infrared spectrum of LON1.

Analysis of this spectrum allowed us to identify the absorption bands located around 3694.11 cm^{-1}, 3621.33 cm^{-1} and 911.63 cm^{-1}. These bands are attributable to the hydroxyl group OH characterizing the presence of dioctahedral minerals, in particular Kaolinite [15]. This is in agreement with the results of XRD which revealed the presence of kaolinite. On the other hand, the absorption bands located around 1026.24 cm^{-1}, 100.24 cm^{-1}, correspond to the elongation vibrations of Si-O also characterizing the presence of Kaolinite [15]. The band observed around 795.89 cm^{-1} may correspond to the different modes of vibration of the Si-O-Fe bond characteristic of illite [15]. The absorption bands located around 778.70 cm^{-1}, 692.54 cm^{-1}, 776.96 cm^{-1}, 691.78 cm^{-1} may correspond to quartz [16]. All peaks between 600 and 400 cm^{-1} correspond to angular deformations of Si-O-M bonds (M = Al, Mg, Fe, Li) [14].

3.3. Differential and Thermogravimetric Analysis

Figure 4 gives the ATG/ATD curves for LON1 clay.

Analysis of these results shows that the ATG curve shows an overall weight loss at 1200˚C of 7.7% which mainly breaks down into 3 steps:

- Ambient at 150˚C characteristic of an endothermic phenomenon with a maximum at 70˚C: It would result from the departure of residual humidity (loss of mass of 1.5%), therefore from the desorption of the physically adsorbed water. The results of the ATG/ATD of the clay soil of LOUTETE show us a mass loss of 1.16% with a maximum at 130˚C [8]. In view of the results of LON1, the increase in mass loss may be due to the presence of illite which is a 2:1 clay species reported by the XRD

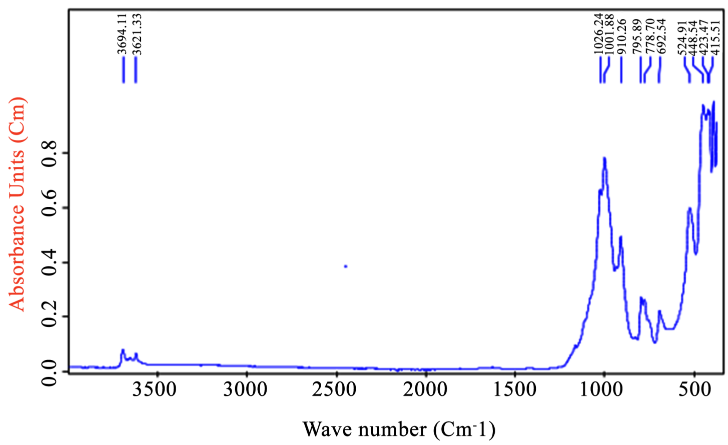

Figure 3. Infrared spectrum of LON1.

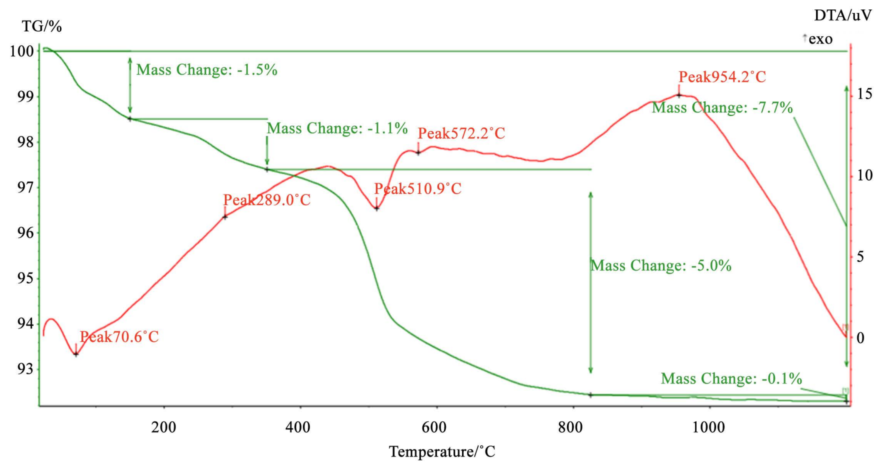

Figure 4. ATG (green)/ATD (red) curves of LON1.

- Between 150˚C and 350˚C: a very weakly exothermic behavior is observed with a mass loss of 1.1% which may be due to the probable decomposition of weak organic residues, the shoulder at around 289˚C would correspond to the expulsion of water bound to exchangeable stocks in illite.
- Between 350˚C and 825˚C: there is an endothermic phenomenon with characteristic peaks at 511˚C and 572˚C which can be explained respectively by the dehydroxylation of 1:1 clays and therefore of kaolinite as indicated by the XRD with a loss of mass of 5.0% deduced by the ATG curve [17] and by the transformation of quartz (passage from the α phase to β).

Above 825˚C, the ATG curve shows a low loss in mass (0.1%). The ATD curve for its part reveals an exothermic peak around 954˚C, characteristic of the structural reorganization of metakaolinite [14].

3.4. Dilatometric Analysis

Figure 5 shows the dilatometric curve recorded for LON1.

We observe on this curve, a weak expansion of a few tenths of a percentage at most below 450˚C with a rate of 0.05%. The dropout with sudden expansion again by a few tenths of a percentage around 555˚C reflects the transformation of quartz (passage from α phase to β phase) as we have just observed in ATD; the reversibility of this phenomenon on cooling, however, not being recorded as shown in the figure. The withdrawal from 800˚C and 850˚C shows a first setback after 900˚C and mainly after 1000˚C. This phenomenon is therefore recorded at low temperatures, thus exhibiting a markedly greater final shrinkage with a rate of 14.4. The shrinkage between 930˚C and 1000˚C is related to the structural reorganization of metakaolinite [14], this is generally associated with an increase in the density of the skeleton without elimination of porosity. A large shrinkage is observed after 1000˚C, due to the densification corresponding to the sintering of LON1. The rapid sintering observed suggests the formation of a significant amount of viscous flux. This flow would then be characterized by a viscosity low enough to lead to creep under the effect of pressure gradients generated by

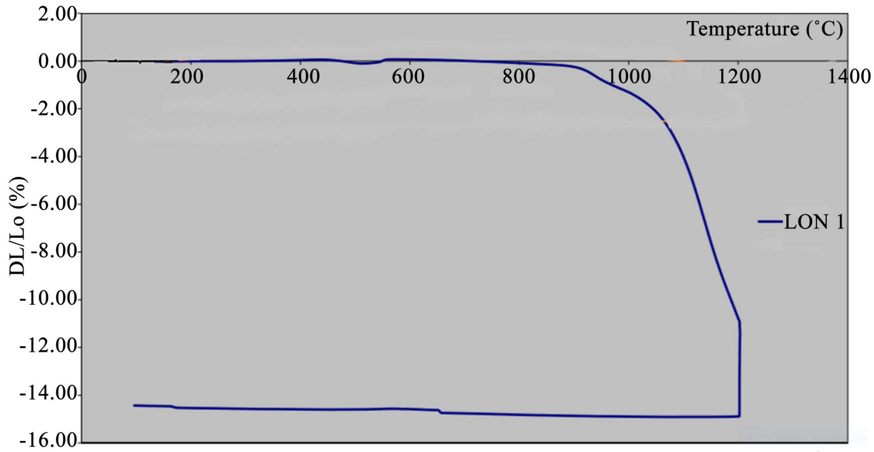

Figure 5. LON1 dilatometric analysis curve.

surface tensions and curvatures between the grains [18]. The dilatometric curve shows that this clay cannot be fired at low temperature around 930˚C.

3.5. Chemical Analysis

The results obtained are shown in Table 2.

The Al_2O_3/SiO_2 ratio of LON1 is 0.30 provides information on the material's permeability to moisture. The greater this ratio, the greater the permeability does not change [19]. This value obtained shows that LON1 is very permeable clay. This permeability can be justified by the presence of illite identified in XRD. The iron oxides present, in large quantities (6.93%) can cause staining of ceramic products [14]. XRD did not reveal the presence of a characteristic iron peak, which suggests that the iron in LON1 is found in the structure of clay species. The large quantity of iron oxides shows that this clay can be used as energetic fluxes which in addition make the materials conductive of heat, resulting in a decrease in the firing temperature [19]. The percentage sum of oxides, sodium and potassium in LON1 is 2.42; accepted value for the use of ceramic clays as a flux [14].

The percentage of titanium oxides in LON1 (1.16%) is high, the peak of anatase not observed in the X-ray spectrum may be masked by the peak of kaolinite.

3.6. SEM and EDS Spectra of Raw LON1 Samples

Figure 6 and Figure 7 show the images obtained by scanning electron microscopy coupled to the EDX of LON1.

The observation of Figure 6 shows that the clay particles are in the form of platelets in sheets which is in agreement with the results of the XRD which revealed the presence of two types of phyllosilicates. We distinguish the classical flattened accordion shape of kaolinite as observed by Kanon for poorly crystallized kaolinite [20]. The observation of white color aggregate confirms the presence of quartz identified in IR spectrum and in DRX of LON1.

Figure 7 gives the EDS results of LON1. Chemical elements such as: Silicon (Si), Aluminum (Al), Iron (Fe), potassium (K), magnesium (Mg), phosphorus

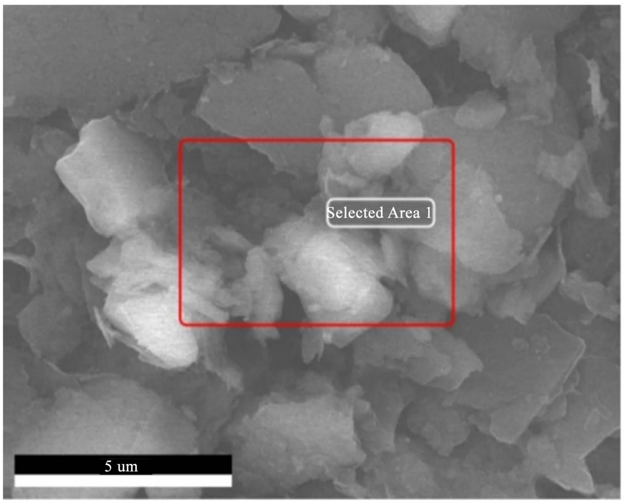

Figure 6. Observation of LON1 with SEM.

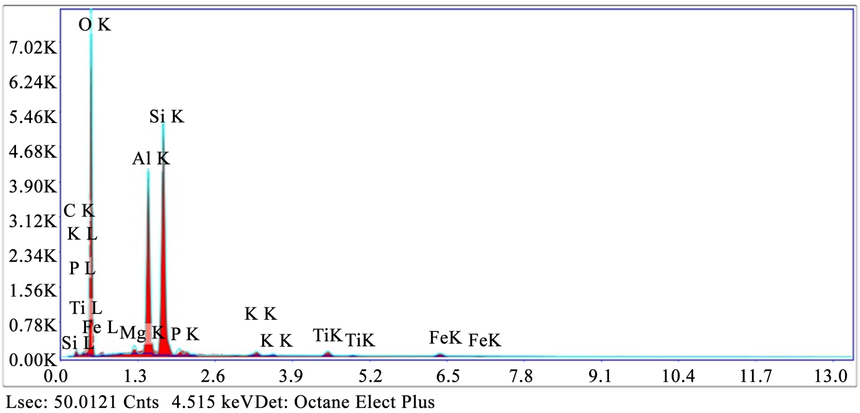

Figure 7. EDS spectrum of LON1.

Table 2. Chemical composition of LON1.

Sample	SiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O	PF	Total
LON1	60.97	18.53	6.93	<LD	0.90	<LD	0.03	2.39	1.16	<LD	8.87	99.88

(P), carbon (C), titanium (Ti) and oxygen (O) are present, which is in agreement with the results of the chemical analysis. The strong presence of Oxygen (O) and Potassium (K) may be due to the presence of illite in LON1. The presence of carbon is certainly due to organic matter.

3.7. Particle Size Analysis

Figure 8 illustrates the particle size curve of LON1.

The particle size curve thus obtained allowed us to obtain the distribution of particles shown in Table 3.

In view of the results obtained, we can say that LON1 is a soil very rich in silt. The results of the particle size analysis allowed us to position LON1 in the Texture triangle (Figure 9).

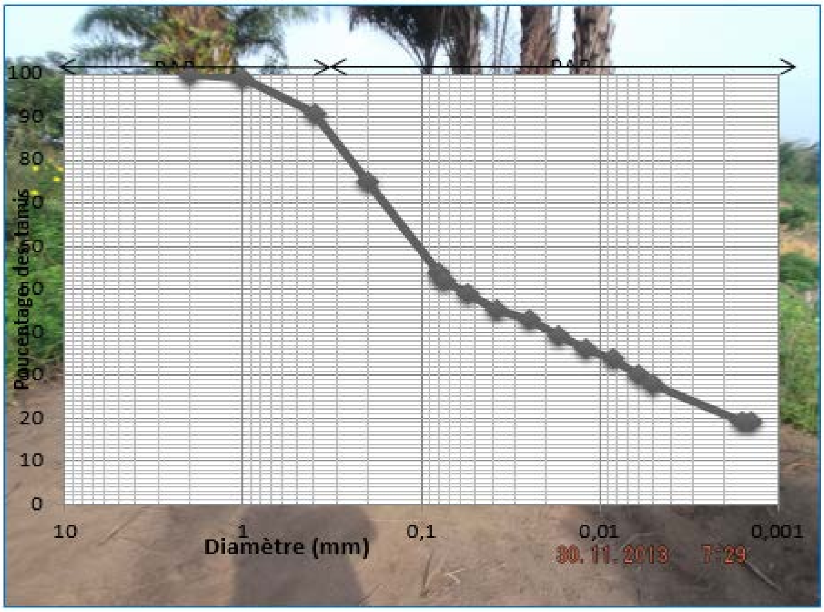

Figure 8. Particle size curve of LON1.

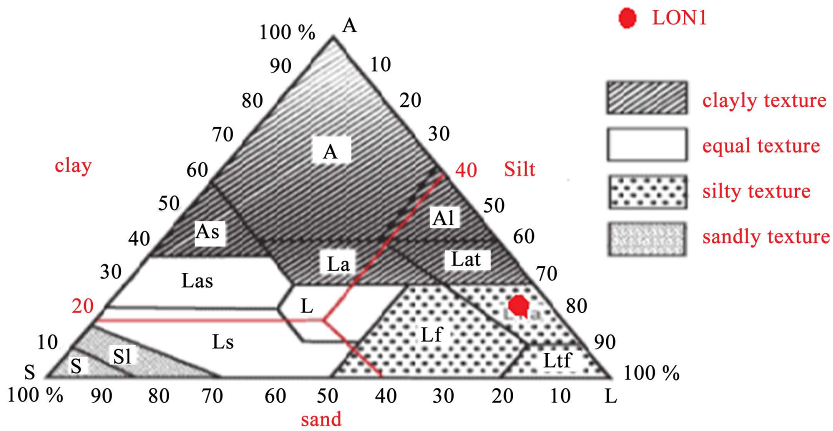

Figure 9. Positioning of LON1 in the Texture triangle.

Table 3. Particle size analysis of LON1.

Particuls / Sample	Clay < 2 µm	Silt 2 - 50 µm	Fines sands 50 - 200 µm
LON1	25%	70%	5%

Observation of this triangle shows that LON1 is found in the area of a soil with a silty texture. By positioning LON1 in the Winkler triangle (**Figure 10**).

LON1 is not found in any brick manufacturing area. By also positioning LON1 in the Shepard triangle (**Figure 11**).

We find that LON1 is in the low frequency area.

3.8. ATTERBERG Limits

Table 4 gives the results of the ATTERBERG limits of LON1.

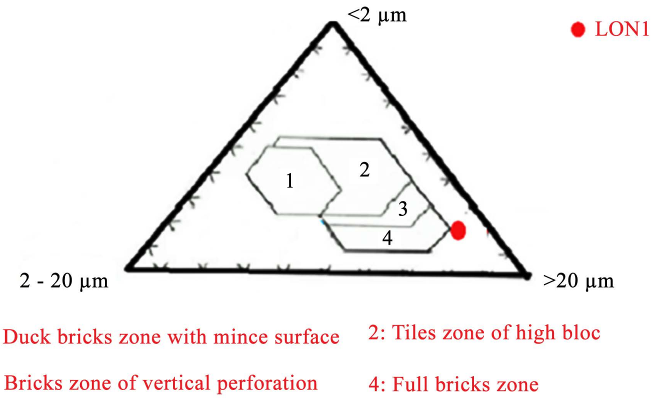

Figure 10. Positioning of LON1 in the Winkler diagram.

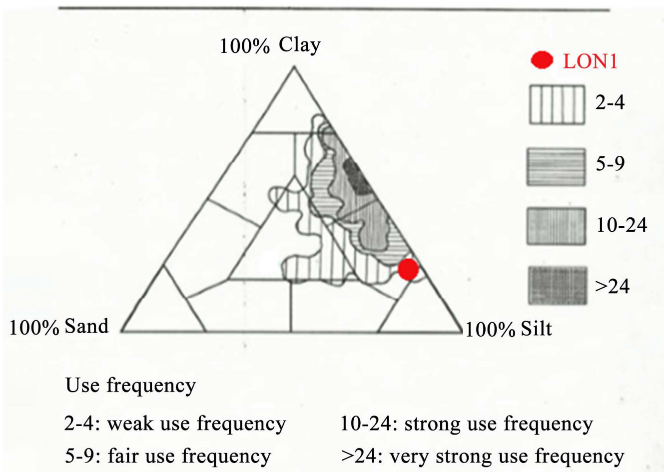

Figure 11. Positioning of LON1 in the Shepard diagram.

Table 4. ATTERBERG limits of LON1 Limits.

Limits / Samples	liquidity Limit	Plasticity Limit	liquidity Indice
LON1	**41.5%**	**26.6%**	**17.9%**

The results of ATTERBERG Limits of LON1 allowed us, while using the Casagrande diagram (**Figure 12**), to give the classification of LON1.

LON1 is found in the zone corresponding to the section of moderately compressible inorganic silts and organic silt. The moderately compressible inorganic clays section of LON1 can be explained by its high percentage of silt, since its mineralogical composition consists of illite, which has in its structure the presence of alkalis and alkaline earths. The results of ATTERBERG Limits also allowed us, using the fecundability map (**Figure 13**), to predict molding properties and soil shrinkage based on criteria related to soil plasticity.

3.9. Specific Surface

The results are reported in **Table 5**.

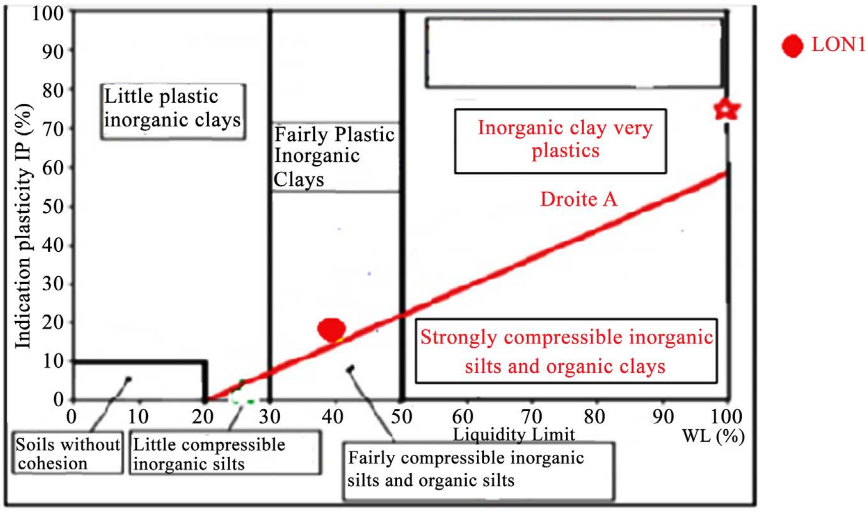

Figure 12. Positioning of LON1 in the casagrande baque.

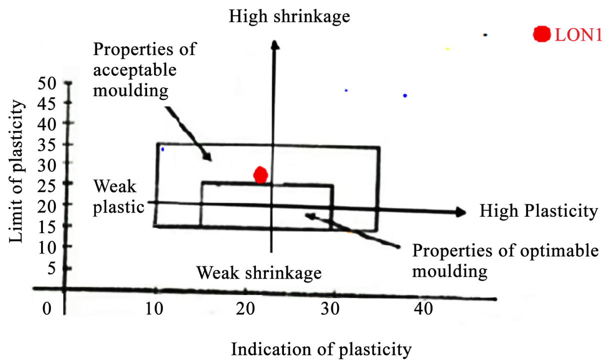

Figure 13. Positioning of LON1 in the card.

Table 5. BET specific surface area of LON1 and LON2.

Sample	BET Specific surface (m^2/g)
LON1	15.5

The value of the specific surface area thus obtained is characteristic of a kaolinite [21]. Kaolinite therefore represents the most abundant mineral in LON1.

3.10. Linear Shrinkage during Firing of Fired Bricks

Figure 14 represents the linear shrinkage of the test pieces obtained.

The results obtained show that the shrinkage increases as a function of the cooking temperature. The high shrinkage of LON1 can be explained by the large amount of alkali and alkaline earth oxides and iron oxides, leading to an increase in the liquid phase during sintering. The significant amount of viscous flux would be characterized by creep under the effect of pressure gradients generated by surface tensions and curvatures between grains causing greater densification generally involving greater linear firing shrinkage[21]. This high retention is in agreement with the result obtained from dilatometry. LON1 alone cannot be

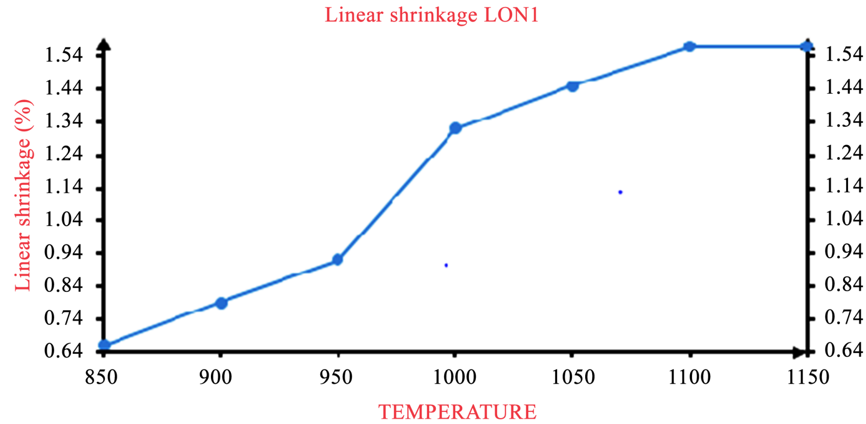

Figure 14. Linear shrinkage curve during firing of LON1.

used in the manufacture of fired bricks, the addition of additives or other clays will be necessary to reduce its shrinkage [22] [23].

3.11. Water Absorption Rate

Figure 15 shows the absorption rate of the test pieces obtained after firing LON1.

Analysis of the results obtained shows that the absorption rate of LON1 decreases with increasing temperature. According to Dondi, the rate of fusible material does not provide sufficient flux to improve densification and reduce pores [24]. This result is in agreement with the high silt and clay contents revealed by the particle size analysis. The loamy clayier the soil, the more water it takes to make it plastic. Using less water would reduce the friction between the grains less and also reduce the pores in the bricks less. Blanchard and his collaborators mentioned in their research work on silicate ceramics that the open porosity after firing of terracotta (briquettes, tiles, tiles, etc.) between 950˚C and 1150˚C should be between 10% and 25% [14]. The sample studied has values greater than 25%. This clay cannot be used in the manufacture of fired bricks. The addition of an adjuvant would be important to improve the absorption values of LON1.

3.12. Mechanical Resistance to Bending

Figure 16 represents the values of Mechanical resistance to bending.

The results obtained show that the mechanical flexural strength increases up to a temperature of 1000˚C and then decreases at a temperature of 1150˚C. The increase in the values of mechanical strength can be explained by the presence of sodium and potassium oxides in LON1 which provide fusible material, thus causing the consolidation of the grains of sand. The decrease in mechanical flexural strength can be explained by a more homogeneous distribution of the porous network within the clay matrix and in particular the formation of a new crystalline phase [21]. The mechanical resistance values obtained compared to

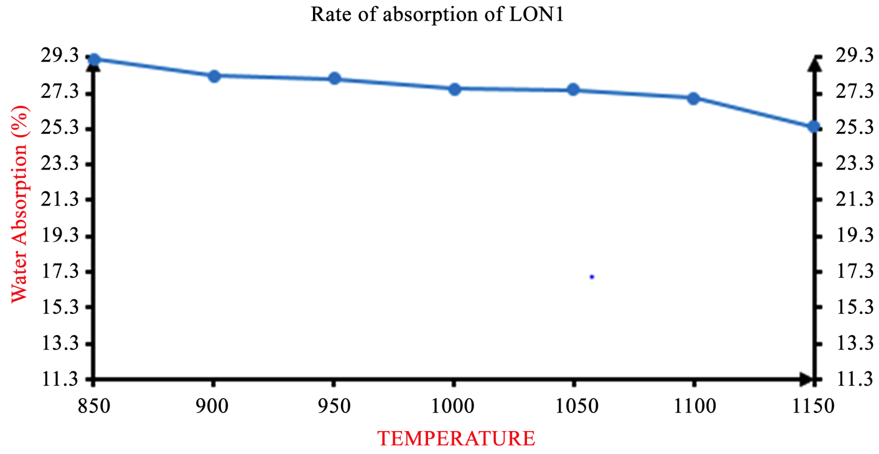

Figure 15. LON1 absorption rate curve.

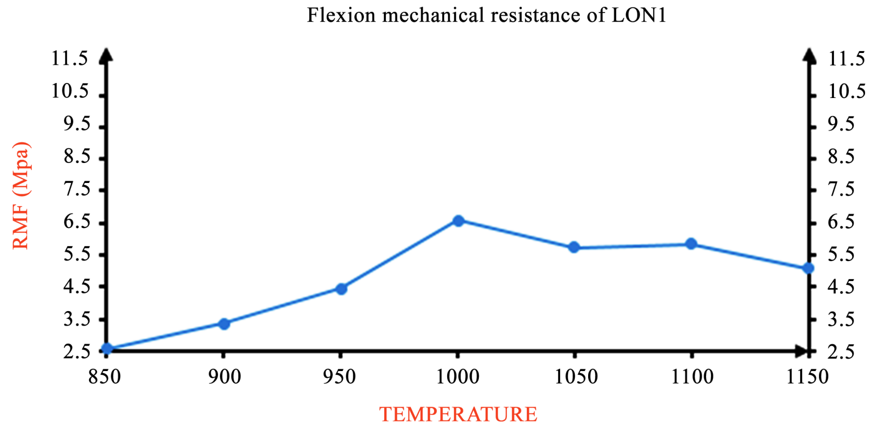

Figure 16. Mechanical flexural resistance curve of LON1.

European standards are low. The low values can be explained by the particle size composition. In fact, silts, the majority fractions in LON1, have almost no cohesion between the particles. On the other hand, the fractions clays less represented in LON1 than the silts, make up the finest fraction of soils (less than two microns) do not have the same characteristics as silts and sand. Each clay particle is surrounded by a film of water that is absorbed very strongly. This gives the clay its cohesion and most of its mechanical strength. The clay therefore gives the finished products its cohesion and acts as a bond between the coarser elements that make up the skeleton [14].

3.13. Mechanical Resistance to Compression

Figure 17 shows the mechanical resistance to compression.

The curve obtained shows that between 850˚C and 1050˚C. There is an increasing function with the cooking temperature and between 1050˚C and 1150˚C. a decreasing function with the cooking temperature. The increase in this parameter is more pronounced from the cooking temperature of 1050˚C. For this material, the temperature of 1050˚C represents this limit of mechanical

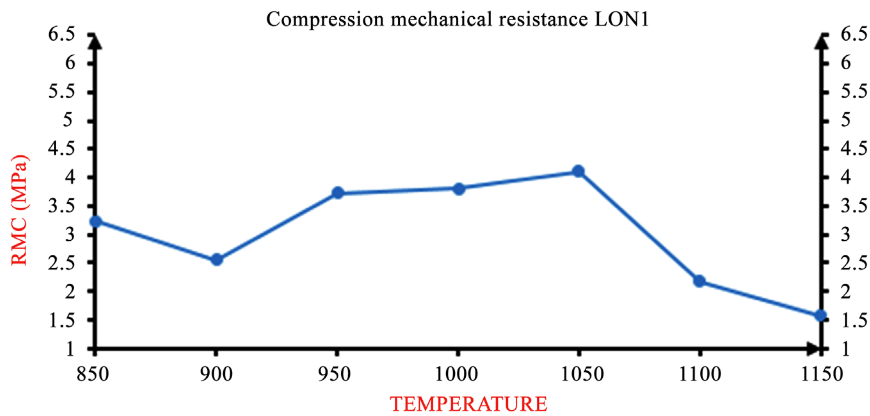

Figure 17. Curve of mechanical resistance to compression of LON1.

resistance to compression. The strong decrease in the mechanical resistance in compression observed from the firing temperature of 1050˚C makes it possible to formulate the hypothesis of the distribution and the nature of the main crystalline phases present in the skeleton of the fired material [14]. In general, the values of mechanical resistance to compression are low in LON1. This can be explained by higher values of absorption rate as a result of porosity.

4. Conclusion

This work had for general objective, the characterization and the valorization of clay taken in Londéla-kayes. Its specific objectives were to carry out a mineralogical characterization and to study the technological properties in order to provide the necessary answers linked to the breakage of bricks made by rural populations. To carry out this study, we used as characterization methods: XRD, IR, ATD/ATG, SEM/EDS, specific surface area by BET method, chemical analysis and geotechnical analyzes. We evaluated the technological properties by linear shrinkage, absorption rate, mechanical resistance to bending and compression. The results of characterization revealed that this soil exhibits kaolinite and illite as clay minerals. This clay can be fired at a low temperature around 930˚C. Analysis of different ceramic tools has shown that this soil has a low frequency to be used in ceramic. The addition of adjuvants would therefore be necessary to improve these geotechnical properties. The results of technological properties compared to standards have shown that this clay cannot be used in the manufacture of fired bricks. The high shrinkage observed in this clay would therefore be the cause causing the breakage of the fired bricks. We plan to continue this study by adding an adjuvant to the Londela-kayes clay to correct this shrinkage in order to obtain products that meet the requirements of the fired brick manufacturing standards.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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