

## Characterization of a clay used in the terracotta sector in the locality of Dongou

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### Abstract

The overall objective of this study is to characterize the clay soil used in the terracotta sector in the locality of Dongou.

The soil texture of Dongou was determined by its particle size distribution (wet sieving). The Dongou clay was characterized by its plasticity (Atterberg limits), chemical composition (XRF), and mineralogical composition (XRF). Shrinkage before and after firing was measured. Water absorption rates and three-point bending strength were determined. Potential uses were studied.

A varied grain size (Clay: 62%, Silt: 36% and Sand: 2%) giving the soil of Dongou a silty-clay texture, a mineralogy composed of Kaolinite as the predominant mineral species, quartz and other associated minerals, a chemical composition predominantly of: SiO<sub>2</sub> (49.92%), Al<sub>2</sub>O<sub>3</sub> (28.27%), Fe<sub>2</sub>O<sub>3</sub> (2.93%), CaO (0.13%), MgO (1.04%) with 11.1 as total loss on ignition, a plasticity index of 40% giving the clay of Dongou a very plastic nature with optimal molding, a linear shrinkage between 2 and 5%, an absorption rate between 19 and 5% beyond 1050°C, very diverse mineral transformations during firing.

The clay soil of Dongou is to be used in the manufacture of lightweight block tiles.

**Keywords:** Clay; Terracotta; Properties; Characterization; Manufacturing; Dongou.

### 1. Introduction

Humans have used clay for centuries. Japan and China can be cited as key countries in the discovery of art objects and pottery between 8000 and 9000 BC [1]. Clay, increasingly known to modern society thanks to the various technological applications of clay materials in industry, is used in the manufacture of cement (heat-resistant cement, geopolymer cement, etc.), paints, ceramics (pottery, art objects, bone, teeth, water filters, porcelain, bidets, etc.), building materials (floor and wall tiles, terracotta bricks, roof tiles, etc.), paper (absorbent paper), petroleum (drilling mud), and pharmaceuticals (foundation, stomach remedies) [2]. The properties of clays are highly varied and can be modified depending on specific applications.

In the Republic of Congo, clay is very abundant, from north to south. Dongou is a town located in the Likouala department, in the north of the country. Clay is used primarily in the manufacture of terracotta bricks, and other uses are virtually nonexistent in this department. However, some studies on the characterization and valorization of clay minerals have been initiated, both in the southern and northern parts of the country [3,4,5,6,7]. Clays have been more extensively characterized in the southern part of the country. The results of this work have shown that the Congolese

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subsoil is rich in a wide variety of mineral species. Despite this diversification of mineral species, the use of clay remains traditional and very limited, with insignificant yields. Yet this sector could positively boost the Congolese economy.

The nature of the clay minerals and the manufacturing processes influence the quality of the ceramic product. In the Dongou area, there is a lack of knowledge about the physicochemical properties of clays.

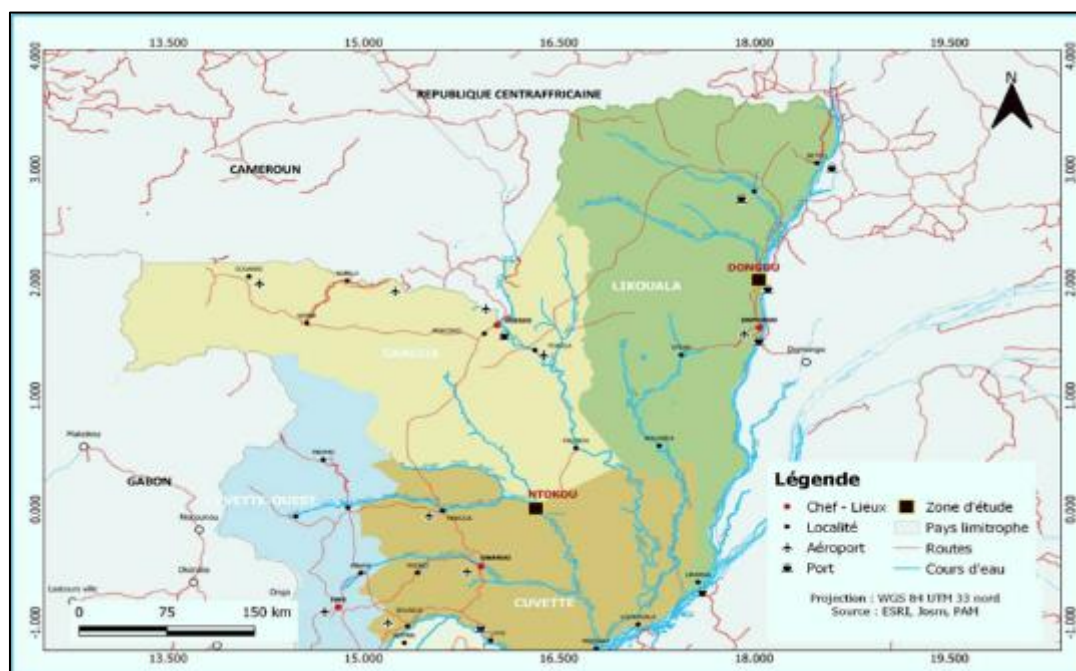
The overall objective of this work is to characterize the clay used for terracotta in the Dongou area. This will allow for the creation of a substantial database, facilitating the use of clays from this locality. The texture of the clay soil in Dongou will be determined by its particle size distribution (wet sieving and sedimentation analysis), while its maximum water content and plasticity will be determined using the Atterberg limits. The mineralogical composition of the clay soil in Dongou will be determined by XRD, and chemical analysis by XRF. The potential uses and technological properties of the clay soil in Dongou will be studied.

## 2. Materials and methods

This section presents the geographical situation of Dongou and the methodology required for carrying out the work.

### 2.1. Geographical locations of Dongou.

The soil sample from Dongou was taken from the Likouala department, specifically from the Mbala neighborhood in the Dongou district. The sample was taken at a depth of 50 cm and a height of approximately 4 m. This area had not undergone any prior treatment.



**Figure 1** Location map of Dongou.

### 2.2. Characterization methods

The limits of Atterberg are measured according to standards NF P94-051 [8]. The proportions of the following particle classes are determined according to standards NF P 94-056 [9]. X-ray diffractogram is recorded at Ceramic Technology Transfer Center (CTTC) in Limoges using a PANalytical X'Pert PRO brand diffractometer using the K wavelength of copper [10]. Chemical composition in major elements is determined in Dangote's laboratory in the Republic of Congo. The temperature increase is carried out at a rate of 5°C/min. The maximum temperature is 1200°C. Cooling is freely dependent on the inertia of the furnace.

## 2.3. Technological properties

### 2.3.1. Linear withdrawal.

Shrinkage is the decrease in a material's dimensions compared to its initial values following drying or heat treatment. When it relates to the change in a single dimension, as in our case, it is referred to as linear shrinkage (LS). In our work, linear shrinkage was determined along the length of the parallelepiped specimens. The measurements were taken using a caliper (ROCH France, patented SGD G).

The linear shrinkage is given by the following relationship:

$$RL(\%) = \frac{L_0 - L_c}{L_0} \times 100 ; RI(\%) = \frac{l_0 - l_c}{l_0} \times 100 ; Re(\%) = \frac{e_0 - e_c}{e_0} \times 100. (1)$$

With :

RL: Setback relative to Length; RI : Setback relative to Width;

Re : Shrinkage relative to thickness.

$L_0$ ,  $l_0$  and  $e_0$  are respectively the Length, width and thickness before heat treatment ;

$L_c$ ,  $l_c$  and  $e_c$  are respectively the Length, width and thickness after cooking.

### 2.3.2. Percentage of water absorbed.

The water absorption coefficient (Abs), or percentage of water absorbed by a material, is the ratio of the increase in its mass caused by partial water imbibition to its dry mass ( $M_s$ ). This measurement is performed on cylindrical specimens. This partial imbibition is achieved by completely immersing the sample in demineralized water while the entire system is brought to a boil and maintained at atmospheric pressure for two hours. After 24 hours of cooling at room temperature, the sample is removed from the bath, dried with very fine absorbent paper, weighed, and its wet mass ( $M_a$ ) is determined.

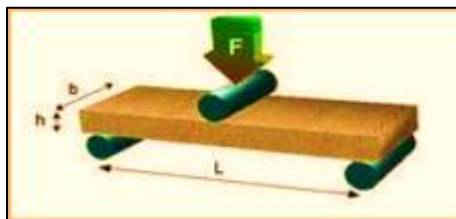
The percentage of water absorption is given by relation (2).

$$Abs = \frac{M_a - M_s}{M_s} \times 100 (2)$$

### 2.3.3. Three-point bending strength

This test measures the breaking strength of a material. The EZ20 device is used with a crosshead travel speed of 0.35 mm/s. A parallelepiped specimen of the material to be tested is placed on two supports, and an increasing force is applied until failure occurs at the center of the specimen. The distance between the specimens is 80 mm (Figure 2) [11, 12].

$$\sigma(\text{MPa}) = \frac{3 F(N) \times L(\text{mm})}{2 b(\text{mm}) \times h^2(\text{mm})} (3)$$



**Figure 2** A test specimen in three-point bending.

### 3. Results and discussion

The different methods used yielded the following results:

#### 3.1. Geotechnical characterization

The geotechnical characterization yielded the following results:

##### 3.1.1. Particle size analysis

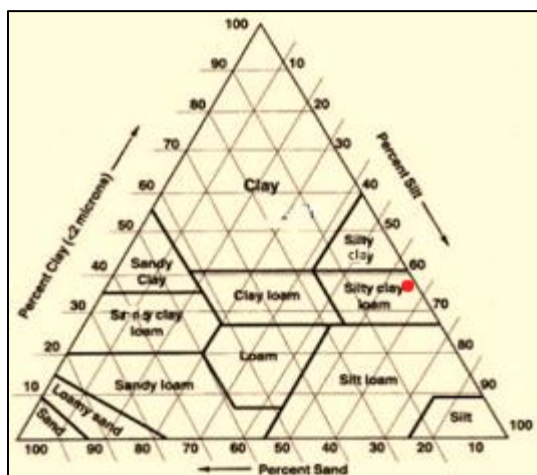
Table 1 presents the results of the granulometric analysis of the DON soil.

**Table 1** Results of the particle size analysis.

Sample	<2 $\mu\text{m}$ (%)	2-20 $\mu\text{m}$ (%)	>20 $\mu\text{m}$ (%)	Liquid limit (%)	Plasticity limit (%)	Plasticity index (%)
DON	62	36	2	70.1	30.1	40

Thanks to the particle size distribution:

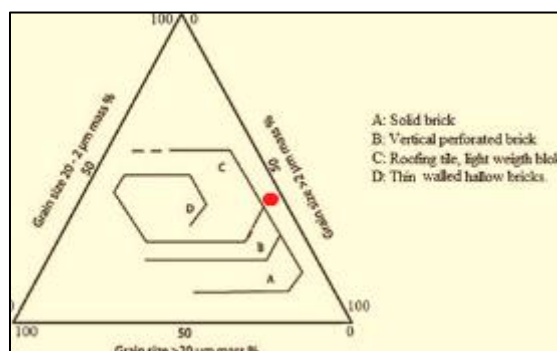
- Figure 3 shows the positioning of the DON soil in the Texture Triangle



**Figure 3** Positioning of DON in the texture triangle [13] .

And it turns out that the DON soil has a silty-clay texture;

- Figure 4 shows the positioning of the DON soil in the Winkler Diagram

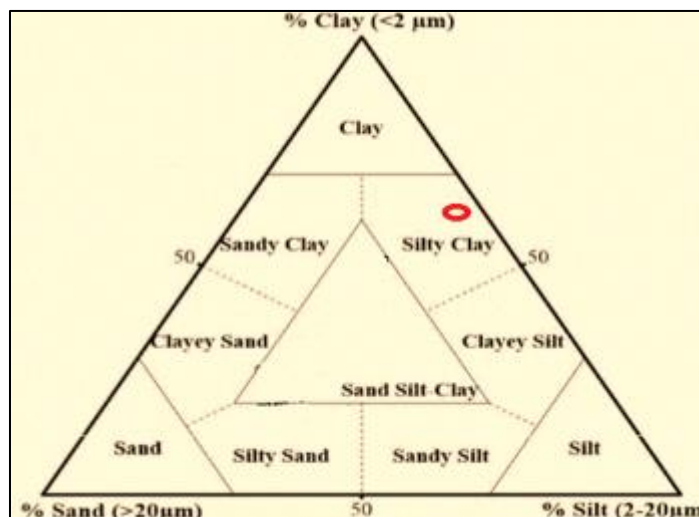


**Figure 4** Positioning of DON in the Winkler Diagram [14].

It appears that the DON soil is not suitable for the manufacture of terracotta products. However, due to its proximity to zone C (Figure 4), it may be possible to use it in the production of lightweight block tiles by adding a small proportion of degreaser.

For bricks, coarse particle sizes are acceptable, whereas for tiles, a particle size with a percentage of fine elements (20-50) is preferred [15]. This soil, with 62% clay, could be used in tile production.

- Figure 5 shows the positioning of the DON ground in the Shepard diagram.



**Figure 5** Positioning of DON in the Shepard Diagram.

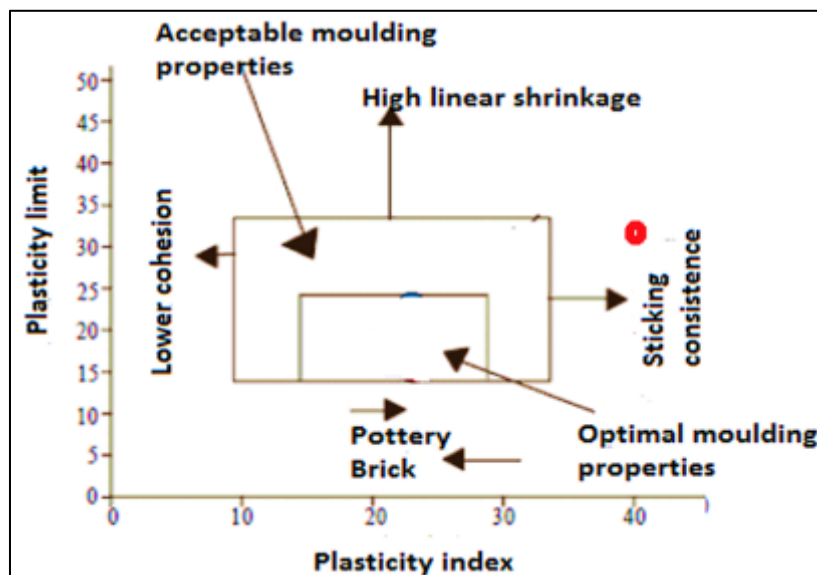
The DON sample is located in the clay loam field, an area of very high frequency of use.

### 3.1.2. The limits of Atterberg

Table 1 shows the results of the Atterberg limits.

Thanks to the results of the Atterberg limits:

- Figure 6 shows the position of the DON ground in the Casagrande Abacus.

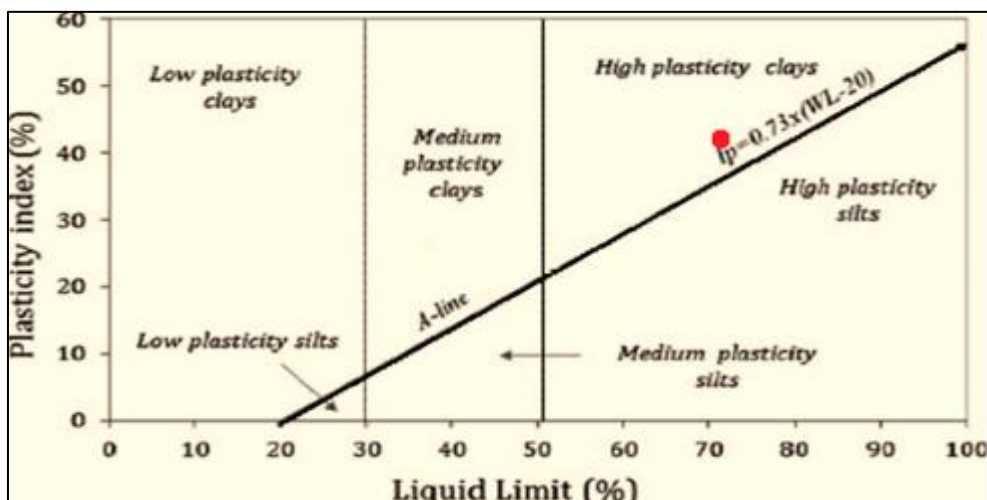


**Figure 6** Positioning of DON in the Casagrande Abacus [16]



DON It is a very plastic clay. This could be explained by the fact that it contains 62% clay.

- Figure 7 shows the position of DON in the Formability Diagram.



**Figure 7** Positioning of DON in the Shapeability Diagram [17].

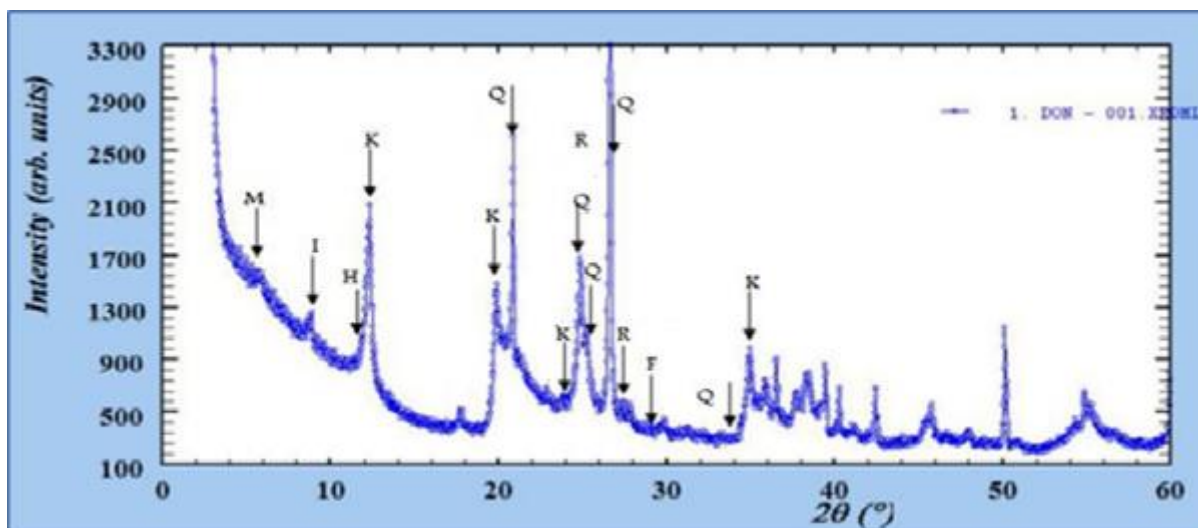
DON falls within the range of materials with optimal molding properties. DON has high plasticity. All of this is consistent with the results of the particle size analysis (with a clay content of 62% and Atterberg limits showing a plasticity index of 40).

### 3.2. Mineralogical Characterization.

The characterizations were carried out according to:

#### 3.2.1. Identification of structural species by XRD

Figure 8 shows the DON diffractogram in a range of angular analyses from 3 to 90°C.



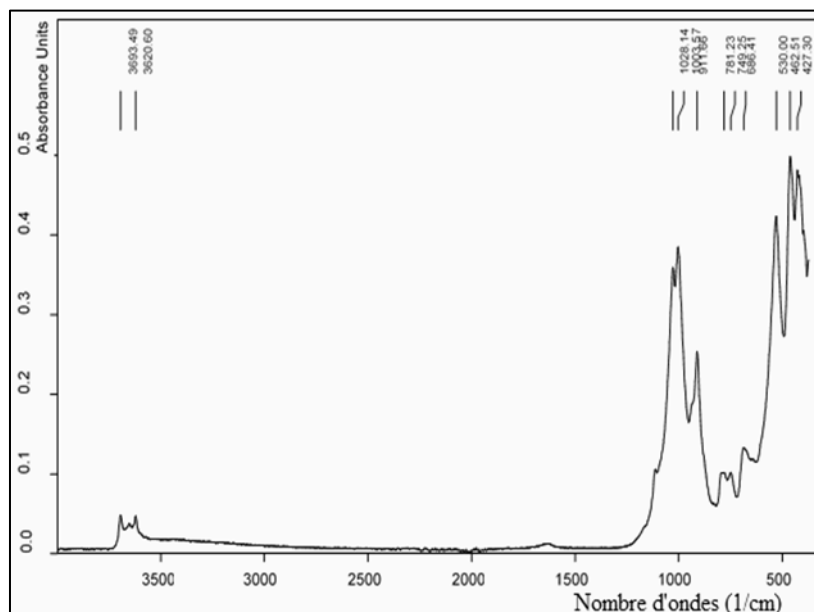
**Figure 8** Diffractogram of the raw DON sample

M: Montmorillonite ; K: kaolinite; Q: Quartz; Goethite R: Rutile; H: Holloysite.

The crystalline phases in DON consist of kaolinite, traces of montmorillonite, quartz, rutile, traces of halloysite, and traces of orthoclase. Kaolinite is predominant. DON is therefore a clay that is mainly kaolinite.

### 3.2.2. IR identification.

Figure 9 shows us the IR spectrum of DON.



**Figure 9** DON IR Spectrum

Analysis of Figure 7 shows wavenumbers of  $3693.49\text{ cm}^{-1}$  and  $3620.60\text{ cm}^{-1}$  in the valence vibration range, and  $911.66\text{ cm}^{-1}$  and  $781.23\text{ cm}^{-1}$  in the angular strain vibration range. These hydroxyl groups correspond to the characteristic hydroxyl vibrations of dioctahedral minerals, particularly kaolinite [18]. The shape of the IR spectrum confirms the information provided by X-ray diffraction.

### 3.2.3. Elemental chemical analysis of DON

It is presented in the form of major oxides in Table 3.

**Table 2** Chemical composition (in % oxide mass) of DON clay materials

Specimen	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	PF	Total
DONATION	49.92	28.27	2.93	1.04	0.13	- - - -	11.1	93.39				

Table 2 gives the results of chemical analysis of major elements (Si, Al, Fe, Mg, and Ca) expressed as the percentage of the most stable oxides. The SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> mass ratio allows for the classification of clays. DON clay has a SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> mass ratio of 1.76 instead of 1.17 for pure kaolinite. This could explain the slightly higher percentage of silica in DON clay or the presence of 2:1 phyllosilicate materials [19].

The iron oxide content (2.93%) could explain the presence of ferric montmorillonite, justifying the high plasticity of DON clay [19].

The absence of alkali oxides could explain the lack of fluxing materials that reduce porosity and play the role of cement in the material.

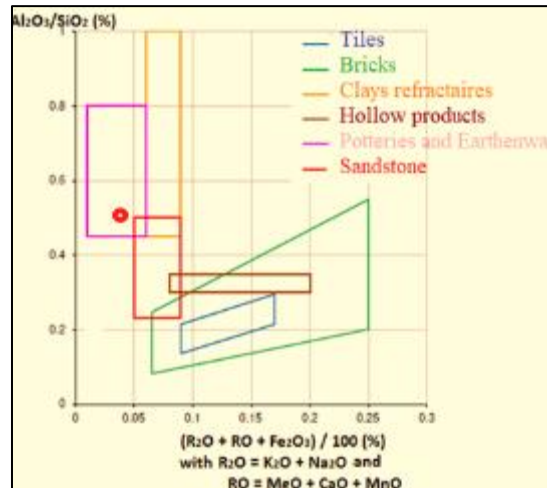
Magnesium oxide content (1.04%) could explain the presence of traces of talc in the DON clay and give the sherd good resistance to thermal shock and low expansion. The presence of colored oxides (iron oxide at 2.93%) would indicate that the firing products would be colored [20].

The value of loss on ignition (11.1%), not very different from that of kaolinite (13.9%), would explain the presence of disordered kaolinite consistent with a refractory character of these samples heated to different temperatures [21].

### 3.3. Potential uses of DON clay soil in the ceramics industry

Thanks to its chemical composition, DON clay has been compared to other clay materials in the world such as those from Italy, France and some African countries:

The Figure 10 shows the positioning of DON clay in the Fiori Diagram.

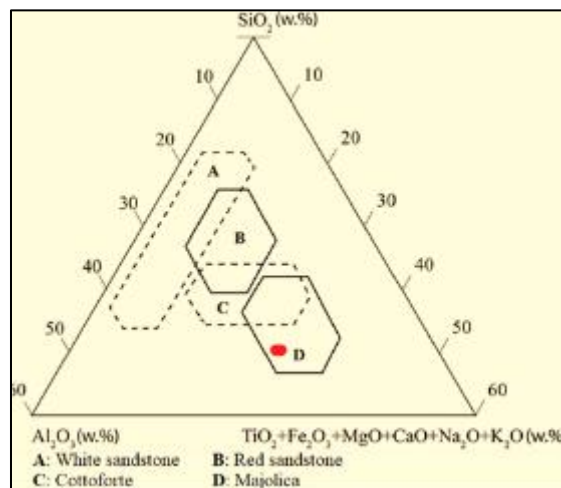


**Figure 10** Positioning of DON sample in Fiori diagram [22]

The chemical composition, according to the Fiori Diagram, shows that DON clay is in the D domain, the domain of ceramics used for the manufacture of decorative objects (plates, tiles, fabrics, etc.) [22].

Furthermore, since DON clay is poor in alkali and alkaline-earth oxides, enriching this clay with fluxing materials could bring it closer to domain C (domain close to domain D) and promote the manufacture of impregnating agents that act as protection for ceramics (terracotta bricks, tiles, porous cements) against water, oils, and greases.

The Figure 11 shows the position of DON clay in the Augustinik Diagram.



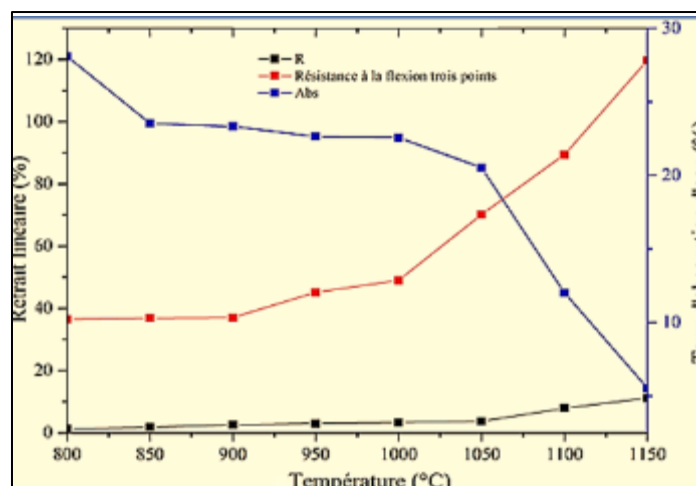
**Figure 11** Positioning of DON sample in Augustining diagram

The DON sample is positioned in domain D. We observe that DON clay could be used for the manufacture of pottery and earthenware.

#### 3.3.1. Technological properties

There Figure 12 shows the different linear shrinkage, water absorption and three-point bending curves.





**Figure 12** Linear shrinkage, water absorption, and three-point bending strength curves

Linear shrinkage is a parameter that reflects the reactivity of a material during firing [23]. From 800 to 1050°C, we observe a slight increase in the linear shrinkage curve. We can deduce that the shrinkage is low. This may be due to the loss of water of constitution. This result is normal because the quartz present in DON, although in a small percentage (2%), is a degreasing agent over this temperature range. Above 1050°C, linear shrinkage increases sharply. A rearrangement reaction occurs within the product (transition from metakaolinite to mullite or the formation of a spinel phase and possibly the appearance of alumina) [24].

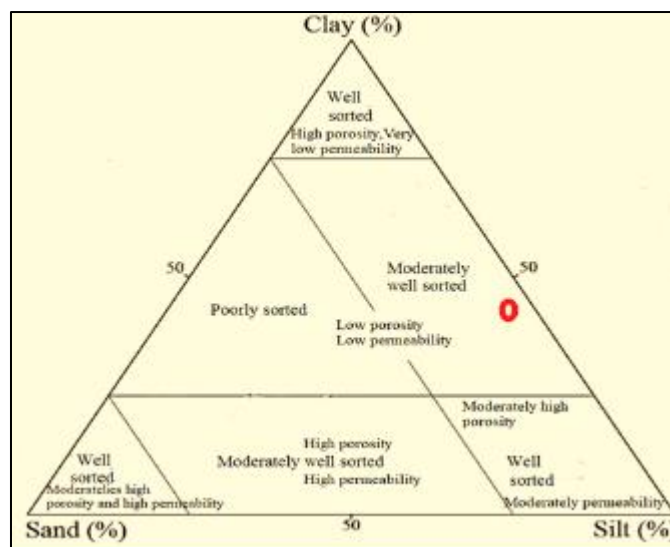
The absorption rate is a property that provides insight into the density of the shard. Figure 12 shows a decrease in the water absorption rate as a function of temperature. From 800 to 1050°C, we observe a slight decrease in the water absorption rate curve. We can therefore deduce that the shard absorbs a significant amount of water (21.3 to 27.9%). This may be due to a low density of the shard. Above 1050°C, the curve decreases more rapidly and reaches a lower rate of 5.54%. This indicates a high density of the shard caused by consolidation reactions.

We also observe that linear shrinkage during cooking is associated with a decrease in the absorption rate, with the intersection at 1050°C. Therefore, the higher the absorption rate, the lower the shrinkage, and conversely, the lower the absorption rate, the greater the shrinkage.

Figure 12 shows the growth of the three-point bending curve as a function of temperature. Between 800 and 950°C, it increases slightly. This is due to a low densification of the sherd. Above 1000°C, we observe a strong increase in the ceramic size. This is due to a strong densification caused by the formation of mullite [25].

**The Figure 13 shows a ternary diagram, presenting the DON paste according to the relationship between sand, silt on the one hand and its control on sorting, porosity and permeability on the other.**

Permeability and porosity are properties that provide insight into a raw material's suitability for molding or shaping. Permeability characterizes the clay's absorption of water. A ceramic paste forms from this water-saturated clay, and the resulting ceramic materials exhibit a certain degree of cohesion after molding or shaping. El Ouahabi et al. demonstrated that raw materials with high permeability have low cohesion and are difficult to mold or shape, while those with low permeability have good consistency and are easy to shape [26]. DON clay with moderately well-sorted grains falls within a range of low permeability and porosity, resulting in good suitability for molding, throwing, or shaping.



**Figure 13** Ternary diagram: relationship between sand, silt on the one hand and its control on sorting, porosity and permeability [26]

#### 4. Conclusion

This work involved the mineralogical, physicochemical, and technological characterization of a clay used for terracotta in the locality of Dongou, in the Likouala department of the Republic of Congo. The soil from Dongou exhibited a silty-clay texture due to its particle size distribution. This also indicates that the clay soil from Dongou is suitable for the manufacture of lightweight block tiles. Atterberg limits confirmed its clayey nature and allowed it to be classified as a highly plastic soil with optimal molding properties. The clay soil from Dongou showed low permeability and porosity, which gives it good shaping capabilities and relatively high flexural strength. Kaolinite and quartz were identified by XRD. Chemical analysis of this soil revealed the following major oxides:  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{MgO}$ , and  $\text{CaO}$ . The Dongou clay has a  $\text{SiO}_2/\text{Al}_2\text{O}_3$  mass ratio of 1.76, which has led to the suspicion of the presence of type 2 phyllosilicate materials. With a  $\text{MgO}$  oxide content (1.04%), this could suggest the presence of traces of talc in the DON clay. The presence of the colour oxide (iron oxide at 2.93%) shows that the products obtained after cooking will be coloured.

#### Compliance with ethical standards

##### *Disclosure of conflict of interest*

No conflict of interest to be disclosed.

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