PHYSICO CHEMICAL PROPERTIES OF SANGARÉCLAY IN THE TRADITIONAL CERAMIC INDUSTRY AND PROPERTIES OF BUILDING BRICK: INFLUENCE OF TEMPERATURE ON MECHANICAL PROPERTIES OF FIRED PRODUCT.

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ABSTRACT

The clay material of the locality of Garoua has been the subject of a mineralogical and physical characterization in order to assess the potential of its use in the field of ceramics. Firing experiments were carried out on experimental briquettes at temperatures of 900°C, 1000°C and 1100°C. Then physical tests and mineralogical analyzes have been made on the cooked products. The results obtained in the Mineralogy show that the clay material contains kaolinite, illite, quartz and feldspar, and once cooked the presence of a new crystal phase is detected: the mullite. From the geotechnical point of view, the material has a particle size spread with a plasticity index greater than 20%. Its activity is less than 0.75. It is a kaolinite sandy clay with a low plasticity. The pH measurement show the character weakly basic of this clay. The technological parameters of the experimental briquettes show that the percentage of water absorbed is less than 15%. The linear withdrawal of all experimental briquettes presents values less than or equal to 10%. The values of the mechanicals resistances of the briquettes increase globally with the

temperature, not exceeding 10 MPa for the bending, but reaching 38 MPa for the compression. All these parameters except the linear withdrawal present optima at 1100°C. The clay material of Garouais suitable for the manufacture of bricks, and tiles at 1100℃.

Keywords: clay material, mineralogy, ceramic.

INTRODUCTION

Clays have been used by man since the dawn of time. The natural abundance and the immediate availability of clays explain their great uses over time. The particularity of this material is its capacity to be able to pass to the plastic state for appropriate contents in water [1]. This material also has the power to be shaped, to shrink, to harden after drying and to consolidate thanks to a firing that allows the formation of a vitreous phase more or less important [2]. This last find was in the same way as the agriculture at the base of the first human civilizations. Today, they are used as a minor constituent as a majority raw material, for example for the production of widely used ceramic materials such as building materials and decorative objects. While the consumption of these products is becoming more widespread, their production remains very unsustainable in some developing countries [3]. In Cameroon, for example, most industrial clay ceramics are imported. This situation can be explained by the virtual absence of a real industrial fabric and a poor estimate of the potential of local resources. In addition to this, the low level of popularization of high-quality ceramic products in our context may also stem from the poor mastery of production technologies that must first be based on the mineralogical and physicochemical characterization of the natural material. The clay materials used for the production of ceramics are often complex natural mixtures of minerals whose granulometry and physicochemical properties are highly variable [4]. User choice criteria are less related to the overall chemical and mineralogical composition of clay materials than to their behavior during the various stages of the manufacture of ceramic products, but this is of paramount importance because, for lack of knowledge, it is often accused of drying and cooking cause the poor quality of the ceramic products and the waste observed at the exit of the oven and much more rarely the preparation of the ceramic paste and its composition [5]. The mastery of the intrinsic parameters of the crystalline material passes through essential analyzes among which the X-ray diffraction analysis which makes it possible to identify the crystalline phases, to follow the evolution of these during the sintering in order to be able to detect the different physicochemical phenomena occurring during this one. Once the sintering has been carried out, an equally important element is the evaluation of the technological parameters of the baked test pieces in order to confuse the precise field of technological application of the ceramic thus obtained. A number of studies have already been carried out on Cameroon clay materials based on kaolinite, illite, smectite or talc [6]. They concerned the study of the physico-chemical characteristics, the catalytic properties

and the thermal behavior of some sources of clay matter. The results of this work aim to provide a sufficiently reliable database to support the start-up of industrial projects for local clay materials. It aims to study the technological ability of this material for the manufacture of ceramic products for wide dissemination and sustainable local development.

MATERIALS AND METHODS

Presentation of the study area

During the construction of a well for access to underground water, a strange observation was made. On a lateritic horizon, at a depth of about 3 m, we notice the appearance of a layer of soil of a whitish and greyish color, layer of soil to which we pay all the interest of this work. The objective of this part is to present the major features of the studied area. Regarding soils, the parent material is mainly composed of clay and sand.

Geological context

Geologically, Garoua is based on sedimentary formations mainly sandstone which is a detrital rock formed in the Cretaceous and resulting from the aggregation of grains of sand [7]. There is also the presence of massifs of gneiss which is a metamorphic rock composed of quartz, mica and feldspars. The Sudanese tropical climate of the region combined with the presence of a river leads to the alteration of parent rocks and the formation of lacustrine clays [8] dating from the Quaternary period and which sediment in alluvium and eluvium and are sometimes covered with a humic horizon and lateritic. These phenomena explain the abundant presence of kaolinitic and illitic clay and vermiculites in the locality of Sangare-Paul [7].

Material sampling

On the whitish and grayish soil layer extracted from the well, a sampling targeted at the point of coordinates in decimal degrees, Latitude 9.285126 °and Longitude 13.455767 ° was carried out. More attention was paid to the compact and gray masses of uncontaminated soil which crumbled to the touch because the other fraction of completely whitish soil was macroscopically only sand. Documentary studies [7] suggested the clayey nature of this greyish material(figure 1). The samples taken are shown in Figure 2.

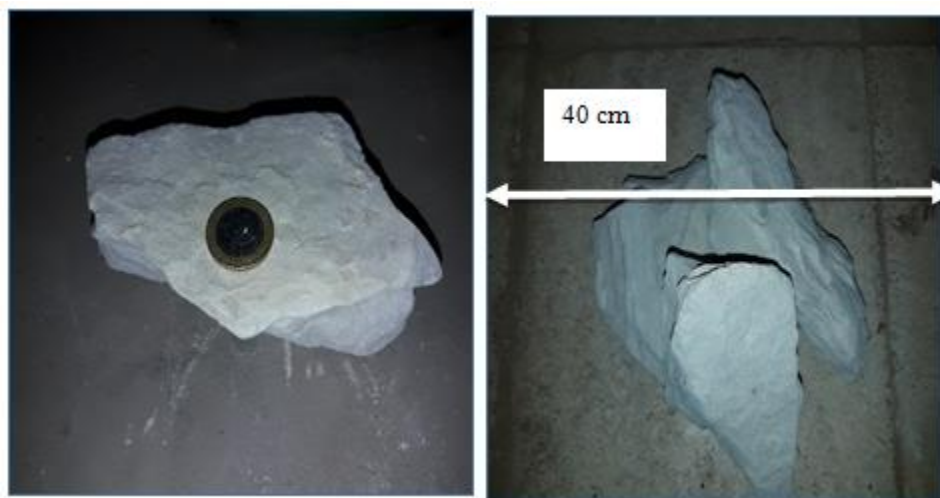**Figure 1: View of the situation at the sampling point (Well)**

Figure 2: Presentation of the samples taken

Particle size analysis

The particle size analysis allowed us to determine the dimensional distribution by weight of the grains constituting the sample. It is carried out in two steps: the particle size analysis by dry sieving after washing for particles larger than 90 µm and the particle size analysis by sedimentometry for particles smaller than 90 µm.

Particle size analysis by dry sieving after washing

The particle size analysis is carried out according to the French standard NF P 94-056 [10]. It consists of separating the agglomerated grains of a known mass of material by stirring under water through a sieve with a 90 µm opening. Once the residue has dried, the accumulated residue is weighed on a series of sieves nested one on top of the other and the dimensions of the openings of which decrease from top to bottom. The mass of residue accumulated on each sieve is related to the total dry mass of the sample.

Particle size analysis by sedimentometry

The test is based on the fact that in a liquid medium at rest, the speed V of settling of a solid particle is a function of its diameter. It is based on the law of STOKES which gives in the case of spherical grains of the same density, the relationship between the diameter of the grains and their sedimentation rate. The determination of the diameter of the grains based on the dispersion and the precipitation of the clay grains within the liquid. STOKES's law is given by the equation:

$$V = \frac{(\gamma S - \gamma W)}{18\eta} D^2$$ in cm.s-1 With

γ_S: density of the particle in g.cm^{-3} D: diameter of the particle in cm.

γ_W: density of liquid (water) g.cm^{-3} η: dynamic viscosity in poises

Having carried out the complete particle size analysis of the clay material, its coefficient of uniformity Cu can be calculated in order to classify the soil according to Table 1 [11].

Cu = D_{60}/D_{10} with D_{60} = effective diameter of the particles which corresponds to 60% of the passing D_{10} = effective diameter of the particles which corresponds to 10% of the passing.

Table 1: Particle size class of soils according to the Coefficient of Uniformity (CU)

Coefficient of Uniformity (CU)	Particle size class
CU≤2	very tight grain size
2<CU≤5	tight grain size
5<CU≤20	semi-spread grain size
20<CU≤200	spread grain size
200<CU	Very spread grain size

Atterberg Limits

Atterberg limits are water contents by weight corresponding to particular conditions of a soil. They are determined on the fraction of the soil passing through a 400 µm mesh sieve and are expressed as a percentage. It is :

- Liquidity limit (ωL): water content of the reworked soil characterizing the transition between a plastic state and a liquid state.

- Plasticity limit (ωp): water content of a reworked soil characterizing the transition between a solid state and a plastic state.

The plasticity index (Ip): it is the difference between the liquidity limit and the plasticity limit. Its formula is $Ip = \omega L - \omega p$. The test is carried out in two distinct phases according to the French Standard NF P 94-051 [12].

Classification of materials based on Atterberg limits

This classification of the state of a soil is made according to its plasticity index and is summarized in Table 2 below [12].

Table 2: Classification of materials according to the plasticity index

Plasticity index	Soil Condition
0-5	Not Plastic
5-15	Little Plastic Very Plastic
15-40	Plastic
>40	Very Plastic

The values of the Atterberg limits are linked to the quantity of water retained at the surface of the particles and therefore to the quantity of clay present in the soil [13]. The activity of clay is therefore defined by the ratio the plasticity index (Ip) and the percentage of elements less than two microns ($C_2 < 2$ µm).

$A = Ip/C_2$ Skempton [13] proposed the following nomenclature:

- Inactive clays: clays which have an activity lower than 0.75µm. These clays are based on kaolinite, or clays containing few clay minerals.

- Normal clays: which have an activity between 0.75 and 1.25µm.

- Active clays: which have an activity between 1.25 and 2µm.

- Very active clays: which have an activity greater than 2µm.

Apparent density

It is about a physical quantity used in order to account for the mass of material contained in a given volume, including the interstitial air. The apparent density Da of the material will be given by the formula: $Da = m_1 \times d / (d \times V - (m_2 - m_1))$ in g/cm^3 with d: density of the paraffin = 0.8 and $V = (V_1 - V_0)$

Actual density

The real density of the clay material is given by the following formula:

$Dr = ((m_2 - m_1) / (m_4 - m_1 - m_3 + m_2)) \times \rho$ in (g/cm^3) with ρ: the density of water

Mineralogical analyzes

They include X-Ray Diffractometry (XRD) and Infrared Spectrometry (IR).

X-Ray Diffractometry

XRD allows the identification and semi-quantitative determination of the main minerals present in a sample.

Infrared Spectrometry

IR spectrometry is a method of identifying mineral species in a sample from the vibration frequencies of the main bonds of these mineral species. This method was used in addition to DRX.

pH measurement

The pH of clay materials has a notable influence on the behavior and preparation of ceramic paste.

Test on prepared specimens

This part presents the production process of the experimental specimens as well as the evaluation of the properties obtained on the fired specimens such as: sound, linear shrinkage, apparent density, mechanical resistance to bending, mechanical resistance to compression and water absorption rate test.

Preparation of experimental specimens and firing

Test on the prepared specimens

In this part are presented the process of making the experimental specimens as well as the evaluation of the properties obtained on the fired specimens such as that: sound, linear shrinkage, bulk density, mechanical flexural strength, compressive strength and water absorption rate test.

Preparation of experimental specimens and firing

The clay material was previously ground in a porcelain mortar and oven dried at 105 ° C for 24 hours. Then the clay material is introduced into a jar with aluminum balls. The whole is closed and placed on a ball mill for one hour. The powder obtained is sieved at 200 μm. Two types of test specimens are then made using the hydraulic press before firing the materials at three experimental temperatures:

- Prismatic test pieces: These are $80 \times 40 \times 20$ mm sized test pieces containing 100 g of material for a water supply of 12% by mass. They were called Sp1, Sp2, Sp3 for baking at 950℃; Sp5, Sp6, Sp7 for that at 1000℃ and finally Sp8, Sp9, Sp10 for cooking at 1100℃.

- Cubic test tubes: These are $40 \times 40 \times 40$ mm test tubes containing 150 g of material for a water supply of 14% by mass. These test pieces were called Sp16, Sp17, Sp18 for baking at 950℃ .; Sp20, Sp21, Sp22 for that at 1000℃ and finally Sp23, Sp24, Sp25 for cooking at 1100℃.

The test pieces thus obtained were dried in the open air for three days then baked at 105° C. for 24 hours in order to avoid sudden drying during baking. The baking was carried out in an oven of maximum temperature 1200℃, the temperature increasing by 5℃ per minute to the desired temperature where the specimens are baked for one hour. The figure 3 shows the samples made

Figure 3: Prepared experimental specimens

Physical properties of ceramics

The following tests focused on determining the physical and mechanical characteristics of fired products in order to evaluate their performance.

Color

The color of the test pieces was determined by visual comparison with the color range of the Munsell chart [14].

The sound

The sound of the test pieces was determined after a few taps of the latter on the tile of the laboratory bench. The sound quality of the fired specimens reflects the good cohesion of the ceramics and the maturity of the fired products [15]. We can have matte, metallic, very metallic tones.

Linear firing shrinkage

Linear firing shrinkage is the reduction in length of material dimensions after heat treatment. The test is carried out according to standard ASTM C531-2000 [16]. One of the fundamental criteria during the manufacture of ceramics is the control of contractions during firing because, the strong contractions can lead to deformations [17].

Loss of mass during cooking

The loss of mass during cooking makes it possible to know the quantity of products liable to decompose or volatilize during cooking. Between the drying phase and the firing phase of the briquettes at different temperatures, there was a decrease in mass which is determined by the following relation [18].

Apparent density

It is defined by the quotient of the dry mass of the sample by the volume occupied by the solid matter, including the voids contained in the grains (apparent volume).

Procedure: Determine the dry mass Ms of the test piece of material after firing. Then directly calculate its volume V from its dimensions by applying the formula $V = L{\times}l{\times}e$. The apparent density φ is given by the formula below: $\varphi = Ms/V$ where in (g/cm^3) Ms: dry mass (g) V: volume of the test piece (cm^3)

Water absorption rate test

The water absorption rate T_{ab} is the percentage of water absorbed by an immersed material for 24 hours. It makes it possible to follow the evolution of the open porosity with the sintering. The test is carried out according to standard ASTM C 20-2000 [19]. The percentage of absorbed water Tab is given by the relation: $\%Tab = ((Ma-Ms)/Ma){*}100$ where Ms: mass of the test piece after baking (g) Ma: mass of the test piece after 24 hours of immersion (g)

Mechanical flexural strength

Mechanical flexural strength is the limiting stress before flexural failure of a material. It makes it possible to determine in a relative manner in a material, the variation of the bonding forces between the particles with respect to one another. The tests are carried out according to Standard ASTM F 417-996 [20], using a bending device which uses the three-point method. The flexural strength, in MPa is given by the following relation:

$\varepsilon f = 1.5 P.d/le^2$ With d: distance between the cylindrical supports (mm) e: thickness of the test piece (mm) P: load applied at break (KN) l: width of the test piece (mm) and εf in Mpa.

Mechanical resistance to compression

The mechanical resistance to compression is the limiting stress before the compression failure of a material. It makes it possible to determine in a relative manner in a material, the variation of the cohesive forces of the material tested.

RESULTS AND DISCUSSION

These results concern the geotechnical, physical, mineralogical characterization and global evaluation of the properties of the Sangaré-Paul clay material once transformed into ceramics.

Particle size analysis

Table 3: Granulometric analysis of the clay material of Sangaré-Paul

Proportion of materials (%)				
(%)engravings d<2000µm	(%) sand 20< d<2000µm	(%) slit 2< d<20µm	(%) clay d<2µm	Total
0,53	41,15	12,28	46,04	100

The granulometric analysis data are grouped in Table 1. The clay material consists of 0.53% gravel, 41.15% sand, and 12.28% silt and 46.04% clay. It is apparent from the exploitation of the results that the Sangaré-Paul clay material has a very wide granulometry because its uniformity coefficient is greater than 200. It is a sandy clay with a few silts and traces of gravel.

Atterberg limits

The results of tests on the clay material which allowed the determination of the values of the Atterberg limits are contained in Table 2. In view of this table, the value of the plasticity limit ωP is 9.21%. The liquidity limit is ωL 30.63%. The plasticity index Ip is 21.42%. The activity value of this clay is 0.5. It is an inactive clay with a clay mineral dominating kaolinite [13].

Table 4: Results of the Atterberg limits of the Garoua clay material

Liquidity limit ωL(%)	30.63
Plasticity limit ωP(%)	9.21
Plasticity index Ip(%)	21.42

Mineralogical analyzes

X-ray diffractometry

Examination of the total powder diffractogram obtained for the sample tested shows the presence of the following minerals with the inter-reticular distances of their main lines: Kaolinite (Kao): reflection at 7.17 Å, 1.48 Å, 3.576 Å ; Illite (Ill): reflection at9.995 Å, 4.996 Å, 4.45 Å; Quartz

(Qz): reflection at 4,255 Å, 3,343 Å, 1,81 Å. Potassium Feldspars (Fds): reflection at 3,240 Å, 2,753 Å, 2,159 Å. We also note the presence of the peak corresponding to the total clay inter-reticular distance 4.45 Å. It is at this peak that we find all the clay minerals.
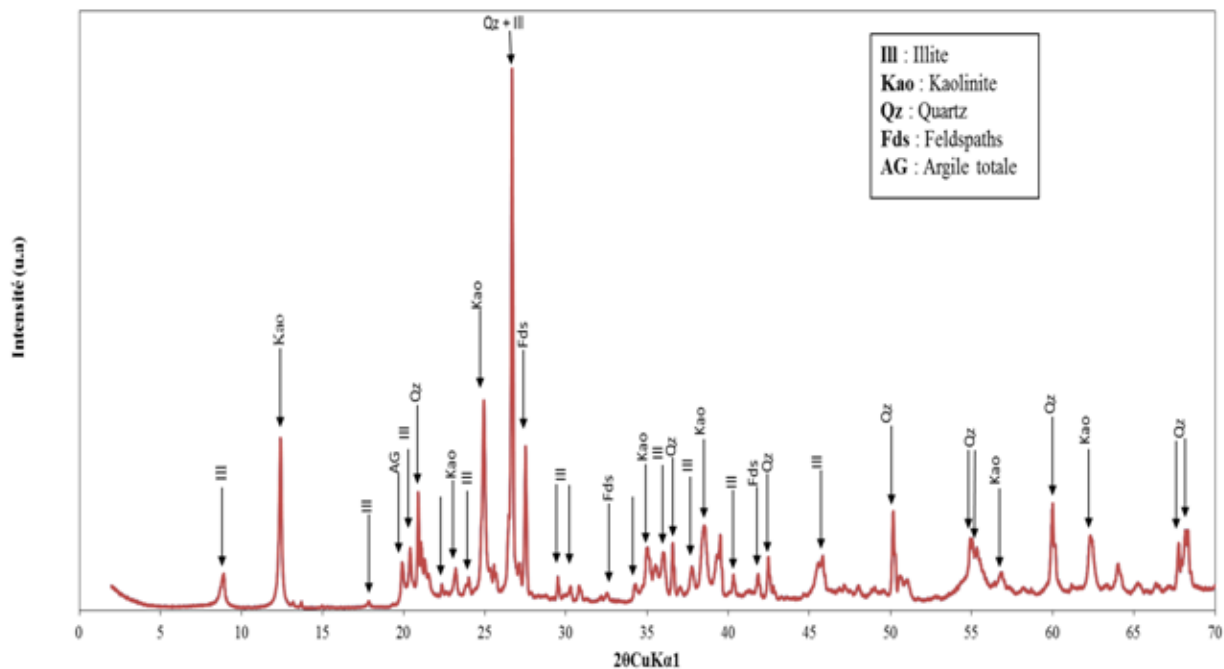

Figure 4: Diffractogram with total clay powder

Figure 3 shows the oriented blade curves obtained from the clay fraction less than 2 microns. The diffractogram obtained on the normal powder shows the presence of kaolinite and illite. The diffractogram obtained shows that the peak of kaolinite detected on the diffractogram of the normal oriented slide disappears, which confirms that it is indeed kaolinite because it is very sensitive to heat. The illite is present with a large peak of inter-reticular distance 9.97 Å.
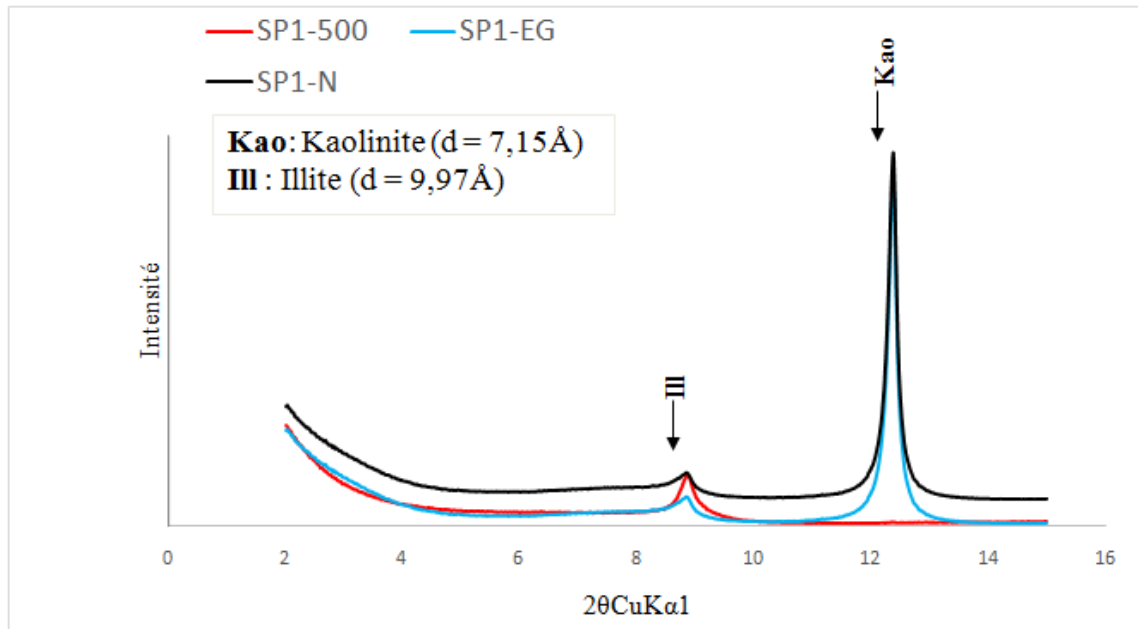
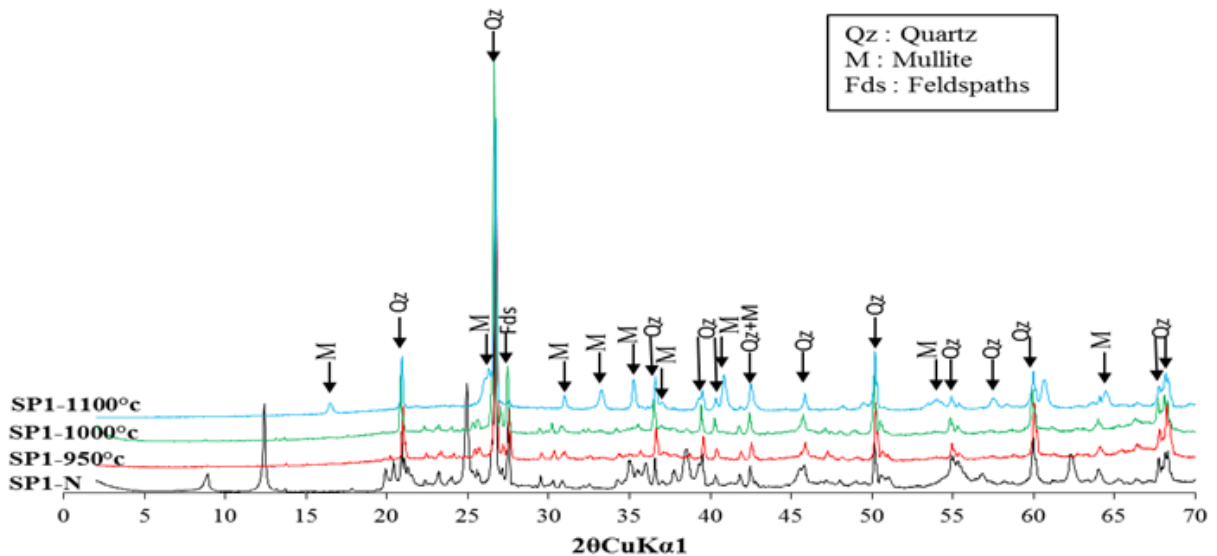**Figure 5: Diffractogram obtained in preparation oriented on clay fraction.**


Figure 6: Superposition of the diffractograms obtained on the powders of the test pieces baked at 950, 1000 and 1100°C.

Figure 6 shows the superposition of the diffractograms obtained on the powder of the raw material (SP1-N) and on the powders of the materials baked at 950℃. (SP1-950℃.) at 1000 ℃.

(SP1-1000°C.) and at 1100°C (SP1-1100°C).This figure shows the disappearance with the evolution of the temperature of certain phases. On the SP-950°C diffractogram, the characteristic lines of kaolinite disappeared. Pabst, Gregorov and Ticha suggest that from 450 °C up to 750°C the clay minerals decompose, losing their build water and deconstructing completely. This is the case of kaolinite which transforms into metakaolin ($2SiO_2$, Al_2O_3) by dehydroxylation following the reaction [21]:

$$Al_2Si_2O_5(OH)_4 \xrightarrow{\text{450 °C}} 2SiO_2,\ Al_2O_3 + 2H_2O$$
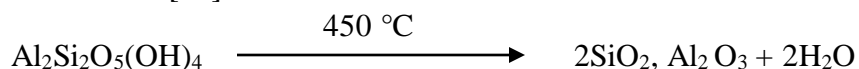
Then the metakaolin obtained which is characterized by a lack of order at great distance in the arrangement of the constitutive elements its solid [22] thus not detectable by XRD, will know a structural reorganization to 1000°C. It is transformed into the spinel type phase ($Si_3Al_4O_{12}$) according to the reaction below [23] with the addition of quartz β (SiO_2) [24], which is a polymorph of silica at a temperature of 1000°C at low temperature and pressure.

$$2(2SiO_2,\ Al_2O_3) \xrightarrow{\text{1000°C}} 3SiO_2,\ 2Al_2O_3 +\ SiO_2$$
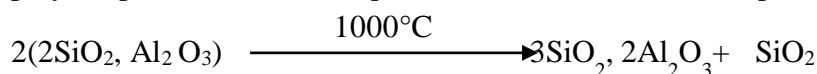
The increase of the sintering temperature (1100°C.) leads to the disappearance of the spinel phase and to the appearance of a high mullite content as shown by the diffractograms SP1-1100°C and SP1-1000°C with the multiplication of the main lines of mullite between 1000°C and 1100°C. These lines are detected at 3.39Å, 2.20Å and 5.38 Å inter-reticular distance. Mullite is an aluminum silicate that forms at high temperatures and low pressures [2]. The mullite crystals ($3Al_2O_3$, $2SiO_2$) appear from 1000°C by forming from the metakaolin and the spinel phase according to the reaction below and their continuous growth with the increase of the sintering temperature [2].

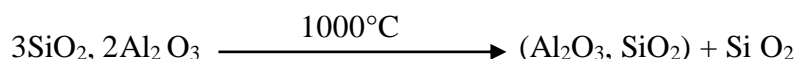$$3SiO_2,\ 2Al_2O_3 \xrightarrow{\text{1000°C}} (Al_2O_3,\ SiO_2) + Si\ O_2$$

Figure 4 also shows the disappearance of another crystalline phase: that of illite. Huger [25] suggests that illite, just below 1050°C, becomes mullite. He speaks of primary mullitization. This is explained by observing the superposition of diffractograms. Up to the sintering temperature of 1000°C, residual peaks of illite which has dehydroxylated from 450°C are observed. At 1100°C (SP1-1100°C), these peaks disappear: the illite completely transformed into mullite during a mullitization parallel to that of metakaolin. [2].

Obtaining mullite from metakaolin and the spinel phase is reminiscent of primary mullitization as suggested by the Jouenne work [26].

Quartz and feldspars are present on all the diffractograms obtained from the different sintering temperatures tested. The melting point of quartz (1700°C) [24] and that of potassium feldspars

(1300°C) is much higher [24]. Quartz is nevertheless subjected to allotropic transformations which occur around 573°C. The transformation of quartz from the so-called low-temperature form α to the so-called high-temperature form β is accompanied by a volume expansion. Around 600°C, there is a reversible dimensional change characteristic of the quartz transition. Around 850°C, it is transformed into β-quartz or tridymite according to the pressure gradient [24]. As long as it is not partially dissolved in the viscous flow, the quartz forms a percolating rigid skeleton which opposes the densification of the materials. The densification of ceramics is due to the formation of viscous flux from the dehydroxylation products of illite and kaolinite [2].

Infrared spectrometry

In the clay material, bands characteristic of the presence of kaolinite (3689-3649cm^{-1}) have been found because the valence vibrations of four OH groups composing the elementary mesh of kaolinite result in four bands centered at 3695, 3667, 3651, and 3620 cm^{-1} [27]. The 3619 cm^{-1} band reflects the O-H bond deformation and materializes the presence of kaolinite [27]. The 1114 cm^{-1} band and the 1004 cm^{-1} band correspond to the elongation vibration of the Si-O bond of kaolinite. The bands observed around 914 and 916 cm^{-1} correspond to the deformation vibrations of the Al-O bond of the clay minerals [28]. The presence of Quartz is indicated by bands between 600 and 800 cm^{-1} [3]. The spectrum obtained for the studied material is illustrated in Figure 7. Figure 9 crosses the bands between 3500 and 4000 cm^{-1} of the infrared spectrum and corresponding to the main vibrations characteristic of kaolinite. Figure 8 illustrates the peaks of the infrared spectrum whose corresponding bands are between 500 and 1900 cm^{-1}.
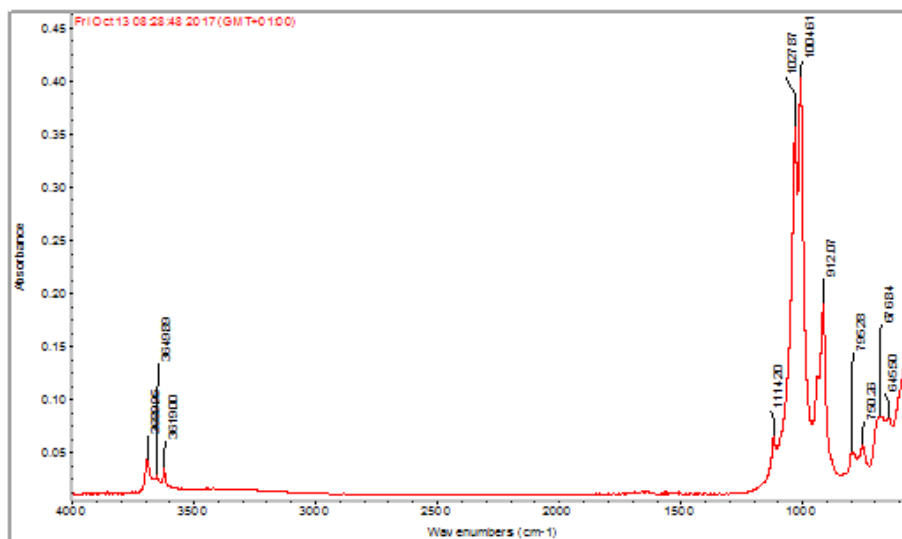

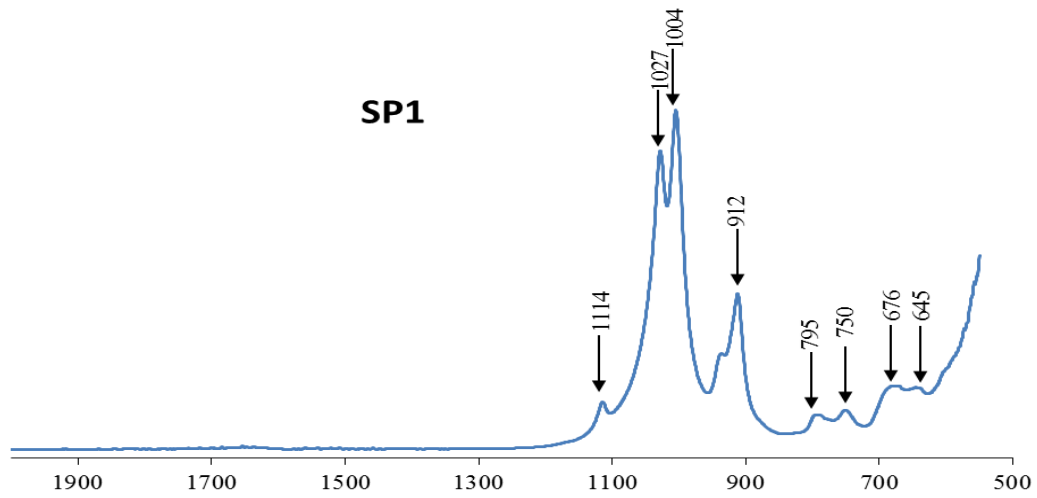**Figure 7: Infrared spectrum of the analyzed material**

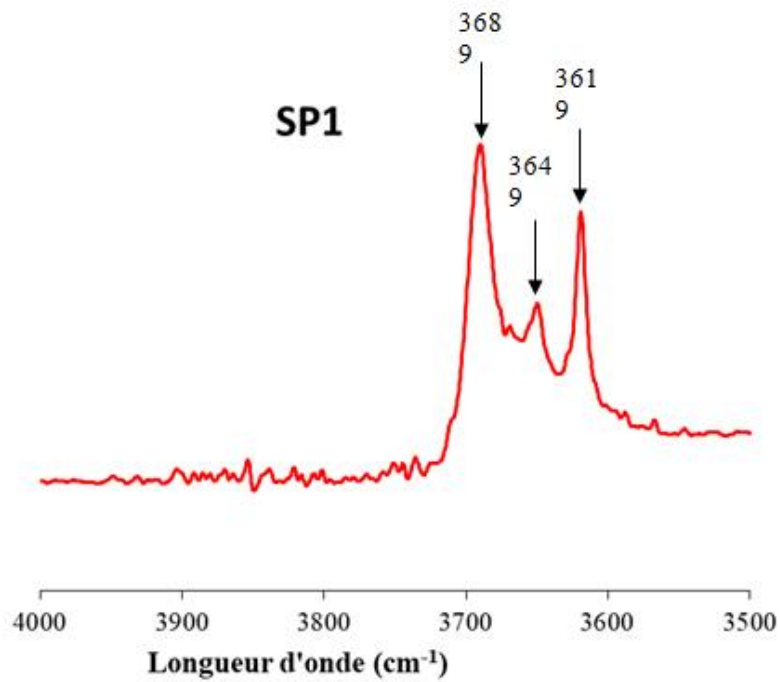**Figure 8: Infrared spectrum of the analyzed material**

Figure 9: Infrared spectrum of the analyzed material

Table 5: Bands of the IR spectrum corresponding to the main minerals present in the clay material of Sangaré-Paul

Wave number (en cm^{-1})	Matching bands
3689	Vibration of elongation of the external hydroxyl (OH) bonds of the Kaolinite network (Al-OH-Al)
3649	Valence vibration of the OH group composing the elementary cell kaolinite
3619	Vibration of elongation of hydroxyl bonds (OH) materializing the presence of kaolinite
1114	Vibration of the Si-O bond of kaolinite.
1027	Deformation vibration of the Al-O-Al bonds of kaolinite
1004	Vibration d'allongement de la liaison Si-O de la Kaolinite
912	Kaolinite Si-O bond elongation vibration
795	Vibration characteristics of the SiO$_4$ group of Quartz
750	vibration of the Si-O-Al bond of kaolinite
676	Vibration characteristic of the SiO$_4$ group of Kaolinite and Quartz
645	

Quality test on the ceramics obtained

Quality control tests are performed to evaluate certain properties such as bulk density, bending strength, compressive strength, linear shrinkage, loss of mass and water absorption rate. Table 6 below summarizes all these properties.

Table 6: Physical and Mechanical Properties of the Cooking Products

Technologicalparameters	Temperature		
	950°C	1000°C	1100°C
Linear withdrawal (%)	1.5±0.18	2.5±0.09	10±1.14
Mass loss (%)	7,76±0.91	7,83±1.43	8,04±0.98
Mass density (g/cm^3)	1,87±0.21	1,92±0.07	2 ,06±0.08
Water absorption (%)	14,06±1.56	12,24±0.98	6,38±0.85

Flexural Strength (MPa)	6,34±0.29	8,02±1.08	13,76±1.00
Resistance to compression (MPa)	19,23±1.53	21,53±1.98	37,44±2.45

Color, cohesion and sound

Figure 10shows the color of the ceramic obtained as a function of the cooking temperature. At 950 and 1000°C the products are of a light beige color while at 1100°C the color is a slightly dark beige. The sonority of the ceramics was examined by lightly tapping the baked test pieces on the laboratory bench and noting the nature of the sound obtained; as a result, it was found that the sound for the sample at 950°C was slightly metallic while at 1000 and 1100°C, the sound produced by the test pieces was completely metallic. The sound quality of the test specimens reflects the baking maturity of the ceramic. For the cohesion, it was evaluated to the touch, it was question to examine the capacity of the material to crumble or not to the touch and force was to note that the specimens had a good cohesion whatever the temperature of cooking.
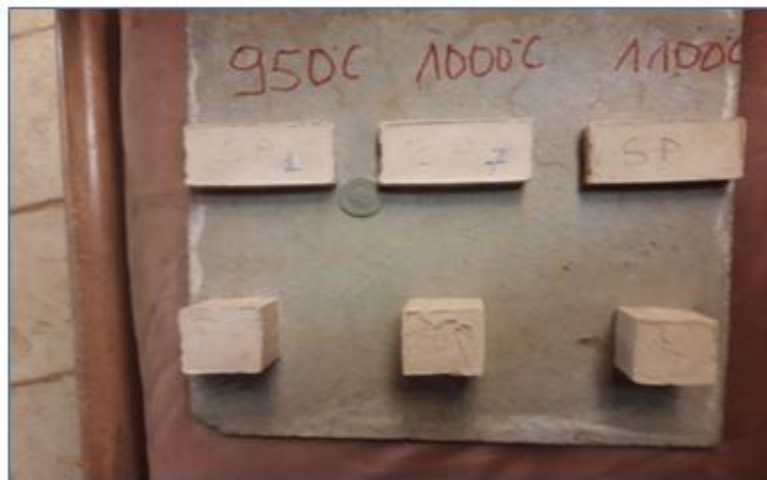

Figure 10: Evolution of the color of the test pieces cooked according to the temperature

Results Interpretation

Mineralogy

On the mineralogical plane, we find in this material kaolinite whose presence could be explained by an in situ alteration of rocks rich in silico-aluminous minerals by meteorite waters and surface

waters in a hot and humid environment [3]. Such deposits are important along fracture and grinding zones that favor the penetration of water into great depths of the bedrock. Kaolinite can also come from a monossialitic alteration that affects feldspars [29]. The presence of illite in our clay material could be linked to a hydrolytically dominant weathering climate that affects feldspathic minerals and may also affect newly formed minerals such as kaolinite. The partial leaching of silica and basic cations (K^+, Ca^+) also leads to the construction of silicates of type 2: 1 or T-O-T with the example of illite. We speak of bissialitic alteration [9]. Quartz is a mineral that strongly resists chemical alteration processes. Its presence is thus linked to a detrital input, probably a mechanical erosion of the source rock [30]. Feldspars are minerals from the source rock. Their presence is due to the mechanical alteration of the bedrock accompanied by a more or less short transport [58]. In addition, the feldspars have a notable ceramic importance because they act as fluxes and constitute the sanding flow at low temperature [2]. The minerals found in the argillaceous material of Sangaré-Paul are by comparison the same minerals found in predominantly kaolinitic formations. [31]. Mullite, a new crystalline phase obtained after sintering, is a mineral that forms at high temperatures and low pressures. These conditions were met in the atmosphere of the baking oven. It is a mineral found in ceramics from kaolino-illitic clay materials and in refractory ceramics because of its high pyroscopic resistance [23].

The Atterberg limits and the plasticity index

The plasticity of a clay is mainly ensured by its high content of clay and loam particles [15]. The clay material can hold up to 30.63% of water without sinking under its own weight. It can deform plastically when it contains 9.21% of water. It is a little plastic clay, it will not require a lot of water to form a paste of consistency called "normal" and will support inert bodies (degreasing) while retaining an appreciable processability and mechanical strength dry enough to ensure manufacture [32]. Its plasticity index is 21.42 %. Compared to the reference clays used in industrial and traditional ceramics, the plasticity index of our clay material is certainly high ($\geq$ 20%) but still remains appreciable because it is recommended a plasticity index between 9 and 20% [32] for the manufacture of terracotta bricks [32].

Physical properties of ceramics

Color, cohesion and sound of ceramic products

The metallic sound for a ceramic product reflects the good performance of the ceramic and the achievement of the baking maturity of the clay material because at this stage there is a significant presence of the glassy phase [15]. This is the manifestation of transformation reactions during sintering and the presence of oxides which have reached their melting point. On this basis, the material is suitable for industrial ceramics (bricks, tiles) [3]. The cohesion of the cooked

materials improving with the cooking temperature is the characteristic of the progressive vitrification of the materials with the temperature. It should be noted that the presence of quartz tends to reduce the cohesion of the materials because its grains behave like defects in the matrix of the ceramic products [2]. The beige color of the test specimens would probably be due to the red-white mixture provided respectively by the presence of a small amount of iron oxide [3] and alumina in the natural material. These oxides can be quantitatively identified through chemical analysis. The baking products of the Sangaré-Paul clay material still have very important positive commercial dyes for use in the ceramic industry [33].

Linear shrinkage during cooking

This is a parameter that reflects the reactivity of the clay during cooking. The linear shrinkage during firing is due to the gradual disappearance of the porosity by the sintering process leading to consolidation of the granular assembly with formation of solid phases bound by liquid phases. All in all, these steps lead to the densification of elaborate materials [4]. The shrinkage variation during firing depends mainly on the degree of sintering and the initial porosity of the raw clay [34]. The shrinkage values for our material are increasing. Between 950 and 1000 ° C the shrinkage is not high: sintering is present but not important. At 1100°C, the particles are finer with the presence of neoformed crystals; in addition, the viscous melt flow coalesces the particles and contributes to the formation of the vitreous phase, which considerably shrinks the ceramic material [18]. This explains why a relatively high linear shrinkage (10%) is obtained at this temperature. It is therefore not favorable for the manufacture of bricks of good quality at this temperature. This problem can be corrected by adding degreasers or any other non-hydraulic substance (sand, feldspars) [30]. Additional laboratory tests will be needed to determine optimal proportions.

Bulk density

The sintering of clay materials leads to the reorganization of the matrix of the material during the appearance of the glassy phases with increasing kinetics with temperature [2]. This crystalline reorganization is done through solid-solid reactions and interactions between minerals that change structure. The increase with sintering of the bulk density from 1.87g /cm 3 (950°C) to 2.06 g/cm 3 (1100°C) is due to a consolidation of the grains of the green material and a decrease in pore volume. Furthermore, the apparent density required for use in the field of traditional ceramics must not exceed 2.2g/cm3 [35], a value which remains higher than the different mass densities of the ceramics obtained from the clay material.

Loss of mase during cooking

The loss of mass during cooking makes it possible to know the quantity of the products likely to decompose or to volatilize during cooking. It is due to the release of CO_2 by removal of carbonates, dehydroxylation of clays and decomposition of organic matter [36]. For the Sangaré-Paul clay material, it is noted that the loss in weight is approximately constant and around 7.5% regardless of the temperature of the experimentally cooked products manufactured. A loss on ignition of less than 10% is interesting for the profitability of a possible exploitation [3].

Water absorption rate

In general, it is assumed that the water absorption rate of ceramic materials should decrease with the sintering temperature [34]. This is due to the decrease in the open porosity available to accommodate the immersion water and the increase in the rate of greyening with temperature. We also note that the large sandy fraction of the Sangaré-Paul clay material has the effect of slightly lowering the percentage of water absorption at low temperature by causing the rupture of capillary currents [2]. Between 950 and 1000°C, values of the rate of absorption of water higher than 12% are noted. From 1000 to 1100°C there is a sharp decrease (6%) of water absorption, which shows that the densification at this temperature is quite important compared to other sintering temperatures. All these values are still acceptable for use in the manufacture of building materials including clay bricks because they remain well below the recommended values of 25%. The tiles can also be made at 1100°C because the recommended value is 9 etand [37]. However, the value of the water absorption rate should not be too low because the texture of a terracotta product plays a key role in adhering to a cement mortar or plaster [38].

Mechanical compressive and flexural strengths.

A determining element of the mechanical properties of the ceramic matrix is porosity [39]. The evolution of the rate of water absorption has allowed us to observe a decrease in open porosity. The approach models linking the porosity to the mechanical properties of the materials are empirical, but in general, an increase in the pore volume fraction implies a decrease in the mechanical properties [40]. The mechanical compressive strength of the ceramic obtained from the Sangaré-Paul clay material increases with the sintering temperature rising from 19.23 MPa to 38 MPa at 1100°C. This could be explained by the decrease in the pore volume because of the sintering which consolidates the material and promotes the densification thereof. At 1100°C, the mechanical strength obtained (38 MPa) is fairly close to or comparable to the mechanical strengths (30-50 MPa) developed by ordinary concrete [3]. At this temperature, the toughness of the material is significant and constitutes an important argument as to the potentiality of the use of ceramics from this clay material in the field of building materials. As regards the mechanical resistance to bending, it should be remembered that it increases strongly with the vitrification of the material after firing, to a point where the vitreous phase becomes very important and the

ceramic material becomes brittle [4]. The constituents of so-called fusible or so-called fluxing samples: alkalis and alkaline earths play an important role during sintering. These constituents melt at low temperature and will form a liquid phase which will react with the solid phases including mullite. This reaction is responsible for improving the mechanical performance of the material [41]. On cooling, densified products are obtained, the elements of which are bound by a vitreous phase that is more or less important depending on the sintering temperature. This, combined with the decreased porosity of the fired materials, explains why the mechanical strength of our fired ceramic specimen increases with the temperature from 6.34 MPa (950°C) to 13.76 MPa (1100 °C). It should also be noted that the recommended bending resistance values are for dense bricks $\sigma \geq 2$ MPa, for ceramic blocks $\sigma \geq 5.5$ MPa and for tiles $\sigma \geq 6.5$ MPa [37]. It is found that at a firing temperature of 1100°C, the clay material of Sangaré-Paul on the basis of its mechanical resistance to bending is suitable for the manufacture of ceramic blocks, tiles and dense bricks.

Determination of the ideal firing temperature of the clay material of Sangaré-Paul

To obtain bricks and even tiles of good quality as suggested by the diagram of Wrinkler and Niesper [31], from the clay material of Sangaré-Paul, it is essential to bake it at a temperature which includes the most satisfactions from the point of view of technological parameters. These parameters are among others the loss of mass on firing, linear shrinkage on firing, the water absorption rate, the mechanical resistance to compression and to bending. Thanks to the graph presented in figure 11it can be seen that of the three experimental temperatures, the optimum cooking temperature is 1100°C. Because it is a good compromise for all the technological parameters mentioned. It avoids a ratewater absorption greater than 10%, to have maximum mechanical performance, loss ofof mass during conventional cooking and a linear shrinkage during cooking which is consistent but adjustable when formulating the raw dough.
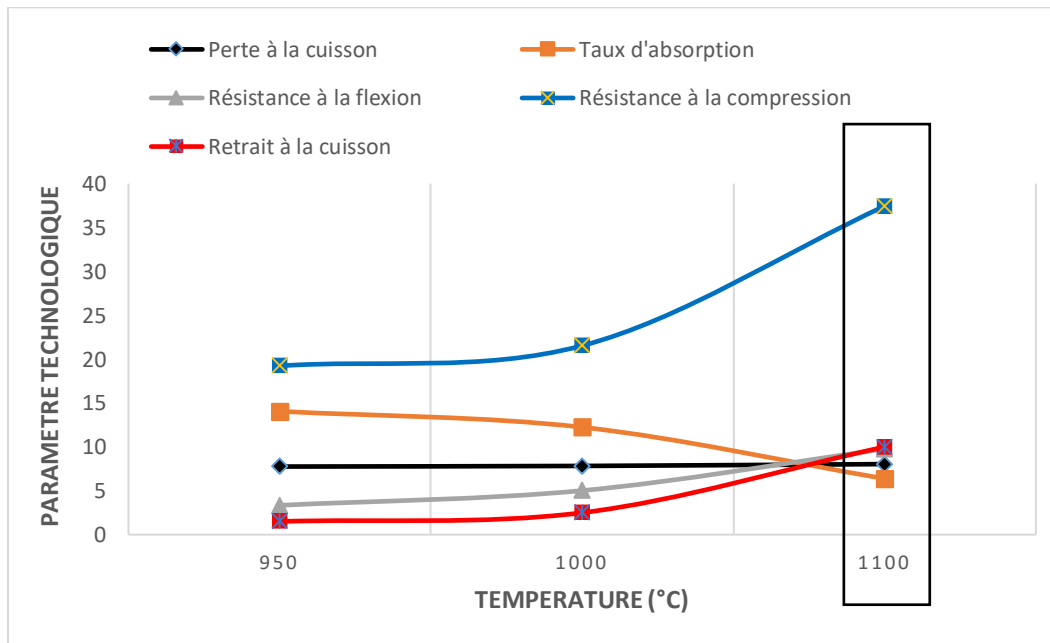
Figure 11: Determining the ideal cooking temperature

The previous results made it possible to identify the clay material of Sangarégeo technically and to highlight the mineralogical composition of the material in its natural state and when it is transformed into ceramic. The investigation of technological parameters tends to describe the physico-chemical phenomena occurring during sintering. The values thus obtained, when compared with those recommended, made it possible to attribute to this clay material a viable application in the manufacture of fired products.

CONCLUSION

From a development perspective, this work has set itself the objective of carrying out the physical and mineralogical characterization of the clay material in order to evaluate the potential of its use in the ceramics sector. It presents a double interest, scientific and industrial. It contributes to the promotion of local materials and the development of the region. Developed under the alteration mantle of the sandstone rock very present in Garoua and covered with a lateritic horizon, the Sangaré-Paul clay material has a very wide particle size distribution with a very high coefficient of uniformity, an activity less than 0, 75 and a Ph = 8. This clay material is not plastic. Mineralogical analyzes show that it consists of quartz, potassium feldspar and the main clay minerals present are kaolinite and illite. In short, it is a sandy kaolinino-illitic clay with a little silt and traces of gravel. The mineralogical composition of the fired material shows the disappearance of the starting clay minerals to give way to a new crystalline phase: mullite. It is

accompanied by silica and potassium feldspars. Physical tests carried out on the products baked respectively at 950, 1000 and 1100 ° C, it is found that the color is beige and it darkens slightly with the increase in the cooking temperature. The sound is overall metallic. The loss of mass during cooking is average and does not exceed 8% for all temperatures tested. The rate of water absorption decreases with the evolution of the temperature to stabilize at 6%; This reflects a decrease in open porosity as the cooking temperature increases. This decrease in porosity results in an increase in the mechanical performance of cooked briquettes which go from 3.4 to 9.8 MPa for the mechanical resistance to bending and from 19 to 38 MPa for the mechanical resistance to compression. It should also be noted that at the temperature of 1100 ° C, the maximum linear shrinkage of 10% found is quite important but remains adjustable by the addition to the raw dough degreasers. The scientific knowledge acquired on natural materials and on the manufactured cooked test pieces make it possible to justify the use of Sangaré-Paul clay materials in the coarse die of ceramic or ceramic construction. Indeed the sintering observed at 1100 ° C shows the sizzling and fuse nature of the natural material. In addition, the ideal particle size distribution for the manufacture of the fired bricks, the mechanical performance comparable to that of certain concretes and tiles, the low mass loss and the rate of water absorption in accordance with the brick production criterion show the potential for use of this clay material in coarse ceramics. It emerges from the superimposition of these results that the clay material of Sangaré-Paul is suitable for the manufacture of bricks and terracotta tiles at a temperature of 1100°C. In order to complete this work, future studies may focus on the quantitative estimation of the natural resource, the chemical analysis of the material by X-ray fluorescence spectrometry to identify the different oxides present in this material as well as their proportions. Thermo differential analysis (TDA) and thermo gravimetric analysis (TGA) can also be used to better understand heat-induced transformations that affect the performance of fired products obtained from this clay material. In order to improve the low temperature properties of the fired materials, the influence of an addition to the raw paste of low temperature melting and sanding materials with the example of feldspars will also have to be studied in order to save energy.

ACKNOWLEDGEMENTS

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