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## Mineralogy and Thermal Behaviour of the Kaolinitic Clay of Loutété (Congo-Brazzaville)

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**Abstract:** The mineralogical and chemical composition and the technological properties of the raw material used by craftsmen of Loutete, locality in the south of Congo republic, for manufacturing fired bricks were characterized. The X-ray Diffraction, IR spectroscopy, ATD, ATG and AEG were used. The Atterberg limits and the particle size distribution were measured. The linear shrinkage, the water absorption, open porosity, apparent density and flexural strength of different bodies at temperatures from 800 to 1150°C were measured. The diffraction patterns of different bodies were registered. Kaolinite is the major clay mineral in this sample. The Winkler diagram and clay workability chart allowed to consider this material convenient for roof tiles manufacturing. The open porosity and the chemical composition are close to that of stoneware. The addition of sintering agent and shortening material are necessary for improving the technological properties in stoneware, floor and wall tiles.

**Key words:** Kaolinitic clay, mineral composition, physical properties, thermal behavior, Winkler diagram, workability chart

### INTRODUCTION

Most of the usual clay ceramics are materials containing alumina silicates, more or less complex, obtained from natural clayey raw materials. The clayey resources usable in Congo are indexed and indicated in Fig. 1 (Plan quinquennal, 1980).

Many brick-yards were worked in the colonial period by the expatriates and provided materials for the construction of many public buildings. These brick-yards are now given up in spite of the autochthones craftsmen attempts to continue this activity. The random success of their undertakings constitutes one of the causes of the abandonment of this activity and thus the ceramic industry is now declining.

The important presence of baked bricks, sanitary, floor and wall tiles and earthen wares on the Congolese market expresses the interest which the Congolese population grants to the products of ceramic industry. Though the consumption of these products is increasing, their local production remains very low. Notwithstanding the large quantities of available clay raw materials, the lack of knowledge about the volume and nature of clay materials in the deposits, as well as the misunderstanding

of their chemical and mineralogical composition, limits their promotion in ceramic industry.

In the locality of Loutete, department of Bouenza, in the South of Congo, at the west of Brazzaville, the populations are incited to produce fired clay bricks in artisanal furnaces. However, the resulting products show uncontrolled variations of the quality, due to the lack of rational investigations on used raw materials and manufacturing processes.

Then, this study aimed at characterizing the raw material used by the craftsmen of Loutete and studying the influence of the manufacturing conditions on their technological properties. Improvements of the preparation and firing conditions of the test samples are expected.

### MATERIALS AND EXPERIMENTAL METHODS

**Mineralogical and chemical analysis:** The clayed material from the deposit used by the craftsmen in the locality of Loutété are located between longitudes 13°50'-13°51' and latitudes 4°19'-4°21', in the department of Bouenza in the south-west of Congo (Brazzaville) (Fig. 2).

Sampling has been carried out at a depth between 2 and 3 m. The valley of Niari in which the department

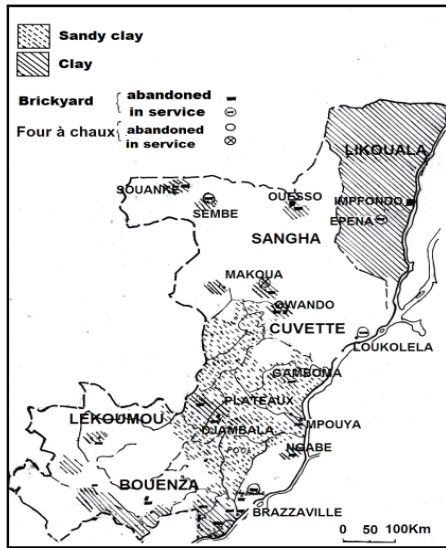


Fig. 1: Building material resources (Plan quinquennal, 1980, Ministère du Plan, Congo)

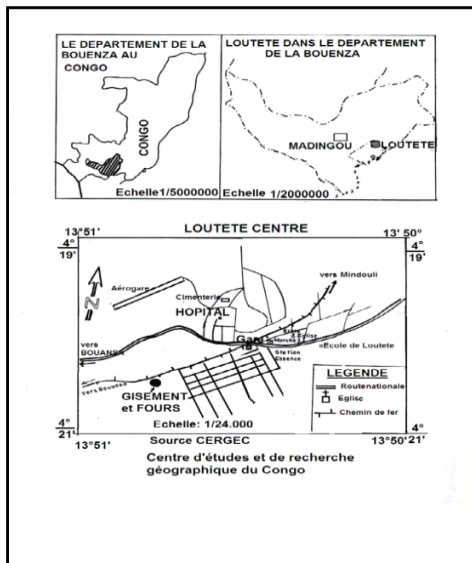


Fig. 2: Localization of the locality and the deposit of Loutété

of Bouenza is situated, presents soils, the mother rock of which consists in schisto-limestones represented by pink and grey dolomites, clayey limestones, built limestones and crystalline limestones, stony sands, sandy and siliceous limestones and finally by grey dolomites on oolitic levels (Martin, 1970).

The particle-size analysis was performed by wet sieving for the  $>80 \mu\text{m}$  fraction and by gravity sedimentation for the  $<2 \mu\text{m}$  fraction using the Stokes law (AFNOR, 1984).

The plasticity was measured by the Atterberg indices: Liquid Limit (LL), Plastic Limit (PL) and Plastic Index

(PI) according to the norms NF P 94-051 NF P 94-052 (1982).

The XRD of sample powder was performed using a Philips model diffractometer operating by reflexion under the  $\text{Cu-K}\alpha$  radiation for  $2\theta$  angle ranging from  $5^\circ$  to  $60^\circ$ . Diffuse Reflectance Infra-red Fourier Transform Spectroscopy (DRIFTS) was performed over a wave number domain between 600 and 4000 per cm using a Bruker IFS 55 spectrometer equipped with a broad band detector of the type MCT (Mercury and Cadmium Tellurium) cooled with 77K and with an accessory of diffuse reflexion (Harrick Corporation). The powdered sample was diluted in KBr (50 mg of sample in 350 mg of KBr). The spectra were recorded by accumulating 200 scans at 2.0 per cm resolution.

The spectra IR obtained in transmission mode were recorded with an infra-red spectrometer with Fourier Transform BRUKER IFS 55 equipped with a detector DTGS (sulphate of deuterated triglycine), a spectral resolution of 2 per cm and a time of measurement of approximately 1mn30. The pastille is carried out by mixing approximately 0.5% weight of sample in potassium bromide (KBr) preserved in a drying oven at  $80^\circ\text{C}$ . The mixture is closely crushed in an agate mortar then deposited in a mould to pelletize 13 mm in diameter which one places in a press to pelletize. The pressure applied is of  $10 \text{ T/cm}^2$ . A spectrum IR (mode of transmission) was obtained at room temperature and after heating in the drying oven with  $80^\circ\text{C}$ .

For the transmission mode (TIFTS), the dilution was 0.75 mg of sample in 150 mg of KBr. The spectra were collected by accumulating 64 scans at a 2.0 per cm resolution.

The Differential Thermal Analysis (DTA) and Thermo Gravimetry (TGA) associated with the Analysis of the Evolved Gas (AEG) were carried out with a device coupled with a thermobalance and a mass spectrometer THERMOSTAR. The speed of heating was  $10^\circ\text{C/mn}$ .

The chemical analysis of the major elements was carried out according to Carignan *et al.* (2001) procedure.

### Technological properties:

**Preparation and firing of the test samples:** 10 g and 45 g of clay powder were mixed respectively with 10 and 5 mL of distilled water in a porcelain mortar and then put respectively in cylindrical and parallelepipedic steel mould. An axial vertical pressure of 342 MPa was applied to obtain two types of test specimens:

- Discs of 4 mm diameter and 6 mm in thickness in order to measure the physical properties (open porosity, gross density and water absorption)
- Parallelepipedic test specimens for the determination of linear firing shrinkage and flexural strength

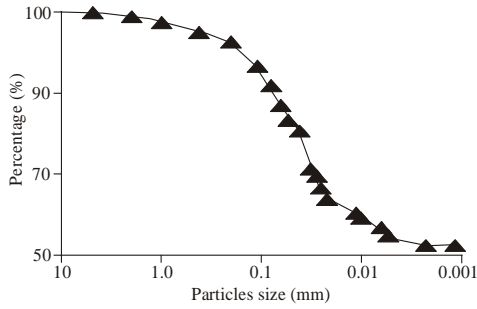


Fig. 3: Particle size distribution de LOU

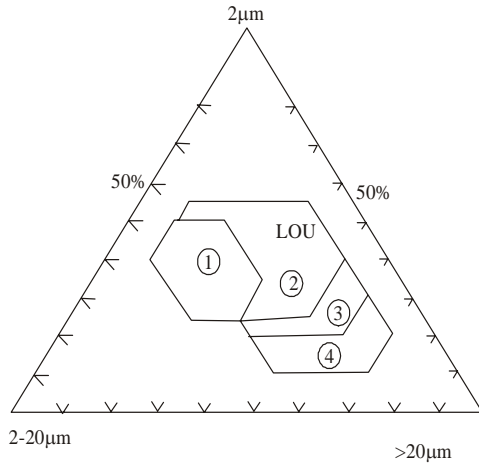


Fig. 4: Positioning of LOU sample in Winkler diagram

The test specimens obtained were first dried at 110°C for 24 h and then fired at 800°C, 850°C, 1000°C, 1050°C, 1100°C and 1150°C in a NABERTHERM model kiln with a heating rate of 5° C/mn and a 2 h evaluation time.

**Determinations of technological properties:** The linear shrinkage, open porosity, bulk density and water absorption were measured according to the European Refractories norm (Aliprandi, 1979) and the protocol P 18-554 of the French norms (AFNOR, 1984). The flexural strength was determined by the three points method (Aliprandi, 1979; Houmba, 1987) with an electro-hydraulic press (M&O, type 11.50 N°21).

**RESULTS AND DISCUSSION**

Figure 3 presents the curve of particle size distribution for the investigated clays.

In this curve, it can be observed that the <2 µm fraction represents 52% of the sample, whereas the 2-20 µm and >20 µm fractions represent respectively 27% and 21%. Argillaceous texture is thus allotted to this sample by using the triangle of texture of Soil Survey Manual (United States Department of Agriculture, 1993). The

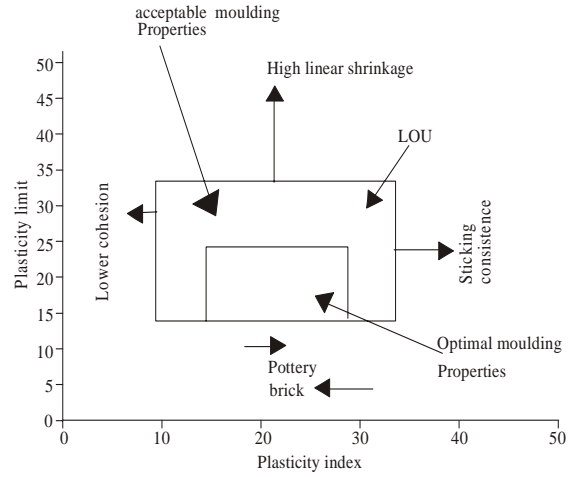


Fig. 5: LOU sample in clay workability chart (Bain and Highly, 1978)

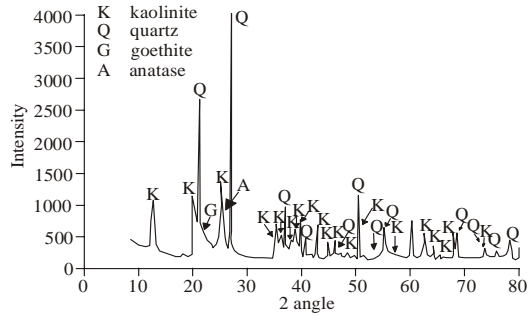


Fig. 6: XRD pattern of LOU sample

particle size distribution of this clay is typical of raw materials for roof tiles, considering the Winkler diagram (Fig. 4) (Capitâneo *et al.*, 2005; Dondi *et al.*, 1998; Monteiro and Vieira, 2004).

The Liquidity Limit (LL), Plasticity Limit (PL) and Plasticity Index (PI) are respectively 62.2, 31.5 and 30.8. In fact, the high value of PI (30,8) allows to characterize this clay as highly plastic according to the Casagrande abacus. The plasticity limit value of this sample is in the domain (17.2 to 32%) indicated to red ceramic, as well as that for extrusion process (26 to 32%) (Teixera *et al.*, 2001). The clay workability chart (Bain and Highly, 1978) (Fig. 5) allows to consider that the Loutete clay possesses acceptable moulding properties. A high linear shrinkage is expected. This raw material is not optimal for pottery and bricks.

The X-ray diffraction pattern of this material reveals the presence of kaolinite, quartz and anatase (Fig. 6).

The analysis of this diffractogram makes it possible to identify the following mineral species: kaolinite, quartz, anatase, and goethite. The broad bump of low intensity around 18° (2q) indicates the presence of smectites. This presence would justify rather high

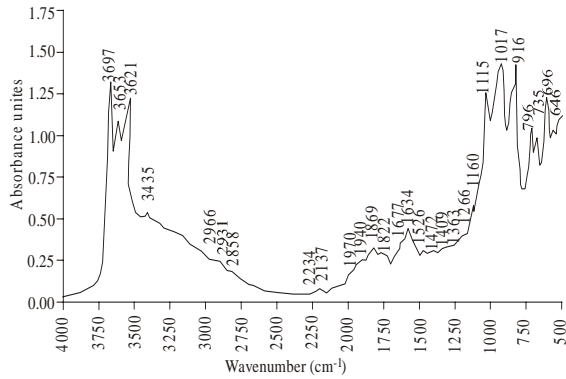


Fig. 7: FTIR spectrum for LOU (diffuse reflectance)

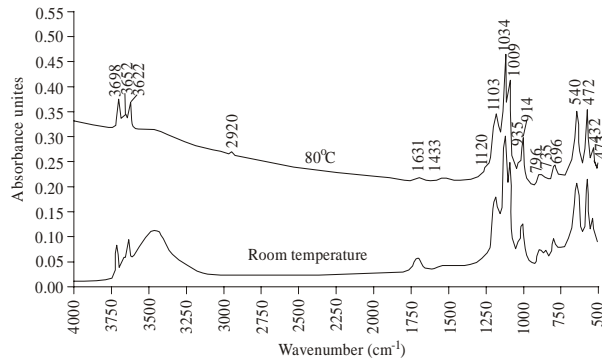


Fig. 8: FTIR spectrum for LOU (transmission mode)

plasticity and the strong awaited linear withdrawal. The sequences of the 021, 111 reflections in the range 20°-30° (2q) and of the 201, 131 in the range 35°-40° (2q) show a poor resolution compared to diffractograms of ideal kaolinites what indicates that the present kaolinite is disordered. (Plançon and Tchoubar, 1975).

Figure 7 and 8, respectively give us spectrum DRIFTF and the spectra in transmission mode at ordinary temperature and after heating at 80°C.

The DRIFTS spectrum shows peaks at 3697, 3653, 3621/cm (lattice O-H stretching), 1115-1017 per cm (Si-O stretching) 938-916 per cm (OH deformation) in kaolinite (Saika and al. 2003, Van Olphen and Fripiat, 1979). The intense bands at 3420 and 1631 per cm are due to the molecular water molecules. (Lappi *et al.*, 2004). In TFTIS spectrum at 80°C, the intensities of these bands highly decrease (Hajjaji *et al.*, 2001).

The peaks between 797 and 753 per cm is characteristic of quartz (Hajjaji *et al.*, 2001). The well defined band at 3435 per cm in DRIFTS reveals small amounts of gibbsite. The presence of organic matter is supported by the slightly intense bands at 2931 and 2820 per cm. The disordered character of the kaolinite is confirmed by the profile of the proton elongation bands. In addition, the small shoulder centred on 3600 per cm is

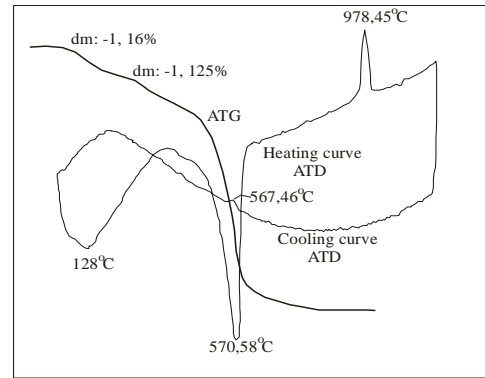


Fig. 9: ATD-TG curves of LOU sample

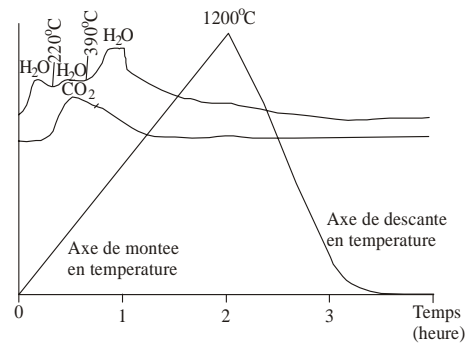


Fig. 10: AEG curve of LOU sample

typical of octahedral ferric iron environment (Delineau *et al.*, 1994).

The band to 646 per cm expresses the presence of the goethite (Wilson, 1996). The carbonates indicated by the very weak bands to 1479 and 1409 per cm are certainly in very weak amounts. (Van Olphen and Fripiat, 1979).

The curves of differential and thermogravimetric thermal analysis are given in Fig. 9

The curve relating to the analysis of the evolved gases (AEG) associated with the thermal analysis is given in Fig. 10.

The following observations are made:

- An endothermic peak of which the maximum is at 130°C (during the heating in DTA) and which corresponds to a loss of mass of 1,16% (in TGA) and water release (in AEG)
- An exothermic bump between 226 and 400°C (during the heating in DTA) which is associated with a carbon dioxide and water release (in AEG) and a loss of mass of 1,125% (in TGA)
- An endothermic peak at 571°C (in DTA), accompanied by a loss of mass of 7.83% between 400 and 1200°C ; in this interval of temperature, the AEG reveals a water release

Table I: Chemical composition of LOU sample

	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>	P.F.
LOU	59	23.99	3.11	0.01	0.27	0.11	<L.D.	0.67	1.28	0.12	11.36 99.91

Table II: Bodies colours at various temperatures (Munsell soil color charts 1975)

	800°C	850°C	1000°C	1050°C	1100°C	1150°C
Colour	Pink	Pink	Pink	Pink	Pink	Very pale brown
Munsell code	5YR 7/4	5YR 7/4	5YR 8/3	5YR 8/3	7,5YR 8/4	10YR 8/4

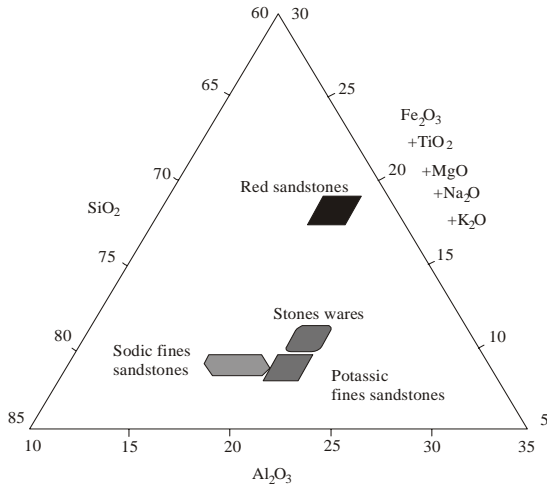


Fig. 11: Chemical composition field of wall or floor tiles

- An exothermic peak at 979°C : It corresponds to the métakaolinite-mullite transformation, and this temperature is very close to that indicated in the case of low contents iron oxides (980°C) (Soro, 2003).

These observations characterize the presence of kaolinite (Mackenzie, 1957; Bouaziz and Rollet, 1972; Jouenne, 2001).

The water departure between 220 and 390°C could be attributed to the presence of goethite and/or gibbsite (Mackenzie, 1957).

During cooling, the DTA curve shows an exothermic peak to 568°C corresponding to the quartz β-quartz α reversible transition and therefore confirms the presence of quartz. This transition has not been observed during the heating translating a relatively low presence of quartz (<30% sample) (Pialy, 2009).

The organic matter in this sample is revealed by the CO<sub>2</sub> signal. By considering the very low loss of mass observed between 220 and 390°C, the contents of goethite, gibbsite and organic matter would be certainly very low in this sample. The depth beyond two meters could explain that. The chemical analysis of this clay is provided in Table 1.

Figure 11 (Boch, 2001) provides the field of chemical composition of various types of stoneware wall or floor tiles.

It is indicated there that SiO<sub>2</sub> must be ranging between 60 to 85%, Al<sub>2</sub>O<sub>3</sub> from 10 to 35% and the oxides sum (Fe<sub>2</sub>O<sub>3</sub> + TiO<sub>2</sub> + MgO + CaO + K<sub>2</sub>O) varying from 5 to 30%. By considering the sum of the oxides contents (Fe<sub>2</sub>O<sub>3</sub> + TiO<sub>2</sub> + MgO + CaO + K<sub>2</sub>O) (5.44%), the content of Al<sub>2</sub>O<sub>3</sub> (23.99%) and that of SiO<sub>2</sub> (59.00%) we are practically in the field of chemical composition of stoneware tiles.

The kaolinite content deduced from chemical analysis is 55% by using the follow formula (Njopwouo, 1984; Yvon *et al.*, 1990).

$$T(a) = \sum_{i=1}^n M_i \cdot P_i(a)$$

with:

T(a) = Mass percent of the element oxyde “ a “ in the sample

M<sub>i</sub> = Mass percent of the mineral “ i “ in the studied material

P<sub>i</sub>(a) = Massic amount of the oxyde of the “ a “ element in the mineral “ i “ deduced from the ideal formula of the minéral “ i “

Calculating the kaolinite content from the loss of mass at 570°C (TGA), leads to 56%.

We can think that the <2 μm fraction could be more important. Then this clay would be over the roof tiles domain in the Winkler diagram (Dondi *et al.*, 1998).

Though the particle size distribution ranges in the roof tiles domain in the Winkler diagram, some bodies having this particle size distribution are used for floor tiles (Capitâneo *et al.*, 2005).

Table II shows the bodies colours at firing temperatures.

These observations correspond to with the presence of colouring oxides (Fe<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>) (Djangang, 2007). These colours distinguish from the red colour relating to an important presence of colouring oxides. Indeed, the global percentage (TiO<sub>2</sub>+Fe<sub>2</sub>O<sub>3</sub>) is not high.

The X-ray diffraction patterns of fired specimens are shows in the Fig. 12.

The crystalline phases appearing in bodies are:

- At 800°C: quartz, anatase and rutile already detected in the X-ray diffractogram of the raw material, illite and/or dehydroxylated smectite revealed by the peaks of low intensity at 10,07 Å and 4,47 Å were not



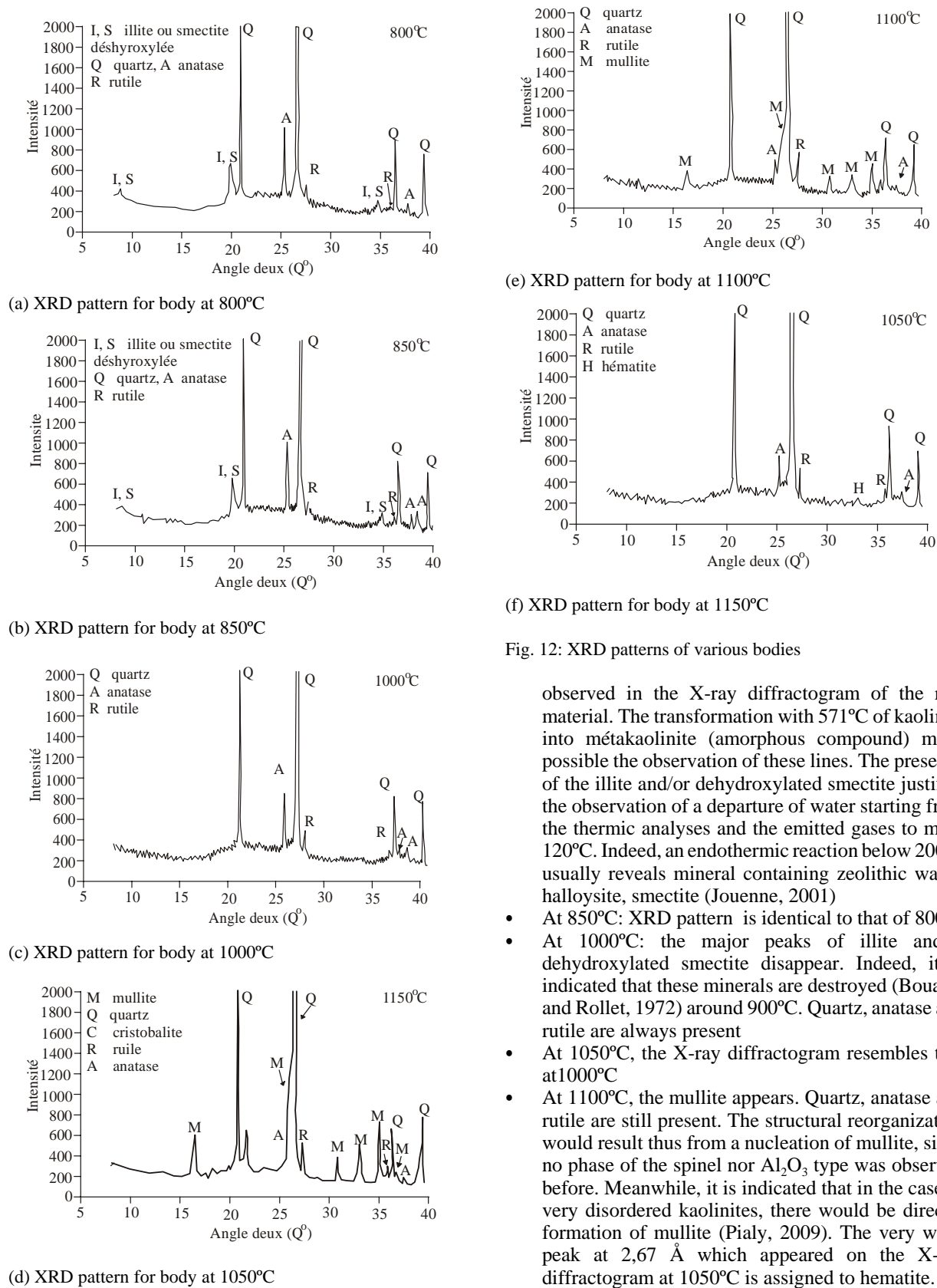


Fig. 12: XRD patterns of various bodies

observed in the X-ray diffractogram of the raw material. The transformation with 571°C of kaolinite into métakaolinite (amorphous compound) made possible the observation of these lines. The presence of the illite and/or dehydroxylated smectite justifies the observation of a departure of water starting from the thermic analyses and the emitted gases to more 120°C. Indeed, an endothermic reaction below 200°C usually reveals mineral containing zeolitic water: halloysite, smectite (Jouenne, 2001)

- At 850°C: XRD pattern is identical to that of 800°C
- At 1000°C: the major peaks of illite and/or dehydroxylated smectite disappear. Indeed, it is indicated that these minerals are destroyed (Bouaziz and Rollet, 1972) around 900°C. Quartz, anatase and rutile are always present
- At 1050°C, the X-ray diffractogram resembles that at 1000°C
- At 1100°C, the mullite appears. Quartz, anatase and rutile are still present. The structural reorganization would result thus from a nucleation of mullite, since no phase of the spinel nor  $Al_2O_3$  type was observed before. Meanwhile, it is indicated that in the case of very disordered kaolinites, there would be directly formation of mullite (Pialy, 2009). The very weak peak at 2,67 Å which appeared on the X-ray diffractogram at 1050°C is assigned to hematite.

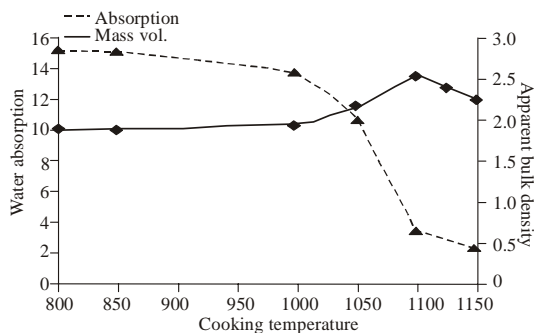


Fig. 13: Evolution of water absorption and apparent density with temperature

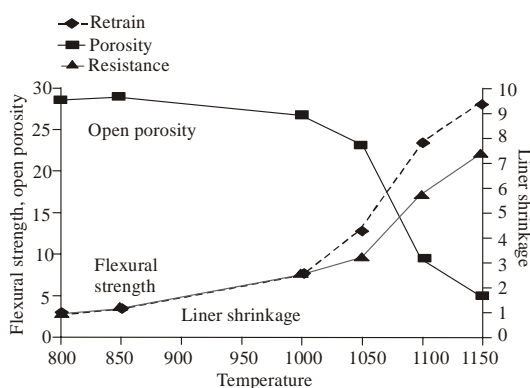


Fig. 14: Evolution of flexural strength, open porosity and linear shrinkage with temperature

At 1150°C the height of the peaks of mullite grows. This observation is in agreement with the raising of the right base of the exothermic peak which translates the continuation of the germination of mullite (Jouenne, 2001). Moreover, the appearance of the peak at 4,08Å indicates the presence of cristobalite. Indeed, it is well known that during the firing of a clay material to high temperatures, the quartz can be transformed into cristobalite or dissolved in a vitreous phase. (Aliprandi, 1979). The abundance of a vitreous phase likely to dissolve quartz is showed by the reduction of the quartz lines height at 4,26 and 3,34 Å. (Elimbi *et al.*, 2004). However, we cannot conclude that this line decreases in spectra X of bodies at least until 1150°C.

The Fig. 13 and 14 shows the temperature dependence curves for technological properties of this clay material.

Figure 14 Evolution of flexural strength, open porosity and linear shrinkage with temperature. These curves display a decrease in the water absorption and the open porosity values on the one hand, on the other hand an increase in firing shrinkage and flexural strength values, with increasing firing temperatures. They show two principal branches, taking in account the importance of the slope:

- The first branch with a weak slope from 850°C to 1000°C
- The second branch with a stronger slope from 1000°C to 1150°C

These strong variations of technological properties above 1000°C could be associated with a liquid phase formation. The presence of iron and potassium revealed by chemical analysis, though with low contents, indicates the presence of fusible matters that would produce a liquid phase likely to induce a low open porosity (Boch, 2001).

The weak slope can be related to a weak densification that would correspond to a sintering in solid phase in its first stage: a light reorganization by slip of grains occurs due to the defects of stacking of initial tests specimens. In absence of liquid phase, the rearrangement would not lead to a strong densification. Consequently, the firing shrinkage is still weak. From 1000 to 1150°C, the strong slope corresponds to a great variation of the linear shrinkage which would be the result of an intense densification. The appearance of a liquid phase is certainly the cause. Indeed, the formation of a liquid phase favours the slip of the particles on the others under the effect of the constraints existing within stacking. Then, the solubility of the solid increases at the contact points between particles. The transfer of matter followed by precipitation in the zones of weak energy allows the densification (Boch, 2001). The fact that at 1150°C the slope shows still a possible evolution of shrinkage let to think that the densification is not finished.

The open porosity at 1050°C is 23.17%. Porosity around 26% favours a better circulation of the liquid phase (Boch, 2001). The strong reduction of open porosity from 23.17% (1050°C) to 9.4% (1100°C) confirms the presence of a liquid phase which intensifies the gresification.

The value (5%) of open porosity reached at 1150°C indicates that the addition of sintering agent as feldspar is necessary, for the open porosity is ranging from 0 to 3% as indicated for ceramic stonewares (Boch, 2001).

The strong reduction of open porosity accompanied by the increase of flexural strength confirms the fact that the densification improves the mechanical strength (Monteiro and Vieira, 2004; Boch, 2001).

The water absorption evolution is in agreement with that of open porosity. According to the Brazilian norms, the recommended values for water absorption for extruded floor tiles are  $\leq 10\%$ . (Capitâneo *et al.*, 2005). With a value of 2.28% at 1150°C, the Loutete clay can be used for extruded floor tiles. Although weak at 1150°C, the water absorption does not reach to zero in accordance with the refractory behaviour of kaolinite (Monteiro and Vieira, 2004).

The value of firing shrinkage obtained at 1150°C is higher than that indicated in literature for floor and wall tiles (Swapan *et al.*, 2005). The controlling of shrinkage



requires the addition of shortening materials. The value reached at 1150°C (22.4 MPa) is higher than the usual recommended values for flexural strength [Brazilian Norms] for extruded floor tiles. The evolution of the apparent bulk density expresses a light fall with 1150°C. The presence of cristobalite could be the cause of it. Indeed, cristobalite and mullite becoming abundant in comparison with the height of their lines in spectrum X with 1150°C would cause an increase in ceramics which leads to the reduction in the apparent bulk density (Elimbi *et al.*, 2004).

### CONCLUSION

The characteristics and the influence of firing temperature on the technological properties of the clay from a deposit in Loutete (Congo Brazzaville) used by craftsmen for making fired solid bricks are studied.

The particle size distribution of this material offers potentialities of use in manufacturing roof tiles but excludes the possibility for this clay as raw material for solids bricks.

The high plasticity of this raw material is showed. It has the acceptable moulding properties and can be used in extrusion process.

The mineralogical composition of this Loutete clay has been carried out by different techniques (XRD, IR spectroscopy, ATD, ATG and AGE). This raw material presents kaolinite, quartz, anatase and rutile. The illite is observed only in XRD pattern at 800 and 850°C. The chemical composition very close to the composition of the stoneware wall or floor tiles can insert this clay in the production of ceramics which requires a vitrified shard. The presence of colouring oxides (Fe<sub>2</sub>O<sub>3</sub>, TiO<sub>2</sub>) indicates coloured ceramic bodies.

The XRD patterns of fired products reveal the appearance of the mullite at 1100°C and the cristobalite at 1150°C. No spinel phase precedes the presence of the mullite.

The technological properties have a weak variation from 850 to 1050°C, and a strong growth from 1050° to 1150°C. The sudden change at 1050°C is explained by the presence of liquid phase. The low content of alkaline oxides gives a little liquid phase that does not allow porosity below 3%. The presence with 1150°C of cristobalite would not support its use as aluminosilicate refractory. The addition of flux material (feldspar) and shortening material is necessary for improving the linear shrinkage and the porosity.

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