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The Characterization and Valorization of Clay Extracted from Ntokou Locality (Brazzaville, Republic of Congo)

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Abstract
The purpose of this study is to characterize a clay soil chemically, physically, mineralogically and technologically, taken from the locality of Ntokou, located near Makoua (Brazzaville, Republic of Congo). This clay is analyzed. The relevant revelations are made about its mineralogical composition determined by X-ray diffraction. The ternary diagrams, constructed on the basis of chemical and mineralogical compositions, are necessary and important for the production of ceramic products (which are still traditional until today), by improving the technological parameters (Atterberg limits, linear shrinkage, water absorption, porosity and flexural strength) of this clay. The technological parameters are measured from the temperature of 900 to 1150°C. The thermal behavior is studied from DTA/TGA curves. After cooking, collocated ceramic products are observed: this is confirmed by the diagrams of chemical and mineralogical compositions of Fiori. The clay soil of Ntokou can also be used as a complementary material in the manufacture of hollow ceramic products, according to Augustinik diagram.

Keywords: Ntokou clay soil, technological properties, to determine, use domain

INTRODUCTION

The use of clay resources made by man, mainly in the manufacture of building materials and pottery has been used since antiquity (Konta, 1995). The natural abundance and immediate availability of clays explain their great uses, over time.

Nowadays the use of clays, especially rich in SiO_2 and Al_2O_3, is experiencing a new boom in construction, ceramics and craftsman industry, pharmaceutical industry and pottery (Celik, 2010). In Republic of Congo, most ceramics (tiles, bidets, porcelains, etc.) made from clays are imported from foreign countries. This importation can be explained by the lack of a real ceramic industry which results from a lack of knowledge of clays in Congo. The use of these materials depends on, on the one hand, their mineralogical and chemical compositions. On the other hand, it depends on certain physical characteristics (granularity, plasticity, shrinkage and bending strength) and firing conditions (temperature, atmosphere and firing time) (Azzeddine and Abdelali, 2014). The projects which consist in

diversifying the Congolese economy by setting up ceramic production unities in Makoua and Maloukou by the Congolese government, those of cement factories in the southern part of the country and the development of a production of pottery products by craftsmen, open a wider field to the use of Congolese clays. The clay is the basic raw material for ceramic industry (Grim, 1960). In order to support this industrial development, Marien Ngouabi University, through these doctoral courses, has embarked on a vast program to study the physical and chemical properties of clays in Congo (Moutou *et al*., 2012; Moutou *et al*., 2018). These studies aim at identifying potential resources for the ceramic industry. The purpose of this work is to characterize a clay extracted from Ntokou locality which is located in the vicinity of Makoua town. Thus, the mineralogy of this clay soil is going to be determine by the use of several techniques (XRD, DTA/TGA, and Chemical Analysis). The geotechnical properties are going to be studied. The specific area is going to be measured. As clay, sintering generally takes place between temperatures 1000 and 1200°C and the characteristics of ceramic parts are more or less around these temperatures. We have chosen to determine the technological properties of Ntokou clay at 1150°C. We are going to estimate the use of this clay soil in ceramic industry.

MATERIALS AND METHODS

Location of Ntokou: Ntokou is a district of the department of Cuvette of which geographical coordinates are 0° 01' 35" North 16°20' 02" East. It is located on the right bank of Likouala-Mossaka River, 14 h by canoe from Makoua. By road, it is <40 km from Makoua. The sampling site is the Yombe one <1 km from Likouala-Mossaka River, precisely in a forest called Andziga. Material is collected from a 1.5 m deep well.

Characterization methods: The limits of Atterberg are measured according to standards NF P94-051 (AFNOR, 1993). The proportions of the following particles classes are determined according to standards NF P 94-056 (AFNOR, 1996) and NF P 94-057 (AFNOR, 1992). X-ray diffractogram is recorded at Ceramic Technology Transfer Centre (CTTC) in Limoges using a PANalytical X'Pert PRO brand diffractometer using the K wavelength of copper (Brindley and Brown, 1980). Chemical composition in major elements is determined in Petrographic and Geological Research Center (CRPG) at Nancy (France) (Carignan *et al*., 2001). The DTA/TGA of our sample is performed at Ceramic Technology Transfer Centre (CTTC) in Limoges (France) using a NIETZSCH STA 409°C Thermobalance. The temperature increase is carried out at a rate of 5°C/min. Maximum temperature is 1200°C. Cooling is freely depending on the inertia of furnace. MICROMERITICS TRISTAR II 3020 adsorption gas specific area analyzer - internal code: BET is used to record the nitrogen adsorption-desorption isotherm on Ntokou clay. The prior degassing of sample is carried out during night at room temperature.

Technological properties: Technological properties are determined at Ceramic Technology Transfer Centre (CTTC) in Limoges (France):

- **Specimens preparation:** Fifteen gram of clay are weighed with precision by using a balance, then it is introduced into a 90×12 mm steel mould, and it is pressed by using an ENERPAC brand press at force of 20 KN. 05 specimens are, therefore, realized. The approximate dimensions of specimens are as follows:

  90×12×7.3 mm (Length × Width × Height h)

  N.B: Specimens are not dry before cooking.
- **Cooking:** Specimens are cooked in a cell furnace with Kantal A1 quality resistances wound on ceramic tubes (T°max = 1250°C). Table 1 gives us thermal cycle programmed for test.

Shrinkage calculation: The dimensions of test pieces (Length L, width l, thickness e) are measured before cooking (Lo, lo, eo) and after cooking (Lc, lc, ec) using a caliper (to display accuracy of 0.01 mm). The shrinkage R is calculated as follows:

$$RL\ (\%) = \frac{Lo - Lc}{Lo} \times 100$$

$$Rl\ (\%) = \frac{lo - lc}{lo} \times 100$$

$$Re\ (\%) = \frac{eo - ec}{eo} \times 100$$

Mechanical strength: It is determined to use a LLOD LR 50 K brand Traction/mechanical compression testing machine used in compression mode and fitted with a 500 N force sensor at Ceramic Technology Transfer Centre (CTTC) in Limoges (France).

The determination of 3-point bending stress is carried out according to protocol inspired by standard NF EN 843-1 under operation conditions presented below:

- The application rate of force: 1 mm/min
- Distance between supports: 80 mm

Table 1: Thermal cycle for the ramp test

N° segment	Rampe (°C/min)	Temperature (°C)	Level time (h)
1	5	1150	/
2	/	1150	2
3	Free descent according to inertia of hearth		

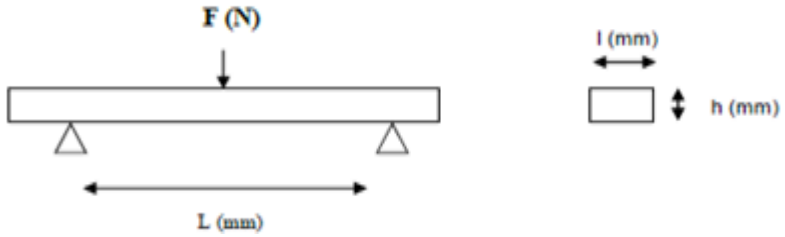

Fig. 1: Flexural strength

- The number and dimensions of parts are tested: 05 specimens are used to perform mechanical tests in 3-point bending.

Mechanical resistance, tensile strength $\sigma_{(MPa)}$ is calculated after the automatic detection of maximum load $F_{(N)}$, causing the bending into two pieces of sample. The software is associated with the machine according to following formula:

$$\sigma(\mathrm{MPa}) = \frac{3FL}{2lh^2}$$

Figure 1 shows us mechanical tests in 3 point. Sample is heated at 120°C for a minimum of 16 h to remove all the traces of moisture. The mass Ms of dry sample is, thus, determined. Sample is placed in a pressure cooker filled with boiling water and maintained for 2 h. Then, boiling water under pressure is going to penetrate into the pores of material. After impregnation, sample is immersed in water. For, we use a suspended basket attached under a weighing pan. The tare of this set is carried out. Sample is placed in a basket. Thus, the mass Mi of immersed sample, subjected only to the pressure of water, is determined. So, sample is rolled on a damp cloth to remove the excess of surface water and immediately weighed to minimize errors due to the evaporation of water in sample. The last mass, determined, is that of the wet sample Mh. By a hydrostatic weighing technique, open porosity is determined by calculation:

$$Open\ porosity(\%) = \frac{M_h - M_s}{M_h - M_i} x100$$

$$Water\ absorption(\%) = \frac{M_h - M_s}{M_s} x100$$

RESULTS AND DISCUSSION

Grain size analysis: Figure 2 gives us the grain size curve of Ntokou clay. These results are summarized in Table 2.

According to Taylor's triangular classification, particles size analysis results show that the NTO soil sample is in the category of clay soils (United States Department of Agriculture, 1993) (Fig. 3).

Figure 4 gives us the position of NTO sample in Winkler diagram (Capitaneo *et al*., 2005). NTO sample may be suitable for the manufacture of light block tiles.

Figure 5 shows us the positioning of NTO sample in the Shepard diagram of Italian clays taking into account their frequency of use in ceramic industry. NTO sample is in the zone of medium frequency of utilization.

Atterberg limits: Atterberg limits results are given in Table 3.

Atterberg limits results are interpreted in the Casagrande chart (Casagrande, 1948) which gives information on the plasticity of clays and in the workability chart (Bain and Highly, 1978), which guides the molding properties of sample. From Casagrande classification, used in Fig. 6, we can see that NTO sample can be assimilated to medium plastic clay soil, since its liquidity limit is <50%.

Activity values, obtained according to Skempton and Seed formula, are given in Table 4.

According to Skempton nomenclature, clays with an activity of <0.75 are based on Kaolinite, or clays containing few clay minerals (inactive clays). The value of activity reveals the presence of clay, precisely of kaolinitic type. These confirm the results of granulometric analysis (NTO soil is a clay soil). The Bain and highly shaping map (Bain and Highly, 1978) from plasticity limit and plasticity index makes it possible to estimate the molding properties of soil.

Table 2: Distribution of Ntokou particles

Specimen	Sands >50 µm	2 µm< Silts <50 µm	Clay <2 µm
NTO	28%	20%	52%

Table 3: Results of Atterberg indices

Specimen	Liquidity limit (W_L)	Plasticity limit (W_P)	Plasticity index (Ip)
NTO	40	16.9	23.2

Table 4: Activity values

Activity	Skempton	Seed
A	0.446	0.493

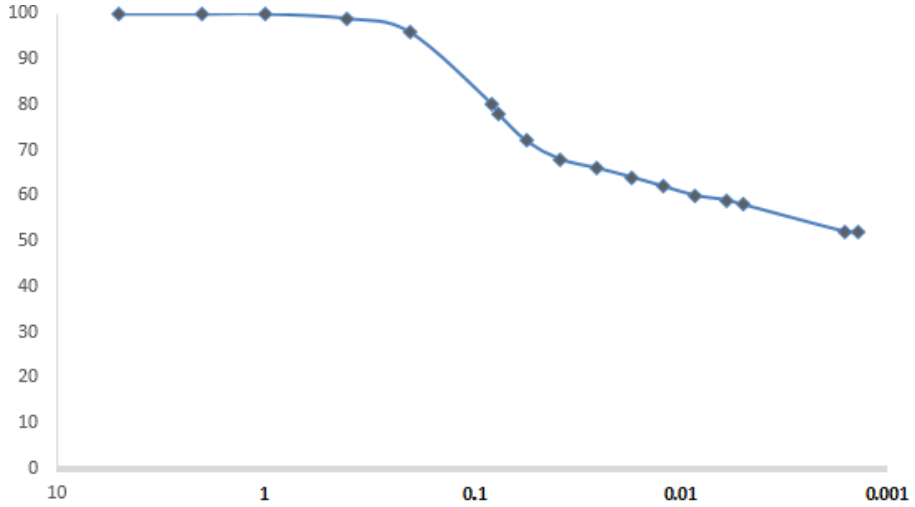

Fig. 2: Granulometric distribution of Ntokou clay

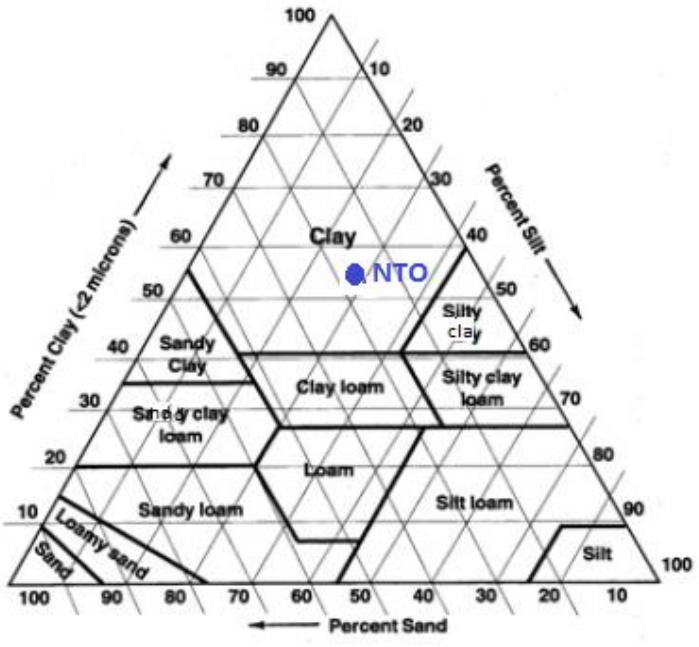

Fig. 3: Positioning of NTO sample in Taylor diagram (United States Department of Agriculture, 1993)

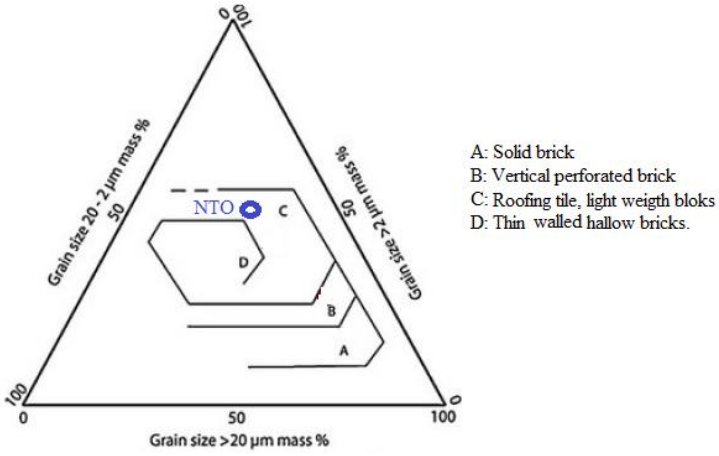

Fig. 4: Positioning of NTO sample in Winkler diagram (Capitâneo *et al*., 2005; Dondi *et al*., 1998; Monteiro and Vieira, 2004)

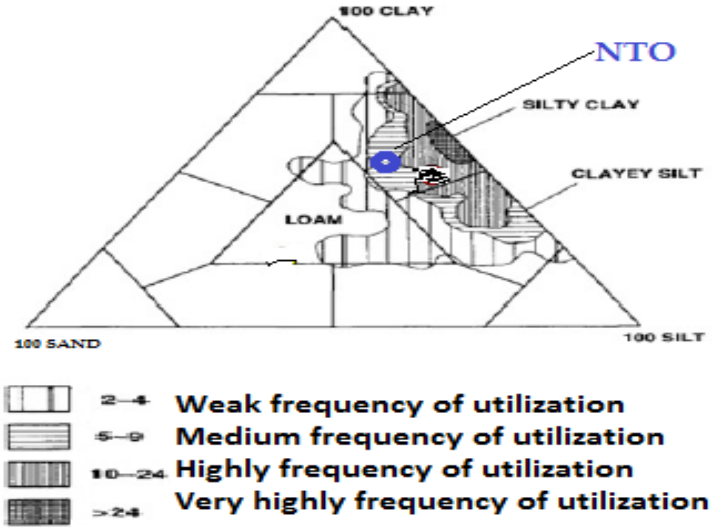

Fig. 5: Positioning of NTO sample in Shepard diagram (Dondi *et al*., 1998)

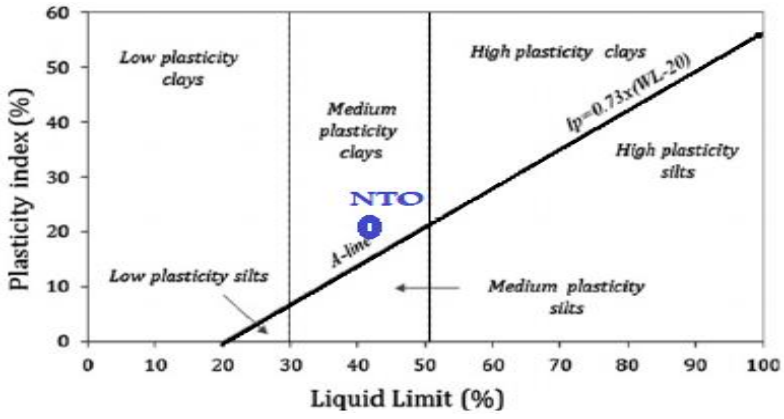

Fig. 6: Positioning of NTO sample in Casagrande chart (Casagrande, 1948)

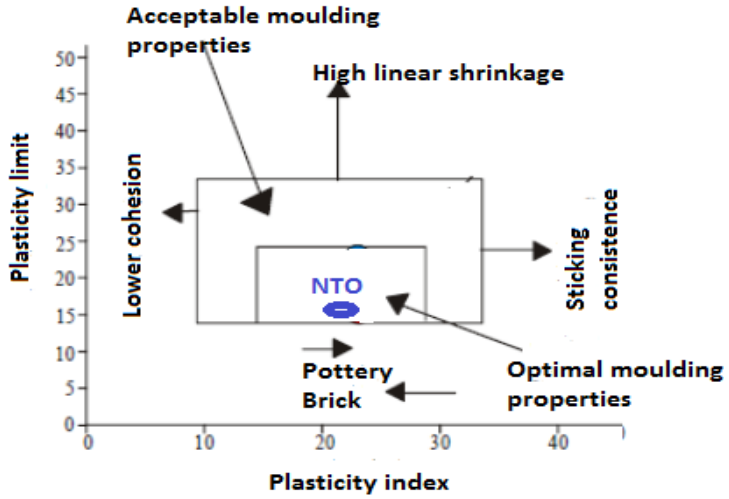

Fig. 7: NTO sample in clay workability chart (Bain and Highly, 1978)

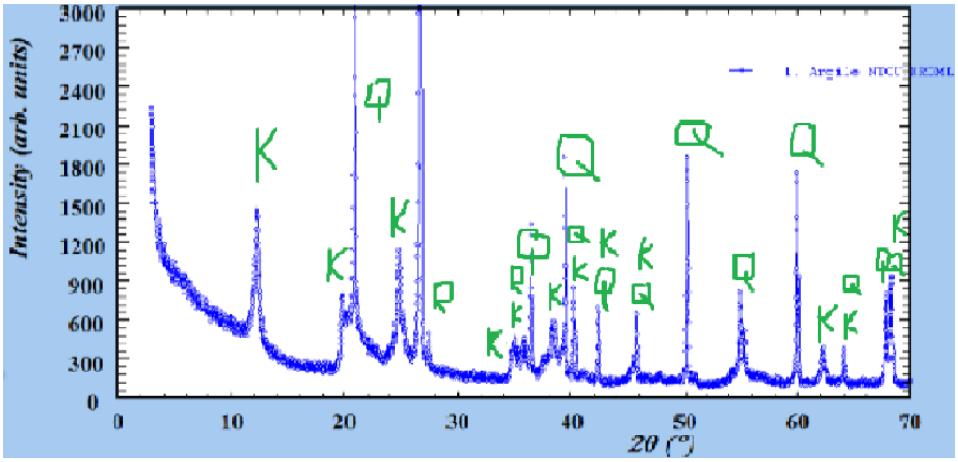

Fig. 8: XRD pattern NTO sample

Figure 7 gives us the position of NTO sample in clay workability chart. NTO sample has optimal molding properties.

X-ray diffraction: Figure 8 gives us the diffractogram X of Ntokou clay.

X-ray diffractogram, recorded on NTO raw sample obtained in using a PANalytical X'Pert PRO diffractometer, reveals the presence of:

- Kaolinite (K) (7.31, 4.49, 4.29; 3.50, 3.37, 2.51, 2.36, 2.29, 2.13, 1.99, 1.89, 1.81, 1.81, 1.54, 1.54; 50, 1.42, 1.38, 1.29 Å)
- Quartz (4.29, 3.37, 2.46, 2.29, 1.99, 1.54)
- Rutile (Brindley and Brown, 1980)

Chemical analysis: The chemical analysis of NTO sample is analyzed at Center for Petrographic and Geological Research (C.R.P.G.) at Nancy (FRANCE) using method: Inductive Coupled Plasma-Atomic Emission Spectrometry (ICP-AES). Table 5 gives chemical analysis results of major elements (Si, Al, Fe, Mn, Mg, Ca, Na, K, Ti and P) as the most stable percentage of oxide.

Weight ratio SiO_2/Al_2O_3 is 4.40 much higher than that in kaolinite (1.17). The SiO_2/Al_2O_3 ratio is a classification parameter for high temperature clays. NTO with a SiO_2/Al_2O_3 ratio of 4.40 can be classified either as a plastic refractory clay (2.4-4.0) or a siliceous refractory clay (3.5-6.7) (Bouaziz and Rollet, 1972). Iron oxide content (1.63%) indicates the presence of goethite. Indeed, by amplifying angular range between 18 and 22°

Table 5: Chemical composition (in % oxide mass) of NTO clay materials

Specimen	SiO_2 %	Al_2O_3 %	Fe_2O_3 %	MnO %	MgO %	CaO %	Na_2O %	K_2O %	TiO_2 %	P_2O_5 %	PF %	Total %
NTO	71.28	16.19	1.63	<LD	0.12	<LD	0.02	0.36	1.43	<LD	9.04	100.07

LD: Limit of detection; PF: Loss on ignition

Table 6: Mineralogical balance

Specimen	Kaolinite	Quartz	Goethite	Sum
NTO	42.75	19.88	1.63	64.26

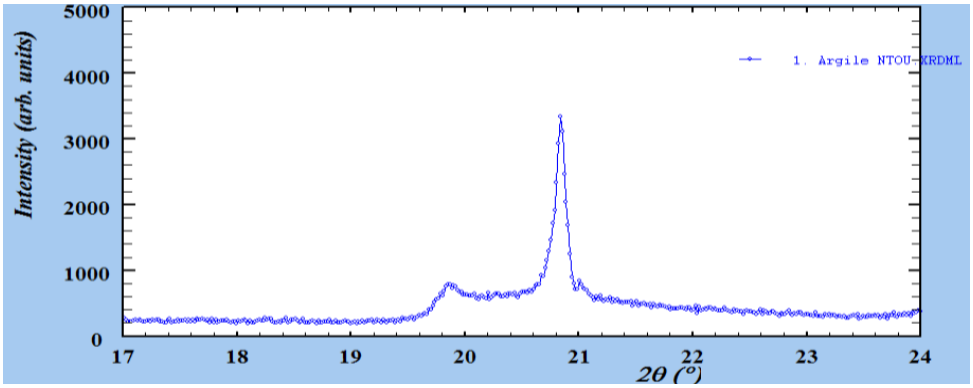

Fig. 9: NTO K_2O sample in Ntokou diffractogram

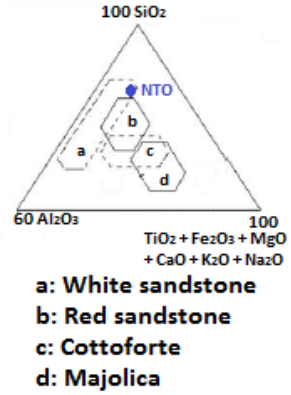

Fig. 10: Positioning of NTO sample in Fiori diagram (chemical composition) (Boch, 2001; Fiori *et al*., 1989)

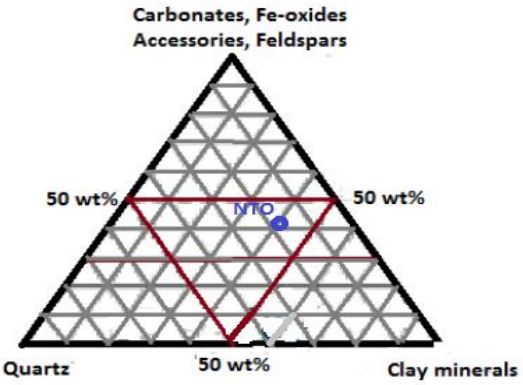

Fig. 11: Positioning of NTO sample in Fiori diagram (mineralogical composition) (Fiori *et al*., 1989)

(2θ) (Fig. 9), we find the presence of a line at distance 4,22Å, which can be attributed to goethite.

The very low content of K_2O in NTO does not augur the sufficient amount of fuse material that can induce low porosity during sintering (Ali, 2002; Elimbi *et al*., 2004). The 3.25 Å line consistent with the significant concentration of TiO_2 can normally be attributed to rutile. The loss on ignition is (9.04%), a value, somewhat, different from that of kaolinite (13.9%). The presence of disordered kaolinite is in agreement with a refraction of these samples (Brindley and Brown, 1980). Alkaline, alkaline-earth oxides are almost absent in NTO, indicating a very weakly fusible clay. In NTO clay, oxides dyes (Fe_2O_3, TiO_2) exist in a remarkable quantity (3.06% in total), which indicates that it is going to give colored cooking products (Bouaziz and Rollet, 1972). Figure 10 gives us the positioning of NTO clay in Fiori diagram.

According to Fiori diagram, chemical composition shows that NTO soil sample is in the domain a. NTO can be used to manufacture white sandstone (Boch, 2001; Fiori *et al*., 1989). Table 6 gives us mineralogical balance.

Figure 11, illustrates a ternary diagram established on mineralogical composition. NTO sample is in red

zone such as showed in the mineralogical composition in Fiori diagram (Fig. 11). NTO is favorable for the manufacturing of red stoneware. As MAD sample (Moutou *et al*., 2018), in Augustinik diagram (Fig. 11), NTO sample is not also positioned in the manufacture's domains of ceramic bodies. NTO sample is close to sandstone domain. The positioning of NTO in Augustinik diagram is given in Fig. 12.

BET specific area: Data collected in Table 7 allows us to draw the line of BET.

Figure 13 gives us BET line. To this straight line, Y-intercept (Table 8) and slope, Slope values and Y-intercept allow to calculate the following parameters (Table 9).

The BET area of obtained sample is 17.5 m^2/g (table). It corresponds with that of Kaolinite (between 10-20 m^2/g) (Chamayou and Legros, 1989; Morel, 1996) but is much lower than that of other species. It shows that clay species, presented in this sample, are kaolinite. The value of specific area of NTO measured by BET method of order of 17.5 m^2/g, given in Table 9 is in range 10-20 m^2/g, compared with Kaolinite presented in literature (Fantozzi *et al*., 2011).

It is noted that the measurement of specific area by BET method does not make it possible to determine the external surface of clay particle (Ali, 2002).

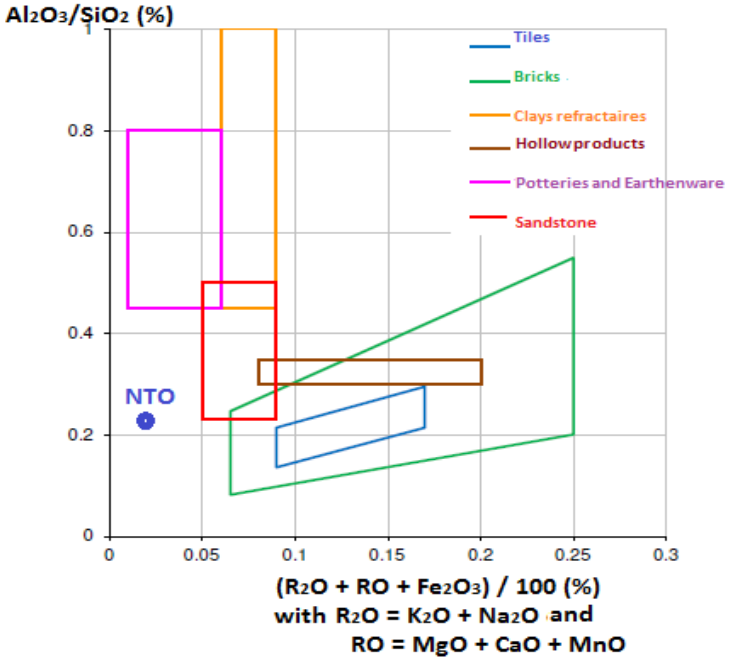

Fig. 12: Positioning of NTO sample in Augustining diagram

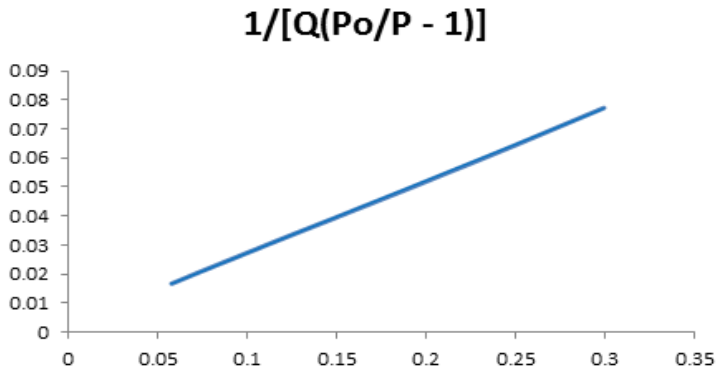

Fig. 13: The BET straight line

Table 7: Parameters values permitting to plot BET line

Relative pressure (P/Po)	Quantity adsorbed (cm^3/g STP)	1/ [Q (Po/P - 1)]
0.05770389	3.65918303	0.01673530
0.07807583	3.86666958	0.02190203
0.11985807	4.22038833	0.03226727
0.14010754	4.37772353	0.03721937
0.16016536	4.52707045	0.04212671
0.19936331	4.80978504	0.05177071
0.24950039	5.16709124	0.06433905
0.29957219	5.53397364	0.07728604

Table 8: Parameters (slope and Y-intercept) values

Parameters	Values
Slope:	0.249276±0.000852 g/cm³ STP
Y-intercept:	0.002313±0.000154 g/cm³ STP

Table 9: Parameters values

Parameters	Values
BET surface area:	17.3028±0.0595 m²/g
C:	108.767842
Qm:	3.9747 cm³/g STP

Differential thermal and thermogravimetric analysis: Figure 14 gives us DTA/TGA curves NTO sample.

The DTA/TGA curves are recorded on NTO sample using a NIETZSCH STA 409°C thermo balance show the thermal behavior of this clay for ATD, a global weight loss 1200°C of 7.7% which mainly breaks down in 3 stages for ATG.

The TGA curve has an overall weight loss at 1200°C of 7.7% which is mainly broken down in 3 steps:

- **From ambient to 150°C:** The departure of residual moisture (the loss of mass of 0.9%) with an endothermic peak on DTA curve at 75°C
- **Between 150 and 350°C:** The probable decomposition of small organic residues (the loss of mass of 0.9%) with rather exothermic behavior around 300°C without a real peak identifiable by software on DTA curve
- **Between 350 and 850°C:** The dehydroxylation of kaolinite (the loss of mass of 5.7%) with endothermic peak at about 519°C, and the transformation of quartz (the passage of phase a in b) with endothermic peak on DTA curve at 572°C, (Bouaziz and Rollet, 1972)

Above 850°C, TGA curve has a loss of mass of (0.2%) rather located after 1100 to 1150°C. DTA curve, for its part, reveals a small exothermal setback at 838°C and an exothermic peak around 965°C, the characteristic of structural reorganization of metakaolinite. (Traore, 2003) Mineralogical balance with regard to diffractogram, muscovite non-detection leads to consider the content of SiO_2 which comes from kaolinite and quartz. Let SiO_2 be the part from quartz and $SiO_2(k)$ from kaolinite. So:

$$\frac{SiO_2}{Al_2O_3} = \frac{[SiO_2(q) + SiO_2(k)]}{Al_2O_3} = \frac{SiO_2(q)}{Al_2O_3} + \frac{SiO_2(k)}{Al_2O_3} = \frac{SiO_2(q)}{Al_2O_3} + 1.17 = 4.40$$

$$SiO_2(q) = Al_2O_3(3.23) = 16.19\ \% \times 3.23 = 52.23\%$$

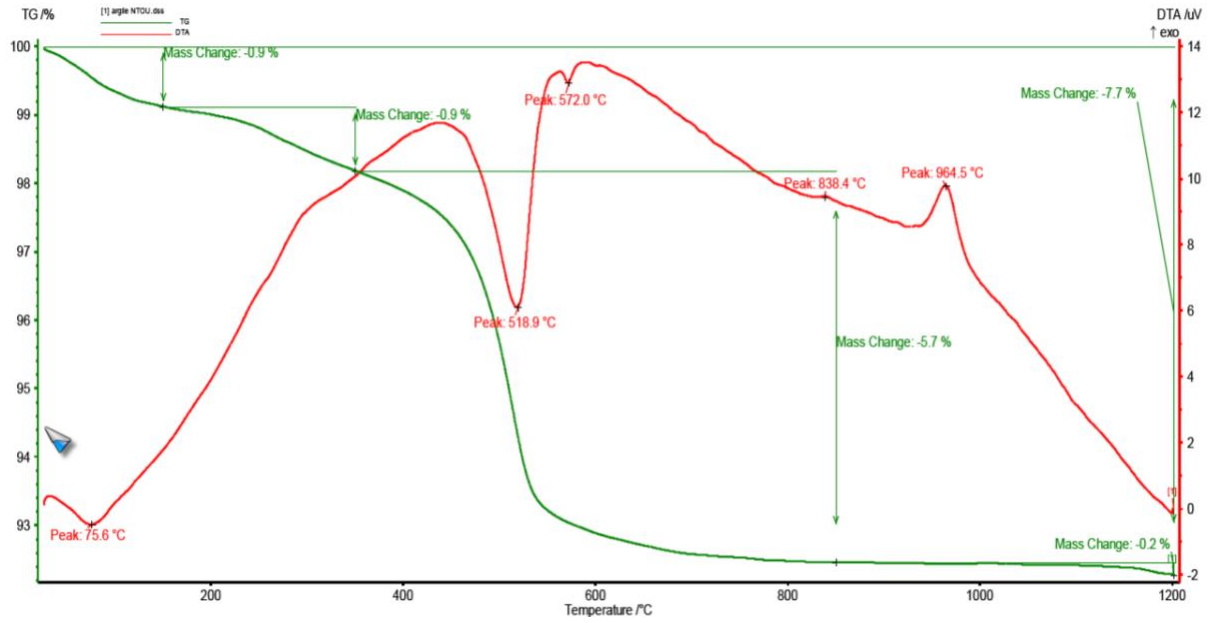

Fig. 14: DTA/TGA curves NTO sample

Table 10: Technological properties

Experiment	Results	Medium results
Shrinkage linear	R (length) 2.02±0.02 R (breadth) 1.64±0.25 R (width) 1.99±0.15	1.6%<R<2%
Open porosity	35.397±1.580%	
Water absorption	16.928±1.580%	
Flexural strength	High flexural strength $F_{(N)}$ 8.55±1.41	Flexural constraint $\sigma_{(MPa)}$ 1.61±0.30

The deduced quartz rate (52.23%) higher than 50% justifies the observation of peak of quartz-quartz, quartz transition at 572.8°C. This observation is made by Pialy and on quartz clays (Pialy, 2009; Boussak *et al*., 2015).

Knowing that [SiO_2 (k)] / [Al_2O_3)] = 1.17 we can estimate SiO_2 (k) = 18.94%. The content in water in corresponding kaolinite 5.71 or 5.68% we deduce the kaolinite 40.84%. It is comparable to that is deduced from the mass loss (TGA) at 518.9°C corresponding to the deshydroxylation of kaolinite (40.78%). With a composition of kaolinite between 25 and 50% clay sample taken from Ntokou can be classified as raw kaolin (Dondi *et al*., 1998). According to Dondi, raw kaolins are hardly used alone as ceramic raw materials. The low plasticity of these clays (raw kaolin) requires additions of other clays and non-clay materials to have an adequate technological behavior.

The technological properties of Ntokou clay at 1150ºC are determined and collated in Table 10.

The significant presence of quartz in Ntokou clay reducing plasticity remains the cause of very low cupping. The very low fuse content is revealed by low concentrations of alkaline, and alkaline earth oxides do not favor the densification of NTO clay shard; therefore, material is going to have an open porosity (35.4%) and an absorption (16.98%) higher. At 1150°C according to classification established by Dondi *et al*. (1998) with absorption rate greater than 10% we get a porous body. Ntokou clay also has low flexural strength. With identical, chemical and mineral compositions, the clays of Tresnuraghes (Salamura, Fongarazza and Patalza) and those of Romana-Cossoine are characterized by the low values of shrinkage and resistance to flexion (Allegato 51034). An absorption rate, greater than 10%, it is likely to manufacture wall tiles with a porous support (Dondi *et al*., 1998), but the modulus of rupture with NTO clay is also weak compared with those found in literature.

CONCLUSION

This study aims at the physical-chemical, mineralogical and technological characterization of clay extracted from the locality of Ntokou, located near Makoua (Brazzaville, Republic of Congo).

The clay soil of Ntokou has a texture in clay soil category. The particle size composition indicates that this soil is located in the area of medium frequency materials and has potentially to be used in the manufacture of ceramic products such as tiles and sandstone. Atterberg limits have confirmed the nature of this soil (clay soil). They allow it to be classified as medium plasticity soil and to place in area materials with optimal molding properties. As water absorption rate is higher, the soil has low shrinkage and flexural strength. The thermal behavior of this clay was given by DTA/TGA curves. Kaolinite and quartz are detected by X-ray diffraction in this soil. Chemical analysis indicates the presence of three major oxides: SiO_2, AL_2O_3 and Fe_2O_3. The report SiO_2/Al_2O_3 (4.40) makes it possible to classify this clay in the family of siliceous refractory clays (3.5-6.7) or in the family of plastic refractory clays (2.4-4.0). The report Al_2O_3/SiO_2 (0.227) allows it to be used as a complementary material for the manufacture of stoneware or hollow ceramic products. After cooking products are colored due to the presence of oxides (Fe_2O_3, TiO_2). In sum, a treatment (the addition of plasticizing clays or reduction of silica) is necessary to improve granilotry, technological properties (plasticity, porosity, shrinkage and flexural strength) and for use in fine ceramics.

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