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## JMST Advances

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# Physico-chemical, mineralogical characterization, and ceramic properties of clay materials from South Mindif (Far North, Cameroon)

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## Abstract

Characterization studies of clays are generally carried out to identify possible applications of these materials. This paper describes the physicochemical, mineralogical, and thermal characterization of residual clays from Mindif commune in Cameroon's Far North Region (Doyang, Mendeo, Mobono, Hoppo, Mindif Center, Tchouake, and Gagadje). Ten samples were taken and studied in the laboratory. The methods used in this work were X-ray diffraction (XRD), X-ray fluorescence (XRF), infrared spectrometry (IR), thermogravimetric analysis (DTA/TG), particle size analysis by sedimentometry, Atterberg limits, and physical and firing properties at 900°C, 1000°C, and 1100°C. It appears that the particle size distribution is dominated by sand (25 to 74%), clay (10 to 48%), and silt (6 to 27%). They mostly correspond to sandy clay soils (blue values : 1.5–5), with low (7–13%) to high (23%) plastic characteristics. Chemical analyses showed high SiO<sub>2</sub> (53–75%) and low Al<sub>2</sub>O<sub>3</sub> (6–14%) contents, followed by Fe<sub>2</sub>O<sub>3</sub>, CaO, K<sub>2</sub>O, and MgO. The clay materials are mainly made up of quartz, which is associated with feldspar, illite, smectite, and kaolinite. Between 900°C and 1100°C, the color of the specimens was mainly orange. The characteristics of the bricks are varied: water absorption (6-27%), linear shrinkage (1-10%), and flexural strength (4-17 Mpa). A significant development in the densification of ceramic behavior could be noticed at firing temperatures above 1000°C. The results presented in this article indicate that these clay materials are suitable for the manufacture of earthenware and ceramic products, with or without addition.

**Keywords :** Clayey soil characteristics, Mineralogy, Physico-chemical, Ceramic properties, Mindif.

## 1. Introduction

Clays are aluminosilicates composed of fine grains, more or less hydrated, whose structure consists of a stack of sheets [1]; which pass to the plastic state in the presence of appropriate water contents, and which harden by drying or by heating. Their natural abundance and availability justify their great use by humans; they are thus an integral part of our daily lives. They are natural raw materials widely used in the fields of housing and ceramics [2–15], the pharmaceutical and cosmetics industries [16–18], the discoloration of oils, and the adsorption and elimination of pollutants [19–25].

From the synthesis of research and thanks to some university and professional work, it appears that clayey formations (sedimentary, alluvial, and residual) in Cameroon are distributed in almost all regions [26]. Many avenues of technological research have been carried out on clay materials throughout Cameroon in general and are still being explored in the North region (Cameroon) [12, 27] and more particularly in the Far North region (Cameroon) [28–30]. However, the Mindif clays in the Mayo-Kani department did not report any such characterization. These clays are mainly used in ceramics and the construction of local houses. To better valorize a raw material, knowledge of its intrinsic parameters is important before directing it towards the best artisanal and/or industrial applications. This study is the first from this locality, and the results of this work will be used to feed a database to support the start-up of artisanal exploitation projects for local clay materials (creation of non-relocatable local jobs) and the optimization of the manufacture of ceramics. These

experimental and scientific approaches contribute to the promotion of local materials and sustainable development, as well as other research.

The physicochemical and mineralogy characterization of Mindif clays (Far North, Cameroon) is the focus of this work. The aim of this study was to characterize the Mindif clays in order to determine their potential uses. This study constitutes an important scientific contribution to the knowledge and characterization of the mineral substances of Cameroon, more particularly the clayey materials of the commune of Mindif and its surroundings.

## 2. Materials and Methods

### a. Study sites

The natural clays that are the subject of this study were sampled in Mindif, located in the north of the department of Mayo-Kani (Far North, Cameroon); with geographical coordinates are Latitude  $10^{\circ}23'51''$  North and longitude  $14^{\circ}26'10''$  East. The geographical map of our study area is shown in figure 1. Geologically, Mindif is mainly composed of sedimentary and eruptive rocks. The mineralogy of granites is mainly composed of quartz, microcline, oligoclase, biotite, green hornblend, and locally augite. The alluvial deposits (fine clay, sandy clay, and more or less feldspathic or micaceous sand) form a belt all around the peak of Mindif (up to 700 m). At Mindif East, the accumulation of eolian sand takes precedence over fluvial backfilling [31]. Literature studies [31–32] have suggested the clayey nature of the material.

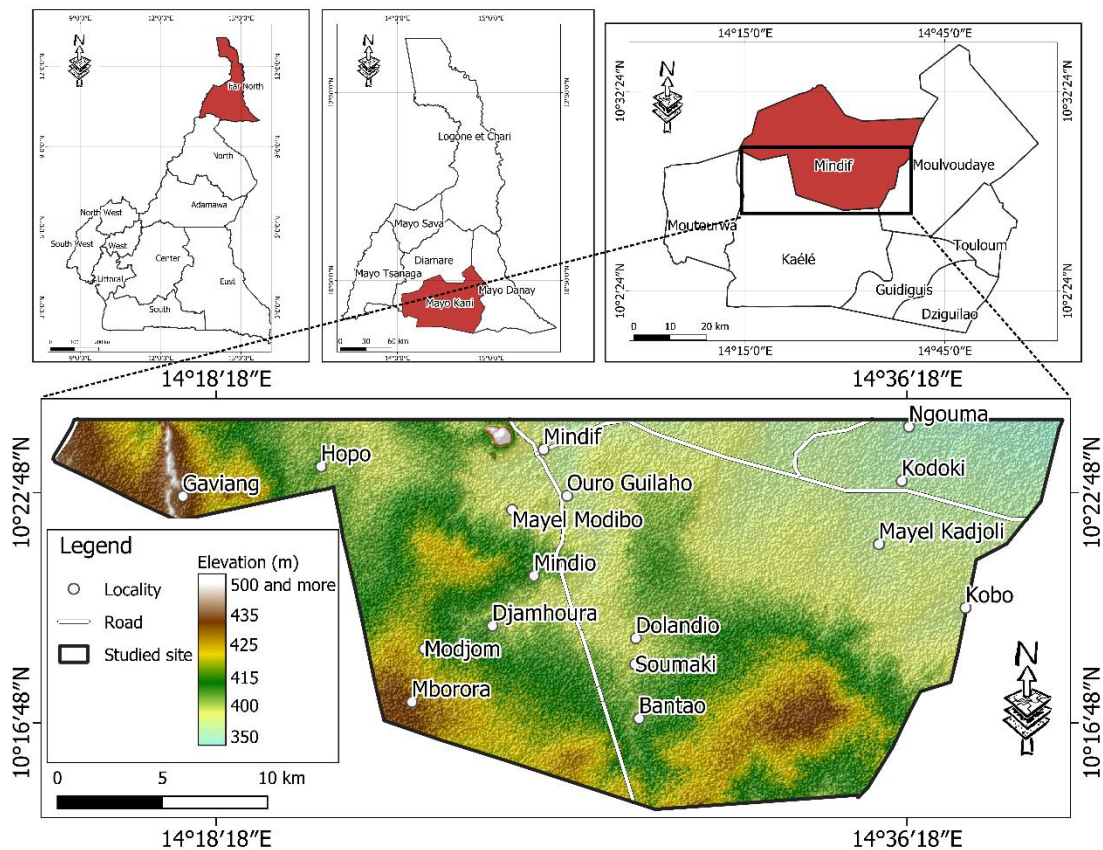


Figure 1: Location map of the study area

### b. Materials

Fieldwork was carried out during the dry and rainy seasons and consisted of direct observations, descriptions of environmental aspects, and a soil study in order to plan the sampling of clay materials. Ten

samples were taken from seven sites : Doyang, Mendeo, Mobono, Hoppo, Mindif Center, Tchouake, and Gagadje, which had codes DO2, DO3, DO6, and DO7, respectively for Doyang, and ME12, MO17, HO19, MI21, TC22, and GA23 for the others. Figure 2 shows the clay material sampling map. The colors of these samples are presented in Table 1. This color variation may be due to climatic conditions, bad weather (redox reactions) [33], heat, organic matter, and mineral and elemental compositions. The samples were selected on the basis of their color and homogeneity. Thus, about 30 kg of each sample were collected and placed in polyethylene bags. 1 kg of each sample was dried, pulverized, and sieved before analysis.

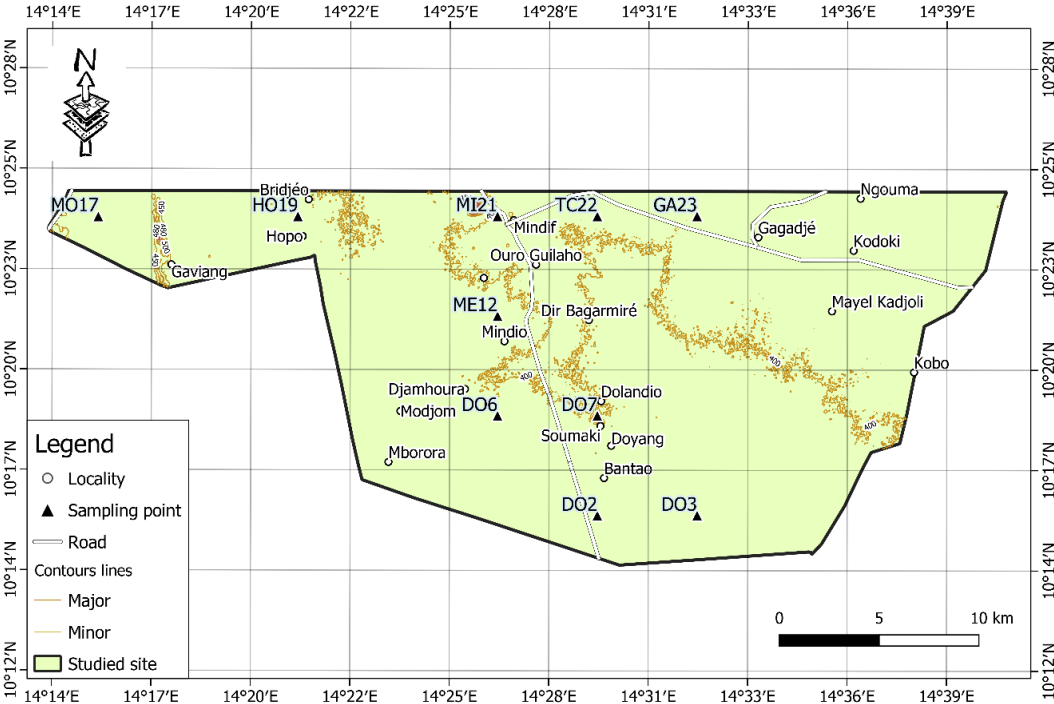


Figure 2: Sample map of materials studied

Table 1: Color of clay sample

Site	Sample	Color
Doyang	DO2	Light Brown
	DO3	Brown
	DO6	Grey
	DO7	Dark grey
Mendeo	ME12	Dark grey
Mobono	MO17	Light grey
Hoppo	HO19	Light grey
Mindif center	MI21	Pale black
Tchouake	TC22	Black
Gagadje	GA23	Light Brown

**c. Experimental methods**

The particle size distribution, the Atterberg limits, and the methylene blue test were considered geotechnical characteristics of the clay materials studied. To determine the particle size distribution, the fraction  $\geq 80 \mu\text{m}$  of clay materials was subjected to sieving, then to sedimentation for the fraction  $< 80 \mu\text{m}$  according to ASTM D-422 standards. These Atterberg limits (liquid limit (LL), plastic limit (PL), and plasticity

index (PI)) were determined using the Casagrande method (ASTM D-4318 standards) [34]. According to the Skempton formula, the ability of clay to absorb cations from a solution is determined by the methylene blue test (VBS). The water content (w) was obtained by double weighing, the first for the raw sample (W) and the second after drying in an oven (at 105 °C) until constant mass (Wd).

The mineralogical composition was obtained from X-ray diffraction on the raw sample powders, both disoriented powders and oriented aggregates. A Bruker Advance D8 diffractometer (copper K $\alpha$ 1 radiations,  $\lambda = 1.5418 \text{ \AA}$ ,  $V = 40 \text{ kV}$ , and  $I = 30 \text{ mA}$ ) at the Laboratoire des Argiles (Geochemistry and Sedimentary Environment, University of Liège, Belgium) was used according to the methodology of Moore and Reynolds [35]. Diffractograms were processed using X'Pert High Score Plus software (version 3.0).

The diffuse reflectance infrared spectra were recorded using a Bruker brand Alpha-p IR spectrophotometer in transmittance mode for a wavenumber scan that varies between 4000 and 400  $\text{cm}^{-1}$  by analyzing the surface areas and volume.

The analysis of the chemical composition was obtained using X-ray fluorescence spectroscopy (Bruker S8 Tiger, 4 kW) at the Laboratory of "Endogenous Petrology, Geochemistry, and Petrophysics (PGEP)" at the University of Liège, Belgium. The degree of material alteration was determined by the Mineralogical Alteration Index (MIA). It was deduced from the data on the major elements:

$$\text{MIA} = 2 \left( \left[ \frac{\text{Al}_2\text{O}_3}{\text{Al}_2\text{O}_3 + \text{CaO} + \text{Na}_2\text{O} + \text{K}_2\text{O}} \right] \times 100 \right) - 50 \quad (\text{E1})$$

The differential thermogravimetric analysis of clays (DTA/TG) was carried out using a SETARAM Cahn device. Each analysis was performed on 40 mg of powder at a heating rate of 20°C/min in platinum-rhodium crucibles using calcined alumina as a reference. The analysis was carried out over a range of 25 to 1000°C.

Baking tests were carried out on the materials studied. They were dried for 24 hours at 105 °C, ground, and sieved (100  $\mu\text{m}$  mesh). The clay materials were wetted, then molded into specimens 40 mm in diameter and 80 mm in length. They were dried in a shaded and ventilated room. These samples were fired at 900 °C, 1000 °C and 1100 °C for 5 hours at a heating rate of 5 °C per minute using a MultiMate oven. Color, sonority, linear shrinkage, flexural strength, and water absorption were determined. In order to determine the sonority, the parallelepiped Fred specimens were struck with a metal rod, and the Munsell Soil Color Chart was used to determine the color. Linear shrinkage was determined on Fred's test specimens using a vernier caliper. Water absorption was determined from ASTM C373-72. The mass  $M_s$  of the specimen is first determined just after it leaves the oven. Then the specimen is immersed in water for 24 hours and carefully blotted with paper, and its mass  $M_a$  is determined. The relationship that gives the percentage of water absorbed is:

$$\text{WA} = \left[ \frac{(M_a - M_s)}{M_a} \right] \times 100 \quad (\text{E2})$$

Flexural strength was determined from ASTM C674-77. This experiment was carried out using a bending device using the three-point method. The flexural strength, in Mpa, is given by the following relation:

$$\sigma_f = \left( \frac{3}{2} \right) \times \left( \frac{P d}{l e^2} \right) \quad (\text{E3})$$

With d: distance between the cylindrical supports (mm) e: thickness of the specimen (mm) P: load applied at break (KN) l: width of the specimen (mm)

### 3. Results and Discussions

#### 3.1 Geotechnical properties of raw materials

Table 2 presents the geotechnical characteristics of the materials studied (particle size distribution, Atterberg limits, methylene blue test, water content, and organic matter).

**Table 2: Geotechnical properties of clay soils in Mindif**

SITES	DOYANG				MENDEO	MOBONO	HOPPO	MINDIF CENTER	TCHOUAKE	GAGADJE
Samples	DO2	DO3	DO6	DO7	ME12	MO17	HO19	MI21	TC22	GA23
Organic Matter (%)	1,65	2,08	3,24	3,28	2,13	2,85	1,94	2,09	4,32	2,32
Water content (%)	2,02	1,77	20,87	2,18	7,81	5,28	18,08	15,09	16,58	7,64
Gravel (%)	2,2	2,3	0,2	0,0	4,1	0,0	0,6	4,4	0,0	0,8

$\Phi > 2$ mm										
Sand (%) $2 > \Phi > 0.02$ mm	61,8	68,4	59,5	46,9	53,8	37,1	62,4	74,7	25,1	62,6
Silt (%) $0.02 > \Phi > 0.002$ mm	13,9	18,6	14,8	19,8	11,3	25,8	14,4	6,7	27,0	16,0
Clay (%) $\Phi < 0.002$ mm	22,1	10,7	25,5	33,3	30,8	37,1	22,6	14,2	47,9	17,6
Liquid limit (%)	26,5	28,3	27,9	34,9	32,2	29,1	33,5	31,8	52,8	31,9
Plastic limit (%)	18,0	18,9	17,2	21,6	20,0	20,2	23,7	24,3	30,1	25,2
Plasticity index (%)	8,5	9,4	10,7	13,3	12,2	8,9	9,8	7,5	22,7	6,7
Blue value		1,5	2,5	2	2	3,5	1,5	2	5	1,5
Consistency index (= (LL-w)/PI)	1,88	1,82	--	1,46	0,99	1,67	0,57	1,22	0,59	2,62

The grain size curves are presented in Figure 3. From these results, it can be observed that clays MI21 (4.1%) and ME12 (4.4%) contain the largest amounts of gravel fraction, while DO2 and DO3 contain around 2%, and the others (DO6, DO7, ME12, MO17, HO19, TC22, and GA23) are all below 1%. The sandy fraction is the most important of the materials studied. The highest proportions are observed in all samples except those of DO7, MO17, and TC22 (46.9%, 37.1%, and 25.1%, respectively). The silt fraction of these clays has average amounts except MI21 (6.7%), which is relatively low. Except for TC22, which has a clay fraction of 47.9%, all the other samples are below 37.1%.

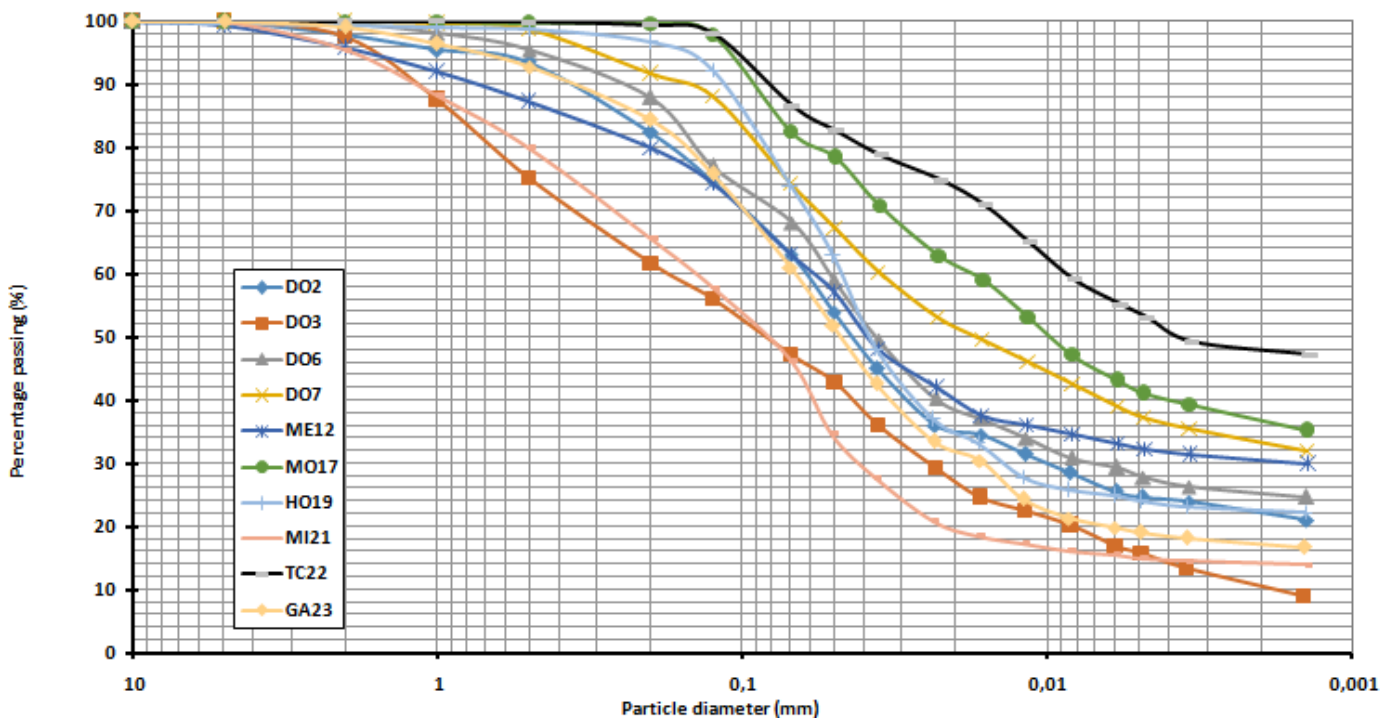


Figure 3: Particle size curves of the materials studied

Using the ternary diagram [36], the clays studied were classified (Fig. 4) into heavy sandy clay (MO17 and TC22), sandy clay (DO2, DO6, DO7, ME12, HO19), clayey sand (DO3 and MI21), and heavy sandy silt (GA23).

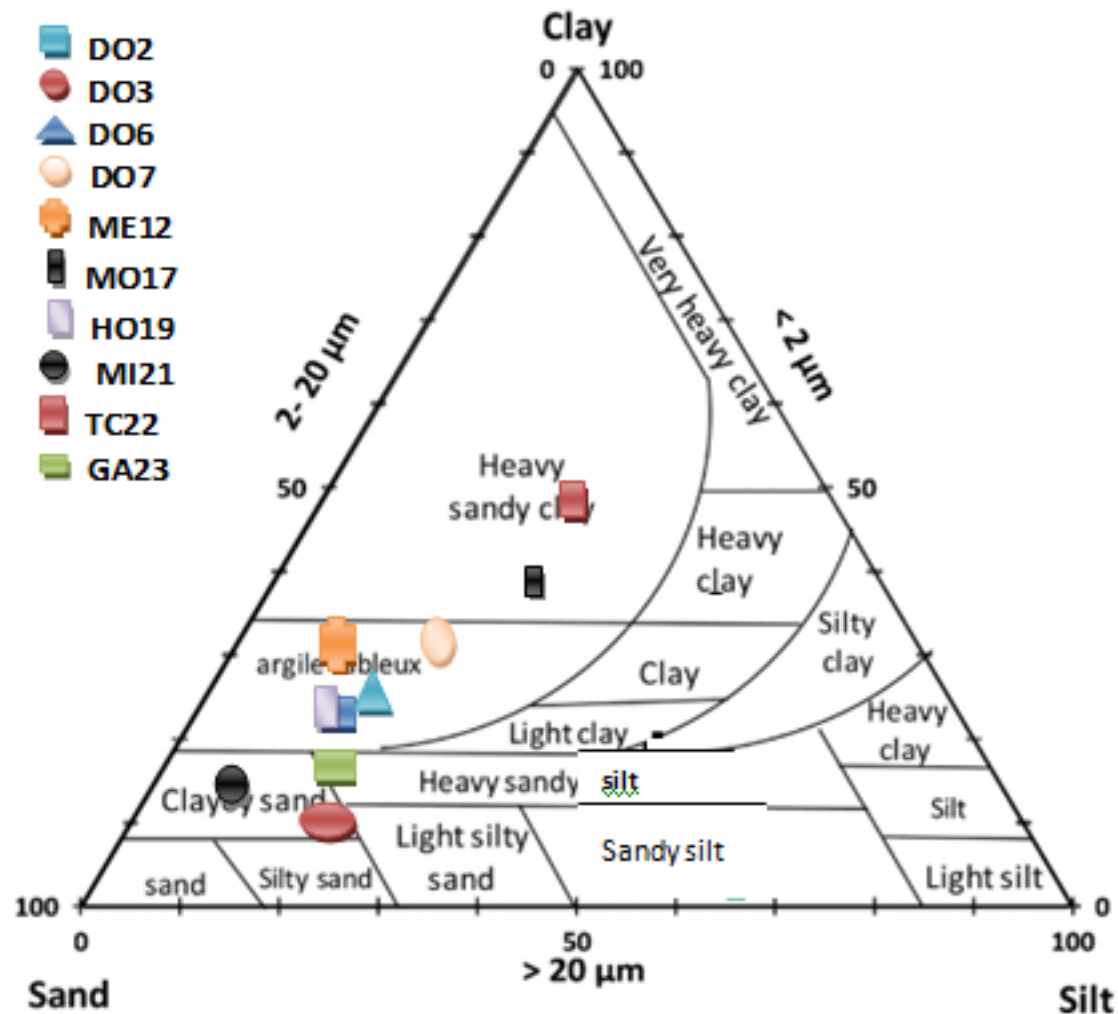


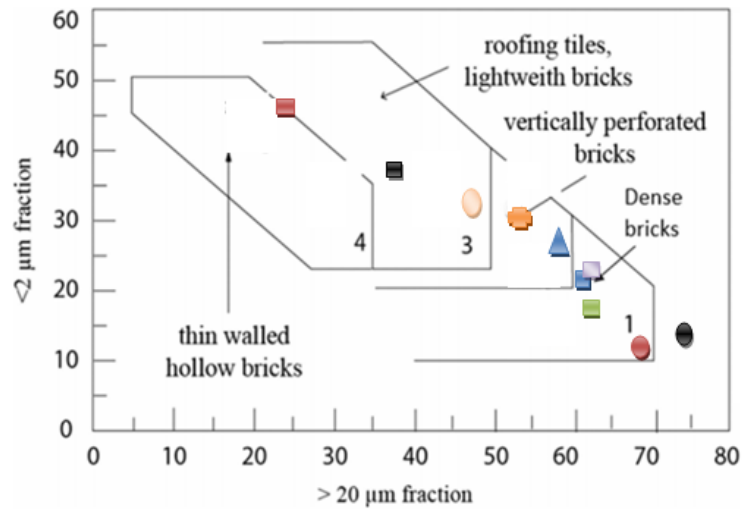
Figure 4: Ternary diagram of the studied clay materials samples [20]

On Winkler's ternary diagram [37] (Fig. 5.a), all clay materials show an ability to produce building materials and ceramics except MI21, which is not classified. The particle size distribution of clay materials has a significant influence on the technological properties of drying and firing [38]. Compared to the clay materials of Boulgou (North, Cameroon) [39], they are less sandy and more clayey, thus facilitating pre-treatments for engineering purposes.

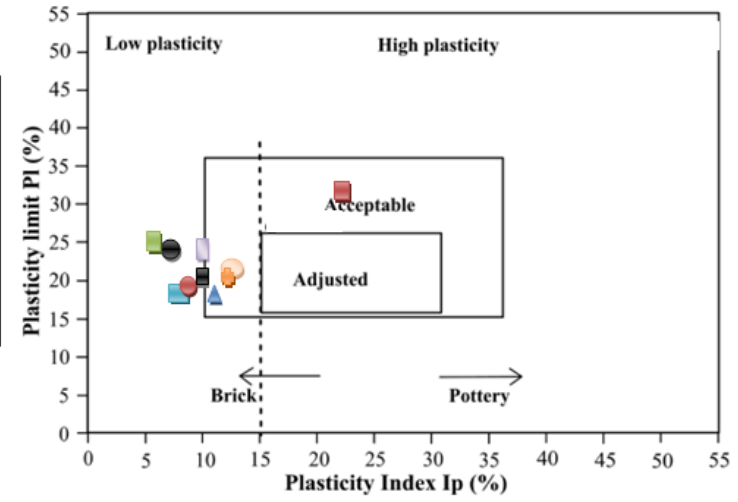
The water content [40] contained in these clays varies from 1.77 to 20.87% by weight. The organic matter present in the raw materials (Table 2) varies from 1.65 to 4.32 %. It is linked to vegetation and the water regime. The resistance of a building material is considerably reduced by the presence of organic matter. They lead to corrosion and softening of the material [41].

The clay materials studied have a plasticity index (Table 2) ranging from 6.7 to 22.7%. From the results, TC22 is plastic (22.7%). Samples DO7, ME12, and DO6 have a plasticity index slightly above the threshold (10), while the others are relatively low (with  $PI < 10\%$ ). According to the Bain and Highley diagram [42] and Figs. 5. c and 5.d; all these samples (exception of TC22, which is plastic) have a low plasticity. Samples DO6, DO7, ME12, and TC22 are acceptable for brick making, while the others are rather unsuitable for extrusion due to their low plasticities. The latter require the appropriate addition of a plastic material (greaser) in order to improve their handling. The plasticity index of clay materials is an important parameter in construction and ceramics [43].

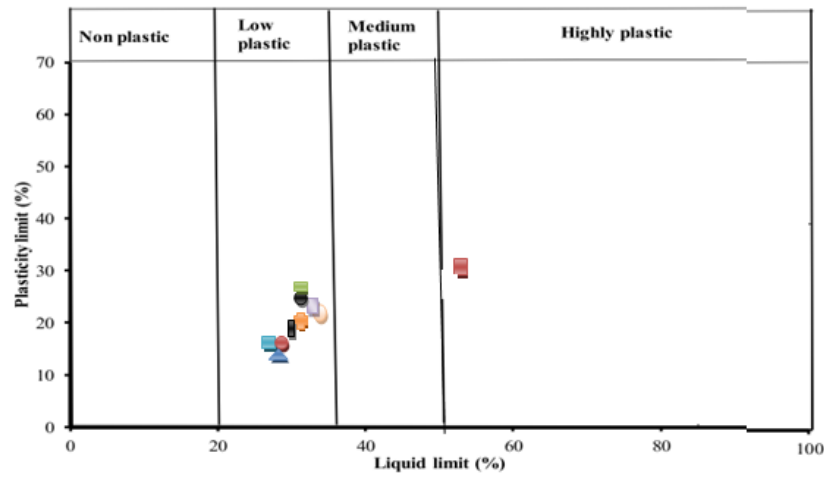
Methylene blue values vary between 1.9 and 5.0. The TC22 sample records the highest blue value (5.0), followed by MO17 with 3.5, while all the other samples are below 3. These values confirm that the clay soils studied are rich in type 2:1 clay materials, explaining the swelling behavior observed at the sites [39].



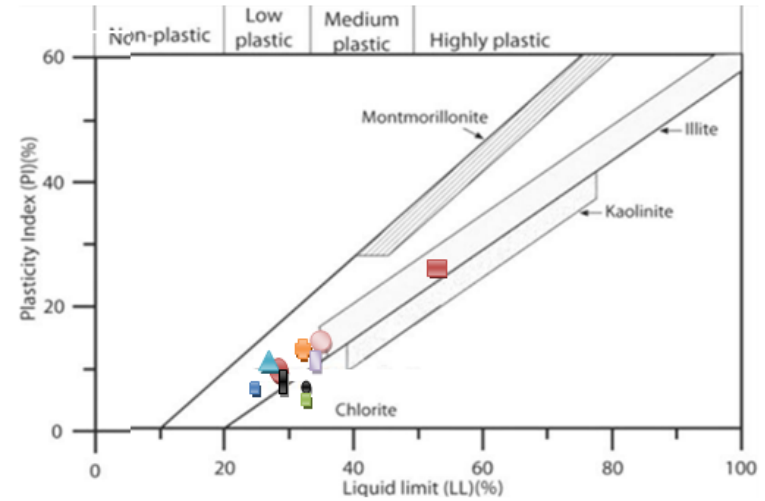
(a)



(b)



(c)



(d)

Figure 5: Position of Mindif soils in some empirical diagrams: (a) Diagram of Winkler [21], (b) Diagram of Bain and Highley [25], (c) Diagram of Holtz and Kovacs [28] in [26] (d) Casagrande's plasticity chart (Holtz & Kovacs) [28] in [26]

### 3.2 Mineralogical and physico-chemical compositions

#### 3.2.1- Mineralogical composition

Mineralogical composition was determined for samples DO7, ME12, MO17, and TC22. The different mineralogical phases of these clays are presented in Figure 6, which shows polymineralized materials. The diffractograms (Fig. 6) show that quartz (Q) (peaks around 4,26 Å, 2,46 Å, 2,28 Å, 2,23 Å, 2,13 Å, 1,98 Å, 1,81 Å, 1,67 Å, 1,54 Å, 1,45 Å) and feldspar (3,25 Å) predominate in all of the samples. Clay minerals are composed by illite (I) peaks around 4,48 Å, 2,50 Å, 2,24 Å, smectite (Sm) (14,91 Å, 1,49 Å), and kaolinite (K) (7,16 Å, 3,57 Å, 3,37 Å). They are present in all samples except for DO7, where we note the absence of smectite. As associated mineralogical phases, we observed albite (Ab), goethite (Go), and calcite (C). The mineralogical composition of clay materials is in agreement with many studies carried out in the northern part of Cameroon [28] [45] [43]. The bisiallization alteration processes produced in the Far North region (Cameroon) favor the presence of smectite [43]. The kaolinite present in these clays exploited for pottery is an important mineral in its composition since it does not promote a phenomenon of shrinkage or swelling during drying and rewetting [46]. The plasticity of clay is greatly improved by the presence of illite [47]. When compared to the strongly smectic logone valley clays (Cameroon) [45], the mineralogical composition of these clays provides adequate ceramic properties. On the basis of the semi-quantitative method (table 3), quartz is dominant in Mindif, clays but the proportions vary from one sample to another. The lowest proportion of quartz is observed in the Tchouake residual clay sample (TC22), as indicated by the low SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> ratio (5,7%) and high LOI (12,7%) shown in Table 4. All samples are characterized by a low proportion of kaolinite; the samples DO7 and TC22 show the highest smectite content. The existence of the smectite group suspected by field observation (swelling) and the methylene blue test is confirmed by the XRD results obtained (Fig.6, Table 3).

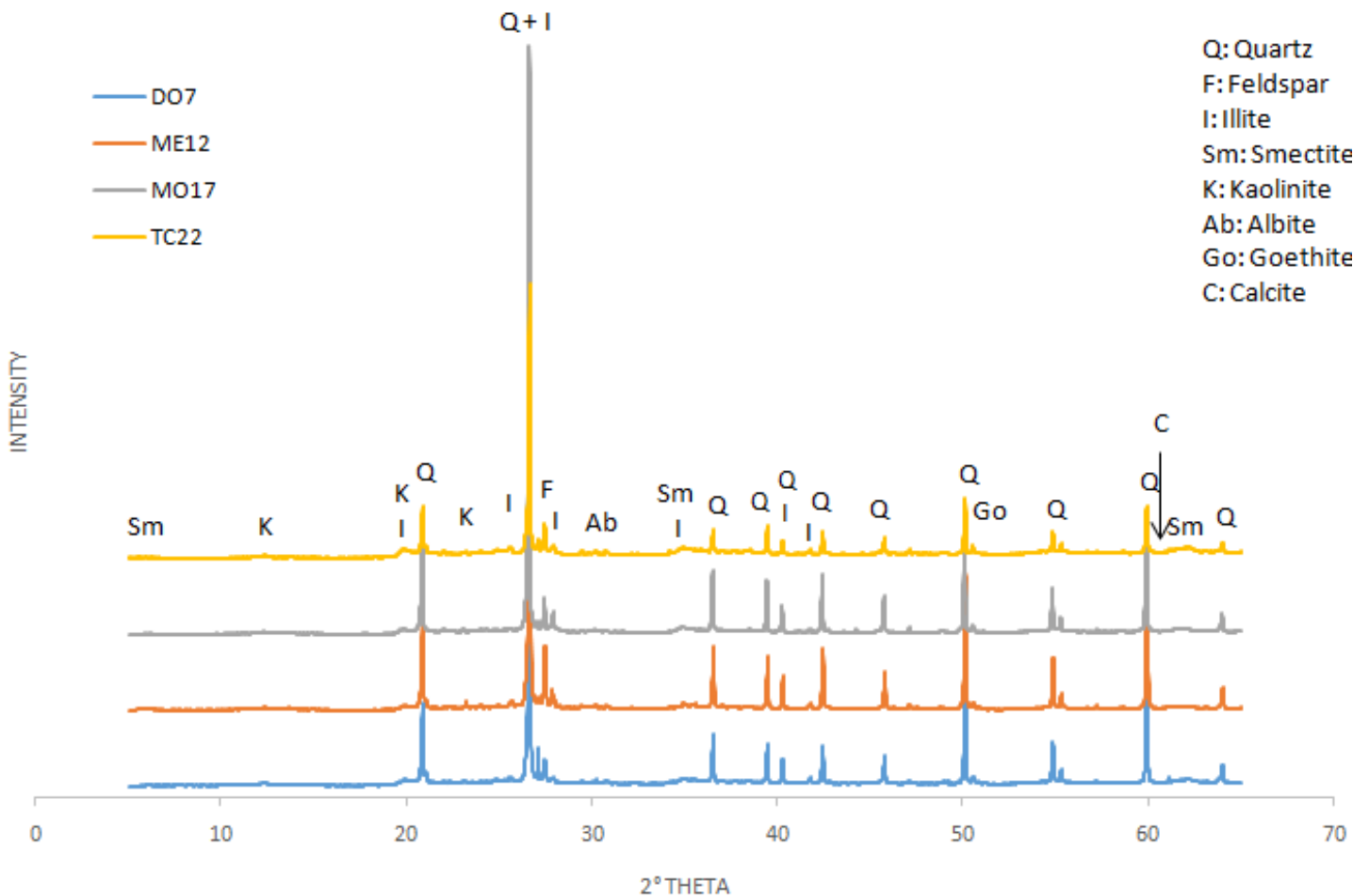
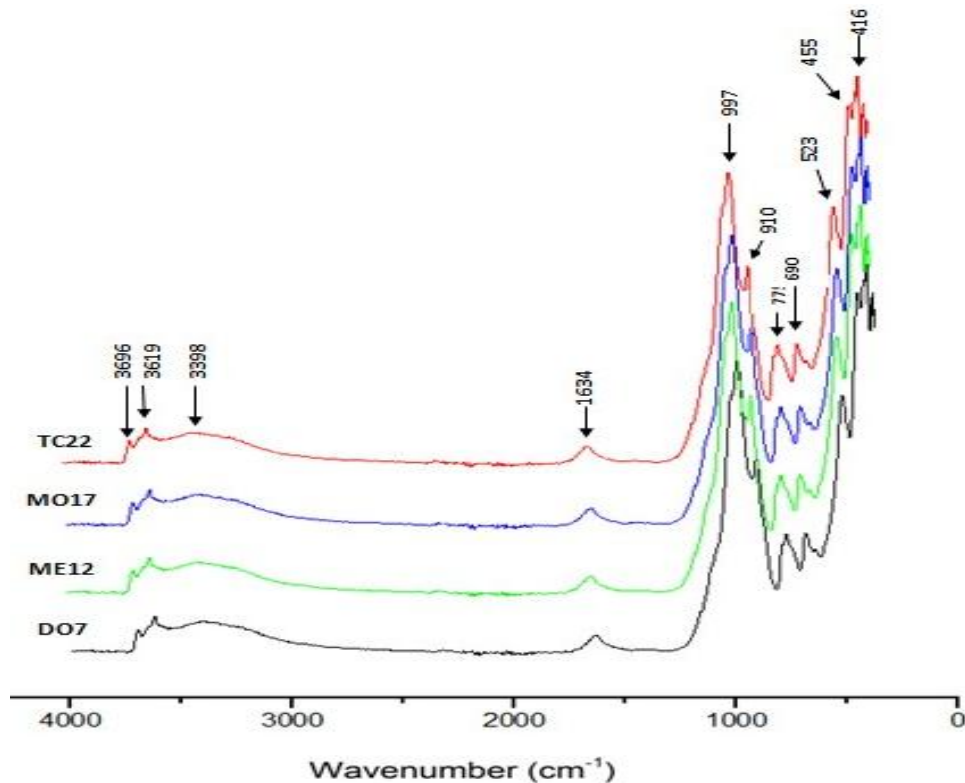


Figure 6: XRD patterns of Doyang, Mendeo, Mobono and Tchouake clays

**Table 3: Semi-quantitative estimation, based on X-ray diffraction, of the mineralogical composition of of Mindif clays Materials**

Samples	Quartz (%)	Illite (%)	Kaolinite (%)	Smectite (%)	K-feldspar (%)	Goethite (%)	Albite (%)	Calcite (%)
DO7	+++++	++	+	++	+++	+	+	+
ME12	+++++	++	+	+	++++	+	+	/
MO17	+++++	++	+	+	+++	+	/	+
TC22	++++	+	+	++	+++	+	+	+

Figure 7 depicts the results of the infrared spectrometry analysis. These spectra show two large absorbance zones; the first zone, between 3696 and 3619  $\text{cm}^{-1}$ , reveals the characteristic bands of the external hydroxyl bonds (OH) of the kaolinite network and possibly of illites [48, 49]. The presence of two bands instead of four indicates poorly crystallized kaolinite [39]. Water adsorbed at the interlayer of smectitic clays is responsible for the absorption band at 3398  $\text{cm}^{-1}$  (OH- stretching vibration) [50]. In contrast to the kaolinitic clays found in the Central [51] and Adamaoua [33] regions of Cameroon, the inclusion of smectite in these clays may have uses in the depollution of sewage. The second zone is between 2500 and 400  $\text{cm}^{-1}$ . The "bumps" observed around 2000  $\text{cm}^{-1}$  are linked to a fault in the analysis equipment. Broad low-intensity absorption bands centered around 1634  $\text{cm}^{-1}$  on all the materials analyzed are attributed to water molecules or possibly to the other constituent radicals of amorphous aluminosilicates [52]. The bands located around 993-997  $\text{cm}^{-1}$  and 909-910  $\text{cm}^{-1}$  are respectively attributable to the symmetrical stretching vibrations of the Si-O-Si bond and the vibration of the Al-O bond in Kaolinite. The bands characteristic of the deformation vibrations of the Si-O bond of the quartz appear between 690 and 775  $\text{cm}^{-1}$ . The vibrations around 523  $\text{cm}^{-1}$  and 416-455  $\text{cm}^{-1}$  are respectively attributable to the deformations of the Si-O-Al<sup>IV</sup> and Si-O-Si bonds in the kaolinite [53]. These results are in agreement with those of the DRX (Fig. 6) and confirm the presence of quartz, kaolinite, illite, and/or smectite in the clays studied. The mineralogical composition of clay materials is in agreement with many studies carried out in the northern part of Cameroon [39, 53].



**Figure 7: Full FTIR Spectra of Samples**

### 3.2.2 Chemical composition

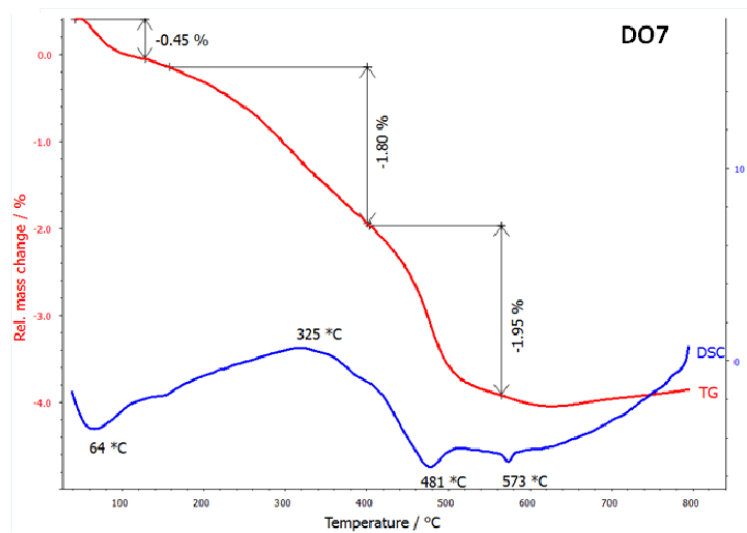
The chemical compositions of the materials studied are presented in Table 4. The clay materials are mainly composed of silica  $\text{SiO}_2$  (53.16 to 75.20%) and alumina  $\text{Al}_2\text{O}_3$  (8.10 to 14.74%). These two oxides are followed by  $\text{Fe}_2\text{O}_3$ ,  $\text{K}_2\text{O}$  and  $\text{CaO}$ . The proportions of  $\text{Fe}_2\text{O}_3$  are relatively low, with values generally between 2.39 and 5.20% for all the samples. The proportions of  $\text{K}_2\text{O}$ , and  $\text{CaO}$  are also low. With the exception of ME12, which contains 6.39% of  $\text{CaO}$ , all samples are below 2%. The weight percentages of  $\text{K}_2\text{O}$  vary between 1.23 and 4.77%. These values are in agreement with numerous studies [42-43, 55]. The high proportions of silica  $\text{SiO}_2$  indicate a high quartz content, implying a sandy fraction. They are confirmed by the results of XRD and particle size analysis. The reddish traces observed on the samples confirm the presence of hematite  $\text{Fe}_2\text{O}_3$ . The presence of K-feldspars may be associated with potassium oxide  $\text{K}_2\text{O}$  [43]. The Si/Al ratio ranging from 4.16 to 11.67% reflects the presence of 2/1-type clays [52, 45]. The MIA of clay materials varying between 19.05 and 58.11% shows that these materials are strongly altered [45]. These values also reflect the abundance of smectite in the materials. The TC22 sample records a high loss on ignition of around 12.73%, while those of the other samples vary between 1.23 and 8.47%. This loss could be due to the presence of smectite or kaolinite in different samples and/or may likely indicate the presence of organic matter [56].

**Table 4: chemical composition of clay materials (% by weight)**

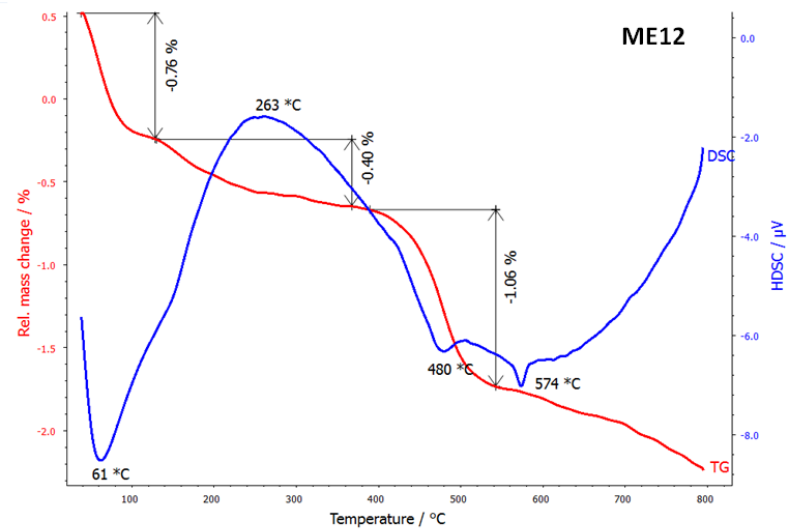
Sites	Doyang				Mendeo	Mobono	Hoppo	Mindif center	Tchouake	Gagadje
Samples	DO2	DO3	DO6	DO7	ME12	MO17	HO19	MI21	TC22	GA23
$\text{SiO}_2$	70,75	73,09	55,38	55,78	60,32	68,19	61,85	53,16	55,95	75,20
$\text{Al}_2\text{O}_3$	8,38	9,73	9,35	10,51	11,81	8,84	8,10	12,77	14,74	6,44
$\text{Fe}_2\text{O}_3$	3,70	2,88	4,80	4,27	5,27	3,10	3,13	5,20	5,37	2,39
$\text{CaO}$	0,47	1,64	1,63	1,45	6,39	1,27	1,29	1,87	0,75	1,00
$\text{MgO}$	0,21	0,34	0,42	0,32	0,45	0,43	0,34	0,37	0,61	0,22
$\text{SO}_3$	0,06	0,08	0,08	0,09	0,10	0,14	0,05	0,08	0,07	0,11
$\text{K}_2\text{O}$	1,58	1,51	2,06	1,85	1,37	1,58	2,44	4,77	2,76	1,23
$\text{Na}_2\text{O}$	0,17	0,64	0,37	0,28	0,27	0,70	0,35	1,25	0,42	0,18
$\text{P}_2\text{O}_5$	0,01	0,01	0,03	0,03	0,01	0,02	0,04	0,05	0,10	0,10
L.O. I	5,57	6,23	8,47	8,28	1,23	7,32	5,89	7,32	12,73	5,57
TOTAL	90,90	96,15	82,59	82,86	87,22	91,59	83,48	86,84	93,50	92,44
Si/Al	8,44	7,51	5,92	5,30	5,10	7,71	7,63	4,16	3,79	11,67
MIA	58,11	43,93	39,44	49,18	19,05	42,69	33,00	23,62	57,90	45,53
$\text{Fe}_2\text{O}_3+$ $\text{MgO}+$ $\text{K}_2\text{O}+$ $\text{Na}_2\text{O}$	5,66	5,37	7,65	6,72	7,36	5,81	6,26	11,59	9,16	4,02

### 3.2.3- Thermal analysis

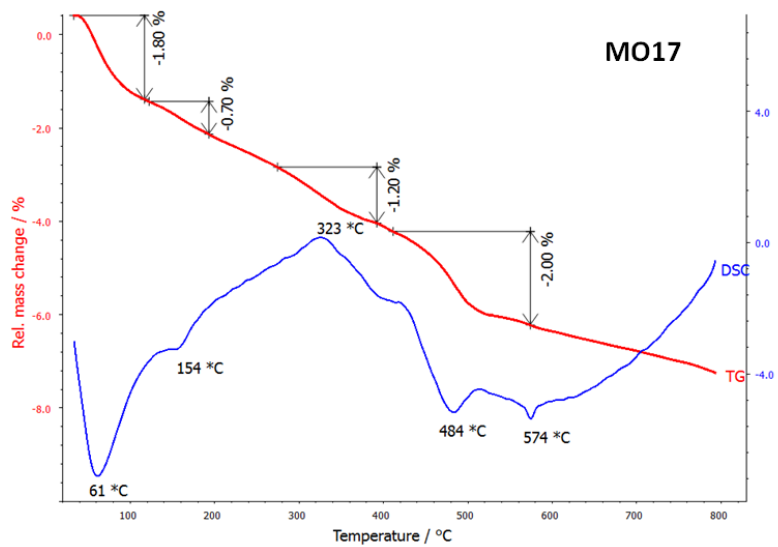
The differential thermogravimetric analysis (DTA/TG) carried out on clay materials is presented in Figure 8. These four thermograms all have the same appearance and each show five zones of anomalies (four endothermic peaks and one exothermic peak each). The endothermic peaks between 25 and 100°C are attributed to the loss of hygroscopic water, absorbed water, and the network of the material [57]. The corresponding mass losses are all less than 2%, with the exception of TC22, which is 3.25%. We can also observe, between 100 and 200°C, the peaks corresponding to the transformation of goethite into hematite [33]. The optimum temperatures for MO17 and TC22 are 154 and 153°C, respectively. These peaks are accompanied by mass losses of around 0.70%. The DTA curves of the materials (DO7, ME12, MO17, and TC22) show maxima at 325, 263, 323, and 351 °C, respectively, with corresponding mass losses of 1.8, 0.40, 0.29, and 1.56% on the TG curves between 210 and 400 °C. These exothermic peaks are attributed to the combustion of organic matter present in the sampled materials [58]. Between 420 and 510 °C, the endothermic peaks centered at 481, 480, 484, and 481 °C of samples DO7, ME12, MO17, and TC22, respectively, correspond to the dehydroxylation of kaolinite and smectite/illite minerals. This phenomenon leads to the conversion of kaolinite into metakaolinite [51]. These results are in agreement with the smectite/illite phase identified in XRD and FTIR. This interpretation is confirmed by the large mass losses at 1.95, 1.06, 2.00, and 3.01% of clays DO7, ME12, MO17, and TC22, respectively. The endothermic peaks observed at temperatures between 520 and 600 °C indicate the transformation of quartz alpha into quartz beta without mass loss [59]. Thus, the quantity and transformation parameters of quartz have an influence on the thermal transformation of clay materials.



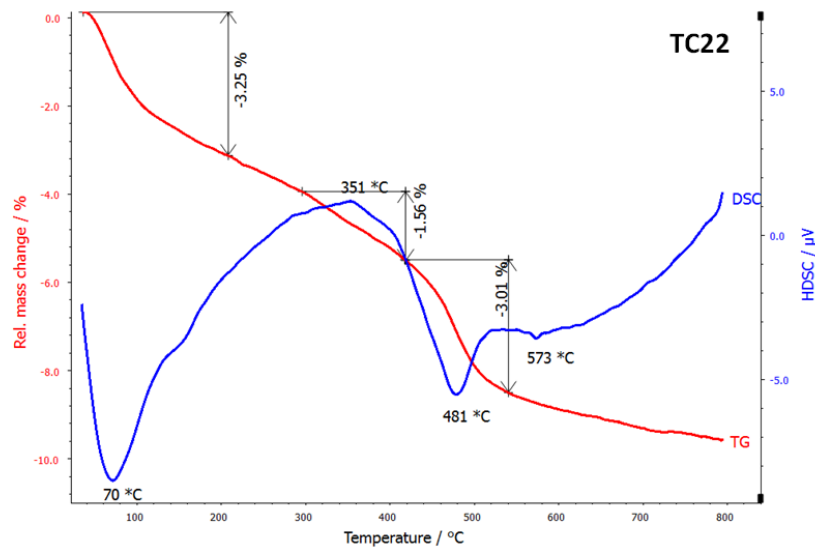
(a)



(b)



(c)



(d)

Figure 8: Differential thermogravimetric analysis of clays (a) DO7, (b) ME12, (c) MO17, (d) TC22

### 3.3- Firing properties

After drying for 24 hours, the clay samples DO7, ME12, MO17, and TC22 were fired at different temperatures (900, 1000, and 1100 °C). Sonority, coloration, water absorption, linear shrinkage, and flexural strength were measured. The firing temperature had an influence on all the properties mentioned above. Table 5 presents the sonority and color tests carried out. The sonority varies from slightly metallic to metallic. Samples DO7, ME12, and TC22 exhibit a metallic sound at all temperatures. Only MO17 is slightly metallic from 900 to 1000 °C and metallic at 1100 °C. This metallic sound reflects the maturity of the firing products and occurs when the cohesion is high [60]. Between 900 and 1100°C, their color varied between dull orange and orange, with the exception of MO17, which showed an orange color at all temperatures. The chemical composition of clays, their temperatures, and firing methods influence the color of fired products [61]. This color depends greatly on the small proportions of iron oxides in the form of hematite (2.39-5.20%) and aluminum oxide impurities (8.10-14.74%) when they are exposed to fire.

**Table 5: Color and Sonority tests**

Site	Sample	Temperature	Color	Sonority
Doyang	DO7	900	5YR 7/4 Dull orange	Metallic
		1000	5YR 6/8 Orange	Metallic
		1100	5YR 7/3 Dull orange	Metallic
Mendeo	ME12	900	7,5YR 7/6 Orange	Metallic
		1000	5YR 6/6 Orange	Metallic
		1100	5YR 6/4 Dull orange	Metallic
Mobono	MO17	900	5YR 7/8 Orange	Slightly metallic
		1000	5YR 7/8 Orange	Slightly metallic
		1100	5YR 7/8 Orange	Metallic
Tchouake	TC22	900	5YR 6/4 Dull orange	Metallic
		1000	7,5YR 6/6 Orange	Metallic
		1100	5YR 6/4 Dull orange	Metallic

Figure 9 and Table 6 show the results of the water absorption, linear shrinkage, and flexural strength tests performed. These parameters have frequently been used in quality control processes and manufacturing development stages of ceramic products [28 -29,33]. Water retention of clay materials is closely related to clay content and its characteristics, such as mineralogy and particle size [62]. The water absorption values (Fig.9.a) increase very rapidly from 900°C (5.8 to 15.0%) to 1000 °C (14.2 to 26.9%), before dropping to 1100 °C (11.9 to 14.2%). This increase is related to the phenomenon of swelling generally observed during the hydration of smectites [63], and the decrease indicates the vitrification of clays. At all temperatures, TC22 has the highest absorption capacities. This could be explained by its low proportions of sand and its high clay content compared to the other samples [9]. For all the samples (except TC22 at 1000 °C) the water absorption values are less than 20%, representing the recommended water absorption value for fired bricks [60].

**Table 6: Physical properties of fired products.**

Sample name				
	DO7	ME12	MO17	TC22
Temperature (°C)	Water absorption (%)			
900	12,2	14,5	5,8	15,0
1000	14,2	16,3	14,4	26,9
1100	13,4	11,9	13,7	14,2
Linear shrinkage (%)				

<b>900</b>	4,0	1,3	2,5	1,9
<b>1000</b>	9,7	2,6	3,7	1,3
<b>1100</b>	2,6	3,9	3,8	1,9
<b>Flexural strength (MPa)</b>				
<b>900</b>	7,6	6,4	3,9	16,7
<b>1000</b>	8,7	16,0	5,4	11,2
<b>1100</b>	5,8	10,3	4,1	7,4

It can be observed that the linear shrinkage values (Fig.9.b, Table 6) of all the samples (except DO7) increase slightly with temperature. This increase reflects the densification of fired clay. They were less than 5% (900 °C), 10% (1000 °C), and 4% (1100 °C). Sample DO7, which has low plasticity (13.3%), high clay content (33.3 %), and low melting agents (6.72 %), shows the highest shrinkage. The low proportions of melting agents (5.81-9.16%) present in the smectites do not favor the reduction of the vitrification temperature, resulting in low densification. The latter strongly affected the linear shrinkage of the fired materials. Recommended shrinkage values for brickmaking should be less than 4% [64]. The materials showed values within the recommended limits (exception of DO7 at 1000 °C).

The results of the flexural strength test of samples fired at different temperatures are shown in figure 9.c. Resistances at 900 °C range between 3.9 and 16.7 MPa, with the high value corresponding to TC22. These resistances increase with temperature (1000 °C) from 5.4 to 16 MPa due to the reduction of porosity, except for TC22, which decreases to 11.2 Mpa. These peaks indicate the optimum firing temperature. At 1100 °C, the resistance decreases (4.1 to 10.3 MPa) for all the samples. This decrease is caused by the expansion and deformation of the ceramic bodies. At 1000 °C, all samples were vitrified. An early vitrification of TC22 at 900 °C is observed. All materials with strengths  $\geq 7$  Mpa (except MO17) could be used for the manufacture of fired brick [65]. The minimum flexural strength (7 MPa) of these clays is reached at 900 °C compared to that of Ndop Plain (Northwest, Cameroon) [66], which is only reached after 1050 °C.

Overall, from these firing properties, it can be seen that the TC22 sample has poor properties due to its high clay content (48%), making it less suitable for the manufacture of bricks and tiles. It requires the addition of degreaser (sand) or additives (lime). The addition of flux to the sample MO17 is necessary to decrease the sintering temperature. ME12 can be used to make bricks without the addition of any other materials. Clays must be balanced using appropriate admixtures [61].

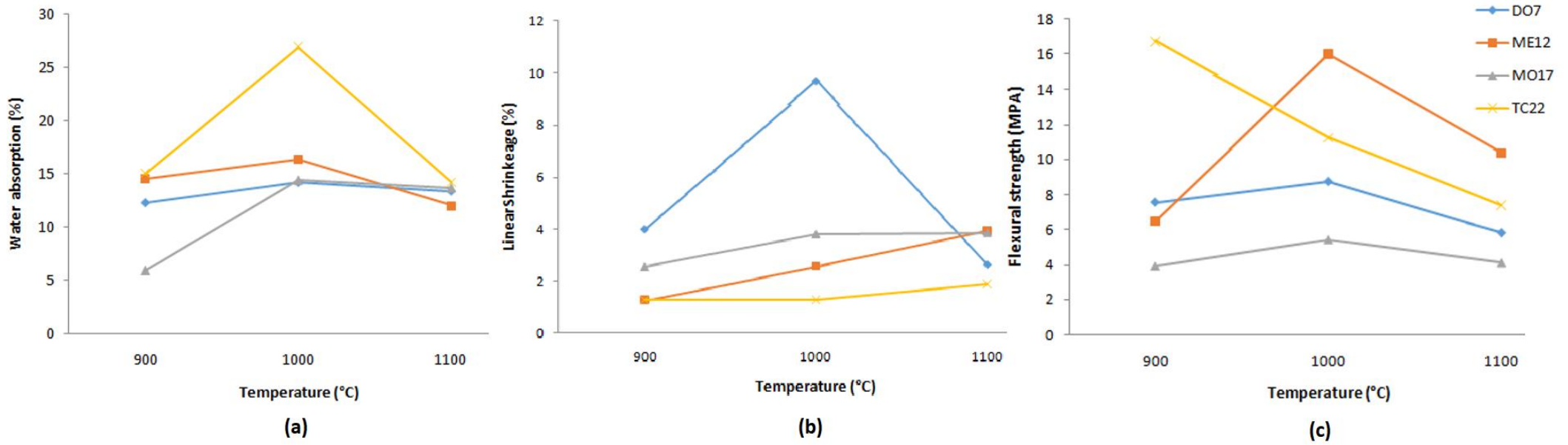


Figure 9: Firing characteristics of selected clay samples from Mindif as a function of firing temperatures (a) water absorption, (b) linear shrinkage, (c) flexural strength

## Conclusion

The use of a material requires the control of its physical, chemical, mineralogical, thermal, and geotechnical properties in order to determine its potential uses. The clay soils in the commune of Mindif (Far North, Cameroon) were subjected to several analyses. Based on the results obtained, we draw the following conclusions:

- ❖ The grain size of clay materials consisted of gravel (0–4%), sand (25–74%), silt (6–27%), and clay (10–48%). The plastic limit test showed that the TC22 clay is very plastic and the others have low plasticity. Samples DO2, DO3, MO17, HO19, MI21, and GA23 show an extrusion behavior (non-plastic clay) that is not acceptable for the production of ceramics (while DO6, DO7, ME12, and TC22 are). These samples require the appropriate addition of a plastic material to improve their workability.
- ❖ XRD diffractograms showed that the clays were composed of quartz, feldspar, illite, smectite, kaolinite, and traces of albite, goethite, and calcite. These crystalline phases are in agreement with the absorption bands of the FTIR infrared spectra and the DTA/TG curves.
- ❖ The clays were mainly composed of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, followed by Fe<sub>2</sub>O<sub>3</sub>, CaO, K<sub>2</sub>O, and MgO. These minerals are very good melting agents in the manufacture of ceramics, as observed by the pottery activities carried out in the localities.
- ❖ The firing properties of clay materials are sufficient for the manufacture of ceramics. Nevertheless, the sample MO17 requires an adequate adjustment of its particle size distribution, with or without additives, above 1000°C. TC22 is unsuitable for brickmaking due to its high clay content. While ME12 is suitable without addition to the production of ceramics.

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