Mineralogical, Physico-Chemical Characterization And Ceramic Properties Of Babouantou Clay Materials (Haut-Nkam, West Cameroon)

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***Abstract*—This work concerns the mineralogical, physico-chemical characterization and ceramic properties of the Babouantou clay materials (lower area of the Western flank of the Bana volcano-plutonic complex). Four samples of clayey materials were collected from wells in Nguieu and Batchieu localities precisely from the illuvial and elluvial horizons (BE1, BE2, BE3 and BE4). Mineralogical analysis showed that they are made of relictual minerals (quartz and feldspar), clays mineral (kaolinite), weathering minerals (illite), hydroxides (gibbsite) and oxyhydroxydes (goethite). The chemistry shows a silico-aluminous character with moderate iron (Fe_2O_3) content and small quantities of alkali (Na_2O, K_2O) and alkaline earth metals (CaO). It is poor in organic matter (< 1%). The physical parameters display a continuous and wide spread granulometry with variable plasticity (5-13).**

The ceramic tests conducted on fired bricks at 900°, 1000°, 1100°C respectively, presented some standard physico-mechanical properties which are acceptable in the domain of traditional ceramics. Based on test performed on fired bricks, these materials can be classified as either malleable fusible clays (samples BE1, BE3, BE4) and malleable refractory clays (sample BE2). Therefore, these clay materials can be used in ceramics at 1000°C of firing after crushing and screening.

***Keywords*—Babouantou, clay materials, mineralogical, physico-chemical analysis and ceramics properties**

I. INTRODUCTION

Clays have been used by humans since older-days to make fire or growth bricks, kitchen materials and to design products further according to their particular properties before and after firing [25,31,33]. Today, clays are the most important materials used by manufacturing and environmental industries. With regards to their wide industrial applications, they are gradually becoming an indispensable tool for development in developing as well developed countries.

They play a vital role in economic development [16,23,32,47].The use of clay covers many applications such as: ceramics, paper, paint, [10,36,50] rubber and plastics, insecticides, making of additives for food [1,45], cosmetics, pharmaceuticals, drilling fluids, fertilizer carriers and geochemical barriers [6,21,40,46].

In ceramic industry, properties of immense importance include: mineralogical and chemical composition [45], malleability, thermal behavior, color and strength after cooking [4,9].

In Cameroon, most ceramic products are imported [34,45]. This point can be explained by the lack of local industries or poor evaluation of potentially available clay materials. For some decades, a lot of research has been made on clay materials in Cameroon, particularly those containing kaolinite [7,29,34,37,38,45] and smectite [28,35].

These studies were concerned with the mineralogical, physico-chemical properties, mechanical strength after firing and clay materials activation in other to increase reliable data base for the starting of industrial exploitation.

This paper is aimed at characterizing the mineralogical, physico-chemical and ceramic properties of some clay materials from Babouantou (West-Cameroon) in order to increase the data base of industrial application of Cameroonian clays.

II. MATERIAL AND METHODS

A. Raw materials

The locality of Babouantou (Figure 1) is situated at the lower Western flank of the Bana volcano-plutonic complex, 15 km from Bafang.

The prevailing climate is equatorial and a surrounding pseudo-altitude tropical type, with two seasons: a long rainy season from March to October (eight months) and a short dry season that range from November to March (4 months). The average annual precipitation is about 1734 mm and the average temperature is 22°C [13].

The hydrographic network of the study area is dendritic in the South and dendritic to Sub-parallel in the North. The landscape presents isolated hills of different altitudes separated by low marshy areas which serve as potential sites for clay deposit.

The samples analyzed during this study were collected from three trial pits particularly in elluvial and illuvial horizon developed on porphyritic granite in Babouantou.

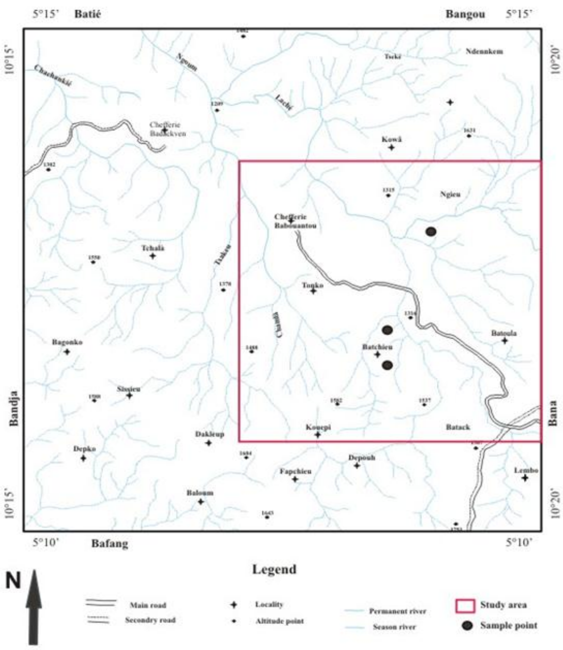

Figure. 1. Hydrographic map and sample point of clay materials

B. Mineralogical analysis

X-ray diffractometric method was used for mineralogical analysis in the mineralogy laboratory of the University of Liège (Belgium). Before analysis, the samples were first crushed in an agate mortar until the particle size is smaller than 50µm, then, dried at 105°c for 24 hours. Twelve milligrams of *diffractogram* each sample were placed on the sample carrier of the diffractometer.The scanned angular range is between 5 ° ≤ 2θ ≤ 90 ° with an angular pitch of 0.020 ° C. The copper Kα1 radiation (λ = 1.5406 A °) used was produced at a voltage of 40 KV and a current 30 mA**.** The ASTM sheets are used to identify the minerals on.

C. Physico-chemical analysis

Particle size distribution and plasticity were considered as the two important parameters to evaluate the suitability of clays as raw material in ceramic domain [14,36]. Bulk chemical composition was also considered as a starting point for ceramic bodies [30].

The particle size was determined by wet sieving for fraction higher than eighty micron (80µm) and sedimentry for fraction less than eighty micron (80µm) according to [41].

Plasticity is a technological parameter that influences the characteristics of ceramic materials [19]. It was performed on fractions less than 400µm which consist of varying the water content on the material in order to evaluate its consistency.

The method called Atterberg's limits was used in order to define the consistence limit between the solid and the plastic state (plastic limit: W_P) and the plastic state to the liquid state (liquid limit: W_L). The interval between the plastic limit and liquidity defines the plasticity index (plasticity index: I_P).

The plasticity test was carried out according to the AFNOR prescription [42] in the material laboratory of MIPROMALO (Mission Local Materials Promotion) Yaoundé Cameroon.

Methylene blue test was used to determine the amount of clay content (quality and quantity) on clayey materials.

Chemical analysis was performed by X-ray fluorescence spectrometer. It is a quantitative analysis of the most stable oxides (major elements and minor elements) in the clay materials. The major elements were determined by assay ICP-AES (Inductively Coupled Plasma-Atomic Emission Spectrometry).

The proportions of major elements were calculated and expressed in the form of oxides percentage.

Organic matter was determined indirectly by oxidation with a potassium dichromate (K_2Cr_2O7) solution in a highly acidic medium (H_2SO_4). The potassium dichromate solution is used in excess**.** This excess potassium dichromate is titrated with ferrous sulfate ($FeSO_4$, $7H_2O$) to determine the quantity that is needed for the oxidation of organic carbon. The formula below is then used to compute the quantity of organic matter.

$$\%M.O = 1.724 \times \%C.O \qquad \textbf{(1)}$$

M.O: organic matter**;**

C.O: organic carbon

D. Ceramic properties

The samples were homogenized and previously separated in four parts. Half a fraction is removed and dried in an oven for 24 hours at a temperature of 105°C.The particle size was reduced by crushing in a porcelain mortar and sieved using a mesh of diameter 1 mm. The under flow obtained was humidified at 5%.

The test specimens (80mm× 40mm ×10 mm) were made using a 50 kN hydraulic press, and then dried at room temperature and in an oven at a temperature of 105°C for twenty four hours respectively.

The dried samples were fired at 900°C, 1000°C and 1100°C for a period of 5 hours at a heating rate of 5°C/min in an electric furnace-type FP 34 G in MIPROMALO ceramic laboratory.

The ceramic properties like colour, tone, firing shrinkage, compressive strength, flexural strength, water absorption were evaluated using [3] respectively

III. RESULTS AND DISCUSSION

A. *Morpho- structural description of profiles*

- Nguieu Profile

This profile (figure 2a) has as coordinates (North: 05°12'37, 9''; East 10°19'18, 5''; altitude: 1298 m). It is made of five horizons and the depth is about three hundred and seventy centimeters (370 cm)

Isalteritic horizon greater than 360 cm thick. It is reddish at polychrome trend. It has a clayey sand texture and a massive structure. The figurative elements are the ghosts' granite and fine quartz gravel**.** Its limit is distinct and progressive.

Alloteritic horizon has a thickness of about 25cm and is dark gray (5YR4 / 1: dark gray). Its texture is sandy silt and structure is lumpy. Its upper limit is frank and the lower limit is progressive.

Iluvial horizon (210 cm) is moonlight black (5YR4 / 2: dark gray). Its texture is sandy clay and the structure is massive. The limits are progressive. The upper limit is covered by a thicks and bars of about 20 cm. This sandbar has a particular structure and as andy-loam texture.

eluvial horizon (60 cm) is yellowish to light red (7,5YR6 / 8:reddishyellow).

Its texture is sandy loam and the structure is lumpy. Its lower limit is brutal while the upper limit is diffuse

Mineral- organic horizon (50cm) is reddish brown (5YR4 / 4: reddish brown). Its texture is silty sand and the structure is lumpy. It contains many rootlets.

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- **Batchieu Profile (Upper slope)**

This profile (figure 2b) has as coordinates (North:10°18'13,70;East:11'45,3"";altitude:1312m). The depth is about 160 centimeters. It is constituted of four horizons.

Alloteritic horizon is constituted of a slightly thick alteration zone embedded in greyish matrix which characterizes the granite fragments in weathering process. The texture is sandy clay and the structure is massive. Its limit with the iluvial horizon is regular.

Iluvial horizon (62 cm)is light gray (5Y7 / 1: light gray). The texture is sandy clay and the structure is friable**.** Its boundaries are diffuse and regular. The sample (BE2) has been collected at this depth.

Eluvial horizon (64 cm) is pale brown (2,5Y8 / 2: pale brown). The texture is sandy clay and the structure is friable. On this horizon, we observe quartz gravel scattered throughout. Its upper limit is progressive.

Organo-mineral horizon is illustrated by the thick layer of about 24 centimeters and is yellowish red (5YR5 / 6: yellowish red). The texture is silty sand and the structure is friable. Here, we observe many roots with fine quartz gravel ranging from millimeter to centimeter

- **Batchieu Profile (middle slope)**

This mid-slope profile is described in a well of about 640 centimeters at coordinates points (North: 05 °11'45,4', East: 10°13'15,1'; altitude 1323m). It is constituted of six horizons (figure 2 c)

Isalteritic horizon has a variable coloration (reddish to yellowish). Its thickness is greater than 590 centimeters and the texture is sandy to sandy clay with a massive structure is massive. This horizon shows the ghosts of granite on weathering process. Its upper limit is diffuse and progressive.

Alloteritic horizon is about 80 centimeters thick. thas varying colours (brown, gray, and pale yellow).The texture is sandy clay and the structure is polyhedral. Its boundaries are irregulars.

Iluvial horizon (250 cm) is reddish yellow (5YR6 / 8: reddish yellow). Its texture is sandy clay and the structure is lumpy. Its limits are regulars. This horizon is crossed by a thick relictual vein of quartz-feldspar of about 14 centimeters. The sample (BE3) was taken from this horizon.

Primary eluvial horizon (125 cm) is yellow brown (10YR5 / 8: yellow brown). The texture is clay loam and the structure is polyhedral. The sample (BE4) was taken in this horizon.

Secondary eluvial horizon (120 cm**)** is yellowish red (5YR4 / 6: Yellow red). The texture is sandy clay and the structure is massive. Its upper and lower limits are progressive.

Organo-mineral horizon is represented by a thick layer of about 15 centimeters. The colour is dark red brown (2,5YR3 / 3: dark reddish brown). Its structure is lumpy and the texture is sandy loam. It contains rootlets. Its lower limit is diffuse.

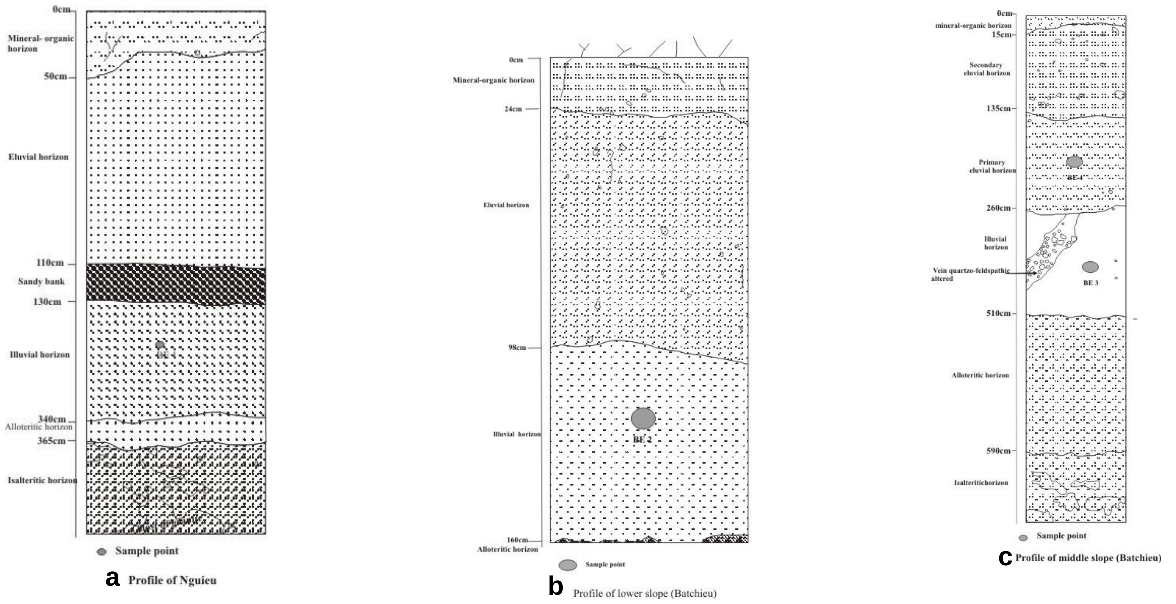

Figure.2. Profiles of different sample points

B. Description of sample collected in study area

TABLE I. CHARACTERISTIC OF SAMPLES COLLECTED FROM TRIAL PITS IN DIFFERENT LOCALITIES OF BABOUANTOU

Locality	Sample	Position	Depth (cm)	Thickness (cm)	Texture	**Colour**
Nguieu	BE1	Illuvial horizon	130	210	Clay sandy	Light black (5YR4/2)
Batchieu	BE2	Illuvial horizon	98	62	Clay sandy	greyish (5Y7/1)
	BE3	illuvial horizon	260	250	Sandy Clay	Reddishyellow 5YR6/8
	BE4	Elluvial horizon	135	125	Sandy Clay	Yellow brown 10YR5/8

C. Mineralogy

The main mineral phases (Figure 3) which are well expressed in these materials are: kaolinite (K), quartz (Q), illite (I), gibbsite (Gi), feldspar (Fd) and goethite (Goe). Table 2 below shows the relative proportions of minerals based on the peak intensity. However, kaolinite, the most requested mineral in the ceramic industry [45]is abundant (32% - 41%), and feldspar and iron that carry ceramic properties. Thus, these materials can be used in ceramic paste.

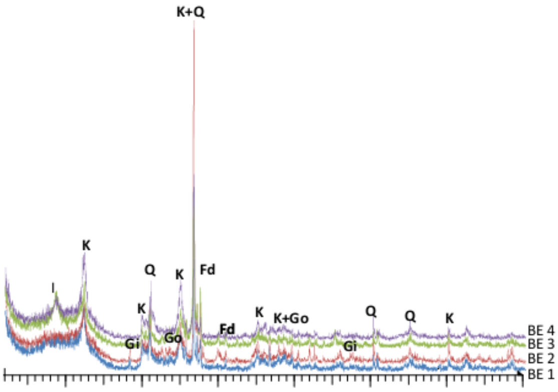

Figure. 3. .X-ray diffraction pattern (Cu K$\alpha_{1,2}$) of randomly non-oriented bulk sample showing the composition of sample analysis: **I**: illite; **K**: kaolinite; **Gi**: gibbsite; **Go**: goethite; **Q**: quartz; **Fd**: feldspath

TABLE II. MINERALOGICAL COMPOSITION OF SAMPLE STUDIED BASE ON SEMI QUANTITATIVE ANALYSIS (%)

Minerals	Kaolinite	Illite	Quartz	Feldspath (%)	Gibbsite	Goethite
BE1	35	18	24	16		7
BE2	32		26	27	8	7
BE3	33	31	8	21		7
BE4	41		20	10		29

D Physico-chemical analysis

- The particle size distribution of clay materials (table 3) is homogeneous and wide spread where all fractions are represented. Sample BE2 is characterized by a high proportion of fine sand (48%).Sample BE1 has the highest rate of clay (28%) and sample BE3 has the highest percentage of silt (22%). Generally, the clay content of the material analyzed is between 16% and 28%. The homogeneity and particle size distribution is consistent for the manufacture of firing bricks[22] and requires another processing such as crushing and screening before being used in pottery.
- Atterberg's limit**:** table 3 shows that the samples BE1, BE2, BE3 and BE4 may contain32%, 29%, 57% and 57% of water without sinking under their own weight. In addition, they can deform

plastically with lower water contents of 25%, 24%, 44% and 45% respectively.
The low plasticity (5-13) is due to the high content of sand [22].Which is confirmed by the low absorption of methylene blue (table 3) by the samples.

According to the diagram, (figure 4), these samples are suitable for firing bricks.

TABLE III. PHYSICAL CHARACTERIZATION OF CLAYS SAMPLES COLLECTED ON BABOUANTOU LOCALITY (%)

Physic parameters	Samples types	BE1	BE2	BE3	BE4
Particule size	Gravel	0	6	3	6
	Sand	18	48	12	32
	Fine Sand	42	22	42	38
	Silts	14	8	22	8
	clay	28	16	21	1
Atterberg limit	W_l	32	29	57	57
	Wp	25	24	44	45
	Ip	7	5	13	12
Rate of methylen blue	VBS	1,13	0,93	2,80	1,33

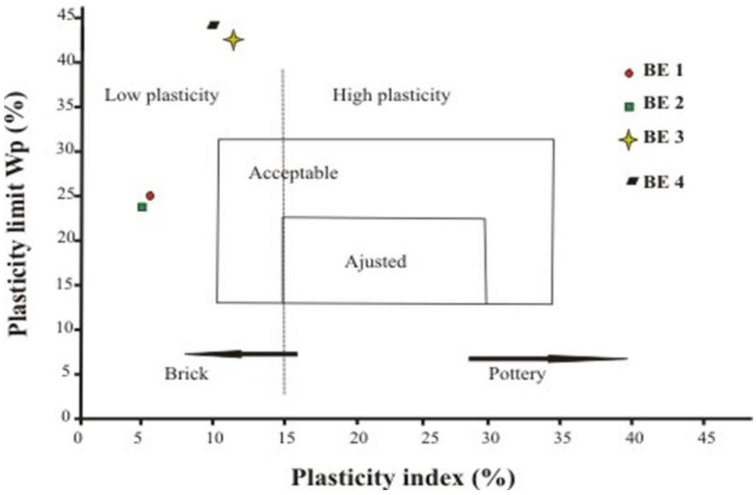

Figure. 4. Bain &Highley Diagram (1979) showing the position of our sample

- The geochemical analysis in table 4 shows a silico-aluminous character with moderate quantity of alkali and alkaline earth metals. In the words, the variation of potassium (K_2O: 2,74 -5,35) and iron (Fe_2O_3: 1,94 – 8,39) confirms the presence of feldspar and illite [45] and an iron compound like hematite which is of big importance in the ceramic industry.

The SiO_2 / Al_2O_3 ratio is greater than 2% and the Fe_20_3 / Al_20_3 ratio lower than 1% respectively reflecting the abundance of quartz [37,45] and the presence of iron compound.

- Organic matter content is relatively low (<1%) and varies from 0.75% to 0.91%.

TABLE IV. MAJOR ELEMENT COMPOSITIONS (WT %) AND ORGANIC MATTER CONTENT IN CLAY MATERIALS FROM BABOUANTOU

Major elements (%)	BE1	BE2	BE3	BE4
SiO_2	55,4	60,35	55,89	52,74
Al_2O_3	23,13	21,83	20,38	23,27
Fe_2O_3	4,13	1,94	6,98	**8,39**
MnO	0,02	0,01	0,02	0,01
MgO	1,04	0,51	1,65	0,75
CaO	0,32	0,18	0,21	0,02
NaO	0,10	0,14	/	/
K_2O	**2,74**	**4,19**	**5,35**	**2,55**
TiO_2	1,52	2,14	1,43	0,99
P_2O_5	0,11	0,09	0,22	0,19
LOI (1000°)	11,04	10,88	7,36	8,15
Total	99,99	100	99,99	99,99
$SiO_2/Al2O_3$	**2,39**	**2,76**	**2,74**	**2,26**
Fe_2O_3/Al_2O_3	**0,17**	**0,1**	**0,34**	**0,36**
Organic matter	0,91	0,80	0,69	0,75

E Ceramic properties

- The colour of fired bricks (table 5) changes with an increase in temperature. This change in colour is related to the presence of goethite **(**FeOOH) or hematite [45] and the presence of Calcium (CaO), Magnesium (MgO), Titanium (TiO_2), Aluminum oxide (Al_2O_3) or temperature of the oven [5].
- Sound transmission is summarized on table 4.We noticed that, the specimens BE1, BE3 and BE4 have a metallic sound which is a characteristic of good quality ceramic product [12]. However, the BE2 specimen doesn't have a metallic sound in the three temperature ranges. This lack of metallic sound is explained by the absence of conversion reaction during firing or the presence of refractory oxides such as silica (SiO_2, aluminum oxide (Al_2O_3), calcium oxide (CaO), magnesium oxide (MgO) which has not yet reached their respective melting temperatures (1710 ° C, 2020 ° C, 2570 ° C and 2800°C).

TABLE V. PHYSICAL PROPERTIES OF CERAMIC TEST

Ceramic properties	Sample	900°C	1000°C	1100°C
Color	BE1	Yellowish light	Yellowish light	Yellowish light
	BE2	white Yellow	Whitishyellow	Whitishyellow
	BE3	Light red	Reddish	Red
	BE4	Light red	Reddish	Reddish
Sonority	BE1	Metallic sound	Metallic sound	Metallic sound
	BE2	Non metallic sound	Non metallic sound	Non metallic sound
	BE3	Metallic sound	Metallic sound	Metallic sound
	BE4	Metallic sound	Metallic sound	Metallic sound

- Firing shrinkage of the samples BE1 and BE4 (Figure 5) increases gradually from1000 °C as a result of chemical transformation [17]marked by the passage from metakaolin to mullite [27] or the formation of a spinel phase or the appearance of alumina [45]. In addition, the linear shrinkage of the sample BE3 increases with temperature thus reflecting the vitrification initiated at temperatures below 900°C [49]. Furthermore, nonlinear shrinkage for the BE2 specimen confirms the absence of chemical reaction during sintering or excess degreaser [26].
- Compressive strength is illustrated in figure 6. In this figure, we observed that the BE1, BE2 and BE3 specimen reach their maximum resistance at 1000°C with values between (10-20 MPa) generally recommended for traditional ceramics [12,20]. In contrary, the BE4 specimen begins its compressive strength from 1000° C. This behavior can be explained by theironcontent (8.9%> 6% which is recommended for traditional ceramics [45]whose melting point is at1200 ° C where an eutectic

mixtures is formed [24].This elevated temperature of iron confers poor textural properties to the sintered products

- Flexural strength of ceramic specimen (figure 7) increases gradually with temperature.

This increase shows that, the vitreous phase in charge of the mechanical properties has not reached its optimum [11]. However, the flexural strength ranges from0.36 to 4.32MPa, but remains lower than 5 MPa which corresponds to the lower limit for traditional ceramics [12].

- Water absorption **(figure 8)** decreases from1000 °C following the appearance of the vitreous phase which fills the pores between the grains during firing [24].Thus, the temperature of 1000 °C is recommended to reduce the porosity of the products during sintering.

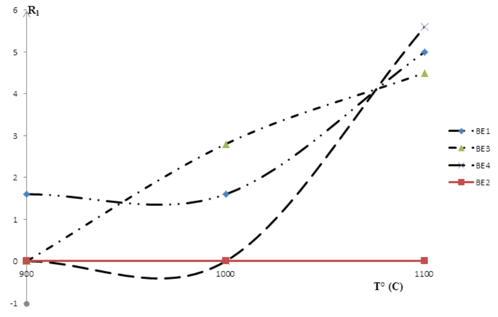

Figure.5. Variation of linear shrinkage with the temperature

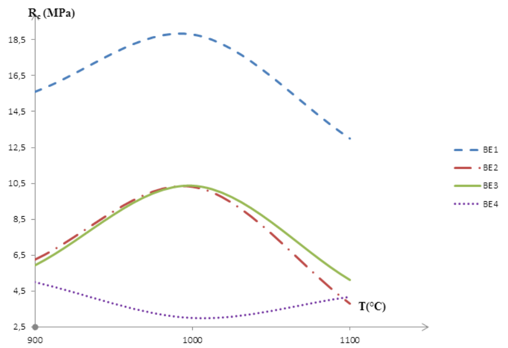

Figure. 6. Variation of compressive strength with the temperature

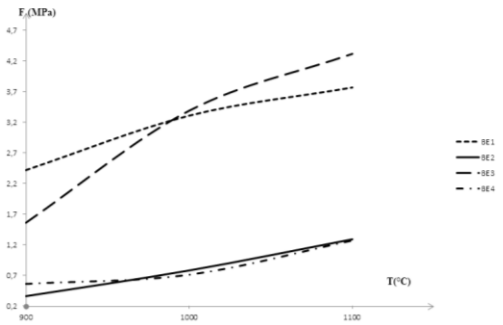

Figure. 7. Variation of flexural strength with the temperature

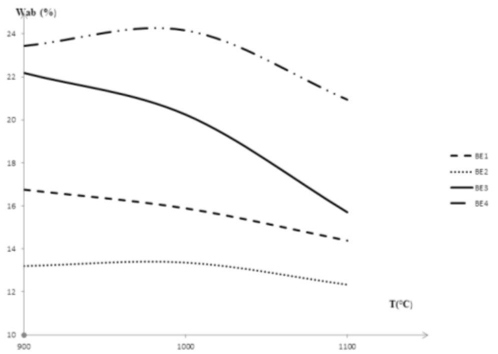

Figure. 8. Variation of water absorption with the temperature

IV CONCLUSION

The Babouantou clay materials are developed on porphyritic granite and concentrated principally on the iluvial and eluvial horizons. The mineralogical analysis of four samples (BE1, BE2, BE3 and BE4) is made up essentially of kaolinite, illite, quartz, orthoclase, goethite and gibbsite accessorily. Physical properties showed a homogeneous swide spread granulometry. It has a low plasticity (5-13) and confirms the low methylene blue absorption rate. Chemical analysis reveals the silico-aluminous character of all samples with varying amounts of iron (FeO) and smaller alkaline (Na_2O, K_2O) and alkaline earth metal (CaO).

The ceramic test on firing bricks reveals that the physical parameters (color, sound, linear shrinkage, absorption rate) and mechanical parameters (flexural strength and compressive strength) are acceptable in the standards of traditional ceramics. According to the plasticity and sintering temperature, the samples BE1, BE3, BE4 are a little bit plastics and fusible while sample BE2is a bit plastic and refractory. These clay materials may be used for the manufacture of firing bricks and roofing tiles after crushing, sieve and sintering at 1000°C.

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