Mineralogy, geochemistry and physico-mechanical characterization of clay mixtures from Sa'a (Center Cameroon): possibly use as construction materials

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Abstract

Six lateritic clay samples and lateritic and alluvial clay mixtures were studied in order to evaluate their potential use as raw material for bricks and tiles manufacturing. Their mineralogical and geochemical compositions were determined by X-ray diffraction and X-ray spectrometry, respectively, together with some physico-mechanical characteristics. The main minerals for the raw materials are kaolinite, quartz, goethite, hematite, and muscovite. Fluxing oxides are in low proportion (≤ 1.55 wt%) and causes insufficient sintering during firing. The flexural strength (FS) values are lower than 2 MPa for lateritic clay bricks, due to the Fe_2O_3 content. This justifies their amendment with alluvial clays from the near Sanaga River. The lateritic and alluvial clay mixtures were done with respectively 100, 80, 60, 40, and 0 wt% of lateritic clay and fired at 900, 950, 1000, 1050, and 1100 °C. Linear shrinkage values increase with temperature and globally with alluvial clay content. Except for one sample-based product at 900 and 950 °C, water absorption values overall remain lower than 20% which is adequate for dense bricks and roofing tiles. For the mixtures, FS values globally increase with alluvial clay content except for a few samples at some temperatures. The studied clay mixtures can be used for bricks manufacture at the five tested temperatures. Some compositions with 40, 60, and 100 wt% of alluvial clays may be used for the production of roofing tiles above 1000 °C. Fluxes amendment is needed at 1050 and 1100 °C for other mixtures to increase their FS.

Keywords Lateritic and alluvial clays · Mineralogical and geochemical features · Linear shrinkage · Water absorption · Flexural strength · Bricks and roofing tiles

1 Introduction

The interest of clay material is not to be demonstrated either by their areas of use [1], or their abundance. Many works have focused on the valuation of these materials during the last decades. It, however, arises the problem of quality than quantity. The limiting factor in the use of lateritic clays (residual clays) is their high iron (Fe_2O_3) content [2, 7]. What do we then do with these abundant

materials, which by their genesis feature has a high level of Fe_2O_3 resulting in low mechanical properties of the final products for the recommended uses? Many authors have tried to improve the properties of these clayey materials. Among them, Andji et al. [2] proposed the addition of calcium clay (10 wt% CaCO_3) to significantly improve the mechanical strength of Gounioubé clays (Ivory Coast), while Ergul et al. [8] proposed the substitution of clayey materials by basaltic tuffs. The approach of lateritic and

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alluvial clay mixtures has also been addressed in Cameroon by Ngon Ngon et al. [5] and Ntoulala et al. [6]; Onana et al. [7]. Six lateritic clay samples have been collected and analyzed in the area of Sa'a. Experimental results obtained have shown the need to amend these materials. The aim of the present work is to highlight the effect that would have an addition of alluvial clays on the technological properties of lateritic clays of Sa'a to bring out their interest in construction.

2 Geographical and geological setting

The morphology of the study area shows quartzitic reliefs with asymmetrical flanks in the Sa'a area. There are two types of hills: half-orange-shaped hills and asymmetrical hillsides. The half-orange-shaped hills are few in number. The asymmetrical hills are separated by more recessed valleys. The current climate is of transitional equatorial type [9]. The total annual rainfall is between 1400 and 1500 mm. Mean annual air temperatures range from 22.9 °C to 25.8 °C with minimum temperatures in august (19.1 °C) and highs in March and April (31.1 °C). This area belongs to the Sanaga watershed, the largest in Cameroon. The hydrographic network is dense and structurally parallel to the North and dendritic to the South (Fig. 1).

The basement of the Sa'a area (series of Sa'a) consists of rocks belonging to the pan-African mobile zone [10, 11]. The geological formations are mainly metamorphic rocks consisting of mica schists, micaceous quartzites, and gneisses. Mica schists are flaky rocks formed of muscovite, quartz, biotite, and garnet. They show intercalations of quartzites. Micaceous quartzites are the most abundant rocks in the study area and consist essentially of quartz associated with muscovite [11]. They form thick benches and are at the origin of more or less uneven microreliefs frequent in the zone. Gneisses are in association with mica schists and micaceous quartzites. They are fine-grained rocks with regular quartzite intercalations. The study area is mainly the domain of ferrallitic soils. They are of variable thickness (2–5 m) depending on the nature of the bedrock and are mainly red at altitudes greater than 600 m, yellow under 500 m and ocher between these altitudes.

3 Materials and methods

Field investigation allows the collection of six lateritic clay samples by shaft sinking (Fig. 1). They are indexed EB, EY, KK, NL, NM, and PO. Alluvial clay sample (AL) was collected in the swampy valley of the Sanaga River.

Fig. 1 Study area and samples location

The mineralogy of samples was determined by X-ray powder diffraction (XRPD) on powder samples. The analytical instrument used is a PANalytical X'Pert Pro diffractometer with a monochromator equipped with a cobalt K α radiation ($\lambda = 1.7890 \text{ \AA}$) over a range of 2.5 to $35^\circ 2\theta$ and a step size of $0.05^\circ 2\theta/\text{min}$ at 40 kV and 45 mA . Infrared spectra were recorded in diffuse reflectance mode using a Bruker Alpha-P Fourier Transform Interferometer. The spectra were recorded from 4000 to 400 cm^{-1} with a resolution step of 4 cm^{-1} . Chemical analysis for major elements was done after sample ignition by X-ray fluorescence on PAN analytical Axios Advanced PW 4400 equipment. Titrimetry is used for iron (II) oxide (FeO) dosage. The color of dry and fired samples was determined using the Munsell Soil colour charts.

Particle size data were obtained by sieving (AFNOR, 1996) for sizes greater than $80 \text{ }\mu\text{m}$ and by sedimentation (AFNOR, 1992) for sizes less than $80 \text{ }\mu\text{m}$.

The liquid limit (LL) was measured by Casagrande dish method and plastic limit (PL) by roller method. Measurements were realized according to ASTM D4318–2005 standard. Plasticity index (PI) was calculated as the difference between LL and PL.

For each lateritic clay sample, lateritic and alluvial clayey mixture was done with, respectively, $100 \text{ wt\%}$, $80 \text{ wt\%}$, $60 \text{ wt\%}$, $40 \text{ wt\%}$ and $0 \text{ wt\%}$ of lateritic clay. For the obtained mixtures, four test briquettes with length $\times$ width dimension of $8 \times 4 \text{ cm}$ were elaborated using a laboratory hydraulic press. For each briquette, a mixture of 100 g of clay associated with 17 – 20% of water (on dry basis) was used. Bricks were air-dried for 7 days and oven-dried at 105°C for 24 h to remove moisture prior to its firing. The firing was done in a muffle furnace with a maximum temperature of 1250°C . These briquettes were subjected to five firing temperatures: 900 , 950 , 1000 , 1050 , and 1100°C . For all firing cycles, a heating rate of $5^\circ \text{C}/\text{min}$ and soaking time of 2 h at the required temperature was observed before furnace cooling at room temperature.

Linear shrinkage (LS) was determined using Eq. (1) below (ASTM C531–2000):

$$\text{LS}(\%) = [(L_0 - L_1)/L_0] \times 100 \quad (1)$$

where L_0 is the initial length (mm) of the brick after drying at 105°C for 24 h and L_1 is the length (mm) after firing.

Water absorption (WA), from Eq. (2) below, is evaluated according to the ASTM C20–2000 standard using the Archimedes' water displacement method [12]:

$$\text{WA}(\%) = [(W_2 - W_1)/W_1] \times 100 \quad (2)$$

where W_2 is the mass of 24 h water-soaked specimen and W_1 is the mass of dry fired briquette.

Flexural strength value (σ) was evaluated according to ASTM F417–1996 norm, following Eq. (3) below for each briquette test:

$$\sigma = 3PL/2lh^2 \quad (3)$$

where P is the load at fracture (N), L is the distance between supporting knife edge (50 mm), l is the width of the briquette and h is the thickness of the briquette (mm).

4 Results and discussion

4.1 Mineralogy and geochemistry

Sa'a clayey materials consist of kaolinite ($7.20 \text{ \AA}$, $4.46 \text{ \AA}$, $3.58 \text{ \AA}$, $2.34 \text{ \AA}$, $1.98 \text{ \AA}$), quartz ($4.26 \text{ \AA}$, $3.34 \text{ \AA}$, $1.82 \text{ \AA}$), goethite ($4.99 \text{ \AA}$, $4.16 \text{ \AA}$, $2.69 \text{ \AA}$, $1.67 \text{ \AA}$), hematite ($2.69 \text{ \AA}$, $1.67 \text{ \AA}$, $1.45 \text{ \AA}$), and muscovite ($10.03 \text{ \AA}$, $4.46 \text{ \AA}$, $3.34 \text{ \AA}$) (Fig. 2). SiO_2 which exist as the chief oxide (Table 1) with the proportion between 76.04 and 53.61% is associated with the presence of quartz, kaolinite, and muscovite. This proportion of quartz in the raw material influences the durability of bricks [13] and provided a uniform shape of the bricks [14].

Because kaolinite and gibbsite are present, SiO_2 is followed by Al_2O_3 (12.00 – 23.23%). The content of Fe_2O_3 (3.76 – 11.58%) can result in poor mechanical strength of the final products [2–7]. Alkali and alkali-earth oxides persist in very low proportions ($\leq 1.55\%$) which may result in inadequate sintering during firing due to poor formation of the vitreous phase. The very low proportion of alkalis in Sa'a clayey materials required a relatively high temperature for their maturation [15].

The high proportion of Fe_2O_3 present in the clay leads to the reddish color of the fired products and thus these clays cannot be used for white body production [16]. The loss on ignition of lateritic clays in KK, NM, and NL is fairly high ($> 10 \text{ wt\%}$), primarily due to the removal of structural water and organic matter in the upper weathering profile. During the firing phase, the organic matter in these materials may serve as a pore former [17].

4.2 Physical and mechanical characterization

The color of fired bricks codified by Munsell code (Table 2) are globally light red ($2.5\text{YR } 7/8$, $2.5\text{YR } 6/8$, $2.5\text{YR } 6/6$) at the five firing temperatures for alluvial clay, light red from 900 to 1000°C and red at 1050 and 1100°C for NL and EB base bricks. For NM base product, the color is globally light red from 900 to 1000°C for mixtures with 40 and $60 \text{ wt\%}$ of lateritic clays and red for mixtures with 80 and $100 \text{ wt\%}$, red at 1050 and 1100°C for the first group and reddish

Fig. 2 XRD patterns of Sa'a lateritic clays (Q: quartz, M: muscovite, K: kaolinite, G: goethite, H: hematite)

Table 1 Chemical composition of raw material (wt%)

Oxides (wt%)	dl	NL-m	NM-m	EY -m	EB-m	KK-m	PO-m	AL
SiO ₂	0.04	53.61	54.77	63.26	76.04	59.91	70.35	61.43
Al ₂ O ₃	0.02	23.23	19.71	12.91	12.06	19.12	12.00	18.01
MnO	0.002	0.070	0.122	0.576	0.030	0.084	0.161	0.076
FeO	0.06	0.41	0.51	0.17	0.24	0.43	0.42	1.23
MgO	0.01	0.21	0.27	0.20	0.19	0.17	0.21	0.36
CaO	0.006	0.109	0.316	0.062	0.077	0.148	0.224	0.236
Na ₂ O	0.02	0.03	0.11	0.05	0.03	0.04	0.07	0.25
K ₂ O	0.01	0.76	1.55	0.93	0.82	0.17	1.11	1.29
Fe ₂ O ₃	0.01	7.53	9.31	11.58	3.76	8.04	6.02	4.85
TiO ₂	0.01	1.17	1.20	0.74	0.80	1.02	1.22	1.58
P ₂ O ₅	0.002	0.053	0.197	0.062	0.033	0.102	0.175	0.106
Cr ₂ O ₃	0.002	0.01	0.01	0.05	0.01	0.01	0.01	0.01
LOI	0.05	12.65	11.51	8.85	5.73	11.20	7.70	9.77
Total	–	99.43	99.08	99.26	99.58	100.02	99.24	99.20

brown to dark reddish brown for the second. EY, KK and PO base products display light red color with 40 wt% of lateritic clay from 900 to 1000 °C and red color at the same temperatures for the three other mixtures. Their colors are red, dark red, reddish brown, or dark reddish brown at 1050 and 1100 °C. The 2.5YR colors are highly allied to the presence of Fe₂O₃ in the raw materials [18, 19].

Particle size distribution shows that Sa'a upper set weathering materials are mainly composed of sand, silts and clays (Table 3). The grain size is spread out (Fig. 3). In lateritic materials, sand proportion varies between 42.83 (EY) and 17.20 wt% (NL) while silt content varies between 40.00 (NM) and 11.50 wt% (EY). Clay's proportion varies from 48.30 (NL) and 24.00 wt% (NM). The

alluvial clayey material consists of 6.00, 40.00 and 54.00 wt% of sand, silt and clay respectively. NL and EB are sandy-clay, while PO, KK, and EY are heavy sandy-clay and NM is silty-sand according to the Belgian textural diagram.

From Winkler's diagram (Fig. 4), granulometry of NM materials is sufficient for common bricks, while EY, EB, KK and PO are fine for roofing tiles and masonry bricks, whereas NL is not graded. The size of these materials spreads encourages compaction [20]. The effects are decrease in porosity, permeability, compressibility, water absorption and swelling, and mechanical resistance increase. The fine-grained size of the studied clayey materials confers good binding properties [21].

Table 2 Munsell code (color) of fired bricks prepared with lateritic and alluvial clays mixtures from Sa'a region

Sample	Lateritic clay wt%	900 °C	950 °C	1000 °C	1050 °C	1100 °C
AL	00	2.5YR 7/8	2.5YR 7/8	2.5YR 7/8	2.5YR 6/8	2.5YR 6/6
NL	40	2.5YR 6/6	2.5YR 6/8	2.5YR 6/8	2.5YR 5/6	2.5YR 4/6
	60	2.5YR 6/8	2.5YR 6/8	2.5YR 6/8	2.5YR 5/6	2.5YR 4/8
	80	2.5YR 5/8	2.5YR 5/8	2.5YR 6/6	2.5YR 4/8	2.5YR 4/6
	100	2.5YR 6/8	2.5YR 6/8	2.5YR 6/8	2.5YR 5/8	2.5YR 3/6
NM	40	2.5YR 6/8	2.5YR 6/8	2.5YR 6/8	2.5YR 5/6	2.5YR 4/4
	60	2.5YR 6/8	2.5YR 6/8	2.5YR 5/8	2.5YR 4/8	2.5YR 4/6
	80	2.5YR 5/8	2.5YR 5/8	2.5YR 4/8	2.5YR 4/6	2.5YR 4/4
	100	2.5YR 5/8	2.5YR 5/8	2.5YR 4/6	2.5YR 4/4	2.5YR 3/4
EY	40	2.5YR 6/8	2.5YR 6/8	2.5YR 6/8	2.5YR 6/6	2.5YR 5/6
	60	2.5YR 5/8	2.5YR 5/8	2.5YR 5/8	2.5YR 5/6	2.5YR 4/4
	80	2.5YR 5/8	2.5YR 5/8	2.5YR 5/8	2.5YR 3/4	2.5YR 2.5/3
	100	2.5YR 5/6	2.5YR 5/6	2.5YR 5/6	2.5YR 3/3	2.5YR 3/2
EB	40	2.5YR 7/8	2.5YR 7/8	2.5YR 7/8	2.5YR 6/8	2.5YR 5/8
	60	2.5YR 6/8	2.5YR 6/8	2.5YR 6/6	2.5YR 5/6	2.5YR 4/6
	80	2.5YR 6/8	2.5YR 6/8	2.5YR 6/6	2.5YR 5/8	2.5YR 4/8
	100	2.5YR 6/8	2.5YR 6/8	2.5YR 5/8	2.5YR 4/6	2.5YR 3/6
KK	40	2.5YR 6/8	2.5YR 6/8	2.5YR 6/8	2.5YR 5/8	2.5YR 5/6
	60	2.5YR 5/8	2.5YR 5/8	2.5YR 5/6	2.5YR 5/6	2.5YR 4/6
	80	2.5YR 5/8	2.5YR 5/8	2.5YR 5/6	2.5YR 4/6	2.5YR 3/6
	100	2.5YR 5/8	2.5YR 5/8	2.5YR 5/8	2.5YR 4/6	2.5YR 3/6
PO	40	2.5YR 6/8	2.5YR 6/8	2.5YR 6/8	2.5YR 4/8	2.5YR 4/6
	60	2.5YR 5/8	2.5YR 5/8	2.5YR 5/8	2.5YR 4/6	2.5YR 4/4
	80	2.5YR 5/8	2.5YR 5/8	2.5YR 5/8	2.5YR 5/6	2.5YR 3/4
	100	2.5YR 5/8	2.5YR 5/8	2.5YR 5/8	2.5YR 4/4	2.5YR 4/4

Table 3 Physical properties of raw material

Parameter	Sample	NL	NM	EY	EB	KK	PO	AL
PSD (wt%)	Sand	17.20	34.33	42.83	36.20	37.33	37.60	6.00
	Silt	33.70	40.00	11.50	17.80	31.00	33.00	40.00
	Clay	48.30	24.00	26.00	46.00	31.00	28.00	54.00
Atterberg limits (%)	LL	46	44	40	40	41	30	43
	PL	31	28	28	24	28	19	25
	IP	15	16	12	16	13	11	18
VBS (g/100 g)		2.87	2.53	2.80	2.80	2.33	2.20	5.07

About the Atterberg limits, for EY and NM, the liquid limit (LL) ranges between 30% (PO) and 46% (NL) and the plasticity index (PI) between 12 and 16%, respectively (Table 3). The Casagrande plasticity chart classification of these materials shows that they are less cohesive soils with medium plasticity. The plasticity index projection and the plasticity limit values in the workability map (Fig. 5) indicates that all the clayey material tested has suitable molding properties. The plastic behavior of the samples and their adaptability for pressing are closely linked [22]. High plasticity would have been good for the extrusion process or hand-molding shaping [15].

Linear shrinkage values are rising with temperature and with alluvial clay content globally (Fig. 6). High shrinkage for ceramic applications should be avoided because contractions in ceramic products cause major distortions. NL briquettes give the highest shrinkage and are consistent with their high clay content. An amendment which may consist of enrichment with silt or sand may assist in the dimensional regulation of the materials studied. The small LS values at temperatures below 1050 °C are in line with the workability chart (Fig. 5). This weak shrinkage is also correlated with the sandy nature

Fig. 3 Particle size distribution curves of the lateritic and alluvial clays

Fig. 4 Winkler diagram, (I) Common bricks; (II) Vertically perforated bricks; (III) Roofing tiles and masonry bricks; and (IV) Hollow products

of the sample, where free silica (sand) acts as a filler and enhances shrinkage control [23].

Water absorption values overall remain lower than 25% (Fig. 7) which is good for dense bricks [24]. The specified values for WA of Brazilian clay-based products by the same author are WA < 20 wt% for roofing tiles. These WA values are obtained at all the temperatures for mixtures with EB, EY, KK, NM and PO, and at a temperature above 950 °C for NL clay-based products. For the NL clayey mixtures, the highest WA values are obtained by the alluvial clay. WA values are most influenced by

Fig. 5 Workability chart (after brain and highly in Moutou et al. [33])

temperature than alluvial clay content of the mixture. The same pattern is observed with of Ayos clayey mixtures [6]. Low drops in water absorption values in all samples are usually due to the absence or very low development of a vitreous phase [20, 25]. This is associated with a minimal amount of fluxing agents in all the samples (Table 1). The vitreous phase produced allows pores to be enclosed or adjacent pores to be separated by penetration of the liquid vitreous layer within the pores [13, 26, 27]. Globally flexural strength remains lower than 2 MPa for lateritic clays bricks except for KK and NM samples where FS is slightly greater than this standard. For the mixtures, FS increase with alluvial clay content

Fig. 6 Linear shrinkage of the fired products

Fig. 7 Water absorption of the fired products

Fig. 8 Flexural strength of the fired products

except for a few samples at some temperatures (Fig. 8). The firing process heightens the flexural strength. It is due to the reduction of crack formation by decreasing porosity [28]. Higher values are obtained for NM materials. EB based product which has lower Fe_2O_3 content exhibit a low FS due to high wt% of SiO_2 (76.04%) while for EY bricks, low flexural strength is related to the high percentage of sand. Andji et al. [2] increase the mechanical strength of Gounioubé clays (Ivory Coast) by incorporating calcium clay (10 wt% CaCO_3); according to them, this is due to the formation of anorthite from the alumino-silicate content between 1100 and 1300 °C. Ergul et al. [8] conclude that low porosity ceramic products (13 wt%) and water absorption products (4 wt.%) are produced from a material consisting of 50 wt% tuffs and 50 wt% clay at a temperature of 1150 °C. In the present research, the temperatures range between 900 and 1100 °C. FS increase can be chemically related to mullite formation from 900 °C [29, 30]. Except for NL mixtures at 900 °C, the studied materials exhibit flexural strength values > 2 MPa and can be used for structural bricks manufacturing [24]. Flexural strength values quantified by Souza et al. [24] are 6.5 MPa for roofing tiles. This FS value is surpassed by NM_60 and NM_40 at 1000, 1050 and 1100 °C, by AL, NL_60, EY_40 at 1050 and 1100 °C and by KK_40 at 1100 °C. Fluxes amendment is needed

at 1050 and 1100 °C for the others mixtures with 60 wt% and 40 wt% of lateritic clay to increase their FS.

Lateritic clay materials from Sa'a area exhibit mineralogical and geochemical characteristics comparable to those of similar materials in the equatorial zone [31, 33]. They are predominantly silicic but characterized by relatively high Fe_2O_3 contents responsible for their red coloring. The parameters studied show the impact of the addition of alluvial clays with a low Fe_2O_3 content on these lateritic clays. The same trends are observed with the mixtures of Yaounde [5], Ayos [6] and Monatele-Ebebda [7]. All this allows to conjecture these results to the equatorial zone.

5 Conclusions

The amendment of lateritic clay by alluvial clay changes the mineralogical and chemical properties of former materials and is favorable to the sintering at low temperatures. Mechanical characteristics such as WA and FS are improved with firing temperature but it's not the case of LS. This improvement is not always proportional to alluvial clay content and would be due to high Fe_2O_3 content on the alluvial clays of the Sanaga. The lateritic and alluvial clays mixtures of Sa'a could be used for bricks manufacture. Some compositions may be used for the production

of roofing tiles at temperatures above 1000 °C. Fluxes amendment is needed at 1050 and 1100 °C for other mixtures to increase their FS.

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Compliance with ethical standards

Conflict of interest We have no concurrent submissions on the same topic and with this analysis data. There is no conflicts of interest.

Availability of data and material The raw data provide by the different laboratory are available.

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