Research article

Mineralogical, physico-chemical and ceramic properties of clay materials from Sekandé and Gashiga (North, Cameroon) and their suitability in earthenware production

B.P. Kagonbé a,b,*, D. Tsozué a, A.N. Nzeukou b, S. Ngos III c

a Department of Earth Sciences, Faculty of Science, University of Maroua, P.O. Box 814 Maroua, Cameroon
b Local Materials Authority Promotion (MIPROMALO), P.O. Box 2396 Yaoundé, Cameroon
c Faculty of Mines, Petroleum, Gas and Water Resources Exploration, University of Maroua, P.O. Box 8 Kaité, Cameroon

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ABSTRACT

Clayey materials from Sekandé and Gashiga localities in the northern part in Cameroon traditionally used for pottery were studied. Eight samples were collected and investigated by XRD, IR, XRF, physical and firing properties from 900, 1000 and 1100 °C. In term of mineralogy, studied clays are rich in smectites, illite, kaolinite, quartz, feldspar and mica. Physicochemically, results show large particle size variations with high plasticity (18–30%). Clay fraction (< 2 μm) ranges from 18 to 38% and sand fraction from 40 to 53%. SiO2 (46–55%) and Al2O3 (19–21%) are major chemical oxides, followed by Fe2O3 (6.68–10.82%) and alcali and alcali-earth elements (8–10%). These characteristics govern their behavior on firing with temperature development. The color of specimens was predominantly red after firing. Between 900 and 1100 °C, their color ranged from reddish brown, red yellow or brown red to red. The linear shrinkage values vary from 0.5% to 7%, weight loss from 4% to 9% and bulk density from 1.74 to 2.21 g/m3. These values increased gradually for specimens fired from 900 to 1100 °C. The water absorption decreased from 17% to 14%. The evaluation of the studied clays based on their mineralogical, physicochemical and ceramic properties revealed that they are suitable for the manufacture of earthenware products like fired bricks.

1. Introduction

Clays have been widely used as the main component of raw materials in the fabrication of diversified ceramic products (Harvey and Murray, 1997; Murray, 2007; Semiz, 2017; Tsozué et al., 2017). Their applications are highly dependent on their structure, composition, physicochemical characteristics and their abundance associated with their relatively low cost (Grim, 1960; Giulio, 1996; Reeves et al., 2006). Today, they 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 worldwide. They play a vital role in economic development (Ekosse, 1994; Blanco Garcia et al., 2005; Reeves et al., 2006). The properties of clays that were interested to the ceramic industries are their plasticity which facilitates the shape of the body, chemical and mineralogical composition, thermal properties, color and mechanical strength after firing (Burst, 1991; Baccour et al., 2009). Most products obtained are earthenware, porcelain and stoneware. In Cameroon, the valorization of clay materials in the domain of earthenware is of growing interest. Currently, the industrial use of clays is increasing in brickworks and tile mills (Ojangang et al., 2007; Elimbi et al., 2011; Ngon et al., 2009; Nzeugang et al., 2013; Touogam Touolak and Tchangnwa Nya, 2014; Padil-Djenabou et al., 2015; Temga et al., 2015; Ndjigui et al., 2016). These studies carried out in Cameroon belong essentially to the warm and humid tropical zone of the country. In the sudanian and sudano-sahelian zones of the country on contrary, works on clay materials are very rare (Nguetnkam et al., 2008; Tsozué et al., 2017; Temga et al., 2018, 2019). Indeed, this part of the country is made up of vast alluvial plains with abundant clay materials. Despite its high proportion, clay materials from this area are only exploited for traditional uses, mainly for pottery and traditional fired bricks. These traditional processing methods of ceramic products do not take into account mineralogical, physicochemical properties and appropriate processing (mixing, shaping and drying) procedure. Nevertheless, the quality of pottery...
produced and fired brick by local manufacturers is generally poor. They show low mechanical resistance and durability. Thus, this paper aims to contribute to the study of mineralogical and physicochemical properties of Sekandé and Gaschiga clays, analyze their suitability in ceramic making and to open ways for other potential industrial applications.

2. Material and methods

2.1. Study sites

Geographically, the studied area is located in northern Cameroon between latitude 06°05’-06°11’ N and longitude 09°58’-10°11’ E (Figure 1). The climate is Sudanian with two contrasted seasons: a humid one from May to October and a dry one from November to April. Total yearly precipitations vary between 900 and 1500 mm and mean annual temperature is 28°C (Ezi, 1980). On the geomorphological point of view, the landscape is characterized by an extensive horizontal plain whose monotony is interrupted here and there by small hills. The natural vegetation is a savannah which is strongly modified by human activities mainly farming and demographic pressure, imposed by a rapidly increasing population (Letouzey, 1985). The hydrographic network is composed of temporary and perennial streams with a dendritic network. It is centrally dissected by the Benue river, main collector. The bedrock is mainly constituted of sandstone (Bessong, 2012). The main soil types are ferruginous soils and vertisols (Gavaud et al., 1976; Ségalen, 1976; Brabant and Gavaud, 1985; Azinwi et al., 2011).

2.2. Sampling techniques

The studied clay materials were collected from two different locations, Sekandé in Pitoa Sub-division and Gaschiga in Demsa Sub-division, respectively in the North and the North-East of Garoua town. Field campaigns consisted of direct observations, description of environmental conditions and soil survey in order to choose the position of pits. Four pits were dug in Sekandé (SK1, SK2, SK3 and SK4) and three in Gaschiga (GA1, GA2 and GA3). All pits were described in detail according to the standard procedure (Figures 2 and 3). The Munsell Soil Color Chart was used for color appreciation. From these pits, eight samples of 30–50 kg were collected in the clayey layers: five in Sekandé (SK1C2, SK1C3, SK2C2, SK3C2 and SK4C2) and three in Gaschiga (GA1C2, GA2C2 and GA3C2). The samples were selected on the basis of their color and homogeneity.

2.3. Analytical techniques

In the laboratory, clay materials were quarried to provide statistically valid samples. Initially, all samples were dried at 105°C for 24 h; aliquots of dried samples were ground using agate mortar for further analysis.

The particle size distribution analysis was performed by wet sieving method for particles of size greater than 80 μm and by sedimentometry for particles of size less than <80 μm. The liquid limit (LL), plasticity Limit (PL) and plasticity index (PI) analyses were performed according to the ASTM norm, D-422 and D-4318 respectively, by the Casagrande method.

The mineral phase analysis was carried out by X ray diffraction (XRD) on disoriented powders and on oriented aggregates and by Fourier transform infrared spectroscopy (FTIR) on total powder. Samples were scanned in the 2θ range from 2° to 45° with step size of 0.02° and time per step 2s, with CuKα radiance, λ = 1, 5418 Å, at 40 kV and 30 mA. Further tests on clay fraction for detailed identification were air-drying (24 h), glycolation (22 h) and heating (500°C for 4 h). All analyses were performed in the laboratory of "Argiles, Géochimie et Environments sedimentaire (AGEs)" at the University of Liège, Belgium, according to the methodology of Moore Duane and Reynolds Robert (1989).

Diffuse reflectance infrared spectra were recorded between 4000 cm⁻¹ and 500 cm⁻¹ using a FTIR spectrometer (IFS 55). The spectrum resolution was 4 cm⁻¹ and spectra were obtained by accumulation of 200 scans.

Elemental composition analysis was performed by X-ray fluorescence spectroscopy (XRF) in the Laboratory of "Pétrologie, Géochimie Endogènes et Pétrophysique (PGEP)" at the University of Liège in Belgium.

For firing test, clay samples were dried at 105°C for 24h and ground to a fine powder and then sieved using a mesh-size of 100 μm. Specimen cylinders of 40 mm in diameter and 80 mm in length were made

Figure 1. Location of the study area.
manually, using wetted clay samples, and then dried in a shaded and ventilated room. 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 a MultiMate furnace at Local Materials Promotion Authority (MIPROMALO), Cameroon. Colors of raw and fired samples were determined using the Munsell Soil Color Chart. The sound test consisted of knocking the fired specimens with a metal rod to obtain the sound. The linear shrinkage (LS) was calculated following the formula \[ LS (\%) = \frac{(L_0 - L)/L_0}{100} \] according to the relative variation length of the briquette, where \( L_0 \) is the length of the briquette before firing and \( L \) that of the briquette after firing. Weight loss (WL) was calculated through the following formula \[ WL (\%) = \frac{[(M_d - M_f)/M_d] \times 100}{M_f} \] with \( M_d \) corresponding to the dry mass at 105 °C and \( M_f \) to that of the fired mass at each final firing temperature. Bulk density (Bd) of a briquette was obtained by dividing the fired briquette mass (M_f) by the measured volume of the briquette (V): \[ Bd (g/cm^3) = \frac{M_f}{V} \]. Water absorption capacity (WA) was measured according to ASTM norm C373-72 by weighing the fired briquette (M_f) and the wet briquette after immersion in water for 24h: \[ WA (\%) = \frac{(W - M_f)/M_f}{100} \]. Each result obtained is the average value of five tested samples.

3. Results and discussion

3.1. Mineralogical composition

The results obtained from the analysis of the diffractograms of bulk clay samples show the same mineralogical phases with a variable amount (Figure 4a). The mineral composition includes quartz (peaks around 4.25 Å, 3.34 Å, 3.24 Å, 2.97 Å, 2.56 Å, 2.45 Å, 2.33 Å, 2.12 Å, 1.98 Å, 1.67 Å and 1.54 Å), kaolinite (7.48 Å) and smectite (15.46 Å and 14.47 Å), associated to feldspars (15.46 Å and 14.47 Å), associated to feldspars (8.82 Å, 15.46 Å and 14.47 Å), associated to feldspars (3.24 Å, 3.19 Å) and amphibole (8.82 Å, 15.46 Å and 14.47 Å).
4.38 Å) (Figure 4a). The oriented samples were analyzed to further confirm the nature of the clay phases in the samples (Figure 4b). The occurrence of smectite was confirmed by the displacement of diffraction peak from 14 Å to 17.92 Å, after the ethylene glycol solvatation treatment (Figure 4b). Furthermore, the main peaks of kaolinite (7.16 Å) and illite (10.01 Å) were not affected by the ethylene glycol solvatation treatment. Also, the main diffraction peak of kaolinite is absent in diffractograms after firing at 550 °C contrary to that of illite which still present. These elements confirm the presence of kaolinite and illite in the studied clay materials. Globally, there was an increase in the intensity of the 10.01 Å diffraction peak after thermal treatment at 550 °C (Figure 4b). According to Fadil-Djenabou et al. (2015), this increase in the intensity might be due to the collapse of the smectite diffraction peak at 10 Å which also confirmed the presence of this 2:1 expansive clay mineral (smectite). The mineralogical composition of Sekandé and Gaschiga clay is comparable to that obtained in Ngaye alluvial clay (Fadil-Djenabou et al., 2015), Logone valley clays and Lake Chad Basin clay in Central Africa (Temga et al., 2019), Benoue floodplain clays (Azinwi et al., 2016) and Maroua clays (Tsozu et al., 2017), in the northern part of Cameroon. It differs however from the mineralogical composition of clay material in southern par of Cameroon by the presence of 2:1 clay minerals, illite and smectite as consequence of sudanian contrasted climate which prevailed in the study area.

The study of clay materials by FTIR spectroscopy confirms the presence of kaolinite, illite, smectite and quartz as observed in X-ray patterns illustrated in Figure 4a. The characteristic bands of O–H stretching of kaolinite and eventually illites appeared between 3696 cm⁻¹ and 3618 cm⁻¹ (Famer and Russel, 1964) and the absence of well-defined peak suggests disordered or poorly crystallized character, also evidenced by the absence of Al₂–OH bending bands at 938 cm⁻¹. The band at 1630 cm⁻¹ is attributed to hydration water for both samples. This band showed significant decrease in intensity with increasing carboxylic acids concentration. This progressive decreasing of the band is related to the removal of octahedral cations, causing the loss of water and hydroxyl groups and indicating the dissolution of dioctahedral structure smectite. The presence of smectites confirmed by the vibration band around 3412 cm⁻¹ (OH- stretching vibration) is reliable to the absorbed water by 2:1 clay minerals (Famer and Palmeri, 1975). The weak bands observed at 685 cm⁻¹ and 788 cm⁻¹ for the eight samples may be related to quartz crystal (Madejova and Komadel, 2001), which is in good agreement with the XRD results.

3.2. Physico-chemical composition

The particle size distribution is presented in Table 1. The results show large particle size variations from one sample to another, with clay fraction ranges from 17.8 to 38.3 %, silt fraction from 11.9 to 36.3 %, sand fraction from 39.7 to 53.2 % and gravel from 1.7 to 8.8%. Higher clay contents were observed in SK3C2 (36.2%) and SK4C2 (38.3%). Previous studies have shown that particle size distribution can have a significant influence on plasticity (Holtz and Kovacs, 1981). The grain-size distribution of raw materials influences the technological parameters during the drying and firing processes (Dondi et al., 1998) and sands are predictably reactive upon firing at lower temperatures range. The plastic limit values and the plasticity index (PI) of all samples vary

| Profile SK1 | Profile SK2 | Profile SK3 | Profile SK4 |
|-------------|-------------|-------------|-------------|
| N 09° 24’ 57.2”; E 013° 32’02.1” Elevation: 223 m | N 09° 24’ 50.5”; E 013° 33’ 55.6” Elevation: 228 m | N 09° 24’ 59.7”; E 013° 31’ 58.8” Elevation: 226 m | N 09°24’ 34.9”; E 013°37’ 52.1” Elevation: 239 m |
| Depth (cm) | Depth (cm) | Depth (cm) | Depth (cm) |
| Layer | Layer | Layer | Layer |

Figure 3. Macroscopic organization of clay soil profiles (SK1, SK2, SK3, SK4) in Sekandé. Analyzed samples are SK1C2 and SK1C3 (Profile: SK1), SK2C2 (Profile: SK2), SK3C2 (Profile: SK3) and SK4C2 (Profile: SK4).
between 24.4 and 36.9% and 17.7 and 30% respectively (Table 1). The positioning of the various samples in the Bain and Highley (1970) diagram permit to classify them as high plasticity materials (Figure 6a). The plasticity is favorable for extrusion and manual processing but the proportion of silt and sand may affect the process ability. Plasticity is one of the most important parameters that influence for manufacturing clayey products (Abdelmalek et al., 2017; Semiz, 2017). There are various factors affecting the plasticity properties such as origin of geological formation and mineral composition, particle size distribution, impurities (no clay minerals), and organic matter (Bergaya et al., 2006; De Oliveira

Figure 4. XRD patterns of Gaschiga and Sekandé clays (a) Bulk XRD patterns; identified minerals: K = kaolinite, Q = Quartz, K-Felds = Feldspar, Pla = Plagioclase; (b) Clay <2 μm XRD patterns showing the basal spacings d in Å and the corresponding minerals with coexistence of kaolinite, illite and smectite in the clay fraction of the analyzed samples; N: air dried condition, EG: glycolated condition with ethylene glycol for 22 h, H: heated condition at 500 °C for 4 h.
Modesto and Bernardin, 2008; Hajjaji et al., 2010). The positioning of the plasticity values obtained in the Diagram of Holtz and Kovacs (1981) (Figure 6b) shows that the Sekand/C19 and Gaschiga clays approximate clays of medium plasticity (SK2C2, SK1C3, SK3C2, GA2C2 and GA1C2). According to Winkler’s diagram, samples SK1C2, SK3C2, SK4C3 and GA2C2 were suitable for thin-walled hollow bricks while SK1C3, SK2C2, GA1C2 and GA3C2 are not suitable for the manufacture of traditional ceramics (Figure 6c).

Table 1 also presents the results of the chemical analysis of the samples. The most abundant oxides in the studied samples were SiO2 (46.46–54.88%) followed by Al2O3 (19.14–21.15%), Fe2O3 (6.68–10.82%) and K2O (1.85–4.30). The SiO2/Al2O3 ratio varying between 2.25 and 3.01. The SiO2/Al2O3 ratios >2% are indicative of an excess of SiO2 in the studied samples, confirming the presence of 2:1 phyllosilicates of smectites type (Nguetnkam et al., 2008; Tsozu/C19 et al., 2017). The Fe2O3 content is relatively high (7–11%) and may be responsible for the reddish color in fired specimens (Dondi et al., 1998). The highest LOI values observed (8.88–15.28%) are related to the deshydroxylation of clay minerals, organic matter oxidation, and decomposition of carbonates and hydroxides (Semiz, 2017). The sum of alkali and alkaline earth elements was high (around 8%). Theses bases, in addition to the presence of feldspars, illite and smectite might guarantee a good fusibility for earthenware production (Tsozu et al., 2017).

3.3. Technological properties of the fired products

After drying for 24 h, the clay samples were fired at different temperatures (900, 1000 and 1100 °C) and coloration, sonority, firing shrinkage, water absorption, weight loss and bulk density were measured. All properties showed a dependence on the firing temperature. Table 2 summarizes the results of different tests. Beyond 900 °C, the sound varies from slightly metallic to metallic and offers very good cohesion for all samples except GA1C2 which did not have a metallic sound at 900 °C. This metallic sound reflects the maturity of the fired products and is produced when the cohesion is high (Melo et al., 2003). It is also a characteristic of good quality of ceramic products (Melo et al., 2003; Damle, 2008), but the reverse is not necessarily true (Melo et al., 2003). This is principally due to sintering facilitated by high

![Figure 5. IR curves of the Gaschiga and Sekandé clays.](image_url)
Figure 6. Evaluation of the suitability of Gaschiga and Sekandé clays base on empirical diagrams, (a) Diagram of Bain and Highley (1970), (b) Diagram of Holtz and Kovacs (1981) in Tsozu et al. (2017), (c) Diagram of Winkler (1954) in Tsozu et al. (2017) showing suitable mixtures of grains fractions for solid brick (I), vertically perforated bricks (II), roofing tiles lightweight bricks (III) and thin walled hollow bricks (IV).
content in fluxing oxides and sand in raw material (Fadil-Djenabou et al., 2015; El Ouahabi et al., 2014). No cracks were observed in all specimens except sample GA1C2. The color of firing specimens changes with an increase in temperature. Between 900 and 1100 °C, their hue/value/chroma ranged from reddish brown, red yellow or brown red to red. Their color was predominantly red due to the presence of iron oxides in the raw materials (6.68–10.84%, Fe₂O₃), in the form of hematite (Pialy, 2009). The variation of color can also be attributed to more amount of some oxide impurities, such as titanium (TiO₂) minerals (Pialy, 2009), magnesium (MgO), aluminum oxide (Al₂O₃) or temperature of the oven (Boch, 2001) and especially the presence of calcium (CaO) which the presence in large quantities slightly decreases the effect of iron giving pink coloring (Boussen et al., 2016).

The firing shrinkage evolution of the eight samples investigated are shown in Table 2. Globally, at 900 °C, significant changes were observed; the values increased from 1.06% to 7.36%. The high value of the linear shrinkage for SK3C2 is indicating the occurrence of sintering reaction facilitated by their relatively high contents in fluxing agents. Generally, the shrinking behavior, primarily attributed to the increase of sintering, is probably due to rearrangement reactions within the firing product associated to partial sintering of some phases and the development of glassy phase, resulting from the occurrence of K₂O (Lemaître et al., 1977; Lemaître and Delmon, 1980).

The water absorption test reflects water retention capacity of each sample. Water absorption decreases with increasing firing temperature. At 900 °C, their values ranged between 15.88 and 20.81% and decreased until 8.57–13.86 % at 1100 °C. This decrease is associated to glassy phase formation that penetrates into pores closing them and isolating them from neighboring pores (Lambercy, 1993). The decrease in water absorption translates the dehydration reactions, decarbonation and combustion of organic matter (Ngun et al., 2011). The decrease in water absorption coefficient influences the mechanical properties and durability of the sample, making it more resistant and more durable.

The weight loss is an important feature to express the amount of organic matter present in the starting materials. It reflects the degree of vacuum and the percentage of water absorption. Table 2 shows that the weight loss of the fired products which increased with the firing temperature, varied between 5.37% at 900 °C and 8.86 % at 1100 °C. The higher value (8.86%) was observed at 1100 °C for GA1C2 sample. The increasing of weight loss can be attributed to the elimination of organic matter by combustion and water by dehydration and the decomposition of some minerals and especially clay minerals during firing (Baulz et al., 2004; Fadil-Djenabou et al., 2015).

Variation of bulk density of the fired clays in relation to firing temperatures is reported in Table 2. Globally, their value slightly increases with temperature (1.74–2.21 g/cm³). The high value of bulk density (2.21 g/cm³) was observed at 1100 °C, confirming the mineralogical transformations that occurred (Tsouzé et al., 2017). This is most likely due to the formation of substantial glassy phase since the study clay contains large amounts in fluxing agents (Ngun et al., 2011).

4. Conclusion

In this study, mineralogical, physico-chemical and ceramic properties of the Sekandé and Gaschiga clays were investigated in order to produce earthenware products. The mineralogical composition of samples includes quartz, feldspar, plagioclase, kaolinite, illite and smectite. From the physico-chemical properties, the results show large particle size distribution from one sample to another, with clay fraction ranges from 17.8 to 38.3 %, silt fraction from 11.9 to 36.3 %, sand fraction from 39.7 to 53.2 % and gravel from 1.7 to 8.8%. No significance change in term of granulometry is observed on the two sites. The studied clays were classified as highly plastic clays. Major oxides
present in the eight samples are SiO₂, Al₂O₃ and Fe₂O₃ associated to alkali and alkali-earth elements (8–10%). In view of these evaluations, these raw materials are suitable for porous colored structural ceramics. According to the firing properties obtained in the studied temperature range (900–1100 °C), the studied clays are suitable for the production of earthenware from 900 °C. Their relatively important content in fluxing agents such as feldspar, alkali and alkali-earth elements could reveal potential stating material for the production of stoneware. Subsequent studies at higher firing temperature are required to confirm this hypothesis.

Declarations

Author contribution statement

B.P. Kagonbé, D. Tsouze, A.N. Nzeukou, S. Ngos III: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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The authors declare no conflict of interest.

Additional information

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