Characteristics of Kaolinitic Raw Materials from the Lokoundje River (Kribi, Cameroon) for Ceramic Applications

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Abstract: Eight kaolinitic materials from the Lokoundje River at Kribi were sampled and investigated for their physical, chemical, mineralogical and thermal characteristics in order to evaluate their potential suitability as raw materials in ceramics. The Lokoundje kaolinitic materials are clayey to silty clayey and are predominantly composed of kaolinite and quartz. The alkali (Na2O + K2O) content ranges between 1 and 2.5 wt.%; these low values do not favor vitrification of the ceramics but may be improved through flux amendment. The presence of goethite in some samples limits their utilization in white ceramics. The minerals content, color, metallic sound, cohesion, linear shrinkage, flexural strength, bulk density, water absorption and microstructure were determined. The XRD data reveal that kaolinite and goethite were transformed, respectively, into mullite and hematite. The colors of the fired products are characteristic of their mineral assemblage. The metallic sound is indicative of low vitrification which is confirmed by the presence of cracks due to low flux contents. The cohesion is good to very good, due to the abundance of kaolinite. The physicomechanical properties increase with temperature as well as densification. The geochemical data show that the Lokoundje alluvial clays are suitable for the manufacture of white stoneware tiles.

Keywords: Kribi; alluvial clays; kaolinite; valorization; low mullitization; white stoneware tiles

1. Introduction

The investigation of clays as raw materials for ceramic industry has been a long-standing research activity spanning over many centuries. Clays mineral are used in traditional ceramics for fired bricks, stoneware or porcelain [1,2]. The interest human societies have in clay materials is due to the several varieties of applications [3]. Clay materials are used to promote sustainable development in many countries due to their low cost and availability for use in the building industry [3]. Kaolin occurs as a ubiquitous raw material with many applications, such as filler in paper, plastics, paints, and rubber. In view of promoting the quality used of available clay resources around the world, many studies are being conducted on their valorization in various applications, such as cementitious-product making [4,5], water treatment [6–10], pollution remediation [11,12], earthen ceramic production [13–15], and polymer–clay composites [16,17]. The present study focused on the evaluation of the potentiality of alluvial kaolinite from Kribi as a potential raw material for the production of earthen ceramics. The testing of the material from this region is motivated by the deposit size, which may allow a large-scale production
if the material properties are favorable. To this end, the chosen samples were subjected to chemical, mineralogical, physical, and textural analyses to determine their properties, as well as performance evaluation of the fired specimens.

2. Geological Data on Lokoundje Alluvial Clays

The study area is located in the Lokoundje watershed [18]. The watershed is mostly developed on the Nyong Unit, which is made up of pyroxenites, migmatites, amphibolites and gneisses [19,20]. Ferralsols and gleysols are dominant [21–23]. Three geomorphological units were recorded (Figure 1a–c). The study site is the river valley terrace in a low geomorphological unit (0 to 100 m; Figure 1b). According to Ndjigui et al. [18], the Lokoundje kaolinitic materials are subdivided into four groups: yellow, reddish yellow, white and light grey (Figure 1c). These clays are partially covered by a clayey-sandy layer enriched with organic matter. The mineralogical investigation revealed that kaolinite and quartz are dominant, in addition to small amounts of gibbsite, rutile, illite, goethite, and interstratified illite-vermiculite [18]. FTIR revealed the disordering of kaolinite [18]. Eight samples were selected due to their high proportion of kaolinite and their high Al$_2$O$_3$ contents. These samples were selected among eighteen samples of which sampling and mineralogical and geochemical studies were presented in Ndjigui et al. [18].

![Figure 1.](image-url) Location map of the study site in the Kribi area around the Cameroonian Atlantic coast (a), a geomorphological map of the Kribi area, including the study site (b), and spatial distribution of alluvial clays in the Lokoundje terrace (c).
3. Materials and Experimental Technics

The clayey samples used were described in Ndjigui et al., 2018 [18]. All the samples were mainly composed of kaolinite, quartz and goethite. Illite was mainly observed as an interstratification with kaolinite. Mineralogical analyses using XRD and FTIR indicate that the kaolinite in all the samples was disordered.

For making the test briquettes, humidified powders were pressed to form prismatic bars, which were heated to 800, 900, 1000, and 1100 °C. The firing temperatures were chosen within the classical temperatures for fired clay bricks. The specimen dimensions were 40 mm × 20 mm × 20 mm. The colors of fired products were determined using the Munsell’s Color Chart. The specimens were elaborated as briquettes, given that mechanical behaviors are accessed through flexural strength in a three-point bending mode. The physical and mechanical properties were determined at each sintering temperature using the elaborated briquette.

The X-ray (XRD) patterns, for mineralogical evolution were obtained using a PAN Analytical X’PERT PRO diffractometer operating under a voltage of 40 kV, a current of 45 mA, and using cobalt Kα radiation (λ = 1.789 Å) at the Geoscience Laboratories, Sudbury, Canada. The mineralogical evolution was evaluated to understand the change in structure within the briquette and to justify the mechanical properties. Thermal analyses on the raw samples were carried out at the University of Liege, Belgium. Clay mineral transformation under thermal analyses are a complement to mineralogical analyses as they help to confirm the presence of some clay minerals through the analysis of their dehydroxylation. Tests were conducted using ~15 mg of the <250 µm fractions on an automatic multiple sample thermogravimetric analyzer (TGA-2000) by Las Navas Instruments. The analysis was done under air at a heating rate of 5 °C/min from ambient temperature to 1000 °C. Some fired products were subjected to scanning electron microscopy (SEM) for microstructure observations at the University of Lausanne (Switzerland). A Tescan Mira LMU emission-SEM operated at 20 kV with a 30 mm working distance was used. The images were acquired using a backscatter electron (BSE) probe.

4. Results and Discussion

4.1. Particle Size Distribution and Atterberg Limits

Lokoundje kaolinitic materials are characterized by high proportions of clay-sized materials (Table 1). This distribution points out the predominance of clay and silty clay textures. The projection of the PSD is the texture ternary diagram concluded that these raw materials are favorable for ceramic products having a low porosity and low permeability. This is probably associated with the low amounts of sand (Figure 2). The Atterberg limits in Table 1 are coherent with materials having medium plasticity, which is of interest for the workability of the pastes from these raw materials.

Table 1. Particle size distribution and Atterberg limits.

| BR-5 | BR-8 | BR-9 | BR-11 | GR-2 | GR-3 | GR-6 | GR-8 |
|------|------|------|-------|------|------|------|------|
| **Particle size distribution** | | | | | | | |
| Clay (<2 µm) | 75.66 | 71.66 | 65.15 | 61.57 | 75.85 | 58.86 | 73.22 | 66.39 |
| Silt (2–50 µm) | 24.12 | 28.09 | 33.14 | 37.45 | 23.81 | 20.02 | 25.41 | 32.63 |
| Sand (>50 µm) | 0.22 | 0.25 | 1.71 | 0.99 | 0.94 | 21.12 | 11.37 | 0.98 |
| **Atterberg Limits** | | | | | | | | |
| LL (%) | 85.2 | 86.1 | 87.1 | 84.2 | 80.2 | 82.7 | 90.1 | 82.4 |
| PL (%) | 44.6 | 42.8 | 43.2 | 43.8 | 41.5 | 43.5 | 46.5 | 43.1 |
| PI (%) | 40.6 | 43.4 | 43.9 | 40.4 | 38.7 | 39.2 | 43.6 | 39.3 |
4.2. Chemical Composition

The chemical data showed that SiO$_2$ and Al$_2$O$_3$ are the most abundant oxides (SiO$_2$ + Al$_2$O$_3$ > 75 wt.%). The SiO$_2$/Al$_2$O$_3$ is less than 2 in several samples (Table 2) and is an indication of a predominance of kaolinite associated to quartz. The Lokoundje alluvial clays are mainly aluminous with Al$_2$O$_3$ contents varying between 23.29 to 30.26 wt.% (Table 2; Figure 3). These sediments are also characterized by low contents in Fe$_2$O$_3$ which vary between 1 and 5.42 wt.% (Table 2). These contents are less than 3 wt.% in white and light grey materials (Table 2). The color of the products after sintering is due to the iron contents (Table 2). The white samples are characterized by lowest iron contents, and the color of products remains the same after sintering (Table 2). The flux oxides (Na$_2$O + K$_2$O) are very low (1.08–2.50 wt.%), these low flux contents may induce poor vitrification. Fluxes, in reaction with silica and alumina, stimulate liquid phase formations that facilitate densification [24]. The low Na$_2$O and K$_2$O contents can be related to the absence of Na-/K-feldspars. In addition, due to low flux contents, a high firing temperature for improved sintering may be needed. Figure 4 shows that these alluvial clays are suitable for the manufacture of white stoneware tiles.

Table 2. Major element compositions (in wt.%) and element ratios of the Lokoundje alluvial clays [18].

| Element | Yellow Materials | Reddish Yellow Materials | White Materials | Light Grey Materials |
|---------|------------------|--------------------------|-----------------|---------------------|
|         | BR-5 | BR-8 | BR-9 | BR-11 | GR-2 | GR-3 | GR-6 | GR-8 |
| SiO$_2$ | 0.01  | 44.33 | 46.73 | 51.12 | 51.41 | 45.64 | 57.61 | 57.84 | 48.48 |
| Al$_2$O$_3$ | 0.01  | 30.26 | 29.51 | 26.07 | 24.77 | 29.36 | 24.23 | 23.29 | 27.13 |
| Fe$_2$O$_3$ | 0.01  | 4.20  | 4.31  | 5.42  | 4.33  | 3.33  | 1.28  | 2.16  | 2.90  |
| MnO     | 0.01  | 0.01  | 0.02  | 0.02  | 0.01  | 0.01  | 0.01  | 0.01  | 0.01  |
| MgO     | 0.01  | 0.34  | 0.38  | 0.36  | 0.40  | 0.37  | 0.15  | 0.27  | 0.38  |
| CaO     | 0.01  | 0.03  | 0.03  | 0.04  | 0.04  | 0.03  | 0.02  | 0.03  | 0.04  |
| Na$_2$O | 0.01  | 0.06  | 0.08  | 0.10  | 0.13  | 0.08  | 0.09  | 0.12  | 0.10  |
| K$_2$O  | 0.01  | 1.00  | 1.03  | 1.06  | 2.37  | 1.00  | 1.85  | 1.68  | 1.71  |
| TiO$_2$ | 0.01  | 1.73  | 1.48  | 1.24  | 2.26  | 1.55  | 1.97  | 1.39  | 2.18  |
| P$_2$O$_5$ | 0.01  | 0.31  | 0.19  | 0.12  | 0.34  | 0.25  | 0.35  | 0.12  | 0.37  |
| LOI     | 0.01  | 16.89 | 15.58 | 13.79 | 16.14 | 17.60 | 11.55 | 12.36 | 15.77 |
| Total   | -    | 99.15 | 99.33 | 99.36 | 99.21 | 99.22 | 99.10 | 99.26 | 99.07 |
| SiO$_2$/Al$_2$O$_3$ | -    | 1.46  | 1.58  | 1.96  | 2.08  | 1.55  | 2.38  | 2.48  | 1.79  |
| Na$_2$O + K$_2$O | -    | 1.06  | 1.11  | 1.16  | 2.50  | 1.08  | 1.94  | 1.80  | 1.81  |

d.l.: detection limits.
Figure 3. Plotting of the Lokoundje alluvial clays in Al₂O₃-CaO-(Na₂O + K₂O) (a) and Al₂O₃-CaO-Fe₂O₃ (b).

4.3. Thermal Behavior and XRD Mineralogical Evolution in Fired Samples

The thermal analyses (TGA-DTG) measurements are reported in Figure 5. The thermal analyses show three mass losses: the first loss occurs around 60–66 °C and corresponds to the loss of adsorbed water [25–27]. The second mass loss at 241–252 °C is associated with the conversion of goethite into hematite, and the last one at 489–492 °C is related to the dehydroxylation of kaolinite [28]. The goethite to hematite conversions confirm that iron is mainly present in form of goethite and that this will probably lead to a reddish color in the fired sample. The variation in the temperature of the last mass loss (489–492 °C) is associated with the crystallinity difference of the kaolinite [25]. As previously reported, FTIR revealed that kaolinite has a high degree of disorder, characteristic of several alluvial environments [18,29]. The intensity of the dehydroxylation peaks (489–492 °C) is coherent with kaolinite-rich materials and the associated mass loss varied between 14% and 18%; these values are consistent with the theoretical weight loss from the kaolinite ideal formula of 14%.

The color of the specimens is variable as a function of the initial color of the raw materials, as well as their mineral compositions. The pale brown sample becomes light red; the light gray becomes dark red or white; and brownish gray becomes pink (Table 3, Figure 6). This is associated with the fact that, during the sintering process, goethite is

Table 2. Major element compositions (in wt.%) and element ratios of the Lokoundje alluvial clays

| Element | Composition (wt.%) |
|---------|-------------------|
| Fe₂O₃   | 4.34              |
| Total   | 99.15             |
| LOI     | 0.85              |
| Al₂O₃   | 16.89             |
| CaO     | 30.26             |
| Na₂O    | 3.05              |
| K₂O     | 0.31              |
| TiO₂    | 0.01              |
| MgO     | 0.01              |
| SiO₂    | 0.01              |

Figure 4. Empirical diagram defining suitable domains for different types of tiles: (a) white stoneware tiles; (b) red stoneware tiles; (c) and (d) porous tiles.
converted into hematite, which justifies the reddish and brownish colors, as suggested by the thermal analyses. The whitish color is mainly the mark of a sample with low iron content [30]. The sound of the fired products is metallic (Table 3), which is indicative of vitrification that accounts for improved sintering. The cohesion of the fired products is good to very good (Table 3). The weak cohesion of sample BR9 may be related to the proportion of coarse particles. The mineral assemblage of the fired products, from XRD analyses (Figure 7), is made of quartz, mullite, rutile and hematite. Mullite is developed from the spinel phase, which is formed from the demixion of metakaolin that is formed after kaolinite dehydroxylation [31,32]. The illite peak, although low, is still observable in the products fired at 800 °C and 900 °C. Illite dehydroxylation evolved from 300 °C to 700 °C. The relic illite structure breaks down between 700 °C and 850 °C, leading to a liquid phase formation, which crystallizes to form a spinel which crystallizes until it changes to mullite above 1100 °C [33]. In the particular case of these materials, the mullitization peaks, observed from 800 °C, are associated with the structural disorder of the clay minerals present in the raw samples.

4.4. Technological Characterization of Fired Products

The linear shrinkage increases with the firing temperature (Figure 8a). The loss of weight varies between 5 and 17%. The values are almost constant for all the firing temperatures (Figure 8b). This may be justified by the low organic contamination of the samples or to the predominance of clay minerals.

The flexural strength increases with the firing temperature because of an increase in densification (Figure 8c). This densification is supported by the XRD patterns, in which an increase in mullite formation with temperature is noticeable. The highest value obtained was 15.75 MPa for sample BR5 fired at 1100 °C. For all other samples, the highest value of flexural strength was reached at 1100 °C (Figure 8c). This can be explained by the high Al₂O₃ content [34] and/or the increased formation of a dense phase, such as mullite during sintering [35]. The bulk density (Figure 8d) followed the same trend as the flexural strength,
which was consistent with the proposed increase in density upon firing, because of dense phase formation. Between 800 and 1000 °C, the bulk density is almost constant. This may be due to the similar mineralogical composition of the samples, in which the main dense phases are quartz and a primary mullite from 1000 °C (Figure 7). The density of the BR5 sample was also the highest among the series. Given that its clay size fraction, from particle size analyses, was the highest (76%) for the GR2 sample, then the mullite formation from sintering was higher in these samples compared to others. The difference reached in the flexural strength was probably due to crystallinity differences, which may affect the kinetics of mullite formation.

Table 3. Color, sound, and cohesion of the raw materials and fired products.

| Sample | Temperature (°C) | Color          | Sound  | Cohesion |
|--------|------------------|----------------|--------|----------|
| BR-5   | 25               | Pale brown     | -      | -        |
|        | 800              | Light red      | Metallic | Good    |
|        | 900              | Light red      | Metallic | Good    |
|        | 1000             | Light red      | Metallic | Very good |
|        | 1100             | Light red      | Metallic | Very good |
| BR-8   | 25               | Pale brown     | -      | -        |
|        | 800              | Reddish yellow | Metallic | Good    |
|        | 900              | Reddish yellow | Metallic | Good    |
|        | 1000             | Light red      | Metallic | Good    |
|        | 1100             | Light red      | Metallic | Very good |
| BR-9   | 25               | Light gray     | -      | -        |
|        | 800              | Reddish yellow | Metallic | Weak    |
|        | 900              | Light red      | Metallic | Average |
|        | 1000             | Red            | Metallic | Average |
|        | 1100             | Dark red       | Metallic | Good    |
| BR-11  | 25               | Reddish yellow | -      | -        |
|        | 800              | Reddish yellow | Metallic | Good    |
|        | 900              | Reddish yellow | Metallic | Good    |
|        | 1000             | Reddish yellow | Metallic | Very good |
|        | 1100             | Light red      | Metallic | Good    |
| GR-2   | 25               | Brownish gray  | -      | -        |
|        | 800              | Reddish brown  | Metallic | Good    |
|        | 900              | Pink           | Metallic | Good    |
|        | 1000             | Pink           | Metallic | Very good |
|        | 1100             | Pink           | Metallic | Good    |
| GR-3   | 25               | Light gray     | -      | -        |
|        | 800              | Pinkish white  | Metallic | Good    |
|        | 900              | Pinkish white  | Metallic | Very good |
|        | 1000             | Pinkish white  | Metallic | Very good |
|        | 1100             | White          | Metallic | Good    |
| GR-6   | 25               | Brownish gray  | -      | -        |
|        | 800              | Reddish yellow | Metallic | Good    |
|        | 900              | Reddish yellow | Metallic | Good    |
|        | 1000             | Pink           | Metallic | Very good |
|        | 1100             | Pink           | Metallic | Very good |
| GR-8   | 25               | Pale brown     | -      | -        |
|        | 800              | Light red      | Metallic | Good    |
|        | 900              | Light red      | Metallic | Good    |
|        | 1000             | Light red      | Metallic | Very good |
|        | 1100             | Light red      | Metallic | Very good |
Figure 6. Pictures of specimens.

Figure 7. X-ray patterns of the clay sintered at different temperatures: (a) GR3 sample; (b) GR6 sample; I: illite; Q: quartz; M: mullite; R: rutile; He: hematite.
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The decrease in the water absorption (Figure 8e) is in agreement with the change in the flexural strength and the bulk density as a result of improved sintering. These values were considerably reduced to less than 20% at 1100 °C, except for in two samples (GR-2 and BR-11), probably because of a glassy phase formation that diffuses between the grains, causing pore enclosure. The water absorption was less than 25%, indicating that these clayey materials can be of use for tile manufacturing. At 800 °C, the sintering was not sufficient to improve densification, which explains the high level of water adsorption.

4.5. Microstructure

An examination of the microstructure under SEM reveals the presence of several cracks at 1000 °C (Figure 9a–e,g) and rounded pores at 1100 °C (Figure 9f,h). The presence of cracks confirms low densification during sintering at temperatures <1100 °C. The abundance of alumina, the low flux contents, as well as the low Fe2O3 contents (Table 3) can confirm this behavior [36]. Additionally, at 1000 °C, the densification is still poor as mullite formation is still limited (Figure 9a,b). The low densification might also be linked to the low mullite formation. As reported in [37], before 1100 °C, XRD peaks associated to mullite more often account for the spinel that will crystalize as mullite. The mullite needle starts to be observable from 1100 °C; at this temperature, the strength is relatively high.
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Figure 9. SEM images of the fired products from the Kribi alluvial clays: Gr-3 sample at 1000 °C (a–d); Gr-3 sample at 1100 °C (e–h).

5. Conclusions

The present study aimed to investigate the physical, chemical, mineralogical and technological properties of fired alluvial clay specimens from Lokoundje in order to evaluate their potential suitability as raw materials in ceramics.

Mineralogical analyses of the Lokoundje alluvial clays indicate that they are mainly composed of kaolinite and quartz. The flexural strength, linear shrinkage, bulk density, and weight loss increase for fired products, while the water uptake decreases. These property changes are consistent with an increased sintering upon firing temperature increase. All the shrinkage remains low (<10%); the values are relatively high for samples GR2, GR8, BR5, and BR8, as a result their relatively high clay contents in comparison to others. Except for GR3 and BR11, the flexural strengths of the other samples were >2 MPa from 900 °C, which make these materials potentially usable for structural-brick making. The mineral assemblage after firing is essentially made of mullite and quartz. However, the quartz proportion and the low vitrification associated with the low feldspars content explained sample cracking for firing at temperatures >900 °C. The high Al₂O₃ contents and the small amounts of goethite indicate that Lokoundje alluvial clays could be used for white ceramic manufacturing. However, for improvements in the performance of fired products, an amendment with fluxes should be considered.
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