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Geotechnical Properties and Geochemical Composition of Mudrock from the Douala Sub Basin, Cameroon: Implication for Industrial Potentials

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Abstract The geotechnical and compositional characteristics of mudrock deposits in the Douala Sub basin were investigated using a combination of analytical methods, including particle size distribution, Atterberg limits, mineralogical (X-ray diffraction) analyses, and whole rock geochemistry. The goal is to characterize the nature and physicochemical properties of mudrock in order to determine its applicability in industries. Grain size analysis reveals that clay-sized particles dominate the samples, with a plasticity index ranging from 6.8% to 20.67%. The mudrock materials are primarily composed of kaolinite (16.8-49.4%), quartz (15.8-68.9%), and illite (00-15.3%), which are typical of the Douala Sub- basin sedimentary environment and morphoclimatic conditions. SiO_2 (42.77-73.5%) and Al_2O_3 (13.13-29.98%) are the most abundant oxides in the samples. Iron oxide content is moderate (1.73- 17.18%). Methylene blue values range from 1.12 to 6.95, confirming the clay content of (39.43-45.43%) and also attesting that the sediments in the study area are rich in 1:1 clay. They are suitable for ceramic applications such as (refractory bricks and tiles) and pottery due to the physicochemical parameters associated with mineralogical and geochemical data

Keywords: *Mudrock, clay mineral, characterization, valorisation, Douala Sub basin Cameroon*

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1. Introduction

The use of clay-rich materials is critical in the production of ceramics and building materials. However, clayey deposits are known to form from sedimentary, alluvial, and residual sources. Since antiquity, humans have valued clay-rich materials for the production of bricks and other articles [1]. It is necessary to have a good understanding of the occurrence, quantity, and properties of clay deposits in order to properly and efficiently exploit them [2]. The development of the global economy, infrastructure growth, and economic sector growth are all dependent on industrial materials [3]. Clay-rich materials are used in a variety of applications, including ceramics, paper, paint, [4], rubber and plastics, insecticides, food additives, cosmetics, pharmaceuticals, drilling fluids, fertilizer carriers, and geochemical barriers [5]. Due to its wide range of industrial applications, clay is now regarded as an indispensable development tool. Mineralogical and chemical composition, malleability, thermal behaviour, colour, and strength after baking are all important properties in the ceramic industry [6]. Many authors have produced scientifically significant documents in the Douala Sub basin, particularly in the fields of stratigraphy and tectonic evolution [7,8,9,10]. In the 1980s, the presence of clay in the Douala sedimentary basin attracted an industrial set for the production of ceramics and construction materials [11]. Several clay deposits have been discovered in the Douala Sub Basin, including Missole, Bomkoul, and Yansoki. The chemical and mineralogical composition of Bomkoul clay revealed its kaolinitic nature [12,13,14]. The firing properties of ceramics was depicted by [15]. Preliminary studies on clayey materials from the Bomkoul area was carried out by [16] and [17] highlighted the physicochemical and mineralogical characterization of Yansoki area. Most of the research works cited above within the Douala Sub basin on fine grained sediment are limited to particular localities. Despite the impressive work done on clayey materials in the Douala Sub basin, no comparative study has been conducted to bring out the physico-chemical characteristics of mudrock found in various localities such as Kombe, Kompina, Tonde, Ngoma, Djapoma, Missole, and Loungahe. The goal of this research is to characterize mudrock samples using geotechnical, mineralogical, and chemical composition in order to assess their suitability for industrial applications. This research will add to the existing literature on the industrial application of clays in Cameroon.

2. Geological Setting and Geographical Setting

The study area includes the central and north-western parts of the Douala Sub Sedimentary basin. It is situated between the latitudes of 3°10'-4°12' N and the longitudes of 9°50'- 9°52". (Figure 1). The Douala Sub-basin was formed during the opening of the South Atlantic Ocean and is bounded to the south by the Kribi-Campo Sub-basin and to the north by the Rio Del Rey basin (Figure 1). The sedimentary formations are related to the three stages of geodynamic and sedimentary evolution that have affected the basins since their formation [18,19].

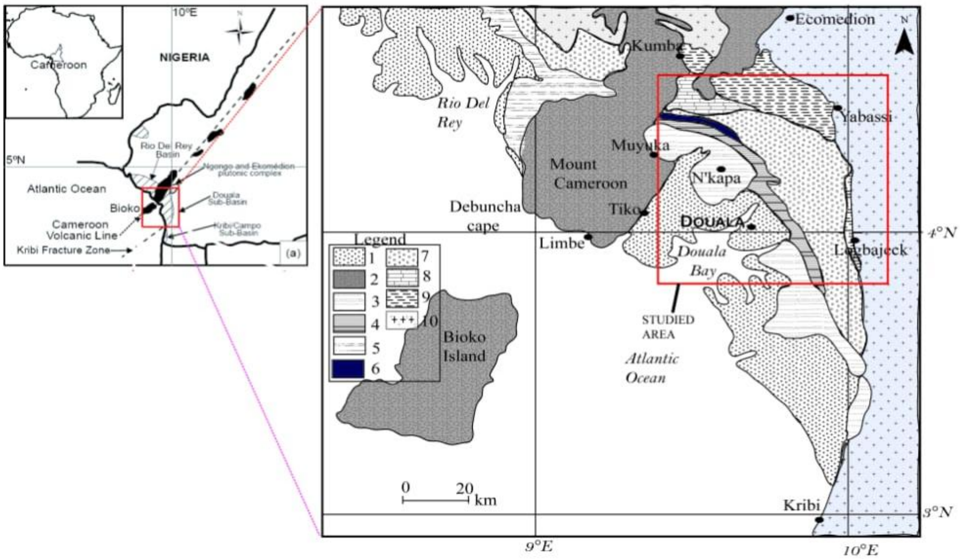

Figure 1. Location of the study area including (a) map of Cameroon highlighting the location of the study area within the Douala Basin in the Littoral Region and (b) geological map of Douala Basin modified from [20]: (1) Recent alluvium, (2) Tertiary volcanic rocks (basalts and trachytes), (3) Neogene (siltstones, sandstones), (4) Lower Eocene (bedded clays, claystones, silts, loose sandstones), (5) Undifferenciated Tertiary, (6) Paleocene (marine facies: claystones, dolomites, sandstones, silts), (7) Paleocene (continental facies: small conglomerates, loose sandstones), (8) Upper Cretaceous (clays, sands, sandstones, marly and calcareous limestones), (9) Lower Cretaceous (Basalt sandstone), (10) Precambrian basement (migmatitic gneisses + granites)

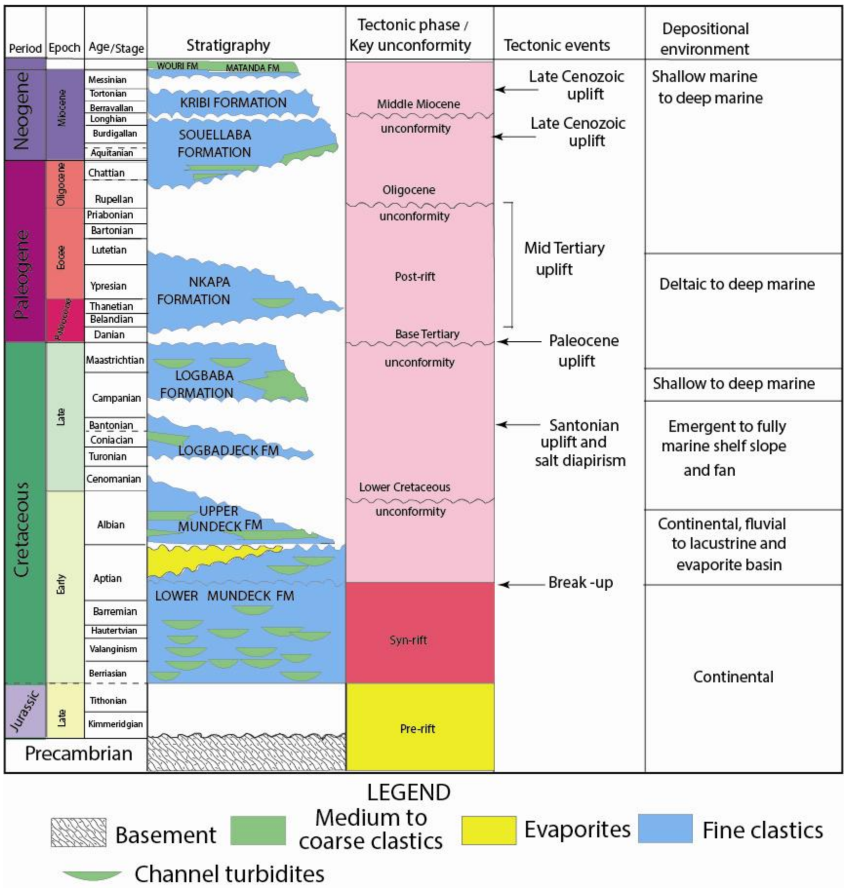

Figure 2. Tectono-stratigraphic framework of the DKC (modified from [21])

The lithostratigraphic entities of the Douala Sub basin are made up of seven formations that are linked to its geodynamic and sedimentary evolution (Figure 2). These formations are distinguished by Cretaceous, Tertiary, and Quaternary sediments that are locally covered with lateritic cuirasses [19]. The Mundeck Formation, which represents the syn-rift period (Aptian-Cenomanian), is composed of continental and fluvio-deltaic deposits such as clays, coarse-grained sandstones, and conglomerates. The remaining six formations representing the post-rift period are: (1) the Logbadjeck Formation (Cenomanian-Campanian), which is discordant to the Mundeck Formation and is composed of microconglomerates, sand, sandstone, limestone, and clay; (2) the Logbaba Formation (Maastrichtian), which is composed primarily of sandstone, sand, and fossiliferous clay; and (3) the Paleocene-Eocene Nkapa Formation, rich in marl and clay with sand lenses and fine to coarse-grained sandstone; (4) the Souellaba Formation (Oligocene) lying unconformably on Nkapa deposits and characterized by marl deposits with some interstratified lenses and sand channels; (5) the Matanda Formation (Miocene), characterised by deltaic facies interstratified with volcano-clastic layers; and (6) the Wouri Formation (Plio-Pleistocene) which is composed of gravelly and sandy deposits and a clayey matrix.

3. Materials and Methods

The raw materials for this study came from Kombe, Kompina, Tonde, Ngoma, Djapoma, Missole, and Loungahe (Figure 3), and samples were collected along road cuts, valleys, hills, and river channels. To avoid contamination, 13 representative samples were assembled and stored in sample bags. Physical and chemical analyses were performed on the samples. The samples were analyzed using XRD. Prior to laboratory analysis, the samples were air dried and gently crushed to increase surface area. The liquid limit, plastic limit, and plasticity index, particle size, and methylene value were then determined. The physicochemical properties mentioned above were determined at the Geotechnical laboratory of Fotso Victor University Institute of Technology in Bandjoun. The laboratory experiments performed on each sample, as well as the methods employed, are briefly described below.

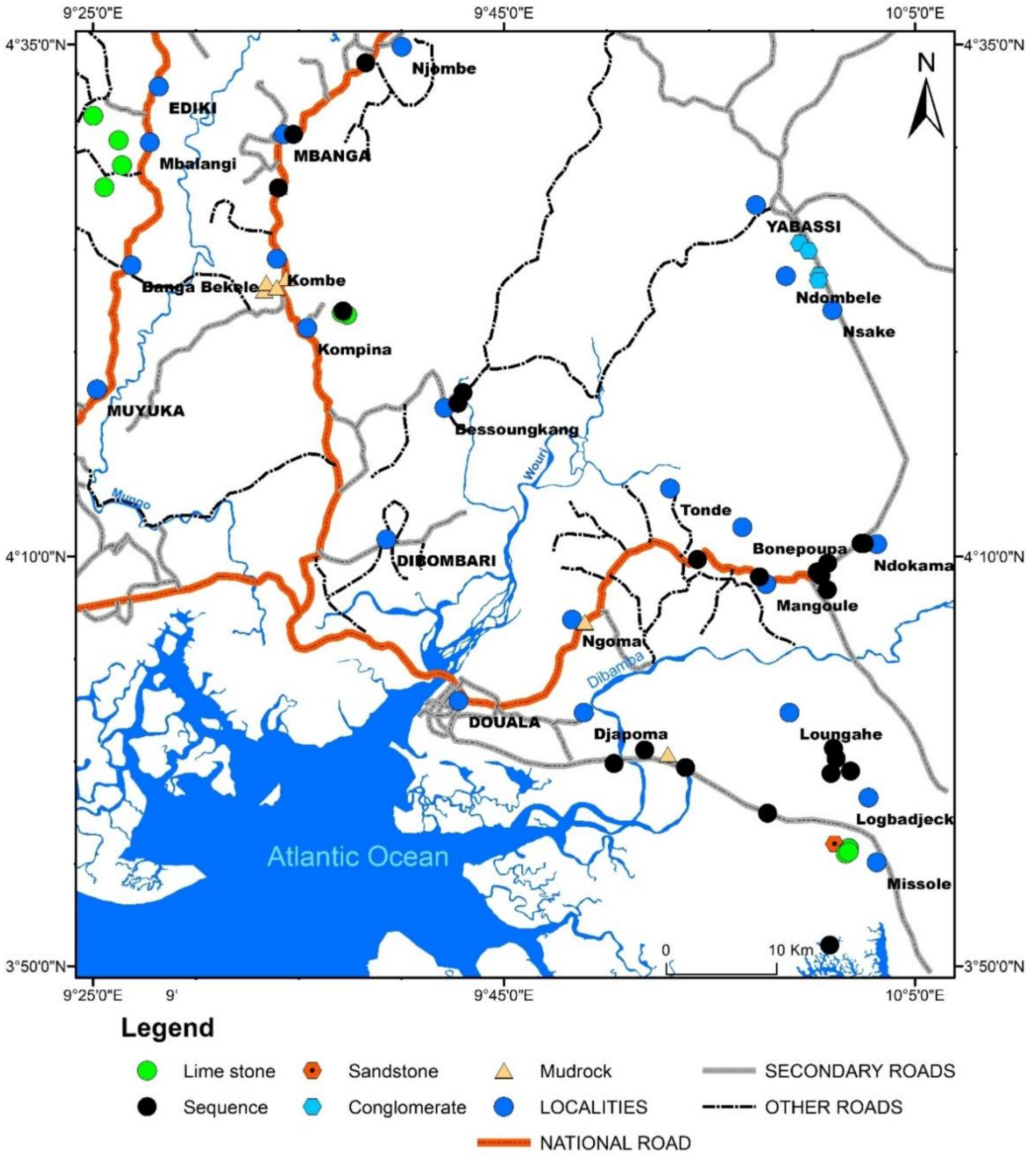

Figure 3. Sample map of the study area

The Atterberg limit was used to determine the plastic behaviour of materials in accordance with the French standard NFP 94- 051 [22]. The Cassagrande apparatus was used to calculate the liquid limit (LL) and plastic limit (PL) (PL). The plasticity index was calculated by subtracting LL from PL.

Sieve analysis was performed on a 500 g sample using ASTM C 136 as a reference, and the soil was washed with an 800 µm sieve. The sediment which remains on the sieve relates to sand and pebble group, while the part that goes through accounts for clay-silt fraction. After 24 hours in the oven, the material that accumulated on the 800 µm sieve was used for sieve analysis, while the fraction that passed through was used for hydrometer analysis. The clay particle size distribution was determined using wet sieving in accordance with the NFP 94-093 French standard [22] and a dispersive agent of sodium hexametaphosphate ($(NaPO_3)_6$, 5wt%).

The methylene blue test was performed using a methylene blue standard solution made by combining 10 g ± 0.1 g of the powder with distilled water and stirring for 45 minutes at room temperature. A sample that has been baked for 24 hours at 105℃ is weighed and gently crushed with a spatula before being placed in a beaker. Before adding 500 ml distilled water, the sediment is stirred for 5 minutes with a speed adjustable blender at 600 ± 60 rpm (revolutions per minute). Following the above procedure, 5 ml of solution is added to the mixture and blended for 1 minute at a rate of 400 ± 40 rpm and a drop of the suspension is taken out. The first drop will typically have a dark blue stain and a water circle surrounding it. The procedure is repeated, adding solution until a light-blue circle (halo) forms around the central blue stain. In the first 5 minutes, another 5ml of solution is added. After 5 minutes, the solution volume is reduced to 2ml and stirred for 1 minute while a drop of the suspension is extracted using a glass rod and dropped on filter paper to observe the circle formation. When a light blue circle forms around the central blue stain, the experiment is considered complete. The volume of methylene blue (VBS) is obtained through; (used methylene blue/ dry mass of sample *100).

The mineralogical and bulk chemical analysis was performed in the clay, geochemistry and sedimentary environments (AGEs) laboratory of the University of Liège in Belgium. In this study, the advanced Burker D8 diffractometer (copper radiation K1, = 1.5418X, V = 40 kV, I = 30 mA) was used in accordance with the methodology proposed by [23]. The diffractograms are obtained from disoriented powder total fractions with measurements in 2 ranging from 2° to 45°, a scanning pitch size of 0.02°, and a time per step of 2s).

4. Results and Discussion

4.1. Geotechnical Characteristics

Grain size distribution and plasticity index are two important factors that influence the suitability of fine-grained materials for various industrial applications, and special consideration should be given to the finer (< 2 um) fraction for ceramic [24,25,26]. The particle size distribution result is shown in (Table 1). The particle distribution of the studied fine-grained materials varied from facies to facies, with proportions ranging from (39.43-49.33) for clay, (29.23-35.57) for silt, and (15.10-26.98) for sand.

The results of the samples show that the clay size particle (0.002 mm) is the most dominant, accounting for more than 39% (Table 1) of all samples. The high amounts of fine particles (silt + clay) and fine clay minerals in the samples support the plasticity index (6.8% to 20.67%). As shown in (Table 2), [27], the amount of clay fraction is used to determine the manufactured products. However, particle size and plasticity index are two of the most important geotechnical characteristics used to select a suitable construction material.

The results of the samples show that clay size particle of (<0.002 mm) is the most dominant, constituting greater than 39% (Table 1) in all the samples.

In this case, considering the amount of sand (22, 19 to 26.89 wt. %), fine particles (40. 07 to 48.58 wt. %), and plasticity index, samples MT, KC, NO, LR3, TD1, and KR1 are suitable for manufacturing bricks, tiles, and sandstones [28].

Methylene blue values range from 1.12 to 6.95, confirming that the sediments in the study area are rich in 1:1 clay (39.43-45.43%). (Table 1).

The Belgian textural classification diagram [29] allows for the identification of specific fields of application for ceramic clay materials. Particle size plotting in this textural classification diagram of clay materials reveals that the samples fall into the clay domains (Figure 4). Grain size data were also plotted on ternary diagram proposed by [30] to demonstrate clay material suitability for ceramics (Figure 5). The plots show that the samples can be used to make tiles and masonry blocks.

Table 1. Grain size distribution of the studied samples

Sample code	MT	BG1	JA2	KC	MZ1	KW	DA1	NO	KB	LR3	TD1	KR1	BE1
Sand (2> Ø >0,02mm) %	26,89	26,87	25,54	23	26,81	25,45	26,84	22,19	26,75	22,37	25,78	26,46	26,82
Silt (0,02>Ø >0,002 mm) %	30,01	32,58	31,9	31,57	32,66	32,4	33,73	29,23	33,01	32,42	31,41	32,76	33,11
Clay (Ø <0,002mm) %	43,1	40,55	42,56	45,43	40,54	42,15	39,43	48,58	40,24	45,21	42,81	40,78	40,07

Table 2. Relation between particles size and type of ceramic product (Cere and Mazel, 1993).

Ø <2µm (Wt.%)	Ceramic products
5-25	Full bricks
25-35	Perforated brick
35-45	Hollow bricks, drains
45-50	Tiles, hordes

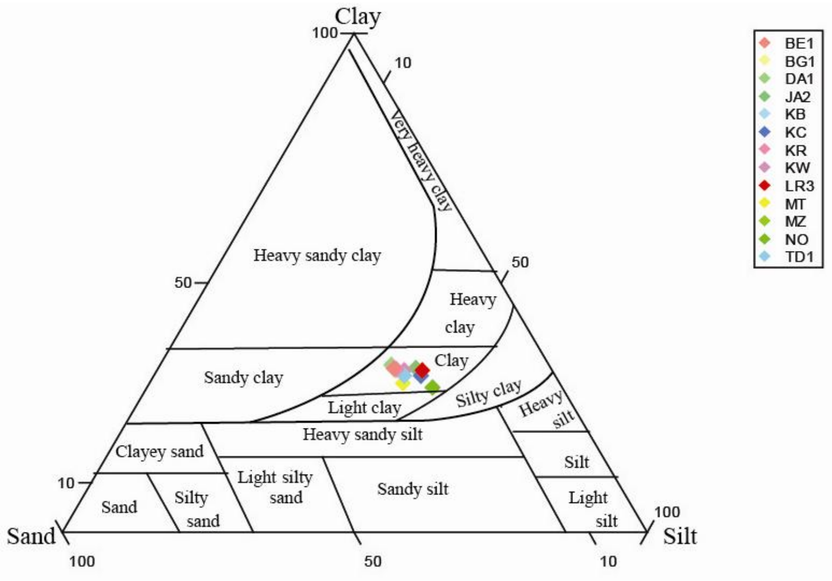

Figure 4. Representation of mudrock samples on the Belgian textural ternary diagram [29]

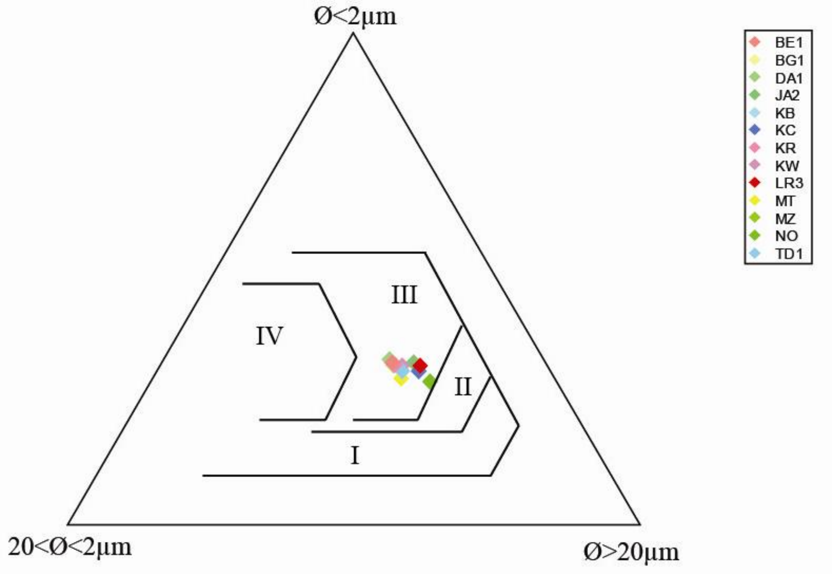

Figure 5. Ternary diagram defining the domains of granulometric composition of diverse raw materials for baked brick [30]. I. Solid brick II. Perforated vertical blocks III. Tiles and masonry blocks IV. Hollow products

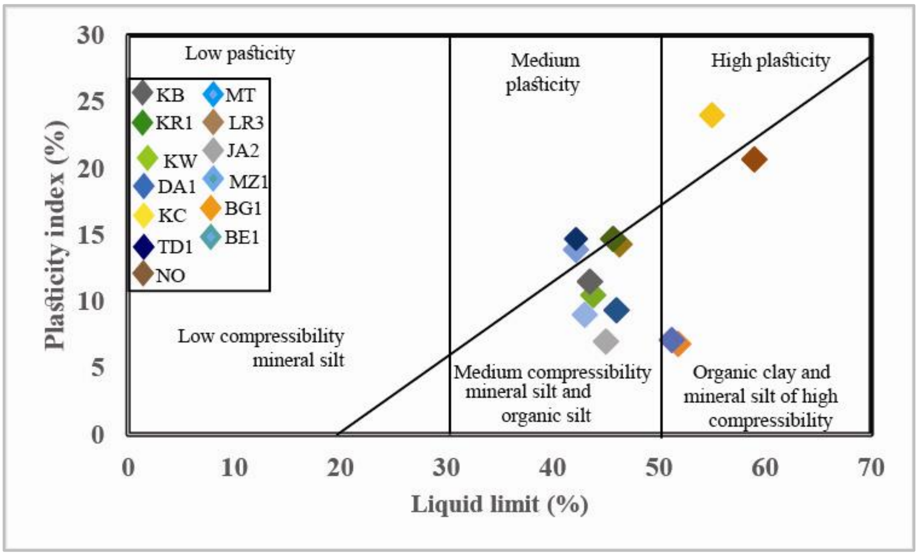

Figure 6. Cassagrande plasticity chart for mudrock samples [31]

Plasticity is an important parameter to consider when manufacturing ceramics products [24] because it allows easier shaping and cohesion of manufactured products. The liquidity limit (LL), plasticity limit (PL), and plasticity index (PI) results are reported in (Table 3). The plasticity index ranges between 5.5 and 24.The plasticity index threshold limit at which a raw material is considered good for the ceramics industry is 10% [32]. Clay materials with a plasticity index of <10% can cause cracks in manufactured products, particularly during extrusion, and are thus unsuitable for building construction due to the significant variation in water quantity [33]. Nonetheless, samples with IP values of <10% could be used in raw structural ceramics. According to the findings of this study, the majority of samples have a plasticity index > 10%, validating their use as a good building material (Table 3). The clay plasticity diagram proposed by [31] shows that the majority of the samples fall in the medium compressibility domain, while samples KC, NO, BG1 and MZ fall in the high compressibility domain of organic clays and mineral silts (Figure 6).

Plasticity and particle size of sediment are two most important and related parameters. Particle size distribution and mineralogical composition influence the plasticity of clayey materials [34]. Similarly, the dominance of kaolin is attributed to the plastic nature of clays [35]. The diagram proposed by [36] was used to determine the ceramic properties of the investigated sediments (Figure 7), and it revealed that the materials are suitable for ceramics with the exception of samples MZ1 and TD1, which are suitable for pottery. Nonetheless, the addition of degreasers is required to reduce plasticity and improve material cohesion, making them suitable for ceramics.

Table 3.

Sample code	MT	BG1	JA2	KC	MZ1	KW	DA1	NO	KB	LR3	TD1	KR1	BE1
Liquid limit, LL (%)	42,2	51,8	45	55	51,2	43,8	46	59	43,5	46,3	42,2	45,7	43
Plastic limit ,PL (%)	28,3	45	38	31	44	33,33	36,67	38,33	32	32	27,5	31	34
Plasticity index ,PI (%)	13,9	6,8	7	24	7,1	10,47	9,33	20,67	11,5	14,3	14,7	14,7	9
Methylene blue value	1,45	2	4,3	6,95	0,75	2,3	1,85	2,75	1,95	1,12	3,4	3,25	2,05

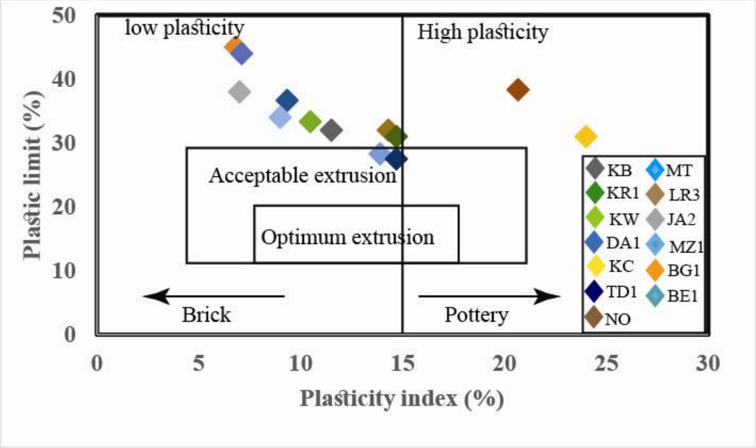

Figure 7. Evaluation of extrusion prognostic on Bain's diagram [36]. Mudrock materials are suitable for brick (BG1, JA2, KC, MZ1, KW, DA1, NO, KB, LR3, KR1, BE1) and pottery making (list them). Samples MT and TD1 are acceptable for brick production by extrusion

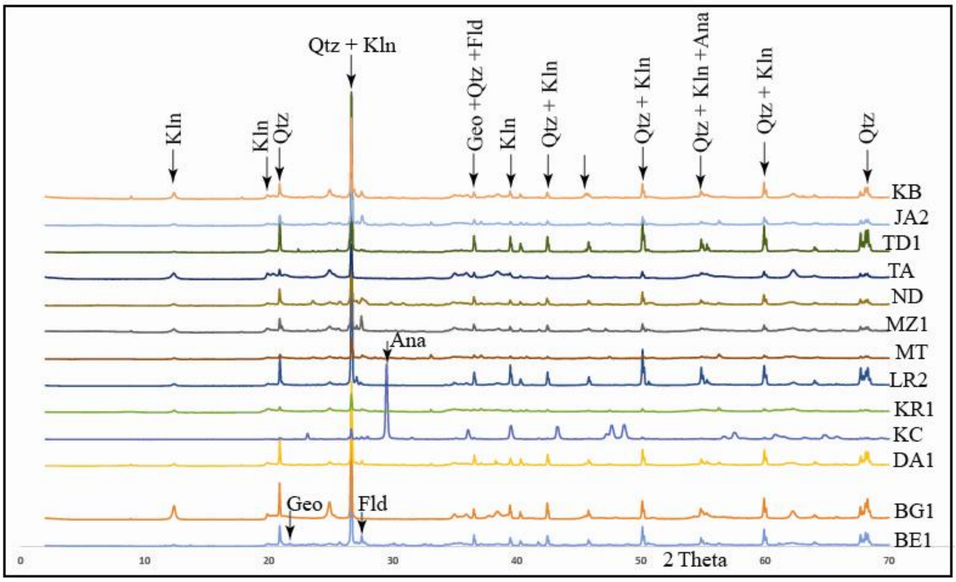

Figure 8. X-ray diffactogram of mudrock samples

4.2. Mineralogy

The mineralogical analyses of the studied materials are presented in (Figure 8) and (Table 3). The mineralogical composition of mudrock from the study reveals several mineral phases comprising of kaolinite, illite, quartz, K-feldspar, plagioclase, anatase, gibbsite, actinolite and goethite.

Clay mineral represents the bulk of the mineralogical assemblage in most of the samples (Table 4). Amongst these mineral variation, clay minerals constitute the greatest proportion with respective percentages being; 10.2% (BG1) - 49.4% (KB). Aside clay minerals, a considerable proportion of quartz is equally noticed in all the samples with percentage varying from 15.8 % (MT) to 68.9% (DA1).

The percentage of goethite ranges from 2.2% (DA1) to 20.4%. (MT). It is high in samples MT, NO, and BG1 (12.2-20.4), but low in the other samples (2-6%). Gibbsite content ranges from 9.8% (JA2) to 15.3%. (BG1). Orthoclase has a relatively higher percentage in all samples, ranging from 7.2% (BE1) to 22.7%. (KR1). Actinolite content ranges from 2% (KC) to 10.2%. (MT). Anatase accounts for a smaller proportion (1%-3%). The clay fraction is primarily composed of kaolinite and illite. The most abundant mineral in the clay fraction is kaolinite. Its percentage ranges between 10 (BG1) and 49.47%. (KB).

The mineralogical composition of clay material influences its industrial valorisation [37]. The majority of the peaks in the studied material are quartz (Figure 7), implying a high silica content [37], which is attributed to its resistance to weathering. The presence of quartz is critical, as it can significantly improve soil geotechnical properties such as compaction, dry density, stiffness, and shear resistance, as well as reduce the shrinkage behaviour of sintered ceramic products [38]. The presence of kaolinite in the studied sediments demonstrates that monosiallitisation is the dominant process in the study area [37]. The availability of quartz and kaolinite in a material facilitates the shaping and drying of the ceramic products [39]. An elevated quantity of clay material increases plasticity and vitrification of ceramic products [39]. Based on the refractory properties of ceramics, samples KC and KB, which are plastic and rich in kaolinite, are good. The presence of Illite is detected in some samples, albeit at a low concentration (Table 4). This mineral improves clay plasticity by promoting the location of glazed phases during course firing [37].

4.4. Chemical Composition of Clay Materials

The chemical composition of the materials under consideration is shown in (Table 5). The materials are distinguished by a relatively high SiO_2 and Al_2O_3 content. Fe_2O_3 concentrations are moderate, while alkali and alkaline earth oxide concentrations are low. The high concentration of SiO_2 in most samples supports the DRX results that show quartz peaks in materials. The content of aluminum oxide (Al_2O_3) is relatively high, especially in samples KC and LR3, confirming the presence of kaolinite in all samples as seen on X-ray diffractogram peaks. The high concentration of kaolinite in the study area is linked to the morphology and climatic characteristics [3,40], with monosiallitization being the primary process. This process is usually accompanied by abundant rainfall and good drainage.

Table 4. Mineral proportions of mudrock samples

Sample code	MT	JA2	KC	MZ1	DA1	NO	BG1	BE1	KR1	TD1	LR3	KB	KW
Kaolinite	38.5	34.6	45,3	16.8	28.7	41.8	10.2	41.2	42.0	31.1	30.8	49,4	22.4
Illite	0	0	0	0	0	15.3	10.2	12.3	0	6.6	6.6	6.3	0
Gibbsite	0	9.8	15.0	12.3	0	0	15.3	0	0	0	0	0	4.0
Goethite	20.4	2.9	3.0	3.3	2.2	12.2	12.2	4.1	5.6	6.6	6.6	4.2	1.0
Anatase	1.1	1.9	2.0	0	0	0	3.0	2.0	1.1	2.2	2.2	1.0	0
Quartz	15.8	50.4	23.7	67.4	68.9	18.3	34.6	32.9	28.4	45.5	45.1	23.1	36.7
Plagioclase	0.1	0	0	0	0	0	2	0	0	0	0	0	0
Orthoclase	13.6	0	8.2	0	0	8.1	12.2	7.2	22,7	8.8	8.8	15.7	28.5
Actinolite	10.2	0	2.0	0	0	4.0	0	0	0	0	0	0	7.14

Table 5. Chemical composition of the materials studied

Sample code	SiO_2	TiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	Na_2O	K_2O	P_2O_5	LOI	SIO_2/AL_2O_3
MT	42,77	1,56	15,51	17,18	0,05	1,48	0,27	0,14	2,15	0,08	18,82	2,76
BG1	52,95	2,81	16,8	10,71	0,05	1,38	0,64	0,03	2,97	0,06	11,59	3,15
JA2	68,53	1,51	20,18	1,71	0,01	0	0,05	0	0,1	0,04	7,87	3,40
KC	49,14	2,43	29,98	3,72	0,01	0,18	0,06	0	0,09	0,09	14,31	1,64
MZ1	68,47	1,07	13,13	2,84	0,01	0,15	0,06	0	0,97	0,08	13,22	5,21
KW	68,36	0,6	16,13	1,73	0,02	0,4	1,46	2,47	5,21	0,05	3,57	4,24
DA1	73,5	1,36	11,07	2,91	0,01	0,34	0,09	0	1,34	0,28	9,11	6,64
NO	48,42	0,88	18,98	11,9	0,02	1,86	0,15	0	2,67	0,13	14,99	2,55
KB	64,61	2,73	15,34	4,4	0,03	0,35	0,14	0,2	3,1	0,04	9,05	4,21
LR3	56,89	0,93	22,12	3,39	0,01	1	0,03	0	3,08	0,05	12,5	2,57
TD1	63,13	1,07	14,35	4,68	0,02	0,62	0,09	0	1,3	0,04	14,72	4,40
KR1	57,03	1,49	18,35	4,82	0,02	0,81	0,3	0,38	3,18	0,09	13,54	3,11
BE1	60,86	2,09	19,53	3,81	0,01	0,19	0,05	0	2,03	0,07	11,37	3,12

This may clarify why materials of the Douala sedimentary sub-basin have relatively low to high proportion of kaolinite (10.2-49.4), greater than those of illite whose formation is related to different climatic conditions such as poor drainage, less precipitation, low gradient slopes and high evaporation [41].

This explicate the low concentration and complete absence of illite in some samples from the study area. The ternary diagram proposed by [42] was used to decipher the degree chemical weathering in the studied material (Figure 9). The projection of samples on the ternary diagram [42] indicates a high degree of alteration, confirming the morphology and climatic conditions of the basin. The presence of goethite [43] accounts for the low to moderate concentration of iron oxide (1.73-17.17). The very low alkali (Na_2O and K_2O) contents of the mudrock can be attributed to the low amount of feldspar [44]. The degree of chemical alteration of the studied materials was demonstrated using a ternary diagram of the evolution of the chemical index. The placement of the samples in the diagram proposed by [42] indicates a significant change (Figure 9). This is consistent with the morphology and climatic conditions of the sub region. The SiO_2/Al_2O_3 ratio indicates the amount of quartz and kaolinite present in clay materials. This ratio ranges from 1.64 to 6.64 (Table 5), confirming the DRX results that showed the predominance of kaolinite associated with quartz in the samples studied.

The geotechnical and mechanical characteristics of clayey materials are essential depending on their chemical and mineralogical compositions, as well as the mineral distribution [25]. The samples in this study are primarily composed of poorly crystallized kaolinite (10.2 to 49.4 wt. %), which is consistent with the loss on ignition (3.57% to 18.82%), illite (0,00 to 15.3 wt.%) and quartz (15.8 to 68.9 wt.%), are suitable for the composition of ceramic pastry [30] and for the manufacturing of bricks and tiles [14]. Also suitable for the manufacture of tiles and bricks are samples JA2, MZ1, KW, DA1, KB, TDI BE1 with silica percentage (SiO_2> 60 wt. %), alumina (Al_2O_3) (35 wt. %), and iron (10 wt. %) [45]. These materials are suitable for the production of tiles (LR3, BE1, and KR1) and stoneware (NO), according to plots on the ternary diagram proposed by [46], (Figure 10).

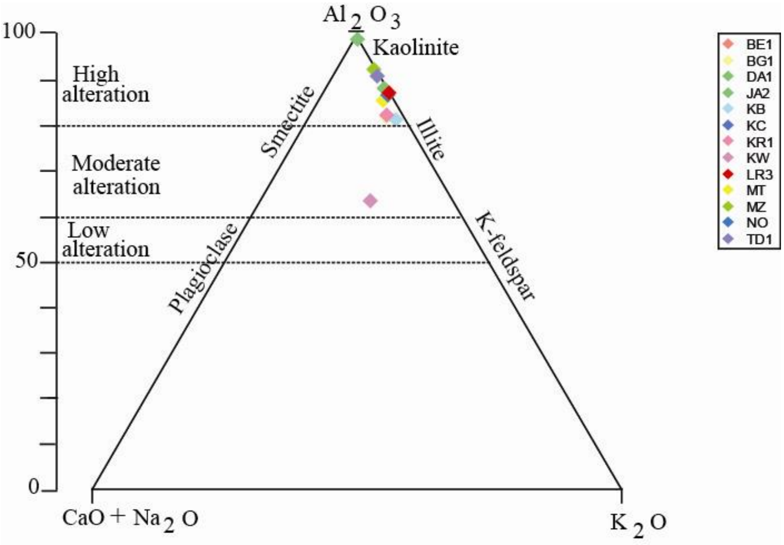

Figure 9. A-CN-K diagram of the studied mudrock materials [42]

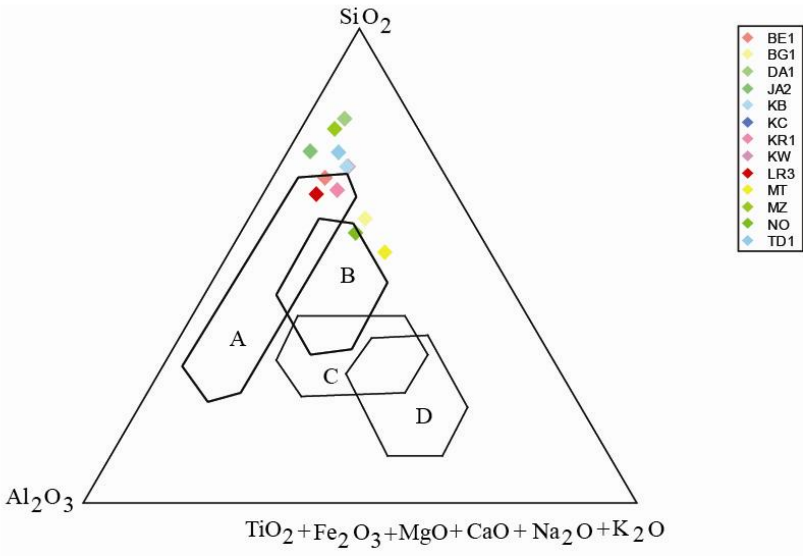

Figure 10. Ternary diagram defining the domains of chemical composition of raw materials for diverse ceramics products [46]; A: Tiles, B: Stoneware C & D: Porous tiles

5. Conclusion

Thirteen mudrock samples collected from various localities within the Douala Sub basin were subjected to mineralogical and physicochemical analysis. This research yielded the following conclusions:

The mudrock materials are primarily composed of sand (23 to 45 % wt), silts (17 to 33 weight percent), and clays (34 to 45 % wt), with a plasticity index ranging from 13.8% to 21.6%. Methylene values range from 1.12 to 3.25.

The dominant clay mineral in the mudrock samples is kaolinite while quartz, goethite, orthoclase, and illite are also present. The samples are siliceous and aluminous, and most clayey samples contain a significant amount of iron (Fe_2O_3 less than 10% wt). The materials studied are suitable for the production of ceramic and pottery based on the geotechnical, mineralogical, and chemical results. This research will add to the existing literature on the nature and properties of fine-grained materials, as well as project them in various fields of industrial application.

Conflict of Interest

The authors declare no conflict of interest.

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Author Contributions

Ndengwe Alexander Tangwa took part in the project design, did the field work, sample collection, sample processing, data interpretation and compilation, conceived and wrote the manuscript draft.

Njoh Oliver Anoh conceived the project, developed the field work strategy, analytical techniques used and data interpretation. He edited, read and amended the manuscript.

Nowel Yinkfu Njamnsi assisted during field work and equally read through the manuscript

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