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Physico-Chemical and Mineralogical Characterisation of Clay Geomaterials and a Typology of Quarries in the Korama Valley, South-East Niger

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Abstract

In Niger, clay geomaterials are widely distributed and extensively quarried. Unfortunately, these materials are still poorly investigated. This limits their potential for utilisation. The aim of this study is to characterise the clay geomaterials and to propose a typology of the various quarries in the Korama Valley in Niger. The clay geomaterials from these various quarries were characterised using X-ray diffraction and fluorescence, the mapping of lithological sections, and Principal Component Analysis (PCA) of the results. All the geomaterials are deposited in two types of horizons: superficial sedimentary horizons and deep alterite horizons. Two groups of geomaterials emerge: those characterised by an abundance of silica (mean SiO_2 = 73.67 percent) and a relatively high proportion of alkaline and alkaline earth elements (mean alkaline and alkaline earth elements = 2.54 percent). From a mineralogical perspective, they are characterised by the presence of montmorillonite. Geomaterials characterised by a relatively lower proportion of silica and alkaline and alkaline earth elements have relatively higher proportions of Fe_2O_3 (mean Fe_2O_3 = 13.75%) and alumina (mean Al_2O_3 = 25.69%). Their proportion of titanium dioxide (mean TiO_2 = 3.07%) is relatively high. From a mineralogical perspective, they are characterised by the presence of kaolinite. The proposed classification system has categorised the five (05) quarries sampled into two types; surface quarries and deep quarries. The surface quarries—Kolleram 1 and Garaké—are quarries containing montmorillonite-rich clay geomaterials,

which are rich in silica and alkaline earth elements, dark in colour and not very hardened; the deep quarries—Kolleram 2, Menari and Yaouri, are quarries containing kaolinite-based clay geomaterials, which are relatively low in silica and alkaline and alkaline earth elements, more compacted and reddish in colour. All these characteristics mean that the geomaterials from both the shallow and deep quarries differ from a physico-chemical and mineralogical perspective.

Keywords

Clay Geomaterials, Superficial Quarries, Deep Quarries, X-Ray Diffraction, X-Ray Fluorescence

1. Introduction

Quarries producing clay materials are extensively used because of the physico-chemical and mineralogical properties of their geomaterials [1] [2]. In Niger, the majority of applications for clay geomaterials are in the construction of houses and in pottery or ceramics in the broadest sense. The Zinder region—and more specifically the Korama Valley, given its geology—is an area with significant potential for clay materials. The pottery of Mirriah, a commune in the Zinder region, is well known beyond our borders. However, to make better use of these materials, it is important to understand them through their characterisation. The clay geomaterials exploited in Niger, and in Zinder in particular, remain scientifically poorly understood. Only a few publications [3]-[6] are available. The objectives of this study are to characterise (physico-chemically and mineralogically) the clay geomaterials and to propose a typology of the quarries operated in the Korama Valley, specifically at the Kolleram 1, Garaké, Kolleram 2, Menari and Yaouri quarries.

2. Presentation of the Study Area

The Korama Valley, the study area, covers an area of approximately 12,000 km^2 and occupies the southern part of the Zinder Region; it covers the whole of the Kantché and Magaria departments and the southern part of the Mirriah and Gouré departments [7]. It is a vast alluvial plain, oriented north-west to south-east, situated on the edges of the western Niger basin (Iullemeden Basin) and the eastern Niger basin (Lake Chad Basin). With a population of over 1,320,788 in 2009, distributed across twenty-six (26) municipalities—23 rural and three (3) urban—the Korama Valley is also the most densely populated area in the Zinder Region.

Its climate varies from the Northern Sudanese type in the south to the Sahelian type in the north, and is characterised by two distinct seasons: a short four-month rainy season (from June to September), followed by a longer dry season lasting around eight months. In parts of the area, rainfall exceeds 600 mm per year. It

receives the highest rainfall in the Zinder Region. In this part of the Zinder Region, floods are recurrent and devastating. The people of the Korama Valley use several types of banco, mainly black and red banco, to build their homes. These building materials are vulnerable to flooding. This explains the significant collapses of habitations and the resulting loss of life [8].

3. Materials and Methods

3.1. Materials

The quarries and clay geomaterials that were the subject of this study are situated in the localities of Kolleram (2 quarries), Garaké (1 quarry), Menari (1 quarry) and Yaouri (1 quarry), all of which are communes in the Korama Valley, Zinder's Region. These are quarries of clay geomaterials that have been used for many decades by the local populations in the construction of habitations and for other uses such as pottery.

Sampling:

The sampling operation focused on the most extensively exploited quarries in these four localities. Consequently, five (05) quarries were sampled. Two (02) in Kolleram, one (01) in Garaké, one (01) in Menari and one (01) in Yaouri. Two (02) profiles were surveyed per quarry. At each profile, samples were taken from three levels (upper, middle and lower), giving a total of three (03) samples per profile. A total of 30 samples were collected and label as follows:

- Kolleram quarry: KOL;
- Garaké quarry: GRK;
- Menari quarry: Nri
- Yaouri quarry: Yri
- Upper level: a
- Middle level: b
- Lower level: c

3.2. Experimental Methods

The methods used in this study require the material to be ground into a powder. Preliminary grinding, carried out using an agate mortar and pestle measuring 20 ml in diameter and 70 mm in height, produced millimetre-sized powders or fragments, free from contamination, from all 30 samples. This operation was carried out at the Spectrum Analytical Services Laboratory at the University of Kaduna in Nigeria.

X-ray fluorescence

The chemical compositions of the raw samples were determined by X-ray fluorescence spectrometry. These X-ray fluorescence (XRF) analyses enabled the chemical composition of the samples to be determined. Thus, in this study, the mass percentages of the compounds (major oxides) constituting the raw samples were determined. An "Epsilon 4" energy-dispersive X-ray fluorescence (EDXRF) spectrometer manufactured by PanAnalytical (Netherlands) was used for the

analysis. 20 g of the prepared sample were weighed and placed in each sample dish. The dishes, containing their contents, were carefully positioned in their respective measurement positions on the instrument's sample changer. The following parameters were set when the instrument was switched on.

- Determination of elemental composition
- Type of samples to be analysed: loose powder
- Voltage used: 14 kV for major oxides, 20 kV for trace elements and rare-earth metals.
- Filters selected: "Kapton" for major oxides, Ag/Al-thin for trace elements and rare-earth metals.

The selection of filters was guided by a given periodic table used for elemental analysis. Time of measurement for each sample was 100 seconds and the medium used was air throughout. The machine was then celebrated by the machines gain control, after which the respective samples were measured by clicking the respective positions of the sample changer. LOI was determined gravimetrically by heating 1 g of the powdered sample in a cleaned weighed crucible at 1000 oc. After which the crucible and the content was weighed to get the difference in weight before and after heating.

$$\text{LOI} = (a - b/1) \times 100\%$$

where a = weight of crucible + 1 g of the sample before heating; b = weight of crucible + 1 g of the sample after heating.

X-ray diffraction (XRD)

The mineralogical compositions were obtained from powder X-ray diffraction (XRD) using the Rigaku Miniflex XRD diffractometer, manufactured by the Japanese company Rigaku Corporation. This is a benchtop X-ray diffractometer used for the identification and quantification of phases, the determination of the percentage (%) of crystallinity and crystallite size, and the study of molecular structure. It is widely used in research, particularly in materials science, chemistry, the minerals and mining sectors, as well as in other relevant industries, for research and quality control purposes. The goniometer employs Bragg-Brentano geometry, with a radius of 150 mm, and has a measurement range (2θ) between 4˚ and 75˚. The X-ray source is a copper anode (λ Cu Kα = 1.5418 Å), I = 15 mA (fixed), U = 30 kV (fixed). Measurement speed ranges from 0.01 to 100˚/min.

The powdered samples are taken to the Analytical Unit for an XRD Analysis to be carried out on them.

One to two grams of powdered sample is weighed into the sample Holder and with the use of a glass slid, the sample is compacted to give an even surface area.

The sample holder is then placed on the XRD multi-samples holders chamber.

The machine is then calibrated (SET) to begin the analysis using the following conditions:

*MEAS_COND_XG_CURRENT "15"

*MEAS_COND_XG_VOLTAGE "40"

*MEAS_COND_XG_WAVE_TYPE "Ka"

*MEAS_DATA_COUNT "3401"
*MEAS_SCAN_AXIS_X "TwoThetaTheta"
*MEAS_SCAN_AXIS_X_INTERNAL "TwoThetaTheta"
*MEAS_SCAN_END_TIME "07/14/2023 09:17:09"
*MEAS_SCAN_MODE "CONTINUOUS"
*MEAS_SCAN_MODE_INTERNAL "CONTINUOUS"
*MEAS_SCAN_RESOLUTION_X "0.0025"
*MEAS_SCAN_SPEED "20.0000"
*MEAS_SCAN_SPEED_UNIT "deg/min"
*MEAS_SCAN_START "2.0000"
*MEAS_SCAN_START_TIME "07/14/2023 09:13:35"
*MEAS_SCAN_STEP "0.0200"
*MEAS_SCAN_STOP "70.0000"
*MEAS_SCAN_UNEQUALY_SPACED "False"
*MEAS_SCAN_UNIT_X "deg"
*MEAS_SCAN_UNIT_Y "cps"
#Intensity_unit=cps
#Attenuator_coefficient=1.0000

The results of analyses are automatically saved into a directory folder which are further uploaded on STUDIO SMART LAB II in conjunction with ICDD PDF-4 to perform an automatic/manual search-match.

3.3. Characterisation of Geomaterials

Based on the results of X-ray diffraction and X-ray fluorescence analysis of the various samples, the proportions of the major oxides and the mineralogy of all samples were determined. A Principal Component Analysis (PCA) was used for in-depth analysis to highlight the characteristics of the geomaterials from each quarry.

3.4. Proposal for a Typology of Quarries

A cross-analysis based on field observations of the type of deposit, the lithological nature of the geomaterials, the condition of the deposit and accessibility conditions has enabled us to propose a typology of the different quarries sampled.

4. Results

4.1. Lithology of the Quarries and Geochemistry of the Geomaterials

Kolleram 1 quarry

At Kolleram 1, t wo (02) sedimentary horizons are observed: a sandy loam surface horizon averaging 30 cm in thickness and a clayey-silty horizon over 320 cm thick (Figure 1). These horizons are relatively richer in silica (Mean SiO_2 = 68.61 percent) and alkaline and alkaline earth elements (Mean (Mg, Na and K) = 2.66 percent) and relatively poor in TiO_2 (Mean TiO_2 = 1.01 percent) (Tables 1-3).

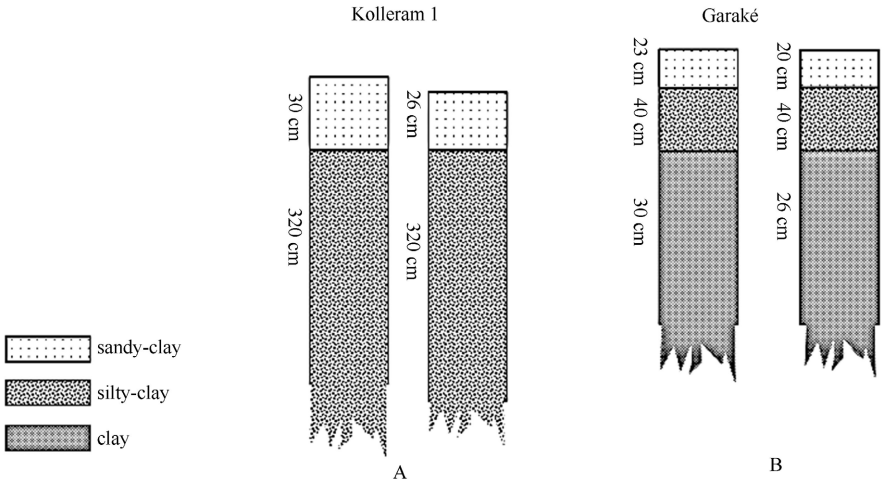


Figure 1. Lithological sections: A: Kolleram 1; B: Garaké.

Table 1. Silica (SiO_2) proportion in quarries.

	Level position	Kolloram 1	Garaké	Kolleram 2	Menari	Yaouri
Profil 1	Upper level	65.43	73.23	47.32	65.43	51.21
	Middle level	60.21	76.65	48.13	42.1	58.2
	Lower level	58.86	80.12	43.8	53.93	52.68
Profil 2	Upper level	74.6	77.86	46.25	42.59	40.48
	Middle level	75.98	81.3	51.17	44.46	32.92
	Lower level	76.56	83.32	54.42	44.12	35.74
Mean		**68.61**	**78.75**	**48.52**	**48.77**	**45.21**
Mean per group		**73.68**			**47.5**	

Table 2. Proportion of alkaline and alkaline earth elements (Mg, Na and K).

	Level position	Kolloram 1	Garaké	Kolleram 2	Menari	Yaouri
Profil 1	Upper level	0.61	1.99	0.96	1.23	1.9
	Middle level	1.55	2.23	0.55	1.2	1.34
	Lower level	4.25	1.77	1.21	1.08	1.4
Profil 2	Upper level	1.34	2.53	0.93	0.78	1.6
	Middle level	4.93	2.14	1.02	1.04	2
	Lower level	3.32	1.55	2.02	1.71	1.87
Mean		**2.67**	**2.04**	**1.12**	**1.17**	**1.69**
Mean per group		**2.35**			**1.32**	

Table 3. TiO_2 proportions.

	Level position	Kolloram 1	Garaké	Kolleram 2	Menari	Yaouri
Profil 1	Upper level	1.74	2.91	3.3	3.22	2.47
	Middle level	0.56	2.46	3.8	2.91	2.52
	Lower level	1.27	1.87	2.94	3.46	3.28

Continued

Profil 2	Upper level	0.8	1.66	2.45	3.72	3.01
	Middle level	0.86	1.84	2.88	3.07	3.17
	Lower level	0.88	1.51	2.67	4.26	3.25
Mean		**1.02**	**2.04**	**3.01**	**3.44**	**2.95**
Mean per group		**1.52**			**3.13**	

Garaké quarry

Here at Garaké, three (03) sedimentary horizons can be distinguished: a sandy-clay horizon 23 cm thick, a silty-clay horizon 40 cm thick and a clay horizon 30 cm thick (Figure 2). At the Garaké quarry too, the horizons are relatively richer in silica (Mean SiO_2 = 78.74 percent) and alkaline and alkaline earth elements (Mean (Mg, Na and K) = 2.035 percent) and relatively poor in TiO_2 (Mean TiO_2 = 2.04 percent) (Tables 1-3).

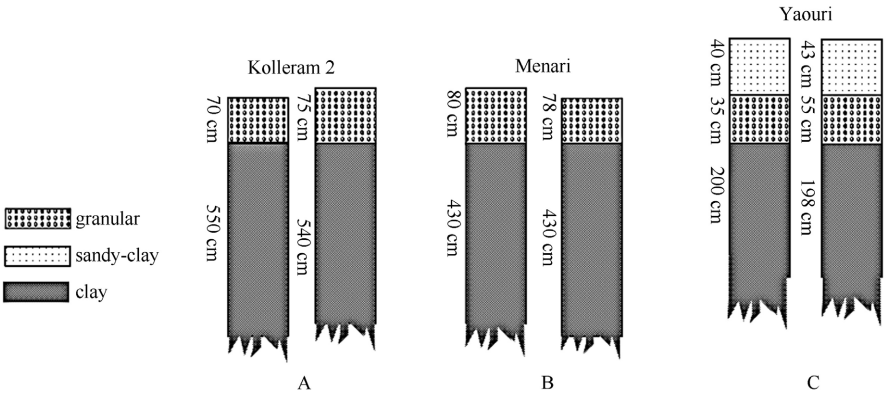


Figure 2. Lithological sections: A: Kolleram 2; B: Menari; C: Yaouri.

Kolleram 2 Quarry

At the Kolleram 2 quarry, two (02) horizons are observed: a superficial sedimentary horizon of granular material (micro-gravel and clay) 70 cm in depth, and a clayey alterite horizon over 550 cm in depth (Figure 2). At Kolleram 2, the horizons are comparatively low in silica (Mean SiO_2 = 48.51 percent) and alkaline and alkaline earth elements (Mean (Mg, Na and K) = 1.11 percent) and comparatively rich in TiO_2 (Mean TiO_2 = 3.00 percent).

Menari quarry

At Menari, two (02) horizons are observed: a superficial sedimentary horizon of granular material (micro-gravel and clay) 80 cm deep and an alterite horizon over 430 cm deep, which is clayey (Figure 3). Similarly, at the Menari site, the horizons are comparatively poorer in silica (Mean SiO_2 = 48.77 percent) and alkaline and alkaline earth elements (Mean (Mg, Na and K) = 1.71 percent) and comparatively richer in TiO_2 (Mean TiO_2 = 3.44 percent) (Tables 1-3).

Yaouri Quarry

At the Yaouri Quarry, three (03) horizons are observed: a 40 cm deep agilo-

sandy surface horizon, a 55 cm deep layer of granular material (micro-gravel and clay), and a clay horizon over 200 cm deep (Figure 3). At Yaouri too, the horizons are relatively poorer in silica (Mean SiO_2 = 45.20 percent) and alkaline and alkaline earth elements (Mean (Mg, Na and K) = 1.68 percent) and relatively richer in TiO_2 (Mean TiO_2 = 2.95 percent) (Tables 1-3).

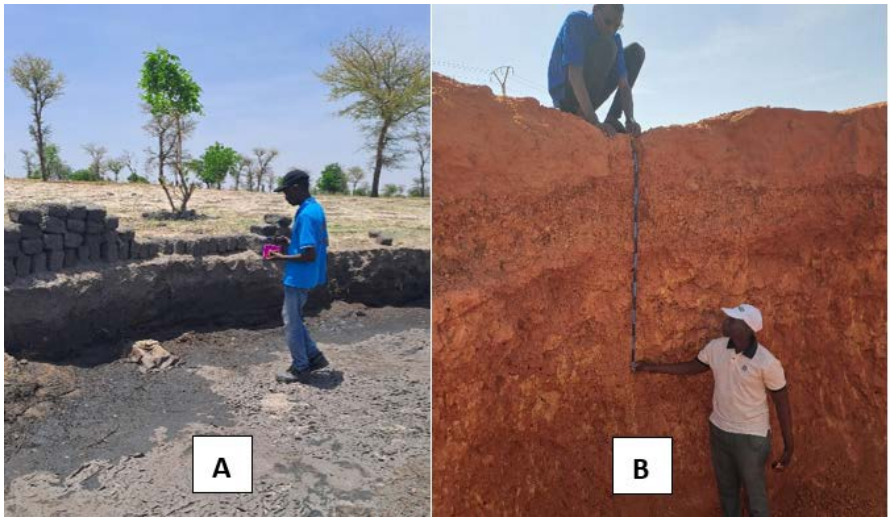


Figure 3. Photos of quarries: A: Type 1 (Garaké); B: Category 2 (Yaouri).

Thus, two types of quarry are identified: Type 1 quarries, where the material is dark in colour, not very compact and can be extracted simply by digging with a spade; these are characterised by their high content of SiO_2 and alkaline and alkaline earth elements (Na, Mg and K) and their low content of TiO_2; and Type 2 quarries, where the material is well-compacted, dense and red in colour (Figure 3). Extracting these geomaterials requires pickaxes, considerable physical strength and deeper digging. The latter are characterised by their relative low content of SiO_2 and alkaline and alkaline earth elements (Na, Mg and K) and their relatively high content of TiO_2 (Tables 1-3).

4.2. Clay Mineralogy

Kolleram 1 and Garaké quarries

The diffractograms of the various samples from the Kolleram 1 and Garaké quarries showed a crystalline clay phase of montmorillonite. The other minerals observed were quartz, albite and orthoclase (Figure 4 and Figure 5).

Kolleram 2, Menari and Yaouri quarries

The diffractograms of the various samples from the Kolleram, Menari and Yaouri quarries showed a crystalline clay phase of kaolinite. The other minerals observed were quartz and muscovite (Figures 6-8).

The Kolleram 1 and Garaké quarries are characterised by the presence of montmorillonite (Figure 9, Figure 10), indicating a confined environment with moderate alteration.

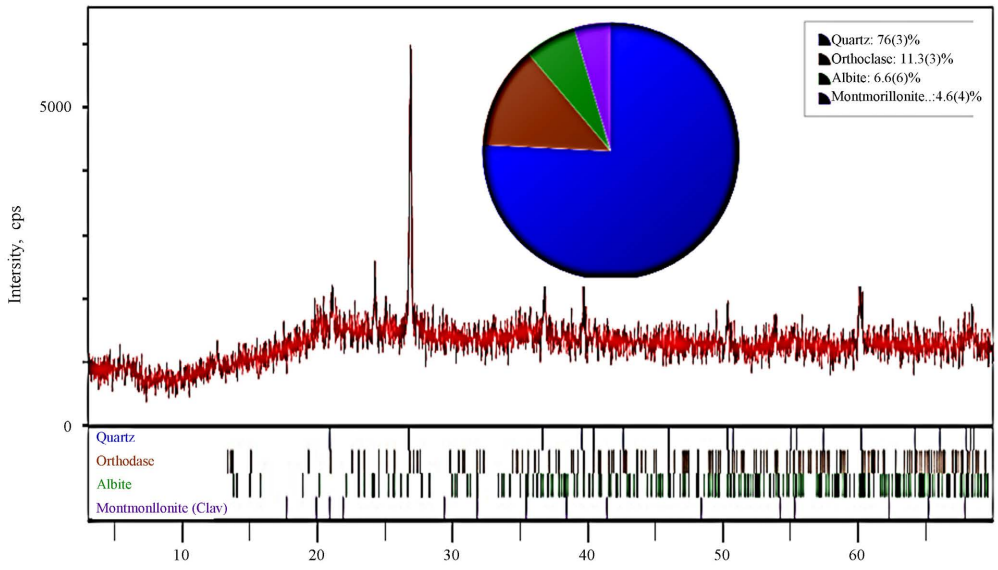


Figure 4. Diffractogram of Sample Kolleram 1 (Upper level).

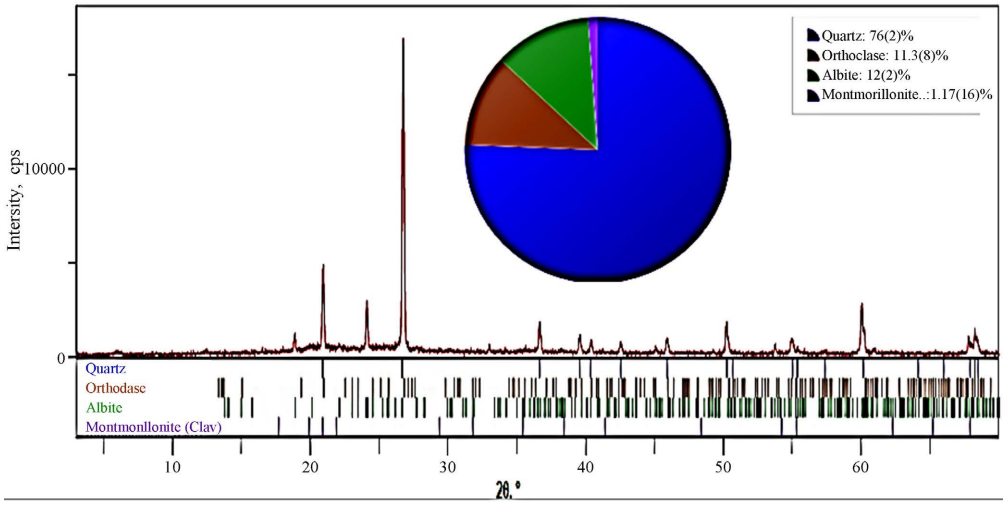


Figure 5. Diffractogram of the Garaké sample (upper level).

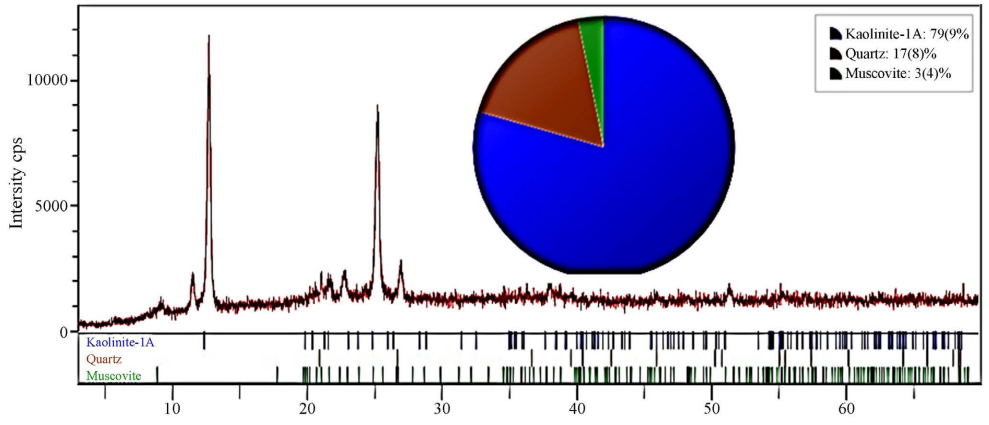


Figure 6. Diffractogram of Kolleram Sample 2 (Upper level).

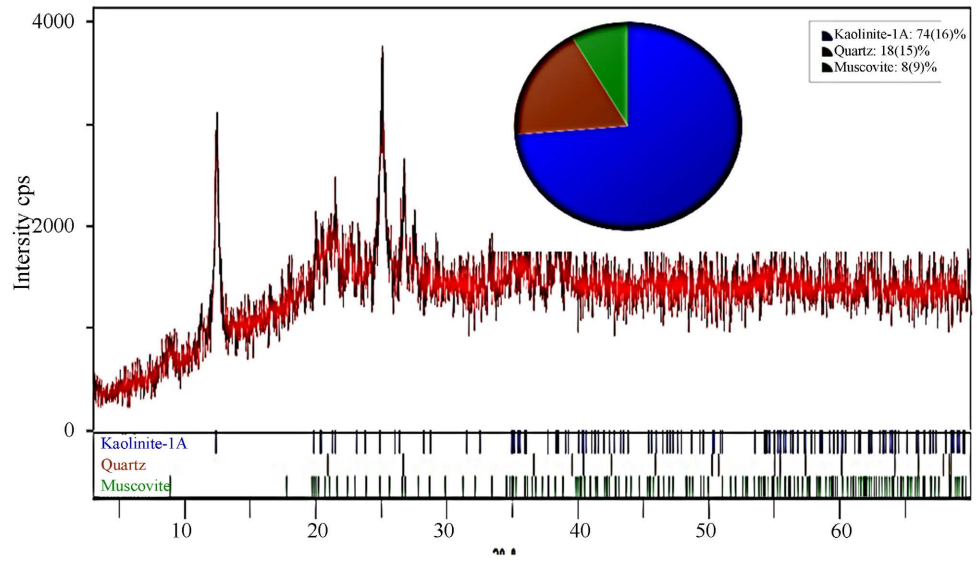


Figure 7. Diffractogram of the Menari sample (upper level).

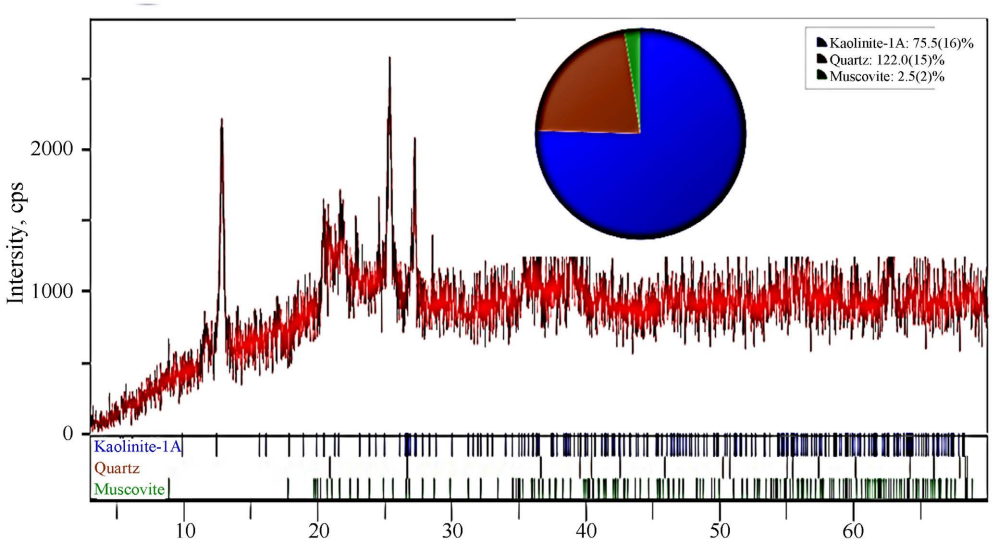


Figure 8. Diffractogram of the Yaouri sample (upper level).

The Kolleram 2, Menari and Yaouri quarries are characterised by the presence of kaolinite. This clay mineral indicates that the formation environment is sufficiently drained and weathered. This explains their low silica and alkaline and alkaline earth elements content. In the study area, this corresponds to the alteration profiles.

4.3. Typology of Quarries

Based on the composition of major oxides and the identified clay minerals, the sampled quarries can be grouped into two different types:

- Montmorillonite's quarries, rich in SiO_2 and alkaline and alkaline earth elements, whose material is dark in colour and not very hardened, found in ba-

sins. Given their lithology, consisting of superficial sedimentary horizons, these quarries are classified as superficial quarries (Figure 11).

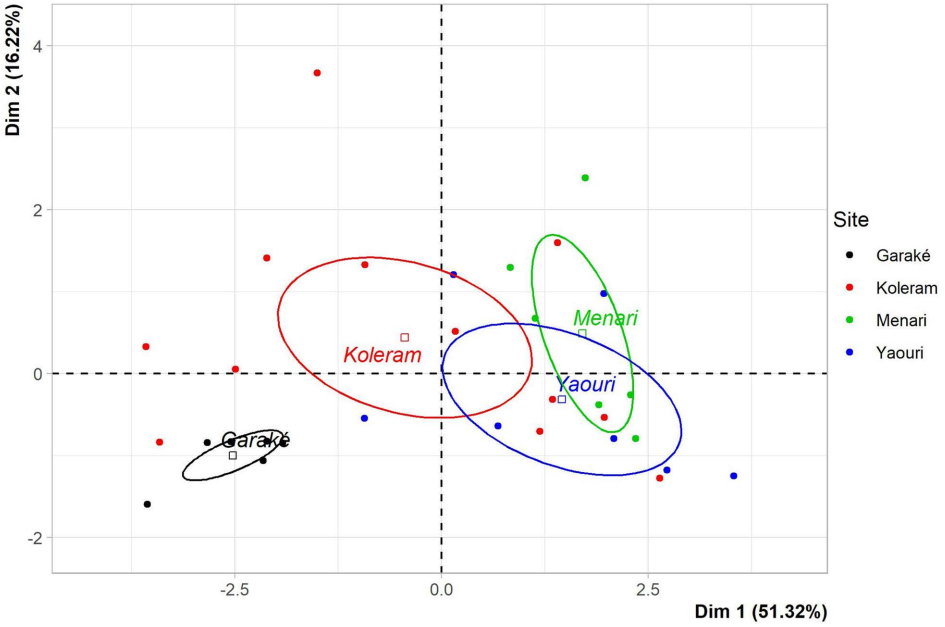


Figure 9. Graph of ACP individuals by site.

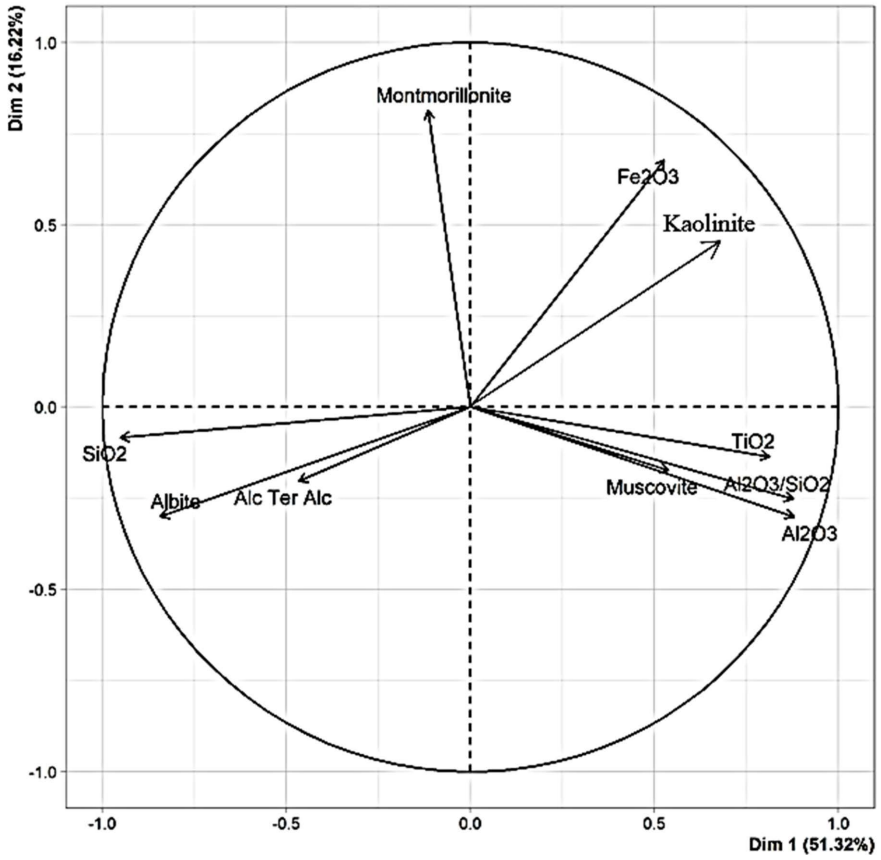


Figure 10. Graph of PCA variables (major oxides and mineralogy).

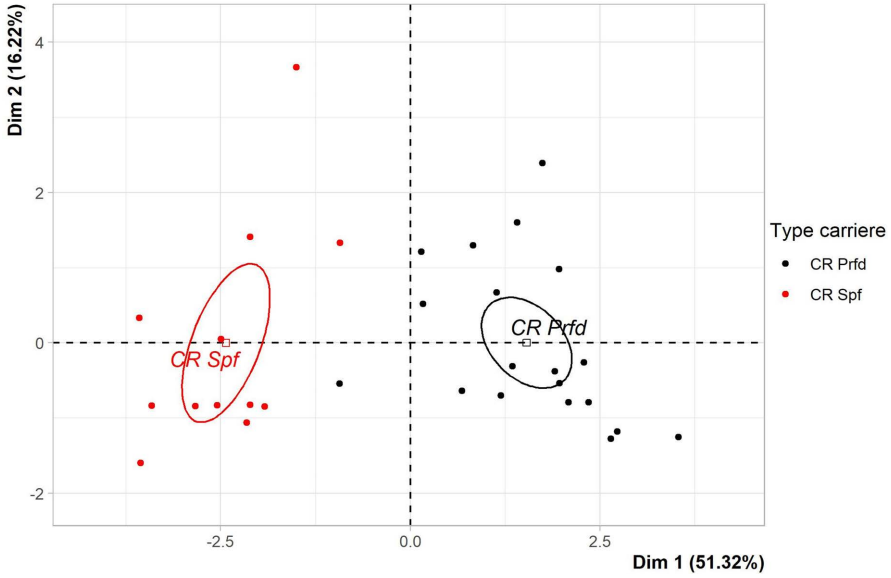


Figure 11. Graph of PCA Variables (quarry type).

- Kaolinite's quarries, which are relatively low in silica and alkaline and alkaline earth elements and contain TiO_2, are reddish in colour and consist of highly consolidated material; they are found in weathering profiles. These quarries, on the other hand, with a lithology consisting of deep alterite horizons overlain by a sedimentary layer, are classified as deep quarries (Figure 11).

5. Discussion

The lithological analysis of the 10 lithological sections from the five quarries selected for this study reveals two types of horizons: superficial sedimentary horizons and deeper alterite horizons. Superficial sedimentary horizons are characteristic of depositional environments. In the study area, these environments correspond to the valley itself, the floodplains and the basins [9]. The decreasing grain size observed at the Kolleram 1 and Garaké quarries demonstrates that this is a fluvial system characterised by a gradual loss of energy. This confirms that the environment is more of a valley than a basin or a shallow water area [10]. Thus, the superficial position and sedimentary nature of these horizons explain their low degree of consolidation. This low degree of consolidation is mainly due to the fact that these recent sediments have not yet undergone prolonged diagenesis, the geological process that transforms loose sediment into solid rock under the influence of pressure and temperature [11]. As for the deep alterite horizons, their degree of consolidation is explained by their position and the alteration process. In a complete alteration profile, hardening does not occur uniformly; instead, vertical zoning is observed from top to bottom, due to greater pressure and also temperature. The weathering process, in this case by hydrolysis, increases the cohesion of the particles through cementation. This cementation results from the transformation of the primary minerals of the protolith (feldspars and micas) into secondary clay minerals. The characteristics of these secondary minerals act as a

cementing agent for the disintegrated particles [12]. The geochemistry of the 30 samples collected shows that they are all composed predominantly of SiO_2 and Al_2O_3. This confirms that they are alumina silicates, like any clay material. However, the proportions of these two elements and the other major oxides varied from one sample to another and from one site to another. Indeed, these proportions of the various oxides are good indicators that enable the differentiation of these different geomaterials [1]-[13]. Thus, two groups emerge: one characterised by high levels of SiO_2 and alkaline and alkaline earth elements containing montmorillonite and being dark in colour; and another group relatively low in silica and alkaline and alkaline earth elements, containing TiO_2 and kaolinite, and being red in colour. For the first group, the high levels of these elements can be explained by the transport process. Indeed, at these quarries, **Figure 11** shows a strong correlation (angle 40˚, **Figure 11**) between these high proportions of alkaline and alkaline earth elements and the high proportions of quartz (SiO_2). This confirms that this is an environment where alkaline earth elements and silica accumulate. These clay deposits are classified as secondary or reworked deposits (Yven B., 2007). Remobilized clays are clay-based geomaterials whose natural structure has been destroyed or altered by transport or mechanical actions, such as mixing. Upon deposition, these clays lose a significant proportion of their cohesion and shear strength compared with intact clays. They are also referred to as loose clays with low consolidation pressures [14] [15]. However, in quarries that are relatively poor in alkaline and alkaline earth elements and silica, strong correlations emerge between Al_2O_3 and Fe_2O_3, as well as with TiO_2. The presence of TiO_2 indicates that no transport has taken place and that the material was therefore formed in situ, consistent with a weathering profile. The proportions of Fe_2O_3 account for the reddish colour of the material. It is well known that during the hydrolysis of rock formations, water leaches out silica (SiO_2) and alkaline earth elements. It breaks down the original mineral, and the aluminium (Al) combines to form clays. The leaching of silica is called desiallitisation; it is a gradual process and can lead to complete leaching, in which case the term allitisation is used [16]. The montmorillonite found in the silica-rich samples (**Figure 11**) is a key indicator of climatic conditions, the depositional environment and the origin of the sediments. Montmorillonite forms in an environment characterised by poor or slow drainage—or even confined drainage—and an alkaline environment resulting from moderate chemical alteration of the parent rocks. This explains their high content of alkaline and alkaline earth elements (Mg^{2+}, K^+, Na^+). In the case of our study area, the formation environment would correspond well to lowland pools [5]. The kaolinite found in the second category of geomaterials indicates that this is an environment that has undergone strong, if not total, hydrolysis. Kaolinite is a mineral that has lost a proportion of its silica and many other elements, a sign of very extensive leaching of the parent rock. The residual alumina hydrates and silica react to form kaolinitic clays. This reaction is known as siallitisation. When this formation occurs in situ due to meteoric and/or hydrothermal fluids, it is referred to as a primary deposit. This is the case for the geomaterials from the Kolleram 2, Menari

and Yaouri quarries in the Korama Valley. Kaolinite is thought to result from the alteration of feldspars in a sufficiently well-drained environment, whilst micas—ferromagnesian minerals—release a significant amount of iron in the form of ferric oxide. This is consistent with the high percentage of iron oxide in the materials from these quarries. This particularly high Fe_2O_3 content accounts for the reddish colour observed in these samples [17]. All these results have made it possible to determine a typology of the quarries sampled. The Kollerem 1 and Garaké quarries are classified as surface quarries. These are quarries containing montmorillonite-rich clay geomaterials, which are rich in silica and alkaline and alkaline earth elements, dark in colour and not very consolidated. The Kolleram 2, Menari and Yaouri quarries are classified as deep quarries. These consist of kaolinite-rich clay geomaterials, which are relatively low in silica and alkaline and alkaline earth elements, more consolidated and red in colour.

6. Conclusion

The results of this study indicate that the 30 samples from five (05) quarries—Kolleram 1, Garaké, Kolleram 2, Menari and Yaouri—are aluminosilicates, with varying proportions of major oxides, and are blackish in colour (Kolleram 1, Garaké) and red in colour (Kolleram 2, Menari and Yaouri). Two types of horizons are observed in the lithology of these quarries: shallow, slightly compacted sedimentary horizons and deeper, more compacted alterite horizons. The geochemistry of the major oxides and clay minerals in the various samples from the five quarries has enabled a typology of these quarries to be established. Two types of quarry emerge: the surface quarries—Kolleram 1 and Garaké—which contain montmorillonite-rich clay geomaterials, high in silica and alkaline and alkaline earth elements, dark in colour and relatively unconsolidated; and the deep quarries—Kolleram 2, Menari and Yaouri, which are quarries containing kaolinite-based clay geomaterials, relatively low in silica and alkaline and alkaline earth elements, more compacted and reddish in colour. These results make it possible to identify suitable clay geomaterials for various forms of utilisation.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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