

Stabilization of a Lateric Clay from Burkina Faso with Cement-Metakaolin for an Application in Road Construction

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Abstract

The objective of this work is to obtain a composite of clay-cement-metakaolin having good mechanical properties and geotechnical. To do this, a lateritic clay from Burkina Faso referenced ALK was characterized by various methods (X-ray diffraction, infrared spectrometry, thermal analysis and Inductively Coupled Plasma, Atomic Emission Spectrometry) in order to be used as a base course after adding cement and metakaolin. The results of the mineralogical characterization of this clay showed that it is composed of kaolinite (65.7 wt.%), quartz (19.3 wt.%) and goethite (10.8 wt.%). The geotechnical tests carried out showed that ALK is moderately plastic with a plasticity index Ip = 22%. The optimum moisture content and the maximum dry density are respectively 15.9% and 1.76 g·cm⁻³. Simple compressive strength and splitting tensile strength are Rc = 1.59 MPa and ft = 0.149 MPa respectively. The California Bearing Ratio (CBR) index at 95% is 40% and above the minimum value of 30% shows that ALK can be used as a sub-base course in road construction. The addition of cement and metakaolin in various proportions improved the CBR index and the mechanical strength of the composites produced. This improvement is due to the formation of hydrated calcium silicate (CSH) resulting from the pozzolanic reaction between the portlandite of the cement and the amorphous silica of the metakaolin. Thus the 2 wt.% metakaolin and 6 wt.% cement formulation with a 95%CBR index of 81% is suitable for the development of a base course in road construction.

Keywords

Lateritic Clay, Cement, Metakaolin, Road Construction, The California

Bearing Ratio Index

1. Introduction

The landlocked countries of Africa, Burkina Faso in particular, concerned with integral and sustainable development must think about improving their road network. However, the construction and ongoing maintenance of these roads is very expensive. Despite the various efforts made by the states to improve the roads, their quality still leaves something to be desired. Indeed the road network in Burkina Faso is subject to deformations such as those called corrugated sheets, the loss of materials, and the depression of the roads. This poor quality of the roads is probably due to the nature of the raw materials used and the insufficiency of geotechnical tests for the choice of materials for road construction. The geotechnical characterization of the raw materials used in road construction is essential in order to define an appropriate treatment. Thus, several research works have been carried out on the stabilization of roads by the use of mineral binders such as cement and lime [1] [2] [3] [4]. These binders, although essential, are expensive and their method of manufacture is energy-intensive and produces large quantities of greenhouse gases such as carbon dioxide. Indeed, the production of one kilogram of cement generates on average the same amount of CO₂ [5] [6]. In addition, the use of these hydraulic mineral binders leaves solid and liquid residues that pollute the environment. However, the use of metakaolin in view of its proven pozzolanic properties as a partial substitute for cement in construction could constitute an alternative to reducing the content of cement and improving the quality of roads [7] [8]. In this present work, the mineralogical composition and the geotechnical characteristics of a lateritic clay from Burkina Faso will be determined. Then clay-cement-metakaolin composites will be developed in order to evaluate their geotechnical, mechanical and mineralogical properties.

2. Materials and Experimental Methods

2.1. Materials

2.1.1. Lateritic Clay Raw

The lateritic raw material subject of this study was taken from the village of Kamboinse located 15 km from the capital Ouagadougou (12°27'44" north and 1°33'17" west). This site was formerly exploited by local companies for the construction of pavement layers in road construction. This clay is also used by the resident populations for making raw bricks (adobes) in the construction of habitats.

2.1.2. The Cement

The cement used in this study for the various tests comes from Diamond Ce-

ment, a cement factory in Burkina Faso. It is a CEM I 45 cement whose chemical, physical and mineralogical properties are recorded in **Table 1** [9].

2.1.3. The Metakaolin

The metakaolin used in this work was obtained by calcination at 730°C for two hours with a rise rate of 10°C /min of a local clay from Burkina Faso taken from the Loulouka clay site (12°31' North and 1°47' West). The raw material as well as the metakaolin has already been the subject of previous scientific work [10]. In addition, the chemical and mineralogical composition of Loulouka clay and the pozzolanic indices of metakaolin are recorded respectively in **Table 2** and **Table 3**. The pozzolanic indices obtained for a substitution of 20, 25 and 30 wt.% of the cement by metakaolin are greater than the minimum value of 75% set by standard ASTM C 618 shows the pozzolanic character of metakaolin [11]. Thus, the metakaolin produced can be used as a pozzolan in the replacement of cement.

2.2. Formulation of the Specimens

The specimens were developed according to the type of test: modified Proctor, CBR, simple compression or tension by splitting. Thus, after sampling, heaps of 6 kg of lateritic clay are mixed with quantities of water with different mass percentages. Homogenize before compacting. For the CBR test, a clay-water mixture is made taking into account the optimum moisture content and the maximum dry density given by the modified Proctor test. The prepared specimen is kept in the mould, immersed directly in a water tank for four days or cured in the open air for three days before immersion. For the specimens to be subjected to simple compression and to tension by splitting, the confection is of the modified Proctor type in a split Proctor mould. A compaction of 56 strokes on 5 layers is made, immediately unmolded and then dried for at least 28 days. The mixture to be compacted is identical to that of the CBR type. For composites (laterite-cement-metakaolin), the manufacturing techniques remain the same. Only the preparation of the different mixtures changes. For the modified Proctor tests, the mixtures are made taking into account the percentages of cement and metakaolin given in Table 4 [12] [13].

2.3. Experimental Methods

2.3.1. Techniques for the Chemical and Mineralogical Characterization of Raw Clay and Specimens

In order to determine the chemical and mineralogical composition of the lateritic raw material and the specimens several experimental techniques were used.

Chemical analysis of the lateritic clay raw was carried out by inductively coupled plasma atomic emission spectrometry (ICP-AES). A sample of raw material was crushed to particles size less than 80 μ m and melted with lithium tetraborate (Li₂B₄O₇) to form a glass bead. The glass bead was dissolved in a nitric acid solution, and the solution obtained was analysed by an ICP-AES device.

X-ray diffraction patterns of the lateritic clay raw and specimens sample powders were obtained with a Siemens D5000 type diffractometer, equipped

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| Chemical | SiO ₂ | Al_2O_3 | Fe ₂ O ₃ | MgO | CaO | P_2O_5 | SO ₃ | Na ₂ O | K ₂ O | FL | IR | LOI |
|-----------------------|------------------|--------------------------------------|--------------------------------|------|----------------------|----------|-----------------|-------------------|------------------|-----|---------|------|
| composition (wt.%) | 20.12 | 5.73 | 4.06 | 1.18 | 64.8 | 0.39 | 2.68 | 0.08 | 0.17 | 0.8 | 0.28 | 0.27 |
| Mineralogy | | C ₃ S | | | C_2S | | | C ₃ A | | | C_4AF | |
| (wt.%) | | 55.7 | | | 15.68 | | | 8.31 | | | 12.34 | |
| Physical | Sp | ecific dens (g·cm ⁻³) | ity | | arent De (g∙cm⁻³) | - | | | Setting (h) | | | |
| properties | | 3.02 | | | 1.06 | | | | 3 | | | |

Table 1. Chemical and mineralogical composition and some physicals properties of cement use.

FL: Free lime; IR: Insoluble residues; LOI: Loss on ignition (1000°C).

Table 2. Chemical and mineralogical compositions (in wt.%) of the Loulouka clay [9].

| | | Chemi | cal compo | sition | | | | | |
|------------------|--------------------------------|--------------------------------|--|---|---|--|---|--|--|
| SiO ₂ | Al ₂ O ₃ | Fe ₂ O ₃ | MnO | MgO | Na ₂ O | K ₂ O | TiO ₂ | LOI | Tota |
| 71.42 | 19.5 | 0.4 | 0.0 | 0.23 | 0.36 | 1.68 | 0.82 | 5.30 | 99.7 |
| | | Mineralo | gical comp | position | | | | | |
| Kaolinite | | Quartz | | | Illite | | Total | | |
| 34 49 | | | 15 98 | | | | | | |
| - | 71.42 Kaol | 71.42 19.5 Kaolinite | SiO2 Al2O3 Fe2O3 71.42 19.5 0.4 Minerale Kaolinite | SiO ₂ Al ₂ O ₃ Fe ₂ O ₃ MnO 71.42 19.5 0.4 0.0 Mineralogical comp Kaolinite Quartz | 71.42 19.5 0.4 0.0 0.23 Mineralogical composition Kaolinite | SiO ₂ Al ₂ O ₃ Fe ₂ O ₃ MnO MgO Na ₂ O 71.42 19.5 0.4 0.0 0.23 0.36 Mineralogical composition Kaolinite Quartz 111 | SiO ₂ Al ₂ O ₃ Fe ₂ O ₃ MnO MgO Na ₂ O K ₂ O 71.42 19.5 0.4 0.0 0.23 0.36 1.68 Mineralogical composition Kaolinite Quartz Illite | SiO ₂ Al ₂ O ₃ Fe ₂ O ₃ MnO MgO Na ₂ O K ₂ O TiO ₂ 71.42 19.5 0.4 0.0 0.23 0.36 1.68 0.82 Mineralogical composition Kaolinite Illite | SiO ₂ Al ₂ O ₃ Fe ₂ O ₃ MnO MgO Na ₂ O K ₂ O TiO ₂ LOI 71.42 19.5 0.4 0.0 0.23 0.36 1.68 0.82 5.30 Mineralogical composition Kaolinite Quartz Illite Total |

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Table 4. Composition of the different formulated test specimens.

Activity index (%)

| Codes | Specimens description | Lateritic clay (g) | Cement (g) | Metakaolin (g) |
|-----------------|---------------------------------|--------------------|------------|----------------|
| M ₀₀ | 0wt.%métakaolin + 0wt.% cement | 6000 | 0 | 0 |
| M ₆₂ | 6wt.%métakaolin + 2 wt.% cement | 5520 | 120 | 360 |
| M ₄₄ | 4wt.%métakaolin + 4wt.% cement | 5520 | 240 | 240 |
| M ₂₆ | 2wt.%métakaolin + 6 wt.% cement | 5520 | 360 | 120 |

with a monochromator using cobalt K*a* radiation (Co K*a*, λ = 1.789 Å) and graphite back monochromator.

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The range of analysis was between 5° and 75°, with a step size of 0.04°. The acquisition time of the analyses was 2 s. The analyses were all performed on powders previously crushed to particles size less than 80 μ m and placed in a rotating sample holder.

Infra-red spectrometry on crushed the lateritic clay raw and specimens sample (particles size less than 80 μ m) was carried out on an attenuated total reflectance-Fourier transform infra-red spectrometer (ATR-FTIR Frontier, 4000 - 500 cm⁻¹, diamond crystal-Perkin Elmer). The device used for the analysis was a Perkin Elmer L125000 P Frontier. Analysis by differential scanning calorimetry

(DSC) consisted of monitoring the difference in the heat flow between the two containers-one contained the analyzed sample, and the second contained the thermally inert sample (calcined alumina). The heating rate during the analyses was 10°C/min. Thermogravimetric analysis (TGA) consists of record the variation in mass during a thermal cycle.

These changes are related to chemical reactions or the departure of volatile constituents adsorbed or combined in a material. The two techniques are often applied simultaneously on the same apparatus and sample. The DSC-ATG thermograms were recorded with a Netzsch SATA 449F3 Jupiter apparatus, under an argon atmosphere between 30°C and 1100°C. A quantity between 10 and 30 mg of the sample, with a maximum particle size not exceeding 80 μ m, was tested.

2.3.2. Geotechnical Characteristics

The determined geotechnical parameters essentially consist of sieving particle size analysis, Atterberg limits, methylene blue test as well as Proctor and CBR tests.

The particle size distribution has been achieved according to NF P18-560 [14] (particle size distribution by dry sieving) standards. The liquid limit was measured by the method of the dish of Casagrande (W_L) and the plastic limit by the method of the roller (W_P). The tests were realized according to NF P94-051 [15] standard. The blue methylene value (MVB) was determined on the total sample according to NF P94-068 [16].

The use of a material in road construction requires the performance of the Proctor test and the CBR (California Bearing Ratio) test.

The objective of the Proctor test is to determine the optimum moisture content (OMC) in percentage and the maximum dry density (MDD) for a standardized compaction of a given intensity. It consists of identically compacting samples of the same soil with different water contents. It can be seen that the dry density varies and goes through a maximum for a determined water content called the modified Proctor optimum [12]. With regard to pavement base materials and draining materials, the modified Proctor test is carried out according to the classification standard NF P 11-300 [17], and then supplemented by standard NF P 98-231-1 [13]. There are two types of Proctor tests. Depending on the intensity of compaction used, the test will be called normal Proctor test or modified Proctor test. The modified Proctor test was carried out because it is the one recommended for road construction.

For the various elements of the body of a pavement, performances are required which are based on a standardized type test, the CBR test. This test is an essential parameter for the mechanical design of pavements. The CBR index is a quantity used to characterize a soil, as a support or constituent of a pavement structure. The general principle of the test used to determine the CBR lift index consists of measuring the forces to be applied to a cylindrical punch with a cross-section of 19.35 cm² to make it penetrate at a speed of 1.27 mm/min into a soil specimen. The particular values of the two forces having caused the depressions of 2.5 and 5 mm are then related, to the values of 13.35 and 20 kN, which are respectively the forces observed on a reference material for the same depressions. The CBR indices are then determined from relations 1 and 2:

$$\operatorname{CBR}_{1}(\%) = 100 \frac{F_{1}}{13.35}$$
 (relation 1)

$$\operatorname{CBR}_{2}(\%) = 100 \frac{F_{2}}{20} \qquad (\text{relation } 2)$$

where F_1 and F_2 correspond to the forces measured during the test, they are expressed in kN. The CBR index is conventionally defined by the highest value of the ratios thus calculated. The test was carried out according to standard NF P 94-093 [12].

2.3.3. Mechanical Characteristics

The mechanical tests carried out on the specimens relate to the simple compressive strength and the Tensile strength by splitting (Figure 1(a) and Figure 1(b)).

For simple compressive strength, the specimen is subjected to a monotonically increasing load until failure (**Figure 1(a)**). The compressive strength is the ratio of the breaking load to the cross section of the specimen. The value of the compressive strength is given by relation 3:

$$R_c = 10\frac{F}{S}$$
 (relation 3)

 R_c : compressive strength of the specimen in MPa; F: maximum load supported by the specimen in kN; S: average value of the section in cm². The test was carried out according to standard NF P18-406 [18].

The Tensile strength by splitting is generally evaluated on cylindrical specimens (**Figure 1(b)**). In this test, a compressive force is applied to the specimen along two opposite generatrices. This compressive force induces tensile stresses in the plane passing through these two generatrices. Failure, due to these tensile stresses, occurs in this plane. The specimen is placed between the two platens of the press, the contact between the plates and the specimen being made through the two strips of plywood. The test was carried out according to standard NF P 18-408 [19]. The loading rate was 0.05 MPa/second. The breaking stress is given by relation 4:

$$f_t = 20\frac{F}{\pi ah} = 6.37\frac{F}{ah}$$
(relation 4)

with: f_t breaking stress in (MPa); F load applied in (kN); a diameter of the specimen in (cm); h height of the specimen in (cm).

3. Results and Discussion

3.1. Chemical and Mineralogical Characterization of Lateritic Clay

The chemical composition of ALK lateritic clay is given in **Table 5**. Analysis of the table shows that the sample contains a large amount of silica (49.88 wt.%)

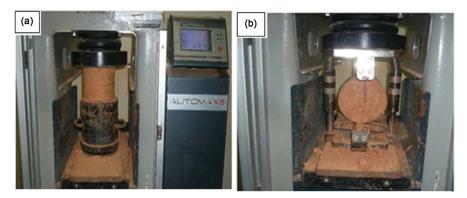


Figure 1. Performing mechanical tests. (a) The simple compressive strength; (b) Tensile strength by splitting.

 Table 5. Chemical composition (in wt.%) of lateritic clay.

| Oxides | SiO ₂ | Al_2O_3 | Fe ₂ O ₃ | MnO | MgO | CaO | Na ₂ O | K ₂ O | TiO_2 | P_2O_5 | LOI | Total |
|--------|------------------|-----------|--------------------------------|------|------|------|-------------------|------------------|------------------|----------|-------|-------|
| (wt.%) | 49.88 | 25.95 | 9.69 | 0.05 | 0.17 | 0.05 | 0.03 | 0.39 | 1.12 | 0.04 | 11.78 | 99.15 |

and alumina (25.95 wt.%) as well as an appreciable amount of iron oxide (9.69 wt.%). These results suggest that quartz, aluminosilicates and iron minerals are predominant in the sample studied.

Analysis of the powder diffractogram of the lateritic clay shown in **Figure 2** shows that it is essentially composed of kaolinite $(Si_2O_5Al_2(OH)_4)$, quartz (SiO_2) and goethite (FeO(OH)). The presence of these minerals is also confirmed by the infrared spectrum in **Figure 3**. The spectrum shows bands of quartz (Si-O-Si vibration at 786 cm⁻¹); of kaolinite: 3620and 3690 cm⁻¹ (O-H stretching vibrations); 1113, 1000 cm⁻¹ (Si-O vibrations); 913 cm⁻¹ (Al-OH bending vibration) [10] [20] [21].

The DSC/TG thermogram in **Figure 4** shows three endothermic peaks and one exothermic peak. The first endothermic peak at 140°C corresponds to the departure of hydration or hygroscopic water. This thermal phenomenon is associated with a mass loss of 0.63 wt.%. The second endothermic peak at 310°C corresponds to the dehydroxylation of goethite and its transformation into hematite [22], with a mass loss of 1.93 wt.%. This dehydroxylation of the iron oxyhyoxides present in the sample takes place following reactions 1 and 2:

$$2\gamma \text{-FeOOH} \xrightarrow{255^{\circ}\text{C}} \gamma \text{Fe}_2\text{O}_3 + \text{H}_2\text{O}$$
(1)

$$2\alpha - \text{FeOOH} \xrightarrow{279^{\circ}\text{C}} \alpha \text{Fe}_2\text{O}_3 + \text{H}_2\text{O}$$
(2)

As for the last endothermic peak around 515°C, it corresponds to the dehydroxylation of kaolinite and its transformation into metakaolinite followed by a mass loss of 9.23 wt.% [23].

The overall dihydroxylation reaction can be schematized according to reaction 3:

$$Si_2O_5Al_2(OH)_4(kaolinite) \rightarrow 2SiO_2-Al_2O_3(métakaolinite) + 2H_2O$$
 (3)

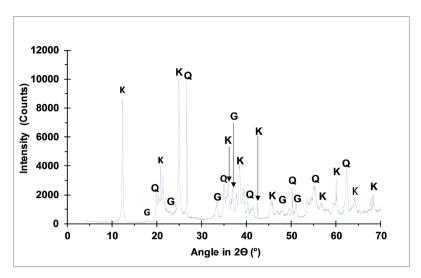


Figure 2. X-ray diffraction pattern of ALK sample. G: Goethite; K: Kaolinite; Q: Quartz.

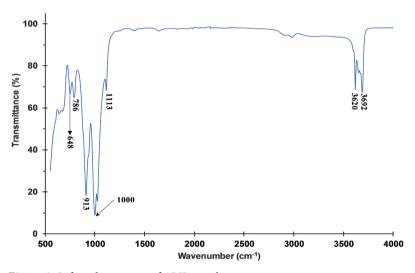
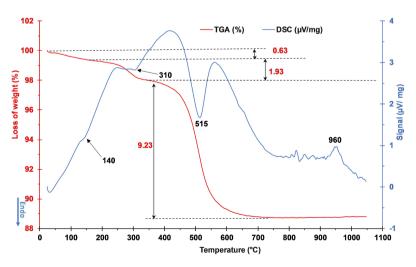


Figure 3. Infrared spectrum of ALK sample.





The dehydroxylation temperature of an ordered kaolinite is around 600°C while that of a disordered kaolinite is lower. Thus, the ALK sample would therefore be composed of disordered kaolinite. The only exothermic peak at 960°C corresponds to the recrystallization or the structural reorganization of the meta-kaolinite [9]. This is the reorganization of mekaolinite into more stable structures (spinel, mullite) in addition to amorphous silica following reaction 4:

 $2(2SiO_2-Al_2O_3)(\text{métakaolinite}) \rightarrow Si_3Al_4O_{12}(Al-Si-spinelle) + SiO_2(\text{amorphe})$ (4)

The results of the elementary chemical analysis and the mineralogy given by X-ray diffraction from relation 5 of Yvon *et al.* [24] made it possible to draw up the mineralogy composition of the ALK sample.

$$T(a) = \sum M_i P_i(a) \qquad (relation 5)$$

with: T(a): content (oxide%) of the chemical element "*a*"; M_i ; content (%) of mineral "*i*" in the material studied and containing the element "*i*"; $P_i(a)$: proportion of the element "*a*" in the mineral "*i*".

Table 6 gives the semi-quantitative composition of the mineral phases of the sample studied. It consists of a high proportion of kaolinite (42 wt.%). The quantities of quartz (19.3 wt.%) and goethite (10.8 wt.%) are significant. The balance (4.2 wt.%) consists mainly of organic matter and amorphous mineral phases.

3.2. Geotechnical and Mechanical Characterization of the Specimen of the Pure Sample ALK

The geotechnical properties of lateritic clay are recorded in **Table 7**. The Atterberg limits indicate that the raw material is moderately plastic clay. This is probably due to the presence of clay minerals such as kaolinite which makes it plastic [25]. The methylene blue value of 1.57 g/100 g attests that the sample belongs to the category of sandy clay soils with low plasticity. This result corroborates well with those of the Atterberg limits. However, the values of the plasticity index and the methylene blue value remain low compared to those reported by Millogo *et al.* [26] in 2008 on lateritic gravel from Sapouy (Ip = 10.5% and VBS = 0.17 g/100 g). This is justified by the clayey nature of the sample studied compared to that of Sapouy which is very gravelly and therefore poor in clay minerals.

As for the particle size composition, it is deduced from the particle size curve of **Figure 5** determined by sieving, it indicates that ALK would be made up of 70% fines fraction. This confirms the clayey nature of the sample.

The results of the modified Proctor test presented graphically in **Figure 6** showed that the optimum moisture content and maximum dry density are 15.9 wt.% and 1.76, respectively. The optimal high moisture content of the ALK sample and its low density compared to those obtained by Millogo *et al.* on the lateritic gravel of Sapouy can be explained by the clayey nature of the ALK sample. These values are in agreement with the values of the Atterberg limits and that of methylene blue.

| Table 6. Mineralogical compositions (i | in wt.%) of the lateritic clay. |
|--|---------------------------------|
|--|---------------------------------|

| Minerals | Kaolinite | Quartz | Goethite | Balance | |
|--------------------|-----------|--------|----------|---------|--|
| Composition (wt.%) | 65.7 | 19.3 | 10.8 | 4.2 | |

Table 7. Some geotechnical properties of lateritic clay.

| Geotechnical properties | Results | | | | |
|---|----------------|--|--|--|--|
| Atterberg limits | | | | | |
| Liquid limit W _L (%) | 44 | | | | |
| Plasticity limit W_p (%) | 22 | | | | |
| Plasticity index PI (%) | 22 | | | | |
| The methylene blue value | (g/100 g) 1.57 | | | | |
| Particle size distribution > 80 μ m (%) | | | | | |
| Gravel | 10 | | | | |
| Coarse sand | 15 | | | | |
| Fine sand | 5 | | | | |
| Modified Proctor | | | | | |
| Optimum moisture content (OMC) (%) | 15.9 | | | | |
| Maximum dry density (MDD) (g·cm ⁻³) | 1.76 | | | | |
| CBR at 95% (%) | 40 | | | | |

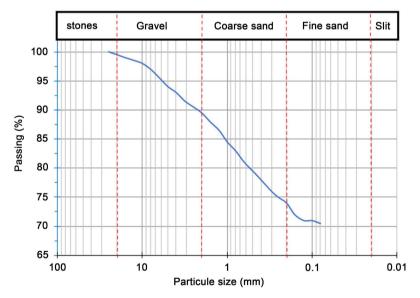


Figure 5. Particle size curve by sieving the ALK sample.

The value of the CBR lift index at 95% after four days of immersion is 40% for the natural sample. This value between 30% and 80% shows that the sample can be used as a sub-base course raw material in road construction [27]. Its use as a base course raw material in road construction requires a chemical improvement (by mineral binders) or mechanical improvement (by adding crushed stones).

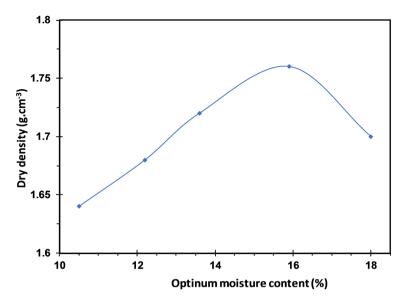


Figure 6. The results of the modified Proctor test of the ALK sample.

After the geotechnical parameters, the various specimens were subjected to mechanical tests by simple compression and tension by splitting. Simple compressive strength and splitting tensile strength are Rc = 1.59 MPa and ft = 0.149 MPa respectively. This compressive strength value is greater than 1.25 MPa and that of the tensile strength by splitting is greater than 0.125 MPa, values obtained by Messou on the stabilization of laterites in Ivory Coast [28]. These results are justified by the fact that the laterites studied by Messou are richer in clay minerals (therefore more plastic) than the laterite clay ALK studied. In order to improve the geotechnical and mechanical properties of the raw material and at the same time reduce cement consumption, cement-metakaolin-clay composites have been produced.

3.3. Study of the Mineralogy of Clay-Cement-Metakaolin Composites

The pieces of the specimens prepared (lateritic clay-cement-metakaolin) according to the modified Proctor test process then subjected to the simple compression and tensile tests by splitting after 28 days of curing in the open air were finely ground and analyzed by X-ray diffraction, infrared spectrometry and differential scanning calorimetry.

The powder diffractograms of these composites are shown in **Figure 7**. Analysis of these diffractograms shows the presence of laterite raw material minerals such as kaolinite, quartz and goethite. Also we note the presence of new phases such as calcite and portlandite.

The low presence of portlandite is probably due to its involvement in the pozzolanic reaction with amorphous silica of metakaolin. To complete the mineralogy of the composites and particularly the demonstration of any amorphous phases formed, samples of ground clay-cement-metakaolin mixtures were analyzed by infrared spectrometry and then by differential scanning calorimetry

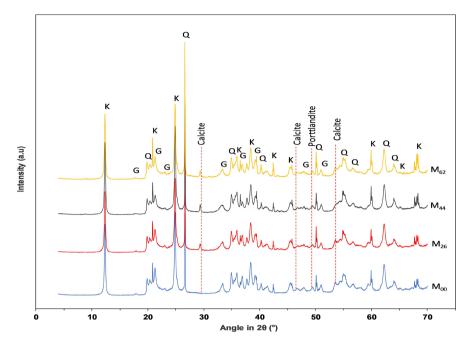


Figure 7. X-ray diffractogram of composites.

(DSC). The analysis of Figure 8 shows that in the high frequencies, we have the characteristic bands of the vibration of the OH bond of kaolinite between 3692 and 3620 cm^{-1} [9] [29]. It can be seen that the intensity of these bands decreases with the addition of cement. This shows the reaction between the cement and some minerals of the lateritic raw material. Indeed, the kaolinite contained in the clay matrix is weakly crystallized, thus the cement can react with the non-crystalline fraction then causing a reduction in the quantity of kaolinite in the material. Also, the band at 913 cm⁻¹ corresponds to the vibration of the Al-OH bond while those at 1000 and 1113 cm⁻¹ would be due to the vibration of the Si-O bond of the kaolinite. At 748 cm⁻¹ we have the vibration of the Si-O-Al bond also of the kaolinite. The band at 786 cm⁻¹ is attributable to quartz. The spectra also show new vibration bands around 1450 and 3400 cm⁻¹. The discreet band around 1450 cm^{-1} is due to the combined effect of calcite [9]. On the other hand, that around 3400 cm⁻¹ is attributable to the vibrations of the CSH hydroxyls [30] [31]. On the DSC thermograms of the composites presented in Figure 9, we can identify thermal accidents attributable both to the minerals of the clay sample and those due to the formation of calcite and CSH. Composites containing cement-metakaolin exhibit a large endothermic phenomenon around 100°C. This is the phenomenon of dehydration corresponding to the loss of hygroscopic water and/or the dehydration of hydrated calcium silicates (CSH) [32]. The thermal accidents around 310°C, 515°C and 960°C correspond respectively to the loss of water from crystallization of goethite, to the dehydroxylation of kaolinite and to the recrystallization of metakaolinite from the lateritic raw material. The area of the kaolinite dehydroxylation peak decreases with the addition of cement. This would be due to the involvement of kaolinite in the pozzolanic

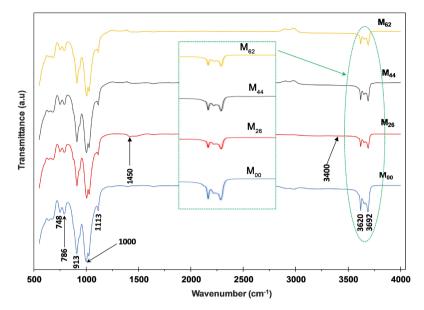


Figure 8. Infrared spectra of composites.

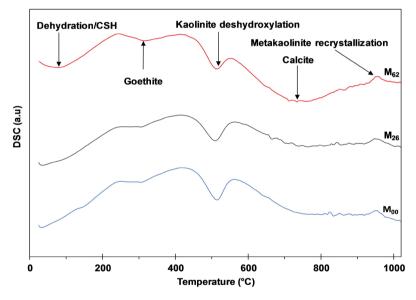


Figure 9. Thermograms DSC-TGA of composites.

reaction [33]. The broad peak around 700°C corresponds to the decomposition of calcite into lime (CaO) and carbon dioxide [9].

3.4. Mechanism of Formation of New Phases

The newly formed phases are essentially calcite $(CaCO_3)$, portlandite (CH) and hydrated calcium silicate (CSH). Portlandite is formed as a result of the hydration of alite (C₃S) and belite (C₂S) according to the following reactions 5 and 6 [9] [34]:

$$2C_3S + 6H \rightarrow C_3S_2H_3(CSH) + 3CH$$
(5)

$$2C_2S + 4H \rightarrow C_3S_2H_3(CSH) + CH$$
 (6)

with: CaO = C; $SiO_2 = S$ et $H_2O = H$.

Calcite is formed as a result of a carbonation reaction involving portlandite produced by the hydration of cement and carbon dioxide from the atmosphere according to reaction 7 [29]:

$$Ca(OH)_2 + CO_2 \rightarrow CaCO_3 + H_2O$$
 (7)

Hydrated calcium silicate (CSH) is formed according to two synthetic pathways, including the direct hydration of alite (reaction 5) and belite (reaction 6) of cement and according to the pozzolanic reaction involving the silicate of metakaolinite and hydrated lime (portlandite).

The mechanism of CSH synthesis by the pozzolanic reaction is as follows [9]

• dissociation of portlandite according to reaction 8:

$$Ca(OH)_2 \rightleftharpoons Ca^{2+} + 2OH^-$$
 (8)

• dissolution in basic medium of the silica of the metakaolinite according to the chemical reaction 9:

$$SiO_2(silica of the metakaolinite) + 2OH^- \rightarrow H_2SiO_4^{2-}$$
 (9)

 reaction in aqueous medium of Ca²⁺ ions released with solubilized silica (H₂SiO₄²⁻) to form CSH according to reaction 10:

$$Ca^{2+} + H_2 SiO_4^{2-} \rightarrow CaO \cdot SiO_2 \cdot H_2O(CSH)$$
 (10)

3.5. Influence of Cement and Metakaolin on the Geotechnical and Mechanical Properties of the Raw Material

The variations of the optimal moisture content and the maximum dry density as a function of the quantity of cement-metakaolin are illustrated in **Figure 10**. The OMC increases while the MDD decreases with the addition of cement and metakaolin. The increase in OMC is due to the strong affinity of the cement-metakaolin mixture with water to initiate the hydration reactions of their anhydrous components. In addition, the synthesis of hydrated calcium silicate (CSH) also needs water for the dissociation of the portlandite produced in order to produce Ca^{2+} ions. The OMC increases more when the metakaolin content in the mixture increases. This is linked to the high demand for water to initiate the process of the pozzolanic reaction, especially in the portlandite dissociation stage [35]. Also this significant water demand would be due to the fineness of the metakaolin.

The reduction of the MDD would be due to the fact that the addition of cement-metakaolin creates the aggregation of the particles. As a result, the volume of the material increases, leading to a change in the particle size distribution. This result is in agreement with that reported by Osulaon cement-amended laterites in Nigeria [33]. The decrease in MDD is more felt for composites rich in metakaolin and this is justified by the low density of metakaolin compared to that of cement.

The results of the California Bearing Ratio (CBR) test of the composites are presented in **Figure 11**. All the CBR index values are greater than 40%, which is

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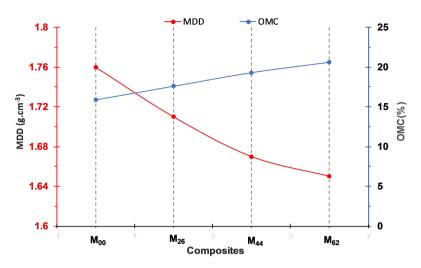


Figure 10. Variations of the optimum moisture content (OMC) and maximum dry density (MDD) of composites.

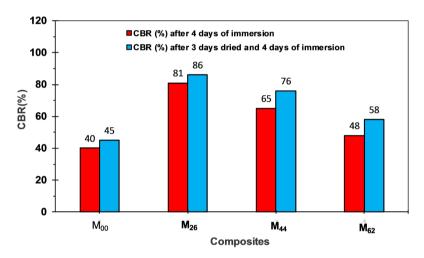


Figure 11. Evolution of California Bearing Ratio (CBR) (after 4 days water immersed, 3 days dried and 4 days of immersion) of composites.

the natural sample. Also, the CBR for the formulations at three (3) days of air cure and after four (4) days of immersion are better than the CBR after 4 days of immersion. Finally, formulations containing more cement than metakaolin have the best CBR.

The increase in CBR would be essentially due to the formation of CSH following the pozzolanic reaction involving the portlandite of the cement and the amorphous silica of the metakaolin. The lower values of the CBR for the specimens immediately immersed result from the uninterrupted process of the hydration of the cement and the lack of consolidation of the CSH. For specimens having undergone a 3-day cure in the air before being immersed, the cement had time to set at a young age. Specimens containing more cement have the best CBR due to the high content of CSH formed by the hydration pathway of alite and belite compared to CSH formed by the pozzolanic reaction [34]. The CBR obtained are lower than those reported by Millogo *et al.* [26] on lateritic gravels improved with cement and lime. This difference is explained by the nature of the two samples. Indeed, the sample studied is more plastic than the sample studied by Millogo *et al.* The 2 wt.% metakaolin + 6 wt.% cement composite is suitable for a base course in road construction regardless of the type of traffic. The composite (4 wt.% metakaolin + 4 wt.%) cement is better suited for the sub-base course in road construction but could be used as a base course for low traffic [26]. As for the composite low in cement (6 wt.% metakaolin + 2 wt.% cement), it can only be used for the sub-base course like the natural sample. Nevertheless, the natural sample was improved in terms of CBR. These results prove that with the contribution of the cement-metakaolin mixture, the material reacts favorably both to the possible poor hygrometric conditions observable in practice and to the constraints imposed on a pavement. The addition of cement and metakaolin markedly improved the geotechnical properties of the base lateritic clay. The maximum observed (81%) for 2 wt.% metakaolin + 6 wt.% cement is the most favorable case with a CBR index greater than 80%.

The results of the simple compressive strength and tensile splitting strength tests for the different composites are presented in Figure 12. Overall, the simple compressive and tensile splitting strengths of the clay-cement-metakaolin composites are better than those of the raw sample. As with CBR, composites rich in cement compared to metakaolin have the best strengths. Thus, the resistances increase until reaching the maximum value of 4.5 MPa in simple compression against a value of 0.5 MPa in tension then decrease for the other formulations. The improvement in resistance is explained, as for CBR, by the formation of CSH, a cementitious compound responsible for the mechanical strength. These CSH then play the role of glue that will bind the isolated particles of the clay matrix while reducing the porosity [34]. The drop in resistance is related to the formation of an excessive amount of calcite in the mixture.

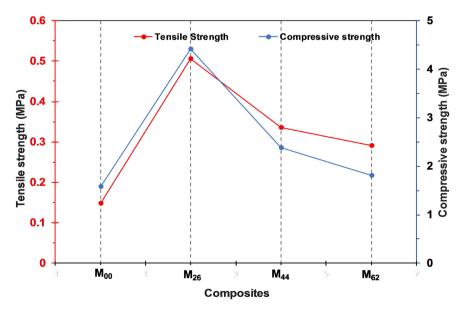


Figure 12. Variation of the compressive strength and tensile strength of composites.

Calcite is formed in large quantities because the portlandite formed is not fully used for the pozzolanic reaction, so the rest will undergo a carbonation reaction [36].

The simple compressive strengths and the splitting tensile strengths for the engineered composites are higher than those reported by Messou, Bahar and Kolias [28] [37] [38] [39] on lateritic soils improved with cement. This could be due to the mineralogical composition of the soils studied by these authors which would be richer in clay minerals compared to our basic raw material ALK.

4. Conclusions

This work has made it possible to characterize the lateritic raw material of Kamboinse and then to develop composites from clay-cement-metakaolin mixtures for application in road construction. The following conclusions emerge from this study:

- The chemical and mineralogical characterizations showed that the raw material is essentially composed of kaolinite (65.7 wt.%), quartz (20.5 wt.%) and goethite (10.78 wt.%) with a very low content of materials organic and in amorphous phases;
- The geotechnical and mechanical characterizations indicated that the ALK clay is not very plastic and rich in fine particles. In view of the value of the CBR at 95% which is 40%, ALK could be used as a sub-base course in road construction;
- The mineralogical analysis of the various composites obtained showed the formation of new phases such as calcite, portlandite and especially hydrated calcium silicate CSH resulting from the hydration of the cement and the pozzolanic reaction;
- The value of the CBR of all the composites at 95% compactness is better than that of the natural sample, showing a contribution of these mineral binders in terms of mechanical resistance. Only the composite containing 6 wt.% cement and 2 wt.% metakaolin is suitable for the development of a base course in road construction. However, a study of sustainability on the mechanical performance in the long term is necessary in order to understand the behavior of composites in time.

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Conflicts of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

References

- Millogo, Y., Hajjaji, M., Ouedraogo, R. and Gomina, M. (2008) Cement-Lateritic Gravels Mixtures: Microstructure, and Strength Characteristics. *Construction and Building Materials*, 22, 2078-2086. https://doi.org/10.1016/j.conbuildmat.2007.07.019
- [2] SouleyIssiakou, M. (2016) Caractérisation et valorisation des matérieux latéritiques utilisés en construction routière au Niger. Master's Thèse, Université de Bordeaux, Bordeaux.
- [3] Attoh-Okine, N.O. (1995) Lime Treatment of Laterite Soils and Gravels Revisited. *Construction and Building Materials*, 9, 283-287. <u>https://doi.org/10.1016/0950-0618(95)00030-J</u>
- [4] Autret, P. (1986) Latérites et graveleux lateéritiques. Laboratoire Central des Ponts et Chaussées France.
- [5] Gartner, E. (2004) Industrially Interesting Approches to "Low-CO₂" Cements. *Cement and Concrete Research*, 34, 1489-1498. https://doi.org/10.1016/j.cemconres.2004.01.021
- [6] Ouedraogo, M., Sawadogo, M., Sanou, I., Barro, M., Nassio, S., Seynou, M. and Zerbo, L. (2022) Characterization of Sugar Cane Bagasse Ash from Burkina Faso for Cleaner Cement Production: Influence of Calcination Temperature and Duration. *Result in Materials*, 14, Article ID: 100275. https://doi.org/10.1016/j.rinma.2022.100275
- [7] Oriol, M. and Pera, J. (1995) Pozzolanic Activity of Metakaolin Binder under Microware Treatment. *Cement and Concretes Research*, 25, 265-270. https://doi.org/10.1016/0008-8846(95)00007-0
- [8] Bich, C., Ambroise, J. and Péra, J. (2009) Influence of Degree of Deshydroxylation on the Pozzolanic Activity of Metakaolin. *Applied Clay Science*, 44, 194-200. https://doi.org/10.1016/j.clay.2009.01.014
- [9] Dao, K., Ouedraogo, M., Millogo, Y., Aubert, J.E. and Gomina, M. (2018) Thermal, Hydric and Mechanical Behaviours of Adobes Stabilized with Cement. *Construction and Building Materials*, **158**, 84-96. https://doi.org/10.1016/j.conbuildmat.2017.10.001
- [10] Seynou, M., Millogo, Y., Zerbo, L., Sanou, I., Ganon, F., Ouedraogo, R. and Kaboré, K. (2016) Production and Characterization of Pozzolan with Raw Clay for Burkina Faso. *Journal of Minerals and Materials Characterization and Engineering*, 4, 195-209. <u>https://doi.org/10.4236/jmmce.2016.43018</u>
- [11] ASTM International (2008) ASTM Standard, C618-22: Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolan for Use in Concrete. Annual Book of ASTM standards, West Conshohocken.
- [12] AFNOR (1999) NF P94-093. Détermination des références de compactage d'un matériau.
- [13] AFNOR (1999) NF P98-231-1. Comportement au compactage des matériaux autres que traités aux liants hydrocarbonés: Partie 1, Essai Proctor modifié adapté aux graves et sables utilisés en assises de chaussées.
- [14] AFNOR (1978) NF P18-560. Analyse granulométrique par tamisage.
- [15] AFNOR (1993) NF P94-051. Détermination des limites d'Atterberg.
- [16] AFNOR (1993) NF P94-068. Mesure de la quantité et de l'activité de la fraction argileuse. Détermination de la valeur de bleu de méthylène d'un sol par l'essai à la tache.

- [17] AFNOR (1992) NF P11-300. Exécution des terrassements Classification des matériaux utilisablesdans la construction des remblaiset des couches de formed'infrastructures routières.
- [18] AFNOR (1981) NF P18-406. Bétons—Essai de compression.
- [19] AFNOR (1981) NF P18-408. Bétons—Essai de fendage.
- [20] Elfil, H., Srasra, E. and Dogguy, M. (1995) Caractérisation physico-chimique de certaines argiles utilisées dans l'industrie céramique. *Journal of thermal Analysis*, 44, 663-683. <u>https://doi.org/10.1007/BF02636285</u>
- [21] Qtaitat, M.A. and Al-Trawneh, I.N. (2005) Characterization of Kaolinite of the Baten El-Ghoul Region/South Jordan by Infrared Spectroscopy. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, **61**, 1519-1523. https://doi.org/10.1016/j.saa.2004.11.008
- [22] Konan, L.K., Seil, J., Andji, J.Y.Y., Soro, N.S., Oyetola, S., Toure, A.A. and Kra, G.
 (2001) Etude de quelques échantillons d'argiles du site de Sekoudé (Côte d'Ivoire). *Journal de la Société Ouest-Africaine de Chimie*, 11, 181-196.
- [23] Ouédraogo, M., Dao, K., Millogo, Y., Aubert, J.E., Messan, A., Seynou, M., Zerbo, L. and Gomina, M. (2019) Physical, Thermal and Mechanical Properties of Adobes Stabilized with Fonio (*Digitaria exils*) Straw. *Journal of Building Engineering*, 23, 250-258. <u>https://doi.org/10.1016/j.jobe.2019.02.005</u>
- [24] Yvon, J., Garin, P., Delon, J.F. and Cases, J.M. (1982) Valorisation des argiles kaolinitiques des Charentes dans le caoutchouc naturel. *Bulletin de Minéralogie*, 10, 431-437. <u>https://doi.org/10.3406/bulmi.1982.7565</u>
- [25] Alliprandi, G. (1979) Matériaux Réfractaires et Céramiques Techniques, Eléments de Céramurgie et de Technologie. Septima, Paris, 237-250.
- [26] Millogo, Y., Traore, K., Ouedraogo, R., Kabore, K., Blanchart, P. and Thoassin, J.H. (2008) Geotechnical, Mechanical, Chemical and Mineralogical Characterization of a Lateritic Gravels of Sapouy (Burkina Faso) Used in Road Construction. *Construction and Building Materials*, 22, 70-76. https://doi.org/10.1016/j.conbuildmat.2006.07.014
- [27] CEBTP (1984) Guide pratique du dimensionnement des routes pour les pays tropicaux.
- [28] Messou, M. (1980) Comportement mécanique d'une couche de base en graveleuxlatéritiques améliorés au ciment: Cas des routes en Côte d'Ivoire. Master's Thèse, Ecole Nationale des Ponts et Chaussées, Paris.
- [29] Millogo, Y., Morel, J.C., Traore, K. and Ouedraogo, R. (2012) Microstructure, Geotechnical and Mechanical Characteristics of Quicklime-Lateritic Gravels Mixtures Used in Road Construction. *Construction and Building Materials*, 26, 663-669. https://doi.org/10.1016/j.conbuildmat.2011.06.069
- [30] Dutta, D.K., Bordoloi, D. and Prakash, C. (1995) Hydratation of Portland Cement Clinker in the Presence of Carbonaceous Materials. *Cement and Concrete Research*, 25, 1095-1102. <u>https://doi.org/10.1016/0008-8846(95)00104-K</u>
- [31] Dias, C.M.R., Cincotto, M.A., Savastano, J.H. and John, V.M. (2008) Long-Term Aging of Fibercement Corrugated Sheets—The Effect of Carbonation, Leaching and Acid Rain. *Cement and Concrete Composites*, 21, 255-265. https://doi.org/10.1016/j.cemconcomp.2007.11.001
- [32] Rojas, M.F. and Cabrera, J. (2002) The Effect of Temperature on the Hydration Rate and Stability of Hydration Phases of Metakaolin-Lime-Water Systems. *Cement and Concrete Research*, 32, 133-138. https://doi.org/10.1016/S0008-8846(01)00642-1

- [33] Osula, D. (1996) A Comparative Evaluation of Cement and Lime Modification of Laterite. *Engineering Geology*, 42, 71-81.
 https://doi.org/10.1016/0013-7952(95)00067-4
- [34] Sanou, I., Seynou, M., Zerbo, L. and Ouedraogo, R. (2019) Mineralogy, Physical and Mechanical Properties of Adobes Stabilized with Cement and Rice Husk Ash. *Science Journal of Chemistry*, 7, 1-10. <u>https://doi.org/10.11648/j.sjc.20190701.11</u>
- [35] Frias, M. and Cabrera, J. (2001) Influence of MK on the Reaction Kinetics in MK/Lime and MK-Blended Cement Systems at 20°C. *Cement and Concrete Research*, 31, 519-527. <u>https://doi.org/10.1016/S0008-8846(00)00465-8</u>
- [36] Akpokodje, E.G. (1985) The Stabilization of Some Arid Zone Soils with Cement and Lime. *Quarterly Journal of Engineering Geology*, 18, 173-180. https://doi.org/10.1144/GSL.QJEG.1985.018.02.06
- [37] Bahar, R., Benazzoug, M. and Kenai, S. (2004) Performance of Compacted Cement-Stabilised Soil. *Cement and Concrete Composites*, 26, 811-820. https://doi.org/10.1016/j.cemconcomp.2004.01.003
- [38] Basha, E., Hashim, R., Mahmud, H. and Muntohar, A. (2005) Stabilization of Residual Soil with Rice Husk Ash and Cement. *Construction and Building Materials*, 19, 448-453. <u>https://doi.org/10.1016/j.conbuildmat.2004.08.001</u>
- [39] Kolias, S., Kasselouri-Rigopoulou, V. and Karahalios, A. (2005) Stabilization of Clayey Soils with High Calcium Fly as Hand Cement. *Cement and Concrete Composites*, 27, 301-313. <u>https://doi.org/10.1016/j.cemconcomp.2004.02.019</u>