# Characterization and potential use of Dingui clay material as partial substitute for clinker in the manufacture of construction cement


## Abstract

This work focuses on the valorization of local clay materials to reduce the environmental impact of construction cement production. Clinker production during Portland cement manufacturing is known to be responsible for significant amounts of global carbon dioxide ($CO_2$) emissions. The objective of this study is the physicochemical and mineralogical characterization of raw clay from Dingui, in the Bouenza region of the Republic of Congo, with a view to its potential use as a partial substitute for clinker in cement manufacturing.
To assess the performance of this clay, the following analyses were carried out: particle size analysis and Atterberg limits, X-ray diffraction (XRD), chemical analysis by X-ray fluorescence spectroscopy, followed by calcination of the clays and an evaluation of the mechanical properties of the formulated cements.
According to the results obtained, the Dingui sample is a silty clay, highly plastic with a plasticity index of 42, composed of 56% clay, 43% silt, and 1% sand. It consists primarily of kaolinite as the clay mineral and quartz. After substitution for clinker in well-defined proportions, cements formulated with calcined Dingui clay exhibit interesting pozzolanic properties comparable to those of CEM II cement. Mechanical tests carried out on cements formulated with this clay show results that meet the requirements of standards EN 197-1 and NCGO 0004-1 2017-09. Thus, this clay can be used in the manufacture of eco-responsible cement capable of significantly reducing the carbon footprint by up to 30%.



**Keywords**: valorization, characterization, calcination, clinker substitution, Dingui.


## 1. Introduction

The global cement industry is undergoing a critical transition, driven by the need to reduce its carbon footprint, which currently accounts for approximately 8% of global anthropogenic CO2 emissions [1,2]. This environmental burden stems primarily from the thermal decarbonation of limestone during clinker production, a process occurring at around 1450°C and releasing massive amounts of carbon dioxide [3]. In the Republic of Congo, this issue is exacerbated by a structural dependence on clinker imports, weakening the rapidly expanding construction sector.
Faced with these challenges, the development of local mineral resources, such as the clay deposits of Dingui in the Bouenza region, represents a promising technological breakthrough. The transformation of an inert crystalline structure (kaolinite) into an amorphous metakaolin is essential to make it highly reactive by promoting its pozzolanic activity [4,5,6].

The incorporation of these activated phases into innovative binding systems, such as calcined clay limestone cement (LC3), promotes heterogeneous nucleation of hydration products. This chemical mechanism, through the active consumption of released portlandite, leads to the formation of hydrated calcium silicate and aluminosilicate gels (C-S-H and C-A-S-H), which densify the porous matrix [7]. This micro-architectural restructuring provides structures with increased resistance to the chemical aggressions typical of tropical environments, thus creating a physico-chemical shield essential to their durability [8].

However, the rational exploitation of the Dingui deposit remains contingent upon overcoming scientific challenges related to its intrinsic mineralogical reactivity.

The objective of this work is to characterize the Dingui clay for its potential use as a partial substitute for clinker in the manufacture of construction cement. Thus, this study aims to elucidate the mechanisms by which the thermal activation of this clay enables the synthesis of a high-performance hydraulic binder capable of reconciling regulatory requirements with industrial sovereignty. To this end, a multiscale characterization (XRD, X-ray fluorescence, particle size analysis, and Atterberg limits) was implemented, followed by the calcination of these clays and the evaluation of the mechanical properties of the formulated cements, thereby paving the way for endogenous and eco-responsible production in the Republic of Congo.

## 2. Materials and methods

### 2.1. Clay sample

The raw clay material was taken from Dingui, a locality located in the Bouenza department in the Republic of Congo. The clay sample was dried, crushed and sieved to obtain a fine granulometry.

### 2.2. Characterization of raw clay material

**Particle size analysis**

Particle size analysis was carried out by sieving in accordance with standard NFP94-056 [9] and by sedimentometry in accordance with standard NFP94-057 [10]. The test sample size was 200g.

**Plasticity study**

The plasticity study was carried out by studying the Atterberg limits using 0.08µm sieve passes in accordance with standard NFP94-051 [11]. The test sample weighed 70g.

**Chemical composition**

The chemical composition of raw Dingui clay was determined by QCX (Quality Control X-ray) X-ray fluorescence spectrometry.

**Mineralogical composition (DRX)**

The mineralogical analysis of the Dingui clay was carried out by X-ray Diffraction (DRX), recorded using a PANaltycalX'Pert Pro Diffractometer in Bragg Brentano theta/2theta geometry. The angular analysis range was 3° to 93°.

### 2.3 Cement formulation

**Calcination and Pozzolanic Activation**

Pozzolanic activity was promoted by calcining the Dingui clay sample in a kiln to the optimum temperature of 750°C.

**Cement manufacture**

Two types of cement were formulated: Control Cement (CC) and Modified Cement (MC). Control Cement was formulated by mixing 95% Clinker + 5% Gypsum. Modified Cement was formulated by adding to the clinker-gypsum mixture, raw clay heat-treated at 750°C and finely ground, so as to partially substitute the clinker. We obtained three modified cement formulations named A1, A2 and A3 (Table 2).

**Performance evaluation**

To evaluate the performance of the formulated cements, the following analyses were carried out on these cements: pozzolanicity tests, fineness measurement, standard consistency measurement, setting time measurement and mechanical strength measurement.

Pozzolanicity tests were carried out to assess the reactivity of the modified clay.

Fineness was measured using the Blaine method (air permeability) in accordance with standard EN 196-6 [12]. It was measured by observing the time taken for a fixed volume of air to pass through a compact cement bed with a specific porosity of 0.5. The test sample was 2.96g of cement. The specific surface area (in $cm^2$/ g) of the Blaine was calculated using the following formula:

$$S = K\sqrt{t} \quad (1)$$

K= 41.02 (Apparatus constant; t: Time)

**Standard consistency** was measured using the Vicat method or the Vicat normal consistency test, in accordance with European standard EN 196 - 3 [13]. Standard consistency was assessed by measuring the penetration of a cylindrical rod into the paste under a constant load. The greater the penetration, the more fluid the consistency. 140ml of water were added to 500g of cement introduced into the mixer tank.

The following formula (2) was applied to calculate the standard consistency:

$$\textbf{Standard consistency} = \frac{\text{Mass of water}}{\text{Mass of cement}} \times 100 \quad (2)$$

**The setting time** was measured using the Vicat method by determining the start and end of the setting of cement pastes, by monitoring the consistency of a paste using the Vicat apparatus equipped with a needle 1.13 mm in diameter. The setting time was measured from the start of mixing until the needle stopped at a distance d= 4mm (± 1 mm) from the bottom of the mold, under the effect of a 300 g load. The time at the end of the set was taken when the needle was only 0.5mm from the bottom of the mold.

Mechanical strength was measured by bending and compression on prismatic specimens (40mm x 40mm x 160mm) of standard mortar (a mixture of cement, standard sand and water) in two stages:

- Preparation of the specimens and

- Fracture of specimens prepared by flexion and compression.

## 3. Results and Discussion

### 3.1 Characterization of raw clay material

**Particle size analysis**

Figure 1 below shows the Particle size analysis curve for raw clay material from Dingui. It summarizes the composition of this material as follows: 56% clay, 43% silt and 1% sand. This result positioned in the texture diagram (figure 2), shows that this material is silty clay.

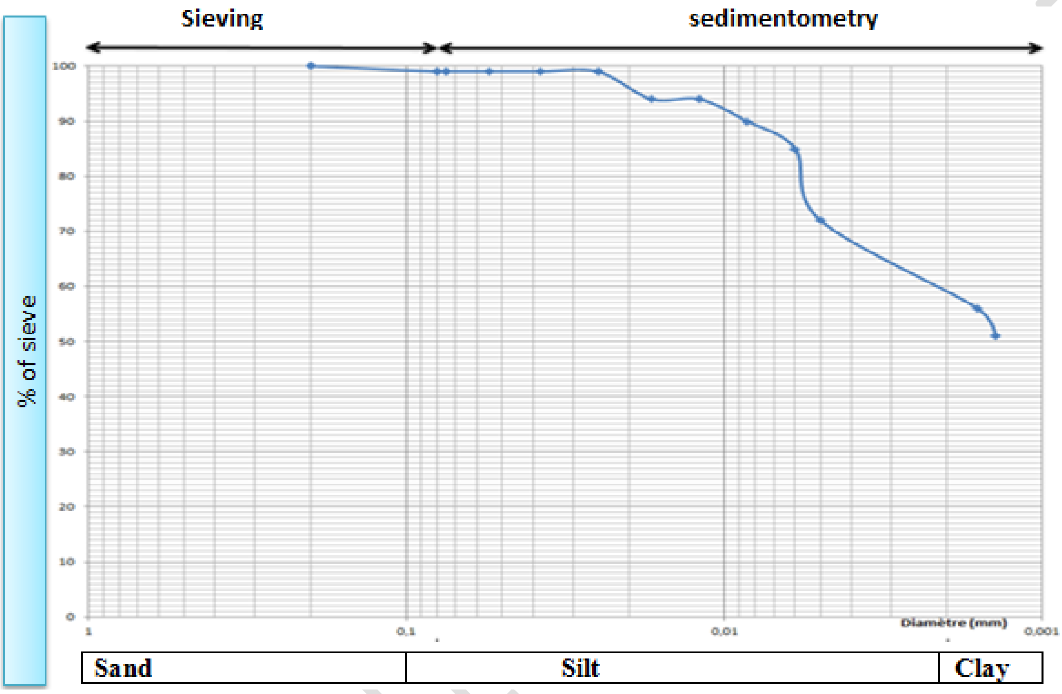


Figure 1: Particle size distribution of the Dingui sample

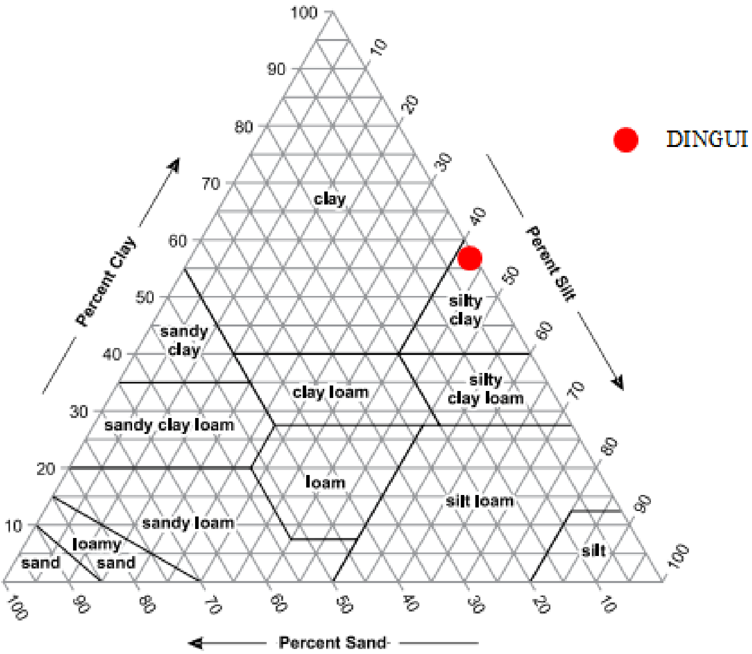


Figure 2: Texture triangle “soil survey”

**Study of plasticity**

The Atterberg limits measured, expressed as a percentage (%), are as follows:

- The liquid limit WL= 84.8;

- The plasticity limit WP= 42.8

The plasticity index (PI) was deduced from the difference between the WL and WP values according to equation 3 below:

PI (%) = WL - WP (3)

In this study, the calculated PI is 42. This means that the PI is greater than 40. In the Casagrande chart (Figure 3), the Dingui sample is placed in the highly plastic silt zone. The relatively high value of this plasticity index is in line with the particle size distribution, which not only has relatively high clay content (56%), but also a high percentage of silt (43%), which reinforces this plasticity.

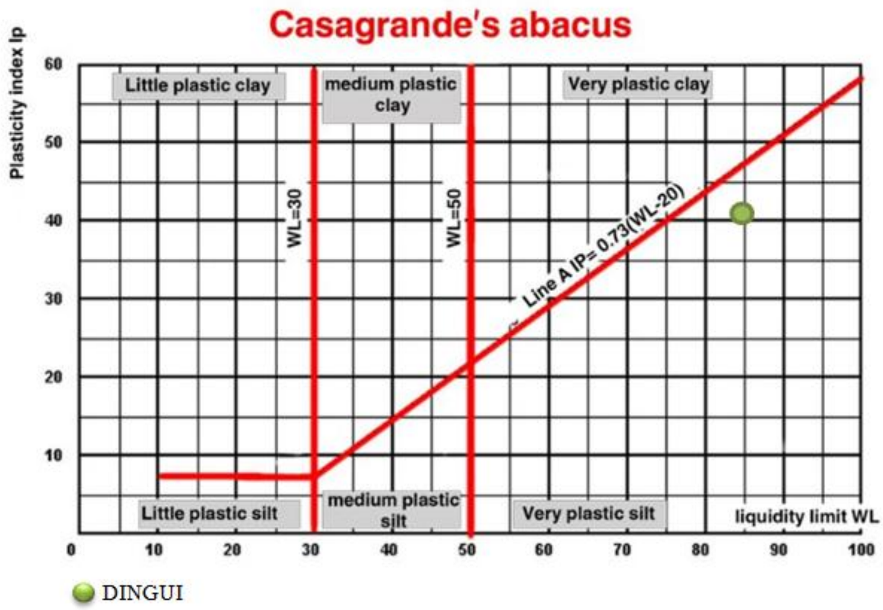


Figure 3: Casagrande plasticity diagram

**Chemical composition:**

Table 1 below gives the chemical composition of Dingui clay. It shows the quantities of major oxides present in this sample

Table 1: Determination of major oxides

| **Oxide** | **$SiO_2$** | **$Al_2O_3$** | **$Fe_2O_3$** | **CaO** | **MgO** | **PAF** |
|---|---|---|---|---|---|---|
| **%** | 62,94 | 17,77 | 3,72 | 0,11 | 1,81 | 11,79 |

**Mineralogical composition (XRD)**

Figure 4 below gives us the x-ray diffractogram of the Dingui sample at 0.01.

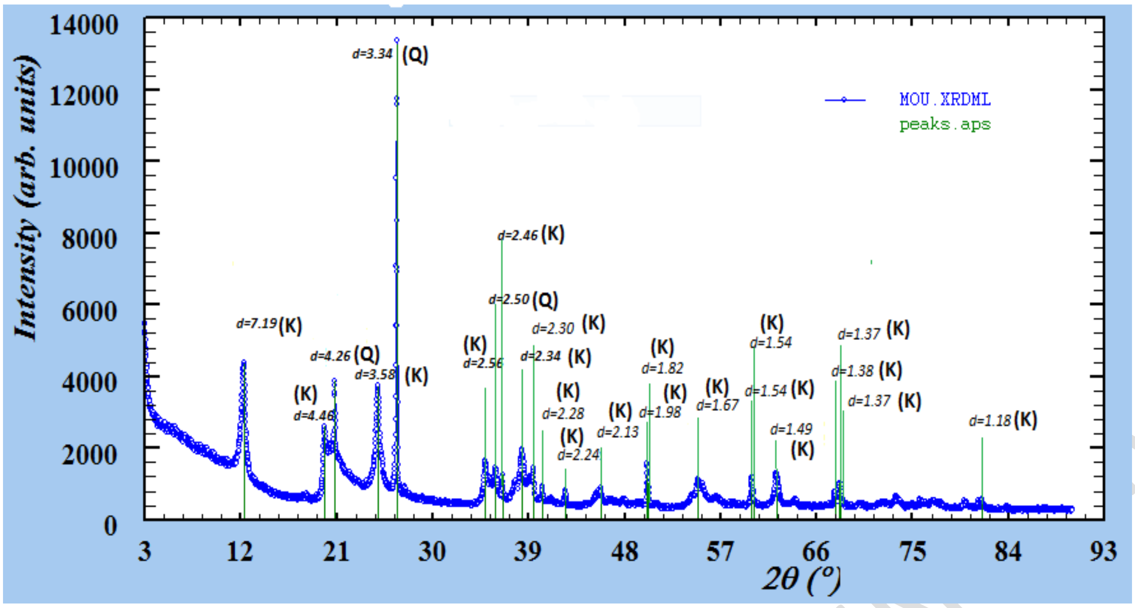


Figure 4: X-ray diffractogram of the Dingui raw clay sample at 0.01

Preliminary examination of the diffractogram of the Dingui raw clay sample gave us the following reflections, which reveal the presence of kaolinite, taking as a reference the JCPDF sheet for kaolinite: 7. 19Å, 4.46Å, 3.58Å, 2.56Å, 2.34Å, 2.24Å, 2.28Å, 1.98Å, 1.82Å, 1.67Å, 1.54Å, 1.49Å, 1.38Å, 1.37Å, 1.18Å.

The lines at 4.26Å, 3.34Å and 2.50Å indicate the presence of quartz in this sample.

Figure 5 below shows the DRX spectrum of the Dingui clay sample in the angular range from 3° to 33° with a maximum intensity of 4700 counts.

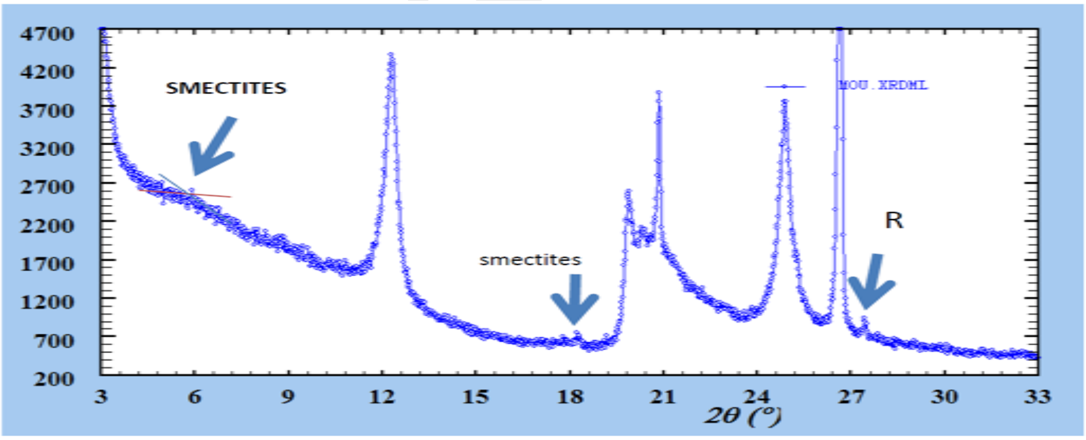


Figure 5: Diffractogram of the Dingui clay sample at 4700 counts over a scan from 3 to 33°

At 6.02549° (2θ) we see a slight change in the slope of the continuous background. This suggests the presence of a peak buried in the continuum background, a broadly spread peak around 14.667Å, probably indicating the presence of a smectite (swelling clay) as noted by Moutou et al. in a study on Loukolela clay [14]. The low-intensity peak at 18.00796° (2θ) corresponding to a lattice distance of 4.93Å generally indicates the presence of montmorillonite [14].

According to Tchoubar et al, the position of the (001) line is shifted towards the small angles as the number of structural defects present in the mineral increases [15].

The observation of this XRD spectrum between 24° and 26° is shown in figure 6.

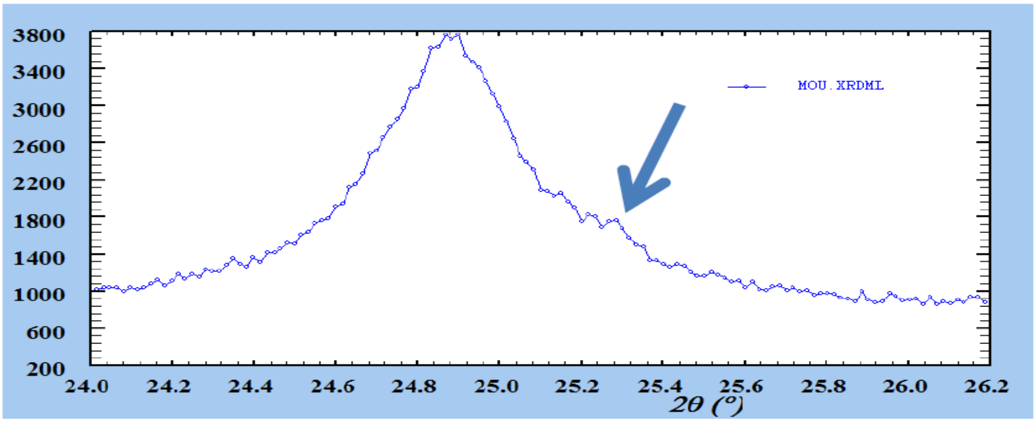


Figure 6: Diffractogram of the Dingui clay sample on a 24 to 26° angle scan

We can think that the shoulder of the 3.58Å peak of the kaolinite, which would be a peak at 3.52Å, is an anatase peak. The peak at 27.376°, i.e. 3.25Å, may correspond to rutile (R) or feldspar (microcline).

## 3.2. Cement formulation

**Calcination and Pozzolanic Activation**

As kaolinite is the predominant clay mineral in this Dingui material, calcination at the optimum of 750°C led to the formation of a highly reactive amorphous structure called metakaolinite, which reacts with the portlandite (calcium hydroxide, $Ca(OH)_2$ released by the hydration of the clinker, to form mainly hydrated calcium aluminosilicates (C-A-S-H) and hydrated calcium aluminates (C-A-H), with pozzolanic properties [16].

$Al_2Si_2O_7$ (Metakaolin) + $Ca(OH)_2$ + $H_2O$ = C-A-S-H + C-A-H (3)

**Cement manufacture**

Table 2 below shows the different cement formulations (CP, A1, A2 and A3) and the proportions of the different constituents.

Table 2: Cement formulation

| Nomination | Clincker (%) | Gypsum (%) | Added clay (%) |
|---|---|---|---|
| CC | 95 | 5 | 0 |
| A1 | 85 | 5 | 10 |
| A2 | 75 | 5 | 20 |
| A3 | 65 | 5 | 30 |

The chemical analysis of formulated cement powders is summarized in the following table 3:

Table 3: Chemical composition of formulated cements

| | $SiO_2$ | $Al_2O_3$ | $Fe_2O_3$ | CaO | MgO | $SO_3$ | $K_2O$ | $Na_2O$ | LOI |
|---|---|---|---|---|---|---|---|---|---|
| **CC** | 19,66 | 4,75 | 4,03 | 62,76 | 2,73 | 2,14 | 1,08 | 0,13 | 1,38 |
| **A1** | 21,5 | 5,13 | 4,09 | 59,81 | 2,64 | 2,16 | 1,38 | 0,14 | 2,22 |
| **A2** | 23,34 | 5,43 | 4,16 | 56,41 | 2,35 | 2,2 | 1,55 | 0,14 | 3,47 |
| **A3** | 24,03 | 5,59 | 4,26 | 53,03 | 2,44 | 2,3 | 1,69 | 0,12 | 4,41 |

The cements obtained meet the requirements of standard EN 197-1 and the Congolese standard (NCGO 0004-1 2017-09). The loss on ignition (LOI) is less than 5%, the sulphate content is less than 3.5% and the MgO content is also less than 3%.
In addition, it can be seen that the sum of the relative quantities of reactive calcium oxide (CaO) and silicon dioxide ($SiO_2$) represents a proportion greater than 50% by mass, for the two types of cement (CC and MC) which can be referred to as CEM cements. The control cement is similar to CEM I Portland cement and the modified cements to CEM II cement. The cements obtained were characterized in accordance with EN 197-1 and NCGO 0004-1 2017-09.

### 3.3 Performance evaluation of formulated cements

**Analysis of the physical properties of formulated cements**

Table 4 below shows the physical properties of formulated cements

Table 4: Physical properties of formulated cements

| | CC | A1 | A2 | A3 |
|---|---|---|---|---|
| **Volume of water (ml)** | 144 | 148 | 155.5 | 168 |
| **Standard Consistency W/C(%)** | 28,8 | 29,6 | 31,1 | 33,6 |
| **Setting time (min)** | 98 | 119 | 115 | 99 |
| **Time to set (min)** | 219 | 285 | 261 | 232 |
| **Finesse ($Cm^2/g$)** | 3599 | 4322 | 4549 | 4512 |

W: Water; C: Cement; ml: millilitre; g: gram

According to this table, the increase in the volume of water needed to moisten 500g of cement, the W/C ratio increases with the percentage of addition. This is because the additives absorb a large proportion of the water used to hydrate the cement. As a result, the quantity of water available for hydrating the anhydrous cement and forming hydrates such as portlandite and ettringite is reduced. This table shows the increase in setting time (start and finish) of the variants compared to portland cement, due to the subtraction of part of the clinker and its replacement by calcined clay. All the materials (clinker, gypsum, clay) were ground separately. The clay has been calcined and the mixes made, and all the blended cements have almost the same fineness.

**Mechanical strength of mortars**

Table 5 below shows the mechanical properties of formulated cements

Table 5: Mechanical properties of formulated cements

| | CC | A1 | A2 | A3 |
|---|---|---|---|---|
| Compressive strength at 2 days (Mpa) | 21,1 | 24,7 | 21,7 | 19,6 |
| Flexural strength at 2 days (Mpa) | 4,6 | 5,6 | 5,1 | 4,7 |
| Compressive strength at 28 days (Mpa) | 44,8 | 44,1 | 40,0 | 37,0 |
| Flexural strength at 28 days (Mpa) | 7,9 | 8,5 | 7,9 | 7,7 |

Specimens of the different variants were tested in flexion and uniaxial compression at 2 days and 28 days respectively. At 2 days, the mechanical strengths of the formulated cements are higher than those of the Portland cement taken as a control (CC), this is explained by the high value of the modulus of fineness, except in the case of A3, because its hydraulic index is low. At 28 days, the Portland cement regained the advantage, as the clinker continued to hydrate and the formulated cements slowed their growth.
Incorporating calcined clay at optimum rates is likely to lead to early strength (2 days) comparable to the control cement, followed by a significant increase in long-term strength (after 28 days), due to the dense formation of C-A-S-H from the pozzolanic reaction on the one hand. On the other, it leads to a denser concrete/mortar microstructure and a reduction in porosity, which is crucial for better durability and resistance to the penetration of aggressive ions (chlorides and sulphates) [17].

### 3.4. Environmental and economic benefits

**Reduction in clinker content:** The substitution of a portion of the clinker by calcined clay in the manufacture of cement leads to a reduction in $CO_2$ emissions. In fact, reducing the clinker content by up to 30% reduces $CO_2$ emissions by approximately 30%.

**Energy savings:** Calcining at much lower temperatures (750°C compared with 1450°C for clinker) reduces energy consumption and, consequently, production costs.

**Local Resource Enhancement:** The use of Dingui silty clay adds value to an abundant local resource.

## 4. Conclusion

The aim of this work was to characterize Dingui clay with a view to its use as a substitute for clinker in the manufacture of construction cement. The results show that the Dingui material is a silty clay, composed mainly of kaolinite and quartz. It can be used as an additive in the manufacture of environmentally-friendly cement. After calcination at the optimum temperature of 750°C, this clay is activated and transformed into an excellent partial substitute for clinker, enabling the manufacture of cement with comparable technical performance to cement known as CEM cement. Unlike the manufacture of clinker, the thermal treatment of this clay does not generate $CO_2$ emissions, so it contributes to a considerable reduction in carbon footprint and energy costs. Its pozzolanic properties have been proven in specific tests. Its introduction at different percentages as an addition in substitution for clinker has made it possible to obtain a modified cement that complies with EN 197-1 and NCGO 0004-1 2017-09. Mortars were formulated using these modified cements and reference cements. This shows that the substitution of part of the clinker by calcined clay leads to mortars that meet the mechanical requirements at a young age (2 days), and that improve long-term mechanical strength (28 days).It can be said that these cements can be used in structures with moderate stresses.

These results highlight the influence of clay additions on the properties of cementitious materials, highlighting the scientific, economic and environmental interest of replacing clinker with calcined clays.

Future work should focus on the precise optimisation of calcination temperature and time, and on a large-scale study of the performance of calcined clay-limestone cement (LC3) formulated with Dingui clay.

Finally, this study highlights the value of an abundant local resource.

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