

CHARACTERISATION OF THE MINERALOGY, DURABILITY AND PHYSICAL PROPERTIES OF TWO EARTH MORTARS: AN EXPERIMENTAL STUDY

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Abstract

Mortars composed of a blend of earth and cement are widely employed in masonry, particularly in the construction of earth blocks. This study is primarily focused on investigating the influence of mineralogical and chemical constituents on the mechanical characteristics of two types of mortars. Furthermore, we have assessed the water absorption coefficient of these mortars, which vary in cement content, with the aim of enhancing their longevity. The findings reveal a significant enhancement in compressive strength, attributed to the interaction between calcium hydroxide in cement and clay minerals containing silico-aluminous compounds. Importantly, the incorporation of cement not only affects the macroscopic properties concerning particle size fractions but also heavily depends on the mineralogical composition, particularly the clay content. Moreover, the addition of cement results in a reduction in water absorption, whereas an increase in magnesium oxide content within the clay leads to higher water absorption rates.

Keywords:

Durability;
Mortar;
Cement;
Mechanical;
Mineralogy;
Earth material.

1 Introduction

Earthen materials have long been fundamental in the construction of human habitats and urban landscapes, marking a rich legacy of earthen architecture worldwide. This heritage reflects a myriad of techniques honed over time and across diverse geographical and cultural settings. Within this spectrum, earth-based mortars serve essential functions as fillers, plasters, and binding agents in structures ranging from adobe to fired brick and stone masonry. Despite sharing primary components of soil and water, these mortars exhibit notable variations in their physical and mechanical properties. This diversity stems from the intrinsic complexity of raw materials, including variations in soil composition, particle sizes, moisture levels during mixing, and the addition of additives such as cement, natural fibers, lime, brick, or straw. Moreover, factors like the method of mortar preparation, the skill of the labor force, and prevailing environmental conditions exert significant influence on their ultimate characteristics [1]. Employing indigenous earth as a construction material or for restoration purposes offers numerous benefits owing to its sustainability and eco-friendliness. With lower carbon emissions and reduced transportation costs due to local sourcing, earth material becomes more affordable and accessible, particularly for rural communities. Furthermore, its usage leads to decreased energy consumption in

maintaining cultural structures. Hence, grasping the chemical and mineral composition of raw earth is crucial, necessitating identification and characterization of its components.

Recently, there's been a proposition suggesting that mortars can be delineated by either their mineralogical composition or microstructural appearance. Understanding the physico-chemical properties of mortar is pivotal for deciphering its composition. Mineralogical and chemical analyses of mortar provide valuable insights into its properties, including binders, aggregate types, grading, and quantitative composition. It's imperative to evaluate the suitability of clay materials before producing stabilized mortar specimens from earth material, considering factors like stabilizer type, constituents, and production processes.

This study has delved into textural and plastic properties to select appropriate clay materials for stabilized mortar production. Hence, understanding chemical interactions emphasizes the significance of understanding how the chemical and mineral composition of clay materials influences their performance in stabilized mortar specimens.

Stabilization is systematically applied to enhance the mechanical qualities of the oldest earth constructions. Chemical and mineralogical earth characterization relies on mechanical analysis and tests, assessing the mechanical characteristics of compressed earth mortar. This study involves the chemical-mineralogical analysis of cement-stabilized earth mortar, determining its mechanical characteristics under both dry and wet conditions. The latter is achieved through simple compression tests, preceded by capillary rise tests, conducted on cylindrical specimens sourced from two cities in Morocco: Fez and Rabat (Fig. 1b).

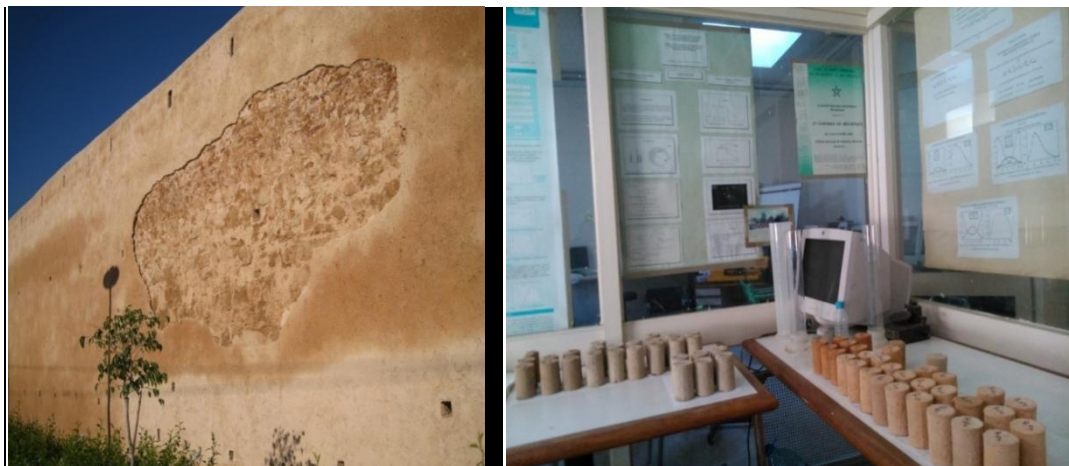


Fig. 1: a- weathering of earth construction

b- Mortar test samples

2 Material and methods

2.1 Material

The earth material utilized for crafting mortar specimens is sourced from the cities of Fez and Rabat in Morocco, identified as F and R respectively throughout this investigation. The particle size distribution of the sample is evaluated by employing wet sieving techniques to segregate the coarser, adhering to the NF 94-056 standard [9]. Analysis indicates that F earth comprises 27.78% fine particles (clay + silt), whereas R earth contains 38.2% (see Table 1). This fine fraction serves as a binding agent for the larger grains, influencing the selection of mortar specimens to be fabricated. Table 1: The particle size distribution of the two types of earth, F and R

	Fraction granular [%]			
	Coarse sands	Fine sand	Silt	clays
Earth Fez	41.67	30.55	16.67	11.11
Earth Rabat	48.46	15.34	17.71	20.49

Table 2 displays the Atterberg limits determined in accordance with standard NF P94-051 [10]. The geotechnical properties of F and R suggest the feasibility of utilizing them for mortar specimen

production. Additionally, the specific surface area is determined as $Sa = 21 * VB$, where VB represents the volume of methylene blue absorbed. The test results are presented in the table below:

Table 2: Consistency Limits of Earth Types R and F

	Wp [%]	WL [%]	IP [%]	Ac	Sa [m ² /g]
Earth Fez	25.32	35.58	10.26	1.28	48.93
Earth Rabat	15.04	21.10	6.06	0.39	77

The coefficient of activity, Ac, presented in Table 3, offers insights into the swelling and shrinkage characteristics of materials, particularly clays. Earths can be categorized based on their activity levels as follows:

Table 3: Activity coefficient (Ac)

Activity	Inactive	Average Activity	Active	Very active
Ac	< 0.75	0.75 < Ac < 1.25	1.25 < Ac < 2	> 2

Based on this data, it is observed that the Fez earth is active, whereas the Rabat earth is inactive.

2.2 Preparation of test materials

Irrespective of the construction technique employed, the preparation of construction soil follows a standardized procedure outlined in detail to produce soil-based constructions [11]. In this study, the analyzed materials were derived from two stabilized earths, F and R, tested at various weight percentages (0%, 4%, 7%, and 10% of cement by weight). Initially, the soil clay is manually crushed and then sifted through a 2-mm sieve, with any remaining particles subsequently crushed. Upon achieving a consistent mass, the sifted soil is blended with cement. Once the dry mixture achieves homogeneity, water is gradually introduced. The inclusion of water in the mixture exerts significant influence on several factors, including compressive strength, porosity, density, aggregate adhesion, and the bond between the mortar and the masonry [12]. Consequently, mortar specimens undergo a drying period lasting several weeks, typically around 4 weeks. The presence of water activates the properties of earth, leading to notable effects such as the occurrence of cracks and changes in volume, which are critical mechanical properties in earth construction masonry. The water-to-binder ratio, often a key consideration in other types of mortar, offers valuable insights into the characteristics of earth mortars, including the type of clay present. Certain clay minerals, such as montmorillonite, exhibit a higher affinity for water absorption compared to others like kaolinite [11].

2.3 Methods

2.2.1 Mineralogical and chemical analysis

X-ray Diffraction (XRD)

One essential analytical technique for determining the presence of minerals and other crystalline phases in a variety of material samples, including sedimentary rocks, is X-ray diffraction (XRD). In this application, the powder XRD patterns were performed using X-ray diffractometer (Siemens D 500) equipped with a copper anticathode ($\lambda_{CuK\alpha} = 1.541838 \text{ \AA}$) with scanning speed of $0.04^\circ/\text{s}$, from 5 to $60^\circ (2\theta)$ and at a tube voltage of 35 kV and a current of 30 mA. Minerals in the samples were identified using database software from the International Centre for Diffraction Data (ICDD) as source of reference diffraction patterns (JCPDS files).

Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared spectroscopy (FTIR) combined with Attenuated Total Reflectance (ATR) was employed to qualitatively identify organic, polymeric, and inorganic materials by analyzing their characteristic absorption bands. The spectra were recorded using a Jasco FT/IR 4600 spectrometer provided with ATR (Pro One) module. The samples were scanned in transmission with 4 cm^{-1} resolution at the range $4000\text{--}400 \text{ cm}^{-1}$.

Scanning Electron Microscopy (SEM)

SEM (Quanta 200) serves the purpose of characterizing the distribution and morphology of minerals, along with identifying organic matter within sedimentary rocks. This technique entails imaging the sample at various magnifications to acquire high-resolution micrographs and achieve enhanced contrast between solid phases and voids.

X-ray Fluorescence (XRF)

XRF analysis measures the amount of minerals in the test samples, enhancing X-ray diffraction analysis. To do this, the FX Axios PW4400 spectrometer system is used. The proportions of the major elements in the samples, represented as oxides, were determined using the instrument's software.

2.2.2 Test mode

In this investigation, cylindrical specimens with dimensions of $\varnothing = 60$ mm in diameter and $h = 100$ mm in height were used in two different test conditions: wet and dry. First, the capillary rise phenomenon was the subject of investigation, with parameters pertaining to the hydric properties of the building materials used being examined. The capillary water absorption coefficient (C_b), one of these factors, is crucial for earth constructions. It controls how liquid moisture flows through capillary pores, giving an indication of how quickly water is absorbed by the material and transferred through capillarity. This coefficient has a significant impact on a number of applications, both in conventional and modern building practices. As such, it is essential to take into account when figuring out the material's hydrometric and mechanical properties.

The specimens were dried at 40 ± 5 °C until they reached a constant mass, and then they were put in room temperature desiccators. One minute was the starting point for measuring the rate of water absorption, followed by 3, 5, 10, 15, 20, 40, 60, 90, 120, 240, and 1440 minutes. In each interval, three samples of each composition are tested. The results of non-stabilised samples are discarded due to material loss from the water submersion.

By dividing the water absorption by the surface area in contact with water and then dividing by the square root of time, the water absorption coefficient, or C_b ($\text{g/mm}^2 \text{min}^{0.5}$), was calculated using the following formula:

$$C_b = 100a \frac{(m_h - m_d)}{s\sqrt{t}} = 100a \frac{(m_h - m_d)}{s\sqrt{10}} \quad (1)$$

Where:

m_d - denotes the initial weight of the sample in grams.

m_h - indicates the weight of the sample at different time points in grams.

s - represents the surface area in contact with water in square centimetres.

t - denotes the time in minutes.

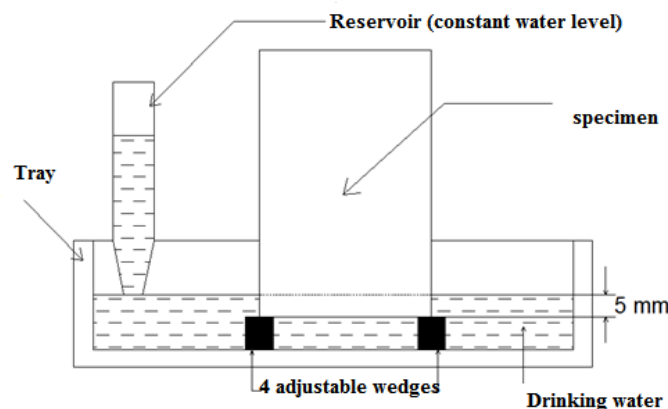


Fig. 2: The capillary absorption test.

The mortar specimens underwent testing after a curing period of 28 days. Following the recommended methods for determining dynamic compressive and tensile strengths for mortar materials by G.H. Houben [35], cylindrical specimens were employed in dynamic compression and tension tests, respectively. The cylindrical specimens, measuring 60 mm in diameter and 100 mm in height, were manufactured from the mortar specimens (Fig.3).



Fig. 3: Experimental Material for Dry and Wet Compressive Testing

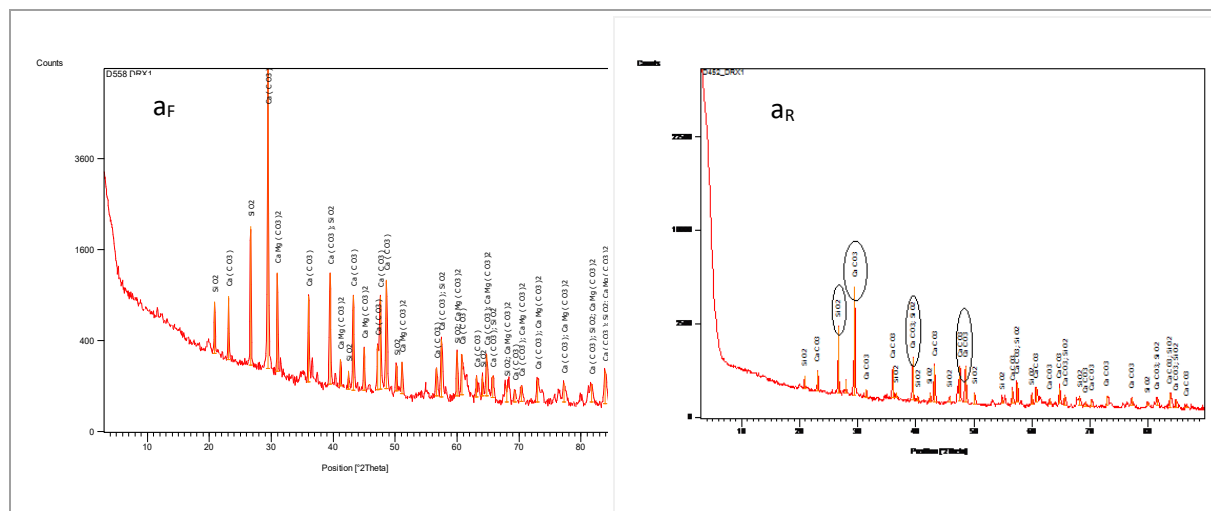
3 Results and discussion

3.1 Mineralogical and chemical analysis findings

Several analytical techniques, such as X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), scanning electron microscopy (SEM), and fluorescence X, were used in the mineralogical and chemical analysis of two types of earth.

3.1.1 X-ray diffraction

The X-ray diffraction shows that the sample F contains mainly calcite, dolomite, and quartz. When cement was added to the mortar test sample, calcite levels increased significantly and small amounts of aluminum silicate compounds were found (Fig. 4.b_F, c_F, d_F). Similarly, quartz and calcite were found in the clay from earth R (Fig. 4.a_R) upon analysis. When cement is added to the mortar test sample, there was also an elevation in calcite content and the detection of aluminum silicate in a weak quantities (Fig. 4.a_R, b_R, c_R).



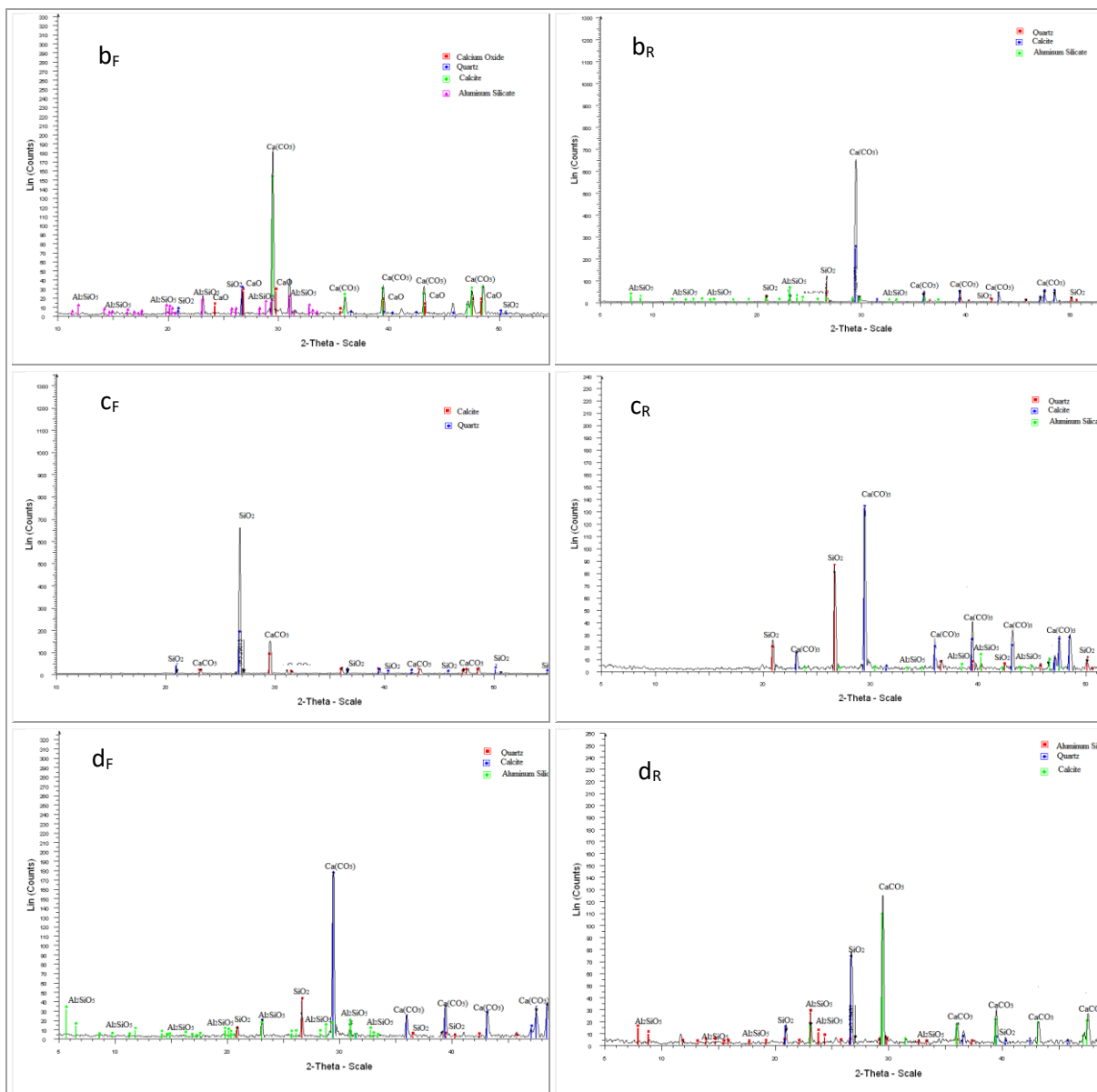
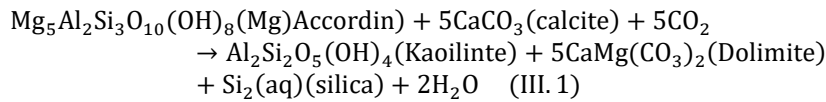


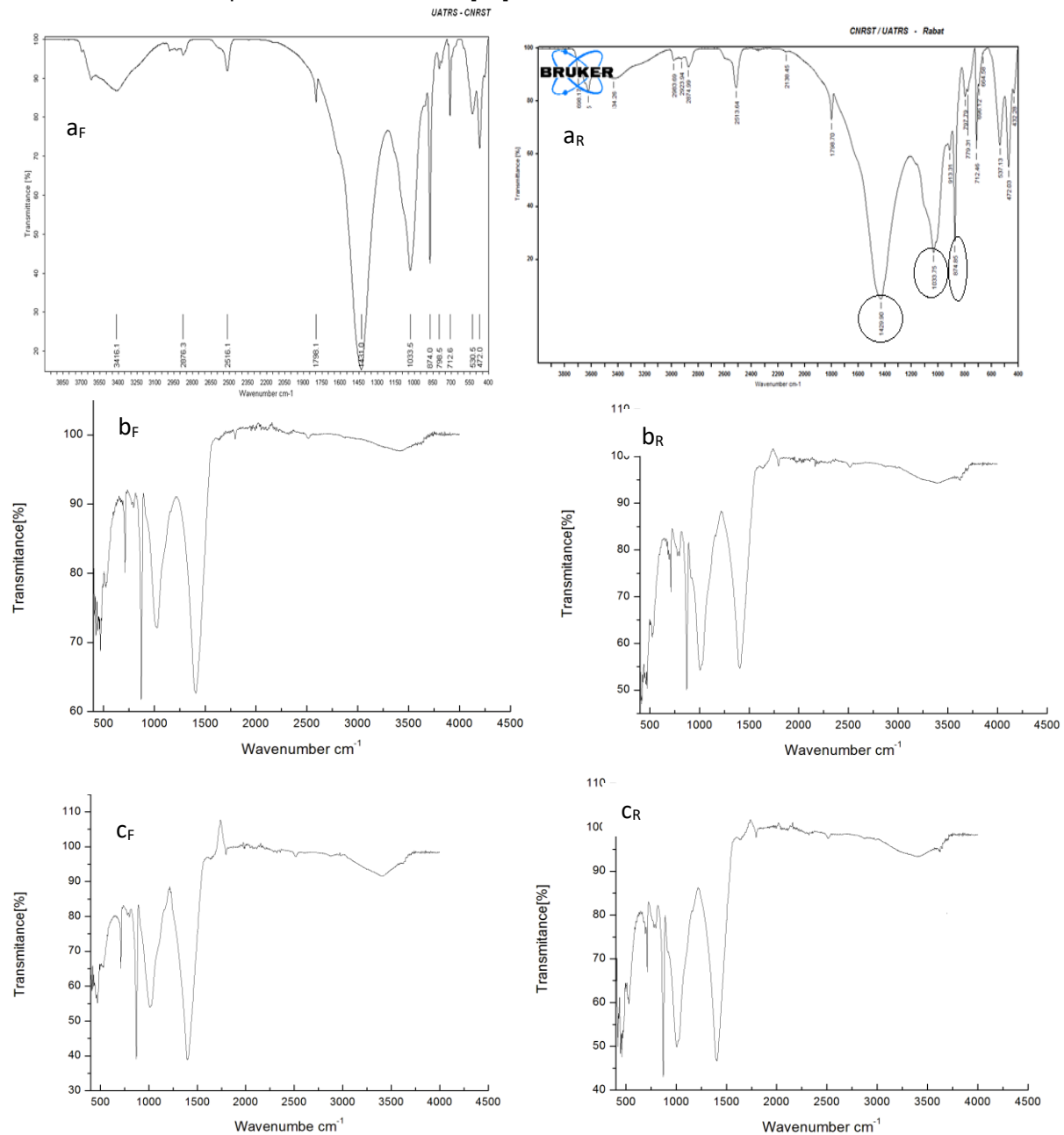
Fig. 4: X-ray diffraction patterns of the cement mixed samples (F and R): (a) raw, (b) 4 wt.% cement, (c) 7 wt.% cement, and (d) 10 wt.% cement.

3.1.2 Fourier Transform Infrared Spectroscopy (FTIR)

The infrared spectroscopy analysis of clay earth F and its mortar specimens with varying cement content are depicted in (Fig. 5.a_F, b_F, c_F, d_F). The main bands characterizing the presence of calcite are identified by peaks at (1798.1, 1431, 874, 712.6) cm⁻¹. Additionally, the infrared spectra exhibit bands associated with quartz, identified by peaks at (1033.5, 798) cm⁻¹, and water at (3416.1 cm⁻¹). Calcite is formed through lime carbonation, which, when mixed with water and tempered with sand, rapidly agglomerates inert particles present in the sample, such as quartz [4, 13, 14]. The analysis of clay earth R, along with its mortar specimens (Fig. 5. a_R, b_R, c_R, d_R), confirms the presence of calcite and quartz as the main secondary minerals. Calcite is distinguished by peaks at (1798.70, 1429.90, 874.85, 2874.99, 712.46) cm⁻¹, whereas quartz has peaks at (1033.75, 797.79, 779.31) cm⁻¹. Furthermore, the presence of kaolinite (Al₂Si₂O₅(OH)₄) and chlorite is indicated by peaks at (472.03, 696.12, 913.31, 3622.25, 3696.17) cm⁻¹ [4, 13-18, 42]. Additionally, the band at 537.13 cm⁻¹ is prominent in chlorite and decreases with the presence of Mg [42]. The hydration water and hydroxyl of hydrated calcium silicate are detected near peaks at 3400 cm⁻¹ and 1636 cm⁻¹ [13, 15]. According to reaction III.1, the presence of Al and Si is responsible for the formation of kaolinite. [19].



Silico-aluminates and similar compounds can impart specific hydraulic properties to the binder. These non-crystalline compounds frequently evade detection by X-ray diffraction tests. The emergence of calcium silico-aluminates in earth mortars containing brick fragments may be due to the pozzolanic reaction between lime and the silico-aluminates compounds found in the burnt clay of brick mortar [35]. We found bands corresponding to humic and fulvic acids at (2513.64, 537.13, 2923.94, 530.5, 2876.3) cm^{-1} [4,13-16, 20-22]. The spectrum of clay earth R shows bands in the 3000-2800 cm^{-1} and 1500-1400 cm^{-1} regions, which are typical of organic matter. Furthermore, strips appearing around 470, 530, and 797 cm^{-1} indicate the presence of muscovite [20].



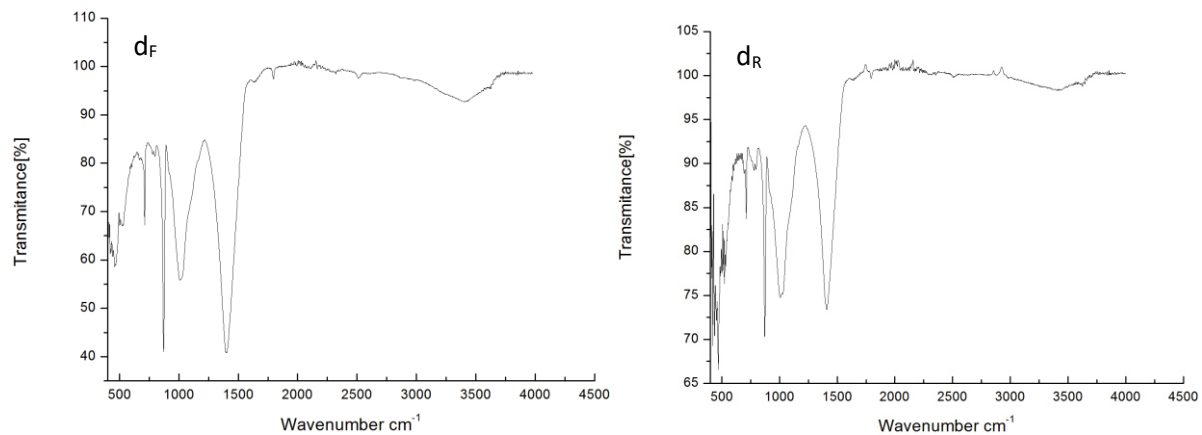


Fig.5: Infrared spectra of the cement mixes samples (F and R): (a) raw, (b) 4 wt.% cement, and (c) 7 wt.% cement and (d) 10 wt.% cement

3.1.3 Scanning Electron Microscope (SEM)

The SEM allows us to generate three-dimensional images of sample surfaces with extremely high resolution at the nanometer scale, providing qualitative information about the sample's homogeneity. The scanning electron microscopy (SEM) images of clays from Earths F and R (Figs. 6 and 7) show a higher and dominant calcite content compared to quartz in both clays, which is due to the high lime content in the earth.

The figures below depict microscopic observations of samples taken at various scales throughout the study.

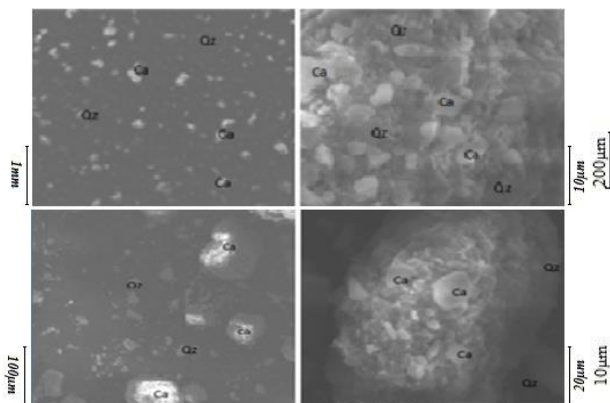


Fig. 6: SEM micrographs of clay F.

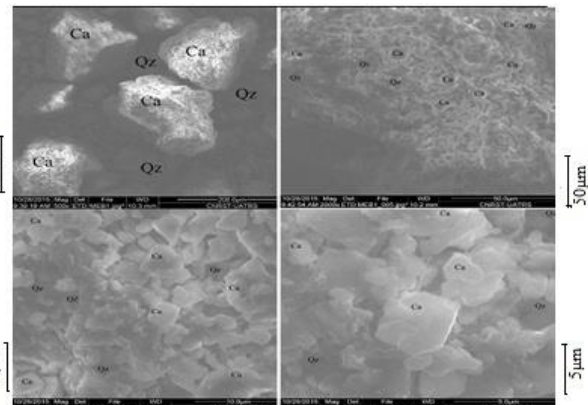


Fig. 7: SEM micrographs of clay R.

(Qz: quartz, Ca: calcite)

3.1.4 Analysis using Fluorescence X

Fluorescence X analysis describes the chemical element proportions found in the clay derived from earth R and F, as well as the CPJ35 cement used to make the mortar specimens. Table 4 summarizes the chemical compositions.

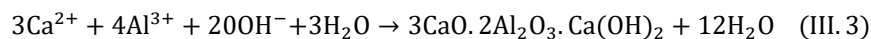
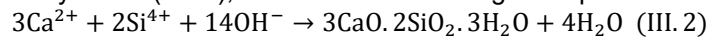
Table 4: Chemical compositions of the concerned raw material of F, R and Cement CPJ35

Composition [%]	SiO ₂	CaO	Al ₂ O ₃	Fe ₂ O ₃	MgO	SO ₃	K ₂ O	TiO ₂	MnO ₂	P ₂ O ₅	Na ₂ O	SrO	PAF 975°C
Ciment CPJ35	12.46	57.02	3.39	1.89	1.35	1.95	0.62	0.18	0.03	0.11	0.03	0.015	20.90
Earth F	22.3	31.8	5.96	1.72	2.13	1.29	0.536	0.226	0.0503	0.753	0.323	0.0125	32.9
Earth R	21.6	35.2	7.77	3.43	0.86	0.204	0.522	0.354	0.104	0.447	0.574	0.0595	28.6

Table 4 illustrates an increase in CaO content in both earth clay and the cement utilized in this study, attributed to the carbonate matrix and a high SiO₂ percentage due to the presence of quartz and siliceous sand.

According to the analysis, carbonates are primarily composed of calcite, with quartz serving as the siliceous species. Moreover, X-ray diffraction has confirmed the presence of pyrite, dolomite, and feldspars, though only in trace amounts. The low potassium oxide concentration (0.5%) indicates that illite is unlikely to be present in the sample [20]. Earth F has a higher magnesium content than earth R, with dolomite accounting for the detected MgO values. The presence of significant amounts of organic matter contributes to the sample's darker coloration, whereas the high iron oxide content in earth sample R produces a yellow-brown hue [16].

The chemical reactions (Equations III.2 and III.3) involving silica (SiO₂), alumina (Al₂O₃), water, and slaked lime [Ca(OH)₂] from quicklime or cement in a pozzolanic reaction lead to the formation of hydrated calcium silicate (CSH) and calcium aluminate hydrate (CAH) [23,24]. This chemical process produces calcium silicate hydrates (CSH), which contribute to high compressive strength [25,26,27].



3.2 Experimental results of mortar durability

The water absorption coefficients of earth F and R vary with cement additions, as shown below:

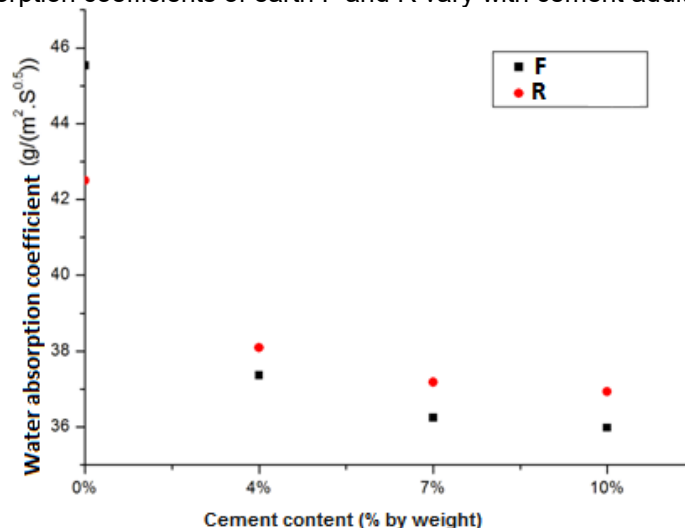


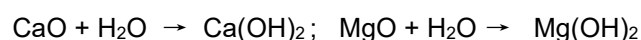
Fig.8: The influence of cement additions on the variation of absorption of water in F and R earth specimens.

Fig. 8 depicts the decrease in the water absorption coefficient of F and R with increasing cement dosages from 0% to 10%. At 0% cement, the water absorption value for earth F is 45.53 (g/ (m².S^{0.5})) and for earth R is 42.50 (g/ (m².S^{0.5})).

However, the results of the granular fraction analysis of the two earths indicate that earth F contains 11.11% clay, while earth R contains 20.49% clay. Typically, in the literature [28–30], when clay content is high in the granular compositions of earth, it tends to absorb a significant amount of water. Surprisingly, our study found opposite results for 0% cement in the mortar specimens of earth F.

According to fluorescence analysis, which provides the content of chemical elements in the constituents of the clay:

- The clay of earth F contains 31.8% quicklime, 22.3% quartz, and 2.13% magnesium oxide.
- For earth R, there is 35.2% quicklime (CaO), 22.6% quartz (SiO₂), and 0.86% magnesium oxide (MgO). It's noteworthy that Magnesium Oxide and Calcium Oxide interact with water according to the following reactions:



The discrepancy in magnesium oxide content between the clay of earth F and earth R is notable, with earth F exhibiting a concentration 2.5 times higher than that of earth R. Consequently, at 0% cement, the water absorption coefficient of earth F surpasses that of earth R. With cement contents

ranging from 4% to 10% in earth F, the water absorption coefficient decreases demonstrates a decline from 38.09 ($\text{g}/(\text{m}^2 \cdot \text{S}^{0.5})$) to 36.93 ($\text{g}/(\text{m}^2 \cdot \text{S}^{0.5})$), while for earth R, it gradually reduces from 37.37 to 35.98 ($\text{g}/(\text{m}^2 \cdot \text{S}^{0.5})$). Studies referenced from [28, 29] suggest that incorporating cement into the earth enhances the micro-pore structure by generating a cement gel and clay agglomerates, thereby modulating water penetration into the mortar specimens. Consequently, the water absorption coefficient decreases diminishes with increasing cement content.

3.3 Experimental results of compressive strength and interpretations

Fig. 9 illustrate the mean experimental outcomes of compressive strength (R_c) relative to cement proportions of 0%, 4%, 7%, and 10% by weight, observed in both the dry and wet conditions of cylindrical test samples of earth mortar F and R.

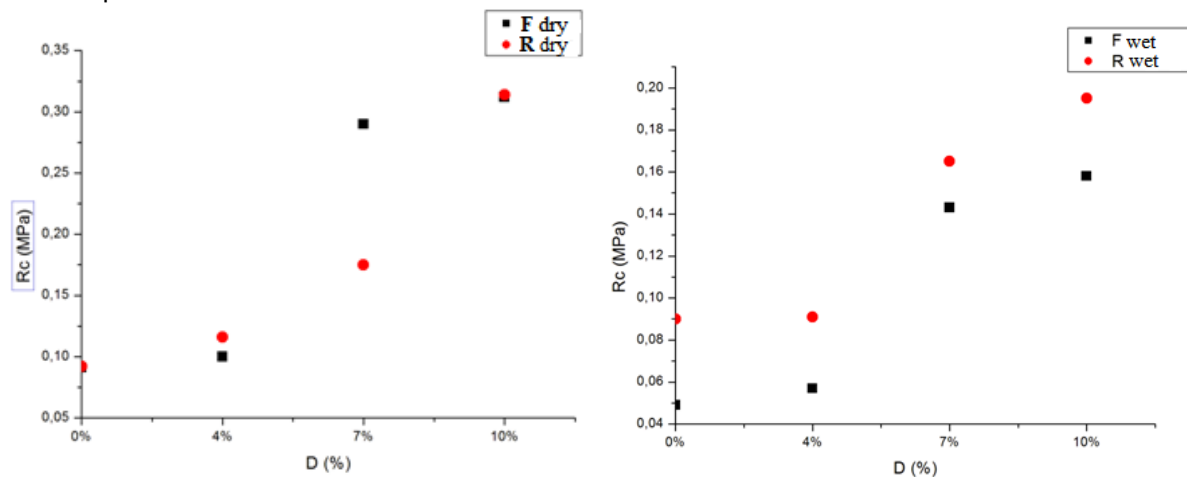


Fig. 9: Compressive strength of mortar F and R in the wet and dry state as a function of the cement content D (%)

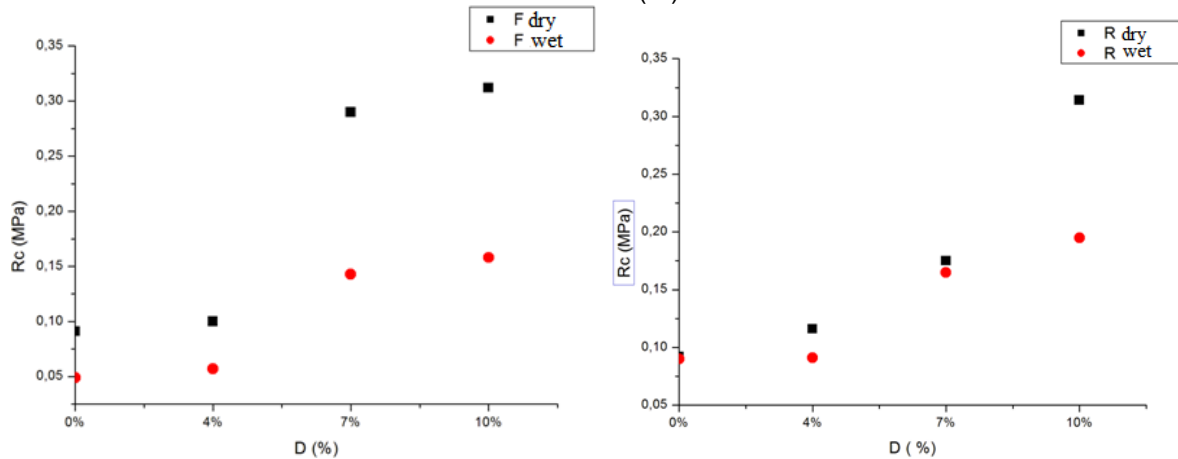


Fig.10: Variation of compressive strength of mortar specimen's F and R in both cases as a function of cement content D (%)

The variation in compressive strength of the mortar, both in wet and dry conditions, for earths F and R increases with the cement content, ranging from 0%, 4%, 7%, to 10% by weight, as depicted in Fig. 9 and 10. This increase in compressive strength differs between the two types of earth. Notably, for earth F, a significant increase in compressive strength (both wet and dry) is observed at 7% by weight of cement ($R_s = 0.29$ MPa and $R_h = 0.143$ MPa). Conversely, the mortar of earth R exhibits enhanced compressive strength (wet and dry) at 10% by weight of cement ($R_s = 0.314$ MPa, $R_h = 0.195$ MPa).

Regarding the granular fraction, the sandy earth F contains 11.11% clay, while the sandy-clayey earth R contains 20.49% clay. Nevertheless, mineralogical assessment via X-ray diffraction of clay from earth F and earth R indicates that both mortar types are primarily composed of calcite (CaCO_3), quartz (SiO_2), and silico-aluminous compounds (kaolinite). According to the study by J. Garcia-Ten et al. [40],

calcite contributes to higher mechanical strength compared to dolomite due to the presence of unreacted MgO. Additionally, the inclusion of potassium feldspar and quartz can decrease mechanical strength. Moreover, kaolinitic clay slightly enhances fired mechanical strength in mortar specimens.

Several researchers [18, 37] have indicated that quartz does not function as a binder but rather constitutes a part of the fine sand fraction. According to Rodriguez, N.C et al. [33], calcite is formed by the carbonation of Ca(OH)_2 slaked lime, which absorbs carbon dioxide (CO_2) from the surroundings. Studies by A. Ammari et al. and K. Bouassria et al. [32, 34] have shown that silico-aluminous compounds function as binders through chemical interactions with calcium hydroxide (Ca(OH)_2) when water is present at room temperature.

In the X-ray diffraction analysis (Fig. 2.c_F) of earth F with 7% cement content by weight, the absence of silico-aluminous (kaolinite) peaks indicates a complete reaction with calcium hydroxide Ca(OH)_2 , leading to the formation of compounds with binding capabilities, thereby enhancing the compressive strength of both dry and wet earth F mortar.

Concerning earth R, which contains 20.49% clay, studies by H. Houben et al. and H. Bouabid et al. [35, 36] have concluded that if clay content exceeds 20%, more cement is needed for stabilization. However, the X-ray spectra of mortar R (Fig. 2.d_R) at 10% cement reveal calcite (CaCO_3) parts, quartz (SiO_2) peaks, and significant silico-aluminous (kaolinite) parts. Portions of the silico-aluminous component have reacted with calcium hydroxide (Ca(OH)_2), while others remain unreacted. The improvement in compressive strength of the earth R mortar, up to 10% cement content, is attributed to the interactions among calcium hydroxide, cement, and clay minerals containing silico-aluminous. These interactions facilitate the formation of connections between clay and cement within the inert earth matrix.

The enhancement in compressive strength of mortar specimens is credited to pozzolanic reactions occurring between the cement content and clay materials [41]. These reactions lead to the formation of cementitious products like CSH and CAH, which bind the matrix of stabilized mortars. However, variations in the reactivity of different clay materials contribute to differences in the extent of compressive strength improvement among mortar specimens.

4 Conclusion

The research underscores the significance of fundamental exploration into the mineralogical, durability, and mechanical attributes of earthen mortar reinforced with cement, especially in heritage building restoration or earthen structure construction. The conclusions drawn from the study are outlined as follows:

Although clay fractions typically exhibit a high water absorption coefficient, a high clay content at 0% cement addition doesn't always result in a high water absorption coefficient. The mineralogy, coupled with grain size, influences water absorption, particularly in the presence of significant calcium hydroxide and magnesium oxide content.

The addition of cement to mortar specimens of both earth types results in an increase in calcite content and the presence of aluminum silicate or silico-aluminous compounds in small amounts. However, these compounds do not act as binders unless they undergo chemical reactions with calcium hydroxide (Ca(OH)_2) and water. Thus, the increase in compressive strength is attributed to the reaction between calcium hydroxide in cement and clay minerals containing silico-aluminous (kaolinite).

The reduction in water absorption coefficient with increasing cement content isn't linear and varies depending on particle size distribution and mineralogy. Cement addition influences not only on a macroscopic scale based on particle size fractions but also significantly depends on mineralogical composition, particularly clay. Despite the initially high absorption coefficients of earth in its raw state (0% cement), this situation can be reversed with increased cement content. Both particle size distribution and mineralogical factors play crucial roles, sometimes in competition.

The inclusion of cement in mortar leads to a significant enhancement in durability only after a certain percentage of calcite is reached.

Silico-aluminous compounds act as binders only after preceding chemical reactions with calcium hydroxide (Ca(OH)_2) and water. This results in increased strength in both dry and wet states with cement content ranging from 4% to 10% by weight.

The absence of silico-aluminous peaks (kaolinite) indicates complete reaction with calcium hydroxide, consequently improving the resistance of earthen mortar in both dry and wet states.

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