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Physical and Thermal Properties of Raw Earthern Bricks from Ksar Ait Benhaddou

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Abstract. The deterioration of historical earthen architecture in the Drâa-Tafilalet region of Morocco is becoming a significant concern. These structures are susceptible to various natural and human-induced factors, leading to their deterioration. Our research focuses on the recycling and valorization of debris from the deteriorated walls of Ksar Ait Benhaddou to potentially facilitate restoration. Various techniques were employed to characterize the debris from Ksar Ait Benhaddou, including geotechnical analysis (Atterberg limits, grain size), physicochemical assessment (X-ray diffraction, Infrared spectroscopy, X-ray fluorescence), and microscopic examination (Scanning electron microscopy). Standardized brick specimens were created from debris waste paste mixed with 22% water by mass. Additional specimens were prepared by incorporating stabilizers (portland cement or lime) or natural plant fibers (wheat straw) into the debris, with a water/solid ratio of 22%. The prepared specimens underwent aging for different periods (0 to 4 days). The study investigated the impact of aging duration and three additives on mechanical properties, material thermal conductivity, and hydrate formation. The debris exhibited medium plasticity, consisting of non-swelling clays and sand. The compressive strength of cement-stabilized samples yielded the best results, reaching 1.90 MPa for the DC5 sample. The thermal conductivity of samples stabilized with cement increased, contrasting with lime and straw, which had the opposite effect.

Introduction

Morocco has had a clay-based material industry since the 1970s. However, the actual development of it began in the 1990s with the implementation of the national construction project known as "200 000 housings"[1,2]. Clay is typically used in artisanal or semi-industrial ceramics, or even in its raw form [3,4]. Clay-based ceramics in Morocco are primarily funded and overseen by small businesses and artisans. Despite their contributions, the national production is still regarded as insufficient and below the desired standard. This situation prompted Morocco to import ceramic materials from neighboring countries like Spain and Italy.[1]. The increases in demand for clayey construction material will be growing due to the devastating earthquake of 8 September 2023 that struck the El Haouz region of Morocco situated at about 70 km south-west of Marrakech, took a heavy toll in terms of both loss of life and damage to infrastructure and people's homes [5]. According to El Bairi et al., (2023), almost 3000 people were killed and more than 5600 injured, and about 50000 homes were totally or partially destroyed [5]. These disastrous consequences mean that this issue is of vital importance to Morocco, and it is therefore vital to supplement existing regulations and update the legislation governing the sector.

Earthen construction has advantages in terms of ecology, society, economy, and culture [6,7]. Each inhabited continent boasts a heritage of earthen architecture, with 20% of the structures listed on the UNESCO World Heritage List constructed using rammed earth materials [4]. Several studies have focused on clays stabilization with cement or lime and natural plant fibres (i.e. wheat straw) to enhance their durability [8–16].

Incorporating lime into the soil has the effect of improving its properties, reducing both swelling and plasticity [8]. Ouedraogo et al., [17], The findings indicate that a combination of 5% cement and lime was effective in improving both the mechanical and thermal properties of the studied soils. Additionally, in the same study, it was determined that approximately 5% of lime was adequate for the short-term reaction, representing the initial lime consumption.

The Ksar Ait Benhaddou is on the UNESCO World Heritage List since 1987. Visitors come from all over the world to see this building. Moreover, it is recognized as a cinematographic location, having been used for the filming of numerous masterpieces. In order to protect the site's natural elements and cultural legacy, the Center for the Restoration and Rehabilitation of the Architectural Heritage of the Atlas and Sub-Atlas areas (CERKAS[1]) has been conducting a number of restoration interventions on the Ksar since the beginning of the 1990s. The United Nations Development Program (UNDP), the Moroccan Ministry of Culture, and UNESCO's technical assistance provided funding for the intervention [18].

The aim of this research is to supply sustainable and suitable earthen material, based on the reuse of clayey ruin material, to restore the Ksar Ait Benhaddou monument. Characterization of the earthen material of the wall of this monument, which has fallen into ruin was previously performed [4]. The additives we have already chosen are cement, lime and wheat straw.

Lime and portland cement, both calcium-based stabilizers, can be incorporated into clayey soils for improvement. This enhancement involves four mechanisms: cation exchange, flocculation and agglomeration, hydration cementitious (geopolymerization of C-S-H gel), and pozzolanic reaction. Portland cement supplies the necessary chemical components for all four processes. Notably, lime is unable to complete the cementitious hydration process [19], Through the carbonation process of portlandite (calcite neoformation), it solidifies the mortar [20].

This analysis will include volume shrinkage, variations in humidity, and alterations in thermal conductivity. The assessment of stabilization-induced changes will involve the examination of scanning electron microscope images and X-ray diffraction data. Combining these tests with those conducted in previous research will enhance our comprehension of the stabilization mechanism, considering physical, chemical, and microstructural perspectives.

Material and methods

Preparation of bricks specimens

A representative sample of debris from the Ksar Ait Benhaddou was collected, and three specimens were created for each aging period as outlined below:

[1] Centre de Conservation, de restauration et de réhabilitation des ksours et kasbahs des zones atlassiques et subatlassiques (CERKAS Ouarzazate)

The debris sample was mixed with water, variable amounts of binders: portland cement, lime, and straw (Table 1). Only 5% of mineral binders (lime and portland cement) were added to earthen debris for economic and environmental coherence. The 5% of mineral binders is adequate to stabilize earthen materials effectively [17,21,22].

Due to the limited use of a hydraulic binder (5%), we opted for pre-curing the mixtures before forming the brick specimens. This method ensures that the microstructure of the hardened earth specimens remains intact without causing increased pliability in the clay. Furthermore, this strategy prevents the evaporation of mixing water, crucial for the consolidation of hydraulic binders and clay particles.

Table 1: Type and quantities of binders employed in the production of brick specimens.

	Brick specimens	Debris (%)	Cement (%)	Lime (%)	Straw (%)
Bricks stabilized without pre-curing	$DC_{NC}1$	99	1	-	-
	$DC_{NC}2$	98	2	-	-
	$DC_{NC}3$	97	3	-	-
	$DC_{NC}4$	96	4	-	-
	$DC_{NC}5$	95	5	-	-
Bricks stabilized with pre-curing	DC1	99	1	-	-
	DC2	98	2	-	-
	DC3	97	3	-	-
	DC4	96	4	-	-
	DC5	95	5	-	-
	DL1	99	-	1	-
	DL2	98	-	2	-
	DL3	97	-	3	-
	DL4	96	-	4	-
	DL5	95	-	5	-
	DS1	99	-	-	1
	DS1.5	98.5	-	-	1.5
	DS2	98	-	-	2
	DS2.5	97.5	-	-	2.5
	DS3	97	-	-	3

Experimental methods

The absorbance of water test allows for the measurement of capillary water absorption. By measuring the mass gain of the specimen in a crystallizer with water positioned at 1 cm above the specimen's underside, the water absorption values of stabilized specimens were determined [22,23].

The geometric parameters (length, breadth, and height) of the specimens were measured with 0.02 mm of errors using a sliding caliper to determine their volume.

Loss on ignition at 105°C for 2H is used to determine moisture content of samples. The moisture reduction percentage change of specimens as a function of curing time was calculated as the following formula (1):

$$Mr\ (\%) = (M\ \%\ (4\ D) - M\ \%\ (0\ D)) / (M\ \%\ (0\ D)) * 100. \quad (1)$$

As:

- Mr: Moisture reduction
- M: Moisture
- D: Days

The thermal conductivity of stabilized samples was determined utilizing the Hot Disk device model TPS 2500 meter (Al Akhawayn University, Ifrane).

Scanning Electron Microscopy was performed on stabilized specimens using a Quanta 200 model at the Technical Support Units for Scientific Research (CNRST, Rabat).

SEM images were taken by the scanning electron microscope for mixtures that gave the best results for compressive strength (DC_5, DL_1, DS_2, DC_{NC5}) to fully understand what is happening microstructurally

Raw material characterisation

The thermal conductivity of the untreated debris sample demonstrates only slight changes over different aging periods. (Fig. 1). Treating with a precuring process has the potential to enhance physical strength [24].

Though the change in thermal conductivity is slight, it could imply differences in bricks formed without additives. Therefore, the chosen duration for the remaining tests is three days.

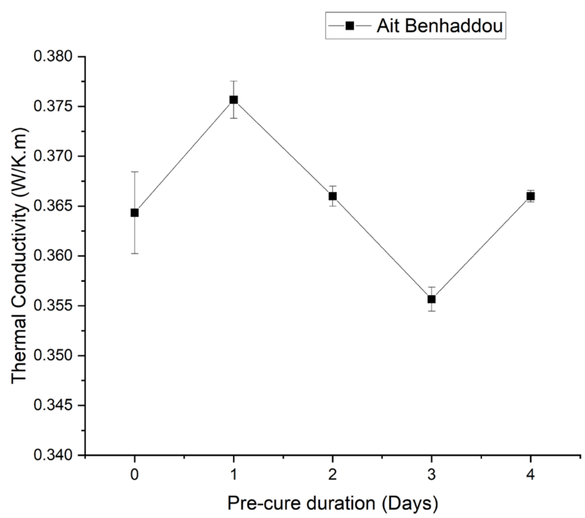

Fig. 1 : Variations in thermal conductivity observed in the untreated sample in relation to pre-curing durations.

Characterisation of stabilized bricks

The introduction of the stabilizer, along with an extended curing period, leads to a reduction in volume shrinkage. (Fig. 2). The type of stabilizer influences the mechanical properties: volume shrinkage decreases by 0.69% per day for lime and 0.88% for straw, and rises to 0.96% per day for cement without curing, and 1.16% for cement with curing.

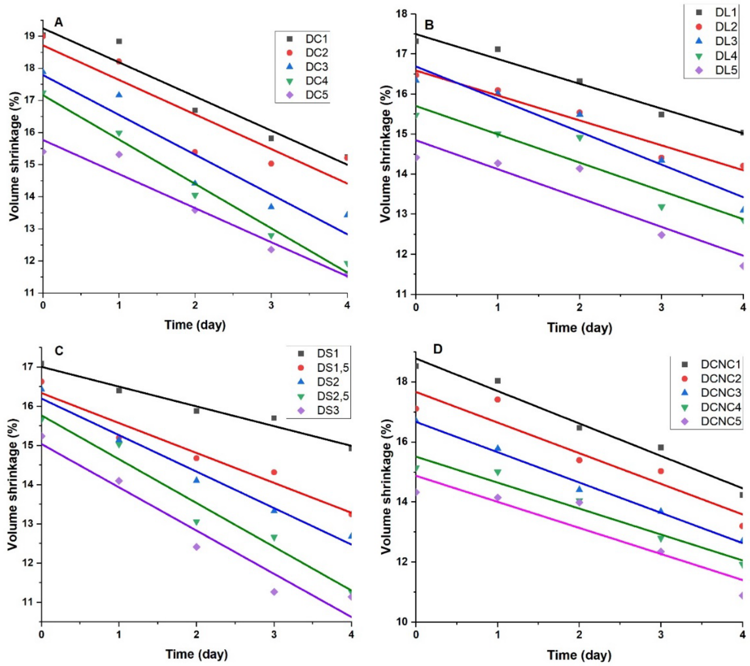

Fig. 2: Changes in volume shrinkage concerning curing duration and the type and amount of additives. A: portland cement (DC); B: lime (DL); C: straw (DS); D: cement without pre-cure.

Table 2: Relationship between changes in volume shrinkage and the curing time of the additives used to manufacture brick specimens.

Additives (%)	Cement		Lime		Straw		Cement (without pre-cure)	
	Slope	R^2	Slope	R^2	Slope	R^2	Slope	R^2
1	-1.06	0.93	-0.61	0.97	-0.50	0.97	-1.07	0.97
1.5	-	-	-	-	-0.76	0.94	-	-
2	-1.07	0.81	-0.62	0.95	-0.93	0.98	-1.01	0.89
2.5	-	-	-	-	-1.11	0.96	-	-
3	-1.23	0.89	-0.81	0.93	-1.10	0.94	-1.00	0.99
4	-1.38	0.98	-0.70	0.89	-	-	-0.86	0.95
5	-1.06	0.95	-0.72	0.85	-	-	-0.86	0.85
Average	-1.16		-0.69		-0.88		-0.96	

The changes in volume shrinkage as a function of the type of stabilizer, calculated from the slopes of the volume shrinkage curves, and their correlation coefficient R^2 are given in **Table 1**. Volumetric shrinkage followed the same trend for samples stabilized with lime (0.69% / day) and straw (0.88% / day). However, it is twice as high for cement (1.16% / day) than for cement without pre-cure (0.96%/day). Cement, unlike the other stabilizers, allows the grains of the material to clump together and improves workability with a long curing time. As a result, the volumetric shrinkage of these specimens is greatest.

The addition of 5% of cement decreases the volume shrinkage less than 15.5% after 4 days of pre-curing, which significantly decreases the occurrence of cracks. The composition of the clays employed affects how shrinkage develops [22,25], indeed, as demonstrated in our previous research, samples obtained from the remains of the wall of the Kasbah of Ait Benhaddou do not exhibit the presence of swelling clay[4]. The amount of shrinkage is significantly reduced by the addition of lime up to 5%, which lessens the likelihood of cracking [26]. Low R^2 values for the shrinkage caused by straw addition can be explained by large and quick water evaporation via capillary action from specimens stabilized by straw as opposed to diffusion through pores and cracks. By integrating natural fibers, earth plasters in straw bale constructions experience significantly less shrinkage [14].

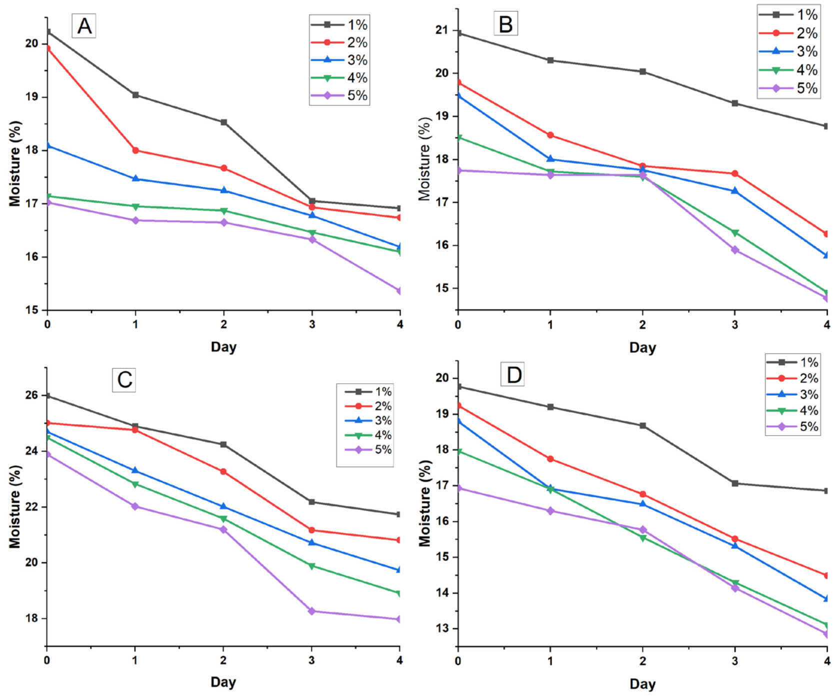

Fig. 3: Variations in moisture development for the specimens relative to curing duration and the type and amount of additives. A: lime (DL); B: cement (DC); C: straw (DS); D: cement without pre-cure (DCNC).

The moisture level diminishes with the duration of pre-curing and the proportion of the additive (Fig. 3). The decrease is more important for straw than for cement without precure and lime, although the evolution of this decrease is almost the same according to the quantity of the added admixture, the lowest decrease was observed for cement-stabilized samples. After 4 days of the pre-cure duration, the moisture decreased (Fig. 4) respectively with a slope of -2.27, -2.10, -2.29 and -1.43, depending on the amount of straw, cement without pre-cure, lime, and cement added.

Zeolite water, adsorbed water, water combined with aluminous silicate hydrates (C-A-H) or calcium silicates (C-S-H) are some of the sources of residual moisture in the paste. This reduction in moisture during the curing process may be caused by pozzolanic processes in addition to evaporation [27]. The low amount of residual moisture compared to straw with lime and cement, which are hydraulic binders, could be explained by the production of stable hydrates at 105°C. For the last reason, evaporative drying during curing may account for the drop in cement paste moisture.

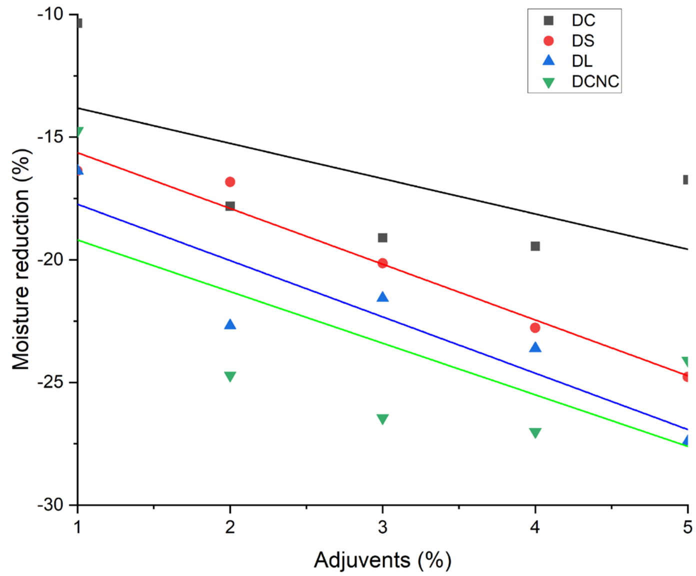

Fig. 4: Moisture reduction as a function of the quantity of additives for the specimens. A: DL : lime; DC : cement ; DS: straw; DC_{NC}: cement without pre-cure.

Fig. 5 illustrates the thermal conductivity results of pastes pre-cured for three days before shaping brick specimens (DL, DC, and DS), as well as those stabilized with cement without pre-curing (DCNC). The figure indicates that the incorporation of cement, regardless of curing, resulted in an elevated thermal conductivity with an increasing percentage of added cement. Conversely, the introduction of lime and wheat straw had an opposing effect.

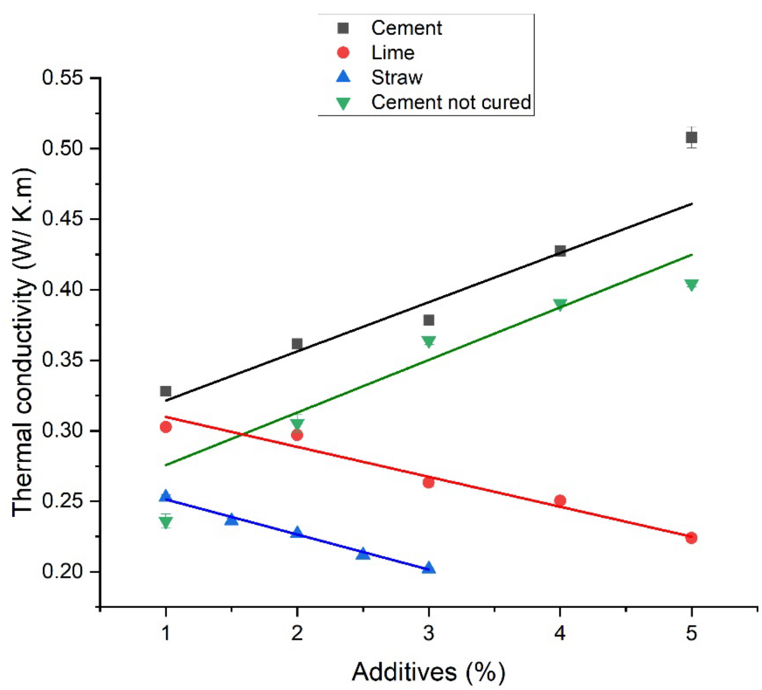

Fig. 5: Fluctuations in thermal conductivity based on the additions made to the specimens.

Table 3: R^2 and slope values derived from the changes in thermal conductivity curves concerning the amount of aggregate introduced into brick specimens.

	Slope ($W.K^{-1}.m^{-1}.\%^{-1}$)	R^2
Lime (DL)	-0.02123	0.98
Straw (DS)	-0.02487	0.99
Cement (DC)	0.03487	0.92
Cement without pre-cure (DC_{NC})	0.03728	0.85

The thermal conductivity variation is influenced by the type of added aggregate. The dimensions of the aggregate and the presence of admixture in the mortar structure contribute to the reduction in thermal conductivity, with larger sizes having a more pronounced impact. Indeed, introducing an aggregate to the debris diminishes thermal conductivity by reducing particle-to-particle contact. [22].

Regarding lime and straw, which had no impact on the compressive strength as already shown in our previous work [4], the conductivity experiences a linear decrease with the added material quantity at a consistent rate. The thermal conductivity of cement surpasses that of the reference sample as the added quantity increases, leading to the consolidation of the matrix. Samples stabilized with cement without pre-curing showed the same fluctuation profile, these samples' thermal conductivity values, however, are lower than those of the cement-stabilized and pre-cured specimens. The drop in the thermal conductivity value is likely caused by pores present in the samples that were not pre-cured.

Considerably reduced thermal conductivity values compared to those documented by Ouedraogo et al., [17] are compatible with the outcomes produced using cement and lime. Due to the insulating properties of the cellulose in the straw and its increased porosity, the addition of straw reduces thermal conductivity [28,29].

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Monitoring of stabilization-induced changes

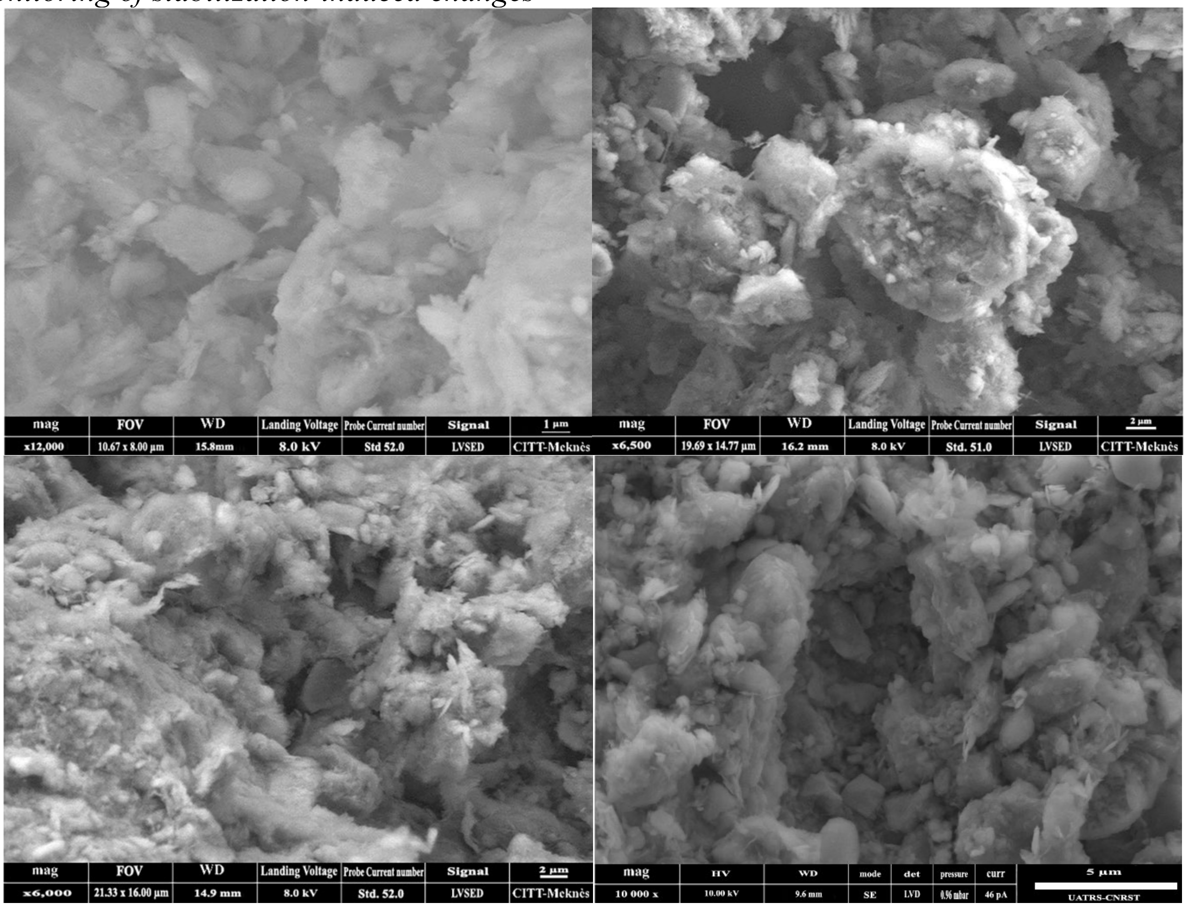

Fig. 6: SEM images of earthen material debris from the Ksar Ait Benhaddou A: DC_5 B: DL_1 C: DS_2 D: DC_{NC5}

Sample DC5 (Fig 6-A) displays a gel-like substance enveloping the grain surface when cement is added. This substance is believed to be calcium silicate hydrate (C-S-H) gel, formed through the hydration of the primary phases of portland cement, belite, and alite. The grains are closely bound to each other, eliminating visible separation, and the particles exhibit significant cohesion with pore sizes ranging from 0.2 to 0.5 µm.

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The straw-stabilised samples have similar mineralogical composition as raw debris sample (Fig.7) of our previous work [4]. The addition of straw reveals a fractured texture. The addition of 2-3% of straw accelerated the formation of cracks in silty clay soil [13].

Comparison of SEM images revealed that cement-stabilized samples DC_5 showed better cohesion and less porosity than DL_1, DS_2, and DC_{NC5}, which explains the enhanced mechanical strength [4]. and thermal results (Fig. 5).

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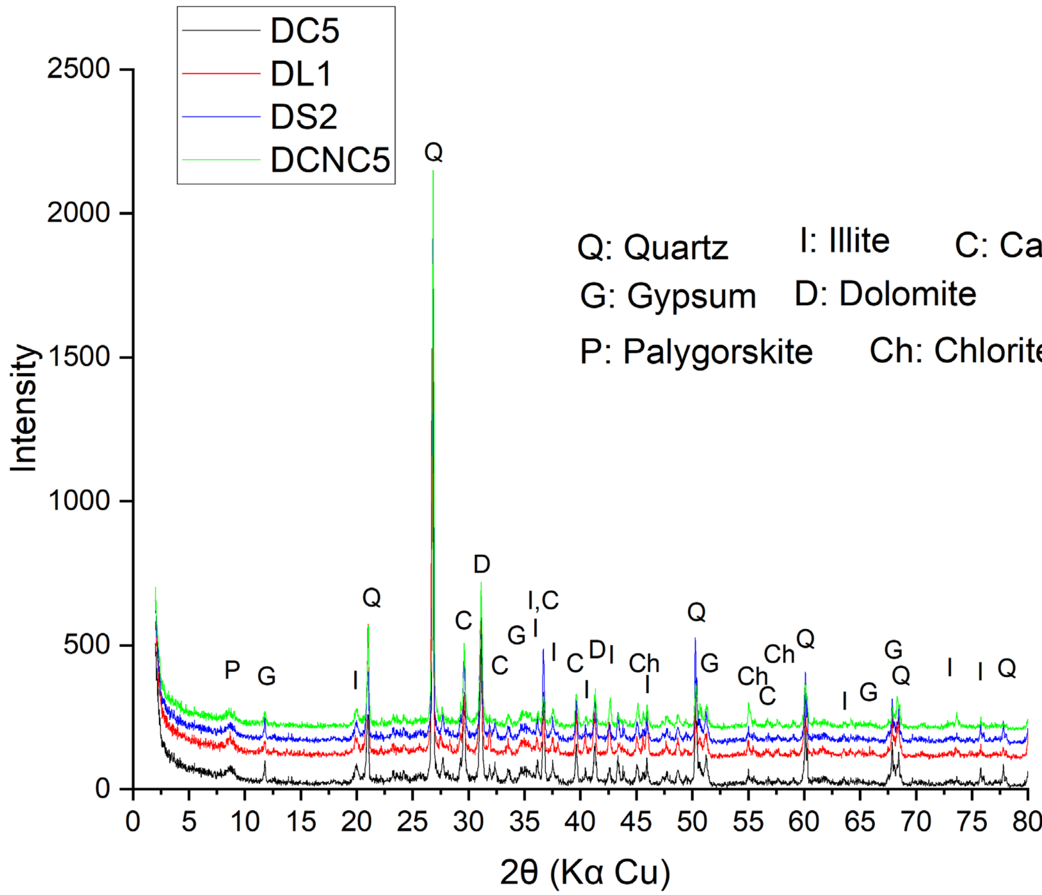

Fig. 7: XRD spectra of stabilized specimens

All stabilized specimens and the raw debris sample show similar mineralogical composition (Fig. 7), suggesting that the stabilisation mechanism is agglomeration of the sample grains around the different grains of hydraulic binders or fibers of straw used to stabilise them, as no new phases were created.

Conclusion

Samples from the Ait Benhaddou Kasbah wall debris were stabilized using portland cement, lime, and straw with the aim of improving their mechanical and potentially thermal properties and efficiency.

Volume shrinkage decreases with the addition of stabilizer and the duration of curing. The shrinkage kinetics is almost high for cement, with or without precure, compared to lime and wheat straw. Cement is a stronger hydraulic binder than lime, while the speed of wheat straw can be explained by significant evaporation at the beginning of the precure as well as the effect of water drainage through the straw channels.

Both the proportion of the additive and the pre-curing duration have an effect on the decrease of moisture content. The increase is more significant for straw than for cement without precure and lime. The least level of decrease was seen in cement-stabilized samples, even if the evolution of this decrease is almost the same for the other admixture.

The size of the binder in the structure of clayey specimens are the causes of the decrease in thermal conductivity; the larger the size, the more pronounced the loss. Indeed, the addition of a binder to the debris decreases the thermal conductivity because it decreases the contact between the particles

Cement, reducing brick porosity, enhances thermal conductivity, as evidenced by SEM images illustrating improved cohesion and, consequently, higher compressive strength. Conversely, lime and straw lead to a decrease in both thermal conductivity and compressive strength.

During setting, no mineralogical composition occurred suggesting that the stabilization mechanism is an agglomeration of the grains of the debris samples around the different stabilizers.

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