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Physico-Chemical and Mechanical Characteristics of Traditional Marrakech Lime

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Abstract. To tackle the problem of construction-related pollution, especially the greenhouse effects mainly caused by carbon dioxide emissions, governments have begun to encourage the use of traditional building materials and techniques characterized by their low carbon impact. Among these alternatives is Marrakech lime, produced using traditional processes. Historically, it has been used to produce a special mortar known as Tadlakt, an ancestral Amazigh skill used to waterproof parts in contact with water. In order to identify other ways of adding value to Marrakech lime, a literature search was launched. Physico-chemical and mechanical characterization was also carried out on samples of traditional lime concrete sold in the Moroccan market under the name "Jer de Gram". Morphological and mineralogical analyses were examined using scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD). The results showed that the lime examined is slightly hydraulic of varied forms, it is mainly composed of calcite, belite, calcium oxides, silicon, and carbons. Crushing tests show a low compressive strength of traditional lime concrete, not exceeding 1 MPa in the best cases, an unexpected result that contradicts the literature. The reason lies in the sandy nature of the aggregates and the traditional lime production process, which does not allow for effective quality control of the final product.

Introduction

Protecting the environment and preserving natural resources has become a major global concern. The world is increasingly aware of the ecological issues we face today, specifically CO_2 emissions and their impact on climate change. The construction sector has been identified as the most responsible for carbon dioxide emissions, due to cement, one of the basic building materials. The most recent studies on air pollution indicate that cement is behind an estimated 10% increase in CO_2 emissions (observed between 2015 and 2016) [1]. With this in mind, and intending to limit climate fluctuations, the European Union has shown global leadership, planning to reduce greenhouse gas emissions by 40% by 2030 and to cut energy consumption by 20% by 2020 and 50% by 2050, in addition to implementing action plans that encourage the use of natural resources as well as improving the energy efficiency of buildings [2].

From antiquity to the first decades of the 20th century, lime mortars played a crucial role in the history of architecture and engineering [3]. However, the knowledge of how to prepare and apply these mortars, which was passed down from parents to sons and from masters to apprentices, thus ensuring good durability and performance, as can be seen in surviving ancient buildings, has been abandoned, and this lack of artisanal knowledge has created difficulties for their practical use [4]. In this context, in-depth scientific studies have been carried out on the nature, quality, and proportions of components, such as the effect of environmental conditions, the evolution of carbonation and change in pore structure, the influence of mortar preparation and application process, and the impact of lime production conditions [4].

The methodological approach adopted in this article is mixed (qualitative and quantitative), combining an intensive bibliographical analysis of the scientific literature related to hydraulic lime, with experimental work in the laboratory, integrating both theory and empirical data.

In this paper, we will focus on the Marrakech traditional lime. First of all, a literature review was launched. Then, using scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD), morphological and mineralogical investigations were performed. Realizations were also applied to samples of the traditional lime concrete known as "Jer de Gram", sold in the Moroccan market, to determine the material's physico-chemical and mechanical properties.

Previous literature

Hydraulic lime

The use of lime has grown considerably in recent years. The "lime revival", a set of rules for good lime practice first developed in 1970, gained momentum after the conservation of the west front of Wells Cathedral under Robert and Eve Baker, and Crowland Abbey and Exeter Cathedral after 1975 [5]. Nevertheless, according to Copsey [5], this "revival" requires an uncompromising reassessment to improve the handling of hydraulic lime while respecting the main foundations of conservation. Referring to the latter, and despite scientific studies encouraging the use of hydraulic lime in this sense, Veiga [4], wondered why the employment of this material was still restricted. He identified three major factors: the need to acquire more scientific knowledge about local conditions and the heterogeneity of raw materials; the importance of the human factor, including the training of technicians and operators in the practical application of the material; and finally, the difficulty of managing the time required for applying and drying lime.

Hydraulic lime is extracted from the calcination of limestone and siliceous rock. During combustion at around 1200°C, silica and calcium oxide (C_aO) are combined to create calcium silicates. The decarbonization of $CaCO_3$ and the extinction of C_aO take place in the same way as for calcium lime. The result is a binder, called belite, composed of $C_a(OH)_2$ and C_2S (dicalcium silicates) [6]. The ratio (C_aO/S_iO_2) influences the lime's hydraulicity, which, in simple terms, affects the amount of belite present in the lime. Three different categories of hydraulic lime can be distinguished: NHL_2, $NHL_{3.5}$, and NHL_5, which are differentiated according to their minimum compressive strength after 28 days, as defined in European standard EN 459-1. NHL_2 has a strength of 2 MPa, $NHL_{3.5}$ has a strength of 3.5 MPa and NHL_5 has a value of 5 MPa. Consequently, the type of lime used in construction basically depends on the strength you need once it has had time to harden [6]. The hydraulic lime cycle is mentioned below in Figure 1:

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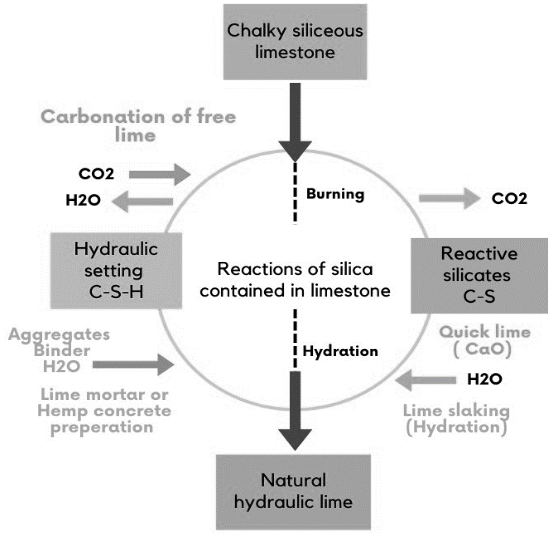

Figure 1: Hydraulic lime cycle [6]

Marrakech traditional lime

Lime was for long an essential conventional material in the construction of Moroccan structures. It's known for its wide range of uses, from building and rehabilitating architectural heritage to husbandry and crafts. For over eight centuries, Marrakech has been producing the lime plaster known as" Tadelakt", which boasts waterproofing and antibacterial rates, from lime made using traditional styles. The original occupants used this mortar admixture specifically to cover areas in contact with water. It consists substantially of lime, water, black cleaner, and several colorings [7].

The Tadelakt method almost disappeared from Morocco in the early 1970s, in favor of new pre-mixed ingredients that allowed for a faster application process. Renewed interest in traditional architecture, particularly in Marrakech, was a major factor in its return in the 1980s. Tadelakt has become a material appreciated all over the world for its waterproofing capabilities, durability, beauty, and variety of colors, as well as its increasingly appreciated ecological components [8]. Local architecture has always been based on this unique blend as shown in the picture bellow (Figure 2).

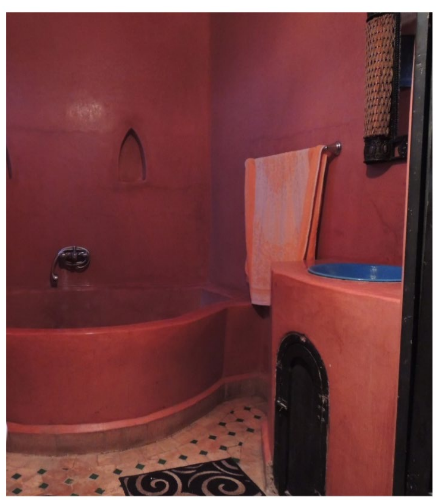

Figure 2: A Marrakech bathroom covered with Tadlakt [8]

Many studies have examined the remarkable properties of hydraulic lime, but few have focused on the elements and characteristics of traditional Marrakech lime. Accordingly, Amrani et al [8] studied the slaked lime of Marrakech and succeeded in extracting its chemical and mineralogical characteristics, which are presented in the tables below:

Table 1 : Mineralogical composition of Marrakech lime [8]

Mineralogical composition (%)	
Calcite	5.7 ± 0.1
Tricalcium aluminate	1.0 ± 0.3
Belite	13.3± 0.4
Katoite	5.7± 0.3
Portlandite	35.5 ± 0.6
Dolomite	2.1 ± 0.2
Quartz	9.5 ± 0.2
Periclase	0.8 ± 0.1
Palygorskite	1.0± 0.1
Brucite	1.0± 0.1
Amorphous and minor phases	24.0± 1.0
on dry sample (105°C)	

Table 2: Chemical composition of Marrakech lime [8]

Chemical composition (%)	
C_aO	65.77±0.5
S_iO_2	23.32±0.5
Al_2O_3	4.01±0.5
MgO	2.54±0.5
Fe_2O_3	1.97±0.5
SO_3	0.14±0.5
K_2O	1.08±0.5
Na_2O	0.59±0.5
TiO_2	0.35±0.5
SrO	0.06±0.5
MnO	0.04±0.5
P_2O_5	0.09±0.5
As_2O_3	0.07±0.5

Chemical characteristics of Marrakech lime

For this study, we used hydraulic lime, which is produced from marly limestone traditionally fired in the Marrakech region. A significant portion of the unfired marly limestone has been elaborated by this process, which was then combined with the siliciclastic fraction to form the aggregates of the mixture [7].

Scanning Electron Microscopy (SEM)

Microstructure analysis of Marrakech lime was carried out using a JEOL scanning electron microscope, to produce images of the sample surface as shown in Figure 3.

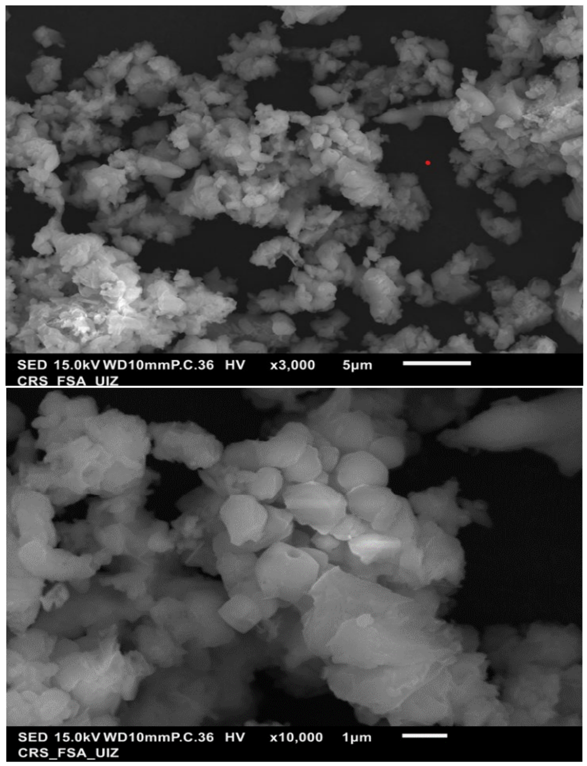

Figure 3: Scanning electron microscope (SEM) photos of Marrakech lime

These images show that Marrakech lime has a highly varied morphology, given its multiple mineralogical components. Minerals formed at low temperatures (around 1200°) are generally poorly crystallized, with small sizes of less than 5 µm [9]. The rhombohedral and orthorhombic crystals in the center of the SEM image represent calcite due either to the unfired portion of the parent rock (traditional firing does not allow decarbonization of the entire mixture) or to carbonation of the portlandite by moisture. The hexagonal layered structures visible in the SEM images represent portlandite, the main component of hydrated lime. Crystals in ovoid or globular form represent belite (C_2S). Further studies are needed to identify the other minority phases (Tricalcium aluminate, Katoite, Periclase, Palygorskite, and Brucite [8]), which appear as interstitial phases between the majority phases [10].

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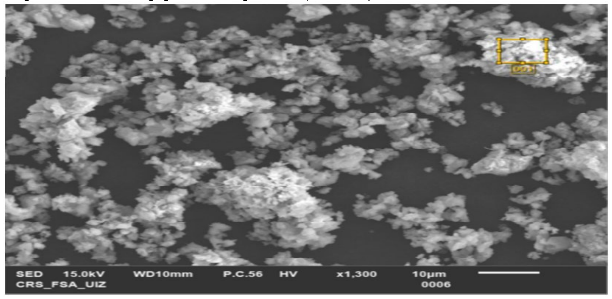
Energy-Dispersive X-ray Spectroscopy analysis (EDS)



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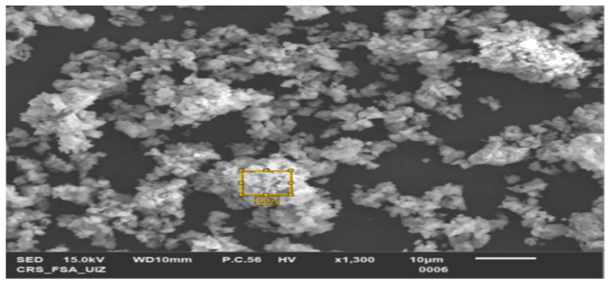

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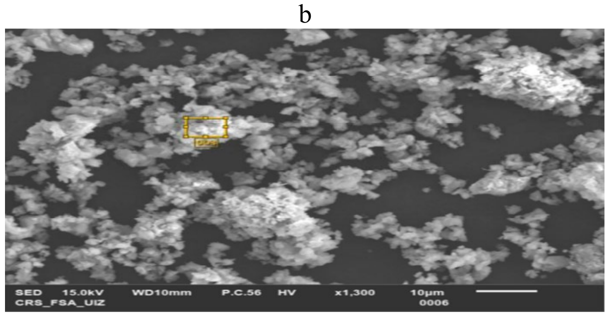

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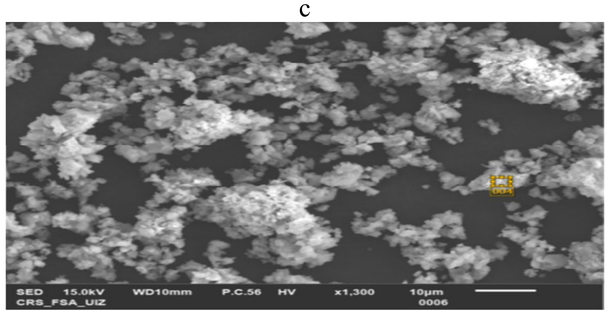

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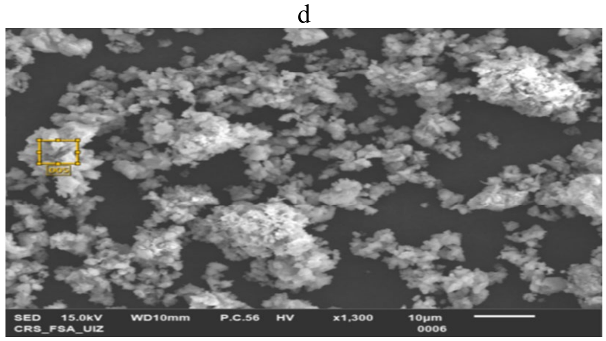

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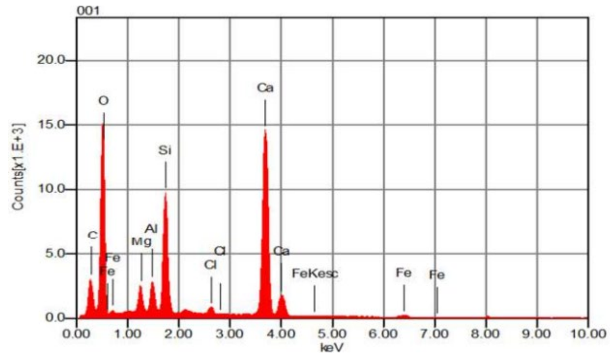
Figure 4: Areas scanned on the Marrakech lime sample concerned by EDS analysis (a: EDS001, b: EDS002, c: EDS003, d: EDS004, e: EDS005)

As shown in Figure 4, we used the energy dispersive spectrometry (EDS) technique to determine the phases present in the sample. Figure 5 below shows the EDS spectra of the scanned areas.



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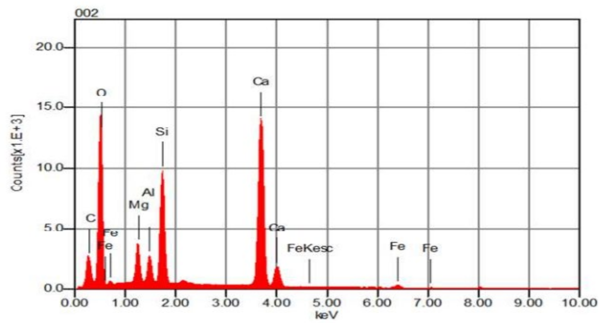

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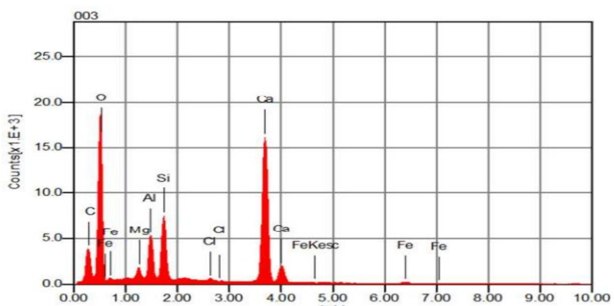

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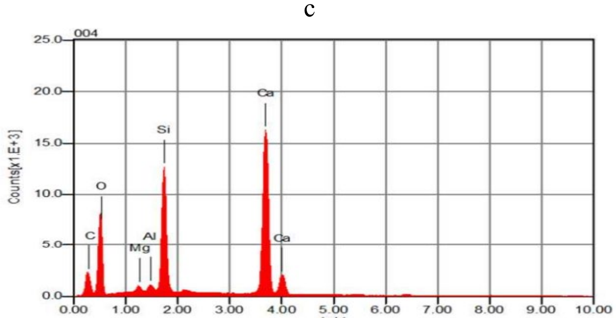

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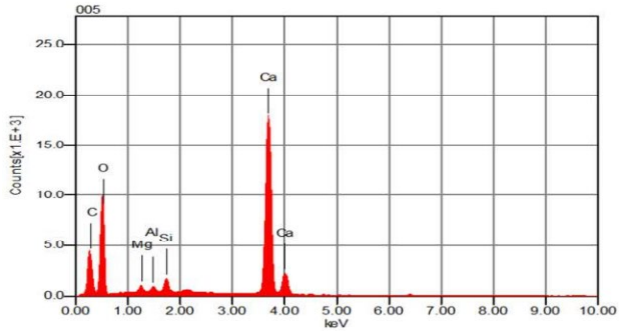

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Figure 5: Spectrum of Marrakech lime in swept areas (a: S001, b: S002, c: S003, d: S004, e: S005)

According to the EDS analysis carried out, Marrakech lime consists mainly of calcium, silicon, and carbon oxides, with a minority chemical components such as magnesium, aluminum dioxides, and traces of chlorine and iron dioxide. Hydrogen and oxygen, the fundamental structural elements of portlandite, were not found in the analysis. Table 3 summarizes the results obtained.

Table 3: The quantification of Marrakech lime

	EDS1 mass %	**EDS2 mass %**	**EDS3 mass %**	**EDS4 mass %**	**EDS5 mass %**
C	8.46	7.32	9.38	6.35	9.38
O					
CaO	57.77	56.28	59.91	62.93	83.13
S_iO_2	21.92	22.75	16.22	28.05	3.98
MgO	3.99	6.34	2.38	1.25	1.84
Al_2O_3	4.99	4.79	9.97	1.42	1.67
CI	0.94		0.53		
FeO		2.53			

X-ray diffraction analysis of Marralech lime

XRD analysis of the material was conducted using a Bruker D8 Eco X-ray diffractometer from the Agadir Faculty of Science. This diffractometer is connected to a computer by EVA operating software. It was used to record all the diffraction lines, identifying the different phases based on peak intensity analysis. Each mineral is characterized by these peaks. The secondary peaks are compared if two minerals have very similar first-order peaks. For XRD testing, CuKa radiation having a wavelength of l.54 A at a voltage of 30 KV, a scan speed of 3/min, and a current of 30 mA, was used.

Figure 6 shows the XRD diagram for Marrakech lime.

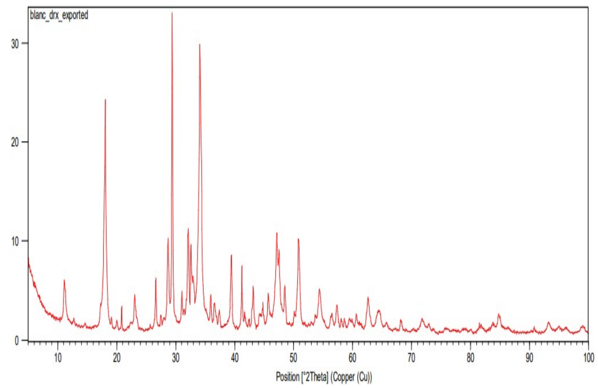

Figure 6: X-ray diffraction pattern of Marrakech slaked lime powder (intensity as a function of peak positions)

The mineral distribution was extracted from the analysis of emission spectra according to the norms indicated by Brindly and Brown [11] using X'pert High Score software. Consequently, the sample consists of the majority phases of portlandite, calcite, quartz, and belite. As the study performed failed to identify the minority phases, further investigations are recommended.

Physico-mechanical characteristics of Marrakech lime

Physical properties

In order to derive the physical properties of the material in question, a particle size analysis was carried out for the fraction of soil larger than 80 µm, accompanied by a sedimentation test for fine particles following French standard NF P94-056, NF P94-057[12], [13]. Figure 7 shows the weight distribution of grain sizes in the lime used.

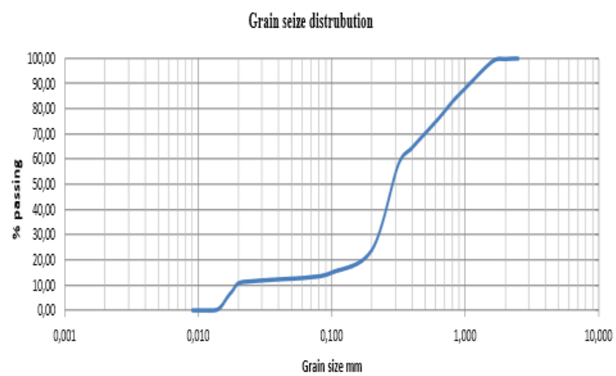

Figure 7: Diagram showing the weight distribution of grain sizes in the lime used.

It has been observed that 24% of the particles have a diameter of less than 200 µm by weight, and this fraction constitutes the binder phase of the mix [8]. Thus, the mixture is made up of three parts binder to three parts aggregate by weight.

Mechanical properties

- Sample preparation

First, we prepared the mixture of Marrakech hydraulic lime and water, adopting a Water/Lime ratio equal to 0.60 [14]. We then proceeded to fill the cylindrical test tubes, tightening them manually with a pricking bar. To get rid of air bubbles, each layer of concrete was struck 25 times. After 24 hours, we moved on to the demolding phase, keeping the specimens for 90 days under controlled laboratory conditions (T=22±3 and Rh= 65% ± 5).

- Testing equipment

To better characterize the material in its hardened state, we opted for a uniaxial compression test to derive ductility parameters, following French standards NF P94-420 and NF P94-425[15],[16]. The experimental setup (Figure 8) comprises a press with a maximum load capacity of 110 KN and a displacement sensor with an accuracy of ±0.05 mm. A displacement speed of 0.1 mm/min was selected for the test. The machine applies a progressively increasing compression force until the sample yields. It is connected to a computer that processes the results obtained and plots the stress-strain curve (Figure 9), facilitating the study of the mechanical behavior of the material in question. All the work was carried out in the laboratory of Agadir National School of Applied Sciences (NSAS).

Figure 8: Picture of the compression machine taken in the NSAS laboratory

- Results and discussion

To evaluate the resistance of Marrakech lime concrete, we were able to determine the ultimate stresses and maximum strains shown in Table 4 from the stress-strain diagram illustrated below:

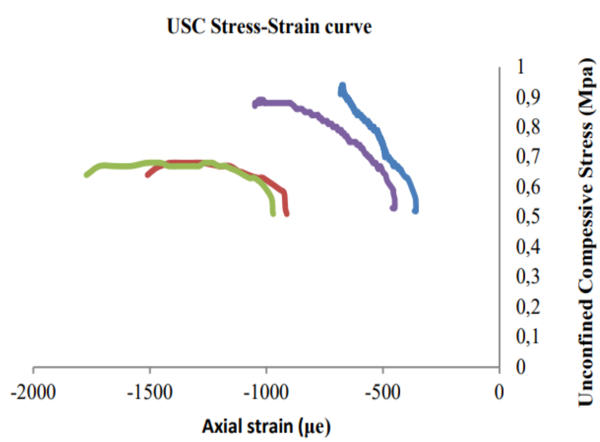

Figure 9: Stress-strain diagram generated by the computer for four specimens

Table 4: Stress-strain results for the four samples generated by the computer

	ρ g/cm^3	Ultim. Strength σ_u Mpa	Fracture Strength σ_f MPa	Strain at maximum stress ε_u µe	Ultimate strain ε_f µe
Specimen 1	1.217	0.93	0.88	-671	-681
Specimen 2	1.143	0.89	0.82	-1017	-1033
Specimen 3	1.110	0.68	0.64	-1129.6	-1798.8
Specimen 4	1.108	0.68	0.6	-1278.8	-1516.6
AVG	1.144	0.795	0.74	-1024.1	-1257.35

The ultimate stresses obtained vary between 0.68 and 0.93 MPa, low values due mainly to the sandy nature of the aggregates, as well as to the traditional hydraulic lime production procedure, which does not effectively control the quality of the final product. Maximum deformations range from 681 to 1798.8 µe. These rather low values correspond well to the low values for stress at break. Given that the device was unable to record deformations relative to stresses under 0.5MPa as shown in Figure 9, we cannot conclude the elastic characteristics of the hydraulic lime concrete used.

Conclusion and recommendations:

This article provides a concise and accurate review of the literature on hydraulic lime, its characteristics, field of use, and utility. It's a rich material, used since antiquity, with ecological and architectural benefits.

Analyses carried out on samples from the Marrakech region have demonstrated the major constituents of this material. It consists mainly of portlandite, calcite, quartz and belite. Calcite was found to have a high percentage due to the presence of unfired marl, in addition to the phenomenon of carbonation due to the CO_2 present in the air. The iron, aluminum, and magnesium oxides detected by EDS analysis are linked to the presence of Katoite, Dolomite, Periclase, Palygoskite, and Brucite minerals, minority phases characterizing Marrakech lime [8].

Marrakech lime was also tested from a mechanical point of view, by assessing its strength in the hardened state, opting for a uniaxial compression test. It was confirmed that lime concrete is not resistant to compression, given the low values obtained, which did not exceed 1MPa. This opens up a wide avenue for researchers to consider ways of strengthening this useful material and contributing to its conservation.

Recommended ideas for future studies include:

- Improve lime performance by incorporating reinforcing fibers.
- Set up units for the production of traditional hydraulic lime in Morocco.
- Draw up a guide to the use of lime to encourage its use in construction.
- Implement reliable methods for reformulating lime concretes.
- Apply Carbon cure technology to improve the performance of lime concretes.

References

[1] A. Aziz *et al.*, « Effect of slaked lime on the geopolymers synthesis of natural pozzolan from Moroccan Middle Atlas », *Journal of the Australian Ceramic Society*, vol. 56, p. 67, mai 2019 . https://doi.org/10.1007/s41779-019-00361-3

[2] B. Abu-Jdayil, A.-H. Mourad, W. Hittini, M. Hassan, et S. Hameedi, « Traditional, state-of-the-art and renewable thermal building insulation materials: An overview », *Construction and Building Materials*, vol. 214, p. 709-735, juill. 2019 . https://doi.org/10.1016/j.conbuildmat.2019.04.102

[3] M. del M. Barbero-Barrera, L. Maldonado-Ramos, K. Van Balen, A. García-Santos, et F. J. Neila-González, « Lime render layers: An overview of their properties », *Journal of Cultural Heritage*, vol. 15, nº 3, p. 326-330, mai 2014 . https://doi.org/10.1016/j.culher.2013.07.004

[4] R. Veiga, « Air lime mortars: What else do we need to know to apply them in conservation and rehabilitation interventions? A review », *Construction and Building Materials*, vol. 157, p. 132-140, déc. 2017 . https://doi.org/10.1016/j.conbuildmat.2017.09.080

[5] N. D. Copsey, « A CRITICAL REVIEW OF HISTORIC LITERATURE CONCERNING TRADITIONAL LIME AND EARTH-LIME MORTARS ».

[6] M. Chabannes, E. Garcia-Diaz, L. Clerc, J.-C. Bénézet, et F. Becquart, *Lime Hemp and Rice Husk-Based Concretes for Building Envelopes*. in SpringerBriefs in Molecular Science. Cham: Springer International Publishing, 2018. doi: 10.1007/978-3-319-67660-9

[7] A. Belabid, H. Elminor, et H. Akhzouz, « Study of parameters influencing the setting of hydraulic lime concrete: an overview », vol. 14, nº 3, 2023.

[8] A. E. Amrani *et al.*, « From the stone to the lime for Tadelakt: Marrakesh traditional plaster », 2018.

[9] « W. Yanmou, D. Junan, and S. Muzhen, "Sub theme 1.3," in ´ Proceedings of the 8th International Congress on the Chemistry of Cement (ICCC '86), vol. 2, pp. 363–371, Rio de Janeiro, Brazil, 1986. »

[10] I. Maki, « Morphology of the so-called prismatic phase in Portland cement clinker », *Cement and Concrete Research*, vol. 4, nº 1, p. 87-97, janv. 1974 . https://doi.org/10.1016/0008-8846(74)90068-4

[11] G. Brown, *Crystal Structures of Clay Minerals and their X-Ray Identification*. The Mineralogical Society of Great Britain and Ireland, 1982.

[12] « (NF P94-056, 1996) Standard AFNOR: NF P94-056, reconnaissance et essais – Analyse granulométrique - Méthode par tamisage à sec après lavage, 1996. »

[13] « (NF P94-057, 1992) Standard AFNOR: NF P94-057, Sols : reconnaissance et essais - Analyse granulométrique des sols - Méthode par sédimentation, 1992. »

[14] M. M. Barbero-Barrera, N. Flores Medina, et C. Guardia-Martín, « Influence of the addition of waste graphite powder on the physical and microstructural performance of hydraulic lime pastes », *Construction and Building Materials*, vol. 149, p. 599-611, sept. 2017 . https://doi.org/10.1016/j.conbuildmat.2017.05.156

[15] « (NF P94-420, 2000) Standard AFNOR: NF P94-420, Détermination de la résistance à la compression uniaxiale, 2000. »

[16] « (NF P94-425, 2002) Standard AFNOR: NF P94-425, Méthodes d'essai pour roches - Détermination du module d'Young et du coefficient de Poisson, 2002. »