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Mechanical evolution of lime mortars during the carbonation process

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Abstract. Lime mortar is one of the most ancient and durable building materials. It is characterized by a slow carbonation during which $Ca(OH)_2$ reacts with CO_2 present in air and forms calcite, giving rise to a stronger and more compact material. This process takes place from the surface to the interior of the material and it is strongly affected by the reaction conditions. The aim of this study is to quantify the increase in strength and elasticity of different lime mortars according to their carbonation degree. For that, six types of mortars were elaborated, with different lime/aggregate proportions and aggregate mineralogy and grading. Mineralogical and textural studies were carried out to follow the carbonation process. Each mortar was tested in a uniaxial compression press after 15, 28, 60 days from the elaboration. In order to differentiate the mechanical behaviour of the external and internal parts of the mortars, two micro-samples (10×10×10 mm) were obtained from the first 10 mm and from the core of each prism. Results show that an increase in strength and especially in the elastic modulus is associated to the carbonation process, but it is different depending on the composition and compactness of the mortars.

Introduction

The mechanical resistance of lime mortars is strictly related with their carbonation degree, which represents the quantity of portlandite ($Ca(OH)_2$) transformed into calcite ($CaCO_3$). This transformation ($Ca(OH)_2 + CO_2 \rightarrow CaCO_3$) depends on CO_2 permeability, temperature and humidity, as well as on the textural characteristics of the mortar [1-3]. In turns, the microstructure of mortars strongly depends on the elaboration and curing conditions, so that a correct choice of components and mixing proportions is essential to ensure good final performances. The aim of this work was to link the microstructure of six different types of mortars with their mechanical properties during early carbonation ages (7, 15 and 60 days). For that, micro-compressive essays were performed on micro-samples of each mortar and their behaviour was related to the textural properties studied at the same time intervals.

Materials and methods

Six types of mortars were prepared with a calcitic dry hydrated lime (CL90-S, UNE-EN 459-1) whose components and mixing proportions are shown in Table 1. Two types of aggregates were used, a calcitic one with continuous (CA, $0.063<\varphi<1.5$ mm) and dicontinuous grading (CDA, $0.1<\varphi<0.8$ mm), and a siliceous one, with a discontinuous grading (SA, $0.1<\varphi<0.5$ mm). After their preparation, mortars were left during 7 days in normalized steel moulds (4×4×12 cm) at HR = 60±5% and T = 20±5°C and after desmoulding, they were cutted in prisms of 4×4×5 cm. Two micro-samples (10×10×10 mm) were obtained from the first 10 mm and from the core of each mortar prism. The observation of the mortars microstructure (morphology, cohesion, porosity) was carried out by means of *optical microscopy (OM)* (Olympus BX-60) equipped with digital microphotography camera (Olympus DP10). *X-ray diffraction* analyses *(XRD)* were performed in order to identify and quantify the mineralogical phases, by means of a Philips PW-1710 diffractometer. The interpretation of diffractograms was carried out using “X-Powder” software package [4]. Total open porosity (P_{tot}, %) and pores size distribution (PSD) were determined using a Micrometics Autopore III 9410 porosimeter (*MIP analysis*).

In order to study the mechanical characteristics of each type of mortar and their evolution during the carbonation process, the whole micro-samples (above described) were tested in an *uniaxial compression press*. This press (Instron 4411) can achieve a maximum load of 5000 kN and a constant load velocity of 0.1 MPa/s was selected for this test. The stress-strain curve was recorded and strength and Young modulus were calculated for each sample.

Results and discussion

The main difference between the textures of each mortar is based on the porosity percentage (Table 1) and predominant pore types.

Table 1: *Sketch of the six types of mortars studied in this work. Several information is showed from each one: aggregate used (CA: calcite aggregate with continuous grading; SA: siliceous aggregate with discontinuous grading; CDA: calcite aggregate with discontinuous grading); binder/sand ratio (B/S, by weight); the amount of water employed in the mass (W/T, expressed in % of the total mass); total porosity (P_{tot}) at 7, 15 and 60 days after mortar preparation; mechanical properties (strength and elastic modulus) of micro-samples obtained from the inner and external part of prisms (Internal and External, respectively).*

Mortar name	Time [days]	Aggregate	B/S	W/T [%]	P_{tot} [%]	Strength [MPa]		Young modulus [MPa]	
						Ext.	Int.	Ext.	Int.
CC1:2	7	CA	1/2	29.5	38.22	-	-	-	-
	15				40.96	1.12	0.83	199.32	182.77
	28				-	1.53	1.08	514.29	269.75
	60				40.84	1.67	0.89	815.01	-
CC1:3	7	CA	1/3	31.3	39.87	-	-	-	-
	15				35.18	0.82	1.01	32.45	251.46
	28				-	0.97	0.98	330.41	218.09
	60				35.03	1.13	0.85	501.23	192.91
CC1:4	7	CA	1/4	24.0	33.64	-	-	-	-
	15				34.50	1.16	1.035	173.91	179.98
	28				-	1.04	1.05	239.66	127.93
	60				34.30	1.56	0.94	623.71	147.59
CC1:6	7	CA	1/6	20.0	29.51	-	-	-	-
	15				30.04	0.93	1.10	296.43	154.86
	28				-	1.15	0.91	378.38	112.07
	60				32.12	1.34	1.11	568.82	178.52
CD1:3	7	CDA	1/3	26.5	35.41	-	-	-	´-
	15				34.50	1.12	1.41	343.14	778.85
	28				-	1.33	1.18	680.98	310.32
	60				36.03	1.60	-	803.80	-
CS1:3	7	SA	1/3	27.0	36.77	-	-	-	-
	15				37.82	0.62	0.68	96.67	99.05
	28				-	0.99	0.93	108.49	105.10
	60				34.25	1.05	0.95	373.36	192.10

Porosity appears in different forms: 1) as shrinkage fissures (present in the matrix and in the interfacial zone (ITZ: surface between the matrix and the grains)); 2) as rounded and big pores typical of water evaporation; 3) as interparticle porosity, especially along the exfoliation planes of calcite.

Mortars with the same binder/sand (B/S) ratio (CC1:3, CS1:3 and CD1:3) present similar total porosity value (Table 1). However, an important difference between these three mortar types was observed in CS1:3. These three varieties present a good cohesion in the matrix. However, the ITZ in the mortar prepared with siliceous aggregates (CS1:3) shows frequently a dishomogeneous contact (shrinkage fissuration processes).

The highest value of P_{tot} was found in CC1:2, because of the high shrinkage caused by the big amount of lime. By means of OM, shrinkage fissures and big pores (Fig. 1) were observed in CC1:2 and CC1:3, although in lower quantity and smaller size in the latter.

In CS1:3, CC1:2, and partially in CC1:4, rounded and big pores (r > 100µm) are observed, due to a rapid water evaporation. CC1:6 mortars present the lowest porosity, because of the lowest B/S ratio and the least amount of water added for their preparation.

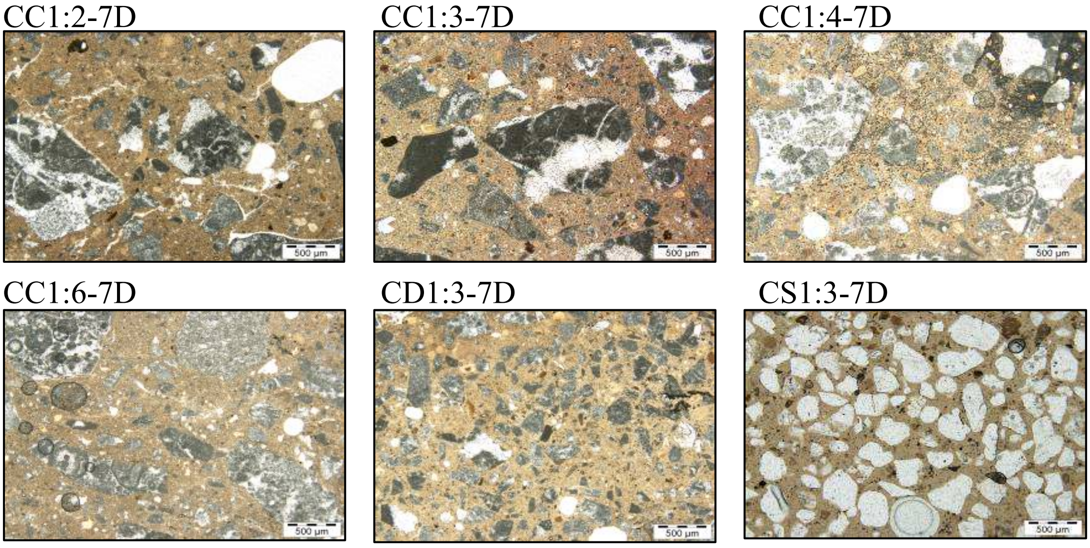
CC1:2-7D CC1:3-7D CC1:4-7D
CC1:6-7D CD1:3-7D CS1:3-7D

Figure 1. OM images of all the mortars at 7 days since the elaboration. Images were taken at same magnification with plane polarized Nicols.

The carbonation curves (Fig.2) show that mortars prepared using calcitic aggregate with continuous grading are the most carbonated. Carbonation degree of mortars was estimated considering the decrease in portlandite content, determined by means of X-ray diffraction quantitative analysis. CC1:3 is the most carbonated mortar almost at every time intervals, while its analogous containing siliceous aggregate (CS1:3) presents at the end of the study the least quantity of portlandite transformed into calcite.

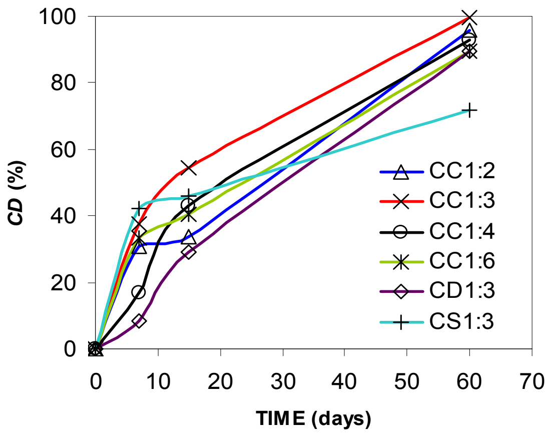

Figure 2. Representation of the carbonation trend in mortar types. CD (%) represents the carbonation degree, estimated considering the decrease in portlandite content, determined by means of X-ray diffraction quantitative analysis.

In Table 1 it is possible to observe the mechanical properties (strength and elastic modulus) of each mortar type, as well as its evolution during the carbonation process. Some interesting results are obtained from these data. External micro-samples (from the first 10 mm of the prism) have always higher strength and Young modulus values at 60 days than at 15 days. Moreover, the values at 60 days obtained in the external micro-samples are always higher than those measured on the internal ones. This is due to the fact that the carbonation process takes place from the surface to the interior of the mortar prisms. Consequently, the external micro-samples will contain progressively more calcite in their matrix from 7 to 60 days. The micro-samples obtained in the prism core, however, contain similar calcite content in every step (there is not strength increase).

In addition to the carbonation process, changes in the porous system of the mortar are caused by water evaporation. In general, mortars present a very open structure and high porosity values (between 30 and 40%). These textures involve a very low strength (lower than 2% in every case). Porosity changes during the curing process do not involve significant variations in the mechanical properties of mortars with calcite aggregate. However, mortar CS1:3 (with siliceous aggregate) present both the lowest strength and elastic modulus. This mortar presents shrinkage fissures in the grain-matrix contact surface, making easy the fissure nucleation and its propagation. Moreover, the lowest carbonation degree at 60 days corresponds to this mortar.

Finally, strength and Young modulus of CC1:2, CC1:3, CC1:4 and CC1:6 are defined between the same limits during the whole process. However, CD1:3 presents values of strength and Young modulus a bit higher with respect to the compositionally similar mortars. Although the only difference between CD1:3 and CC1:3 stays in the grading of the aggregate, the higher value of strength can not be attributed to this factor, because continuous grading always produces more resistant mortars. The strength of CC1:3 is lower because the porosity of this mortar is much higher than that of CD1:3. On the other hand, the lowest value of porosity, obtained in CC1:6, does not correspond to the highest resistance. In this case the reason stays in the low amount of lime of this mortar, which produces less compact and resistant matrix.

Conclusions

This study confirms that the carbonation process affects positively the mechanical properties of mortars, by producing an increase of strength of these materials, always higher in the external zone which is the most carbonated one. It has been found that the use of calcareous aggregates give place to more carbonated and resistant calcite lime mortars.

Among the six types of mortars studied in this work, CC1:3 showed the highest carbonation degree and one of the highest values of resistance and elastic modulus.

On the other hand, we found that the use of a siliceous aggregate gives place to weaker mortars, in spite of the higher hardness of quartz compared to calcite. The reason of that stays in the insufficient cohesion between the grains of quartz and the matrix.

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Acknowledgments

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