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Effect of maturation time on the fresh and hardened properties of an air lime mortar

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ABSTRACT

The restoration and maintenance of old renders is one of the key aspects of correct rehabilitation practice. The ideal course of action is to replace the damaged material by a material with compatible characteristics. This work aims to analyze the effect of the maturation process on hardened state characteristics of hydrated powder lime mortars. The rheological characterization shows an air lime mortar thickening behaviour with the length of the test. The different mixes were subjected to a maturation process consisting on keeping them in the fresh state, covered with water, isolated from CO_2, during seven days. The specimens and applications were prepared both with the non-matured and the matured mortars. Maturation seems to influence the hardened state characteristics causing a decrease in the capillary values, and an increase on the mechanical strength, which are more evident for mortars with higher binder contents.

1. Introduction

According to current guidelines, building on virgin soil should decrease and the construction activity must evolve towards the conservation and restoration of degraded buildings, since they also represent a common heritage [1]. The restoration and maintenance of old renders is one of the key aspects of correct rehabilitation practice. The ideal course of action is to replace the damaged material by a material with compatible characteristics [2,3]. In practice, this can be quite complicated when traditional materials such as air lime mortars are involved, because knowledge of their properties and their preparation is not complete, particularly, the knowledge of their rheological properties and relationship with the characteristics in hardened state. In terms of flow properties, mortars typically behave as a Bingham fluid, being characterized by a yield stress and a plastic viscosity [4,5]. If the effect of mortar constitution on its workability is known, quality could be controlled at the time of production rather than waiting for the results of tests on the hardened product, which imply a long waiting process, involving a minimum time of 28 days but an average time of 90 days for reliable results [4–6].

A non-hydraulic lime mortar sets by a simple chemical reaction between lime (calcium hydroxide) and airborne carbon dioxide. However, the presence of water is essential not only for the material's workability but also for carbonation to occur. Thus, in the preparation of a mortar, the solid components are mixed with kneading water and the amount of kneading water is, as for cement mortars, a very important parameter with high influence on this material's final properties [7].

According to Feldman and Sereda [8], the water incorporated in hydraulic mortars mixing will eventually exist in four different forms or bonding states: (1) chemically bound water or hydrate water, (2) interlayer water, (3) adsorbed water, and (4) capillary water or free water.

Chemically bound water is the water consumed for hydration and needed for the formation of hydration products, including calcium–silicate–hydrate (C–S–H). Interlayer water is composed by the water molecules existing between the layers of C–S–H, and is strongly held by a hydrogen bond. This interlayer water is lost only on strong drying. Adsorbed water molecules are physically adsorbed onto the surface of the hydrated solids. A major portion of the adsorbed water can be lost by drying down to 30% of relative humidity (RH). The loss of this adsorbed water is the main responsible for the shrinkage of mortars on drying [9]. Capillary water is present in voids or channels larger than about 50 Å and up to 1 to 10 µm. This water is free from the influence of the attractive forces exerted by the solid particles surface. Its removal does not result in significant shrinkage of the material but its withdrawal creates capillary pores.

As hydrated powder lime mortars do not have calcium silicate hydrates in their composition, then it is possible to consider that, in this type of mortars, there are only two forms of water: the capillary water and the adsorbed water on the solid particles surface.

Following an initial hardening through drying, air lime mortars set entirely through carbonation. The presence of water is essential for carbonation to occur, since carbonation takes place when CO_2 dissolves in water and reacts with dissolved calcium hydroxide [10,11]. Lawrence et al. [10] consider that there are five stages involved in carbonation:

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(1) diffusion of gaseous CO_2 through the mortar pores; (2) dissolution of CO_2 in pore water; (3) dissolution of Portlandite ($Ca(OH)_2$) in pore water; (4) solution reaction between $Ca(OH)_2$ and CO_2 and, (5) precipitation of solid $CaCO_3$. Van Balen [12,13] had already pointed out that the carbonation process is controlled by two mechanisms:

(i) Carbon dioxide diffusion from the air through the capillary pores up to the reaction front: the diffusivity of gaseous carbon dioxide in capillary pores depends on the openness of the porous structure and on the presence of water in the pores. It was estimated that the diffusion coefficient of CO_2 would drop 100 times when the equilibrium water content of the mortar reached 100% RH. In the initial hardening through drying phase, diffusion resistance will depend on the drying rate of the mortar.

(ii) Reaction of the diffused carbon dioxide with $Ca(OH)_2$: the controlling factor may be the dissolution of portlandite at the water adsorbed surface. The highest reaction rate is proportional to the lime specific surface.

Based on the work of Van Balen [12,13], it seems that the highest rate of carbonation is achieved when there is a minimum of water in capillary pores. However, the presence of adsorbed water in the lime particles surface, is needed in order to promote carbonation. According to this author, the internal relative humidity must be higher than 30% but without reaching saturation level at which CO_2 diffusion is hindered again.

During the first half of the twentieth century, traditional knowledge of building with lime-based mortars (lime putty or air lime powder) started to fade away with the advent of cement based materials. For conservation and rehabilitation purposes, it is of capital importance to recover the expertise involved in making these mortars. Maturation of lime was one of the techniques used to improve mortar properties. Cazalla et al. [19] and Atzeni et al. [20] presented studies on aging (maturation) of lime putty. They have observed the effect of aging in the rheological properties, microstructure and carbonation. However, lime putty and air lime present a different behaviour with water. While lime putty, when placed in contact with water, reacts chemically with it, creating a process of nucleation and growth of portlandite crystals, in the case of the air lime, portlandite crystals are already formed and there is no chemical reaction with water.

Hence, this work aims to analyze the effect of the maturation process of air lime-based mortars (hydrated powder lime mortars) on the fresh behaviour and also on the hardened product properties such as strength and capillary absorption. For all compositions used to characterize the hardened state, mixes were prepared and subjected to a maturation process. This process consists of leaving the fresh mortar mixes in a recipient, covered with water and thus isolated from CO_2 for seven days. The amount of covering water (previously measured) was taken out before the preparation of the specimens or the application of the mortar for characterization tests. Both matured and non-matured mortars were tested.

2. Experimental

2.1. Materials

Mortars with different binder: aggregate volume ratios (1:1, 1:2 and 1:3) were prepared (Table 1) with air lime (hydrated powder lime) as binder, with a purity higher than 99% ($CaOH_2$) and a density of 0.8 kg/dm^3 and, siliceous sand as the aggregate, with controlled granulometric distribution and a density of 1.55 kg/dm^3 (Fig. 1).

The hydrated powder lime mortar maturation process consists of mixing with water the binder and aggregate in the chosen proportions and then isolating this mortar from atmospheric CO_2 remaining as such for seven days and only then they were used to prepare samples for fresh and hardened state characterization.

Table 1
Compositions of air lime-based mortars.

Designation	Binder/aggregate volume ratio	Observations
CA1	1:1	No maturation
CA1M	1:1	7 days of maturation time
CA2	1:2	No maturation
CA2M	1:2	7 days of maturation time
CA3	1:3	No maturation
CA3M	1:3	7 days of maturation time

The kneading water content in the formulations that were used for the hardened state characterization was kept constant at 19% of the total weight of dry mortar. The water content of the samples prepared for the characterization of rheological behaviour was higher (around 30 to 35%) due to working limitations of the used rheometer.

2.2. Rheological characterization

For the characterization of rheological behaviour a specific rheometer for mortars (Viskomat NT) was used. This rheometer measures torque as a function of rotation speed for different speed–time profiles that can be set up. The characteristic Bingham fluid relation of torque (T) with rotation speed (N) is $T = g + hN$, where g (N mm) and h (N mm min) are coefficients related with yield stress and plastic viscosity, respectively [14].

In order to prepare samples for the rheometer the following mixing procedure was involved: (i) addition of the required water volume to the pre-mixed air lime-based mortar powder (ii) 30 s mixing at low rotation speed (~60 rpm), (iii) pause during 45 s to gather the mortar into the centre of the bowl, (iv) further mixing during 60 s at the same rotation speed and (v) resting during 10 min before starting the rheometer test.

It should also be stated that torque measurement in this kind of rheometer is limited to a maximum value of 300 N mm, above which the instrument shuts down automatically. This limits the minimum water content in the tested mortars inside a certain range, as stated in the previous section. Rheological characterization was especially performed on the 1:1 ratio mortars since in that formulation the impact of the maturation effect due to the higher lime content present in this formulation is more evident.

2.3. Hardened state characterization

Samples of matured and non-matured hydrated powder lime mortars were also prepared for hardened state characterization at laboratory and in-situ application scale.

Laboratory samples of each formulation with standard dimensions of 40×40×160 mm were prepared and cured during 28 and 90 days at approximately 20 °C and 60%RH. The mortars were characterized in terms of water transport properties, mechanical behaviour and microstructure porosity.

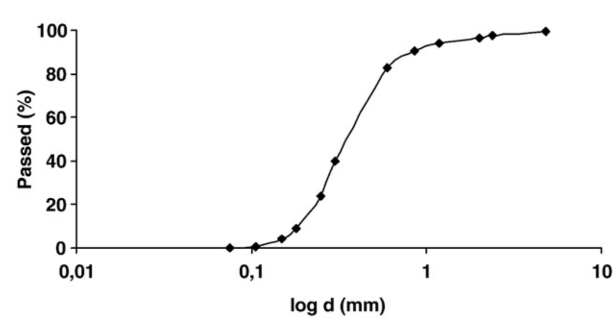

Fig. 1. Grain size distribution of aggregate used in studied formulations.

The water absorption coefficient was measured according to EN 1015-18 European standard. The capillary absorption coefficient C is the water mass absorbed per unit time, through a unitary surface of a dried sample in contact with a water layer, and taken from the $(m/A) = C \cdot t^{0.5}$ relation, where m is the absorbed water mass, A is the contact area between the sample and the water layer and t is the measurement time.

The mechanical characterization involved measurements of dynamic elastic modulus (E) by the frequency resonance method and flexural and compressive strengths (Fs, Cs), following standard methods (EN 1015-11).

The open porosity measurement was carried out by Mercury Intrusion Porosimetry (MIP).

Besides this laboratory characterization, an in-situ test of two selected mortars was also performed to complement this hardened state characterization. The CA2 and CA2M formulations (Table 1) were chosen for pilot tests in adobe walls in order to complement the laboratory results. Mortars were applied as renders on an adobe wall since in many rehabilitation works, this support made of weak material is found coupled with lime-based mortars as renders. This particular application field is located nearby the salt flats that are common in the Aveiro region, which can be an increased difficulty factor in real applications.

CA2 mortar was prepared in situ while CA2M had been previously prepared in order to allow maturation for 7 days, as performed before.

The mortars were set in adobe walls as 3-layer panels and tested in relation to their surface strength and water absorption. Surface strength or hardness was evaluated using the Schmidt hammer test, with which five measurements were performed on each panel. The water absorption test under low pressure was measured using Karsten tubes, also in different panel locations [15].

3. Results and discussion

3.1. Rheological characterization

The rheological characterization was made using samples with high content hydrate lime powder (binder:aggregate volume ratio of 1:1) because, in this formulation, the changes related to the air lime in the mortar fresh state behaviour, during the testing time, is more evident due to the higher binder content.

Fig. 2 shows that the air lime mortar thickens with agitation time and this effect is clearer with the increase of agitation speed. Air lime particles are small and have a great tendency to form agglomerates. In their study on lime putty and commercial air lime, Rodriguez-Navarro et al. [21] observed these agglomerates constitution with scanning electron microscopy. They are made of aggregates of nanometer-sized $Ca(OH)_2$ particles.

When the hydrated powder lime is mixed with the water, initially, there are only adsorbed water connections with the agglomerates and the amount of capillary water, or free water, is enough to lubricate the system. The agitation induces the agglomerates' gradual breakdown, increasing the lime specific surface exposed to the adsorbed water

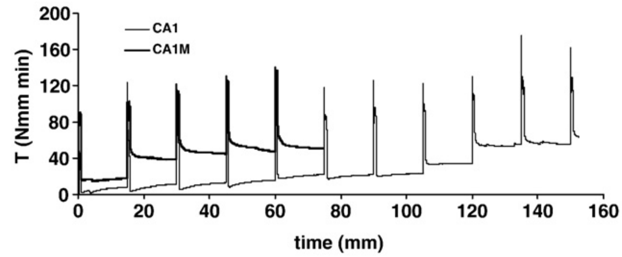

Fig. 3. Torque variation for a mortar (CA1) and a seven days matured mortar (CAM1), prepared with 30% of kneading water over a special conditions test.

leading to a decrease in the amount of free water and a suspension thickening [16,19]. Fig. 2 shows that the thickening effect is more visible for high rotation speed (160 rpm), even with more kneading water (35%) present in the mortar, than for low rotation speed (20 rpm) with 30% of water, since the agglomerates' breakdown rate is much greater with high rotation speed.

However, even without constant agitation, it is possible to observe a thickening effect with time (Fig. 3). With a special designed speed profile in the rheometric test, where the air lime mortar is almost resting during the length of the test, thickening is observed only by increasing rotation speed every 15 min up to 160 rpm. Indeed, torque values show an increase with time, due to the thickening of the suspension, for both mortars. However the matured mortar always displays higher torque values than the non-matured mortar (CA1). Comparing the two mortars' behaviour, the matured mortar (CAM1) presents a torque value at the beginning of the test that is only shown by the non-matured mortar (CA1) after 1 h of test. Thus, the maturation process could possibly be accelerated by increasing the rotation time and speed during the mixing process.

Fig. 4 shows the variation of parameters h and g with the test time for the mortar (CA1) and the seven days matured mortar (CAM1), with 30% of kneading water. Reported values present standard deviation less than $\pm 5\%$. These rheological parameters are taken from the $T = g + hN$ relationship, when N is varied every 15 min from 0 to 160 rpm (see Fig. 3). In terms of non-matured mortar (CA1), the plastic viscosity (h parameter) shows a decrease in the values, due to the agglomerates' breakdown and particle orientation with the flow, since smaller particles flow easier than big particles. There is a minimum h value close to 135 min and after this, there is an increase in viscosity due to the system becoming rheopectic in nature, characterized by an increase in viscosity with stress application time. This behaviour is due to the fact that the agglomerate separation into single particles and their subsequent physical connection with water, promotes a continuous compaction of the system to a point where the particles practically do not have space to flow and there is a thickening with an increase in viscosity, characteristic of the rheopectic behaviour.

The matured mortar (CAM1) does not show any decrease in the viscosity values because the agglomerates' breakdown already

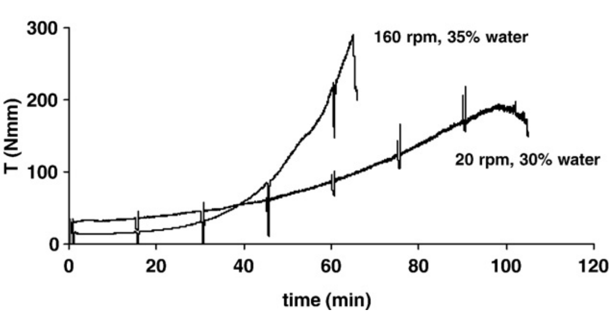

Fig. 2. Torque variation with time for mortars in rheological tests with different rotation speed (CA1 mortars prepared with 30 and 35% of kneading water).

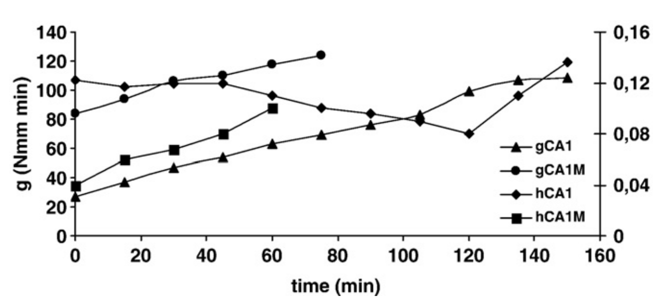

Fig. 4. Variation of h and g parameters as a function of testing time for air lime mortars (CA1 and CA1M) prepared with 30% of kneading water.

Table 2
Mechanical strength, capillarity and apparent density values of mortars.

Sample	Mechanical strength				C		Apparent density	
	Rf (MPa)		Rc (MPa)		(kg/m^2 min$^{0.5}$)		(kg/dm^3)	
	28 days	90 days	28 days	90 days	28 days	90 days	28 days	90 days
CA2	0.46 ± 0.02	0.50 ± 0.04	0.64 ± 0.04	0.86 ± 0.01	11.41 ± 0.24	11.98 ± 0.30	1.70 ± 0.13	1.83 ± 0.08
CA2M	0.39 ± 0.04	0.40 ± 0.02	0.74 ± 0.06	1.03 ± 0.03	9.52 ± 0.36	9.88 ± 0.40	1.78 ± 0.01	1.86 ± 0.05
CA3	0.37 ± 0.03	0.43 ± 0.00	0.76 ± 0.03	0.88 ± 0.03	9.29 ± 0.40	8.24 ± 0.20	1.84 ± 0.02	1.88 ± 0.02
CA3M	0.45 ± 0.01	0.54 ± 0.03	0.64 ± 0.03	1.01 ± 0.02	9.04 ± 0.30	8.06 ± 0.20	1.90 ± 0.06	1.91 ± 0.01

occurred during the maturation process. Since the start of the test, the matured mortar already shows rheopexy behaviour, represented by the increase of viscosity with the length of test. The thickening of the suspension, with the progression of the test time, caused by the decrease in the amount of free water for lubrication of the system, is also reflected in yield stress (*g* parameter) through a constant increase in their values in both mortars. Nevertheless, the matured mortar always presents higher values than the non-matured mortar.

Based on these rheological results, the increase of contact time of lime and kneading water promotes agglomerates' breakdown and so, an exchange of the free water present in capillary pores to the surface of lime particles by a physical bonding. So, if a mortar is submitted to a maturation process, a gradual water adsorption in the lime particles is expected with the consequent reduction of free water, responsible for the formation of capillary pores.

3.2. Hardened state characterization

The aim of using compositions with different binder/aggregate ratios was to observe if the effect of maturation process was the same whatever the mortar composition. Only 1:2 and 1:3 ratio formulations were characterized here since the 1:1 ratio mortars, due to higher lime content, present significant shrinkage, which hinders mechanical strength evaluation.

Table 2 presents the mechanical characteristics and water transport behaviour results for the different studied compositions. The Young modulus was measured for all the studied compositions and found to be between 4000 and 4300 MPa, inside the range recommended for rendering compatible mortars [17]. After 28 days of curing process, the maturation process does not seem to improve the results of the flexural strength values; however, the results of compressive strength already show a trend for higher values in the matured lime mortar samples. In terms of capillary coefficient, the matured samples also show a decline when compared with the non-matured formulations. One must bear in mind that lime mortars usually present a slow hardening process.

After 90 days of curing, the matured samples show higher results for mechanical strength than the samples without maturation. The same behaviour is observed in terms of capillary coefficient since the mortars present slower capillary absorption at 90 days. This behaviour becomes more evident when the content of lime in the mortar increases. The CA2 compositions, with a binder: aggregate ratio of 1:2, presents a higher decrease on the capillary values than the CA3 formulation (binder:aggregate ratio of 1:3).

In terms of apparent density, the matured mortars show higher density values than the other mortars (Table 3). The densification evolution from 28 to 90 days, due to the carbonation process, shows a higher densification rate when compared with the non-matured mortars. According to Van Balen [12,13], one of the mechanisms that controls the carbonation rate is the carbon dioxide diffusion from the air through the capillary pores up to the reaction front and the diffusivity of gaseous carbon dioxide in capillary pores depends on the openness of the porous structure. Hence, the maturation process by promoting the reduction of mortar porosity may reduce the carbonation rate.

Table 3
Surface strength for CA2 and CA2M mortars applied on an adobe wall.

Mortar	Surface strength (Schmidt hammer values) (MPa)	
	60 days	120 days
CA2	21.20 ± 0.20	25.40 ± 0.30
CA2M	21.50 ± 0.20	26.40 ± 0.20

In terms of porosity evaluation (MIP), it is possible to observe in Fig. 5 that the mortars with higher content of binder present a higher volume of pores in the pore size range linked with the binder phase. Lanas et al. [18] have already shown that, in lime-based mortars, a binder content increase leads also to a porosity increase. In Fig. 5, there is a trend for the decrease in the pore content due to the maturation process, more evident in the mortar with higher binder content (CA2M). These results are coherent with the behaviour observed on the matured samples capillary coefficient and with the rheological characterization.

The CA2 and CA2M formulations were chosen for pilot in-situ tests in adobe walls in order to complement the laboratory results obtained previously. While CAFM had been previously prepared in order to allow maturation for 1 week, the CA2 mortar was prepared in situ. Two mortar coats of about 1.5 cm were applied, using a trowel and float. A time span elapsed between the application of each layer, allowing for complete setting of the previous one. Wetting of walls and mortar layers was performed in order to improve adhesion between them.

Results for mortars surface hardness, obtained with the Schmidt hammer, gave an increase with time (after exposure for 60 and 120 days). Table 3 shows that there is an improvement in surface strength from 60 to 120 days and that the matured mortar presents slight higher values than the non-matured mortar.

Water absorption under pressure using Karsten tubes was also evaluated in panels at the age of 120 days. Standard deviation is less than ±5% for these values. As shown in Fig. 6, which presents a average of results, it was verified that there is a great difference between the performances of the matured mortar (CA2M), which displays a slower water intake than the CA2 mortar. In this way, also the in-situ test results are in agreement with what was observed above in the laboratory tests and with the work of Lanas et al. [18].

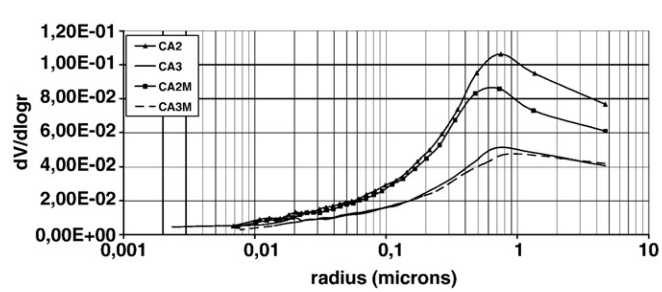

Fig. 5. Pore size distribution of CA2, CA3, CA2M and CA3M at 28 days of curing (Mercury Intrusion Porosimetry).

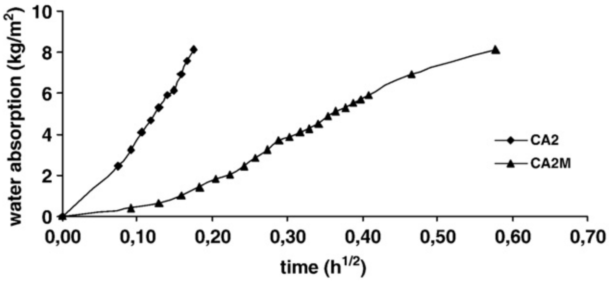

Fig. 6. Water absorption under low pressure for CA2 and CA2M after 120 days of cure.

4. Conclusions

The rheological characterization shows that hydrated powder lime mortars present a thickening behaviour due to the gradual agglomerates breakdown and the binding of water with the fresh surface of the particles. This thickening behaviour is increased with test length and with the increase of agitation speed.

Thus, considering that in the maturation process the agglomerates' breakdown occurs at a very slow speed, all this process could possibly be accelerated by increasing the rotation time and speed during the mixing process.

Both laboratory and in-situ samples show that due to the maturation process, a decrease on the capillary values and a slight increase on the mechanical strength are obtained in matured lime mortars. This behaviour is associated with the porosity reduction promoted by the maturation process, due to the gradual exchange of the free water present in capillary pores to the surface of lime particles by a physical bonding. The adsorbed water of the lime mortar submitted to a maturation process increases and consequently there is a reduction of free water, accountable for the formation of capillary pores.

This process seems to improve the mechanical and physical characteristics of the mortar. However, for long term curing, this reduction of mortar's porosity may reduce the carbonation rate, thus the complete carbonation may be achieved later than in a non-matured, more porous lime mortar, although higher strengths are achieved earlier due to higher compaction of matured lime mortars.

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