

Influence of Curing Conditions on Lime and Lime-Metakaolin Mortars

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ABSTRACT

Air-lime mortars with or without pozzolanic components were largely used in historic buildings. Due to natural or accidental degradation it is often necessary the application of repair mortars, durable and compatible with the masonries of historic buildings.

Within this context and associating the improvement of mortars characteristics to the necessity of sustainable construction practices, some mortars formulated with lime and the addition of pozzolans have been studied. In previous papers, the influence of some types of air-lime (powder hydrated lime or lime putty) and the addition of metakaolin in lime mortars have been presented. Each type of mortar presents its specificities. In pure lime mortars the setting occurs by carbonation and in lime-metakaolin mortars it occurs both by carbonation and hydration. A crucial question in order to optimize the characteristics of the mortars (and its applicability) is related with the curing conditions.

This article describes an experimental campaign with different pure air lime mortars and lime-metakaolin mortars, cured under different conditions of relative humidity and CO₂ content.

Properties of the mortars, mainly in terms of mechanical behaviour and open porosity, capillary water absorption and drying capacity, are obtained, compared and discussed. The benefits in some properties revealed by the different mortars are correlated with the laboratorial curing conditions and with *in situ* applications possibilities.

KEYWORDS

Air lime, metakaolin, mortar, curing condition, characterization.

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1 INTRODUCTION

Air lime-based mortars are present in all Portuguese old buildings, in different types of applications. The most common types of applications in old buildings are as renders, plasters and masonry mortars. As the name implies, these mortars are composed by air lime - as unique or at least main binder -, sand and sometimes pozzolans. They are ecological mortars, in comparison with mortars with hydraulic binders, because air lime is obtained by calcination at lower temperature than the one needed for hydraulic binders production. Air lime can be purchased and used as a hydrated power - after hydration of quicklime with a minimum of water - or as putty - obtained by hydration of quicklime with excess of water [Faria *et al.* 2008]. The hydration of the quicklime occurs with rising temperature and traditionally can be held together with the addition of vegetal or animal fat, for water repellence of the air lime. The pozzolans, fine materials rich in silicates or aluminates in amorphous form, can partially substitute the air lime. In the presence of water, the pozzolans react and combine with the calcium hydroxide of the air lime, developing calcium silicate and calcium aluminate hydrates that confer hydraulic properties to the mortars [Charola *et al.* 2005] and can also increment the mortar durability - but generally maintaining its compatibility with old masonry materials [Faria 2009]. Pure air lime mortars harden by carbonation, while air lime-pozzolan mortars harden by carbonation but also cure by hydration. The carbonation process occurs by combination of Ca(OH)_2 with CO_2 from the environment and depends on the presence and transport of CO_2 through the mortar. The pozzolanic reaction is a slow process as well; depends on the presence of uncarbonated Ca(OH)_2 , on the reactivity of the pozzolan - which also depends on its specific surface - and on the presence of water. The presence of water, as moisture, is then important for the CO_2 transport for carbonation and for the hydration of compounds by pozzolanic reaction.

Since the beginning of the 20th century and until nowadays air lime mortars have been replaced in old buildings, mainly in renders, by cement mortars and since then, the thousands of years knowledge of lime mortars craftsmen abruptly decreased. In the last decades, the origin of many defects that appeared in old buildings was attributed to the new cement mortars that have been applied. Since then, many researches, all over the world, have been trying to fundament the advantages of air lime-based mortars when compared with cement-based mortars. Fortunately also the knowledge of lime mortars craftsmen tends to be regained.

The main problems of cement mortars when applied as substitution renders in old buildings are their mechanical, chemical and physical incompatibility with the masonries and with other old mortars. In fact, cement mortars are much stiffer and stronger than the old masonry walls, inducing stresses in the interface with those walls and, later on, tend to break by the wall, that the render was supposed to protect. Often the cement mortar releases salts, namely sulphates that contaminate those walls they were supposed to protect. Also often the old walls have access to water - for instance by capillary rising from the ground, by problems in the roofs, by migration of the rain water through the porous structure of the exterior layers of the walls, by water vapour generated inside the building that migrate through the thickness of the walls and its protective layers towards the exterior. The water can transport salts from the outside and also salts that were already inside the walls. When the water front faces a layer that is much less permeable to water vapour in comparison to the wall materials - some paint layers or some substitution mortar layers, for instance -, the water and eventual dissolved salts concentrate in the previous layers, often the exterior surface of the original walls, weaker than the impermeable layers. In cold climates that water can originate problems of freeze/thaw, weakening the surrounding material. When salts are involved, they can go through cyclic crystallization/dissolution processes, involving stresses that also weaken the old materials. Even if the exterior rendering seems in good conditions, behind its thickness often there are big voids, due to material without cohesion. Later on the apparently good substitution render detaches, showing a huge degradation in the wall. Nevertheless air lime mortars also have disadvantages mainly regarding actual construction constrains. In fact, in construction sites rapid construction schedules and fast resistant gains are often pursuit and these are not easily achieved with pure air lime mortar renders. Often no one cares if cement renders will behave properly and if they will really protect the walls; but everyone should

remark the fact that lime renders need different application procedures. Some of those different procedures are: the air lime-based mortars should be applied with low water content compared with cement mortars; the lime render have to be applied in separate layers, with about a week between them to achieve some carbonation; each lime mortar layer should be re-tight over the base after suffering initial shrinkage to achieve better compactness.

The characteristics of the walls where the mortars are applied alter significantly the properties of the mortar and that should also be taken in to account when formulating a mortar. But in the study that is presented here, the main focus is on the different properties obtained in air lime-based mortars cured in different relative humidity conditions and CO₂ content. In face of the results, several conclusions can be taken in order to optimize *in situ* curing conditions of pure air lime mortars and of air lime-metakaolin mortars. Those results are obtained within researches held in projects FCT LIMECONTECH and METACAL.

2 EXPERIMENTAL CAMPAIGN

2.1 Preparation of the Material and of the Mortar Samples

For the preparation of mortars two washed sands were used as aggregates: a coarser sand 0/4 and a finer sand 0/2. Two air limes were used as binder in the different mortars: a powder air lime CL90 commercialized by Lusical (AL); a water repellent lime putty CL 90 commercialised by Fradical (PL). A metakaolin was used as a pozzolan (MK) produced from thermal and granulometric treatment of Portuguese kaolin. All mortar volumetric compositions were 1:3, 1 volume of air lime - or air lime+metakaolin - and 3 volumes of sand - 1 volume of finer sand and 2 volumes of coarser sand. By the dried weight of a specimen of lime putty and its apparent density it was possible to assure that the content of air lime in the lime putty mortars was higher than the one in mortars with powder air lime. In air lime-metakaolin mortars, 20% of the mass of the air lime volume was replaced by metakaolin. In mortars with lime putty, once the volume of air lime was heavier - compared with the same volume of powder air lime -, the content in metakaolin was higher. Four different mortars were prepared: mortar AL with powder air lime; mortar AL+MK with powder air lime and 20% of metakaolin; mortar PL with lime putty; PL+MK with lime putty and 20% of metakaolin (Table 1). By weight the mortars composition was 1:12 for AL mortars and 1:3,5 for PL mortars.

The mixture of the mortar components was mechanical and always identical: the water was added in the mechanical mixer tank, followed by the air lime and the sand (previously hand homogenized); mechanical mixture at low speed for 30 seconds; another 30 seconds to scrape the material inside the tank and mechanical mixture for three minutes at high speed. The procedure was based on EN 196-1 [CEN 2005] and EN 1015-2 [CEN 1998] but the period of mixture was enlarged because the one defined in the standard was considered inadequate for air lime-based mortars. In lime putty mortars PL no water was added and only a little amount was added for PL+MK mortars; the other constituents were mixed similarly. The mortar samples were mechanically compacted in two layers inside prismatic metallic moulds 40 mm x 40 mm x160 mm. The samples of each mortar were subjected to four types of curing conditions until the age of test - 60 days or 120 days -, at 20°C temperature, inside conditioned chambers: 50% relative humidity (RH) - cure identified by D; 65% RH – standard cure identified by S; 65% RH and 5% carbon dioxide – cure identified by C; 95% RH – cure identified by H (Table 1). Six samples of each mortar undergone each curing conditions.

2.2 Testing Program and Results

For each type of mortar multiple mixings were made, due to the mechanical mixer capacity and the quantity of mortar samples required for the experimental campaign. For each type of mortar, when needed, always the same quantity of water was added. All mortars seemed to provide good workability for application in real conditions. The influence of the amount of water in the fresh

mortars was evaluated by the consistency flow table test [CEN 1999a]. For all mortars preparation a comparable consistency flow of $153 \text{ mm} \pm 3 \text{ mm}$ was always reached (Table 2).

The mortars shrinkage inside the moulds was evaluated, with 6 samples of each mortar/curing condition, before demoulding, at the age of 7 days, except for mortars cured inside the carbonation chamber (cure C) that could only be demoulded - without registering any visual shrinkage - at the age of 21 days. At the age of 7 days those samples were almost as soft as at the moment of moulding; at the age of 14 days the problem persisted and only at the age of 21 days, with particular care, they could be demoulded. A possible justification for this occurrence was a possible saturation of carbonate ions at the only exterior surface of the mortar samples, inside the moulds, forming a solution rich in hydrogen carbonates - from the reaction of carbon dioxide with water - that diminished the carbonation velocity or even stopped the carbonation front in the mortar sample exterior face. It showed that confined rich CO_2 environments are not adequate for laboratory initial curing of lime-based mortars. Nevertheless and except for mortars C, shrinkage inside the moulds of the different mortar submitted to diverse curing conditions was registered, showing that shrinkage evaluation since moulding - and not only after demoulding - is important to lime-based mortars.

The carbonation velocity intended to be evaluated by the phenolphthalein method. A phenolphthalein solution at 0,5% in alcohol was applied in fresh cutting surfaces - 2 cm thickness - of 3 samples of each type at the ages of 30, 60, 90 and 120 days. It was obvious that mortar C achieved complete carbonation during the test; for the other cure conditions, carbonation were very slow, generally a little faster in mortars D and S, and a little slower in those in cure H.

At 60 days of age, 3 samples of each mortar and cure were dried in an oven at 60°C until constant mass - weight variation in 24 h not higher than 0,1%. They were used to dynamic modulus of elasticity determination by fundamental resonance frequency [CEN 2004], and three points bending flexural strength determination [CEN 1999b]. One half of each specimen from the flexural test were used to compressive strength determination at 60 days [CEN 1999b]. The other half were kept in interior environment at medium temperature of $30 \pm 3^\circ\text{C}$ and $50 \pm 5\%$ RH. Those conditions were not particularly beneficial for the lime-based mortars curing, due to the lack of moisture for carbon dioxide transport and for pozzolanic reaction. At 120 days the half samples were used to compressive strength determination and afterwards the tops of those half samples - which were perfectly undamaged - were used for open porosity determination by vacuum and hydrostatic weighing [RILEM 1980, CEN 1999c].

At 120 days, the half of 3 samples of each mortar and cure, resulting from the ones used before for the carbonation determination, were dried in an oven at 60°C until constant mass. After cooling in dry environment, they were used for capillary water absorption determination (capillary coefficient in terms of initial capillary absorption velocity and capillary absorption in terms of total adsorbed water) [CEN 2002, CEN 2009]. The lateral faces of the samples were not watertight and the test was held inside a box with saturated environment; the samples were over a geotextil with 2 mm water high. When completely saturated by capillary water, the samples were used for the drying index determination [C.Normal 1991, Brito 2009]. During drying they were kept in environmental conditions of $20 \pm 3^\circ\text{C}$ temperature and $50 \pm 5\%$ RH.

3 RESULTS

Test results of mechanical characteristics and internal structure are presented in Table 1; flow table consistency and physical characteristics are presented in Table 2.

3.1 Mechanical Characteristics and Internal Structure

Mortars with powder air lime AL present higher dynamic modulus of elasticity E than mortars with lime putty PL, what may induce a higher deformability of lime putty mortars due to the decrease of

portlandite crystal dimensions of the putty when compared to powder air lime [Hansen *et al.* 1999]. Mortars with metakaolin AL+MK and PL+MK present higher E than similar mortars without metakaolin AL and PL - except in the case of mortars with powder air lime cured with high CO₂ content AL+MK_C, with a very high standard deviation. With regard to mortars of each type (mortar and curing), the higher E is always registered by samples C cured with high CO₂ content; the following values of E are registered by samples cured at 50% or 65% RH (cure D or S) for mortars without metakaolin and by samples cured at 95% RH (cure H) for mortars with metakaolin.

Table 1. Test results (average values and standard deviation) of dynamic modulus of elasticity, flexural and compressive strength and open porosity of mortars and curing

Mortar	Mortar/Curing	$E_{(60d)}$	StDv	$R_{f(60d)}$	StDv	$R_{c(60d)}$	StDv	$R_{c(120d)}$	StDv	Open Poros.	StDv
[ID]	[ID]	[Mpa]		[Mpa]		[Mpa]		[Mpa]		[%]	
AL	AL_D	2671	10	0,2	0,1	0,4	0,1	1,0	0,0	30	0,2
	AL_S	2627	42	0,2	0,0	0,5	0,0	0,9	0,0	30	0,3
	AL_C	5028	227	0,8	0,1	1,4	1,0	1,2	0,2	29	0,4
	AL_H	2412	98	0,2	0,0	0,4	0,1	0,9	0,1	31	0,3
AL+MK	AL+MK_D	3023	123	0,3	0,0	0,5	0,2	1,1	0,1	30	0,1
	AL+MK_S	2822	71	0,3	0,1	0,4	0,2	1,0	0,0	30	0,2
	AL+MK_C	3691	504	0,7	0,1	1,2	0,3	1,2	0,2	29	0,3
	AL+MK_H	3194	76	0,3	0,0	0,7	0,3	1,3	0,0	29	0,2
PL	PL_D	1529	29	0,2	0,0	0,3	0,1	0,6	0,0	35	0,4
	PL_S	1455	4	0,2	0,0	0,3	0,0	0,6	0,0	35	0,1
	PL_C	4587	179	0,7	0,1	1,3	0,4	1,7	0,2	35	0,3
	PL_H	1232	43	0,2	0,0	-	-	0,5	0,0	35	1,0
PL+MK	PL+MK_D	2153	52	0,3	0,1	0,5	0,0	0,9	0,0	34	0,1
	PL+MK_S	2132	61	0,2	0,0	0,5	0,1	0,9	0,1	35	0,1
	PL+MK_C	4518	147	0,8	0,0	1,6	0,1	1,9	0,2	35	0,4
	PL+MK_H	2167	86	0,4	0,1	0,7	0,0	1,0	0,0	35	0,1

Regarding the flexural strength at 60 days of age, mortars with lime putty generally register a slight increase comparatively with mortars with powder air lime. Except for mortar AL+MK_C, mortars with MK register slightly higher values of flexural strength. In what concerns each type of mortar with different type of cure, as for the case of E, also mortars cured with high content of CO₂ register the higher results. Respecting the compressive strength, an increase of the results generally occurs from 60 to 120 days of age of the mortars, although the environmental condition where the samples were kept meanwhile. At 120 days mortars with powder air lime AL register higher values than those with PL (except for mortar with lime putty PL cured with a higher content of CO₂). Mortars with MK generally register an increase of compressive strength compared to the similar ones without MK. In what concerns each type of mortar with different types of cure, as for the case of E and R_f , also mortars cured with high content of CO₂ register the highest results of R_c , except for AL+MK_C; as happened before for E, the following values of R_c are registered by mortars cured at 50% or 65% RH (cure D or S) among mortars without metakaolin and by mortars cured at 95% RH (cure H) among those with metakaolin. In what relates to open porosity, it is higher for mortars with lime putty PL compared with mortars with powder air lime AL; those last mortars are then denser than the previous. Results of similar mortars with or without MK are almost the same. Regarding each type of mortar with different types of cure, only mortars with powder air lime AL cured with high content of CO₂ present a lower open porosity and a higher compactness; that can be related to rapid carbonation evolution. Results of open porosity can justify some of the mechanical characteristics obtained but also underline the particularly different internal structure that may occur in mortars with lime AL compared with lime PL.

Table 2. Test results (average values and standard deviation) of flow consistency, maximum values of capillary absorption, capillary coefficient and drying index of mortars and curing

<i>Mortar</i>	<i>Flow</i>	<i>StDv</i>	<i>Mortar/Curing</i>	<i>Capillary Absorp.</i>	<i>Capillary Coef.</i>	<i>Drying Index</i>
<i>[ID]</i>	<i>[mm]</i>		<i>[ID]</i>	<i>[kg/m²]</i>	<i>[kg/m².min.^{0,5}]</i>	
AL	155	3	AL_D	13,21	1,14	0,25
			AL_S	13,90	1,20	0,23
			AL_C	12,42	1,17	0,24
			AL_H	14,40	1,32	0,23
AL+MK	154	3	AL+MK_D	13,85	1,07	0,15
			AL+MK_S	14,47	1,12	0,21
			AL+MK_C	16,91	0,96	0,13
			AL+MK_H	13,33	0,92	0,24
PL	151	5	PL_D	4,08	0,09	0,35
			PL_S	3,27	0,03	0,33
			PL_C	1,54	0,01	0,27
			PL_H	3,28	0,05	0,42
PL+MK	152	1	PL+MK_D	4,11	0,13	0,56
			PL+MK_S	2,74	0,13	0,47
			PL+MK_C	6,49	0,03	0,25
			PL+MK_H	4,40	0,07	0,51

3.1 Physical Characteristics

Regarding the capillary coefficient, there is a strong difference of capillary coefficient between mortars with powder air lime AL and with lime putty PL; the last mentioned mortars are much less absorbent than the others due to the water repellent natural product incorporated in the lime putty - an olive oil by-product. Capillary coefficient of mortars with air lime AL is lower for mortars with metakaolin; for mortars with lime putty PL, capillary coefficient is a little higher when metakaolin partially substitutes the lime. Regarding each type of mortar with different types of cure, generally mortars cured with high content of CO₂ register the lower results - only mortar with powder lime with metakaolin cured in humid conditions AL+MK_H present a slightly lower value compared to AL+MK_C. But these results should be analysed together with the capillary water absorption. In what concerns the capillary water absorption, also the mortars with lime putty PL register very low absorption compared with those with AL. Comparing mortars with lime AL, there is an increase of water absorption when metakaolin is used, except with mortar with metakaolin and humid cure AL+MK_H. These last mentioned mortar present the best behaviour in terms of capillary absorption, among the ones with AL+MK and particularly in terms of capillary coefficient among mortars with lime AL; mortar AL_C present the best behaviour among the ones with AL without MK, in terms of capillary absorption, and one of the best concerning capillary coefficient. Comparing mortars with lime PL, the best behaviour in terms of capillary absorption is registered by mortar PL_C. Among mortars with PL+MK, the mortar with lower capillary coefficient register the highest values of total absorption and the mortar with lower capillary absorption register the higher capillary coefficient.

Results of capillary absorption must be analysed together with the drying capacity of mortars, fundamental for the elimination of water, once absorbed. Mortars with lime AL generally register lower values of drying index compared to mortars with lime PL, what means the moisture can be easily and faster eliminated from AL mortars than from PL mortars. Among mortars with lime AL, mortars with metakaolin present lower values; between mortars with lime PL, mortars without metakaolin present lower values - except for mortar C cured with high CO₂ content, with similar values. All mortars AL cured in different conditions present very similar values; among mortars with

the same lime but with metakaolin, mortar AL+MK_D and AL+MK_C register the lowest values, and humid cured mortar H present the highest drying index. Among mortars with lime PL, also mortar C present the lowest value and mortar H the highest value; among mortars with metakaolin PL+MK, a low drying index is register by mortar C while the other mortars present the highest values.

4 DISCUSSION AND CONCLUSIONS

The experimental campaign highlighted several aspects: the good workability of air lime-based mortars, even with relatively low flow table consistencies; the difficulty but the need on evaluating shrinkage since moulding; the phenolphthalein test inadequacy to evaluate lime-metakaolin carbonation due to changes registered on PH by pozzolanic reaction; the increase of mechanical characteristics when lime was partially substituted by metakaolin - although the metakaolin that was used was not very reactive and the lime-metakaolin proportion was not optimized; the good deformability, expressed in terms of the dynamic modulus of elasticity, evidenced by all mortars but particularly by mortars with lime putty, with similar flexural strength results; the higher porosity of mortars with lime putty compared to powder air lime mortars, but with similar mechanical resistances when metakaolin replacement occurred; the low capillary water absorption of analyzed lime putty mortars - this lime had a water repellent product - but also the greater difficulty to dry of these mortars - while lime without water repellent mortars absorbed rapidly and more quantity of capillary water but could release that moisture more easily; an improvement of the behavior of powder air lime mortars to initial water capillary absorption and drying capacity when metakaolin was added and the inverse situation for lime putty with water repellent mortars.

Regarding the influence of different curing conditions, the most important aspects that were detected were: the initial difficulty of hardening of lime-based mortars when exposed to high levels of CO₂; the increase of carbonation evolution and on mechanical characteristics, after the initial hardening, when the cure occurred with high level of CO₂. Although that cure situation is not reproducible on site applications, the acceleration of cure of pure air lime mortars that, after initial hardening and demoulding, are submitted to higher CO₂ environments during a defined period of time, can help in the preparation of lime mortar samples to be tested and characterized. Accordingly this study is being extended to the characterization of mortar samples submitted to other curing conditions, combining some of the previous and different ones. The aim is to define cure conditions that potentiate pure lime mortars characteristics, in order to prepare laboratory specimen to be used as support to other products, but also trying to optimize curing conditions that can be reproduced *in situ*. For the time being and from the obtained results of conditions reproducible on site, curing at 65% RH seemed to be the most appropriate for pure air-lime mortars.

For lime-metakaolin mortars, and although the general improvement registered in the mortars characteristics by the partial substitution of lime, the authors think that a substantial improvement can yet be achieved with the use of a more reactive metakaolin and a better defined proportion between each type of lime and metakaolin. In this type of mortars the amount of calcium hydroxide must be able to react with the silicates and aluminates of the metakaolin but also to carbonate. It is important to be aware of the kinetic of both the pozzolanic reaction and the carbonation process, in order to potentiate the best conditions during mortar formulation and curing. Anyway, from the analyzed results, and although not reproducible *in situ*, curing with high level of CO₂ generally potentiated the lime-metakaolin mortar characteristics. Among the curing conditions that can be closer to *in situ* situations, humid curing can potentiate lime-metakaolin mortars characteristics. Humid curing seems fundamental both for the continuity over time of the hydration - the pozzolanic reaction - and the CO₂ transport - for the carbonation process. In most Portuguese exterior environmental conditions cycles occur, between night and day, ranging from very humid to dryer conditions. Nevertheless a geotextile covering frequently wetted could be recommended to be applied *in situ* over lime-metakaolin renders during the first ages. For interior plastering of old walls in very humid environments, the application of lime-metakaolin mortars should be advantageous.

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