The Maturation Time Factor in Lime Putty Quality

by

M G MARGALHA[1], M R VEIGA[2] and J DE BRITO[3]
[1] *Municipality of Beja (CMB), University of Évora (UE)*
[2] *Laboratório Nacional de Engenharia Civil (LNEC)*
[3] *Instituto Superior Técnico (IST)*

ABSTRACT
Until the 19th century, lime in construction renderings was used mostly as a paste. After the transformation of rock into lime, the latter was stored in such a way as to maintain its characteristics. For that purpose, tanks or big ditches were used to store lime, always covered by water to avoid its carbonation.

Through this procedure, the quality of the material was guaranteed for many years.

The evolution that occurred in the material's storing processes led to a change in this procedure, i.e. the binders being used today are in most cases powdery mixtures.

Lime slaking is done at the factory, with the necessary care to avoid its contact with carbon dioxide from the atmosphere, which is not always achieved until its application in the work.

In the present paper, the authors present some results concerning the mechanical behaviour of mortars made with lime putties with different maturation times. The authors also make an analysis of the microstructure of these limes putties and of each one of the mortars prepared with these materials, during the fresh phase and, later, in the hardening phase.

1. INTRODUCTION

Lime has played a unique role in building throughout history, being a material with many qualities. The architectural heritage in Portugal is a good example of this concept, as lime mortar is frequently fund as rendering more than one century old and in a perfect state of conservation.

The special beauty that lime lends to architectural surfaces is not always understood. The original surfaces, frequently with irregularities and characteristic "defects" of the materials and manufacturing processes used, are often replaced with "cold" cement-based mortars or synthetic paints, thus depriving the surface of any features it may have had. However, not only aesthetic and historical questions are raised by covering buildings in cement mortar. There are also problems of compatibility that have arisen with the introduction of new materials that sometimes cause more damage to buildings than the original ones had.

Until cement was discovered in the 19th century, as can still be seen in old buildings, lime was the favourite binder. Cement is, of course, a material of undisputable quality, but its use is not advisable in mortar for restoration of old masonry because of its insufficient water vapour permeability, besides other aspects, such as the content of soluble hygroscopic salts and its mechanical strength [1].

The process of slow carbonation in lime is not propitious to encourage its use. Therefore, an improvement in the quality of mortar through factors such as the quality of its constituent materials is important so that they blend with the characteristics of the mortar without the usual resort to cement.

The aim of the research carried out is to improve the understanding of lime binder in the shape of paste and the characterisation of the materials used. Lime putty mortar subjected to different slaking periods was tested.

2. CHARACTERISATION OF THE MATERIALS USED

The different limes used in the tests were characterised from a chemical and mineralogical point of view and in terms of their microstructure.

The lime tested was industrially produced from a limestone with a high percentage of calcium carbonate (Tables 1 and 2). The lime chemical characteristics are presented in Table 3.

One of the difficulties encountered when making mortar with lime putty is to determine how much binder to add, as putty includes water in its composition, since even when the excess water is decanted, its density cannot be determined through traditional processes [2].

Table 1
Chemical composition of limestone (% normalised to 100%)

Sample	CaO	SiO_2	MgO	Al_2O_3	SO_3	Fe_2O_3	K_2O	MnO	CuO	Loss of ignition
Alcanede	52,3	0,4	1,5	0,7	0,2	0,1	0,8	0,06	0,06	44,0

Table 2
Mineralogical composition of limestone

Sample	$CaCO_3$ (%)	Others (%)
Alcanede	99,5	0,5

Table 3
Chemical composition of hydrated lime (% normalised to 100%)

Sample	CaO	SiO_2	Al_2O_3	SO_3	Fe_2O_3	MnO	CuO	Loss of ignition
Alcanede	71,9	0,2	0,1	0,2	0,03	0,06	0,06	27,6

The density of the lime putty was thus calculated in accordance with the density of the dry hydrated lime, taking into consideration the water the lime incorporated in its composition. The process consisted in drying in a 40ºC oven three different limes with varying slaking periods, until the mass was kept constant, and then checking the difference in weight between the wet and dry samples (Table 4).

From the analysis of Table 4, it can be concluded that the water content in the lime paste is not constant. On the other hand, no direct relationship was established between the slaking period of the lime and the water content value. In order to avoid the risk of adding a low amount of calcium hydroxide in the tests carried out with the lime putty, it was considered that the water contented in lime putty is 20% of its weight, a slightly higher amount than that found in lime paste after 5 years of slaking. As the density of the dry hydrated lime was 667.7kg/m³, the density of the lime putty was increased by 20% to a total of 801.2kg/m³.

Given the interest of knowing the microstructure of the different limes used, they were studied under an Environmental Scanning Electron Microscope (SEM/ESEM), model FEI 400 equipped with an E.T. detector, an LFD detector, and a GSED detector, existing at the University of Trás-os-Montes and Alto Douro, of Portugal.

The aim of the observations made was to study the lime paste in different phases of slaking. Four samples with slaking periods of approximately 2 hours, 1 month, 7 months and 5 years were studied. The samples were prepared by using a small portion of each lime, which was placed in the microscope chamber (Figures 1 and 2). The equipment used allowed the observation of the samples before they were dried, something extremely important with lime putties.

Some results of the observations made can be seen in Figures 3 to 6.

These observations show that the lime's microstructure changes with the slaking period. Figures 3, 4, 5 and 6 show that the crystals that form the lime putty decrease in size and change their shape becoming more elongated as the slaking period increases.

Lime putty that has been slaked for 2 hours remains very compact, with its crystals ill-defined. A larger number of portlandite crystals are more clearly defined in lime putty slaked for 1 and 7 months, there being a clear distinction in these periods as compared to the lime putty slaked for 2 hours. The portlandite crystals in lime putty slaked for 5 years are smaller than the others and several of them have an elongated shape.

3. MORTAR PREPARED WITH LIME PUTTIES

The interest of observing the microstructure of lime putty has to do with the need to know how this parameter influences the quality of the mortar, namely the effect that the size and shape of the crystals have on the lime as a binder of the aggregates.

A ratio of 1 binder: 3 aggregates was used to study the mortar prepared with lime putty with slaking periods of 1 month, 7 months and 5 years. Mortar that was slaked for 2 hours was not prepared as this slaking period did not give the quicklime time to be transformed into calcium hydroxide, which would give rise to expansion processes during its hydration and lead to deterioration of the mortar. The amounts of binder and aggregate to be added were determined by taking into account the density of each of the components. In accordance with previous decisions, a density of 801.2kg/m³ was used for the lime putty.

The mortar prepared was observed under the Environmental Scanning Electron Microscope (SEM/ESEM) described before. The results of this observation can be seen in Figures 7 to 12.

The observations made showed that adhesion of the binder to the aggregate was more effective for lime putty with a longer maturation period, in this case 5 years. This can be seen in Figure 11, where no grain of sand can be seen that has not been enveloped by the binder. On the other hand, observations under the Environmental Scanning Electron Microscope also showed a thicker layer of mortar enveloping the grains of the aggregates. As had been observed in lime putties (Figures 3 to 6), and comparing Figures 8, 10 and 12, the paste with the longest slaking period had a finer grain and some of the grains had a needle-like shape. These needle-like crystals were not visible in mortar prepared with lime putty slaked for 1 and 7 months, which has a similar microstructure. Furthermore, no great difference could be seen between different mortars prepared with these limes. Some grains of sand that had not been enveloped by the binder could be seen in those mortars (Figures 7 and 9).

By studying the different limes and mortars with varying slaking periods, it may be said that the maturation period of the lime putty has a very positive effect on the adhesion of the binder to the aggregates.

Table 4
Loss of weight in lime putties after drying

	Lime putty after 5 years	Lime putty after 3 months	Recent lime putty
Recipient with lime putty (g)	648,8	921,0	765,5
Recipient with dried lime (g)	531,0	806,8	655,0
Loss of weight (%)	18	12	14

Figure 1 Preparation of samples

Figure 2 Placing the samples in the chamber of a microscope (SEM/ESEM)

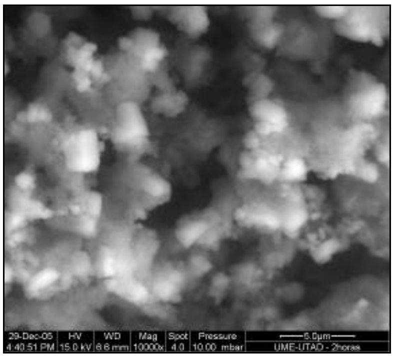

Figure 3 Observation under a microscope (SEM/ESEM): lime ≈ 2 hours (amplified 10 000x)

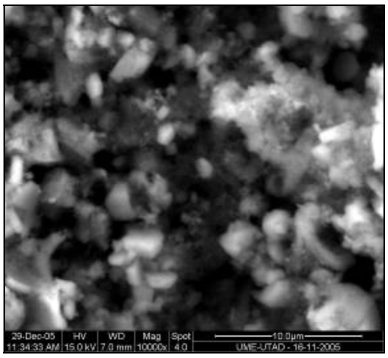

Figure 4 Observation under a microscope (SEM/ESEM): lime ≈ 1 month (amplified 10 000x)

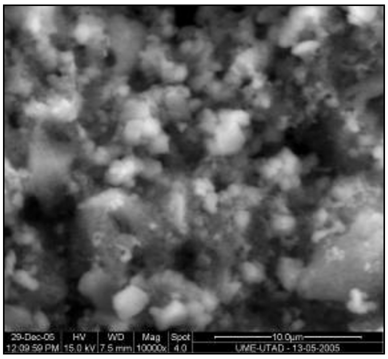

Figure 5 Observation under a microscope (SEM/ESEM): lime ≈ 7 months (amplified 10 000x)

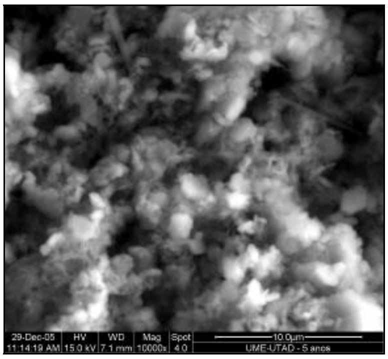

Figure 6 Observation under a microscope (SEM/ESEM): lime ≈ 5 years (amplified 10 000x)

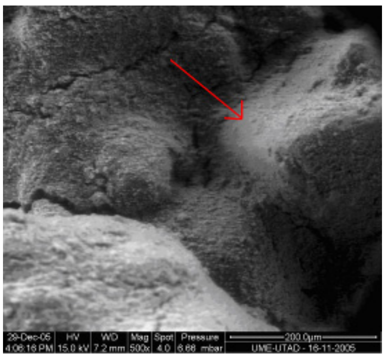

Figure 7 Observation under a microscope (SEM/ ESEM): mortar with 1 month lime (amplified 500x)

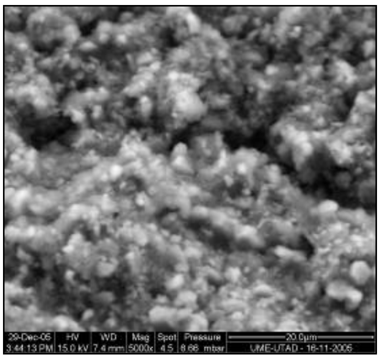

Figure 8 Observation under a microscope (SEM/ ESEM): mortar with 1 month lime (amplified 5 000x)

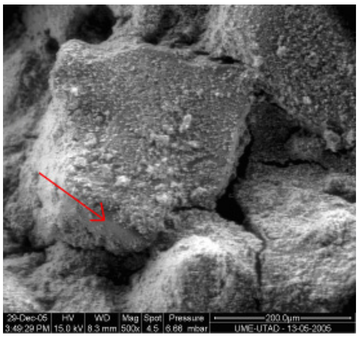

Figure 9 Observation under a microscope (SEM/ ESEM): mortar with 7 month lime (amplified 500x)

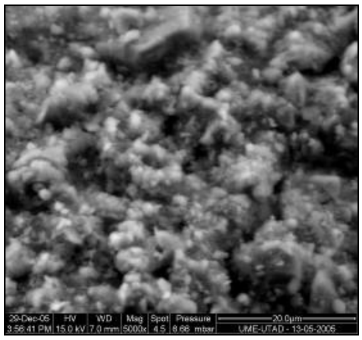

Figure 10 Observation under a microscope (SEM/ ESEM): mortar with 7 month lime (amplified 5 000x)

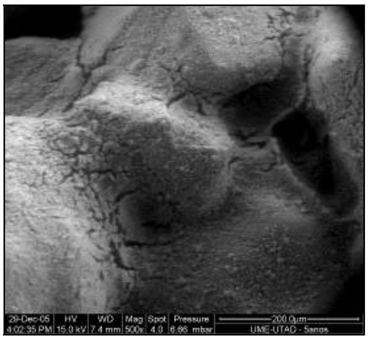

Figure 11 Observation under a microscope (SEM/ ESEM): mortar with 5 year lime (amplified 500x)

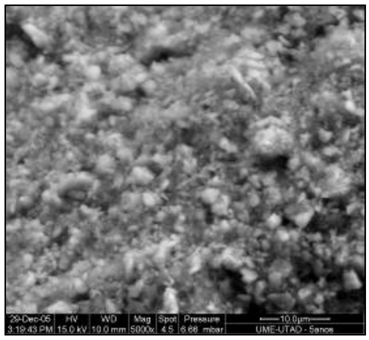

Figure 12 Observation under a microscope (SEM/ ESEM): mortar with 5 year lime (amplified 5 000x)

4. ON-SITE APPLICATIONS AND ANALYSIS OF MORTARS' CHARACTERISTICS IN LABORATORY

One of the difficulties encountered when studying mortars is the problem of reproducing the natural conditions in which they will be used, namely the porosity of the walls, the changes that occur with water vapour between the interior and exterior of buildings, among other aspects. On the other hand, lime in laboratory is tested in a way that does not help the process of carbonation when the only binder is non-hydraulic lime, due to the depth of the moulds. However, these laboratory tests are valid and useful because they allow the comparison of the results with those of other researchers.

The on-site application of mortars is a fundamental complement to the laboratory tests in order to understand performance characteristics, such as their durability in relation to external agents and their compatibility with masonry. Some different mortars were thus applied on-site in July 2005 with the aim of comparing the performance of lime putty of recent slaking (2005) and lime putty with a long period of maturation (2000) [3].

The building chosen to apply these mortars was an historical construction in one of the prettiest squares in the city of Beja in the south of Portugal.

Two panels were prepared with mortars with the proportions shown in Table 5.

The mortars were not applied in the best conditions due to the intense heat that besiege the city in July, temperatures being around 40ºC. Care was taken to keep the walls damp and the mortars were frequently wetted after being applied in order to prevent them from drying too quickly.

The chosen mortar was applied to the lower part of the building, where the previous layer of render and the finishing layer had been removed beforehand, as they showed signs of deterioration due to the application of cement mortar. The first layers of original render remained untouched.

It was seen that the mortar prepared with the old lime was easier to mix and made a perfectly homogeneous paste without addition of water (Figure 13) [4]. It was more difficult to prepare the lime mortar of recent slaking because it was very dense and it was not possible to eliminate all the existing grains of lime when mixed manually, which forced the addition of some water (Figure 14).

The mortar was applied in two layers, the first of which with a mixture of 2/3 coarse sand and 1/3 fine sand from Santa Margarida since the preliminary tests had shown that this composition of sand was favourable in terms of mechanical behaviour. The finishing layer was prepared with only fine sand from Santa Margarida. The first layer was applied with a thickness of about 10mm and the finishing layer with approximately 2 to 3mm [5]. The finishing layer was smoothed with a float and was given a slightly wrinkled finish. The proportions used were the same for the first and second layers of both panels.

The effect achieved with the use of lime putty together with light-coloured sand gave the panels an almost-white colouring and was an excellent base for the application of lime wash.

The technique applied was "wet-wet", fresh layer on top of fresh layer.

A visual inspection made a week after the application of the mortars showed that there was no damage to the panels with the older lime putty. In panels B1 and B2, made with the recent lime putty with a ratio of 1 binder: 3 aggregates, small fissures could be seen everywhere. One of the reasons given is that the addition of water to allow the mortar to achieve the necessary plasticity to be applied had been excessive. A subsequent lime wash prepared with the older lime putty filled all the fissures seen in the rendering and no visible damage can be observed today. As we know the materials are very important for good results when we work with lime, but also the care to preparing and placing the mortars play an important role [6]. As we can say we need a good material but also a good mason.

Table 5
Types of lime and proportions used in on-site tests

Identification	A1	A2	B1	B2
Type of aggregates	1/3 fine sand + 2/3 coarse sand	fine sand	1/3 fine sand + 2/3 coarse sand	fine sand
Type of lime	Recent lime putty		Old lime putty	
Density (kg/m³)	801,2*		801,2*	
Proportion in volume (works)	1 binder: 3 aggregates		1 binder: 3 aggregates	
Proportion in volume (corrected with binder density)	1 binder: 3.75 aggregates		1 binder: 3.75 aggregates	

* considering that the lime putty is made up of 20% water

Figure 13 Aspect of a mortar prepared with old lime putty

Figure 14 Preparing a mortar with recent lime putty

5. CHARACTERISATION TESTS IN LABORATORY

The mortar was transported from the work site in Beja to the National Civil Engineering Laboratory (LNEC) in Lisbon, where the laboratory tests were carried out.

The fresh mortars were tested for density according to EN 1015-2.

The cured mortars were tested at 90 days of age according to the following standards:

- Preparation of the mortars- EN 1015-2;
- Consistency- EN 1015-3;
- Flexural and compressive strength- EN 1015-11;
- Water capillarity- NF B 10-502.

The mortars prisms (40 x 40 x 160mm) were kept, during four days, in controlled conditions of 23 ± 2 ºC and 50 ± 5% relative humidity because the conditioning specified by EN 1015-2, characterised by 95% of relative humidity during the first days, does not permit to achieve the carbonation with air lime mortars. Then, the unmoulded mortars were kept in the same conditioning which has been used since long time at the National Civil Engineering Laboratory (LNEC) for lime mortars [7].

Table 6 shows some results of this work using the materials and proportions shown in Table 5.

The materials were mixed at the work site without control over the amount of water the mason thought necessary to make a workable paste. No water was added, however, to the old lime mortar used for B1 and B2. In fact, the mortar made with recent lime paste used for panels A1 and A2 showed higher values for consistency by flow table than those of the mortar made with old lime paste, but both presented adequate workability for application.

The results obtained can be justified by the amount of water added but mostly by the fact that the old lime makes a stronger binding with the aggregates.

The results are generally high as far as capillarity is concerned, although the test carried out after 28 days is not very significant for lime mortar. Capillarity was found to be lower in mortars prepared with old lime paste, which is positive and indicates a higher compactness.

On the other hand, the weight variations were also lower for the mortar made with old lime putty, which may indicate a better behaviour in terms of shrinkage.

As far as strength is concerned, no significant differences were registered in mortars prepared with lime of different slaking periods. The results of compressive strength were slightly higher in the mortar prepared with coarse and fine sand and old lime, but the trend was inverted with mortar prepared with fine sand and old lime that was lower, which did not allow a definitive conclusion. However the flexural and compressive strength show good results for applications in old masonries.

6. CONCLUSIONS

According to the tests carried out, it can be concluded that lime putties' microstructure changes with the duration of its maturation, since differences in size and shape of the crystals that compose it are visible when comparing fairly old lime (5 years) with more recently slaked lime (1 or 7 months).

It can be seen that the lime's process of maturation exerts a positive influence on the plasticity of the mortar. This eliminates the need to add a lot of water when it is being mixed, which lessens the capillary coefficient and the susceptibility to cracking.

On the other hand, when the maturation period is longer, the adhesion of the binder to the aggregates is better.

Further studies with a larger number of tests over a longer period, plus the correction of some factors that make it difficult to compare results, such as the real content of calcium hydroxide and the amount of water to be added, will hopefully allow clearer conclusions on the use of lime putty and the influence of the slaking period in the preparation of mortars.

Table 6
Laboratory tests results

Designation	A1	A2	B1	B2
Type of aggregates	1/3 fine sand + 2/3 coarse sand	fine sand	1/3 fine sand + 2/3 coarse sand	fine sand
Type of lime	Recent lime putty		Old lime putty	
Consistency (mm)	123,5	133,0	110,0	119,8
Water capillarity (kg/m².min. ½) 28 days, between 10 to 90 min.	2,04	1,86	1,69	1,58
Weight on removal from mould (g)	470,50	436,11	479,33	442,7
Weight (g) 90 days	442,98	409,56	468,22	422,0
Weight variation (%)	-5,85	-6,09	-2,32	-4,66
Flexural strength- f_f (N/mm²) 90 days	0,73	0,87	0,73	0,75
Compressive strength- f_c (N/mm²) 90 days	1,50	1,53	1,65	1,35

ACKNOWLEDGEMENTS

The authors thankfully acknowledge the contribution given by Engineer Nuno Martins of the University of the Trás-os-Montes and Alto Douro in observing the microstructure of the limes and mortars, at the Environmental Scanning Electron Microscopy centre of that Portuguese University.

The authors also thank the technician Ana Maria Francisco, of LNEC, for her special collaboration in moulds preparation and the performance of laboratory tests.

This investigation has been partially financed by a scholarship given by the Calouste Gulbenkian Foundation that the first author has benefited from to develop her PhD thesis.

REFERENCES

1. MARGALHA, M G. Sobre o uso da cal aérea no Alentejo (On the use of lime putty in Alentejo), Seminar on Color and conservation of architectonic surfaces, National Laboratory of Civil Engineering, LNEC, 1999.
2. HANSEN, E F et. al. Effects of ageing on lime putty, Proc. Of RILEM International Workshop, Historic Mortars: Characteristics and Tests, eds. P J M Bartos et. al., RILEM, pp. 197-206, 2000.
3. MARGALHA, M G, VEIGA, M R and DE BRITO, J. Algumas vantagens do uso da cal em pasta em revestimentos (Some advantages of the use of lime in renderings), 2nd Meeting on Pathology and rehabilitation of buildings, Patorreb 2006, pp. 283-293, 2006.
4. CARRINGTON, DAVID and SWALLOW, PETER. Limes and lime mortars- Part two, Journal of Architectural Conservation, nº1, pp. 7-22, 1996.
5. MALINOWSKI, E S. Historic mortars revived, Proc. of RILEM International Workshop, Repair Mortars for Historic Masonry, RILEM, 2005.
6. TEUTONICO, J M, McCAIG, I, BURNS, C and ASHURST, J. The Smeaton Project: Factors affecting the properties of lime-based mortars, Bulletin of the Association for Preservation Technology 25 (3-4), pp. 32-49, 1994.
7. MARGALHA, M G. Lime Renders in Southern Portugal, Masonry (8)- Proceedings of the Fifth International Masonry Conference, British Masonry Society, pp.125-130. 1998. Internet: www.lnec.pt/LNEC/DED/NA/fctproj/jarv/articles.htm.