

Lime mortars with rice husk ash for ancient masonry

Nuno Gouveia de Almeida

*MSc/Civil Engineer, UTL-Instituto Superior Técnico, Lisbon, Portugal,
nunofgalmeida@gmail.com*

Paulina Faria

*Assistant Professor/Civil Engineer, Nova University of Lisbon, Lisbon, Portugal,
mpr@fct.unl.pt*

Ana Paula Ferreira Pinto

*Assistant Professor/Civil Engineer, UTL-Instituto Superior Técnico- ICIST, Lisbon,
Portugal, anapinto@civil.ist.utl.pt*

SUMMARY: *This paper presents some preliminary results achieved in the aim of a research program with the main objective of evaluating the potential interest of using rice husk ashes on aerial lime based mortars for old buildings. The mechanical and physical characteristics and potential durability of lime mortars formulated with commercial rice husk ashes are analysed and their behaviour compared with a pure lime mortar. The results are also analysed taking into account the main aspects that are responsible for the compatibility between the new mortars and the original building materials. The potential durability of the tested mortars is evaluated through salt crystallization tests performed with sodium chloride and sodium sulphate solutions.*

The improvement of some properties of the lime mortars due to the presence of rice husk ashes pointed out the interest of the research in this domain, namely in what concerns the design of mortars with a moderately hydraulic behaviour for old buildings.

KEY-WORDS: *compatibility, lime, pozzolanic additions, rice husk ash, salt crystallization*

1 INTRODUCTION

1.1 Mortars for ancient masonry

Mortars in ancient buildings were largely used in construction, namely on renders, joint bedding and as major constituents of several masonries. Due to degradation in time, it becomes necessary the replacement of these mortars for new ones, which must be mechanically, physically and chemically compatible with ancient materials (stones/bricks and mortars) in order to provide the specific requirements that the interventions on historic buildings impose. The diverse applications of mortars present in old buildings oblige the new mortars to satisfy different requirements (Collepari [1], Henriques [2], Mosquera [3])

The present paper focus on a specific application of mortars: their use as wall renders. In this case, the new mortars need to assure the compatibility with the substrates and their protection against climatic and environmental actions, thus avoiding their decay. Therefore,

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these replacing mortars should have lower mechanical properties than the support, low water absorption characteristics and water vapour permeability similar to the support. Moreover, these mortars should be durable, which means that they also have to present, among other characteristics, a good resistant to salt crystallisation processes as they are the biggest threats to ancient buildings, especially those standing nearest to the sea (Henriques [2], Veiga [4]).

The aerial lime mortars are usually referred as presenting the necessary compatibility with ancient masonries. However, their application is somewhat restricted, which is usually justified by their slow setting time and low mechanical properties at early curing times. On the other hand, their durability is also limited when submitted to degradation processes by salt crystallisation because they can be responsible for the presence of internal stresses higher than the material resistance (Faria [5]).

1.2 Lime mortars with pozzolanic additions

It is referred that lime based mortars with pozzolanic properties were largely used since the Roman time until the earliest of the XX century when the cement mortars took their place (Moropoulou [6]).

However, some recent interventions carried out with cement based mortars make clear that they are incompatible with the old materials. Cement mortars favour the damage of substrates caused by salt crystallisation processes due to its content on alkaline ions and present mechanical properties quite different from the ancient masonries (Moropoulou [6], Arandigoyen [7]).

The use of lime based mortars with pozzolanic materials is often referred as a promising option for replacing mortars due to the experience of their use in the past, the recognised cement mortars incompatibility with the ancient supports, and the low durability of pure lime mortars

Artificial and natural pozzolans are able to produce a pozzolanic reaction with portlandite in presence of water. From this reaction, hydraulic compounds are developed and the resulting mortars present faster setting time than lime mortars which can occur in environments with high relative humidity. Furthermore, those mortars have higher mechanical resistances and durability than lime mortars, as well as a higher deformability and lower resistance than cement mortars.

The research on the potential interest of pozzolanic industrial by-products has been increased due to the necessity of improving the sustainable construction practices, namely with the aim of enhancing the lime mortars properties (Faria [5], Fragata [8], Moropoulou [6], Velosa [9]).

1.3 Rice husk ash as pozzolanic addition

The agricultural products constitution has innumerable minerals from soil. Silicates are present in higher proportions in annually grown plants than, for instance, in the trees which have a longer development period. Rice, wheat, tobacco plants, among others are examples of products that have a considerable potential of reutilization due to the significant amount of silica present in their constitution (Biricik [10]).

Considering the important worldwide rice production, and the waste that the rice husk represent (approximately 20 % of the husk production), several industries have been developing efforts to use this agricultural waste (Chandrasekhar [11], Sousa [12]).

In construction, rice husk could be used directly as an industrial by-product for concrete light panels or bricks production (Sousa [12]). Notwithstanding, their use as a pozzolanic material in concrete and mortars formulation implies their transformation on rice ash. The rice husk when burned under controlled conditions (time, temperature and presence of oxygen) becomes an ash with a large amount of amorphous silica, which is essential for a pozzolanic reaction (James [13], Metha [14], Nair [15], Nehdi [16]). This ash must be grounded to a fine size to get high surface area in order to increase its reactivity (Chandrasekhar [11], Metha [14]). The amorphous silica of ash when mixed with lime and water reacts with the hidroxium calcium, $\text{Ca}(\text{OH})_2$ and forms calcium silicates hydrates (C-S-H) (James [13], Metha [14]).

According to Metha rice husk ash, like silica fume, “*belong to a superior class of pozzolans because they do not suffer the variability in mineralogical character typical of fly ashes. Also both materials possess exceedingly high surface area which is an important reason for their highly active pozzolanic behaviour*” (Metha [14]). However, it should be noticed that only some rice ashes are able to be mineralogical homogeneous due to the dependence of the mineralogical composition with the rice husk and burning conditions (Chandrasekhar [11]).

Several researches have been done with the objective of increasing the cement mortars and concrete properties through the addition of rice husk ash (Coutinho [17], James [13], Metha [14], Nair [15], Nehdi [16], Sousa [12]). Metha was one of the researchers that started the study of this waste as mineral admixture to cement mortars and concretes. He found that it can be achieved a strength increase of 40% on cement mortars with 3, 7 and 28 days of curing with a cement replacement by rice husk ashes of 30% (Metha [14]). Metha concluded that the pozzolanic reaction may start as soon as calcium and hydroxyl ions were available from the cement compounds and so contribute to the initial strength enhance (Metha [14]). This conclusion could be a good intention for the lower lime mortar setting time. Similar results reporting the increase of resistance have been published by others authors (James [16], Nedhi [18]).

Although the diversity of studies of rice husk ashes in cement and concrete, researches with rice husk ashes as a constituent of lime based mortar are not common.

Besides the potential improvement of some mortar properties, the use of this by-product could lead to a substantial reduction of cost and energy consumption when compared to other materials, Table 1.

Table 1 – Energy requirements for production of various cementitious/pozzolanic materials (Ramachandran referred in Chandrasekhar [13])

Materials	Energy (kJ/kg)
Cement	372
Lime	173
Hydrated lime	142
Burnt clay pozzolan	68
Rice husk ash	12
Brick powdered	12
Fly ash	0

The rice husk addition in lime mortars might be an environmentally, technically and economically promising practice (Almeida [18]).

2 EXPERIMENTAL WORK

2.1 Materials

For the present study a river sand was used (from Tagus river). The aerial lime and rice ask ashes used were both Portuguese commercial products. The hydrated lime in powder belongs to the class CL90 according to European standards [EN459-1]. The ashes were previously prepared in two ways:

- Rice ask ashes (**ash**) - The commercial ash was mechanical sieved for two minutes in the sieve n.º 30 (from principal ASTM series - 0,600mm).
- Rice ask ashes (**ash,p**) - The rice ask ash previously sieved (**ash**) was powdered for 3 minutes in a balls-mill (**ash,p**).

Figure 1 shows the particle size distribution of the sand used and Table 2 presents the particle size of lime and both ashes of 10%, 50% and 90% of cumulative mass passed, determined by low angle laser light scattering.

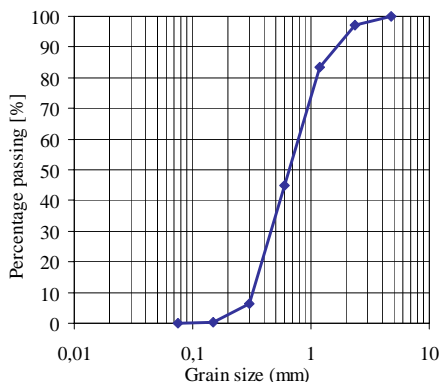


Figure 1 – Particle size distribution of river sand used

Table 2– Lime and ashes particles size distribution

Material	d(0.1) [µm]	d(0.5) [µm]	d(0.9) [µm]
lime	1.14	2.31	4.49
ash	5.09	27.16	92.69
ash,p	3.62	18.23	47.56

Four mortars were made and analysed in order to evaluate the potential interest of using rice husk ashes on lime based mortars, Table 3. All tested mortars have a binder (lime + rice ash)/aggregate ratio of 1:2 by volume. Pure lime mortar (**L**) was considered as reference mortar.

Table 3 – Tested mortars

Mortar	Composition (in volume)			
	lime	ash	ash,p.	sand
L	1			2
AS1	1	0.5		3
AS2	1	1.0		4
ASP	1		0.5	3

The water content of the mortars was established in order to ensure a flow of 65-70% in the table (EN 1015-3 – [19]). Six prismatic specimens (40x40x160mm) and three cylindrical specimens (95mm of diameter and 10mm of thickness) were prepared for each mortar. The cylindrical specimens were manual compacted pressing with a float, while the prismatic were mechanically compacted.

The specimens were stored until their characterisation (60 days of curing time) under controlled conditions of $23\pm 3^{\circ}\text{C}$ and $65\pm 5\%$ RH. The samples were demoulded 5 days after their preparation.

2.2 Experimental work

Tests on compressive and flexural strengths and the determination of the dynamic modulus of elasticity were carried out on prismatic specimens in order to evaluate the mechanical properties of the mortars. The open porosity, water absorption by capillarity and water vapour permeability were the characteristics used for the physical evaluation of the mortars. The behaviour of the mortars to sodium chloride and sodium sulphate crystallization process was also evaluated.

The dynamic modulus of elasticity was determined on all the six prismatic specimens which afterwards were submitted to the flexural test. Compressive strength tests were performed on one of the half resultant from the flexural test and the other six halves were used to perform the capillary water absorption test. After the water absorption by capillarity test, the specimens were dried in an oven at 60°C until constant mass was achieved and submitted to the durability evaluation by salt attack (3 half specimens to sodium chloride and the other to 3 sodium sulphate). The open porosity of the tested mortars was evaluated on small fragments of the specimens resulting from compressive tests. The water vapour permeability was determined on the cylindrical specimens.

3 RESULTS AND DISCUSSION

3.1 Dynamic modulus of elasticity, flexural and compressive strength

The dynamic modulus of elasticity test was performed based on the determination of the longitudinal resonance frequency using a Zeus Computer System according to NF B 10-511 [20]. Flexural and compressive strength were evaluated according to EN 1015-11 [21] (except the preparation of the specimens, described previously). Table 4 presents the average values and standard deviation of the dynamic modulus of elasticity (E) and the flexural (Rt) and compressive strength (Rc).

The results of **AS1** and **ASP** present in Table 4 point out the influence of the particle size distribution of the rice ash to the mechanical behaviour. These mortars have the same

amount of rice ash (in volume); however the rice ash used in **ASP** present lower particle size, which is responsible for the higher mechanical properties of **ASP**. As expected the reduction of particle size, associated to the increment of specific surface, seems to potentiate the pozzalanic activity (Metha [14]). According to James (James [13]), possibly occurred the formation in more proportions of hydrates compounds, like calcium silicates, which are responsible for the resistances increase.

Table 4 – Mechanical resistances of mortars

Argamassa	E (MPa)	Rt (MPa)	Rc (MPa)
L	2130 ± 50	0.30 ± 0.02	0.69 ± 0.02
AS1	1970 ± 60	0.26 ± 0.01	0.59 ± 0.01
AS2	2210 ± 40	0.27 ± 0.01	0.79 ± 0.03
ASP	2720 ± 120	0.43 ± 0.05	1.35 ± 0.05

The enhancement of the mechanical behaviour by adding powdered rice husk, **ASP**, when compared to the pure lime mortar behaviour, is in accordance to other works with lime mortars with pozzolans (Faria [5], Fragata [8], Moropoulou [6], Velosa [9]). Moropoulou [6] reached an increment of compressive and flexural strength in lime mortars with ceramic powder when compared to a pure lime mortar. The same trend was achieved by other authors namely Faria [5], Fragata [8], and Velosa [9] using respectively brick powder and kaolins, kaolins and glass powdered.

Considering the mortars **AS1** and **AS2**, both have a similar mechanical performance to the lime mortar. However, when comparing these two mortars it is observed that the increment of sieved rice husk ash was responsible for a slight enhancement of the mechanical properties, especially for the compressive test.

3.2 Porosity and density

Table 5 presents the average values and standard deviation of porosity and density of the tested mortars determined with a procedure based on EN 1936 [22]. It can be observed that lime mortar presents the highest value of porosity while the porosities of all mortars with rice ashes are similar. This could be explained by the formation of hydrated compounds, which are more expansive than CaCO_3 resulting from the carbonation reaction (Faria [5]). The density values of these mortars show that the rice ash with smaller particle size (**ASP**) may be responsible for the registered increase of density.

In spite of density values of the mortars **L**, **AS1** and **AS2** are similar there porosities were slightly different, maybe because the mortars with sieved hash have more pores not accessible to water than in lime mortar, thus decreasing their open porosity values.

Table 5 – Density and porosity of hardened mortars

Mortar	Density (kg/m^3)	Porosity (%)
L	1730 ± 10	33.3 ± 0,4
AS1	1740 ± 15	31.7 ± 1,3
AS2	1750 ± 55	31.4 ± 0,8
ASP	1810 ± 15	31.1 ± 0,5

3.3 Water absorption by capillarity

The procedure adopted for evaluation of the water absorption by capillarity was based on EN 1925 [23] and EN 1015-18 [24].

The specimens were kept inside a plastic box where saturated ambience was created in order to avoid the evaporation of water from the specimens. The specimens were dried at 60°C and their dry mass evaluated. They were placed vertically inside a tray over absorbent paper and it was guaranteed that a layer of 2mm of water stayed in contact with the lateral surfaces of the specimens. Water was added during the test to maintain the water level. The specimens were weighted after 5, 10, 15, 30, 60, 180 min, 24h from the beginning of the test and afterwards each 24h, until the differences between two successive weightings were not greater than 1%. The coefficient of water absorption by capillarity ($\text{kg/m}^2 \cdot \text{s}^{0.5}$) was determined.

Table 6 presents the average values and standard deviation of coefficients of water absorption by capillarity of the tested mortars.

Table 6- Coefficient of water absorption by capillarity

Mortar	Coefficient of water absorption by capillarity ($\text{kg/m}^2 \cdot \text{s}^{1/2}$)
L	$0.28 \pm 0,00$
AS1	$0.23 \pm 0,00$
AS2	$0.24 \pm 0,01$
ASP	$0.20 \pm 0,00$

It can be observed that pure lime mortar present the highest coefficient of water absorption by capillarity. The results of **AS1** and **ASP** present in Table 6 point out the influence of the particle size of the rice ash to its reactivity. These mortars have the same amount of rice ash (in volume) though the rice ash used in **ASP** presents a smaller particle size, which was responsible for the lower rate of water absorption registered on **ASP**.

The same trend of decreasing the initial water absorption caused by the addition of pozzolanic materials in lime mortars was achieved by Faria with pozzolanic additions like fly ashes, brick powder, kaolins among others (Faria [5]).

3.4 Water vapour permeability

This test was performed based on European Standard EN 1015-19 [25]. After drying, the lateral surface of the specimens were sealed and afterwards positioned on individual circular iron cups, containing a layer of 1cm of water, in order to maintain the 100% of RH inside. The set (cup + specimen) was initially weighted and then placed inside a climatic chamber at 20°C and 40 % RH. Sets were periodically weighted until a vapour flow is stable in order to evaluate the specimens permeability.

Table 7 presents the average values of water vapour permeability and the thickness of the diffusion air layer equivalent to 10 mm of mortar (m).

The water vapour permeability of all mortars was similar for all the tested mortars, Table 7, what seems to indicate that the rice husk ash addition does not modify the capability of the mortars to water vapour permeability.

Table 7 – Water vapour permeability

Mortar	V.P. x10-11(kg/m.s.Pa)	Sd (cm)
L	1.98	9.85
AS1	2.00	9.78
AS2	2.08	9.36
ASP	1.95	9.99

3.5 Salt crystallisation tests

3.5.1 Sodium chloride

The specimens aged through the sodium chloride crystallisation test were contaminated with a saturated salt solution by immersion for 24 hours, which was followed by the drying of the specimens at $105\pm 5^{\circ}\text{C}$ until constant mass was achieved. The amount of sodium chloride retained was determined by the dry mass variation of the specimens before and after immersion. The salt loaded specimens were submitted to 35 cycles inside a climate chamber at 20°C and weighted every week. The cycles had the duration of 24 hours: 12 hours at 90% RH and 12 hours at 40% RH. The ageing of the specimens was monitored by visual observation and through the evaluation of the mass variation expressed in percentage of the initial salt loaded dry mass.

Figure 2 presents the evolution of the mass variation during the sodium chloride salt crystallisation test. Table 8 presents the retained chloride sodium and the average values of mass variation change at the end of tests, in terms of percentage of initial dry mass.

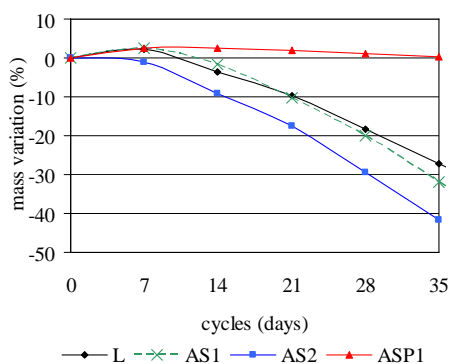


Table 8 –Chloride sodium test, average values of retained sodium and mass variation at the end of the test

Mortar	Retained salts (%)	Mass variation (%)
L	3,6	-27,3
AS1	3,3	-31,9
AS2	3,2	-41,6
ASP	3,1	1,1

Figure 2 - Chloride sodium test, mass variation

The highest amount of sodium chloride retained inside the samples was registered on the pure lime mortar, which was expected due to the fact that lime mortar is the more porous tested mortar. It is observed that the variation of the specimen's mass started before the 7th cycle. The results obtained show that all the mortars reveal approximately similar salt resistance with exception of ASP, which did not present any loss of mass. This different

behaviour probably is justified by the higher resistance of **ASP (ash,p)** induced by the higher specific surface as referred on the mechanical behaviour analysis.

3.5.2 Sodium sulphate

The test of sodium sulphate attack was performed based on EN 12370 [25]. Each cycle corresponds to the specimens contamination with a 6% anhydrous sodium sulphate solution by immersion for 2 hours and afterwards dried at $105\pm 5^{\circ}\text{C}$ for 22 hours. The specimens were submitted to 25 cycles and the ageing process was monitored by visual observation of the specimens and through the evaluation of their mass variation expressed in percentage of the initial dry mass.

Figure 3 presents the evolution of the mass variation during the sodium sulphate salt crystallisation test. Table 9 presents the average values of mass variation change at the end of tests, in terms of percentage of initial dry mass.

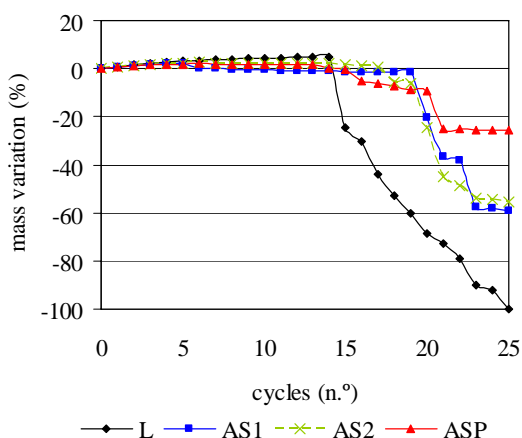


Figure 3 – Sodium sulphate action – mass variation

Table 9 – Sodium sulphate action - average values of mass variation at the end of the test

Mortar	Mass variation (%)
L	-100 (*)
AS1	-58,9
AS2	-55,2
ASP	-25,8

* specimens totally destroyed at the end of the test

Initially the specimens remained apparently more stable to the sodium sulphate attack test than under the sodium chloride ageing. In fact, the variation of the specimens mass started only after the 15th cycle. However, after that time some mortars registered an important rate of degradation. Lime mortar was the most susceptible to the simulated action followed by mortars **AS1** and **AS2** with similar behaviours. **ASP** was the mortar that revealed the highest resistance to sodium sulphate crystallisation. As expected, sodium sulphate was responsible for the highest degradation on all the tested mortars. The higher resistance of **ASP** to both salt crystallisation tests points this mortar as potentially more durable to degradation processes by salt crystallisation. This behaviour is possibly justified by the higher reactivity of the rice ash used (**ash,p**).

4. CONCLUSIONS

The potential interest of using rice husk ash on lime based mortars was evaluated studying the behaviour of different mortars. The behaviour of these mortars was compared with a pure lime mortar with an equal binder/aggregate ratio, since these types of mortars are usually referred as being compatible with ancient masonries.

According to the results achieved, the addition in pure lime mortars of powdered rice husk ash causes a better mechanical behaviour due to the increment of compressive and flexural strength, respectively 43% and 96%, as well as the increase of 28% of dynamic modulus of elasticity that is not sufficient high to compromise the required deformability. This addition is also responsible for a 40% decrease of absorbent water by capillarity which is advantageous for the protection of the support. The other physical characteristics under analysis, porosity and vapour water permeability, do not suffer significant alterations when compared to pure lime mortar. The durability of pure lime mortar, evaluated in terms of salt crystallisation, is also improved with this addition.

On the other hand, the additions in two different proportions of sieved rice husk ashes were not responsible for an important alteration of the lime mortar mechanical behaviour. Thus the main advantages of the use of sieved rice husk ashes were the increment of the value of the resistance to sodium sulphates action and the slightly decrease of the water absorbent by capillarity for the two formulations tested.

It is concluded that for most of the properties under analysis the powdered rice husk ash is better addition to mortars than ash just sieved due to their smaller particle size that is responsible for a higher pozzolanic activity.

This study contributes to verify the potential gains in the addition of rice husk ash to lime mortars.

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