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Traditional organic additives improve lime mortars: New old materials for restoration and building natural stone fabrics

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1. Introduction

Lime mortar is a mixture of lime – used as a binder – an aggregate e.g. sand (normally river sand), and water. Lime mortars have been used in building materials since very ancient times; early examples of their use have been found in Palestine and Turkey, dating back to c.12,000 B.C. [1,2] and later in ancient Greece and the Roman Empire [3–6]. Lime mortars were used continuously in construction up until the 21st century.

In the second half of the 19th century, the appearance of Portland cement led to a considerable fall in the use of lime mortars [7] because cement offered some important advantages such as quick setting times and high mechanical resistance [8,9]. In recent years, a revival of the use of lime mortars in the restoration of historic buildings has occurred as it was discovered that Portland cement had some inadequate properties and that it was incompatible with many natural stones [10–14]. Soluble salts such as calcium sulfates and sodium salts are sometimes found in Portland cement [15,16] and can leach out over time [17–21]. If this occurs, it can quickly damage the immediately surrounding materials.

The tradition and techniques associated with lime mortars were almost entirely lost in many western countries after the industrial

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ABSTRACT

Portland cement has low chemical and physical affinity for traditional building materials. This hinders the restoration of historical buildings and modern rustic architecture where natural stone is used. Lime mortars used in construction since c.12,000 B.C. were recovered, and attempts were made to enhance their properties. Various additives were selected on the basis of their properties and historical use. These include polysaccharides (opuntia, also known as nopal used either as a powder or as mucilage), proteins (animal glue and casein) and fatty acids (olive oil). Six types of lime mortar were formulated and characterized. Compressive strength, water-resistance, carbonation speed, porosity, texture and mineral composition were measured. We propose new lime mortars with added value, *i.e.* increased mechanical properties and water-resistance, different carbonation speeds, and different textures. They are all compatible with traditional building materials, so they can be used in the restoration of architectural heritage and modern architecture where natural stone is used.

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revolution. Moreover, despite the disadvantages of the use of cement based mortars in restoration and modern architecture where natural stone is used, new uses of lime mortars were not always successful because they were handled in the same way as Portland mixtures. In ancient times and indeed until 19th century lime was mixed with many different additives into improve and modify its properties. Such properties included the setting time, adhesion, impermeability, and hardness. These mixtures have been totally lost in the modern "rediscovery" of lime mortars, while commercial mixtures with resins and synthetic organic materials have come to the market, producing a lot of "noise" around the traditional application of ancient techniques and materials.

Unfortunately, in recent decades very little research has been done on lime mortars. There is a lack of rigorous studies with regard to the characteristics and properties of lime-based mortars. Slow carbonation was one of the main causes for the decline in the use of lime-based mortars. Carbonation is of fundamental importance in making mortars harder and therefore more durable [13]. This process depends on many factors including relative humidity, temperature and CO_2 concentration [22–24] and normally involves an increase in mass caused by the transformation of portlandite (Ca(OH₂)) into calcite or aragonite (CaCO₃) [22]. It has been suggested that the total carbonation of mortar could take centuries [8,10]. Shih et al. [25] observed that in conditions of relative humidity above 8%, Ca(OH)₂ would not react with CO₂ to form CaCO₃.

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Weak mechanical properties and low internal cohesion as well as high porosity were other factors that helped Portland cement surpass lime mortars in popularity.

Once placed in the fabrications of a building, the durability of mortars is influenced by external factors (i.e. environmental conditions) and by material properties (e.g. porosity, composition and texture) [26].

The aim of this study is to improve properties of lime mortars such as the carbonation rate, mechanical resistance, dry speed and water- resistance through the use of additives, which will make them more attractive to the construction industry. We also examined how the chemical or physical mechanisms of lime mortar influence the improvement of some of its properties help produce extra properties that may be useful in different restoration scenarios.

This paper is integrated in a research group, focuses on restoration science and modern architecture where natural stone is used [27]. In our research we have verified the harmful effects of Portland cement in various restored and modern buildings from 1984 onwards.

2. Experimental

Several mixtures of lime, sand (used as an aggregate) and water with different organic components (polysaccharides, proteins and fatty acids) were prepared following traditional methods used in South America and Mexico. We also prepared mixtures recommended by construction workers who used them in their previous work.

Six types of non-hydraulic lime mortars were tested:

- 1. Lime + aggregate (river sand) + water (blank sample)
- 2. Lime + aggregate (river sand) + water + animal glue (protein)
- 3. Lime + aggregate (river sand) + water + casein (protein)
- 4. Lime + aggregate (river sand) + water + nopal as powder (polysaccharide)
- 5. Lime + aggregate (river sand) + water + nopal as mucilage (polysaccharide)
- 6. Lime + aggregate (river sand) + water + olive oil (fatty acid)

2.1. Materials

2.1.1. Lime

The lime $(Ca(OH_2))$ used for the tests was a commercial product supplied by Lafac S.L [28]. It is a lime powder of class CL90-S according to Spanish standards [29].

2.1.2. Aggregate

River sand was used as an aggregate. It is the most common aggregate in mortar preparation. The main components are quartz, albite, anorthite and oligoclase grains. The grain size used was $\leq 1 \text{ mm}$.

2.1.3. Additives

All of the additives were previously used in traditional construction since the first quarter of the 20th century and are still in use today in the rural areas of many Latin American countries.

Animal glue: this is a proteinaceous substance with adhesive properties, obtained by hydrolysis from existing collagen forms i.e. the skins, nerves and animal bones. To extract the collagen, animal bones were left in water for one night to allow complete hydration. Later, the sample was heated to 45 °C to obtain a homogeneous liquid, taking care not to surpass 60 °C, at which point the collagen may lose its properties as the molecular structure breaks.

Casein (from Latin caseus "cheese"): this is the predominant phosphoprotein (α S1, α S2, β , κ) that accounts for nearly 80% of proteins in cow milk and cheese. Casein consists of a fairly high number of proline peptides, which do not interact. In addition there are no disulfide bridges. As a result, it has relatively little tertiary structure, meaning it is unable to denature. It is relatively hydrophobic, making it poorly soluble in water.

Opuntia: this is also known as nopales or paddle cactus (from the Nahuatl word *nōpalli* for the pads, or nostle, from the Nahuatl word *nōchtli* for the fruit) and is a genus in the cactus family, Cactaceae. Opuntia is native to Mexico and is commonly used in applications from medicine to construction [30]. In fact, nopal was used in building constructed by the Aztecs.

We used dry powder and mucilage from Opuntia.

Mucilage is a complex carbohydrate with an excellent capacity for absorbing water. It is produced by the *Cactaceae* family and is considered a potential source of industrial hydrocolloids [31]. The main component of this substance is a hetero-polysaccharide with a molecular weight between 2.3×10^4 and 3×10^6 g mol⁻¹ [32–33]. Several uses have been found for this component, for instance as a food

thickener, food emulsifier, as a water purifier (polyelectrolyte molecule), as an adhesive for lime $[Ca(OH)_2]$, as a natural super-plasticizer in mortars and as a food product [31–35]. The traditional procedure involves pricking pieces of Opuntia when a knife and then storing them in water for one night. Following this, the water (which dissolves part of the polysaccharides of the plant) is used to prepare the mortar.

Olive oil: this is an oil obtained from the olive (*Olea europaea*; family Oleaceae), a traditional tree crop grown in the Mediterranean Basin.

2.2. Mortar preparation

Lime/aggregate ratio selected for this study was 1:3 by volume, which is the most common ratio cited in the literature [36–41]. The percentage weight of the additives was 5%. We used this proportion because none of the ancient methods provided definitive measurements.

Mortar mixtures were prepared using the amount of water required to obtain a normal consistency and a good workability (measured by the flow table test [42]; a water/binder ratio ranging from 0.5 (1:1 specimens) to 1.2 (1:5 specimens) provided suitable workability.

All the mortars were manually mixed for 20 min, then put into prismatic $50 \times 50 \times 50$ mm inox steel casts (from here on referred to as mortar cubes). They were then slightly pressed to remove any air bubbles and voids and released from the mold after 28 days following the normal rules applied to cement mortars. Curing was carried out in controlled environmental conditions (RH 60 ± 10% and 20 ± 5 °C) until the test day. For the first 8 days the mixtures were kept in the molds with the upper side open. Thus there was only one side available for carbonation. During the last 20 days, the molds were removed.

2.3. Analytical methodology

All the mortars were analyzed 28 days after being created. During this period water loss was evaluated by carrying out regular weight checks until no further weight loss was detected.

2.3.1. Mechanical resistance

Compressive strength were measured using INSTRON 1175 equipment. The loading rate was 50 N/s. Three samples of each mortar were tested to ensure the validity of the results. The reported results are an average of all the samples.

2.3.2. Water and weight loss

Reduction in weight caused by water loss was assessed by checking the weight of the samples at fixed intervals until a constant weight was achieved.

2.3.3. Carbonation

Carbonation was assessed by dying the broken surfaces of the mortar samples with phenolphthalein (C₂₀H₁₄O₄), which turns colorless in acidic or neutral solutions and pink in basic media.

2.3.4. Pore structure

The method used to assess pore structure was the BET specific surface and porosimetry technique, developed by Brunauer, Emmett and Teller, which is used to determine specific surface area [43,44]. It is based on the absorption of an inert gas over a solid surface at low temperatures. Measurements were collected on a Micromeritics Tristar 3000 system, and the inert gas used was nitrogen.

2.3.5. Mineralogical analysis

X-ray powder diffraction patterns were recorded on a Panalytical diffractometer operated in transmission mode using Cu K α radiation with a double molybdenum crystal as primary monochromator. The XRD patterns were compared with the JCPDS database using X'Pert High Score Plus software [45].

SEM-EDS a JEOL (Tokyo, Japan) instrument model JSM-840 (with secondary and backscattered electron detection) coupled with a LINK AN 10,000 microanalyser was used. The acceleration voltage used for observation and analysis was 20 keV and the working distance was 25 mm. Samples were coated with gold in a sputtering.

2.3.6. Texture analysis

Images were observed using SEM. The instrument used was the same as described above.

3. Results and discussion

3.1. Mechanical properties test

The results of the mechanical resistance test revealed the weight that the mortar sample was capable of resisting in cm²,

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Table 1

Results of the mechanical resistance test for the mortar samples. B: Lime + aggregate (river sand) + water (blank sample), AG: Lime + aggregate (river sand) + water + animal glue, C: Lime + aggregate (river sand) + water + casein, N_p: Lime + aggregate (river sand) + water + nopal as powder, OI: Lime + aggregate (river sand) + water + nopal as mucilage.

	Compressive strength (kg/cm ²)
В	1.75
AG	3.36
С	2.94
Np	2.56
oi	2.67
N _m	1.86

which increased with the use of additives (Table 1). The most significant result was obtained using animal glue, when the resistance increased by a factor of almost 2.

3.2. Loss of water over time

Mortars were applied as a mixture with water. When wet, the mortars demonstrated a certain degree of fluency pressure, so determining the drying time is essential in ensuring an accurate estimation of when an arch or a wall structure will be complete. Water loss is therefore an important parameter to be evaluated, not only in plain mortars but in those containing additives, which could influence the evaporation process and therefore water loss.

Fig. 1 shows the weight loss of the cubes during the 28 days study period. The results show that weight loss was particularly fast in the first 12 days but after day 15, very little evaporation occurred and the cubes maintained an almost constant weight.

An interesting observation was that during water loss, the carbonation reaction that transforms portlandite (calcium hydroxide, $Ca(OH)_2$) into calcite (calcium carbonate, $CaCO_3$) begins. It is possible that the carbonation reaction causes an increase in weight, which compensates for the water weight lost to evaporation. However, in contrast to the carbonation process, water loss is fast and occurs in the first days; carbonation, and the subsequent increase in weight, is a slow process. It is well known that in lime mortars the carbonation process can take several years to complete due to the very low concentration of carbon dioxide in the atmosphere (typically 350 ppm) [46].

3.3. Carbonation test

Once the lime mortar is prepared and the drying process begins, the transformation of calcium hydroxide (portlandite, $Ca(OH)_2$) into calcium carbonate (calcite and/or aragonite, $CaCO_3$) in the presence of carbon dioxide will take place. The carbonation reaction is influenced by many factors, the most important one being moisture content and permeability (open porosity) of the mortar, as well as the carbon dioxide concentration [22].

According to Van Gemert [47], the carbonation process comprises a carbon dioxide diffusion process followed by a chemical reaction in which calcium carbonate crystals are formed. Van Balen et al. 1994 give the following equations for the dissolution of carbon dioxide in water (Eq. (1)) and the reaction of lime with the resulting carbonic acid (Eq. (2)):

$$CO_2 + H_2O = H_2CO_3$$
 (1)

$$Ca(OH)_2 + H_2CO_3 \rightarrow CaCO_3 + H_2O \tag{2}$$

The solubility of the hydrated lime depends on particle size and it is assumed that dissolution occurs at the pore surface. Since the rate of dissolution is faster than the diffusion rate of carbon dioxide, a maximum calcium hydroxide content will be in the water on the pore surface as long as carbonation is not complete [47,48].

Moorehead [22] suggests that the following reactions occur: (i) carbon dioxide dissolves in the water in the smaller capillaries where condensation is favored, forming H^+ , HCO_3^- and CO_3^- ions



Fig. 1. Mortar cube weight evolution over time.

Table 2

The depth of the carbonation front of the mortar samples 28 days after being produced. B: Lime + aggregate (river sand) + water (blank sample), AG: Lime + aggregate (river sand) + water + animal glue, C: Lime + aggregate (river sand) + water + nopal as powder, N_m: Lime + aggregate (river sand) + water + nopal as mucilage and Ol: Lime + aggregate (river sand) + water + olive oil.

	Carbonation front (mm)
В	<1
AG	<1
С	<1
Np	>2
N _m	>2
01	<1

Table 3

Porosity results of mortar samples. B: Lime + aggregate (river sand) + water (blank sample), AG: Lime + aggregate (river sand) + water + animal glue, C: Lime + aggregate (river sand) + water + casein, N_p: Lime + aggregate (river sand) + water + nopal as powder, N_m: Lime + aggregate (river sand) + water + nopal as mucilage and OI: Lime + aggregate (river sand) + water + olive oil.

	Pore volume (cm ³ /g)	Pore size average (Å)		
В	0.029	345		
AG	0.018	290		
С	0.024	303		
Np	0.025	309		
Nm	0.022	315		
01	0.014	300		

and reducing the pH; (ii) calcium hydroxide particles dissolve in the acidified capillary water to form Ca^{++} ions, which interact with CO_3^{-} ions to form calcium carbonate. This process will continue until either all the calcium hydroxide is converted into calcium carbonate or until all the water in the capillaries has evaporated due to the heat generated by the carbonation reaction.

Carbonation can start after the fresh mortar is partially dried. The optimum water content for carbonation is achieved when it corresponds to maximum adsorption on the pore surface before extensive capillary condensation occurs [47,49]. In dry or fully water – saturated mortars, no significant carbonation has been previously detected [22].

The addition of phenolphthalein to the mortar samples allowed us to obtain a qualitative evaluation of the carbonation front for the different cases studied (Table 2). The best results were obtained in the samples with nopal, both as a powder and mucilage as an additive.

3.4. Porosity test

In all the samples, the use of an additive in the mortar reduced porosity and pore size (Table 3). Significant results were observed in two cases in which olive oil and animal glue were added. When using olive oil, pore size was reduced to less than half of the standard pore size. This indicates a significant improvement in water resistance, resulting in a hydrophobic mortar.

3.5. Texture characterization

Fig. 1. Mortar cube weight evolution over time. Fig. 2 shows SEM images obtained from each of the studied mortars in fresh fracture, revealing significant texture differences between the samples. The differences relate to the number of pores, as well as the development of crystalline in the minerals.

Images of the sample with olive oil (Fig. 2f) are consistent with the results of the porosity analysis, which show it to be the sample with the least number of and smallest pores.

An interesting observation is that the mortars containing an additive, acicular crystals of aragonite (CaCO₃) are present (Fig. 2f, c, d, and e), the only exception being when casein was added (Fig. 2b). In the latter case, even when the presence of aragonite was confirmed by X-ray diffraction, the nature of the crystal was not acicular, indicating that casein determines the morphology of the crystal. The growth of acicular crystals of aragonite (CaCO₃), due to the use of an additive, may help to improve the consistency of the mortar and result in better compressive strength.





Fig. 3. XRD patterns of mortar samples. P: portlandite, A: aragonite and C: calcite.

3.6. Mineralogy characterization

No mineralogical changes were observed in samples containing an additive. Other than variations in the proportion of Portlandite $(Ca(OH)_2)$, Calcite $(CaCO_3)$ and Aragonite $(CaCO_3)$ (Fig. 3), the same mineralogical composition was observed in all the samples (Table 4).

4. Conclusions

The use of traditional organic additives in lime mortars prepared following a traditional methods enhanced some of the properties of the mortar, which are consistent with the descriptions found in the ancient records and in the tradition. However, the ancient recipes do not describe the processes used for handling, setting and curing the mortar since it is supposed to be known by workers and so, it cannot be compared with those presented in this paper. Nevertheless, and according with the logical of the building tradition, the procedures proposed in this paper are suitable and adequate for mortar preparation and improve some of the properties.

The most significant results obtained by the experimentation are as follows:

- (1) The addition of animal glue (a protect material) as additive increased the mechanical strength of the mortar by a factor of 2 (after 28 days, most probably it will be more with a longer time). This can be important for uses that occasional load can be applied or for new constructions in which incremental load is applied to the joints while the walls grow-up.
- (2) The carbonation front was significantly improved (almost by a factor of 2) by the addition of nopal both as a powder and as mucilage (mainly a polysaccharide material). In many cases of restoration this is an important factor to avoid mortar decay just after application due to rain o other mechanical erosion.
- (3) The use of olive oil (a fatty material) as additive reduce the pore system by half (in percentage of volume) and decreased the pore size. Additionally it improved the impermeability of the mortar, meaning that it can be used in cases, e.g. when a water-proof surface is needed to protect a particular area from direct rain. Animal glue also reduced the number and size of the pores, although less significantly.

Lime mortars proposed in this work are suitable as joint, repointing, rendering, recovered and restitution. They are fully compatible with traditional building materials, meaning that they can be used in the restoration of architectural heritage and in modern architecture based in traditional techniques.

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Table 4

Results of the XRD analyses of mortar samples. L: Lime, A: aggregate. B: Lime + aggregate (river sand) + water (blank sample), AG: Lime + aggregate (river sand) + water + animal glue, C: Lime + aggregate (river sand) + water + casein, N_p : Lime + aggregate (river sand) + water + nopal as powder, N_m : Lime + aggregate (river sand) + water + nopal as mucilage and OI: Lime + aggregate (river sand) + water + olive oil.

	Portlandite Ca (OH) ₂	Calcite CaCO ₃	Aragonite CaCO ₃	Quartz SiO ₂	Albite Na Al Si ₃ O ₈	Anorthite (Ca, Na) (Al, Si) ₂ Si ₂ O ₈	Oligoclase (Na, Ca) Al (Al, Si)Si ₂ O ₈	
В	х	х	х	х	х	Х	х	
AG	х	х	х	х	х	х	x	
С	х	х	х	х	х	х	х	
Np	х	х	х	х	х	х	х	
Nm	Х	х	х	х	х	х		
Ol	Х	х	х	х	х	х	х	

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