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New natural hydraulic lime mortars – Physical and microstructural properties in different curing conditions

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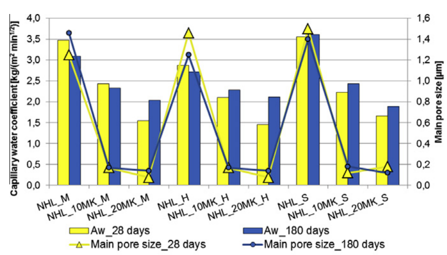
HIGHLIGHTS

- Results suggest that NHL mortars can be suitable for applications in sea aggressive conditions.
- The presence of moisture has a great contribute to the infilling of the mortars porous structure.
- MK introduction showed a clear enhancement in terms of lowering water capillary absorption.
- NHL mortars present the lowest values of drying index, by comparison with NHL–MK mortars.

GRAPHICAL ABSTRACT



NHL_M, H and S – NHL mortar in Marine, Humid and Standard curing conditions.

NHL_10MK_M, H and S – NHL mortar with 10% of MK in lime substitution in Marine, Humid and Standard curing conditions.

NHL_20MK_M, H and S – NHL mortar with 20% of MK in lime substitution in Marine, Humid and Standard curing conditions.

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ABSTRACT

The new version of EN 459-1 standard for building limes redefined the classes of hydraulic limes and made the producers reformulate or reclassify their natural hydraulic limes.

This work evaluates the mechanical, physical and microstructural behavior of mortars formulated with a recently produced natural hydraulic lime NHL3.5 that conforms to EN 459-1, submitted to natural marine environment, humid and standardized conditions, and also the benefits and drawbacks of adding metakaolin in partial replacement of lime.

Mortars with NHL3.5 present positive results at young ages. The metakaolin addition increases strength while decreasing the capillary water coefficient. The behavior in an aggressive marine environment seems promising.

1. Introduction

There are records and archaeological sites which prove that ancient civilizations used limes with pozzolans for the preparation of mortars with hydraulic characteristics namely to be into contact with water, which contributed to the development of limes with hydraulic properties [1]. With the discovery of hydraulic binders during the 18th century, airl limes were gradually replaced by hydraulic limes and by the beginning of the 20th century, mainly by Portland Cement (PC), a binder with a faster hardening and stronger mechanical characteristics [1–5].

Nowadays, it is common knowledge that the PC used in mortars for conservation and repair of old buildings was generally a wrong choice, being responsible for several problems in the repaired area, where it is frequently associated with the origin of the pathology

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[6,7]. Many buildings are prone to moisture action and particularly to marine environment, which can lead to degradation of rendering systems. This situation urges the need to select adequate mortars to be applied for repair purposes. In the last decades, due to better compatibility with masonries and facades of old buildings, lime mortars are slowly returning to repair works. Bearing this in mind, facing the degradation of the housing stock and the global construction crisis, maintenance of buildings arises as both a work and study opportunity, promoting the development of new and compatible mortars based on lime for the repair of old masonries.

Nowadays natural hydraulic lime NHL3.5 can be produced by calcination at around 900 °C of more or less argillaceous or siliceous limestones, forming calcium silicates and aluminates. The implementation of the new version of European Standard EN 459-1:2010 [8] made some producers reformulate or reclassify some of their building limes [9–11]. The new version of the building lime standard establishes three groups of limes with hydraulic properties: the natural hydraulic limes, NHL, the hydraulic limes HL and the formulated limes, FL. Some of the limes formerly classified as NHL by EN 459-1:2001 [12] are now classified as HL or FL by EN 459-1:2010 [8] due to more restricted requirements for NHL. This new version of EN 459-1 standard defines three classes for natural hydraulic limes according to compressive strength developed after 28 days of curing, as well as to $Ca(OH)_2$ content. NHL3.5 limes must present a characteristic value of compressive resistance between 3.5 MPa and 10 MPa at 28 days and a content of $Ca(OH)_2$ of at least 25% (weight percentage).

Pozzolans are defined as materials rich in silica and/or alumina in amorphous form, with high specific surface that have the property of reacting with calcium hydroxide, in the presence of water, forming hydraulic products. The pozzolanic materials can be obtained by many ways: they can be natural, originating from igneous rocks and only need to have their particle size reduced, or they can be artificial. Artificial pozzolans can be produced by thermal treatment. This is are the case of ashes resulting from the combustion of vegetal products (like rice husk ashes), of natural materials such as clays for example metakaolin. They may result directly from ground industrial byproducts (e.g. some ceramics or coal and biomass fly ashes) [13–15]. Their use has great advantages, both economic and environmental. Artificial pozzolans from calcinated materials are produced recurring to thermal treatment at temperatures below the sintering temperature of hydraulic binders. Therefore when incorporated in building materials they contribute to diminishing greenhouse gas emissions, which makes them more sustainable materials than common hydraulic binders. Interest concerning the use of pozzolans has been increasing once the mixture of hydraulic binders and pozzolans results in mortars with improved durability characteristics [15,16].

Metakaolin (MK) is a pozzolanic material resulting from kaolinitic clays thermally treated. After calcination and grinding it can become a highly reactive pozzolan with a high potential for mortars based on lime. However, studies of lime-metakaolin mortars and renders are relatively rare (only about 30–40 references in Web of Science during the last 30 years) [17], compared to cement–metakaolin mortars, and are even more so in the case of natural hydraulic lime–metakaolin mortars.

The curing conditions are an important parameter for the mortars' characterization. Different curing conditions produce changes in characteristics due to the development of chemical reactions in time [16,18] and propitiate different developments in the setting and hardening reactions, which will influence the mortars strength, porosity and microstructure [11,19]. Actually, the onsite curing conditions are completely different from laboratory standardized conditions, so it is important to analyze the influence of this factor, by testing mortars with different curing conditions, either laboratorial or natural.

In this paper, mortars formulated with a new NHL3.5, without and with metakaolin, are characterized in terms of mechanical, water action and porosity behavior after different curing conditions, one of them being a natural marine environment curing condition in. The influence of metakaolin incorporation and of the curing conditions on the evolution of NHL mortars with ageing is evaluated in terms of their durability characteristics.

2. Experimental study

The experimental study involved hydraulic lime mortars preparation, based on a natural hydraulic lime NHL3.5 with binder:aggregate ratio of 1:5 in weight. The mass of binder was maintained (NHL mortar) or partially replaced by metakaolin (MK) in weight percentages of 10% (NHL_10MK mortar) and 20% (NHL_20MK mortar). The mortar samples were exposed to three different curing conditions, and afterwards tested at different ages, up to 180 days. The weight ratio 1:5 was chosen because it corresponds approximately to a commonly used reference volumetric 1:3 binder:aggregate ratio [14], in which the volume of binder fills the voids between the sand grains,

2.1. Mortars preparation: materials and mixture

The mortars were prepared with a Portuguese natural hydraulic lime NHL3.5 [8] produced by SECIL, and a French metakaolin Argical M1200S produced by IMERYS. The chemical compositions of the NHL and the MK are presented in Table 1. The MK's Blaine specific surface is 3.38 m^2/g, and the particle size distribution $d(10\%) = 1.53$ μm, $d(50\%) = 4.35$ μm and $d(90\%) = 11.97$ μm.

A mixture of three washed and well graded siliceous sands was used as aggregate. The mixture of sands was composed of coarse sand, medium sand and finer sand in a volumetric ratio of 1:1.5:1.5, and intended to reduce the volume of voids between the grains, increasing the loose bulk density. The particle size distribution curves of each sand type and of the corresponding mixture are presented in Fig. 1. The loose bulk density of the granular constituents, determined according EN 1097-3:1998 [20], is presented in Table 2. For each NHL–MK mortar, a defined percentage of lime (10% or 20%, in weight) was substituted by the same weight of metakaolin. The mortars' weight percentage of lime substitution by MK, the volumetric and the weight compositions in terms of NHL + MK:Sand and NHL:MK:Sand, are shown in Table 3.

A quantity of potable water, previously determined to obtain mortars with flow consistency around of 150 mm was used. The preparation of the mortars and samples was based on 1015-2:1998/A1:2006 [21] but adapted to lime-based mortars, as follows: each mortar began with the correct weighing and manual homogenization of all dry materials and their introduction into the mechanical mixer container; the mechanical mixer worked at low speed and the water was introduced during the first 15–20 s; after 150 s the machine was stopped to scrape the borders and involve the mortar and turned on for another 30 s to complete the mixture.

The water/(NHL + MK) ratio of the mortars is presented in Table 3, as well as the flow table consistency, which was determined based on the European Standard EN 1015-3:1999 [22]. The mortars were then cast into metallic prismatic moulds with 40 × 40 × 160 (mm), completed with two layers, each of one mechanically compacted with 20 strokes within a mechanical mortars compacter device.

It can be seen from Table 2 that among the different sand types, the finer sand presents the minor loose bulk density value, as expected. As it can be observed by Fig. 1 the sand mixture presents an extended particle size distribution with the objective to obtain more compact mortars. In Table 3 the weight proportions of the constituents are presented for all the mortars.

2.2. Curing conditions

The freshly moulded mortar samples were placed inside polyethylene bags for 7 days for initial curing; after the two first days the samples were demoulded and continued inside the bags. After this pre-curing time, the samples were divided in three groups, each group corresponding to a distinct curing condition. The curing conditions employed were: M – natural marine environment at the experimental station of LNEC in Cabo Raso (Cascais village, Portugal, close to the Atlantic Coast); H – laboratorial controlled humid curing, with temperature (T) = 21 ± 2 °C and relative humidity (RH) = 95 ± 5%; S – laboratorial controlled standard curing, according to EN 1015-11 [23] where the mortars were placed in T = 20 ± 3 °C and RH = 65 ± 5%.

The mortar samples exposed to M curing were placed vertically, with the top protected by a ceramic tile to avoid the risk of damage by weather during the first days, and experienced natural salt water spray and salt fog conditions from January to July 2012 (winter and spring time). The average T and RH conditions during this period in the experimental station are presented in Table 4.