Construction and Building Materials 116 (2016) 384–390

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Effect of fatty acid soap on microstructure of lime-cement mortar

A. Lagazzo [a,*], S. Vicini [b], C. Cattaneo [c], R. Botter [a]

[a] Dep. of Civil, Chemical and Environmental Engineering, University of Genoa, P.le J.F. Kennedy, pad. D, 16129 Genoa, Italy
[b] Dep. of Chemistry and Industrial Chemistry, University of Genoa, Via Dodecaneso 31, 16146 Genoa, Italy
[c] TICASS scrl, Via B. Bosco 57, 16121 Genoa, Italy

HIGHLIGHTS

- Additives for mortars based on fatty acid soaps.
- Method for reducing the water absorption in lime cement-mortars.
- Effect of the microstructure on the mortar water absorption.

ARTICLE INFO

Article history:
Received 23 November 2015
Received in revised form 15 April 2016
Accepted 26 April 2016
Available online 6 May 2016

Keywords:
Mortar
Fatty acid soap
Barrier properties
Water absorption
Durability
TGA
Porosity

ABSTRACT

Different additives based on Na oleate, Na oleate – Ca stearate and Al stearate were used in two different quantities (0.15 %w/w and 0.30 %w/w of the solid amount) to enhance the waterproofing effect of mortars. From capillary water absorption test, Na oleate showed the better performance with a reduction of water absorbed of 86% in comparison with the reference mortar. The microstructure showed the presence of microcavities ranging between 0.01 mm and 0.2 mm, due to air bubbles trapped during the mortar preparation for the surfactant effect of Na oleate. The presence of a higher amount of pores (210 mm^3/g vs. 175 mm^3/g) was confirmed by the Hg porosimetry. Moreover a reduction of the carbonation rate for the presence of additive was verified.

1. Introduction

In recent decades, the apparent acceleration in the rate of building decay and the growing worldwide interest in preserving structures are promoting a significant increase in the studies of their protection [1]. It is well known [2] that masonry materials such as stones, bricks, concrete, mortar and plaster, with high porosity, undergo degradation processes due to the action of pollutants present in the atmosphere carried by rain or by moisture condensation [3].

Therefore the penetration of water inside the mortars porous matrix can be a source of a number factors which promotes the decay and the deterioration of the materials. Among these processes it is worthwhile to recall that during the water freeze/thaw cycles, mechanical stresses can arise inside the porous matrix leading cracks development [4]. Besides, water is a source of biodegradation processes due to the growth of biological organisms that can proliferate inside the aqueous phase.

To reduce the degradation process a number of studies was carried to clarify scientific and technological features of this problem. The hydrophobic treatment of mortars is one of the most important interventions usually adopted [5].

A great number of papers [6,7] was devoted to prove the efficiency of different hydrophobic substances from a technological point of view, however there are still unexplored area of investigations concerning their mechanisms of action and effectiveness. This is not a surprising due to the complex nature of the investigated systems and to the many interactions that can be established between the hydrophobic products and the mortar compounds.

Metallic soaps are popular as waterproofings [8–12] due to their low cost and effectiveness.

In order to clarify their role on the mortars wetting complex phenomena, the influence of different soaps on the lime-cement mortars micro-structure will be considered in the present paper.

* Corresponding author.
E-mail address: alberto.lagazzo@unige.it (A. Lagazzo).

http://dx.doi.org/10.1016/j.conbuildmat.2016.04.122
0950-0618/

To do this, water capillary penetration and the water evaporation tests were carried out in mortar, with and without additives.

It is well know that, during the mortar drying period, calcium carbonate formation in the mortar and particularly at the surface is catalyzed by the presence of water [13]. It is interesting to study whether or not the additives modify the carbonation process. Moreover, the cement Portland component hydration can be affected, directly or indirectly, by the hydrophobic additives. Thermal-gravimetric measurements, porosimetry and electronic microscopic analysis were performed to evaluate the effect on the microstructure of the mortars.

2. Material and methods

2.1. Materials and preparation of samples

Portland cement (CEM II/A-LL 42.5 R), hydraulic lime and quartz sand (density 2.68 g/cm^3, granulometry distribution: <0.063 mm 0.7%, <0.25 mm 28%, <1 mm 99%) were used for the mortar preparation.

Hydrophobic additives, Na oleate (NO), Na oleate – Ca stearate (NOCS) and Al stearate (AS) were used in two different quantities, corresponding to 0.15 %w/w and 0.30 % w/w of the solid amount.

The powders (11 % w/w of cement, 8 % w/w of lime, 81 % w/w of sand and a single kind of additive) was mixed for 30 min in a cylindrical container of 0.4 dm^3, rolling at 120 rpm, then regular water (pH = 7.52, solid residue at 180 °C = 265 mg/l, Ca^{++} = 50.3 mg/l, Na^{+} = 6.0 mg/l) with water/solid ratio equal to 0.22, was added to the solid mixture and manually mixed with a spatula for 3 min at room temperature. The fresh mortar was introduced in cylindrical polystyrene molds (5 cm in diameter × 0.7 cm) and compacted. The shape and the dimension of these "flat" samples were chosen for practical use as well as to simulate the renders both in the thickness and in the conditions of setting and hardening. The samples were stored for 28 days in a ventilated chamber at 70% RH at room temperature.

2.2. Characterization techniques

2.2.1. Capillary water absorption test

The wetting behaviour was studied in terms of the capillary water absorption using a gravimetric sorption technique [14]. Each specimen (5 cm in diameter × 0.7 cm) was laid on a filter paper pad, about 1 cm thick, partially immersed in deionised water. The amount of water absorbed by capillarity was determined by weighing the specimen after 10, 20, and 30 min and 1, 2, 4, 8, 24, 48, 72 and 96 h, to obtain the wet specimen mass, M_i (M ± 0.01 g).

The amount of absorbed water (Δw/S) at the time (t_i) per unit of surface (S in cm^2), is defined as:

$$(\Delta w/S) = (M_i - M_0)/S \quad (1)$$

where M_i is the wet specimen mass (in grams) at the t_i time (in min.) and M_o is the dry specimen mass (in grams), at the beginning of the test. The Δw/S values versus the time (min.) were plotted as capillary absorption curves.

The angular coefficient of the first part of the curve (i. e. the initial rate of water absorption) enables to evaluate the Capillary coefficient C expressed in kg/m^2·min$^{0.5}$.

2.2.2. Evaporation test

The tests were carried out on saturated specimens resulting from capillary tests. The evaporation test [15] was carried out in a desiccator at a constant water vapour pressure of about 1.3 mbar, kept by silica gel at 23 ± 0.5 °C. The amount of evaporated water was determined by weighing the specimen after 10, 20, and 30 min and 1, 4, 6, 8, 24, 48 h, to obtain the dry specimen mass, M_i (M ± 0.01 g). The time to complete the tests was reduced as much as possible to minimize the influence of water condensation (adsorption) on the samples.

At the end of the test the samples were dried in oven at 50 °C ± 2 °C, to reach constant mass (M_f).

The amount of evaporated water (ΔP%) at the time (t_i) is defined as:

$$\Delta P\% = [(M_i - M_f)/M_f] \times 100 \quad (2)$$

where M_i is the wet specimen mass (in grams) at the t_i time (in minutes) and M_f is the dry specimen mass (in grams) at the end of the test. The ΔP% values versus the time (min.) plot the evaporation curve.

2.2.3. Morphological characterization

Morphological features of the mortar with and without additives were observed in the external surface and in the internal section of the samples. Scanning electron microscope Hitachi S-2500 was used. SEM images were recorded using the Secondary Electron (SE) detector at different magnifications of 50×, 100×, 400× and up to 2000×. Samples were observed after coating using a Polaron CC7650 carbon coater to increase the conductivity of the samples.

2.2.4. Air entraining measurements

The measurements of air occluded in the mortar matrix during the mixing was measured according to the standard method [16]. 40 ml of fresh paste were inserted in a graduated cylinder of 100 ml, then the cylinder was filled until 50 ml with a solution of water and ethanol (60 v/v% in alcohol). The cylinder was reversed manually 20 times to remove the trapped air bubbles from the paste and disperse the solution in the paste, then the liquid level was measured. The operation was repeated until the liquid level between two subsequent reversing operations remained constant.

2.2.5. Mercury intrusion porosimetry

Mercury porosimeter measurements (MIP) were made to determine the open porosity in the range 0.0075–100 μm according to Washburn equation. A single block of 0.15 g of mortar, obtained by sawing the cylindrical sample, was dried at 70 °C for 24 h and placed in a glass dilatometer of 15 cm^3 with a capillary tube of 3 mm in diameter. After a degassing time of 15 min the sample was submitted to macropore determination and successively to the high pressure unit. The apparatus was a Pascal 140 for the low pressure and a Pascal 240 for the high pressure measurements until 202 MPa produced by Thermo scientific.

2.2.6. Thermogravimetric experiments

The focus of the Thermal Gravimetry (TG) and of the Differential Thermal Analysis (DTA) was to establish the carbonation degree along the depth of the mortar samples with and without additives.

To obtain this goal, the stored dried samples were cut at three different depths. About 50 mg of powder from each level were placed in alumina crucible inside the furnace of TG apparatus (Netzsch) in air. The heating rate was set at 10 °C/min from 20 °C up to 750 °C in air. The sensitivity of the TG was set at 10^{-2} mg.

3. Results and discussion

3.1. Effects of additives on the formation of mortars microstructure

Hydro-repellent additives produced from renewable and natural source based on oleates and stearates salts present a water solubility depending on the length of the organic chain, on the presence of unsaturated bonds and particularly on the cations type [17]. For instance, sodium stearate is water soluble, whereas calcium, aluminum or zinc stearates are really insoluble compounds. The same is true for sodium oleate (highly soluble) when compared with calcium oleate (a very insoluble compound [9]).

The introduction of certain additives such as surfactants in mortars increases the amount of air mechanically trapped into the mixture, due to a decreasing of the liquid phase surface tension in the slurry or to the introduction of fine insoluble additive particles that transport air into the slurry.

The measurements of air included into the fresh cement paste provided the following results: 11 %v/v of air for the sample containing NO as additive, 6 %v/v for the samples with NOCS and with AS and 4-5 %v/v for the mortar paste without additive (reference sample).

Fig. 1 (A, B, C and D) shows a typical view of the internal fracture surfaces corresponding to the reference mortar (A), NO mortar (B), NOCS mortar (C) and AS mortar (D), containing 0.30 % w/w of additive.

It is possible to observe in NO mortar the presence of spherical cavities with diameter ranging between 0.01 and 0.2 mm. These features were not observed in the reference sample (Fig. 1A). These cavities were typically created by air bubbles trapped in the paste during the mortar preparation. This air entraining was caused by the foaming effect of Na oleate especially at the beginning, successively the chemical reaction between calcium ions and Na oleate chains produced a calcium insoluble soap, without surfactant properties.

Closer observation at enlarge magnification, see Fig. 2 (A, B and C), shows that the internal surface of these bubbles is interconnected with the smaller pores in the network matrix of Portland