Construction and Building Materials 172 (2018) 706–716

Contents lists available at ScienceDirect

Construction and Building Materials

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

The influence of metakaolin on the properties of natural hydraulic lime-based grouts for historic masonry repair

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HIGHLIGHTS

- The influence of MK1 and MK2 on the properties of NHL-based grouts was studied.
- For target fluidity MK increases w/b and water retention of fresh grout.
- MK contributes to early age and long-term increase of grout's compressive strength.
- MK1 is more efficient due to higher reactivity and different evolution of phases.
- MK1 highly improves bond strength of the NHL-based grout.

ARTICLE INFO

Article history:
Received 11 April 2017
Received in revised form 11 March 2018
Accepted 1 April 2018

Keywords:
Natural hydraulic lime
Metakaolin
Injection grouts
Hydration products
Cultural heritage

ABSTRACT

This paper investigates the properties of injection grouts with different amounts of natural hydraulic lime (NHL) replacement by metakaolin MK1 or MK2. The focus is dedicated to the fresh properties of the NHL-based grouts, as well as to a parallel study of the evolution of compressive strength and phase composition of hardened grouts, through tests at 28, 56, 90 and 900 days. Additionally, at 90 days, the influence of the MK1 and MK2 incorporation on the porosity characteristics of hardened grouts was determined. Tensile bond strength, being one of the key hardened properties of the injection grouts, was for the time being determined only at an early age of 28 days. The results showed that by adding metakaolin at constant fluidity, the water demand of the fresh mixture increases with the increase of the metakaolin content. At the same time the water retention capacity of the grout is enhanced. In a hardened state the addition of metakaolin increases early age and long term compressive strength of the grout. Also, its bond strength can be considerably increased, by applying MK1. Mixtures with metakaolin resulted in the formation of hydration products such as ettringite, hemicarboaluminate and monocarboaluminate, whereas stellerite was present only in the case of higher proportions of metakaolin with a higher BET surface area and Al_2O_3 content.

1. Introduction

Among the many techniques that are available for strengthening and/or repairing masonry buildings, the consolidation of walls using grout injection may be the most appropriate technique when there are requirements regarding the preservation of the original appearance of rendered or plastered masonry walls, which are often painted or covered by mosaics. Injection grout (a fluid mixture made out of water, binder and additives) is injected into a wall under an appropriate pressure (usually between 1.5 and 3 bars at the exit of the grout from the container). The quality of the grout injection depends on several factors, such as the morphology of the walls, the consistency of execution (preparation of the walls, the sequence of the working procedures), and the properties of the grout in a fresh and hardened state [1–3]. In order to ensure good consolidation and strengthening of the masonry concerned, apart from having good mechanical properties, the selected grout has to be stable, fluid enough to reach the small cavities and cracks, and able to set in an environment without air [4]. These conditions can be easily achieved in the case of cement based grouts, but due to their salt crystallization, high stiffness and strength, they are not appropriate for use in historic masonry [5]. If they are used for this purpose then severe damage can be caused to the masonry, and the historic fabric may be lost. This is especially harmful in cases where cultural heritage buildings are adorned with wall paintings,

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https://doi.org/10.1016/j.conbuildmat.2018.04.007
0950-0618/

as salt crystallization can led to the detachment of the paint layers, pulverisation of the pigment, and degradation of the layers of mortar [6].

Due to the above-mentioned adverse impact of cement based binders, lime has, to an ever greater extent, become the material that is most commonly used in restoration-conservation practice [7]. Its physical and chemical properties make lime-based grouts (or plasters and mortars) highly compatible with historic materials, which makes them much more suitable for the repair and/or strengthening of historic masonry [3,8].

However, when lime is used, there are certain disadvantages compared to cement based binders: the rate of increase of strength of lime grouts is slow, their final strength is low, and they show high levels of shrinkage, so that additives in the form of pozzolanic material are often added [9–11]. Metakaolin is a highly effective pozzolan which, apart from increasing the mechanical strength of the grout, also improves its resistance to the transportation of potentially harmful substances through the matrix [12].

Several combinations of lime and metakaolin have already been studied in grout as well as mortar preparation, but only a few with natural hydraulic lime [5,9–11,13]. This type of lime differs from air limes due to its hydraulic properties, which are especially suitable for grouts that have to set in a moist environment without air [9]. Grilo et al. [14,15] has already determined some of the effects of metakaolin on natural hydraulic lime in mortars under different curing conditions, indicating that the incorporation of metakaolin improves NHL mortar strength at an early age of 28 days. However, with ageing the compressive strength of the NHL – metakaolin mortar starts to decrease, and the decrease is higher at higher metakaolin content. A similar finding is reported by Luso and Lourenço [11], where the compressive strength of the injection grout with ternary hydrated lime – hydraulic lime – metakaolin binder started to decrease after 180 days.

This paper presents the results of the first phase of a more comprehensive study about the effects of metakaolin on natural hydraulic lime-based injection grouts. Two different types of metakaolin at different substitutional levels were applied and the required workability of the fresh grout was obtained solely by adequate water content. The study focuses on the fresh properties of the lime-based grouts, as well as on a parallel study of compressive strength and phase composition of hardened grouts, at 28, 56, 90 and 900 days. Additionally, at 90 days, the influence of metakaolin incorporation on the porosity characteristics of hardened grouts was determined. Tensile bond strength, being one of the key hardened properties of the injection grouts, was for the time being determined only at an early age of 28 days.

2. Experimental

2.1. Grout mixtures

Information regarding the composition of the studied injection grouts is given in Table 1. Six different grout mixtures were prepared by mixing natural hydraulic lime (NHL) and two different types of metakaolin at substitution levels of 10%, 20% and 30% by weight. The reference mixture consisted entirely of NHL.

Table 1
Composition of the investigated injection grout mixtures.

Mix	Lime	Metakaolin		w/b
	NHL 3.5 ($\%_{mass}$)	MK1 ($\%_{mass}$)	MK2 ($\%_{mass}$)	
NHL	100	–	–	0.75
A1	90	10	–	0.76
A2	80	20	–	0.80
A3	70	30	–	0.84
B1	90	–	10	0.76
B2	80	–	20	0.81
B3	70	–	30	0.85

The grout mixtures were prepared at room temperature 21 ± 2 °C and 60% of relative humidity. First the dry components (NHL and metakaolin) were homogenised by hand mixing. Tap water was gradually added to the dry mixture, and then mixed using a high-speed mixer (1700 rpm) with a helicoidal shaped blade for 90 s. The mixing was then paused for 30 s, when the paste adhering to the wall of bowl was removed and returned to the mix; mixing was then continued for 90 s.

The amount of water used (i.e. the water/binder ratio – w/b) in the preparation of the mixtures was determined by the target fluidity of 16 ± 2 s, which is lower than the recommended upper limit value of 25 s [16]. The fluidity test was performed according to EN 445:1996, with the Cone method cl. 3.2.2 [17]. The NHL used was Chaux Blanche Nathural NHL 3.5, classified according to EN 459-1:2010 [18], produced by Lafarge, France, whereas the metakaolins used were Metaver M (MK1) and Metaver N (MK2), which are produced by Newchem, Switzerland.

2.2. Methods

The chemical composition of the raw materials (the NHL and the metakaolins) was determined by using a Wavelength Dispersive X-ray Fluorescence (WD XRF) analyser produced by Thermo Scientific ARL Perform X. Prior to each measurement, a fused bead was prepared with a mixture of the sample and flux (lithium tetraborate 50% – lithium metaborate 50%) at a ratio of 1:10, heated at 1025 °C. The loss on the ignition of the samples was determined according to EN 196-2:2013 [19].

The total specific area or Brunauer–Emmet–Teller (BET) surface area of the materials used was determined by nitrogen adsorption at 77 K over a relative pressure range of 0.05–0.3 using Micromeritics ASAP 2020 equipment. Prior to the measurements, the samples were heated at 200 °C for 2 h and outgassed to 10–3 Torr using Micromeritics Flowprep equipment. The density of the materials used was determined according to EN 1097-7:2008 [20].

Among the fresh properties of the studied injection grouts, the volume change was assessed using the can method, which is consistent with EN 445:1996, cl. 3.4.3 [17,21]. The cans were left open until the second measurement, after 24 h. Bleeding was determined according to EN 445:1996 [17] and water retention according to prEN 1015-8:1999 [22].

For the determination of the compressive strength, the mixtures were cast into prismatic moulds, 20 × 20 × 80 mm, and demoulded after two days. In the moulds and after de-moulding, the specimens were cured in sealed plastic bags at 21 ± 2 °C until the test day. For each mixture, three specimens were prepared, of which the compressive strengths were determined after 28, 56, 90 and 900 days. Tests were performed according to EN 1015-11: 1999/A1:2006 [23] using a ToniNORM, Toni-Technic by Zwick testing machine (maximum test load 10 kN, loading rate 0.05 kN/s).

Tensile bond strength determination by pull-off was performed according to EN 1542 [24]. The injection grout was applied, using thicknesses of 5 to 7 mm, to horizontal limestone surfaces (dry conditions, Karst limestone (Repen-Povir) with porosity around 1%). The composite specimens were cured in sealed plastic bags at 21 ± 2 °C until testing. The bond strength test was performed at 28 days, using Josef Freundl F15D EASY M equipment. A diamond drill crown and 50 mm diameter steel test discs were used, together with UHU Schnellfest glue.

The phase composition of the raw materials and injection grouts in a hardened state after 28, 56, 90 and 900 days was determined using X-ray powder diffraction in a PANalytical Empyrean