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An approach for a rapid determination of the aging time of lime putty

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ABSTRACT

A method for determining the effective and long aging time of lime putty, a feature which favors better quality of the corresponding lime mortar, is proposed. Different quicklime precursors were slaked under an excess of water for different times ranging from 3 to 66 months. Each of resulting lime putty was dehydrated by lyophilization and after characterized by thermo-gravimetric analysis (TGA), X-ray diffraction (XRD) analyses, scanning electron microscopy (SEM), and surface area. Taking into account the impurity contents present in the starting quicklime, the mineralogical composition of each lime putty was determined from the weight loss related to the amount of adsorbed water and to the weight losses related to the decomposition of mineral phases present in the lime putty such as brucite, $Mg(OH)_2$, portlandite, $Ca(OH)_2$ and calcite, $CaCO_3$, respectively.

The total mineralogical composition of lime putties aged for a time higher than 12 months was close to the expected value of 100%, while such value resulted lower than 100% for lime putties aged for lower times.

To justify such result, an incomplete hydration of the quicklime precursor must be considered for lime putties aged for lower times. The delayed hydration can be related to the presence of over burnt of some particles of lime present in the starting quicklime.

1. Introduction

Lime putty has been used in mortars for masonry construction for millennia reaching the maximum level of quality during the Roman Empire. It is prepared by slaking quicklime under an excess of water. In recent years, there has been an increasing use of hydrated lime, $Ca(OH)_2$, as a binder for restoration and conservation of historic and architectural structures [1]. The main reason is due to the fact that lime mortars are more compatible with materials of ancient buildings [2,3]. Lime putty aged for long time under an excess water manifests improved quality of portlandite in lime mortar or plasters [4]. High plasticity, easy workability, reduced shrinkage and increased speed of portlandite carbonation favour, in fact, better quality of corresponding lime-based mortar or plaster [5–7]. The long exposure of lime putty to water determines on the whole a reduction of portlandite crystals size. Simultaneously plate-like crystals are produced from starting prismatic crystals due to the higher dissolution of prism faces [7,8]. A continuous change in the microstructure was detected for extended periods of aging time which favours broader particles size distribution of portlandite crystals [9] of the more aged lime putty. Such features account for the quality improvement in terms of plasticity and workability of lime putty based mortars [10–13].

How old should lime putty be? As established by an ancient Roman law, an aging time longer than 36 months is necessary [14]. From the commercial point of view, actually there is not a method to determine the effective aging time of lime putty. On the other hand numerous and uncontrollable features affect the characteristics of lime putty with the aging time [5,15,16].

This communication concerns a rapid method for verifying an effective long aging of lime putty using the thermo-gravimetric analysis as main test.

2. Experimental

Three different quicklimes, QL1, QL2 and QL3 with starting composition reported in Table 1, were slaked under an excess of water for times ranging from 3 to 66 months so obtaining numerous lime putties. To avoid contact with the supernatant water, each stored sample was taken from inside the stock of the corresponding aged lime putty and was dehydrated by lyophilization and subsequently characterized.

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Table 1
Chemical composition (wt%) of the quicklime precursors.

Quicklime	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	CO_2	H_2O
QL1	<0.002	<0.001	<0.03	94.3	1.8	0.20	<0.001	0.3	2.5	0.6
QL2	0.91	<0.001	<0.002	93.7	1.2	0.17	<0.02	0.1	2.3	0.5
QL3	1.21	0.4	0.09	88.8	6.5	0.30	0.1	0.2	1.5	0.7

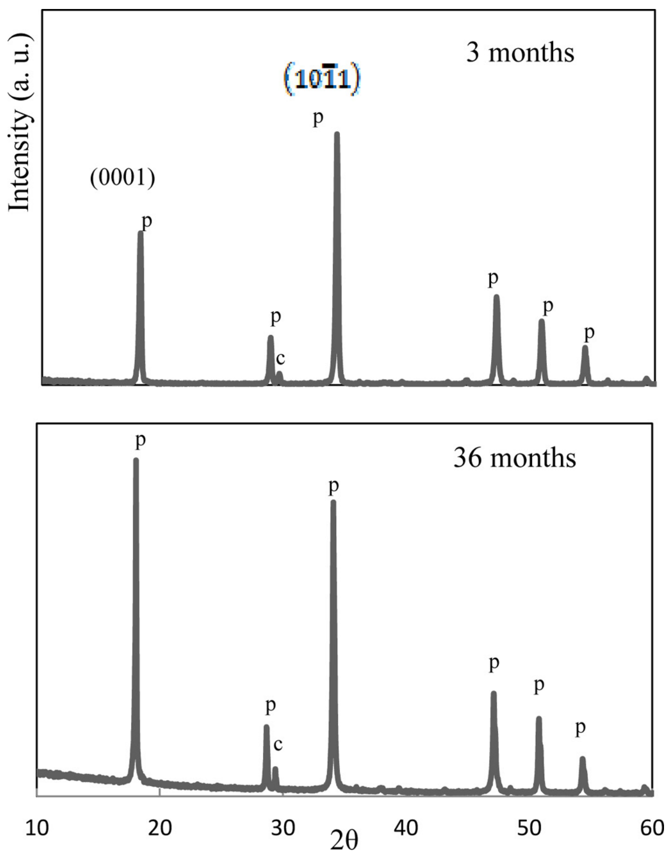

Fig. 1. XRD patterns of the 3-month and 36-month-old aged lime putties, respectively, obtained slaking quicklime QL1 (p = portlandite; c = calcite).

The water retention content was determined on sample directly dehydrated under vacuum at −46 °C for 24 h by lyophilization using a Lio Cinquepascal apparatus (Milan, Italy). Some significant samples were also characterized in the dehydration behaviour measuring the weight loss of water at selected and increasing times of lyophilisation. The mineralogical composition of the samples was investigated by X-ray diffraction (XRD) analyses, using a X'PERT diffractometer of Philips (Almelo, The Nederlands) and Cu Kα radiation. Simultaneous thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were performed on dehydrated samples using a Netzsch model 409 thermoanalyzer (Selb-Bavaria, Germany), with α-Al_2O_3 as reference and a 10 °C/min heating rate. The specific surface area of the dehydrated samples was measured by N_2 adsorption, after a further drying at 80 °C for 12 h, according to the BET method, using a Gemini instrument from Micromeritics (Norcross, GA, USA). The particle morphology was investigated by scanning electron microscopy (SEM), using a Philips microscopy (XL series, Almelo, The Nederland).

3. Results and discussion

Fig. 1 shows the XRD patterns of the 3-month and 36-month-old aged lime putties, respectively obtained slaking quicklime QL1 reported in Table 1. Both samples contain portlandite, $Ca(OH)_2$, as

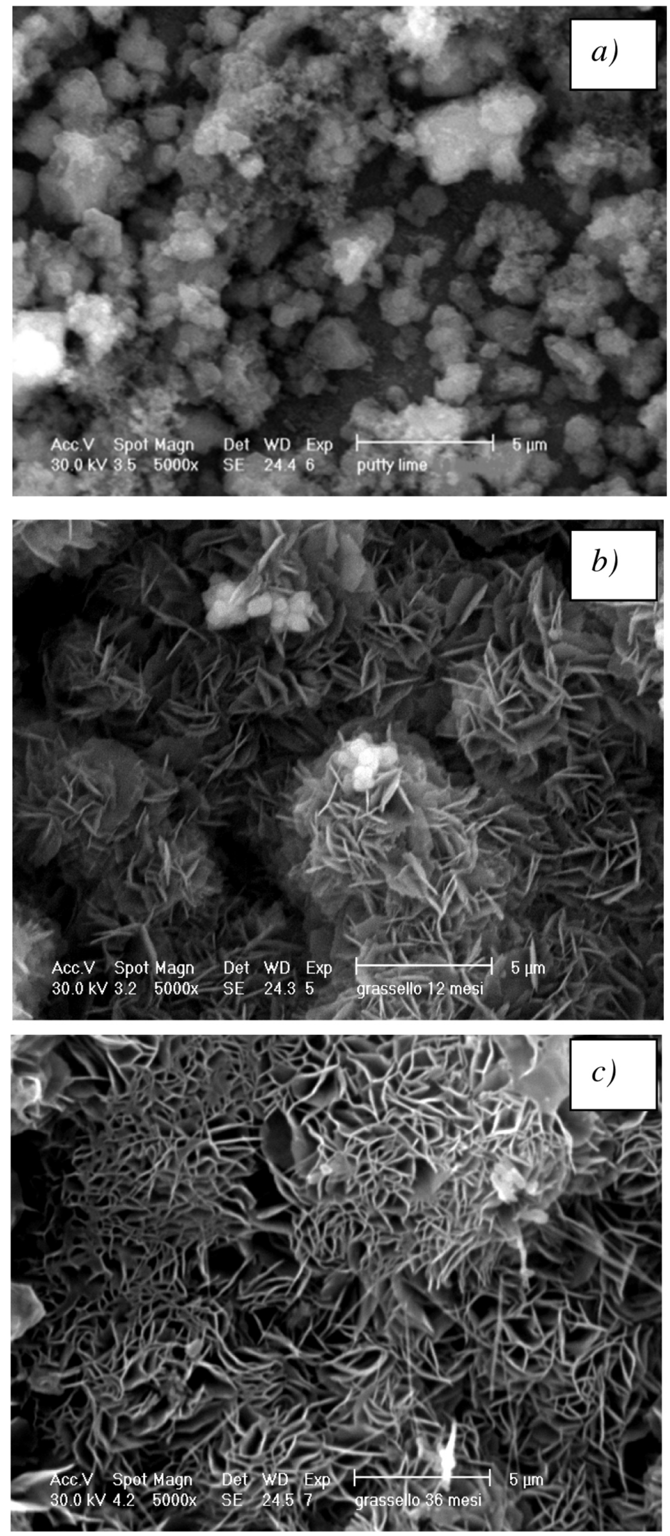

Fig. 2. SEM micrographs of lime putties aged for 3(*a*), 12 (*b*) and 36 (*c*) months, respectively.