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Influence of the traditional slaking process on the lime putty characteristics

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HIGHLIGHTS

- We compare limes from the same calcinated raw stone slaked with different methods.
- We study the influence of water ratio and temperature during slaking of lime.
- Particle size distribution increases with lower water ratios during slaking.
- Traditional slaking with higher water ratios increases the viscosity of lime putty.
- The viscosity of the lime putty increases with age.

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ABSTRACT

The influence of the slaking method on the characteristics of lime putties has been investigated. There has been used the same raw lime, calcined in a traditional kiln, to perform in situ traditional slaking processes as well as laboratory controlled slaking processes. The obtained lime putties were characterized using different instrumental techniques such as X-ray diffraction (XRD), X-ray fluorescence (XRF), thermogravimetric analysis (TGA), specific surface area (BET), particle size using laser dispersion and viscosity. Particle morphology using scanning electron microscope (SEM) was also studied. The amount of water used during slaking as well as the temperatures distribution give raise to differences in the particle size distribution and in the resulting hydration products.

1. Introduction

Lime has been used as a construction material in mortars and plastering since ancient times [1]. In the Mediterranean area the geological conformation, rich in calcareous or dolomitic rock, facilitated the use of lime as building material [2]. Limestone was calcined using traditional furnaces adapted to the characteristics of each region. In the same way, lime slaking was diverse and led to lime putties with different properties. In particular, in the North of Italy rich in dolomitic stone the use of lime was highly extended and during years was one of the main materials in local architecture. Although its use has declined due to the introduction and to the fast implementation of cements in the mid XIX Century, lime is still one of the major materials to be considered in restoration works [3]. This fact is especially relevant in those cases involving heritage conservation, because lime mortars present a better compatibility with traditional materials, such as stone and masonry, than cement and polymeric materials [4].

Lime quality has been traditionally related with factors such as the origin and calcination method of the limestone, the nature of the slaking process or the age of the lime putties [5]. The experience and skills of the professionals involved in the lime cycle, from slaking to application, are also key factors to success in the use of these materials. Nowadays, traditional methods and traditional crafts are being progressively abandoned, circumstances which may lead to a loss of this valuable knowledge.

Different studies are being carried out in order to establish the influence of the effect of different parameters upon the properties of lime putties. Different authors [6–9] analysed the characteristics of lime putties with aging. The main conclusions are that portlandite crystal size is reduced and therefore general surface area increases. The consequences of these changes induce to higher water retention and plasticity resulting in lime putties with excellent workability. In general, viscosity increases with aging but the non-Newtonian behavior of lime putties makes the analysis of the rheological properties more complex. Hydration parameters such as, agitation rate, water–lime ratio and temperature are also object of investigation [1,10]. Stirring has a positive effect on slaking process by homogenizing the mixture and dispersing heat and

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particles. On the other hand optimal lime water ratio strongly depends on the reactivity of lime, as well as on its specific surface. Temperature reached in the slaking process is one of the most affecting factors regarding the particle size and its specific surface area [11,12], especially if the source of CaO and the size of the particle to be hydrated is invariant.

The studies cited have mainly used calcium lime (CL), while in our case we have worked with dolomitic lime (DL) that is the lime mainly used in historical restoration in the North of Italy. CL slaked limes are sometimes preferred to DL, because late hydration of MgO may result in the development of small cavities in a surface owing to phenomena such as disintegration attack by SO_2 from air pollution resulting in the formation of highly soluble magnesium sulfates [6]. On the other hand, the presence of extra phases in DL that also undergo hydration could originate differences in microstructure and flow behavior, which according to Arizzi et al. [13] could explain why DL is traditionally preferred for plastering and rendering applications.

In this work, we use the same limestone burned in the same day and kiln to slake it under different conditions and obtain several lime putties. We have performed in situ traditional slaking methods as well as in laboratory in order to study the influence of the slaking process in the characteristics of the lime products. This fact is relevant because we avoid the differences in the lime putties originated due to the limestone origin and the way it is burned.

An experiment was conducted in Zone (Brescia), consisting of the slaking of lime fired in a traditional early twentieth century kiln. Raw material was obtained from calcination of dolomite rocks from the surroundings. The lime was slaked using different methods detailed in Section 2: aspersion, excess water in an earth-dug pit (*Grande Acqua*) and immersion. During the slaking process, temperature measurements were carried out using temperature sensors and infrared cameras. The samples of slaked lime were characterized using a range of laboratory techniques: X-ray diffraction (XRD), X-ray fluorescence (XRF), thermogravimetry, specific surface (BET), laser granulometry, scanning electron microscope (SEM). In a laboratory environment, the quicklime produced in Zone was slaked using different amounts of water to simulate the different traditional slaking systems and to study the effects of the temperature at which the process is carried out on the quality of the lime putty obtained. The comparison of the two sets of data shows that the different slaking methods result in important differences in the lime putties obtained, especially in terms of the size and granulometric distribution of the crystals.

2. Materials and methods

The different traditional slaking methods used in the work are described below:

2.1. Aspersion (Z2)

This slake process consists in spreading lumps of quicklime on a wooden raft with an area of 3×6 m^2 and 0.4 m height, rejecting those stones apparently undercooked and the impurities. Quicklime was then thoroughly watered, as shown in Fig. 1a and the biggest clods were crumbled using wooden shovels, in order to disaggregate the lumps and to facilitate the mixture. More water was then added, until slurry was obtained, leading to the completion of slake process [14]. Agglomerates and impurities were eliminated by continuous sieving and, finally, lime putty was decanted (see Fig. 1b) and classified in two phases according to its sedimentation.

2.2. Grande Acqua (Z3)

In this second method, a pit was dug in the ground, roughly 1.2×0.6 m^2 and 1 m deep, which was filled up to its half with burned stones form the lime kiln. Approximately 200 l of water were poured into the hole (see Fig. 1c). A few minutes after the entering in contact with the lime the water began to boil and, 400 additional litres of water were added, stirring the lime putty as shown in Fig. 1d). Quicklime-to-water ratio was roughly 1:2 in terms of weight. In some previous experiences using this slaking method, the exothermal nature of the process produced an explosion of some virulence.

2.3. Immersion (Z4)

This third slaking method consisted in filling up a 15–20 l wicker basket with calcined stones and introducing it into a water pit up to its complete immersion [11,14]. As shown in Fig. 1e and f, the basket was removed almost immediately, and then left outdoors while lime was still hydrating, and therefore increasing its volume and temperature in a process that could last several hours.

2.4. Laboratory (Z1)

Slaking processes performed in the laboratory used the burned limestone, right after being crushed in sizes between 10 and 30 mm. The whole amount of water was then added at once, and samples were slightly stirred in a cylindrical recipient of 10 cm of diameter and 20 cm height. The recipient was insulated in order to avoid heat losses.

Table 1 shows nomenclature, slaking process and lime type for the set of samples analysed. In all cases, calcined stones from the same area of the kiln were used.

2.5. Characterization

Monitoring of slaking temperatures, both in situ and in the laboratory was performed using a set of K-type thermocouples connected to a data storing device. During traditional slaking processes, a FLIR Systems InfraCAM SD thermovision equipment was used to measure infra-red radiation. Infra-red receptor was adjusted to an emissivity of 0.98.

The following techniques were used to characterize lime samples:

- X-ray fluorescence (XRF), has been used to determine the chemical composition of the burned limestone used as raw material. A sequential spectrophotometer Philips PW2400 was used.
- X-ray diffraction (XRD). Spectra of samples have been obtained through Cu Ka 1 radiation, using a Panalytical alfa powder diffractometer. To avoid carbonation, lime samples were dried in a nitrogen atmosphere.
- Particle size distribution has been determined with a Beckman Coulter LS 13 320 device. Distilled water has been used as a dispersant agent, and ultrasounds have been used to ensure the homogenization of the samples and the crumbling of the agglomerates.
- Specific surface area has been measured with BET method, using a Micromeritics Tristar 3000 device. Samples Z2 and Z3 were dried at 105 °C in a nitrogen atmosphere.
- Thermogravimetric analysis (TGA) and differential thermal analysis (DTA) were performed in a Mettler Toledo TGA-SDTA 851e/SF/1100 thermobalance, in air atmosphere, with a heating rate of 20 °C/min for a range between 25 and 1000 °C.
- Superficial morphology of the samples has been observed using a Hitachi H-4100FE scanning electron microscope (SEM). Samples were prepared through a drying process inside a N_2 atmosphere drying stove, and were covered with gold in order to facilitate their observation.
- Viscosity of lime putty samples has been obtained using a rotational viscosimeter Thermo Haake Viscotester 7L Plus. Samples have been dispersed in water up to a 30% in solid content. Resulting slurry has been homogenized for 2 min and has been analysed using L3 and L4 standard rods of the equipment.

The sample is then submitted, for approximately four minutes, to a shear stress produced by a constant rotational speed of 10 rpm. Viscosities values are set to be the average of the values obtained between 220 and 240 s.

3. Results and discussion

3.1. Evolution of temperature during lime slaking process

Fig. 2 shows quicklime temperature just before removing it from calcination kiln. Although kiln was inactive for a week, thermography shows that calcined rock is still keeping a temperature nearly 90 °C. Therefore the initial temperature of the limestone in the in situ slaking process was around 50 °C.

3.1.1. Aspersion slaking method (Z2)

As shown in Fig. 3, slaking process through aspersion gives rise to heterogeneity in temperatures related to the distribution of the amount of water. In those areas where lime is completely flooded, heat is faster dissipated, thus temperatures are lower. In the areas where lime is not flooded, temperatures are higher, reaching more than 100 °C in some points.