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Physical-chemical study, characterisation and use of image analysis to assess the durability of earthen plasters exposed to rain water and acid rain

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HIGHLIGHTS

- The binder nature has little influence on physical properties of earthen plasters.
- The curing conditions have an influence on physical properties of earthen plasters.
- Earthen-based plasters have low resistance to rain water and acid rain.
- Gypsum patina formed by sulphuric rain improve the surface resistance of plasters.
- Image analysis allows the assessment of textural alterations produced by acid rain.

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ABSTRACT

This paper aims to determine the physical performance and rain resistance of earthen plasters made with a red clayey binder, rich in silico-aluminates and a yellow binder which is mainly calcareous. The nature of the binder had little influence on practical properties of plasters, such as density, consistency, shrinkage, consumption, thermal conductivity and capillary water absorption. However, important properties of the plaster as for instance, the cohesion of the surface were affected by the type of binder. In addition, the plasters durability was evaluated by rain erosion tests conducted with distilled water and acid rain simulated with hydrochloric and sulphuric acid solutions (5% w/w). It was found that earthen plasters showed a better resistance to sulphuric acid than to hydrochloric acid. Scanning electron microscopy and X-ray diffraction analyses confirmed the formation of gypsum coatings on the surface of plasters attacked by sulphuric acid. In this case, the erosion was smaller due to the passivating role of gypsum, which protects the underneath material from dissolution. The textural alterations assessment, evaluated by optical microscopy and image analysis software, showed that hydrochloric acid rain was the most aggressive attack, followed by sulphuric acid rain and rain water.

1. Introduction

Earthen materials comprise a wide variety of uses and typologies, such as adobe, rammed earth and plasters. The rising interest in earth-based building materials can be explained by a combination of factors, among which the raw materials availability is essential. They are abundant, cheap and eco-friendly materials as lower energy is needed in their quarrying and manufacture comparing with common raw materials (e.g. limestone, granite). Moreover, earthen materials are an excellent choice to regulate temperature and humidity at comfortable levels [1–3]. They are often found in heritage constructions, thus motivating further research on both preservation criteria and methods for testing durability. In this respect, earth-based plasters present inherent limitations, such as low mechanical strength and limited durability against atmospheric agents [4]. In addition, during the drying stage period, cracks may appear due to the volumetric instability of clay [2,5]. In fact, the occurrence of cracks is a serious limitation in plasters, especially when the binder/sand ratio is not properly adjusted in the designing phase [2].

The addition of stabilisers like such cement, lime, fly ash, wastes or coal ash has been proposed to improve a number of features in earthen materials [3,6–8]. It is worth noting that soils are natural materials with a heterogeneous composition, including clay, sand and gravel. Therefore, the optimisation of standard procedures

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for preparing plasters, adobe or earthen-walls is more difficult than that used for cement-based materials. For instance, the chosen soil (binder) would meet several properties regarding mineralogical composition, clay/sand ratio and granulometry. Usually, the binder dosage and aggregates' grain size must be considered for the intended use. In plasters, for instance, the amount of binder should be reduced as much as possible to avoid cracks formation. Similarly, enough compressive strength is required for earthen-walls or adobe bricks taking part in load-bearing or structural elements of buildings. In this case, the dose of binder is clearly a conditioning factor as previously reported by different authors [9,10].

Besides, the mechanical behaviour of earthen materials is influenced by additional factors like manufacturing process, use of stabilisers and curing conditions. Emiroglu et al. [2] evaluated the effect of the clay/sand ratio on the performance of clay plasters and found that the compressive strength was lower for samples with lower clay content. Moreover, samples dried at 60 °C had lower compressive strength than those dried at 100 °C. El-Mahllawy and Kandeel [3] studied the characteristics of clay-sand formulations stabilised with Portland cement and quicklime in different proportions and humidity (85% and 50%). The best mechanical performance was found in samples cured at 85% and moreover, the amount of cement and curing time were critical factors too.

The low rain water resistance of earthen materials [10] is clearly an important limitation of earthen plasters. Although specific tests for measuring the rain water erosion are scarcely addressed, a number of methods have been proposed in the literature to extend the durability of earthen materials. For instance, the use of zinc stearate or polymeric additives has been found to be effective to extend the lifespan of plasters and adobes [4,11]. In the same way, natural agents like resins (latex), animal blood, bitumen or oil have been proposed as effective solutions to improve a number of properties in earth-based materials [8,12,13]. The use of coatings, such as nanostructured emulsions, silane-siloxane emulsions and nanolime suspensions has been studied for strengthening and protecting the surface of earthen materials [13,14]. Finally, phosphogypsum wastes have been confirmed as excellent admixtures for improving both the mechanical resistance and durability of adobes [15,16].

The literature on earthen plasters is quite scarce and very few studies report on durability of plasters, particularly as it relates to acid rain attack. This paper is focused on three main aspects: i) the chemical characterisation of the clayey binder, ii) factors that influence on the physical properties of earthen plasters, and iii) durability of earthen plasters against rain water and acid rain. The curing conditions had a significant effect on the compressive strength, ultrasonic speed, and Young's modulus of plaster samples, whereas the nature of the binder had little effect on practical properties of plasters often provided in technical data sheets. However, functional properties related to durability, such as hardness and rain resistance were to some extent affected by the nature of the binder. The formation of gypsum patina on the surface might explained the additional resistance of plasters against acid rain. Microscopy examination and image analysis software are consistent with the erosion data obtained in rain tests.

2. Materials and methods

2.1. Materials and curing

In earthen materials, the binding behaviour is attributed to the clayey phase and therefore, the terms *binder* or *clay* are used interchangeably. The specimens were made with either red or yellow earth-based binder, two types of sands – termed as sand A and sand B – and distilled water. The binder was a sieved soil fraction (<0.5 mm) used for the manufacture of industrial bricks. The sands, provided by Reverté Minerals (Spain), were eminently calcitic and classified according to the EN 933-1 standard [17]. A previous study was carried out for determining the optimal dosage of binder to avoid shrinkage to occur as much as possible. The optimal composition for the dry mixture consisted of: clay 10% w/w; sand A 30% w/w; and sand B 60% w/w. To obtain adequate workability water was added in a ratio of 16% w/w on the amount of the dry mixture.

Several formats of specimens were confectioned depending on the intended use of the samples. For the mechanical characterisation 4x4x16 cm specimens were prepared in normalised moulds. The specimens were left in the moulds under laboratory conditions (24 ± 2 °C) for 7 days so that the initial hardening might be achieved. After that, they were de-moulded and heated at 60 °C for 24 h, thus completing the hardening process, which basically consists in densification due to water evaporation. As is the usual practice in plasters, a second set of samples was prepared spreading the fresh paste on 12 × 24 cm ceramic surfaces using uniform thickness of 10 ± 1 mm (Fig. 1a–b). Equally, the plaster samples were stored under laboratory conditions and heated at 60 °C for 24 h.

2.2. Methods

2.2.1. XRD, XRF and TG-MS characterisation

The mineral phases present in the binders were analysed by X-ray diffraction (XRD) using a Bruker D8 Advance diffractometer in θ- θ mode. The spectra were registered from 10 to 70° at 0.05° stepping intervals and the X-ray tube was operated at 40 kV and 30 mA using the Cu K-alpha line as source of radiation. The diffraction patterns were evaluated with DIFFRAC.EVA 4.0 software and powder diffraction database PDF4+ (ICCDD, 2015). In addition, the chemical composition of the binders was studied by X-ray fluorescence (XRF) with a Bruker S4 Pioneer fluorescence spectrometer. Finally, thermogravimetric analysis coupled to a QMS 200M3 quadrupole mass spectrometer detector (TG-MS) was used for determining volatile components like H_2O and CO_2. The measurements were performed with a TGA/DSC 1HT Mettler-Toledo thermogravimetric analyser.

2.2.2. General properties: density, consistency, shrinkage, consumption and thermal conductivity

The apparent density of the samples was calculated from cured prismatic specimens dividing their mass by the corresponding volume. Flow table measurements were used to determine the workability of the fresh plasters following the standardised method for determining mortars consistency [18]. Furthermore, the plasters' shrinkage was determined from prismatic specimens once the curing period was completed (n = 3). To do this, the prisms were measured with a calliper along their axes (±0.01 mm resolution) and the data were expressed as percentage variation with respect to the initial length of the specimen. Another practical feature of renders is the actual surface which is coated by the fresh paste, a parameter also referred to as consumption. To investigate this, the samples were spread on 12 × 24 cm ceramic surfaces at uniform thickness as shown in Fig. 1a–c. The thermal conductivity was analysed using a thermal conductivity analyser C-Therm TCi equipped with a MTPS sensor accessory.

2.2.3. Ultrasonic propagation speed, Young's modulus and compressive strength

Ultrasonic propagation speed, Young's modulus and compressive strength were conducted for different curing conditions with the aim of studying the influence of the environment on the materials' physical properties. In this case, the specimens were cured for 28 days at: i) 20 °C/30% RH; ii) 20 °C/60% RH; and iii) 20 °C/95% RH.

Ultrasonic speed measurements provide information about the material stiffness i.e., Young's modulus. The ultrasonic pulse tests were performed following the UNE-EN 12504-4 standard [19] and three determinations were made per sample. This test consists in measuring the time, in μs, necessary for the ultrasonic waves to cross the sample. Contact transducers emitting ultrasonic pulses at 54 kHz were coupled to the lateral sides of the specimens using a coupling agent to increase the reproducibility of the measurements. The wave speed was obtained from the size of the specimen and the propagation time. The Young's modulus was calculated from the theoretical equation:

$$E = 10^{-6} \cdot v^2 \cdot \rho \quad (1)$$

where,

E is the Young's modulus, in N/mm^2.

v is the propagation speed of ultrasonic waves through the material, in m/s.

ρ is the density of the sample, in kg/m^3.

The compressive strength tests were performed on 4x4x16 cm specimens according to the UNE-EN 196-1 standard [20] at a rate of 1 kg/s using a universal testing machine. Since earthen materials typically have low mechanical resistance, a 2 kN load-cell capacity was installed in the testing machine to increase the accuracy of the measurements.

2.2.4. Surface properties: peeling test, hardness and capillary water absorption

The peeling test [21,22] was used to assess the cohesion and consolidation characteristics of both red and yellow plasters. For this purpose, equivalent formulations were spread on clean ceramic substrates as shown in Fig. 1a–d. Three specimens were confectioned per type of plaster (red or yellow) and three test zones were defined on the plasters' surface. Adhesive tapes of 20 x 50 mm were previously