MICRO AND NANOSTRUCTURAL CHARACTERIZATION OF SURFACES AND INTERFACES OF PORTLANT CEMENT MORTARS USING ATOMIC FORCE MICROSCOPY

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ABSTRACT

The characterization of Portland cement mortars is very important in the study the interfaces and surfaces that make up the system grout/ceramic block. In this sense, scanning electron microscopy and energy-dispersive (X-ray) spectrometer are important tools in investigating the morphology and chemical aspects. However, more detailed topographic information can be necessary in the characterization process. In this work, the aim was to characterize topographically surfaces and interfaces of mortars applied onto ceramic blocks. This has been accomplished by using the atomic force microscope (AFM) – MFP-3D-SA Asylum Research. To date, the results obtained from this research show that the characterization of cementitious materials with the help of AFM has an important contribution in the investigation and differentiation of hydrated calcium silicates (CSH), calcium hydroxide (Ca(OH)2, ettringite and calcium carbonate by providing morphological and microtopographical data, which are extremely important and reliable for the understanding of cementitious materials.

Keywords: Microstructural characterization, mortar, atomic force microscopy, interfaces and surfaces, cementitious materials.

1. INTRODUCTION

The need for research and characterization of materials aimed at the construction has been growing due to the need for proper selection of materials, based on the performance of Portland cement based materials, as well as the increasingly shortage of raw materials and market demand for products with adequate performance and durability. Only the evaluation of mechanical properties such as tensile strength tension of mortar is not enough to ensure proper performance of rendering mortar systems, especially in Portland cement materials that have a micro and nanostructures still little explored. In this sense, this paper proposes to contribute on the microstructure characterization of micro/nanostructural aspects of Portland cement mortars used for flooring in civil works using a new approach: by means of atomic force microscopy (AFM) as a tool for the characterization of Portland cement mortar.

2. PORTLAND CEMENT MORTAR

The rendering mortar more used in construction is composed of cement, aggregate and water, called cement mortar and cement, lime, aggregate and water, additives may be added to improve its characteristics. There is also a lime mortar composed only of lime, aggregate and water, which is little used nowadays

2.1 Atomic force microscopy (AFM)

The atomic force microscope (AFM) can be considered as a hybrid between the equipment for measuring the force on surfaces (SFA), and a surface profilometer [1] [2]. The AFM can operate in the work scheme known as contact, similar to the profiler mode, but with much lower power load.

The AFM or scanning force microscope (SFM) can be operated in various ways. However, its basic principle is to measure the deflection of a cantilever with 100 to 200μm in length, whose free extremity is mounted on the probe. These deflections are caused by forces acting between the probe and the sample [3].

When the probe approaches, the sample is first attracted by the surface due to a wide range of attractive forces existing in the region such as van der Waals forces.

This attraction increases until the tip is very close to the sample, then, the atoms of the two masses are so close that their electronic orbitals begin to repel. This electrostatic repulsion weakens the attractive force as the distance decreases. The force vanishes when the distance between atoms is of the order of a few angstroms, the characteristic distance of the order of a chemical bond. When forces become positive, it can be said that the atoms of the tip and the sample are in contact and the repulsive forces eventually dominate [3].

Various types of forces contribute to the deflection of a cantilever in an AFM, as already mentioned [4].

To date there is no record of the use of atomic force microscope in research involving the characterization of masonry blocks used in construction, for this reason and for believing in the importance in investigative potential and uniqueness of this technique, this work has used AFM in the characterization of extremely small morphological aspects of the blocks and mortars under study.

3 CHARACTERIZATION OF PORTLAND CEMENT MORTAR WITH THE USE OF ATOMIC FORCE MICROSCOPY (AFM)

One of the main advantages of the technique of atomic force microscopy is being able to work in different conditions and for studies of the properties of various materials in the ideal conditions for them, ie samples at atmospheric pressure, since no vacuum is needed, and especially in controlled environmental conditions.

Because it provides morphological 3D data, the technique allows to analyze the morphology, the microtopography and the geometric surface area [5] of solid samples. These factors help to study the physical characteristics and chemical reactivity of mineral phases, thus being able to control them through specific physicochemical procedures. Hence, mortar samples are analyzed by this technique in an unprecedented manner.

3.1 Equipment used for the characterization

The atomic force microscope used is installed in the Microscopy Center of UFMG, as follows:

a) atomic force microscope – MFP-3D-SA ASYLUM RESEARCH – Modes Contact, Non-Contact and Semi-Contact (intermittent) in air; scan (x and y) of up to 90 micrometers, with isolation of vibration and external noise; nanoindentation.

3.2 Preparation of the samples of Portland cement mortar

Three panels of masonry were built and mortar was applied over them with roughcast trait (1:3) of Portland cement and washed river sand: on top of these, three different coatings were further applied, one for each panel: a) a plaster cladding with stroke (1:6) of Portland cement and washed river sand; b) a mortar coating (1:1:4) of cement, lime and sand; c) a mortar lining with a stroke (1:2:8) of cement, lime and sand.

For sample preparation, approximately 12cmx12cm pieces of the panels containing the complete system, ceramic block, roughcast mortar and mortar coatings. After the removal, the 12cmx12cm samples were fragmented and reduced to approximately 6,5cm. Sample fracture was used instead of cutting hard, not to interfere with the sample surfaces.

After fragmented, the samples were prepared for analysis in the atomic force microscope (AFM). The preparation followed the basic procedures of ceramic/rock samples, that is, the mortar samples were impregnated within epoxy resin and then polished as seen in Fig. 1.

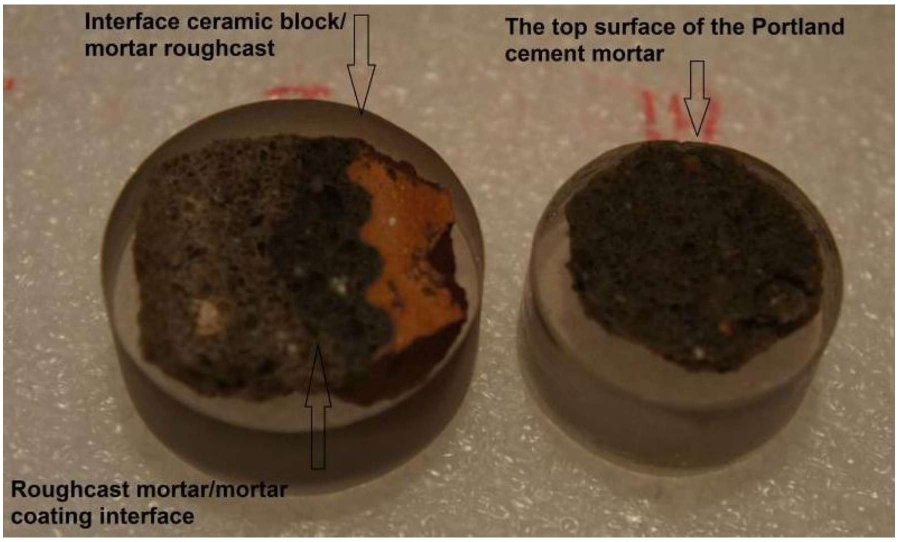

Figure 1 – Portland cement mortar samples prepared for AFM.

4 RESULTS

From the analysis of the Portland cement mortars samples in the atomic force microscope micrographs such as Fig. 2A were obtained; this photo corresponds to the morphology of CSH present in the sample near the interface and in Fig. 2B, a group of ettringite crystals surfacing from inside a pore interface.

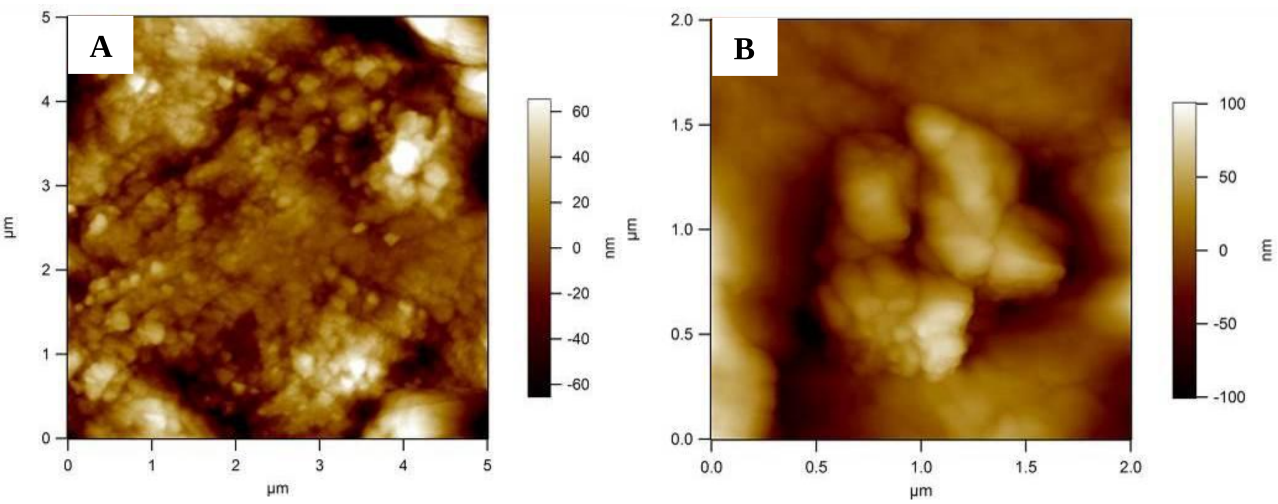

Figure 2 – Micrographs of the microtopographic profile of the region close to the ceramic block/mortar interface.

Figure 3A shows the surface of the region near the ceramic block/mortar interface and Fig. 3B, the three-dimensional (3D) image of the topographic profile shown in Figure 3A. The morphology suggests portlandite and calcite.

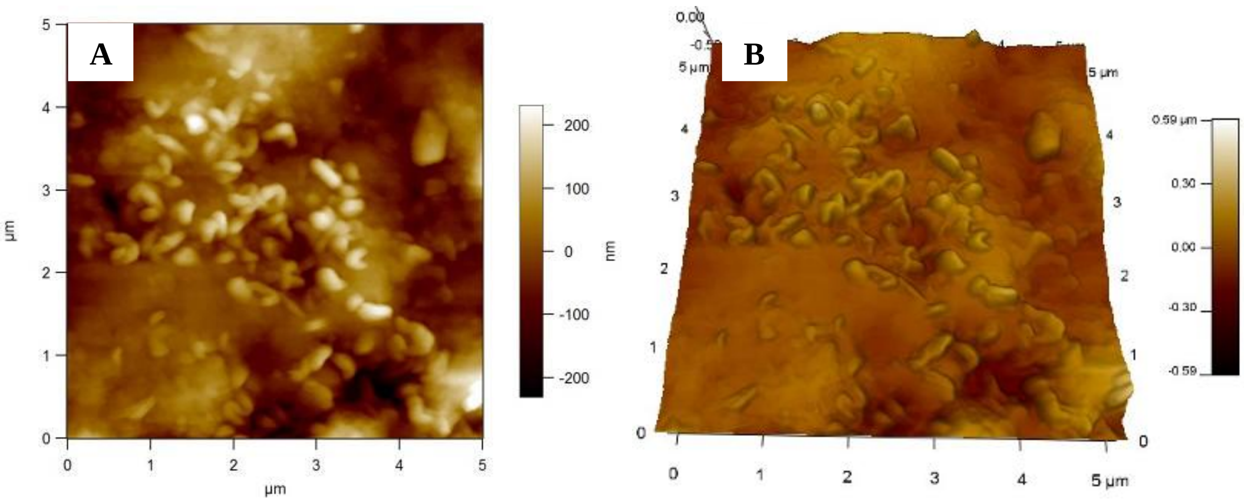

Figure 3 – Micrographs: A) the microtopographic profile of the region close to the ceramic block/mortar interface; B) 3D topographic profile of the region close to the ceramic block/mortar interface.

Figure 4A shows the topographic profile of the region near the ceramic block/mortar interface and Fig. 4B, the 3D version of the same region. The morphology clearly indicates oriented ettringite crystal groups.

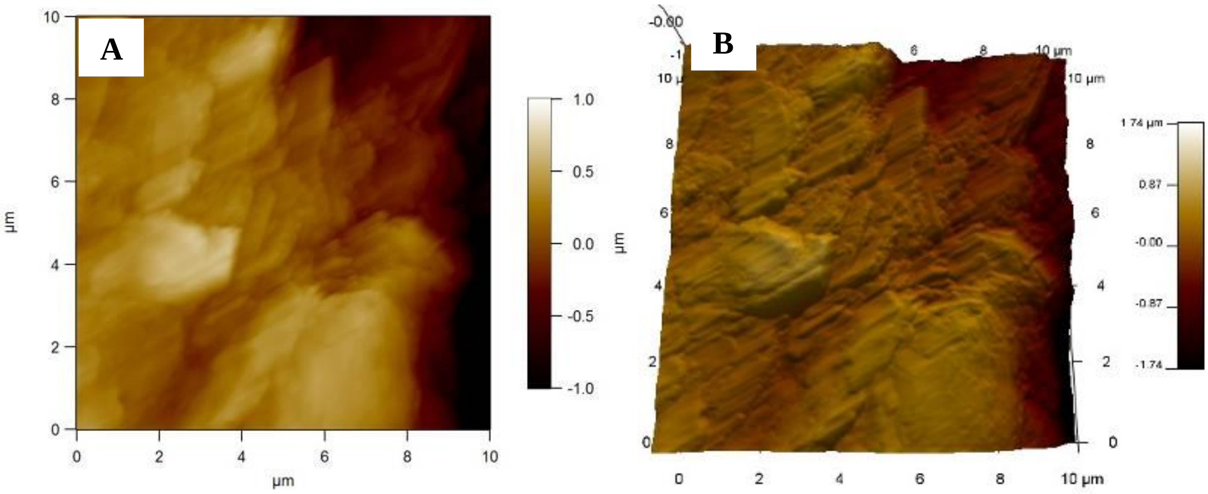

Figure 4 – Micrographs: A) the microtopographic profile of the region close to the ceramic block/mortar interface; B) 3D topographic profile of the same region.

A typical CSH area is seen in Fig. 5, showing very fine, colloform or botryoidal morphology. This aspect, consisting of minute spherules coalesced, is consistent with the amorphous nature of the hydrated calcium silicates (CSH).

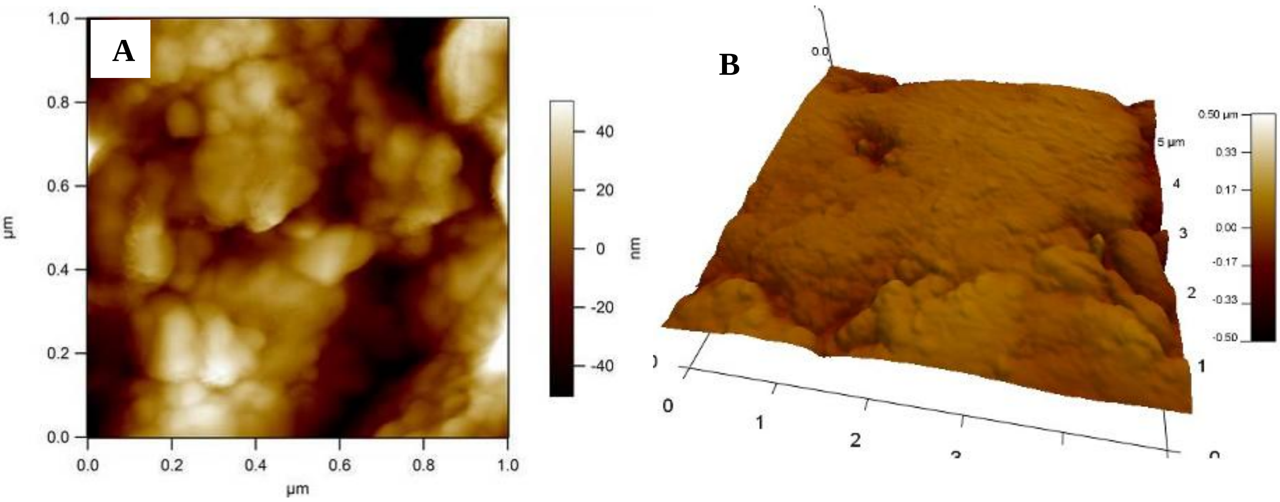

Figure 5 – Micrographs: A) the microtopographic profile of the region near the roughcast mortar/mortar plastering interface, B) 3D topographic profile of the same region.

5 CONCLUSION

The atomic force microscopy (AFM) was used in this work in an unprecedented way for cementitious materials, here for Portland cement mortars. The samples preparation showed positive results, even though a simple procedure was used, which consisted of fracture mortar fragments impregnation in resin and doing the surface polishing.

The microtopographic characterization proved important because it showed the classical morphology of the important phases, such as ettringite, portlandite and calcite in the samples at the nanometer scale. Also, the typical morphology indicating CSH, as very fine, colloform or botryoidal morphology. This aspect, consisting of minute spherules coalesced, is consistent with the amorphous nature of the hydrated calcium silicates (CSH) and is harder to be observed with other microscopy methods like scanning electron microscopy.

These microtopography micrographs and the corresponding three-dimensional profiles have provided very good morphological pictures of the cementitious materials at the ceramic block/mortar interface and at the equivalent roughcast mortar/ rendering mortar. Therefore, the ATM technique demonstrated to be a powerful and effective tool for micro and nanostructural characterization for the Portland-based materials.

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7 REFERENCES

[1] ISRAELACHVILI, J.N., Intermolecular and surface forces. 1 ed. San Diego: Academic Press, 1992. 450p.

[2] TEAGUE, E. C., SCIRE, F. E., BAKER S. M., JENSEN, S. W., 3-dimensional stylus profilometry. Wear, v.83, 12p, 1982.

[3] ORÉFICE, R. L., PEREIRA, M. M., MANSUR, H. S., Biomateriais; fundamentos e aplicações. 1.ed. Rio de Janeiro: Cultura Médica, 2006. 538 p.

[4] HERRMANN JR., P. S. P., COLNAGO, L. A., MATTOSO, L. H. C., VAZ, C. M. P., RUVINEL, P. E., A potencialidade da microscopia de varredura por sonda na pesquisa agropecuária. São Carlos: EMBRAPA. ISSN 1517-4778. 1999. 29p. (Circular Técnica).

[5] VAZ, C.M.P., HERRMANN, P. S.P., CRESTANA, S., Soil particle thickness and size distribution measured through atomic force microscopy. In: 1998 ANNUAL MEETING, Oct. 1998, Baltimore, Maryland, USA. Abstracts... Baltimore: ASA/CSSA/SSSA, 1998. p.180.