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PERMEABILITY CHARACTERIZATION OF CONSTRUCTION MATERIALS USING ADVANCED METHODS

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Abstract

Advanced ^1H Nuclear Magnetic Resonance (NMR) relaxometry is a valuable tool for the characterization of building materials like waterproof mortars. Carr-Purcell-Meiboom-Gill (CPMG) pulse sequence method was used for the measurement of four samples (C65, P88, P288, AQSE) of waterproofing mortars at 7 days after preparation. In general, four dynamics components were observed for the T_2 Laplace distributions at 7 days after preparation. Their characterization shows the evolution and the mobility of protons in the samples. In general, these peaks have T_2 values which can be associated with bound water, free water in small pores, and free water in medium and large pores. The Scanning Electron Microscopy (SEM) was measured for all samples and correlated with NMR parameters.

***Key words**: CPMG, mortars, NMR, pore evolution, relaxometry, SEM, T_2, waterproofing.*

INTRODUCTION

Characterization of building materials has become an increasingly widespread practice in the last decade due to their applicability. Low-field NMR is one of the methods recently used in this characterization and it is a non-invasive method (Moldovan, 2012). 1H NMR method brings a great and quality contribution to the understanding of hydration phenomena. Their characteristic shows the dynamics and mobility of protons (Jumate et. al., 2017; Ardelean, 2021; Fechete 2009).
Mortar cement is a mixture of water, aggregates (different sizes of the particle of sand), and cement materials in different proportions. Water is the main component in producing mortars because it contributes to the chemical hydration reaction between solid compounds (Jaffe, 2001).
Waterproofing mortars are used for many applications in the field of construction. The behavior of waterproofing mortars, after application on the surface, is different due to the use of aggregates and polymers from different sources, even if it satisfies the waterproofing needs of the substrate and meets quality standards (Cadar at al., 2021). Water retention capability is the capacity of the mortar to sustain a rapid loss of mixing water to air and absorbent masonry materials (Pop, 2022).
The purpose of this paper is to characterize four different mortars by NMR and to determine the proton dynamics in the samples, to understand what happens at the microscopic level. Three important characteristics of mortar are constructability, workability, and compressive strength. Their degree of impermeability is given by the rigid components in the sample.

MATERIALS AND METHODS

The four waterproofing mortars C65, P88, P288, AQSE, see Figure 1 were purchased from the local market in Romania. From the preparation phase, the ratio between components A (liquid) and B (solid) was different for each sample. The samples C65 and P88 are waterproofing 1k mortars, the component A is only water and in component, B is aggregate, additives, and polymers.

Figure 1. Pictures of waterproof mortars on day 7 after preparation.

Figure 2. Students Andrada and David are preparing samples for NMR measurement.

The samples P288 and AQSE are waterproofing 2k mortar, component A (liquid) is water plus additives and polymers. The samples have been prepared following the quality standard to SR EN 1015-11:2002/A1:2007 (2007).

Nuclear Magnetic Resonance measurements were performed with the Bruker Minispec low-field NMR spectrometer working at the proton frequency of 19.69 MHz (see Figure 2).

For a qualitative evaluation of the rigid component morphology and the pore dimensions, the scanning electron microscopy (SEM) images were taken at 7 days from preparation, for all samples. The SEM images were captured with a Jeol JSM-5600 LV at an acceleration voltage of 15 kV with magnifications of 1000x. The electron microscope is equipped with an Energy Dispersive X-Ray Detector (EDX), UltimMAX 65 (Oxford Instruments), operated with Aztec Software.

RESULTS AND DISCUSSIONS

In this section, we describe the NMR and SEM measurements for all samples at 7 days after preparation, but this description is a follow-up of a more extensive study done on samples during a more extended period from day 1 to day 28 after preparation to understand the dynamic behavior of the water in the samples (Cadar at al., 2021).

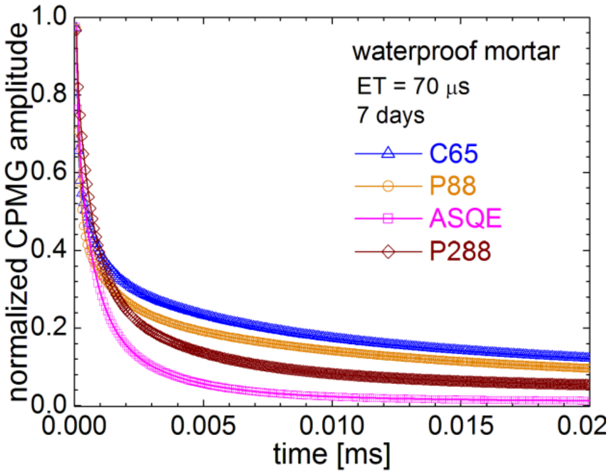

Figure 3. The normalized CPMG echoes decay for a waterproof mortar at day 7 after preparation.

Figure 3 is presented the CMPG echoes decay for all four samples with the echo time of 70 µs for 7 days after preparation. The fastest decay in the representation of Free Induction Decay (FID) measured data is for the ASQE sample and the slowest decay is the C65 sample with magenta respectively blue in figure 3.

To monitor the effects of water consumption and the evolution of pores the curves depicted in Figure 3 can be analyzed using a numerical Laplace inversion algorithm (see Figures 4a and 4b).

In this figure, one can observe a *T2* distribution which is more like that one measured on day 1 after preparation and less than those measured on day 3 for the same P288 mortar according to the previous measurements and characterizations (Cadar at al., 2021). To validate this behaviour, the distributions were measured again for another set of P288 samples, but the result is consistent. The *T2* distribution measured for the sample P288 at 7 days is like those measured for the sample AQSE at 7 days. The difference consists in the fact that the peaks obtained for the sample P288 are better resolved indicating that the mortar P288 is more homogeneous although they are in the same category of 2k mortars. AQSE sample has four peaks but three of them are not so well resolved

(see figure 4a with red). The peaks overlap slightly which indicates that at 7 days after preparation the hydration process has not finished, and water is still migrating from one pore to another. They are between 0.01ms and 0.01s. The peak for the *T2* value under 1 ms can be associated with bound water. A *T2* value of the order of milliseconds is water in small pores and medium pores. The quantity of water content in large pores is quite low in the ASQE sample as compared to the other samples which have a higher water content in these pores.
The NMR data show that the behaviour is similar in these samples even if the amount of water is different in each of them. Also, the amplitude is different. The lowest amplitude of peaks associated with bound water is found in the AQSE sample.
In figure 4b the amplitudes are approximatively equal at both samples and the peaks are well defined. The peak associated with bound water for the C65 sample is at 0.7ms instead for sample P88 which is at 0.1ms.
The pore size and the morphology of the samples were evaluated qualitatively from Scanning Electron Microscopy (SEM) like in figure 5 up.

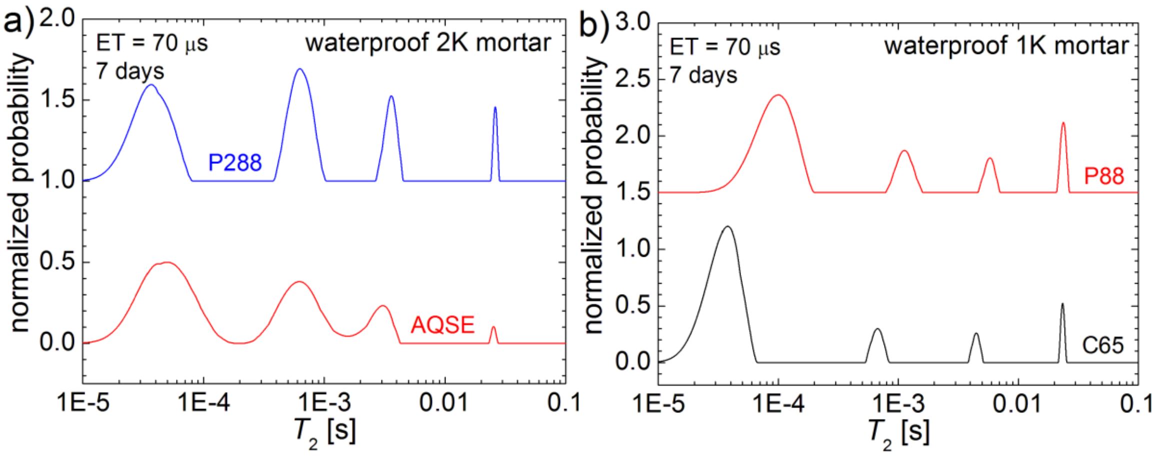

Figure 4. T2 distribution measured for a) waterproof 2K mortar P288, AQSE and b) waterproof 1K mortar P88 and C65.

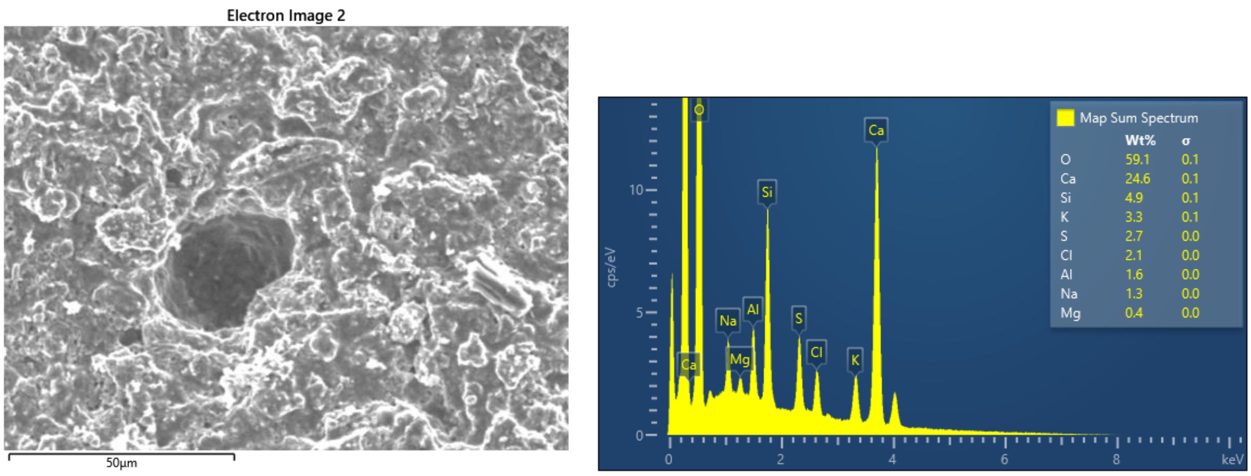

Figure 5. SEM micrographs, at ×1000 magnification for P88, waterproof mortar at day 7 after preparation up to and down the map spectrum in the function of the chemical components

This figure reveals that the homogeneity is high, but the hydration process is not finished yet. The pore sizes are different depending on the sample.
In figure 5 down we can see the chemical structure of sample P88, the highest proportion has 59.1% of oxygen and 24.6% of calcium. The smallest weight has Mg with 0.4% being alkaline-earth metal is supposed to make the sample more rigid.
These NMR and SEM characterizations will

continue, and they will be correlated with measurements at 28 days after preparation since the hydration process was not completed at 7 days in any of the samples. The permeability will be characterized and correlated with classical mechanical tests of building materials, but this will be in future research.

CONCLUSIONS

The 1H NMR Laplace distributions were proved to be a valuable tool to investigate the evolution of permeability of the building's materials. The *T2* distributions reveal changes, in the waterproofing 1k and 2 k mortars. To 7 days after preparation four dynamics components were observed in the *T2* distributions for all samples. AQSE mortar is the most heterogeneous in the early stages of evolution after preparation.

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