

Experimental report

15/04/2024

Proposal: 1-07-6

Council: 10/2022

Title: Permeability Enhancing Mechanisms of Organic Fibers in Refractory Castables

Research area: Engineering

This proposal is a new proposal

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Samples: High Alumina CAC bonded castable

Instrument	Requested days	Allocated days	From	To
NEXT	3	1	17/04/2023	18/04/2023

Abstract:

From the drying of refractory castables, to the safety of concrete structures under fire and even to the performance of rockets, launching pad, the pressurization of water in partially saturated media is crucial for the explosive spalling. One of the most common strategies to decrease its likelihood is the use of organic fibers leading to a more permeable microstructure, and possibly reducing the mechanical strength at high temperatures. The aim of this study is to quantify the water migration in refractory castables with different fibers at high temperatures thanks to the high neutron attenuation of hydrogen at a speed matching the intrinsic speed of the micro-scale. The different hypothesis explaining how the fibers behave during heating can be validated providing guidelines for the selection of the best polymer as a permeability enhancing additive. Besides the intrinsic scientific interest, technological advances such as efficiency gains in the processing of refractory castables can create noticeable positive impact in different segments, as well as improving the safety of high-performance concrete structures at high temperatures, such tunnels and nuclear reactor walls.

ILL EXPERIMENTAL REPORT PROPOSAL 1-07-6

Permeability Enhancing Mechanisms of Organic Fibers in Refractory Castables

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1 Introduction

Hydraulic bonded refractory castables are used as linings of vessels for high temperature processes often found in the heavy industry [1]. For calcium aluminate cement castables, the main challenge for its adoption in the industry and its carbon dioxide emission reduction is the drying stage of the installation process [2]. If the rate of thermal energy supply is too high, the vapor generation from free water evaporation and dehydration of the products from the cement and water reactions can outpace the moisture flux to the surrounding environment, leading to pressurization [2, 3]. It is currently believed that once the combination of vapor pressure and thermomechanical stresses exceeds the material's strength, explosive spalling occurs, leading to damage and even total destruction of the lining [3].

This scenario leads to slow heating-up protocols [3]. Hence, in the industrial context, the use of polypropylene (PP) fibers is common thanks to their low price and (empirically observed) effectiveness to reduce damage during drying [2] linked to the increase of the castable's permeability observed upon heating, through mechanisms that are yet not well understood [4].

Provided that the sequence of events that control this permeability increase is understood, purposefully engineered fibers for drying additives emerges as a viable solution leading to lower carbon footprint and improved efficiency of the heavy industry process.

Thus, the objectives of the current experimental report were i) to compare the mass transport behavior of unidirectional dried refractory castables samples containing Polyethylene (PE), Polypropylene (PP) and Cellulose (Cel - a natural alternative to the synthetic polymers) using neutron tomography; ii) assess the reproducibility of the neutron tomography drying experiment for samples comprising drying additives; and iii) evaluate if the current experimental setup can be used to describe the permeability enhancing mechanisms of polymeric fibers in refractory castables.

2 Materials and Methods

For the sake of brevity, the complete description of the sample preparation, the sample environment and the neutron images acquisition configuration can be found in the published work from the proposers, Moreira et al. [5].

The setup for the heating tests follows the same approach already described by Dauti et al. and Tengattini et al. [6], with a heating rate of the infrared heater of 20°C/min up to 600°C, reaching a plateau afterward for 30 additional minutes. Some of the tests were ended earlier once the drying front reached at least one third of the sample. Finally, the details for the post-processing can be found in [6, 5].

3 Results and Discussions

In total, 10 tests were conducted in the NeXT equipment, and the evolution of the mean raw intensities can be seen in Figure 1. It can be seen that overall the results are reproducible, highlighting an initial increase of the intensity followed by a drop. Some tests represented a larger noise. This behavior was also detected on the voxels outside the sample and upon talks with the specialists at ILL, it was discussed as possible explanations: i) adjusts of the monochromators of instruments upstream the neutron line (as it was the last night of the reactor cycle), or ii) something related to the observed fluctuations on the reactor power output. Upon checking with the upstream equipment operators, no changes on the monochromators were reported.

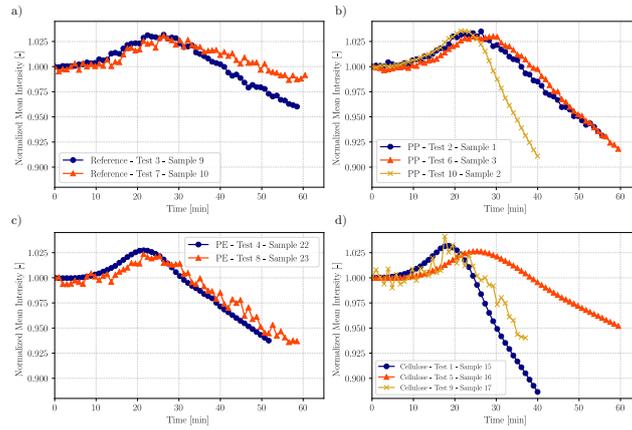


Figure 1: Evolution of the mean raw intensities for compositions a) 5CAC, b) 5CAC-PP, c) 5CAC-Cel and d) 5CAC-PE.

Finally, Test 5 and 10 were seen as outliers, with the distinct behavior - slower one in Test 5 and faster drying observed in Test 10 - attributed to moisture loss during aging of the samples and detachment of the sample from the ceramic casing, respectively. Thus, in the following analysis, these results are disregarded.

The quantitative analyses of the results can be summarized by Figure 2 that shows the average of the voxel intensity over the whole sample at a given height as a function of the drying time. The reference composition, Figure 2 a) shows the slowest water removal and the second-smallest moisture accumulation at the innermost position (being only more intense than the 5CAC-Cel sample), while still being the broadest accumulation zone (25mm). Because of that, the secondary drying front (moving from the bottom towards the top surface) in the reference composition can reach the highest depth of the sample for all compositions.

The addition of PP starts to change the drying dynamics only after the 20-minute mark, as seen in Figure 2 b), when the temperatures within the sample are high enough to activate the permeability enhancing mechanisms. Notably, due to the unidirectional heating boundary conditions, the bottom of the 5CAC-PP sample is at lower temperatures (around 150°C), leading to a permeability level similar to the reference case. Nevertheless, the behavior in this region is different from the 5CAC-Ref case, given that the water migrated from the top towards the center of the sample, leading to a moisture accumulation zone that extends to the lower surface.

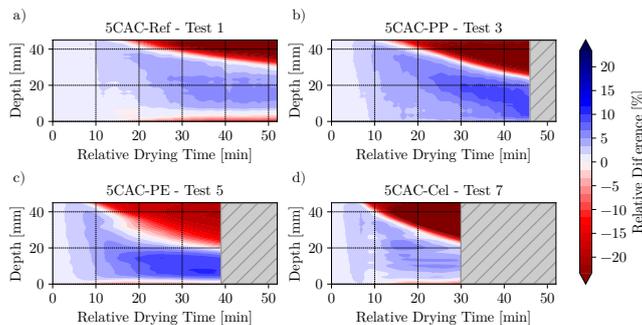


Figure 2: Average of the attenuation coefficient over slices 3.8mm thick in the axial direction for a given height as a function of time. (a), (b), (c), and (d), present the results for the reference sample, the one with PP, PE, or Cel, respectively. Higher values of difference (dark blue) indicate water accumulation, whereas lower values (dark red) represent drying.

Figure 2 d) presents the results of the sample containing cellulose with the smallest water accumulation both in extension and in intensity, suggesting a permeability-enhancing mechanism that is triggered at lower temperatures, inducing a steep increase in the drying rate at early temperatures due to its shrinkage.

Figure 2 c), shows the distinct features of the sample with PE. The drying front progress is significantly faster than 5CAC-Ref and 5CAC-PP, but it is more diffuse (as indicated by the smoother transition between red and blue and by the larger white strip) and less intense (lighter red region behind the white drying front). This shows a reduction of the moisture flux, which limits the benefit of the PE addition as highlighted by the most intense moisture accumulation of all the tests. This effect is also more localized as shown by the secondary drying front at the bottom of the sample which

reaches its maximum height around 15 minutes, and then starts to recede towards the bottom, ultimately stabilizing there, conversely to the constant rate of advancement towards the center observed in the 5CAC-Ref and 5CAC-Cel samples.

This behavior could justify the PE worse performance in avoiding explosive spalling, which was observed both by Bezerra *et al.* [7] and Zhang *et al.* [8].

This behavior underscores the dynamic and varying nature of permeability during the drying process, which raises the question whether the point-wise measurements of permeability at controlled temperatures and in stationary conditions are truly representative of the complex nature of the drying process.

4 Conclusions

The use of neutron tomography for assessing the drying of refractory castables comprising polymeric fibers was found out to be a viable option for *in-situ* observations. The results were reproducible and were used to better understand the unique behaviors attributed to the fiber's properties. Further studies with higher resolution could reveal the local effects of these fibers and provide deeper understanding on the different mechanisms of permeability enhancement at play.

5 Scientific Production

The current project results have enabled the presentation of two proceedings in international conferences (UNITECR 2023 "Permeability-Enhancing Drying Additives - A Perspective from In-Situ Analysis" - and PACC-FMAs 2024 - "Polymeric Fibers Effect on the Unidirectional Drying of Refractory Castables via Neutron Tomography") and scientific paper in the Journal of the American Ceramic Society ("Neutron Tomography Analysis of Permeability-Enhancing Additives in Refractory Castables"), currently accepted with minor revisions, which was pre-selected for the Rising Stars Competition which aims to recognize the best work from emergent researchers in ceramic and glass science and engineering at all levels.

6 Suggestions of Further Tests

- Consider a model material with smaller aggregates and thicker polymeric fibers to enable the direct observation of local effects on the moisture distribution;
- Reduce the sample and the sample environment setup sizes to enable higher resolution neutron tomographies;
- Execution of simultaneous X-ray tomography to assess the porosity evolution.

References

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