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Characterization of EPS lightweight concrete microstructure by X-ray tomography with consideration of thermal variations

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HIGHLIGHTS

- Use of X-ray micro-tomography for a morphological characterization of EPS mortar.
- Material porosity using image processing and assessing the REV as $10 \times 10 \times 10\ mm^3$.
- Cellular structure observation of EPS beads and their good adherence using SEM.
- Temperature higher than 90 °C alters EPS beads structure and causes their shrinkage.

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ABSTRACT

The use of recycled polystyrene beads in cement-based materials is a solution to improve the insulation in buildings and reduce the energy consumption. This also helps limiting landfill by reusing the polystyrene waste.

This paper presents a refined morphological and thermal characterization of a polystyrene-based mortar using X-ray micro-tomography where a dedicated X-ray scanning and volumetric processing protocol was developed. All the reconstructed volumes had a mean voxel size between 10 and 20 µm. In complementary procedure, the interactions between the different phases of the material have been investigated, by observing the interface using scanning electron microscopy (SEM). After a set of volumetric analysis, the obtained representative elementary volume (REV) was $10 \times 10 \times 10\ mm^3$. The microstructure (porosity, pore diameter and interface, etc.) has been studied by 3D image analysis. The material's response to thermal solicitations at a local scale has also been examined, by observing its microstructural variations under thermal loads using an optical microscope. The micro-tomographic results allow us to assess a representative elementary volume for the polystyrene mortar, to evaluate its total porosity using two different approaches, and to have access to the pore size distribution. The SEM images show a good adherence between the two phases of the material, and the microscopic tracing under thermal solicitations indicates possible alterations of the beads from a temperature of 100 °C, unlike the literature results.

1. Introduction

In today's world, the need for quality housing is becoming more pressing, and it has become increasingly common to adopt alternative building materials that use waste or recycled materials, given the overexploitation of natural resources in the construction sector. Moreover, energy consumption in buildings continues to increase [1], and another difficulty to overcome is developing suitable materials in order to reduce this consumption.

Enhancing the thermal properties of construction materials is one way to improve energy efficiency in buildings.

Lightweight concrete is a good alternative for building materials in modern construction applications. It is an eco-material that greatly improves the technical, economical and environmental aspects of the construction sector. The use of lightweight concrete significantly reduces the load and weight of the structures. Compared to ordinary concrete, lightweight concrete presents many

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advantages such as: lower density, lower thermal conductivity, improved durability properties and higher acoustic insulation characteristics [2,3]. However, this type of concrete is associated with lower mechanical properties due to the very porous structure of the material, which limits their applications to non-load-bearing structures, such as curtain walls, coating panels and floor coverings. It is generally used to make non-structural insulating items.

Expanded Polystyrene (EPS) appears as a good choice to produce lightweight concrete given its good thermal properties, very low density and moisture resistance. It is commonly used for insulation and packaging of several products for protection purposes due to its flexible structure, impact absorption and mechanical robustness. However, EPS is most often released into nature after it is used, and being non-biodegradable, it does not disappear by natural means and remains present in nature for many years. For this reason, the recycling of EPS, in its natural form or after some treatments, is beneficial to the economy and to the protection of the environment. EPS is not poisonous because it does not perform chemical reaction under normal atmospheric conditions. It is not a nutrient for bacteria and fungi. Moreover, EPS is an environmental friendly material as it is recyclable and its components do not give harm to the nature and the ozone layer [4].

In the recent years, mechanical properties of polystyrene-based materials were investigated in many studies enumerated here after. Some of them focused on the mechanical performance by studying the compressive and flexural strengths of the material [5–8], the effect of the polystyrene beads size [9,10], and the strength along with durability [11–14].

Some researchers focused on the effect of polystyrene incorporation on the durability properties of this type of materials. Ferrandiz-Mas et al. [15] and Li et al. [16] carried out capillary absorption tests, heat cycles and freeze-thaw cycles on polystyrene mortars and polystyrene concrete, respectively. Their results showed that the addition of expanded polystyrene aggregates improved the durability of the materials. Fathi et al. [17] showed that the presence of expanded polystyrene in a cement paste decreased water absorption due to the hydrophobic nature of the polystyrene, thus improving its durability. More general studies carried out the characterization of the mechanical and hygrothermal properties of this type of materials [18,19], sometimes accompanied by durability tests [20–22].

The previous works focus mostly on the characterization of the polystyrene-based materials and the assessing of its durability at the macroscopic scale. In fact, it is necessary to investigate the mechanisms at the microscopic scale in order to understand its macroscopic behavior. Moreover, the material studied is multiphasic and highly heterogeneous, and a microstructural study is necessary to understand the interactions between the different phases under hygrothermal loads.

In this context, Bouvard et al. [23] evaluated the mechanical and thermal performance of a polystyrene concrete, in addition to its microstructure. They used X-ray micro-tomography to observe the different phases of the material with a voxel size of 40 μm. Lanzon et al. [24] also used X-ray micro-tomography to measure the porosity of a mortar with expanded polystyrene waste and tires, with a voxel size of 7.93 μm. They observed the different forms of porosity (open and closed) but they could not distinguish between the polystyrene and the porosity due to the non-absorbing nature of polystyrene during the scan. They also considered the polystyrene as open porosity, even though the expanded polystyrene has an internal closed pore structure.

On the other side, only few researchers were interested in the adhesion between the polystyrene beads and the cement matrix, and they did not agree on the type of the cement/polystyrene interface. On one hand, Laukaitis et al. [25] whose material of interest was a mix of cement and hydrophilizated polystyrene granulates ensured that the polystyrene beads perfectly adhere to the cement matrix. In addition, Lanzon et al. [24] studied a lightweight concrete composed of cement, crushed marble and EPS waste, and showed the good adherence between polystyrene and cement. On the contrary, Fathi et al. [17] incorporated EPS beads, scoria and nano/micro silica to cement and argued that there is a transition zone between the two phases that do not adhere at all.

It is then important to describe the polystyrene-based materials and the interactions between the different phases at a mesoscopic scale, in order to understand the behavior at the macroscopic level. It is also crucial to master the reactions and variations of the material under thermal loads at the local scale.

In this context, we propose to use recent techniques such as X-ray micro-tomography at a high resolution, to accurately measure the porosity and characterize the morphology of EPS mortar. A suitable post-processing technique is then adopted to select a representative elementary volume (REV) and to obtain the pore size distribution. Also, an investigation of the cement/polystyrene interface was conducted using scanning electron microscopy, to determine whether there is adherence between the expanded polystyrene and the cement, or it is rather a transition zone characterized by a higher porosity than that of the matrix. The material's variations under thermal loads are also examined using microscopic observations to evaluate the variation of the microstructure, and to assess its sensitivity to thermal loads.

2. Materials and methods

The lightweight material studied in this work is a polystyrene mortar (Fig. 1a) made of cement and polystyrene aggregates (Fig. 1b), mixed respecting a water/(cement + polystyrene) mass ratio of 0.32 (Table 1). The cement is of type CEM II and the aggregates are spherical expanded polystyrene beads, and their diameters vary between 2 and 3 mm. The measured density of the polystyrene mortar is 540 kg/m^3.

The expanded polystyrene beads have a spherical shape and are very porous, as they contain 98% air and only 2% polystyrene. The expanded polystyrene is a thermoplastic and hydrophobic material characterized by a closed pore structure and a very low density (between 20 and 30 kg/m^3 [26]). It has a very low mechanical strength because of the specific microstructure and the high porosity resulting from the expansion process. The main purpose of the expanded polystyrene beads is to improve the thermal and acoustic insulation properties, and they are mostly used as filling materials. Indeed, the polystyrene mortar compressive strength is only about 5 MPa, and it cannot be used as a load bearing material.

Since the main objective of this work is to study some properties and the behavior of the material at a microscopic scale, the first step is to select a Representative Elementary Volume (REV).

Jacob Bear first introduced this concept in 1972 [27], as it is the smallest volume from which the macroscopic behavior of a material no longer depends on the size of the sample. Its determination is crucial in multi-scale modeling, which is increasingly used to estimate the properties of complex and heterogeneous materials.

There are several methods for its determination, the most commonly used consists in taking as REV the smallest volume for which the porosity of the sample remains constant without taking into account the macroscopic parameters (Fig. 2).

To do so, four samples of different sizes and shapes were scanned under X-rays with different voxel sizes and parameters (Table 2) using the tomograph X50 at LMT Cachan [29]. The environmental conditions were always of 25 °C and 50% of relative humidity. X-ray tomography is a non-destructive method that allows the 3D reconstruction of the volume of the sample with several resolutions. In the literature, studies have failed to do the exact measure of the porosity of polystyrene-based materials. Indeed, water or mercury porosimetry [15] cannot provide the precise value of porosity because of the closed pore structure of expanded polystyrene, its hydrophobic nature and its compressibility. With X-ray micro-tomography, it is tricky to do so because of the low X-ray absorption of polystyrene that appears as porosity. However, associated with the right image processing, it allows to differentiate the porosity and the polystyrene.

It was possible to obtain the reconstructed volumes of all the samples using efX software compatible with the tomograph X50. Arbitrary cubic zones of interest were selected in the centers of the reconstructed volumes, and the porosity was calculated using Avizo [30], a leading high-performance 3D visualization and analysis solutions for scientific data. This software allows us to select the polystyrene beads and identify them as a separate phase, and then define the porosity apart from the cement matrix using a thresholding tool, which is a simple method of grey level segmentation [31,32]. The processing was not easy and took time, but the result is accurate and precise. In addition, another image processing software "iMorph"