Materials and Design 147 (2018) 80–91

Contents lists available at ScienceDirect

Materials and Design

journal homepage: www.elsevier.com/locate/matdes

Use of tomography to estimate the representative elementary volume in mortars stained with potassium iodide

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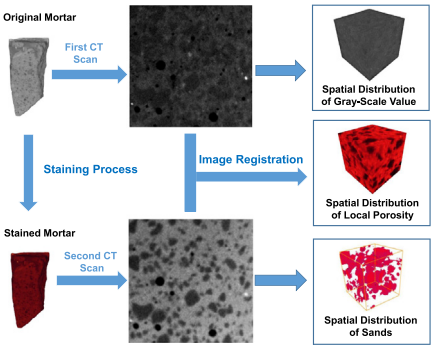
HIGHLIGHTS

- Potassium iodide solution is suitable for the identification of sand grains in mortars.
- A refined dual-scan method can avoid additional damage by drying in traditional attenuation method.
- The RVE size is about 3 to 4 times the maximum sand grain.
- The RVE size is influenced by the mix proportion, local porosity or local sand volume fraction.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 11 October 2017
Received in revised form 9 February 2018
Accepted 12 March 2018
Available online 14 March 2018

Keywords:
Staining
Computed tomography(CT)
Porosity
Mortar
Representative volume element(RVE)
Cement-based material

ABSTRACT

The representative volume element (RVE) plays an important role in the mechanics and physics of random heterogeneous materials with a view to predicting their effective properties. Estimating the RVE size, therefore, is of high importance. In this paper, potassium iodide (KI) was applied first time as the staining agent, and local sand fraction of mortar was calculated combining staining technique and computed tomography (CT). Besides, an extended attenuation method was refined in this study, and spatial distribution of local porosity for mortars was obtained. The RVE size was estimated depending on gray-scale value, local sand fraction and local porosity, respectively, and the results reveal that, the RVE size depending on gray-scale value is smaller than that depending on local porosity or depending on local sand volume fraction; besides, both the RVE sizes depending on local sand fraction and depending on local porosity comply with that obtained from numerical modelling, where a RVE size is suggested to be 3–5 times greater than the maximum sand size.

1. Introduction

For composite materials, the smallest volume over which a measurement can be made that will yield a value representative, is referred as the Representative Volume Element (RVE). The RVE plays an important role in the mechanics and physics of composite materials with a view to predicting their effective properties. Since the pioneering work by Hill et al. [1], a number of researchers have attempted to prove the existence of RVE as well as to estimate the RVE's size [2–5].

When it comes specifically to cement-based material, it is now widely believed that a RVE existed; and, depending on the studied property, the RVE size can vary significantly [2–5]. For example, depending on mechanical properties, a RVE size is estimated to be 3–5 times larger than the maximum aggregate size [4], while based on electromagnetic properties, a volume greater than 3 times of the maximum aggregate

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https://doi.org/10.1016/j.matdes.2018.03.029
0264-1275/

size seems to be sufficient as RVE [5]; and, based on isotherm desorption, a RVE size is suggested to $7d^3$ (d is the maximum aggregate size) [2].

Despite abundant achievements been achieved in the understanding of RVE, the majority of RVE estimation work is based on numerical modelling [6,7]. Unfortunately, the RVE size determined from modelling may not necessarily align with that determined from experiment, as in most situations, models are built under specific preconditions, while the preconditions may be incompatible with the real conditions in cement-based material [8,9]. Nonetheless, understanding the concept of RVE from experimental perspective is necessary, as only under the condition that the overall tested volume surpasses the RVE size can the experimental result be reproducible [2,10]. Application of advanced testing techniques (e.g., Scanning Electron Microscopy (SEM), Computed Tomography (CT), and Nano-Indentation) has brought renewed interest in the RVE estimation because a single test from these techniques only reveals the information from a volume at nano-level or micron-level, which is far below RVE [11–13].

One main reason for the lack of experimental research in RVE is the difficulty involved in sample preparation, especially for mortar: to estimate the RVE size from an experimental perspective, both samples of volume larger and smaller than RVE should be prepared, and the material property at each volume should be taken for investigation; a RVE is attained at a volume above which the oscillation of material property vanishes and a consistent reading is achieved [2,5]. This sample preparation is applicable for concrete, as its RVE is usually of the order of centimeters [2]; however, the difficulty of sample preparation increased dramatically for mortar, as its typical RVE is merely of the order of millimeters [14,15], and it is extremely difficult to control a sample volume precisely at that scale.

With the promise of increased resolution, X-ray computed tomography (CT) has entered the field of RVE estimation in recent years [16–18]. After CT scan, 3D distribution of attenuation coefficient (denoted as "μ") of the scanned object can be obtained, this spatially resolved 3D μ images in voxel element (on the order of tens of microns) can reflect the property of the investigated material, thus innately suitable for RVE estimation. The RVE sizes of graphite electrode [19], cathode [20] and lithium-ion cells [21] have been estimated successfully in previous work using this technique; however, for cement-based material, since a large fraction of capillary pores and all gel pores are above CT's resolving limit, the technique is still insufficient for the microstructure characterization [10]. Besides, CT offers 3D distribution of attenuation coefficient, which is mainly related with the density of the material, while the information of the microstructure cannot be well reflected from this parameter. The deficiency is more severe in composite materials like mortar, where the sand and hydrated matrix acquire a similar attenuation coefficient, but a complete different microstructure [22]. To mitigate these deficiencies, some pioneering work has been done by Wan et al. [10], where the tested sample was scanned in the dried state and in the saturated state respectively, and the obtained local porosity, instead of the local attenuation coefficient, was employed for the RVE estimation. The work itself was pioneering, but the technique requires a complete drying of the scanned object, which may introduce unspecified damage into the specimen, and the obtained porosity in that circumstance, may be different from that of an intact specimen.

In this study, inheriting dual-scan method, local porosity was obtained and was taken as the parameter for RVE estimation in mortars. To avoid extra damage from drying, the drying procedure was substituted by a staining process, and the local porosity was thus, obtained through a dual scan of the specimen in the original state and in the stained state. Potassium iodide, which has been successfully adopted previously in medical science for strengthening image contrast in soft tissues (e.g. embryos [23], tumors [24], fallopian tubes [25]), was adopted in present study for the investigation of mortar's microstructure. To the authors' knowledge, this is the first time use of potassium iodide (KI) for the microstructure investigation of mortar, and the RVE size depending on local porosity was then estimated. Besides, based on the difference of gray-scale value between the hydrated matrix and the sand after staining, a threshold gray-scale value was preset in this study, and local sand fraction was measured; the obtained sand fraction was also employed for the RVE estimation. Moreover, the RVE size depending on the local attenuation coefficient was still estimated in this study. Combining the RVE sizes depending on local porosity, sand volume fraction and local attenuation coefficient, the work aimed at determining the parameter suitable for the RVE estimation, which highlights the degree of heterogeneity in mortar from a microstructural perspective. In addition, local porosity, local sand fraction and the estimated RVE size from this study were compared with that based on other techniques or based on numerical modelling, hence the necessity of present research and its potential application were illuminated.

2. Experimental

2.1. Sample preparation

Mortars were prepared. The P·II 52.5 cement used here was manufactured from Huaxin cement factory, and the quartz sand used was with a maximum diameter of 1.2 mm. Two water to cement ratios (0.3 and 0.5) were selected in this study, and specifically, in one W/C = 0.3 campaign, pulverized cellulose fiber was introduced. The detailed mix designs are listed in Table. 1.

All specimens were cast in rectangular moulds with size of 40 × 40 × 160 mm^3, and were covered by plastic sheets for 24 h under ambient condition. After that, all specimens were de-moulded and cured in lime-saturated solutions for two months. Next, a diamond saw was used to carefully grind the specimen to a size suitable for the CT scanner (with a diameter less than 5 mm). Samples with a smaller size are more easily to be stained thoroughly; and, according to Jin et al. [26], beam-hardening artifact can dampen out in smaller samples. The prepared mortar particles were cured in lime-saturated solution for two more months, during which period the damage around the sample surface was expected to be healed.

2.2. Staining

Direct recognition of sands from mortar is inapplicable, as the attenuation coefficient of the sand is very similar to that of the hydrated paste, and both areas are thus of similar brightness on the reconstructed images [22]. To magnify the color contrast between the sand and the hydrated matrix, a staining technique was applied here.

A CT scan was performed instantly after curing, and the result served as the reference. After that, all samples were immersed in potassium nitrate (KNO_3) solution for 24 h. The concentration of KNO_3 solution was equivalent to the KI concentration used later for staining, and the osmotic movement of iodide trace into the samples was thus minimized. Next, all samples were immersed in prepared KI solution, and a magnetic stirrer was used to accelerate staining. The whole staining process lasted for two weeks, during which period, the temperature in the staining lab was fixed at 20 ± 3 °C. Furthermore, considering that KI is easy to see light decomposition, the whole staining process was performed in a brunet desiccator.

Very limited attempts on KI staining have been made in the field of cement-based materials, and the optimal staining concentration remains unknown yet. In this study, three trial concentrations (0.01 M,

Table 1
Mix designs of the mortars.

	Cement	Water	Sand	Cellulose fiber
CF	367	110	220	1.8
CN	367	110	220	
CNX	367	184	184	