Abstract
The microstructure of building materials importantly influences engineering properties like permeability, strength and durability. To determine this microstructure, different techniques were developed, each with its own limitations. The purpose of this study on concrete and natural building stones was to compare and to combine data obtained by X-ray computed micro-tomography (micro-CT), water absorption under vacuum and mercury intrusion porosimetry (MIP). Pore-size distribution curves ranging from 10 nm to 1 mm and total porosity results were obtained. Furthermore, micro-CT revealed the presence of an interfacial transition zone (ITZ) and of micro-cracks inside the aggregates of the concrete samples after mercury intrusion. Micro-CT visualized mercury inside large air bubbles within the concrete samples. Both micro-CT and MIP were compared and their respective advantages and disadvantages discussed.

Keywords
Concrete, natural building stone, mercury intrusion porosimetry, X-ray computed micro-tomography, porosity.
**Introduction**

The microstructure of concrete and natural building stones has significant influence on their physical and mechanical properties and on their durability. Over the past years several methods were developed to characterize the microstructure of these materials: e.g. gas absorption by the Brunauer-Emmett-Teller (BET) technique, image analysis of thin sections or mercury intrusion porosimetry (MIP). More recently developed techniques like NMR microscopy (Pauli et al., 1997), small angle neutron scattering (Coppola et al., 2002), X-ray computed micro-tomography (micro-CT) (Cnudde & Jacobs, 2004a) allow non-destructive 3D visualization of the internal microstructure of materials. Micro-CT, in combination with at Ghent University developed software can provide not only 3D data of porosity, pore-size distribution and the orientation of pores, but also offers a visualization of the internal structure of the specimen before and after certain applications (Cnudde & Jacobs, 2004b, Cnudde et al., 2004c, De Graef et al., 2005). The main objective of this research was to compare and to combine the results obtained by MIP, water absorption under vacuum and micro-CT and to determine its potential advantages and limitations.

**Materials**

High-performance concretes together were studied in this research with sandstone and limestone samples (Table 1).

Rapid hardening Portland Cement, type CEM I 52,5R was used with 7 % of condensed silica fume and a polycarboxylate-based superplasticizer added during mixing. The aggregates mainly consisted out of basalt. The consistency of the fresh concrete was assessed by the flow table test (according to European Standard EN 12350-5, 1999).

Two types of natural building stones were investigated for this study: a sandstone of Bray (Belgium), which is a quartz arenite of Upper-Ladenian age (Lower Eocene) and a highly-porous bioclastic limestone from Maastricht (The Netherlands, Maastrichtian, Upper Cretaceous). Due to cementation the sandstone has an open but varying porosity ranging from 5 % to 23 %, while the limestone, which mainly consists of skeletal components from foraminifera, sponges, bivalves, bryozoa and brachiopods, has an average porosity of 51 %.

**Table 1. Material properties**
Methods

Micro-CT analysis

For the measurements a “Skyscan 1072” X-ray computed micro-tomograph was used. This device scans a rotating small sample, with a fixed X-ray source, a Hamamatsu micro-focus tube with a focal spot size of 10 µm, and a CCD detector. The spot size limits the spatial resolution of the reconstructed slices to 10 µm in the X, Y and Z directions. Samples were scanned at 130 kV and 76 µA. Random movement of the vertical axis and multiple-frame averaging to minimise the Poisson noise in the projection images were used. Beam hardening was reduced by the presence of a 0.8 mm Al-filter between source and detector.

Cone-beam reconstruction software from Skyscan converted the series of X-ray radiographs (recorded at different angles during step-wise rotation between 0° and 185.85° around the vertical axis) into horizontal cross-sections.

The concrete and stone samples were scanned together with a standard material (calcite) as a grey value reference. After cone-beam reconstruction, the stack of 2D cross-sections were analysed with 3D-software “µCTanalySIS” (Cnudde & Jacobs, 2004a; Steppe et al., 2004). Segmentation of the 2D reconstructed images was performed by double thresholding. A strong thresholding is performed to filter the noise and to select areas that definitely belong to the etched boundaries of the pores, even if they do not completely represent these boundaries. A weak thresholding determines a complete delineation of all the boundaries of the pores, but also includes some noise (Figure 1). The resulting segmented images will combine information of both thresholdings in order to visualize the pores with their corresponding boundaries. The different pores are then labelled in 3D and quantitative 3D interpretation is performed.

Figure 1. Illustration of micro-CT images before and after segmentation by using a strong and a weak thresholding.

The pore-size distribution is determined by using a structuring element that approximates the shape of a sphere and fills every pore, progressing from the smallest inscribed “sphere”, to the largest, which is also known as the maximum opening. Figure 2 illustrates the way the software operates in a 2D example, while in reality spheres are used instead of the shown disks. For each object the software registers the total volume which can be filled inside the object with a “sphere” of a certain diameter. In this way the software
will consider all objects in Figure 3a as objects with the same maximum opening (1 voxel), but a different total volume.

Figure 2. a) Illustration of a 2D-image with labelled pores; b) 2D-filling of the labelled pores with disks in order to determine the maximum opening in combination with the total area that each disk fills inside each pore.

Starting from spheres with 1 voxel, spheres with a diameter of 3 pixels and a volume of 19 voxels (Figure 3b) will fill the objects and the structural element will continue growing (Figure 3c) until the maximum opening is reached.

Figure 3. a) Different pore shapes filled with a sphere with an inscribed diameter of 1 pixel; b) Minimum pore size with 3 pixels diameter c) 2D section of the growing structural element.

Usage of the mentioned 3D-software enables to obtain the total porosity, the maximum opening in each pore, its equivalent diameter and each pore volume. Because micro-CT is a non-destructive technique, small samples can be scanned before and after certain treatments, allowing quantification of changes inside samples. In order to obtain high-resolution images, small samples were preferred that were first scanned with micro-CT and then underwent MIP.

**Water absorption under vacuum.**

Water absorption under vacuum was performed to obtain extra information on the total porosity of each object which could later be compared with the micro-CT and MIP results. The method for the determination of water absorption under vacuum followed the guidelines of the European standard EN 1936 *Natural stone test method – Determination of real density and apparent density, and of total and open porosity* and the TV 205 by the BBRI (BBRI, 1997). In this test procedure dried samples are first put into a vacuum setup to remove all air present inside the pores. Then samples are put into demineralised water, but still under vacuum. Normal air pressure is slowly reached while samples stay for 24 hours under water. Based on the weight of the sample under water and in air, together with the volume of the samples, their open porosity
can be determined. Under vacuum water can penetrate into pores with a diameter larger than 100 nm (Meyer et al., 1994).

**Mercury intrusion porosimetry**

Mercury intrusion porosimetry (MIP) is based on the premise that a non-wetting liquid (contact angle greater than 90°) will only intrude capillaries under pressure according to the following relation:

\[
\Delta \gamma = \gamma \cos \theta - \frac{2 \gamma \cos \theta}{r} \cdot p(pressure)
\]

in which \( p \) is the pressure, \( \gamma \) the surface tension of the liquid, \( \theta \) the contact angle of the liquid and \( r \) the radius of the capillary.

The pore-size distribution is determined from the volume of mercury intruded at each pressure increment, while the total porosity is based on the total intruded volume. The MIP-technique is widely used to study pore structure of materials with pore diameters ranging from 360 to 0.0055 µm. MIP has however some important drawbacks (Cook, 1999; Voka, 2000, Diamond 2000). One of them is the fact that it does not measure the true pore-size distribution due to the so called “ink bottle” effect, since it detects the diameter of the throat entrance instead of the true pore diameter. Secondly, pores are assumed to be cylindrically shaped and fully accessible for mercury penetration from the surface of the specimen what, especially in the case of high performance concretes (HPC), is questionable (Olivier, 1995). The assumption of MIP that the porous medium consists of a bundle of interconnected capillary tubes (De Las Cuevas, 1997) can be very far from reality, because the pore space of rocks is more tortuous and consists of large pores connected by smaller throats (Andriani & Walsh, 2003). Certain assumptions like a constant value for the surface tension and the contact angle of the mercury are used for the calculation of the pore size. MIP can not provide information on closed pores, nor can it give detailed information on pore connectivity.

For the purpose of this research the Carlo Erba Mercury Intrusion Porosimeter was used, with following parameters: contact angle \( \theta = 141,3^\circ \), surface tension of the liquid \( \gamma = 480 \text{ mN/m} \) and compressibility of the mercury \( 0,000001 \text{ bar}^{-1} \).

**Porosity and pore size detected by MIP versus micro-CT**
Figure 4 illustrates a randomly chosen object. In the case of MIP when the mercury would penetrate this object from the top, it would be considered as an object with diameter of 1 pixel and a volume of 29 voxels. If the mercury would penetrate into this object from the bottom, it would be defined as having a diameter of 3 pixels, but the same total volume.

In the case of micro-CT analysis, the software would consider this object in all cases as an object with an inscribed diameter of 3 pixels and a total volume of 29 voxels. Additionally it would also provide the information that a sphere with 3 pixels diameter (figure 3b), would fill 19 voxels of the object, while 10 voxels would only be filled with minimum spheres of 1 voxel size. Based on this information it is possible to define the shape of the object although different variants are possible (Figure 5).

Test procedure

In the first stage, cores having 9 to 10 mm diameter and being 1 cm high were drilled from the concretes and natural building stones. Afterwards, the concrete samples were stored for 28 days in a climate chamber at 65% RH and +20° C. All concrete and stone specimens were dried under vacuum (10⁻³ mbar) for 2 weeks. In the next stage all samples were analyzed by water absorption under vacuum, micro-CT and MIP analysis. Finally, the specimens filled with intruded mercury were analyzed again by the micro-CT in order to determine the extent of the mercury penetration within the specimen.

Test results and discussion

Figure 6 shows a 2D cross-section of concrete sample 0.42-7sf, obtained by micro-CT before MIP. Because the X-rays from the micro-CT are absorbed by the specimen according to composition and density of the material, different features can be detected. Objects with a low atomic number and a low density absorb less
X-rays, resulting in bright areas. Consequently, the higher the atomic number or density, the more X-rays are absorbed, creating darker regions.

Figure 6. Micro-CT image of test concrete 0.42-7sf; 1-matrix (cement paste), 2-aggregate particle, 3-airvoid.

The results calculated according to MIP, micro-CT and water uptake measurements are shown in Table 2. These demonstrate that MIP systematically generates higher total porosity values, except for sample 0.35-7sf. Micro-CT provides always the lowest total porosity results for the concrete samples, while for the natural building stones the micro-CT data are comparable to the total porosity data of the water absorption under vacuum. The main reasons for these different results can be explained by the different measurable pores-size ranges and the physical basis of the measurements. The Carlo Erba Mercury Intrusion Porosimeter can detect pores ranging from 10 nm to 60 µm (according to the manufacturer). The water absorption determines all pores larger bigger than 100 nm, while micro-CT visualizes pores larger than 10 µm. Regarding the methodology, MIP and water absorption tests are base on the intrusion of liquids into the microstructure which ultimately relates the results to the connectivity of the pore system and its access to the external surfaces. On the other hand, micro-CT is an image analysis-based technique which detects all pores as long as their diameter is larger than 10 µm.

The results presented in Table 2 comply with these limitations. For instance, for the dense HPC samples micro-CT showed lower total porosity values compared to water absorption, while for the natural building stones both techniques indicated more comparable data. This phenomenon can probably be explained by the microstructure of the samples in relation with the different analysis techniques. The natural building stones are characterized by a coarser and more interconnected porosity than the HPC concrete samples. Based on the MIP data, the percentage of pores smaller than 10 µm diameter turns out to be relatively higher for the HPC concrete samples than for the natural building stones. This explains why micro-CT systematically detected less pores in the concrete samples. Analysis of the “overlapping range” where direct comparison of the results obtained by MIP and micro-CT was possible, is demonstrated in Table 2. In the case of HPC samples, the MIP values of the overlapping range appeared to be significantly lower in comparison to micro-CT. Also Lange et al. (1994) observed similar underestimation of coarser pores and overestimation
of finer pores by MIP in comparison to the image analysis technique. These authors attributed this phenomenon to the “ink bottle effect” and the different measurable pore-size range of both techniques. The negative influence of the “ink bottle effect” was less pronounced in the case of the natural stones (samples B38, B111 and M49).

Table 2. Comparison of the data obtained with MIP, micro-CT and water uptake under vacuum.

The overlapping pore-size range of MIP and micro-CT is schematically visualized on the pore-size distribution curve in Figure 7, while Figure 8 illustrates the data obtained with MIP and micro-CT. The total porosity calculated by combination of the results from MIP/micro-CT and MIP/water absorption are shown in Table 3. As it was expected, the results showed generally higher values for MIP-micro-CT calculation. The main reason was the higher detectability or overestimation of the pores larger than 10 µm by micro-CT, which detects all pores, including the ones not accessible to the penetrating medium. Surprisingly, the total porosity of concrete sample 0.25-7sf defined by MIP/micro-CT was significantly higher in comparison with two other concrete samples having a higher W/B ratio. This result can be attributed to the combined effect of air voids and of the “ink bottle effect”. Presumably the entrance throat diameters of the air voids were very narrow and thus caused an increase in the amount of pores in the fine pore-size range. This observation can be supported by the high porosity value obtained by micro-CT alone (see Table 2). This example clearly indicates that a combination of MIP and micro-CT techniques can help to determine the extent of the “ink bottle” effect on the measurement results.

Table 3. Total porosity determined by MIP/micro-CT and MIP/water uptake in the rage 5 nm to 1 mm.

Figure 7. Schematical overview of the measuring range of MIP and micro-CT.

Figure 8. Pore-size distribution curves by means of MIP and micro-CT.

The non-destructive character of the micro-CT technique enables to scan samples before and after MIP. After comparing both stacks of images, certain phenomena became visible. Before MIP intrusion pores are
visible in cross-sections as white areas due to the presence of air voids (Figure 9a), while after MIP (Figure 9b) they show up as black areas due to the high absorption for X-rays of the mercury inside these air voids. Despite the fact that Hg caused extensive beam hardening artefacts in the micro-CT images, the infilling of the air voids by the mercury could easily be detected. In cross-sections scanned before mercury intrusion (Figure 9b), the aggregates absorb more X-rays than the matrix (they appear darker than the matrix), while in the cross-section taken after intrusion (Figure 9b), those aggregates absorb less X-rays compared to the matrix (they appear brighter than the matrix), due to the presence of the mercury inside the matrix. The images also demonstrate that the Hg did indeed intrude certain parts of the matrix, while other parts stayed unfilled. This indicates that a large amount of porosity is present in the matrix, which cannot be distinguished in the micro-CT image without mercury, due to resolution limitations. Additional analysis (Figure 9b) revealed the existence of darker areas located on one side of the aggregate particles. According to parallel research (Cwirzen & Penttala, 2004) on the same test concretes by ESEM/BSE-EDS analysis, these shadow areas should be interpreted, as the cement-paste aggregate interfacial transition zone (ITZ). Such zones were detected only under larger aggregate particles. The ITZ is characterized by increased capillary porosity, which becomes visible in micro-CT images as darker areas due to the presence of mercury (figure 9b-1). These results reveal that the contrast in the micro-CT images can be enhanced by intrusion of material with a higher X-ray absorption.

Figure 9. Micro-CT cross-sections of concrete specimens obtained before (a) and after (b) mercury intrusion; 1- interfacial transition zone, 2-aggregate, 3- zone of increased porosity in the cement paste, 4- airvoid filled with mercury

Figure 10a and 10b show cross-sections after micro-CT scanning of concrete sample 0.42-7sf before and after mercury intrusion, but taken at another level. The micro-CT image of the sample filled with Hg revealed a micro-crack in an aggregate particle (Figure 10b-1). This crack was invisible on the micro-CT images of the sample without Hg (Figure 10a). There are two possible explanations for this observation: the cracking of the aggregate occurred during Hg intrusion or the described micro-crack was already present in the sample before MIP, but stayed invisible in the micro-CT images due to insufficient resolution.
Figure 10. Micro-CT cross-sections of concrete specimen 0.42-7sf, obtained before (a) and after (b) mercury intrusion; 1- micro-crack of aggregate particle.

The analysis of micro-CT data allowed the creation of 3D-models of the concrete microstructure and phase composition. Figure 11 demonstrates 3D-images of a concrete phase composition and the microstructure of the concrete and the sandstone specimen. The studied concrete specimen appeared to have a very dense microstructure in the cement paste with just a few air voids, visible as white spheres (Figure 11a). The black particles are presumably agglomerates of the silica fume that was added to the concrete. The aggregate particles are represented by grey areas. It should be noticed that in the case of the concrete sample, the amount of pores with a diameter larger than 10 µm is limited to a few air voids only. The sandstone specimen appeared to have a very complex porous microstructure. This again illustrates that the assumed cylindrical shape of the pores for MIP-calculations is far from reality.

Figure 11. 3D-models created according to the micro-CT data; a) microstructure of test concrete 0.42-7sf; white parts: air bubbles, black spots: presumably agglomerates of the silica fume; grey area: aggregates; b) porosity network of the sandstone specimen.

**Conclusions**

Porosity data obtained from water intrusion porosimetry, micro-CT and water absorption under vacuum have been compared and advantages and disadvantages of these 3 techniques were discussed. The test results indicated that it remains difficult to compare all 3 methods due to their different approach and physical basis, but that the combination of the different techniques can provide additional but valuable data. First of all, micro-CT and MIP measure different pore size ranges in these experiments. The overlapping size range of both methods did not exceed 50 µm. Consequently, direct correlation of the obtained test results, e.g. total porosity and pore-size distribution curves, was difficult. The pore-size distribution curves created from MIP and micro-CT data and covering a range from 10 nm to 1 mm, showed large discrepancies in porosity values. Nevertheless, the supplementary data about the pore-size distribution in the range above 10 µm provide valuable information unavailable when solely MIP is applied. Although MIP is based on liquid intrusion while micro-CT is technique based on image analysis, both data sets can be
of high value for durability studies. For instance the resistance to frost attack depends to a great extent on air-void content and on their distance (Powers, 1949). The combined micro-CT/MIP technique also allows analysis in the pore-size range which includes the air voids. Previously this was normally investigated with polished thin sections and optical microscopy in combination with image analysis-based methods. The creation of 3D-models allows e.g. calculation of the 3D air-void spacing factor, which is detrimental from a frost-durability point of view. Due to the rather limited number of tested specimens, more detailed research is needed especially concerning normal strength concretes (also with air entrainment).

The results also demonstrate that the presence of mercury within the microstructure of the cement paste significantly improved the contrast in the micro-CT images, although Hg created large artefacts as well. Due to the different approach of the 3 techniques, it is relatively difficult to compare MIP and micro-CT test results. Both techniques have their limitations. Depending on pore size and pore structure, MIP-results can be more or less representative (Andriani, G.F., 2003). Results of the 3D-image analysis on micro-CT images mainly depend on their resolution: the higher the resolution is, the more representative the 3D-results will be. Creation of images with a higher signal-to-noise ratio can only improve the quality of the image and thus the 3D-results. For the near future the combination of a better resolution, a still higher image quality and a more powerful 3D-software will increase the application potential of this 3D non-destructive micro-CT technique in the research domain of building materials.

Acknowledgements

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References


### Table 2. Material properties

<table>
<thead>
<tr>
<th>Specimen</th>
<th>W/B *</th>
<th>Silica Fume [% of the cement weight]</th>
<th>Fresh mix air content [%]</th>
<th>Consistency Flow table [cm/cm]</th>
<th>Compr.str. [MPa] 28-days</th>
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<tr>
<td>0.25-7sf</td>
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#### Stone specimens

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<th>Specimen</th>
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<th>Location</th>
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<tr>
<td>B111</td>
<td>sandstone</td>
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<td>Quartz (SiO₂)</td>
<td>20.4</td>
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<td>limestone</td>
<td>Maastricht (the Netherlands)</td>
<td>Calcite (CaO₃)</td>
<td>52.5</td>
</tr>
</tbody>
</table>

*Water to binder (cement + silica fume) ratio*

### Table 2. Comparison of the data obtained with MIP, micro-CT and water uptake under vacuum.

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<tr>
<th>Sample</th>
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<td>Total porosity [%] (10 nm - 60 µm diameter)</td>
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<td>Porosity [%] (10 - 60 µm diameter) by MIP</td>
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<td>4.94</td>
<td>6.52</td>
<td>0.62 (6.742 %) *</td>
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<tr>
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<td>7.92</td>
<td>0.62 (8.618 %)</td>
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<td>0.42-7sf</td>
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*Between hooks the percentage of the pores in this pore class.*

### Table 3. Total porosity determined by MIP/micro-CT and MIP/water uptake in the rage 5 nm to 1 mm.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Percentage pores &lt; 100 nm determined by MIP</th>
<th>Percentage of pores (10 nm – 10 µm) determined by MIP</th>
<th>Total porosity MIP (10 nm-100 nm) + water uptake (&gt; 100nm) [%]</th>
<th>Total porosity MIP (10 nm-10 µm) + micro-CT (&gt;10 µm) [%]</th>
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List of figures

Figure 1. Illustration of micro-CT images before and after segmentation by using a strong and a weak thresholding.

![Original image](image1) ![Segmentation](image2)

Weak Threshold $t_{ww} = 178$ -- $p = 41.0324\%$  Strong Threshold $t_{ww} = 185$ -- $p = 40.3485\%$

Figure 2. a) Illustration of a 2D-image with labelled pores; b) 2D-filling of the labelled pores with disks in order to determine the maximum opening in combination with the total area that each disk fills inside each pore.

![a](image3) ![b](image4)
Figure 3. a) Different pore shapes filled with a sphere with an inscribed diameter of 1 pixel; b) Minimum pore size with 3 pixels diameter c) 2D section of the growing structural element.

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Figure 7. Schematical overview of the measuring range of MIP and micro-CT.

Figure 8. Pore-size distribution curves by means of MIP and micro-CT.
Figure 9. Micro-CT cross-sections of concrete specimens obtained before (a) and after (b) mercury intrusion; 1- interfacial transition zone, 2-aggregate, 3-zone of increased porosity in the cement paste, 4-airvoid filled with mercury.

Figure 10. Micro-CT cross-sections of concrete specimen 0.42-7sf, obtained before (a) and after (b) mercury intrusion; 1-micro-crack of aggregate particle.
Figure 11. 3D-models created according to the micro-CT data; a) microstructure of test concrete 0.42-7sf; white parts: air bubbles, black spots: presumably agglomerates of the silica fume; grey area: aggregates; b) porosity network of the sandstone specimen.