X-ray Computed Tomography Assessment of Air Void Distribution in Concrete

by

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A thesis submitted in conformity with the requirements for the degree of Doctor of Philosophy
Department of Civil Engineering
University of Toronto

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2017

Abstract

Air void size and spatial distribution have long been regarded as critical parameters in the frost resistance of concrete. In cement-based materials, entrained air void systems play an important role in performance as related to durability, permeability, and heat transfer. Many efforts have been made to measure air void parameters in a more efficient and reliable manner in the past several decades. Standardized measurement techniques based on optical microscopy and stereology on flat cut and polished surfaces are widely used in research as well as in quality assurance and quality control applications. Other more automated methods using image processing have also been utilized, but still starting from flat cut and polished surfaces. The emergence of X-ray computed tomography (CT) techniques provides the capability of capturing the inner microstructure of materials at the micrometer and nanometer scale. X-ray CT’s less demanding sample preparation and capability to measure 3D distributions of air voids directly provide ample prospects for its wider use in air void characterization in cement-based materials. However, due to the huge number of air voids that can exist within a limited volume, errors can easily arise in the absence of a formalized data processing procedure. In this study, air void parameters in selected types of cement-based materials (lightweight concrete, structural concrete
elements, pavements, and laboratory mortars) have been measured using micro X-ray CT. The focus of this study is to propose a unified procedure for processing the data and to provide solutions to deal with common problems that arise when measuring air void parameters: primarily the reliable segmentation of objects of interest, uncertainty estimation of measured parameters, and the comparison of competing segmentation parameters.
Acknowledgments

First and foremost, I would like to say thanks to my advisor Prof. Karl Peterson, for his guidance, knowledge, time, financial and moral support in this journey. His commitment to work, his attention to details, and his warm heart to anyone are things I always aspire to;

Also, I like to express gratitude to my committee members: Prof. Douglas Hooton, Prof. Daman Panesar and Prof. Giovanni Grasselli. Without their support and guidance, this dissertation work could not be possible. As well, the constructive feedbacks from my external thesis reviewer, Prof. Julie Vandenbossche in University of Pittsburg, are essential in improving my dissertation;

The always helping hand of the concrete material lab manager Dr. Olga Perebatova and supporting staffs in Civil Engineering Departments all made my study and research here a much easier experience; Thank you.

The permitted access to X-ray CT Machine from Prof. Grasselli’s Geomechanics Group is greatly appreciated. Advices to operate this same machine offered by Ph.D. candidate Zhao Qi, Prof. Nicola Tisato, and Dr. Bryan Tatone all helped me a lot.

For all the fellow graduate students affiliated with U of T Concrete Material Group during 2010-2016 (Ali, Aziz, Bishnu, Chloe, Reza, Rana, Farideh, Soley, Run Xiao, Majella, Eric, Ge-Hung and many more), I enjoy your company and friendship. I am pretty sure these years are going to be a lifelong warm memory. Especially, I would like to say thanks to Ali and Chloe for sitting as a critical audience for my defense practice on several occasions;

I also would like to take this opportunity to say thanks to my master thesis advisor Prof. Yang Jun back in Southeast University for her constant encouragement along these years and Prof. Zhanping You for his generous help at the earlier stage of this study.

Finally and especially, I like to express my thanks and love to my extended family back in China, for all their understanding, huge sacrifice and moral support for these long years when I was far away. To my beloved late uncle, Mr. Qunshan Lu, thanks for all the happy memories.
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<td>micro X-ray computed tomography</td>
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<td>MTO</td>
<td>Ministry of Transportation Ontario</td>
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<td>n</td>
<td>air void intercept frequency</td>
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1 Introduction

The properties of cement-based materials are closely related to the pore structure at varying scales; these include strength, fracture, gas/liquid transport, and thermal conductivity, among others. Many efforts have been made to measure open and closed porosity, and the measurements have been used for different purposes, including quality assurance and quality control (QA/QC), mechanistic investigations, and geometrical inputs for computational simulation of performance. X-ray computed tomography (CT) is a powerful technique to observe at the macro, micro, and nano levels the effectiveness of different processing alternatives or the extent of damage under severe environments. Although X-ray CT has been used as a tool in concrete research for over two decades, within the past few years its use has been more frequently reported. Besides research targeted at characterizing capillary pore structure and tortuosity in cement paste, other commonly reported uses include the characterization of hydration at early stages, and the measurement of the degree of internal damage caused by durability related issues. With few exceptions, most research utilizing X-ray CT has been more focused on qualitative analyses. With the increasing availability of versatile chemical admixtures, developments in material processing on one side and the increasing concerns for more sustainable materials on the other, the design of “engineered materials” is much more common than before. In this process, a technique that can more quantitatively characterize and compare the three-dimensional (3D) structure of alternative material designs will be of great importance. Among these design parameters of interest, air void parameters are of essential importance. The use of X-ray CT for reliable and routine measurement of air voids not only provides an alternative means of measurement, but also presents a unique advantage over other techniques with its capacity to capture the exact 3D location of all objects of interest, and derive air void parameters not available to traditional two-dimensional (2D) test methods. However, some barriers must be overcome before X-ray CT can be used in a practical manner for the measurement of air void parameters. For example, the issues of how to distinguish entrained air voids in the paste from voids in aggregate, how to reliably separate air-void clusters, what volume of sample that can be treated as representative volume element (RVE), and what error propagations could arise from image processing techniques, are still lacking. This dissertation addresses these issues as applied to air-void parameters in cement-based materials.
In Chapter 2 a literature review on existing air void measurement techniques in general is carried out, along with a review of the current scope of micro X-ray CT (µCT) in cement-based material characterization. In Chapter 3 a brief introduction to the work-flow of image processing based quantitative analysis is provided, and influential image segmentation and shape classification methods are reviewed.

In subsequent chapters, selected methods for image processing are demonstrated on lightweight concrete, structural concrete, and laboratory cement mortar. These chapters are presented in chronological order. For most material characterization research programs, a logical approach would be to start with a simple binary two-phase system and then work towards more complicated multi-phase systems. In this research, the opposite occurred: it began with the most complicated multi-phase problem, and worked its way towards simpler problems. This was not by design, but merely a consequence of circumstance, as the complicated problem presented itself first. In Chapter 4, air void size distributions are obtained from lightweight concrete based on the combination of two X-ray CT scans performed at both the macro and micro scale. Difficulties and solutions identified during this initial stage are refined in Chapter 5, where results of air void parameter measurements from field concrete are presented and discussed within the context of QA testing.

In Chapter 6, the results of air void parameters from cement mortar mixes produced using vinsol resin and a synthetic air entrainer are summarized.

Chapter 7 provides a summary of contributions, and a discussion of limitations and recommendations for further research.
2 Methods for air void characterization of cement-based materials

2.1 Stereologic methods

Pores in concrete, whether at the nm inter-layer scale of hydrate gels, the nm to µm scale of capillary voids, the µm to mm scale of entrained air voids, or the mm to cm scale of entrapped air voids, all play a crucial role in the performance of cement-based infrastructure. But in terms of durability, entrained air voids are widely credited with providing resistance to freeze-thaw related damage. In 1939, the Universal Atlas Cement Company was the first to commercialize a cement product with built-in air entrainment, which they named Atlas Duraplastic. In promotional publications of the time, air-entraining portland cement was advertised as “one of the greatest advances in cement and concrete making since the water-cement ratio theory”, complete with an artistic depiction of the new technology (Figure 2.1).

![Figure 2.1](image.png)

Figure 2.1: “A highly magnified section of Atlas Duraplastic air-entrained concrete would look something like this. The two large black areas represent small portions of coarse aggregate; remaining black spots represent sand; gray area represents cement paste; white dots represent entrained air bubbles” –excerpt from Universal Atlas Cement Company (1951).

After air entrained concrete had been demonstrated to provide improved freeze-thaw durability in structures and pavements throughout North America, Powers (1949) proposed a mechanism to
explain the improved performance where the air voids exerted a “sphere of influence” with radius \( r_m \) that protected the paste surrounding an entrained air void with radius \( r_b \) (Figure 2.2).

\[
\frac{r_m^3}{r_b} + r_b^2 - \frac{3}{2} r_m^2 = \frac{3kT}{\mu (1.09 - S^{-1}) u \theta}
\]

Equation 2.1

Where:
- \( k \) = cement paste permeability coefficient
- \( T \) = cement paste tensile strength
- \( \mu \) = pore fluid viscosity
- \( S \) = capillary pore saturation
- \( u \) = volume of water frozen per volume paste, per degree
- \( \theta \) = cooling rate

Depending on the choice of input values, Powers estimated that the maximum shell thickness of paste that lies within the protected sphere of influence, \( L_{max} \), would fall within a range of 270 to 660 \( \mu m \) for most concrete. Powers further recognized that a simple geometrical parameter to
describe the typical distance within a given concrete between points in the paste to the nearest entrained air void was necessary. Powers named this parameter the spacing factor, $L$, and based it on stereological measurements of the volume fraction of air voids, $A$, the volume fraction of paste, $p$, and the void intercept frequency, $n$, as collected from a linear traverse over a flat polished cross section through a concrete specimen. Once $A$ and $n$ are determined, a value for the average air void chord intercept length, $\bar{I}$ with the test line is computed (Powers, 1949):

$$
\bar{I} = A/n 
$$

Equation 2.2

With a value for $\bar{I}$ established, the surface area of the air voids per unit volume, i.e. the specific surface, $\alpha$, is computed according to the stereologic relationship (Powers, 1949):

$$
\alpha = 4/\bar{I} 
$$

Equation 2.3

The radius, $r_v$, of a spherical air void that would satisfy the value for $\alpha$ is easily computed, and Powers used this radius as the basis for computing $L$. Assuming mono-sized air voids with radius $r_v$ arranged in a cubic lattice within the paste, and the lattice sized to satisfy the ratio $A:p$, the maximum distance from a point in the paste to the nearest edge of an air void is computed (Powers, 1949):

$$
L = \frac{3}{\alpha} \left[ 1.4 \left( \frac{p}{A} + 1 \right)^{1/3} - 1 \right]
$$

Equation 2.4

Powers also derived a simpler equation for $L$ based on the thickness of paste required to cover an air void of radius $r_v$ while satisfying the ratio $p:A$ (Powers, 1949):

$$
L = \frac{p}{\alpha A}
$$

Equation 2.5

Equation 2.4 yields smaller values for $L$ than Equation 2.5 when $p:A$ is greater than 1:4.342, and Equation 2.5 yields smaller values for $L$ than Equation 2.4 when $p:A$ is less than 1:4.342. Powers suggested using whichever equation yielded the lowest value for $L$. Powers’ work became the
basis for standards used today worldwide for the characterization of air voids in concrete (ASTM C457, 2012; BS EN 480-11, 2005).

Since Powers initial study, many alternatives to the parameter $\overline{L}$ have been proposed, as summarized by Snyder et al. (2001), along with more recent additions by Mayerćsik (2014), Wawrzeńczyk and Kozak (2016), and Schock et al. (2016). Although the new approaches use more sophisticated geometric parameters as opposed to Powers’ simplified model, they remain highly correlated to $\overline{L}$, and perform similarly in terms of predicting durability in freeze-thaw tests conducted according to ASTM C666 (2015) (Figure 2.3).

![Figure 2.3 Correlation between proposed limits for alternative air-void spacing parameters and $\overline{L}$ plotted by ASTM C666 durability factor (DF) and the air content within the mortar fraction, (a) Philleo factor, $S$ (Peterson & Sutter 2011), and (b) maximum nearest surface, $\overline{M}$ (Mayerćsik et al. 2014).](image)

In Figure 2.3 the durability factor (DF) is calculated as the relative dynamic modulus of elasticity (RDME) after 300 freeze thaw cycles, where RDME is expressed as a percentage of the initial modulus at zero cycles (ASTM C215, 2014; ASTM C666, 2015). If the RDME drops below 60% of the initial modulus before 300 cycles, the RDME is reported at the number of cycles at which the drop occurred. The data in Figure 2.3a were collected using the less aggressive ASTM C666 Procedure B, where the beams are frozen in air and thawed in water. The data in Figure 2.3b was collected using the more aggressive ASTM C666 Procedure A, where the beams are frozen in water and thawed in water. In both cases, there is a trend for lower RDME values as $\overline{L}$ increases. The upper limit to ensure freeze-thaw durability for $\overline{L}$ is suggested as 0.2 mm in the Appendix of ASTM C457 (2012). The corresponding suggested upper limits of 0.1 mm for the Philleo factor $S$ and 0.025 mm for the maximum nearest surface, $\overline{M}$ are also included in Figure...
2.3 (Philleo, 1983; Mayercsik et al. 2014). $S$ is defined as the distance at which only 10% of the paste volume lies farther than that distance from the perimeter of the nearest air void (Philleo, 1983). $\overline{M}$ is defined as the maximum distance between a point in the paste and the periphery of an entrained air void defined (Mayercsik et al. 2014). Based on the data in Figure 2.3.3, neither $\overline{L}$, $S$, or $\overline{M}$ appears to do a perfect job in terms of predicting freeze-thaw durability.

In spite of the limitations of $\overline{L}$, it is currently used in the province of Ontario, Canada to determine contractor payment for Ministry of Transportation (MTO) projects as specified in OPSS.PROV 1350 (2014). After placement, cores are collected and $A$ and $\overline{L}$ are measured according to MTO LS-432 (2013), the provincial equivalent to ASTM C457 (2012). If the concrete has an $A \geq 3\%$ and a $\overline{L} \leq 0.23$ mm, the contractor avoids a penalty. If the concrete has an $A$ between 4 and 7%, and a $\overline{L} \leq 0.19$ mm the contractor receives a bonus.

A common complaint directed towards ASTM C457 is that the test is tedious and prone to operator variability. To conduct a test, a cut surface of concrete must be polished, and the surface examined under a microscope with a mechanical stage. Alternatives have been implemented by many researchers and practitioners based on the same general approach, but with automated means of detecting and measuring the air voids (Browne and Cady 1970; Chatterji and Gudmundsson 1977; Roberts and Schainer 1981; Roberts and Scali 1984; Dewey and Darwin 1991; Laurençot et al. 1992; Scott 1997; Harris 1998; Schorholz 1998; Aligizaki and Cady 1999; Baumgart et al. 2001; Elsen, 2001; Pleau et al., 2001; Peterson et al. 2001; Pade et al. 2002; Zhang et al. 2005; Zalocha and Kasperkiewicz 2005; Carlson et al. 2006; Jakobsen et al. 2006; Jana 2007; Ramezainianpour et al. 2010, Mateusz et al. 2010, Toumi and Resheidat 2010; Fonseca and Scherer 2014; Mayercsik et al. 2014; Liu et al. 2015; Wawrzeńczyk and Kozak 2016).

### 2.2 Methods for fresh concrete

One of the major shortcomings of the previous stereologic methods is that they are performed on concrete after it has hardened; $\overline{L}$ can only be measured after the concrete has been placed. To overcome this, two fresh concrete test methods have been standardized: the Air Void Analyzer, AVA (AASHTO TP 75, 2008) and the Super Air Meter, SAM (AASHTO TP 118, 2015).
To conduct an AVA test, a fixed volume sample of the mortar fraction is extracted from the fresh concrete and injected into the base of a column filled with glycerin. A magnetic stir bar agitates the mortar, releasing the entrained air bubbles, which rise up the column. At the top of the column, the entrained air bubbles are collected by a small cup attached to a balance. Larger bubbles rise faster than the smaller bubbles according to Stokes’ law. By recording displacement versus time, the air content and air void size distribution are determined. Combined with a priori knowledge of the volume of the paste fraction, air-void parameters such as $\bar{L}$ can be computed (Distlehorst and Kurgan, 2007).

The SAM operates on the same principle as a Type B ASTM C231 (2014) pressure meter, where air is pressurized to a set level in an upper chamber, and then opened to a sealed lower chamber that contains the concrete. When exposed to elevated pressure, the air voids in the concrete are compressed, and the drop in overall pressure of the system is related to the bulk air content according to Boyle’s law (Hover, 1988). The SAM goes one step further by taking advantage of the fact that when air voids are compressed, the internal pressure inside the air voids increases, and as the pressure increases some of the gas dissolves into solution according to Henry’s law. Air voids in fresh concrete are under pressure from the curvature of the bubble wall, as well as the load from the overlying concrete; as a result, smaller voids in the size range of 10 µm are expected to dissolve completely in fresh concrete (Mielenz et al., 1958). To conduct a SAM test, the upper chamber is pressurized to a level 3× that of an ASTM C231 test, and then opened to the lower chamber containing the concrete. The pressure of the system at equilibrium is recorded, and the system returned to atmospheric pressure. During the initial pressurization, some fraction of the air voids dissolve, but they do not necessarily re-appear immediately when returned to atmospheric pressure. Next, the process is repeated. The second equilibrium pressure is typically higher than the initial equilibrium pressure, and the pressure differential is attributed to the loss of dissolved air voids during the initial run. Although the exact mechanism has not been fully documented, it has been demonstrated that properly air entrained concrete exhibits lower SAM numbers than inadequately air entrained concrete (Ley and Tabb, 2014).

Ultrasonic measurements of both fresh and hardened concrete have also shown promise for air void characterization. Various methods take advantage of the attenuation and loss of velocity as waves pass through concrete; the higher the entrained air content, the greater the attenuation and velocity loss (Punurai et al., 2007; Lissenden et al. 2010, Zhu et al., 2011; Sun et al. 2013, Guo at
al. 2016). Other approaches include the detection of air voids by changes in the reflected intensity of light emitted from optical fibres inserted in fresh concrete (Ansari, 1990) and by the thermal signature as monitored by flash thermography (Xiao, 2010).

### 2.3 X-ray computed tomography methods

Computed tomography (CT) is an imaging technique where projected two dimensional (2D) images through an object are compiled and used to reconstruct the external and internal features as a three dimensional (3D) image (Stock, 2009). The earliest documented application of the technique as applied to concrete was by Morgan et al., (1980) who used $^{137}$Cs isotopes as a source of gamma rays to pass through a 150 mm dia. concrete cylinder (Figure 2.4a). The cylinder was rotated 360° with a projected image recorded every 3.6°. A linear array of scintillator detectors were positioned 1.2 m from the gamma ray source, and the cylinder positioned 0.6 m away from the source, yielding a projected image with a resolution of 0.8 mm. The general set-up for the X-ray CT equipment used in this dissertation research is shown in Figure 2.4b.

![Fan beam configuration (a), where either the object or the source-detector apparatus is rotated, from Morgan et al. (1980), and cone beam configuration with areal detector for the X-ray CT equipment used in this dissertation research, after Ketcham (2016).](image)

For CT, the resolution is a function of the size of the photon source, the distance between the source and the sample, the distance between the sample and the detector, and the dimensions and spacing of the detector elements (Figure 2.5) with magnification, $M$, computed as (Feser et al., 2008):
\[ M = (a + b)/a \]

Equation 2.3.1

Where:
- \( a \) = source to object distance
- \( b \) = object to detector distance

The size and composition of the sample controls the attenuation of photons. If the sample is too large or too dense, the quantity of photons that reach the detector may be insufficient to yield a quality image. Figure 2.6 shows the typical relationship between sample size and resolution for X-ray sources (Nueser and Suppes, 2007).
Although a variety of photon sources may be used for CT, X-rays have seen widespread use as applied to cement-based materials. Prior to 2010, synchrotron X-ray sources dominate the literature, but with the availability of micro X-ray CT equipment over the past few years, the number of publications in this field has been growing exponentially (Figure 2.7). Table 2.1 summarizes applications of X-ray CT to cement-based materials as taken from the literature.

Figure 2.7: Number of publications in the field of cement-based materials utilizing X-ray CT as compiled from Engineering Village™ database.
Table 2.1: Summary of publications with X-ray CT as applied to cement-based materials.

<table>
<thead>
<tr>
<th>Category</th>
<th>Publication</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aggregate/cement particle shape</td>
<td>Garboczi (2002); Garboczi and Bullard (2004); Masad et al. (2005); Garboczi et al. (2006); Cepuritis et al. (2014); Thomas et al. (2016); Yang et al. (2016)</td>
</tr>
<tr>
<td>characterization</td>
<td>Air void characterization</td>
</tr>
<tr>
<td></td>
<td>Wiese et al. (2000); Kim et al. (2012); Yun et al. (2012); Bernardes et al. (2015); du Plessis et al. (2016); Schock et al. (2016)</td>
</tr>
<tr>
<td>Acid attack</td>
<td>Fan and Luan (2013)</td>
</tr>
<tr>
<td>Alkali-silica reactivity</td>
<td>Farnam et al. (2015); Marinoni et al. (2015)</td>
</tr>
<tr>
<td>Bacterial weathering</td>
<td>De Graef et al. (2005)</td>
</tr>
<tr>
<td>Carbonation</td>
<td>Mason et al. (2014); Liu et al. (2016); Han et al. (2012a); Han et al. (2012b)</td>
</tr>
<tr>
<td>Cryogenic</td>
<td>Kogbara et al. (2014); Kogbara et al. (2015)</td>
</tr>
<tr>
<td>Early-age cracking</td>
<td>Zhang et al. (2014)</td>
</tr>
<tr>
<td>Fire damage</td>
<td>Kim et al. (2013); Henry et al. (2014); Su et al. (2015); Henry et al. (2016)</td>
</tr>
<tr>
<td>Freeze-thaw</td>
<td>Promentilla and Sugiyama (2010); Weise et al. (2012); Yuan et al. (2014)</td>
</tr>
<tr>
<td>Leaching</td>
<td>Burlion et al. (2006); Rougelot et al. (2010); Wang et al. (2012); Walsh et al. (2013); Bywalski et al. (2015)</td>
</tr>
<tr>
<td>Steel corrosion</td>
<td>Neff et al. (2011); Česen et al. (2013); Itty et al. (2014); Šavija et al. (2014); Zhang et al. (2012a)</td>
</tr>
<tr>
<td>Sulfate attack</td>
<td>Bentz et al. (1995); Stock et al. (2002); Naik et al. (2004); Naik et al. (2006); Yuan et al. (2016);</td>
</tr>
<tr>
<td>Fiber reinforcement</td>
<td>Barnett et al. (2010); Krause et al. (2010); Erdem et al. (2011); Kaufmann et al. (2013); Trainor et al. (2013); Bordelon and Roesler (2014); Hernández-Cruz et al. (2014); Zofka et al. (2014); Yi et al. (2015); Chalencon et al. (2016); Schabowicz et al. (2016); Žirgulis et al. (2016a); Žirgulis et al. (2016b); Wang et al. (2014a)</td>
</tr>
<tr>
<td>Hydration</td>
<td>Galluci et al. (2007); Liu et al. (2012); Ba and Qian (2013); Třík et al. (2013); Volotolini et al. (2013); Artioli et al. (2014); Parisatto et al. (2015)</td>
</tr>
<tr>
<td>Lightweight additions</td>
<td>Lanzón et al. (2012); Tekin et al. (2012); Wei et al. (2013); Crandall et al. (2014); Wei et al. (2014); Lanzón et al. (2015); Tekin et al. (2015); Farhan et al. (2016); Lee et al. (2016); Chung et al. (2016a); Chung et al. (2016b)</td>
</tr>
<tr>
<td>Mechanical testing</td>
<td>Landis et al. (1999); Lawler et al. (2001); Landis et al. (2003); Elaqr et al. (2007); Landis et al. (2007); Landis and Bolander (2009); Kocur et al. (2011); Erdem et al. (2012); Poinard et al. (2012); Jivkov et al. (2013); Su et al. (2013); Wan and Xue (2013); de Wolksi et al. (2014); Liu et al. (2014); Pan and Lloyd (2014); Ren et al. (2015); Skarżyński et al. (2015); Skarżyński and Tejchman (2016); Sun et al. (2016); Zhang and Jivkov (2016)</td>
</tr>
<tr>
<td>Novel materials</td>
<td>Rattanasak and Kendall (2005); Chen et al. (2011); Provis et al. (2012); Yoon et al. (2014); Fan et al. (2015); Onutai et al. (2015); Borges et al. (2016); Leite and Monteiro (2016); Murugan et al. (2016); Nunes et al (2016)</td>
</tr>
<tr>
<td>Pervious pavements</td>
<td>Manahiloh et al. (2012); Meulenzyzer et al. (2012); Agar-Ozbek et al. (2013); Jiang et al. (2015); Kuan et al. (2015)</td>
</tr>
<tr>
<td>Self-healing materials</td>
<td>Tittelboom et al. (2011); Fukuda et al. (2012); Fukuda et al. (2013); Fan and Li (2015); Lv et al. (2016); Snoeck et al. (2016); Tittelboom et al. (2016); Wang et al. (2014b)</td>
</tr>
<tr>
<td>Water/gas transport</td>
<td>Lu et al. (2006); Promentilla et al. (2008); Promentilla et al. (2009); Třík et al. (2011); Darma et al. (2013); Shibata et al. (2013); Dolder et al. (2014); Wan and Xu (2014); Bossa et al. (2015); Chung et al. (2015); Ranachowsk et al. (2015); Yang et al. (2015); Lukovic and Ye (2016); Promentilla et al. (2016); Zhang et al. (2012b); Han et al. (2012c)</td>
</tr>
</tbody>
</table>
The broad topic of pore space in concrete covers everything from nano-sized gel pores, nano to micro-sized capillary pores, and micro to macro-sized entrained and entrapped air voids, as well as cracks. While all of these categories of porosity have been studied using X-ray CT methods, given the topic of this dissertation, only the research more closely related to the field of air void characterization warrants further discussion.

Weise et al. (2000) were the first to explore the potential for air void characterization using an industrial cone beam X-ray CT that obtained 20 µm resolution images from an 8 mm dia. hardened mortar cylinder. Although quantitative measurements were never made, they demonstrated the capability to discern air voids with diameters on the order of 0.1 µm. X-ray CT measurements of bulk air content in concrete are commonplace in the literature. However, the quantification of other parameters, particularly those describing the spatial distribution of entrained air voids, has received much less attention.

Cnudde et al. (2009) achieved the isolation of air voids in concrete through a dual grayscale threshold approach, combined with a voxelized sphere-growth algorithm to determine air void size. An initial threshold, termed the “strong threshold” was manually selected to eliminate noise voxels while simultaneously including air voids. A secondary threshold, termed the “weak threshold” was selected manually to more accurately define the boundaries of the air voids that were identified by the initial threshold. After isolation of the air void boundaries, the diameter of the largest voxelized sphere that could be contained within each individual air void was used as a measurement of the air void size (Figure 2.8).

![Figure 2.8: Depiction of voxelized sphere growth within an air void to illustrate practice of Cnudde et al. (2009) where the diameter of the largest voxelized sphere contained within the air void is used as a measurement of air void diameter.](image)

Kim et al. (2012) were the first to compute $\bar{L}$ in air entrained cement paste samples using micro X-ray CT. Given the simple binary system of paste and air voids, Otsu’s method of threshold
determination was sufficient to discern the two phases as based on the grayscale X-ray intensity histogram (Otsu 1975). A variety of grayscale-based thresholding methods, including Otsu’s, are described in more detail in the next chapter. After isolating air voids in the 3D reconstructed image Kim et al. (2012) distributed traverse lines over a series of extracted 2D cross-sections to determine $\bar{L}$.

Subsequent work by Yun et al. (2012) went one step further by utilizing randomly oriented traverse lines projected through 3D reconstructed images of mortars to obtain values for $A$ and $\bar{I}$, with the calculation of $\bar{L}$ based on values of $p$ obtained from the mix design. Although technically a three-phase system (air, paste, and fine aggregate) thresholding was performed using Otsu’s method to discern between just two phases: the solid phase (paste and fine aggregate) and the non-solid phase (air-void). Yun et al. (2012) also made an effort to measure the paste-void proximity distribution that describes the population of distances between points in the paste to the edges of the nearest air voids (Snyder et al. 2001). Since no distinction was made between the paste and fine aggregate, the measurements were actually distances between points in the solid phase and the edges of the nearest air voids. To account for this, all distances were multiplied by the volume fraction $p$ to achieve an approximation of the true paste-void proximity distribution.

Both Kim et al. (2012) and Yun et al. (2012) also used the 3D reconstructed images to obtain air void size distributions based on the diameters of spheres that would have the same volumes as the corresponding air voids. They termed this value the “equivalent void diameter.” Similar measurements of air void distribution were also made by Bernardes et al. (2015) and du Plessis et al. (2016), but within the context of the influence of sampling size and resolution. Bernardes et al. (2015) make no mention of the methodology employed to isolate the air voids, and instead focus on the influence of the regions of interest (ROIs) used to sample 2D circular cross-sections through their 3D reconstructions of 20 mm dia. mortar cylinders. They defined three types of ROIs: global, indented, and central (Figure 2.9), and concluded that air voids are more abundant along the perimeters of the sample directly adjacent to the mold. Although never defined explicitly, the air void size distribution was expressed by the mean diameter, presumably derived from the diameters of circular intercepts of air voids intersected by the 2D cross sections.
du Plessis et al. (2016) took a more sophisticated approach exploring the influence of scan resolution and collection time on measured air void size distributions. Not surprisingly, with increased resolution and collection time the detection of smaller sized air voids improved. Air voids were isolated by the initial selection of the central threshold value between the grayscale histogram peaks that represented the solid and air void phases, followed by fine-tuning through manual threshold selection. du Plessis et al. (2016) then used the 3D reconstructed images to obtain air void size distributions based on volume.

Schock et al. (2016) performed the most thorough air void characterization to date, based on scans collected at two different accelerating voltages: 160 kV and 60 kV. 3D reconstructions were independently performed at each voltage level, and combined to obtain a single high contrast 3D reconstructed image at a voxel resolution of 5.7 µm³. The grayscale intensities for the paste, aggregate, and air void phases were sufficiently distinct to allow for manual selection of appropriate threshold levels to isolate the three phases. Voxels classified as air void, but surrounded by voxels classified as aggregate, were reclassified as aggregate and excluded from subsequent spatial calculations. Air voids composed of < 8 voxels were also excluded. Finally, air voids with a sphericity of < 0.85 were excluded, with sphericity defined as the ratio of the surface area of a sphere with a volume equivalent to the object to the surface area of the object. For their computations of volume and surface area, all air voids were approximated as ellipsoids based on the principal and semi-principal axes. The sphericity cut-off of 0.85 was selected by comparing manual measurements of $\bar{L}$ obtained from a cut and polished slab to calculations of $\bar{L}$ obtained from the 3D reconstruction using direct measurements of $A$, $p$, and $\alpha$. The sphericity cut-off of 0.85 minimized the difference between the manual and X-ray CT based measurements of $\bar{L}$. Schock et al. (2016) also employed a new parameter based on the star length distribution.

Figure 2.9: ROI categories, after Bernardes et al. (2015).
(SLD). To find the SLD, 100,000 points were randomly located within the paste. A series of 600 rays were propagated from each point, and terminated whenever they reached either an air void or an aggregate phase (Figure 2.10).

![Figure 2.10: Rays emanating from random point in the paste; rays intersecting aggregate (crosshatch pattern) are rejected, rays intersecting air voids (circles) are accepted, with the shortest ray recorded as the star length (from Schock et al. 2016).](image)

The minimum length ray that terminated at an air void was selected as the star length for any given point. The 85th percentile of the SLD was found to be equivalent to values found for $\bar{L}$, and was termed the cumulative star length $\bar{s}$.

The size and spatial distribution of air voids has also been well-explored within the context of providing inputs for finite element and other numerical modeling (Elaqra et al. 2007; Kocur et al. 2011; Poinard et al. 2012; Jivkov et al. 2013; Pan and Loyd 2014; Wolski et al. 2014; Ren et al. 2015). With few exceptions, minimal effort is spent describing the specific methodologies used to delineate the phases and measure the air void size distributions. In most cases, grayscale thresholding was performed either manually or by Otsu’s method, with size distributions derived from spherical equivalents based on the voxelized volume of the air voids. One notable exception is the work of Elaqra et al. (2007) who employed Fourier domain band-pass filtering to enhance the edges of air voids on 2D cross-sections as extracted from the 3D reconstructed images. By combining manual grayscale thresholded binary images of the original and band-pass images, the air void boundaries were isolated (Figure 2.11).
Figure 2.11: Steps for detection/isolation of air voids, (a) initial grayscale image, (b) intensity profile of initial image after band-pass filter, (c) corresponding profile from initial image, (d) summation of images (e) and (f) where (e) is the binary outcome of threshold applied to band-pass image and (f) is the binary outcome of threshold applied to the initial image, from Elaqra et al. (2007).
3 Digital image processing

3.1 Typical workflow for image processing based quantitative analysis

Approaches to traditional experimental data collection and processing have many similarities to approaches to image collection and processing, whether using specific image processing software (e.g., Photoshop™ or ImageJ), or using more general programming/scripting language/mathematical packages with image processing modules (e.g., Matlab™, Python, C++, C#, Java, or IDL). The approaches only differ in the original data format and in the techniques used to process them. For image processing, some visual observations can be made directly, but in many cases the "object of interest" is buried within image data behind varying levels of noise. In the context of this research the objects of interest are multitudes of individual air voids. But in more general context, the objects of interest could be particles of any type dispersed in a matrix of some composite material. During the quantitative assessment of the patterns of these objects as a whole, such as their total volume, spatial distribution, size distribution, or shape variation, the errors incurred during segmentation and classification of individual objects should be kept to a minimum. To achieve this goal, images processing usually starts with noise removal (if it exists), followed by the extraction of objects of interest from the background, and the classification of objects based on one or multiple chosen features. Finally, parameters associated with a specific type of objects can be counted, measured or derived. A simplified illustration of these steps is provided in Figure 3.1.

Many techniques are available to extract and classify objects, however, there are no universally accepted guidelines to carry out such processing. The selection of an appropriate technique depends on many factors. These factors include the nature of the material (e.g. grayscale contrast among different phases of material), the textures (i.e., notable brightness patterns), the number of objects within the sample, the available computing resources, and the acceptable uncertainty bounds of the measured parameter.
Fig 3.1 Illustration of general steps for image data processing, with initial image (a), isolation of objects (b), and categorization of objects (c,d,e,f).
Figure 3.2 illustrates a situation where objects can be sorted by applying a uniform grayscale thresholding value; Figure 3.2b illustrates a situation where objects can be sorted based on their shape, and Figure 3.2c shows a case in which texture plays a key role for object classification. However, there are many situations where there are no explicit features available to take advantage of. In such cases, information from different aspects has to be combined in order to classify the objects. One approach is to utilize a moving window with a reasonable field view, and collect information contained within the window (Figure 3.2d). Then, features with varying weights are involved to classify the objects. The optimization criteria for decision-making dictate the classification algorithm; the development of optimization criteria is a rapidly evolving field.
of research. In the following sections, the important segmentation and classification techniques used in this study are reviewed, along with additional techniques that show great promise for future studies.

3.2 Methods for segmentation and classification

3.2.1 Segmentation based on global thresholding

Pixels of digital images are usually indexed with a numerical value corresponding to reflectance or other physical properties. For X-ray CT, this value (the CT Number) is related to X-ray attenuation within the material. However, the scale for this number is usually equipment dependent. For a convenient comparison of measurements from different equipment, a more practical unit called Hounsfield Unit (HU) is often used. It is based on a linear transformation of the original linear attenuation coefficient. Values for HU collected at different scanning energies can be found for selected elements, compounds, and mixtures that are of radiological interest in databases, for example, the compilation by Hubbell and Seltzer (2004). In the HU scale, the radiodensity (i.e., the relative inability of electromagnetic radiation to pass through a particular material) for air is defined -1000 HU and the value of distilled water at standard temperature and pressure is defined as 0 HU. The linear transformation is described as in Equation 3.1:

\[ HU = 1000 \times \frac{\mu - \mu_{water}}{\mu_{water} - \mu_{air}} \]

Equation 3.1

Where:
\(- \mu = \) linear X-ray attenuation coefficient of the tested material
\(- \mu_{water} = \) linear X-ray attenuation coefficient of water
\(- \mu_{air} = \) linear X-ray attenuation coefficient of air

When the linear X-ray attenuation coefficient of a measured material and the machine-specific conversion factors from CT Numbers to HU are available, objects made of a known material can be easily identified. As the aforementioned conveniences are not always available, the approach has seen only limited use as applied to cement-based materials (Aruntaş et al. 2010; Wan et al. 2015). More often, methods purely taking advantage of the differences of grayscale intensity among phases are employed. This group of methods is illustrated in Figure 3.3. The images in the left column are three artificially synthesized images with their corresponding grayscale value histograms in the right column. Each synthesized image is assumed to consist of two phases. For the image in Figure 3.3a, a uniform threshold value can be easily applied to segment the two
Figure 3.3 (a) 100×100 pixel synthesized image with grayscale value of two phases following N(10,2), and N(30,3) distribution respectively; (b) Histogram of images in (a); (c) 100×100 pixel synthesized image with grayscale value of two phases following N(20,2), and N(30,3) respectively; (d) Histogram of images in (c); (e) 100×100 pixel synthesized image with grayscale value of two phases following N(25,2) and N(30,3) respectively; (f) Histogram of images in
(e); phases. For the images in Figures 3.3c and 3.3e, the grayscale values in two phases tend to overlap with each other, and a uniform cut-off threshold value cannot be easily found. Many methods can be used in this situation. Some of the most commonly used as well as simpler methods include:

- Otsu’s method, based on the minimization of variance of grayscale values between phases under different trial threshold values (Otsu 1975).

- Clustering by Gaussian Mixture Models, where the grayscale value histogram is modeled as a mixture of two or more Gaussian distributions from different phases, with an optimum threshold value selected based on the best Gaussian fit for each phase (McLachlan and Peel 2004, Everitt 2011).

- Edge detection, a method that relies on pronounced changes in brightness (Canny 1986; Thakkar and Shah 2011; Spontón and Cardelino 2015).

- Volumetric approach, where bulk air content is measured by an alternative means (e.g. gravimetrically or by pressure meter) and the grayscale threshold varied to minimize the difference between the X-ray CT measurement of bulk air content and the benchmark alternative measurement (Zelelew and Papagiannakis 2011; Hu et al. 2013, Shaheen et al 2016).

A more extensive review of different thresholding methods can be found in Sezgin (2004).

3.2.2 Segmentation based on spectral analysis

It is well known that sound can usually be decomposed into independent signals with different frequencies and aptitudes. Thus, it can be edited via eliminating or transforming some of the decomposed components. For images, a fluctuation in pixel brightness along a specific direction is analogous to a signal with some frequency (how fast the brightness fluctuates) and aptitude (the grayscale contrast for neighboring fluctuation). Under many situations, different objects in an image usually present different brightness patterns. If these patterns can be decomposed, many processing tasks are made possible. A group of methods following these lines, termed spectral analysis, has been developed and is widely used in image processing. Among them, Fourier analysis is one of the most widely used methods. Taking 2D images as an example, they
can be treated as a bivariate function \( f(m, n) \) with \( m \), \( n \) as the spatial coordinate and the value of \( f(m, n) \) as the grayscale value. Then a Fourier transform can be performed on \( f(m, n) \) (Equation 3.2). The result of this transform is a bivariate function \( F(x, y) \), in which image components with different frequencies can be much more easily identified. An inverse Fourier transform to convert \( F(x, y) \) back to \( f(m, n) \) is provided in Equation 3.3:

\[
F(x,y) = \sum_{m=0}^{M-1} \sum_{n=0}^{N-1} f(m,n) e^{-j2\pi(x\frac{m}{M}+y\frac{n}{N})}
\]

Equation 3.2

\[
f(m,n) = \frac{1}{MN} \sum_{x=0}^{M-1} \sum_{y=0}^{N-1} F(x,y) e^{j2\pi(x\frac{m}{M}+y\frac{n}{N})}
\]

Equation 3.3

Where

\( M = x \) dimension of the image
\( N = y \) dimension of the image

To briefly illustrate this method, a Fourier transform is applied to four 30×30 pixel synthetic images (i.e., original images, \( f(m, n) \)) in the top row of Figure 3.4.

![Fourier transform on synthetic images](image)

The corresponding results of the transform are listed below the original images (i.e., the Fourier transform images, \( F(x, y) \)). For the first three images, due to the simple pattern of brightness
fluctuations, it is not difficult to observe how the pixel brightness fluctuates. These patterns are revealed more explicitly and quantitatively in their corresponding Fourier transform images. The direction of the line connecting the two points in Fourier transform images represents the direction of brightness fluctuation. The distance between the two points shows the frequency of the brightness fluctuation. The fourth image is an image with the patterns from the first three images blended together. Based on the information of its Fourier transfer, the three pattern components can be decomposed and thus any of them of can be removed as desired. Also, some or all of the pattern components can constitute a vector of features to differentiate objects in an image. More detailed introductions to Fourier transform theory as well as the implementation algorithms are found in Van Vleck (1914), Brigham (1988), or Zonst (2004).

3.2.3 Segmentation based on active contour line

Compared with the segmentation methods already discussed, there is a relatively new type of method called active contour line, also known colloquially as “snakes” (Kass et al. 1988; Terzopoulos et al. 1987). The basic idea is to evolve a curve, which can move and deform dynamically to delineate the boundary of an object in an image (Micula and Micula 2012). A schematic illustration of the process is shown in Figure 3.5.

![Figure 3.5 Process of active contour line method, from Tang et al. (2016).](image)

The key to the method is the criteria followed to decide the optimum location of this active contour, i.e. the boundary/edge of an object. The criteria of the optimum location are based on the regularity of boundary curve and distribution of grayscale values within the curve (e.g. the uniformity of the grayscale value). The former is usually expressed in a form called “internal energy” and the latter as “external energy”. The combination of the two parts of the energy is called the energy functional, a concept borrowed from energy methods in mechanics (Reddy 2002; Cassel 2013). The best possible boundary curve is found by minimizing the energy functional. Different schemes for defining the internal and external energy has lead to different
types of active contour line methods, which in turn achieve segmentation to varying degrees of accuracy and require varying degrees of computing efficiency. The formulation of the original active contour model developed by Kass et al. (1988) is provided in Equation 3.4.

$$E[x(r)|u, 0 \leq r \leq 1] = \int_0^1 (\alpha|x'(r)|^2 + \beta|x''(r)|^2) dr + \mu \int_0^1 g(|\nabla u|(x(r)))^2 dr$$  \hspace{1cm} \text{Equation 3.4}

Where:
- $E[x(r)]$ = the energy functional with the shape of boundary $x(r)$ as the independent variable.
- $x(r)$ = the arc curve length of the parameterized boundary curve.
- $u$ = the segmented image to seek.

The first term on the right hand side of Equation 3.4 is the internal energy term, which provides the controlled connectivity of the potential boundary curves. The second term is the combined gradient of grayscale values within a potential boundary curve. If a single object needs to be segmented and at the same time a salient gradient of gray values exists, active contour methods work very well. In reality, there are situations where a gradient of grayscale values does not necessarily exist or the level of noise is large enough to obscure the gradient. Furthermore, in most cases the number of objects to segment is greater than one. To solve these problems, various methods have been developed (Leitner and Cinquin 1991; Szeliski et al. 1993, Caselles et al. 1997, Xu and Prince 1998; Chan and Vese 2001). More extensive introductions to active contour lines can be found in Chan and Shen (2005), Metaxas 2012, and Mitiche and Ayed (2010).

### 3.2.4 Segmentation based on random fields

Another relatively new type of segmentation method is called random fields method. In this method, images are first divided into a group of nodes and each node corresponds to pixels or agglomeration of pixels. For each node, a hidden variable is usually attached. In image processing, the grayscale value is often chosen naturally as the hidden variable. The general framework can be formulated as in Equation 3.5:

$$P(x|z) \propto P(z|x)P(x)$$  \hspace{1cm} \text{Equation 3.5}

Where:
- $x$ = a vector variable representing the possible labels of all nodes in an image.
- $z$ = a vector variable representing the grayscale value of all nodes in the same image.
If the distribution of possible labels \( x \) is known (e.g. the percentage of different phases in a certain image), and the statistical distribution of grayscale values for a given phase can be known as well, the segmentation problem then becomes a statistical inference problem that estimates the most possible labels \( x \) based on the observation \( z \). At this point, it is possible in theory to carry out an exhaustive look at all the possible variable spaces and infer the \( P(x|z) \). However, considering the high resolution of modern microscope images, it is impossible or at least impractical to carry out the exhaustive computations. This is where the core idea of random field method has to come into play. It is known that that neighboring nodes tend to be labeled as the same class. In probabilistic terms this can be expressed as in Equation 3.6:

\[
\begin{align*}
P(X_i = x | X_{i-1} = x') &= \alpha; \\
P(X_i = x | X_i = x) &= \beta
\end{align*}
\]

Equation 3.6

In Equation 3.6 \( X_i \) and \( X_{i-1} \) can be regarded as the labels of two neighboring nodes, while \( \alpha \) and \( \beta \) are the possibilities that the neighboring nodes have the different and same labels respectively. For the Markov model this dependency can be thought as only related to its immediate neighbors. In this way, longer-range dependencies can be realized through a short range ‘knock-on effect’. For the degree of the dependency between neighboring nodes, it can be manipulated using different numbers of edges connecting neighboring nodes as in Figure 3.6. Detailed background information, as well as implementation algorithms, are provided in Derin and Elliot (1987), Dubes and Jain (1989), Li (2009), and Blake et al. (2011).
3.2.5 Summary statement of segmentation methodologies

The segmentation methodologies reviewed here include the most commonly used as well as relatively new methods. Not all of them are applied in this study. All of them have their own strengths to attack specific problems. For images with limited noise and limited types of phases, applying a uniform grayscale threshold is the most economic method. For images with distinct frequency components, which usually can be identified with rich patterned textures, spectral analysis based methods can be used effectively. For random fields and active contour line based methods, two of the relative newcomers, improved versions are still active research topics. These methods are usually robust with regard to noise, but at the same time have a much higher computational demand. For images with strong probabilistic components, such as many natural scenes, random fields based methods of segmentation perform very well. On the other hand, active contour line based methods have a distinct advantage when dealing with artificial objects with regular outlines.
4 Measurement of air void system in light-weight concrete

Chapter 4 is derived from a paper published in the Proceedings of the 36th International Conference on Cement Microscopy; International Cement Microscopy Association, Milan, Italy, April 13-17, 2014.

4.1 Introduction

This chapter presents the process and results of characterizing the entrained and entrapped air void size distribution in a lightweight concrete using micro X-ray CT (µCT), and outlines practices for the processing and analysis of µCT images. Practical difficulties as well as possible uncertainties shared by other cement-based materials are emphasized. Considering the many factors that may contribute to the final accuracy of measurement, such as instrument configuration (e.g., nature and spectrum of incident X-ray beam, detector capacity), scanning parameters (e.g., scan resolution, scan time), instrument operation (e.g., field of view, sample motion, image artifacts, sample size), image-based processing (e.g. noise removal, thresholding, segmentation ), and the inherent properties of the scanned material (e.g. homogeneity, contrast among phases, random inclusions), it is impossible to take into account of all the factors required for a comprehensive discussion of the uncertainty of any quantitative measurement. Rapid developments in X-ray beam generation, detection technology, reconstruction algorithms and noise reduction strategies have all led to the ever-increasing resolution of the images as well as improved signal-to-noise levels. With this premise, the discussion of uncertainty in this study is biased towards image-based processing, which is usually where many subjective choices have to be made and thus provides one of the largest sources of uncertainty. Here a typical use of µCT to characterize cement-based materials is demonstrated towards a more quantitative end. The results from such efforts can provide data for people involved in performance simulation, or to estimate the implications of uncertainty from geometric inputs within the larger context of uncertainty quantification. In addition, the results identify issues, and demonstrate the potential for future full-scale validation protocols for the application of µCT analyses in typical cement-based materials, in which standard materials or sometimes parallel testing techniques have to be involved.

The particular lightweight concrete sample examined here is used in insulative coatings on pipes for offshore oil applications. The air-voids within the concrete can be subdivided into three general classes: air voids within the lightweight expanded glass aggregate (LWA), air voids
within the hollow glass microspheres, and the entrained or entrapped air-voids within the hardened cement paste (hereafter referred to as air voids in the paste). Since LWA and hollow glass microspheres are added in known quantities to the mixture, only the air voids in the paste remain as unknown entities. The distinction between the air voids attributed to LWA and hollow glass microspheres (hereafter referred to collectively as air voids in aggregate) from the air voids in the paste poses significant difficulties for the digital images collected by µCT, as outlined in later sections.

µCT methods for the characterization of air voids in lightweight concrete have been widely utilized over the past few years. The work of Tekin et al. (2012) and Tekin et al. (2015) focused on the quantification of total macro porosity in concrete made with ground pumice. Lanzón et al. (2012) examined the relationship between open porosity (connected air voids) and physical measurements of capillary water absorption in concretes produced with expanded perlite LWA, expanded glass LWA, and hollow glass microspheres. The same team of researchers later utilized similar approaches to study the bond between expanded polystyrene waste LWA and the paste matrix, and the bond between crumbled tire rubber and the paste matrix (Lanzón et al. 2015). Farhan et al. (2016) used µCT to assist with visualization of cracks in concrete containing crumbled tire rubber after tensile strength testing, and Lee et al. (2016) used µCT to assess total macro porosity in concrete containing polyethylene and polypropylene LWA. Foamed concrete research is another active area utilizing µCT. Crandall et al. (2014) explored the air void size distributions in foamed concrete produced at both elevated and atmospheric pressures. Wei et al. (2013) and Chung et al. (2016a & 2016b) examined relationships between air void size distributions and thermal characteristics of foamed concrete, and Wei et al. (2014) examined relationships between air void size distributions and acoustic characteristics of foamed concrete.

4.2 Material and method

4.2.1 Specimen preparation and X-ray scan configuration

The constituents of the lightweight concrete specimen are summarized in Table 4.1.
Table 4.1 Mix design of lightweight concrete.

<table>
<thead>
<tr>
<th>Constituents</th>
<th>Reported wt. %</th>
<th>Reported vol. %</th>
<th>Density(^a) g/cc</th>
<th>kg/m(^3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>18.6</td>
<td>17.2</td>
<td>1.00</td>
<td>171.7</td>
</tr>
<tr>
<td>Blended portland cement</td>
<td>46.6</td>
<td>14.3</td>
<td>3.01</td>
<td>430.1</td>
</tr>
<tr>
<td>Hollow glass spheres (20 - 80 µm dia.)</td>
<td>4.1</td>
<td>11.4</td>
<td>0.33</td>
<td>37.8</td>
</tr>
<tr>
<td>Expanded glass LWA (0.25- 1 mm dia.)</td>
<td>29.7</td>
<td>45.7</td>
<td>0.60</td>
<td>274.1</td>
</tr>
<tr>
<td>Alkali-resistant glass fibres</td>
<td>0.6</td>
<td>0.2</td>
<td>2.77</td>
<td>5.5</td>
</tr>
<tr>
<td>Liquid chemical admixtures</td>
<td>0.4(^b)</td>
<td>0.4(^c)</td>
<td>1.02</td>
<td>3.7</td>
</tr>
<tr>
<td>Air voids in the paste</td>
<td>-</td>
<td>10.8(^d)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>100.0</td>
<td>100.0</td>
<td>922.9</td>
<td></td>
</tr>
</tbody>
</table>

\(^a\) Derived from the reported wt. % and vol. % values using a water density of 1.00 g/cc.
\(^b\) Computed as the difference between 100 wt. % and the sum of the other constituents.
\(^c\) Computed based on an assumed density of 1.02 g/cc.
\(^d\) Computed as the difference between 100 vol. % and the sum of the other constituents.

Since the lightweight concrete is a proprietary product, information regarding the manufacturer’s precise mix design data was necessarily limited to some degree. A mix design based on wt. % and vol. % values was provided by the manufacturer for all of the constituents, except for the liquid admixtures, along with a target entrained/entrapped air content value of 10 vol. %. To translate this information into a kg/m\(^3\) mix design framework, several steps were taken as follows:

- The wt. % of liquid admixtures was computed as the difference between 100 % and the sum of the other constituents.
- Assuming a density of 1.00 g/cc for the mix water, densities were derived for the other constituents based on the reported wt. % and vol. % values.
- Since vol. % was not reported for the liquid chemical admixtures, it was computed based on an assumed density of 1.02 g/cc.
- The vol. % for the air voids in the paste was computed as the difference between 100 % and the sum of the other constituents.

With wt. %, density, and air content vol. % values established, expression of the mix design in units of kg/m\(^3\) was straightforward.

A single 20 mm thick section cut from a 50 mm dia. cast cylinder was provided for testing (Figure 4.1). The sample as received, designated LW1, was scanned with a GE Vtome|x240D \(\mu\)CT machine through a complete rotation of 360° with 0.33° rotation increments, at an accelerating voltage of 110 kV, a current of 200 µA, and with a 0.5 mm Cu filter. The resolution of the X-ray detector is fixed at 1018 × 1024 pixels, and the cylinder was positioned to achieve a
projected 2D image resolution of 55 µm/pixel. The selection of this particular resolution was based on an optimization of the maximum possible resolution achievable given X-ray attenuation that occurred as the beam passed through the 50 mm dia. sample.

After the initial scan, a 6 mm dia. core was retrieved from sample LW1, and cut in half to provide two subsamples (Figure 4.1). These subsamples, designated LW2a and LW2b, were both scanned through a complete rotation of 360° with 0.33° rotation increments, at an accelerating voltage of 90 kV, a current of 160 µA, and with a 0.1 mm Cu filter. The core segments were positioned to achieve a projected 2D image resolution of 9 µm/pixel. Again, the selection of this particular resolution was based on an optimization of the maximum possible resolution achievable given X-ray attenuation that occurred as the beam passed through the 6 mm dia. sample. To help account for the effect of beam instability that may occur between scans, an aluminum stud was installed immediately below each core segment to serve as a control for calibration purposes.

The 55 µm/pixel scan of the 50 mm dia. cylinder was performed to capture the large (entrapped) air voids in the paste. The 9 µm/pixel scans of the 6 mm dia. cores were performed to capture the smaller (entrained) air voids in the paste. The methodologies behind the isolation of the air voids in the paste are similar for both scales, and outlined in detail in Sections 4.2.2.1 and 4.2.2.2.

Due to the difficulty in completely avoiding breakaways of small segments during coring, as well as the instabilities in the X-ray beam around the edges of a specimen, the final dimensions
of the regions of interest (ROIs) subjected to analysis were slightly smaller than the specimens themselves, as listed in Table 4.2.

After each scan, a 3D reconstruction was performed using the manufacturer’s software to remove imaging artifacts resulting from variations in sensitivity of individual detector elements (ring artifacts), differential attenuation of the polychromatic X-ray source (beam hardening), and shifting/movement of the sample during data collection (Stock 2009).

Table 4.2: Specimen IDs and dimensions.

<table>
<thead>
<tr>
<th>Specimen ID</th>
<th>Specimen dimensions</th>
<th>Dimensions of ROI</th>
</tr>
</thead>
<tbody>
<tr>
<td>LW1</td>
<td>50.8 mm dia. 19.0 mm h. cylinder</td>
<td>49.50 mm dia., 17.82 mm h. cylinder</td>
</tr>
<tr>
<td>LW2a</td>
<td>6.0 mm dia. 6.0 mm h. core</td>
<td>5.58 mm dia., 5.62 mm h. cylinder</td>
</tr>
<tr>
<td>LW2b</td>
<td>6.0 mm dia. 6.0 mm h. core</td>
<td>5.49 mm dia., 5.62 mm h. cylinder</td>
</tr>
</tbody>
</table>

4.2.2 Air void system measurement

As already mentioned in Section 4.1, the primary objective of this analysis was to differentiate between air voids in aggregate and air voids in the paste. To help illustrate the complexities of this problem, it is useful to approach it from the traditional methodology of air void characterization using a cut and polished surface. Figure 4.2(a) shows a composite false-color red-green-blue (RGB) image constructed using three consecutive scans collected from a cut and polished surface of the lightweight concrete using a flatbed scanner at a pixel resolution of $8 \times 8 \mu$m (3,175 dpi, 125 dpm). In Figure 4.2(a) the red band consists of a grayscale image of the as-polished sample, the green band consists of a grayscale image after the application of a phenolphthalein stain, and the blue band consists of a grayscale image after painting the surface black and pressing white powder into the air voids.
The different surface treatments when overlaid in the RGB color scheme results in LWA particles that appear yellow, paste that appears red, and air voids that appear blue. Figure 4.2(b) consists of a spectrally classified image that isolates three phases: solid aggregate, solid paste, and air voids; this same general approach to air void analysis has been used by several researchers over the past decade (Peterson et al. 2001; Zalocha and Kasperkiewicz 2005; Wawrzeńczyk and Kozak 2016). In the classified image, air void pixels completely surrounded by aggregate pixels can be easily reclassified as air voids in aggregate using a standard image-processing fill-holes operation. However, due to the limitations of sample preparation and image resolution, a significant portion of the air void pixels appear simultaneously in contact with both aggregate and paste pixels, making it difficult to discern between air voids in aggregate and air voids and paste in an automated fashion. To overcome this issue, a step-wise approach was adopted to process the μCT images, as outlined in Sections 4.2.2.1 and 4.2.2.2. A remaining issue is the separation of air voids within the hollow glass spheres from air voids in the paste. This is addressed in the final step covered in Section 4.3.2 after the combination of the air void distributions from the 55 µm/pixel and 9 µm/pixel data sets.

4.2.2.1 Image processing of 9 µm/pixel scans

Figure 4.3 shows the basic steps used to differentiate between phases in the lightweight concrete in the 9 µm/pixel data sets. These steps are described in detail in the subsequent sections.
4.2.2.1.1 Isolation of LWA

Figure 4.4 shows an example grayscale histogram for the full 3D reconstructed image from subsample LW2a. To calibrate the images, the histogram peaks representing the aluminum stud and the empty space surrounding the sample were set as the lower and upper intensity limits respectively, and the image linearly rescaled to the full $2^{16}$ range to increase contrast between the phases, as illustrated in Figure 4.5.
Figure 4.5(a) consists of a single slice extracted from the 3D reconstructed image after cropping to the ROI. A post-stretch histogram representing all of the slices from subsample LW2a is provided in Figure 4.5(b) and the grayscale profiles corresponding to lines AA’ and BB’ are provided in Figures 5(c) and 5(d) respectively. The grayscale profiles show no explicit signals that can be interpreted as noise and removed using a wavelet based method. Furthermore, the mixed contribution of grayscale signals from the multiple phases present poses no easy way for them to be decomposed; especially since knowledge of the individual distributions of grayscale values for the solid phases were not available.

To address this issue, a step-wise approach was employed to reduce the problem into a series of easier-to-solve two-phase problems. The first step was to isolate a significant number of LWA
Figure 4.5: (a) 2D slice from sample LW2a, (b) post-stretch histogram from full 3D image, (c) grayscale profile from line XX’, (d) grayscale profile from line YY’.

particles, thus reducing the grayscale signals present to solid aggregate and air void in aggregate. This was accomplished by using a popular image segmentation algorithm called “active contour line.” Active contour line is based on a variational partial differential equation (PDE) framework where different phases are represented as disjoint regions, and segmented to decide the boundaries of these regions (Mumford and Shah 1989; Rudin et al. 1992; Chan and Vese 2001). As in its name, this algorithm works by starting with an initial ‘seed’ in the form of a geometrical line that can grow itself based on constantly updated information of the grayscale value gradient.
around the line (the external energy) and the geometric regularity of the growing line (the internal energy). This algorithm has been rapidly developed in the past fifteen years to a level that open source codes are readily accessible and adaptable to build into image processing models. The main inputs include the coordinate of one point anywhere within the phase of interest, as well as an initial configuration for the contour line. In this study, the difference between clustered air void patterns within LWA and the dispersed air void patterns within the cement paste makes it a trivial task to locate points within the LWA (Figures 4.6a and 4.6b).

To identify starting points for the initial contour lines within LWA particles, a preliminary grayscale threshold level of 0.70 was selected to isolate both air voids in the paste and LWA particles in a general sense on a 2D slice (Figure 4.6b).

![Figure 4.6: (a) 2D slice showing selected LWA particles, (b) rough threshold to illustrate undifferentiated air voids in aggregate and air voids in the paste.](image)

Next, the 2D solidity was tabulated for each object, where the term ‘object’ is defined by a 2D set of connected air void pixels. As an initial estimate, objects with 2D solidity values \( \leq 0.3 \) were identified as potential LWA particles. 2D solidity is defined as the proportion of area of an object to the area of its convex hull (Clapham and Nicholson 2009). By definition a circle and a rectangle would both have a 2D solidity of 1, while shapes with more complicated indented perimeters would have a 2D solidity \(< 1\). For example, the five-pointed star of Figure 4.7 has a 2D solidity close to 0.5.
Figure 4.7: Five pointed star (a), and pentagon that defines the convex hull of the five pointed star (b). The area of the five pointed star divided by the area of the pentagon is a measure of the star’s 2D solidity.

The contour line ‘seeds’ used in this study were circles with a radius of 10 pixels, and placed within LWA particles identified by the initial grayscale threshold and 2D solidity cut-off level. Two examples of cropping the LWA are shown in Figures 4.8. The general degree of accuracy of aggregate cropping is demonstrated in Figure 4.9 with 256 examples. It is stressed here that the main purpose was not to segment the LWA exactly, but rather to obtain a significant sample of pixels that fell inside the general boundaries of LWA particles.

Figure 4.8: Examples of growth of contour line front for the LWA particles indicated in Figure 4.6.
4.2.2.1.2 Separation of solid and void phases within LWA

After a significant number of cropped images from LWA are available, Otsu’s method, often used for segmenting two-phase materials, can be applied to each cropped image to obtain an appropriate threshold for each individual image (Otsu 1975). The distribution of the threshold values between solid aggregate and voids in aggregate from 5,500 LWA particles is shown in Figure 4.10.
From Figure 4.10, a central tendency of the threshold value clearly emerges. However, to strictly validate the assumption that threshold values will converge to a value that can well represent the entire specimen, empirical measurements based on different numbers of samples were carried out, and modeled on a process to prove that the statistic is consistent and efficient. In this study, the mode of the distribution was chosen as the best estimate. Since knowledge of the underlying distribution of the threshold values is absent, in order to achieve a high accuracy estimate of uncertainty, bootstrapping was employed. Bootstrapping is a computational statistical technique based on resampling that often provides a much more accurate estimation than forcing a distribution assumption (Efron and Tibshirani 1986; Davison and Hinkley 1997). The results of the best estimate of the threshold value as well as the uncertainty based on the different numbers of cropped images based on the bootstrap method are shown in Figure 4.11.
From Figure 4.11, it is found that the proposed method can converge rapidly with well-bounded uncertainty even when a moderate number of cropped LWA samples are available.

### 4.2.2.1.3 Partitioning LWA and paste solid phases

Based on the best estimate for the air void threshold from the previous step, it was applied globally to the entire 3D image. Figure 4.12 shows the results for combined voids in the paste and voids in aggregate using the same bootstrap approach as Figure 4.11. After applying the
global threshold, the air void content portion of the image was removed, leaving a 3D image that had just two phases: the solid portion of the LWA, and the solid cement paste. The same general approach was used as in the previous step to arrive at a grayscale threshold value between the solid portion of the LWA and the cement paste.

Figure 4.12: Convergence on total air void content (air voids in aggregate plus air voids in the paste) from sample LW2a (a) and sample LW2b (b).

To avoid biased sampling of the solid phases (solid aggregate and paste) rectangular ROIs with random locations and sizes were used to sample 2D slices as shown in Figure 4.13. Based on
50,000 individual ROIs, Otsu’s method was used to find an appropriate threshold between solid paste and aggregate, with the distribution shown in Figure 4.14.

Since the uncertainty from the initial air void threshold is included as one of the random factor in the second stage sampling, it is possible to assess the uncertainty in this step as independent from the previous air-void threshold. This provides a practical convenience for the uncertainty estimation presented at the end of this Section.
From Figure 4.14 it is found that the distribution follows a quasi-normal distribution. Same as in the estimation of the threshold value between the LWA air voids and solids, the mode of the distribution was chosen as the best estimate. The speed of the convergence of the estimation against the number of random samples and the associated uncertainty in terms of the threshold value and the paste content are shown in Figures 4.15 and 4.16 respectively. As the number of ROIs increases, a trend of convergence is clearly observed, and validates its use as an estimate of the “true” threshold value for the entire specimen.

Figure 4.15: Convergence on best estimate of threshold between LWA and paste solid phases from sample LW2a (a) and sample LW2b (b).
Figure 4.16: Convergence on total paste content from sample LW2a (a) and sample LW2b (b).

Figure 4.17 provides a visual comparison of the effectiveness of the step-wise approach (Figure 4.17b) to paste and aggregate segmentation as compared to thresholding based on the multiphase Otsu’s method (Figure 4.17a). Although the absence of knowledge of the exact phase boundaries prevents validation of segmentation effectiveness in a strict sense, a much better segmentation of air-voids can be argued for the step-wise method.
As demonstrated in Figures 4.15 and 4.16, the uncertainty is controlled by the number of ROIs. This is a critical factor influencing the general effectiveness of the proposed method or its potential use in segmenting images of other types of materials. The choice of 50,000 ROIs was based on economic considerations in terms of the computation load and in the reduction of uncertainty. As such, an exploration of the use of fewer ROIs is presented in Tables 4.3 and 4.4. Although the results from Tables 4.3 and 4.4 are similar to the results shown in Figures 4.11, 4.12, 4.15, and 4.16, the main purpose here is to treat the uncertainty as it happens over the whole process covering all steps, and provide a more quantitative result when considering the effectiveness of the proposed method.
Table 4.3. Uncertainty of the paste segmentation for specimen LW2a.

<table>
<thead>
<tr>
<th># LWA samples</th>
<th>Air content (%)</th>
<th>95% Confidence interval</th>
<th>Paste content (%)</th>
<th>95% Confidence interval</th>
<th>Aggregate content (%)</th>
<th>95% Confidence interval</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>28.3</td>
<td>(27.4, 31.7)</td>
<td>26.8</td>
<td>(26.2, 28.1)</td>
<td>44.9</td>
<td>(40.2, 46.4)</td>
</tr>
<tr>
<td>500</td>
<td>27.5</td>
<td>(25.9, 28.2)</td>
<td>28.4</td>
<td>(27.5, 29.4)</td>
<td>44.1</td>
<td>(42.4, 46.0)</td>
</tr>
<tr>
<td>1000</td>
<td>27.7</td>
<td>(26.9, 28.0)</td>
<td>28.9</td>
<td>(27.6, 30.2)</td>
<td>43.4</td>
<td>(41.8, 45.5)</td>
</tr>
<tr>
<td>2000</td>
<td>27.7</td>
<td>(27.3, 27.9)</td>
<td>27.7</td>
<td>(27.4, 28.7)</td>
<td>44.6</td>
<td>(43.4, 45.3)</td>
</tr>
<tr>
<td>3000</td>
<td>27.6</td>
<td>(27.1, 27.7)</td>
<td>27.1</td>
<td>(25.4, 27.3)</td>
<td>45.2</td>
<td>(45.0, 47.5)</td>
</tr>
<tr>
<td>4000</td>
<td>27.8</td>
<td>(27.5, 28.1)</td>
<td>28.2</td>
<td>(27.7, 28.9)</td>
<td>44.0</td>
<td>(43.0, 44.8)</td>
</tr>
<tr>
<td>5000</td>
<td>27.6</td>
<td>(27.2, 27.8)</td>
<td>28.0</td>
<td>(27.3, 28.6)</td>
<td>44.4</td>
<td>(43.6, 45.5)</td>
</tr>
</tbody>
</table>

Table 4.4. Uncertainty of the paste segmentation for specimen LW2b.

<table>
<thead>
<tr>
<th># LWA samples</th>
<th>Air content (%)</th>
<th>95% Confidence interval</th>
<th>Paste content (%)</th>
<th>95% Confidence interval</th>
<th>Aggregate content (%)</th>
<th>95% Confidence interval</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>25.6</td>
<td>(23.3, 26.4)</td>
<td>28.5</td>
<td>(28.4, 29.1)</td>
<td>45.9</td>
<td>(44.5, 48.4)</td>
</tr>
<tr>
<td>500</td>
<td>25.9</td>
<td>(24.6, 26.5)</td>
<td>28.3</td>
<td>(27.4, 29.3)</td>
<td>45.8</td>
<td>(44.2, 48.0)</td>
</tr>
<tr>
<td>1000</td>
<td>26.0</td>
<td>(24.8, 26.7)</td>
<td>28.2</td>
<td>(27.2, 29.2)</td>
<td>45.8</td>
<td>(44.1, 48.0)</td>
</tr>
<tr>
<td>2000</td>
<td>25.6</td>
<td>(24.7, 25.9)</td>
<td>27.9</td>
<td>(27.2, 28.5)</td>
<td>46.5</td>
<td>(45.6, 48.1)</td>
</tr>
<tr>
<td>3000</td>
<td>25.5</td>
<td>(24.5, 25.7)</td>
<td>28.4</td>
<td>(27.1, 29.7)</td>
<td>46.1</td>
<td>(44.6, 48.4)</td>
</tr>
<tr>
<td>4000</td>
<td>25.7</td>
<td>(24.7, 26.0)</td>
<td>28.2</td>
<td>(28.0, 29.3)</td>
<td>46.1</td>
<td>(44.7, 47.3)</td>
</tr>
<tr>
<td>5000</td>
<td>25.6</td>
<td>(25.3, 25.9)</td>
<td>28.4</td>
<td>(27.7, 29.7)</td>
<td>46.0</td>
<td>(44.4, 47.0)</td>
</tr>
</tbody>
</table>
4.2.2.1.4 Partitioning voids in LWA from voids in the paste

A careful comparison between the segmented μCT image slice from Figure 4.17b and the segmented flatbed scanner image of Figure 4.2 reveals the same problems for differentiating voids in aggregate from voids in the paste as based on the identities of surrounding pixels. As a result, a different approach based solely on the shape of the air voids was employed.

Figure 4.18a illustrates some of the complexities involved when trying to separate air voids in the paste that are directly adjacent to air voids in LWA, while Figure 4.18b illustrates some of the similarities in appearance between spherical air voids within LWA to spherical air voids in the paste.

To separate air voids in LWA, slice-wise processing was initially tried based on a 2D solidity threshold value of 0.30. This step was applied in a conservative manner that aimed to disrupt the entirety of air-voids in LWA rather than remove the air in LWA altogether. The outcomes of this processing from some randomly selected slices are shown in Figure 4.19. Although the differentiation of air voids in the paste from air voids in LWA appeared satisfactory from a qualitative perspective, it was determined that a more robust 3D approach was necessary for quantitative analysis.
Since a spherical or quasi-spherical shape for air voids in the paste can be reasonably assumed, the degree of overlap between a minimum bounding 3D sphere of an air void and the air void per se is indicative of its degree of sphercity (Larsson 2008). Out of consideration of algorithm speed as well as the accommodation of truncated spheres (caused either by blockage from an LWA particle or by errors in the image reconstruction process that sometimes fail to resolve the tip of an object) a limited number of points contained within the curved part of the surface were randomly chosen to calculate the bounding sphere. Due to the fact that four points that are not on the same plane are sufficient to define a unique sphere, the bounding spheres from the random selection of points on a more spherical air void will tend to overlap with each other more than those from an air void with a less spherical (more angular) shape. Thus, the coefficient of variation (COV) of the bounding sphere radii from these repetitions, as well as the ratio between the volume of original object contained within its minimum bounding sphere and the volume of the minimum bounding sphere (termed 3D spherical solidity) were chosen as the two major parameters used to distinguish between different types of air voids. It is worth mentioning here that 3D spherical solidity proved to be a much more efficient classifier, although the COV of the bounding sphere radii did serve an important role in the fine-tuning of the code. To illustrate the method, Figure 4.20 provides a 3D image of an air void in LWA, along with eight different bounding spheres based on the random selection of ten points and a chart tracking the corresponding 3D spherical solidity and bounding sphere radius values.
Figure 4.20: (a) Air void in LWA (dimensions listed in units of pixels), (b) bounding spheres based on randomly selected points, and (c) the corresponding values for 3D spherical solidity (blue) and bounding sphere radius (green) for each trial.
3D representations of the air voids in the paste from samples LW2a and LW2b based on a 3D spherical solidity threshold of 0.70 are shown in Figure 4.21. The distribution of the 3D spherical solidity and the COV of the bounding sphere radii measurements for different air void radius categories are provided in Figure 4.22.

The general quality of the classification using a 3D spherical solidity threshold of 0.70 is illustrated in Figure 4.23 by a side-by-side comparison between classification outcomes and randomly selected original images. As a sensitivity test, three 3D spherical solidity threshold levels were applied (i.e. 0.65, 0.70, 0.75) with values for number density and total air content tabulated in Table 4.5, and the resulting air void size distributions shown in Figure 4.24.

Figure 4.21: 3D renderings of air voids in the paste contained in sample volume, (a) LW2a, (b) LW2b.
Figure 4.22: Distribution of the 3D spherical solidity and COV of the bounding sphere radii measurements for different air void radius size categories, (a) sample LW2a, (b) sample LW2b.
Figure 4.23: Side-by-side comparisons of 2D grayscale slices and corresponding classified images of air voids in the paste using 3D spherical solidity threshold of 0.70, (a) sample LW2a, (b) sample LW2b.

Table 4.5: Measurements of air-voids in the paste for 9 µm/pixel dataset.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>3D spherical solidity threshold</th>
<th>All entrained air</th>
<th>Diameter ≥ 100 µm</th>
<th>Diameter ≥ 200 µm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Number</td>
<td>Vol. %</td>
<td>Number</td>
</tr>
<tr>
<td>LW2a</td>
<td>0.650</td>
<td>2164</td>
<td>13.69</td>
<td>1309</td>
</tr>
<tr>
<td></td>
<td>0.700</td>
<td>2148</td>
<td>13.59</td>
<td>1289</td>
</tr>
<tr>
<td></td>
<td>0.750</td>
<td>1991</td>
<td>13.35</td>
<td>1213</td>
</tr>
<tr>
<td>LW2b</td>
<td>0.650</td>
<td>2295</td>
<td>11.52</td>
<td>1394</td>
</tr>
<tr>
<td></td>
<td>0.700</td>
<td>2308</td>
<td>11.30</td>
<td>1360</td>
</tr>
<tr>
<td></td>
<td>0.750</td>
<td>2283</td>
<td>11.48</td>
<td>1340</td>
</tr>
</tbody>
</table>
Figure 4.24: Number density of air voids in the paste based on different 3D spherical solidity threshold levels, (a) sample LW2a, (b) sample LW2b.
4.2.2.2 Image processing of 55 µm/pixel scans

Figure 4.25 shows the basic steps used to differentiate between phases in the lightweight concrete for the 55 µm/pixel data set. These steps are described in detail in the subsequent sections.

START

Image enhancement pre-processing and random selection of 2D slices

→ Seed-growing within 2D slices to outline air voids

↓

Application of 3D solidity threshold to full 3D reconstructed image to isolate air voids in the paste

← Selection of range of grayscale thresholds based on pooled histogram from cropped air voids

↓

Sensitivity analysis of output based on initial grayscale threshold levels

→ Selection of best initial grayscale threshold

FINISH

Figure 4.25: Flow sheet of steps taken during processing of 3D reconstructed images from 55 µm/pixel scan data.

4.2.2.2.1 Initial air void grayscale thresholding

Histogram equalization was performed on the 3D reconstructed image to increase the contrast between different phases. In order to remove noise as well as to strengthen the geometrical shape of individual air voids, a low pass (one standard deviation) 3D Gaussian filter with a structural element of 3 × 3 × 3 pixels was also applied. An example of a 2D slice before and after the histogram equalization and filtering, as well as 10 × 10 mm close-ups are provided in Figure 4.26. The histogram of the original 2D slice and the histogram after processing are shown in Figure 4.27.

For scans at 55µm/pixel due to the generally smaller size of air voids within LWA relative to the scan resolution, as well as the level of inherent signal noise associated with the scan resolution, the air voids within LWA are not resolved accurately in most cases. In some ways, this simplified the segmentation of air voids in the paste, but it also posed challenges for the selection of a grayscale threshold that would simultaneously preserve both their number as well as their size. Grayscale threshold values that better preserved the size of the air voids in the paste also tended to inadvertently include some voids in LWA, and lead to an overestimation of the number air voids in the paste, as shown in Figure 4.28.

56
Figure 4.26: Example 2D slice before (a) and after (b) performing histogram stretch and filtering, and corresponding close up 10 x 10 mm region.

Figure 4.27: Histogram from 2D slice prior to equalization (a), histogram from 2D slice after equalization (b), and histogram from 2D slice after Gaussian smoothing (c), with arbitrary choice of initial air void threshold.
Given the even spread of histogram grayscale values, there were no explicit peaks that could be taken advantage of to easily set the threshold between the solid and air phases. Thus, in order to best preserve the number as well as the size of air voids in the paste, a direct method based on pooling the grayscale values was used. The same active contour line strategy described in Section 4.2.2.1.1 for the 9 um/pixel scan data set was used to obtain a population of grayscale values representative of the air voids. An initial grayscale threshold of 0.70 was applied to generally identify both air voids in the paste and LWA particles. Objects with 2D solidity values \( \geq 0.90 \) were identified as air voids in the paste, and contour line seeds were placed within.

Figure 4.29 shows examples of 625 cropped air voids in this manner, and Figure 4.30 shows the pooled grayscale histogram. Pixels in the left-hand tail of the histogram distribution have grayscale intensities somewhere in the intermediate range between solid (darker) phases and (brighter) empty void space. Different threshold levels were selected based on the percentage of pixels contained within this intermediate range. The threshold intervals are provided in Figure 4.30, and their corresponding effects on a 2D slice image are shown in Figure 4.31.
Figure 4.29: (a) Air voids and surrounding matrix, (b) Air voids after active contour line cropping, a scale bar is omitted, since air voids of all different sizes were scaled to prepare the montage image.

Figure 4.30: Pooled grayscale histogram from cropped air voids.
Technically, there is no established criterion to follow in this situation to choose one specific value as the threshold. Considering the final purpose, i.e. to obtain the size distribution of air voids in the paste, maintaining the shape of the air voids in the paste should be a priority during image processing. However, the use of a bounding sphere to represent the air voids in the paste would eliminate sensitivities to small perturbations in the original dimensions of an air void object. In this respect, the selected threshold value is a tradeoff between preserving the size of the air voids in the paste, and the unwanted inclusion of air voids in LWA. From Figure 4.31, it can be seen that grayscale threshold levels corresponding to $< 6\%$ of the intermediate zone result in the inclusion of some air-voids in LWA. However, when the threshold levels
corresponding to $\geq 6\%$ of the intermediate zone are applied, the sizes of air voids in the paste are slightly underestimated. As a practical solution, a range of grayscale threshold values, instead of one specific value, were applied; if the subsequent 3D shape criterion for separating voids in the paste from voids in LWA is robust enough, the final paste air-void distribution should converge to a reasonable bound that will be adequate for engineering purposes. The threshold levels that correspond to the intermediate zone boundaries of 4%, 5%, 6%, 7%, and 8% were selected to go forward to the next stage of 3D shape assessment. Figure 4.32 shows example 10 $\times\ 10\ mm$ 2D slices after application of the 4 – 7 % intermediate zone grayscale threshold levels.

![Figure 4.32: Application of grayscale threshold levels to 2D image slices corresponding to percentages of pixels within intermediate zone from 4 – 7% (a-d).](image-url)
4.2.2.2.2 3D shape based filtering for isolation of air voids in the paste

The same 3D spherical solidity threshold approach introduced in Section 4.2.2.1.4 for the 9 µm/pixel scan data set was used to identify voids in the paste for the 55 µm/pixel scan. Figure 4.33 shows a 3D rendering of a typical air void in the paste, along with eight examples of different bounding spheres based on a random selection of ten points.

The general distribution of the 3D spherical solidity values and the COVs of bounding sphere radii for different size ranges of air voids are shown in Figure 4.34. To identify air voids in the paste with a radius ≤ 150 µm, a 3D spherical solidity cut-off of 0.70 was applied, while for larger sizes, a 3D spherical solidity cut-off of 0.80 was applied mainly to help address situations of separating connected air voids.

Figure 4.35 compares the results for the size distributions of air voids in the paste as identified by the 3D spherical solidity criteria. The data represent processing based on initial grayscale threshold air void inputs covering the intermediate zone boundaries of 4%, 5%, 6%, 7%, and 8%. Table 4.6 provides summary statistics for the bulk volume of air voids in the paste, and frequency of occurrence for different size categories. Understandably, the numbers of air voids in the paste reported in Table 4.6 are sensitive to the specific grayscale threshold applied. However, considering the total number of air voids present, the results of the proposed classification can still be regarded as adequate for engineering purposes. Based on this sensitivity analysis, a grayscale threshold value based on the intermediate zone boundary of 6% was selected. Figures 4.36 and 4.37 show detailed 3D renderings of the air voids in the paste as classified using the combined grayscale threshold and 3D spherical solidity threshold approach.
Figure 4.33 (a) Air void in the paste (dimensions listed in units of pixels), (b) bounding spheres based on randomly selected points, and (c) the corresponding values for 3D spherical solidity (blue) and bounding sphere radius (green) for each trial.
Figure 4.34: Distribution of the 3D spherical solidity and COV of the bounding sphere radii measurements for different air void radius size categories.

Figure 4.35: Resultant size distributions of air voids in the paste based on different initial grayscale threshold levels.
Table 4.6: Measurements on entrained/entrapped air for specimen scanned at 55 µm/pixel.

<table>
<thead>
<tr>
<th>% of pixels in intermediate zone</th>
<th>Corresponding threshold value</th>
<th>All air voids in the paste</th>
<th>Dia. ≥ 400µm</th>
<th>Dia. ≥ 600 µm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Number</td>
<td>Volume %</td>
<td>Number</td>
</tr>
<tr>
<td>4</td>
<td>0.6055</td>
<td>14555</td>
<td>4.75</td>
<td>8184</td>
</tr>
<tr>
<td>5</td>
<td>0.6171</td>
<td>13472</td>
<td>4.46</td>
<td>7595</td>
</tr>
<tr>
<td>6</td>
<td>0.6265</td>
<td>12709</td>
<td>4.31</td>
<td>7191</td>
</tr>
<tr>
<td>7</td>
<td>0.6367</td>
<td>11943</td>
<td>4.09</td>
<td>6826</td>
</tr>
<tr>
<td>8</td>
<td>0.6459</td>
<td>11319</td>
<td>3.94</td>
<td>6494</td>
</tr>
</tbody>
</table>

Figure 4.36: 3D rendering of air voids in the paste for entire LW1 ROI.
4.3 Results

4.3.1 Combined size distribution based on both scanning resolutions

Strict procedures are usually required to determine whether measurements of material properties collected from samples of a certain volumetric are statistically representative, as outlined by Kanita et al. (2003). However, considering the abundance of air voids in the paste as segmented...
in this lightweight concrete material, an assumption can be made that the dimensions of the ROIs scanned at both 9 µm/pixel and 55 µm/pixel are equal to or larger than the relevant representative volume element (RVE) that would correspond to the range of air void sizes that are practically measurable. Thus, the results from scans at 9 µm/pixel were linearly scaled up to the larger specimen. Following this, a full range size distribution of the air voids in the paste can be achieved through the combination of results from the 9 µm/pixel scan (covering the smaller size range of the air voids in the paste) and the 55 µm/pixel scan (covering the larger size range of the air voids in the paste). The cutoffs used were 100 - 400 µm dia. air voids in the paste from the 9 µm/pixel scan and ≥ 400 µm dia. air voids in the paste for the 55 µm/pixel scan. The combined number density size distributions are shown in Figures 4.38a and 4.38b, and the corresponding volume size distributions of the same combinations are shown in Figures 4.39a and 4.39b.

When the size distribution is expressed in terms of number density, the transition that occurs at a dia. of 400 µm from the air voids in the paste detected at the 9 µm/scan resolution to the air voids in the paste detected at the 55 µm/scan resolution looks relatively smooth. But, when expressed in terms of vol. %, a marked shift occurs in the cumulative distribution at this point. The irregularities, or ‘bumpiness’ of the size distribution when expressed in terms of vol. % at the larger end of the spectrum can be expected, as the volume contribution of singular individual entrapped air voids becomes more pronounced. The practical difficulties in uniting size distributions from data collected at different resolutions are addressed in Section 4.3.3.
Figure 4.38: Combined size distribution for air voids in the paste expressed in terms of number density, LW1 & LW2a (a) and LW1 & LW2b (b).
Figure 4.39: Combined size distribution for air voids in the paste expressed in terms of volume percentage, LW1 & LW2a (a) and LW1 & LW2b (b).
4.3.2 Separating air voids from hollow shell glass sphere aggregates

Even at the higher resolution scan of 9 µm/pixel, it was impractical to include air voids with diameters < 100 µm, as the limited number of voxels contained in such objects precluded the reliable application of the 3D spherical solidity threshold. Even if scans were collected at higher resolutions, a 3D spherical solidity based threshold would not be capable of distinguishing between spherical entrained air voids in the paste and air voids in the hollow glass sphere aggregates. However, the quantity of hollow glass sphere aggregates is known from the mix design (Table 4.1), as well as the manufacturer-listed statistics about their size distribution (Figure 4.40). With this information, it would be possible to subtract the hollow shell glass sphere aggregates from the population of air voids in the paste.

Given the limitations of the 9 µm/pixel scans employed in this research, and the small size of the hollow shell glass sphere aggregates (< 100 µm dia.), it was unnecessary to consider their removal from the detected air voids in the paste, as the smallest diameters included were ≥ 100 µm dia.

Figure 4.40: Manufacturer-listed size distribution for hollow glass spheres.
4.3.3 Smoothing of final entrained/entrapped air void size distribution

The ‘jump’ in Figure 4.39 that occurs at the 400 µm dia. transition can be attributed to contrasts in efficiency for the detection of air voids in this size range at the two different scan resolutions. For the 9 µm/pixel resolution dataset, air voids in the paste at the 400 µm dia. end of the spectrum are detected without any problem. But, for the 55 µm/pixel resolution dataset, air voids of this size are more difficult to detect. Unfortunately, given the wide range of sizes covered between entrained and entrapped air voids, it is not possible to simultaneously capture both ends of the spectrum with a single scan. The diameter of a sample required to capture mm sized entrapped air voids is necessarily on the order of a cm or more, but X-ray attenuation through samples of this size prevents the effective use of the resolutions required to detect the µm scale entrained air voids. When the diameter of a sample is brought down to the mm scale conducive for the detection of smaller entrained air voids, the mm sized entrapped air voids are physically too large to be contained within the sample. For situations like this, one practical solution is to smooth the distribution wherever the transition becomes evident. To accomplish this, a power law statistical distribution was employed (Equation 4.1). Detailed explanations of power law properties and how the parameters are estimated can be found in Mitzenmacher (2004), Newman (2005), and Clauset et al. (2009).

\[
p(x) = \frac{x^{\alpha - 1}}{x_{\text{min}}^{\alpha}} \left( \frac{x}{x_{\text{min}}} \right)^{-\alpha}
\]

Equation 4.1

Where:
\(x_{\text{min}}\) = minimum value of \(x\)
\(\alpha\) = scaling parameter

Because the mechanism of formation of large entrapped air voids is distinct from the bubble-generating nature of air entraining admixtures (AEAs), air voids with dia. ≥ 2 mm were not considered during curve fitting. The results of the curve fitting are shown in Figure 4.41, and the fitting parameters are listed in Table 4.7. Only the distribution of air voids with diameters between 400-1000 µm were modified for the final distributions. For bubbles with sizes outside this range, the original values were used. The modified air void size distributions as based on the power law fit for the combined resolution data sets are expressed in terms of number density in Figure 4.42 and in terms of volume percentage in Figure 4.43. The final results for the bulk air void in the paste content fall within a range of 9-10 vol. %, which is in general agreement with the computed mix design value of 10.8 vol. %.
Figure 4.41: Power law curve fit to air void size distribution.

Table 4.7: Parameters for power law fitted curves.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>LW1&amp;LW2A Average</th>
<th>LW1&amp;LW2A Standard deviation</th>
<th>LW1&amp;LW2B Average</th>
<th>LW1&amp;LW2B Standard deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>$x_{\min}$</td>
<td>245.4650</td>
<td>N/A</td>
<td>240.5153</td>
<td>N/A</td>
</tr>
<tr>
<td>$\alpha$</td>
<td>4.6201</td>
<td>0.0066</td>
<td>4.630</td>
<td>0.0064</td>
</tr>
</tbody>
</table>
Figure 4.42: Combined size distribution for air voids in the paste expressed in terms of number density, LW1 & LW2a (a) and LW1 & LW2b (b) after smoothing the 400-1000 µm dia. region according to fitted power law curve.
Figure 4.43: Combined size distribution for air voids in the paste expressed in terms of volume percentage, LW1 & LW2a (a) and LW1 & LW2b (b) after smoothing the 400-1000 µm dia. region according to fitted power law curve.
4.4 Conclusions

This chapter provides a sound methodology for the use of µCT to characterize the entrained and entrapped air void size distribution in a lightweight concrete. Complicated issues involving the distinction between air voids in the paste from air voids in aggregate were addressed by a combination of segmentation strategies.

The utilization of active contour line algorithm played an essential role in simplifying the segmentation by reducing the grayscale based thresholding to a set of binary two-phase problems. In the example of the 9 µm/pixel (high-resolution) scan dataset, the active contour line was used to isolate LWA particles, allowing for the determination of a grayscale threshold for the undifferentiated air void phase as based on Otsu’s method. After removal of the air void phase from the 3D image, Otsu’s method was used again to identify an effective grayscale threshold between the solid paste and solid LWA phases. In the example of the 55 µm/pixel (low-resolution) scan dataset, the active contour line was to isolate potential entrapped air voids, allowing for a sensitivity based analysis of the pooled grayscale values to identify an effective grayscale threshold between the solid paste and air void phases. In both the high- and low-resolution cases, a 3D spherical solidity based criterion was established to distinguish between voids in the paste and voids in aggregate.

The final issue of the unification of air void size distributions collected at two different resolutions was resolved by fitting the combined distribution with a power law curve. The fitted distribution was used as an approximation of the number of air voids contained within the transition zone between the high- and low-resolution datasets. The resolutions of the scans explored here were unable to capture the smallest entrained air voids in the paste and the air voids in hollow shell glass sphere aggregates, although X-ray CT machines capable of nano-resolution have the potential to capture this size-range range of air voids. Although a 3D spherical solidity approach would not be capable of distinguishing small entrained air voids from hollow shell glass sphere aggregates, the hollow shell glass sphere aggregates could be subtracted from the measured air void size distribution since the individual size distribution and addition rates are both known entities. Using the same power law approach, air void size distributions from scans at even higher and lower resolutions could be combined to obtain the full size distribution of air voids covering the range from µm to cm.
5 Measurement of Entrained Air-Void Parameters in Portland Cement Concrete Using Micro X-ray Computed Tomography

Chapter 5 is derived from an article published in the *International Journal of Pavement Engineering*, copyright Taylor & Francis, available online: http://dx.doi.org/10.1080/10298436.2016.1172705

5.1 Introduction

In freeze-thaw environments, air-entrained concrete is often used in structures where durability is a concern. The air-void system in concrete is critical in terms of freeze-thaw durability (Powers 1949, Power and Helmuth 1953, Litvan 1972, Fagerlund 1977, Setzer 2001, Penttala and Al-Neshawy 2002, Sun and Scherer 2010). In general, uniformly dispersed fine air-voids are preferred over clustered or large air-voids. Well-dispersed fine air-voids are believed to shorten the path moisture residing in capillary pores has to travel to release pressure built-up under freezing conditions. To characterize the capability for protection, Powers’ spacing factor ($\bar{L}$) is frequently used to describe a typical distance travelled through the cement paste before encountering an air-void, and is widely used in many standards (CEB 1989; EN 480-11 2005; ACI 201.2R-08 2008; CSA A23.1/A23.2; 2014 ASTM C457/C457M 2012; LS-432 2013). As for the procedure of its measurement, a linear traverse is performed on a polished cross-section using a microscope and mechanical stage, which involves careful sample preparation and skilled operators. The time-consuming nature of this procedure and its dependence on the experience of operators provides incentives for modification, improvement or alternatives (Pleau et al. 2001, Zalocha and Kasperkiewicz 2005, Distlehorst and Kurgan 2007, Jana 2007, Radlinski et al. 2010, Lissenden et al. 2010). With its unique capacity to measure the three-dimensional (3D) structure in a non-destructive manner, micro X-ray computed tomography (µCT) has been widely adopted for microstructural characterization of materials (Stock 2008). Its use in cement-based materials research has increased in recent decades, mostly on characterizing the microstructure (primarily air-voids) of cement paste (Rattanasak and Kendall 2005, Gallucci et.al. 2007, Promentilla et.al. 2008, Promentilla et.al. 2009, Provis et.al. 2012, Kim et al. 2012, Yun et al. 2012) or analysing microstructural changes due to durability related damage (Bentz 1995, Stock et al. 2002, Naik et al. 2006, Burlion 2006, Rougelot 2010, Promentilla et al. 2010). Applied to concrete, literature describing the measurement of air-voids using µCT is more limited (Lu et al. 2006, Ikegami et al. 2010). Some barriers must be overcome before µCT can be used in a practical manner for the
measurement of air-void parameters in concrete. For example, the issues of how to reliably separate air-void clusters, how to distinguish entrained air-voids from voids in aggregate, and how to distinguish aggregate from cement paste all need to be addressed. For sample size, it is well known that a small volume of paste can contain a large number of air-voids, but information defining a sample size that can be treated as representative volume element (RVE) for measuring air-void parameters in concrete is still lacking. This current study is an effort to explore the potential of using µCT to measure entrained air-void parameters with emphasis placed on addressing the aforementioned issues. The results can provide a reference for using µCT to measure air-void parameters and more generally to further explore alternative methods for the characterization of air-void systems in hydraulic cement concrete.

5.2 Method

5.2.1 Specimen preparation and µCT configuration

Two 100-mm dia. concrete cores that had been cut in half lengthwise were acquired. They had been previously tested for air content and \( \bar{L} \) in accordance with a provincial test method for the microscopical determination of air-void parameters (LS-432, 2014) as part of a routine Ministry of Transportation Ontario (MTO) quality assurance (QA) program for new concrete construction. One core had what is considered marginal air entrainment, and the other exhibited adequate air entrainment. No specific information as to the exact locations of the cores was available. In the case of core 044, only the contract number affiliated with the core was provided. From this information, core 044 was traced to the Concession Road 6 Batteaux Creek crossing in Nottawa, Ontario, which was constructed in 2007. For this contract, cores would have been submitted for hardened air-void parameter testing as sampled from the approach slabs, bridge deck, parapets, abutments, wing walls, piers, arches, spandrels, and curbs, in accordance with the provincial standard specification (OPSS.PROV 1350, 2014). Core 052 originated from the column of an unspecified pier constructed in 2008 on the Queen Elizabeth Way somewhere between 7th St. and the Garden City Skyway in St. Catharines, Ontario. Neither of the sites has been visited to assess the current condition of the concrete. Both cores would have been extracted shortly after construction, and neither exhibited any signs of damage.
In addition to the air-void parameters reported by the MTO QA program, the same parameters were also measured using an automated flatbed scanner approach (Peterson et al. 2016). The results are summarized in Table 5.1. Out of consideration of scan resolution relevant to the size of entrained air-voids, and the capacity of the μCT machine involved, a series of 6 mm dia. mini-cores were sub-sampled from the half-cores as shown in Figure 5.1. The decision to use a 6 mm dia. sample size was the result of a trade-off between the resolution achievable by the μCT device employed, and obtaining a sample of sufficient volume to contain a significant number of air-voids for analysis. Breaks in the mini-cores were common, and sub-segments composed primarily of the coarse aggregate phase were excluded from analysis. Six core sub-segments, each with a large mortar fraction were randomly selected from each sample for μCT scanning.

<table>
<thead>
<tr>
<th>ID</th>
<th>Test method</th>
<th>Air content, (%)</th>
<th>Specific surface, (mm⁻¹)</th>
<th>Spacing factor, (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>044</td>
<td>MTO QA</td>
<td>3.70</td>
<td>*</td>
<td>0.235</td>
</tr>
<tr>
<td></td>
<td>Flatbed scanner</td>
<td>3.58</td>
<td>32.40</td>
<td>0.250</td>
</tr>
<tr>
<td>052</td>
<td>MTO QA</td>
<td>6.50</td>
<td>*</td>
<td>0.130</td>
</tr>
<tr>
<td></td>
<td>Flatbed scanner</td>
<td>7.25</td>
<td>36.15</td>
<td>0.082</td>
</tr>
</tbody>
</table>

*Not reported.

In some cases, local variations in entrained air-void content within concrete may occur. For example, entrained air may be locally removed due to excessive vibration or finishing (Whiting and Nagi 1998). In spite of the provincial test method LS-432 instructions that the “top ends of cores shall not be trimmed,” both ends of the as-received core halves examined in this study had
been trimmed, thereby removing the original finished or form-contact surfaces. As such, the exact locations of the sub-segments relative to their placement in the field were not known for this study.

A GE V|tome|x|240D μCT machine was used to collect the images at an accelerating voltage of 80 kV, current of 80 µA, and with a 0.1 mm Cu filter. The resolution of the X-ray detector is fixed at 1018×1018 pixels, and the cores were positioned to achieve a projected 2D image resolution of 7.5 µm/pixel. This resolution was an improvement on the 9 µm/pixel of Chapter 4, and was arrived at after experimentation with various accelerating voltages and currents. For each scan, a total of 1,080 images were recorded at 0.33° rotation increments with a dwell time of 0.4 s through a complete rotation of 360°. A segment of Cu-wire was placed at the base of each segment for calibration purposes.

5.2.2 Image pre-processing

After each scan, a 3D reconstruction was performed on the 2D projected images using the manufacturer software to remove imaging artifacts resulting from variations in sensitivity of individual detector elements (ring artifacts), differential attenuation of the polychromatic X-ray source (beam hardening), and shifting/movement of the sample during data collection (Stock 2009). Each reconstructed 3D image had a resolution of 7.5 µm/voxel. In this application, it is questionable whether a single voxel is sufficient to characterize a small spherical entrained air-void. For the purposes of this study, the practical limit for the detection of a voxelized void was set at a width of 3 pixels, as is discussed in more detail in Section 5.2.5. After reconstruction, the data was exported as a stack of image slices for further image processing in Matlab™. The intensity levels of the image stacks were calibrated based on the histogram peak location of the reference Cu-wire and the peak location of the large empty air-voids in the sample. Specifically, a histogram stretch was performed on each image slice using the modal values for the Cu and air as end points, with intermediate densities distributed linearly using 2^{16} channels. Since the cores were not cylindrical, the images were cropped to a cylindrical region of interest (ROI) for a consistent analysis area (Figure 5.2). After cropping, some air-voids near the perimeter were clipped as seen in Figure 5.2, but considering the large number of air bubbles available within a typical sub-segment (on the order of ten thousand), the number of clipped air-voids included in
the analysis was considered insignificant. No additional image processing steps (e.g. noise reduction filters) were applied to the image slices.

![Figure 5.2](image_url) (a) An example image slice as collected, and (b) after cropping to ROI.

5.2.3 Determination of threshold between void and solid phases

Many factors contribute to the accuracy of the measurement of air-void parameters in concrete materials, but from an image processing perspective, the choice of a global threshold grayscale value to separate the void and solid phases is the most critical factor. This study relied on a region growth based algorithm known as an active contour line to set an appropriate threshold between void space (combined entrained air-voids and voids in aggregate) and solid phases (cement paste and aggregate). The algorithm models the boundary of the object of interest as a controlled continuity line, sometimes referred to as a ‘snake’ (Kass et al. 1988). An equivalent energy functional is defined with the potential line boundary as a variable and with energy contributions from the boundary curvature, and from the grayscale values inside and outside the boundary line. An optimum boundary is found based on variational analysis (Chan and Vese 2001; Mumford and Shah 1989). The motivation of using this method is that there are multiple levels of grayscale values present in the phases of interest, which can be a challenge for other methods. For example, Otsu’s and other similar histogram based methods are based solely on the variation of grayscale values, and are independent of morphology (Otsu 1975). Methods using edge detection (Canny 1986; Deriche 1987) are sensitive to image noise, and this can complicate the continuous definition of a feature boundary.
For this research a preliminary threshold of 0.7 (a value of 45,875 in the $2^{16}$ grayscale image) was applied to each sub-segment to obtain a new 3D binary image that divided the segment between potential void and non-void (solid) phases. For each sub-segment, a series of two-dimensional (2D) image slices were extracted at a fixed interval of every 15\textsuperscript{th} slice. Depending on the length of the segment, this yielded approximately fifty 2D slices. Void intercepts with an eccentricity $<0.9$ and a 2D solidity $>0.9$ were identified as potential air-voids. Eccentricity is the ratio of the distance between the foci of a fitted ellipse to the void intercept divided by the major axis length; a circle has an eccentricity of zero, and a line segment has an eccentricity of unity (Clapham and Nicholson 2009). Solidity in 2D is defined as the proportion of area of the intercept over the area of its convex hull (Clapham and Nicholson 2009). Typically, each individual 2D slice contained anywhere from fifty to three hundred potential air-voids. For each sub-segment, four hundred intercepts through potential air-voids were randomly selected from its population of 2D slices, with the centroid coordinates of each intercept serving as an input for the active contour line algorithm to more accurately locate and record the X-ray intensity histogram threshold value that best defined the boundary for each individual air-void. The optimum threshold value for each air-void ($Th_{opt}$) was in turn treated as a sample of the full population of $Th_{opt}$ values for all of the air-voids in the sub-segment. A statistical fit of the $Th_{opt}$ population was applied to obtain an estimate for a global threshold ($\overline{Th}_{opt}$) to apply to the entire sub-segment 3D data set. Specifically, $\overline{Th}_{opt}$ was selected as the maximum value of the $Th_{opt}$ distribution as fitted using a kernel density estimation (Upton and Cook 2014). Examples of automatically located air-void intercepts with their surrounding solid background, as well as their shape after the final active contour line segmentation are provided in Figure 5.3. The distribution of measured $Th_{opt}$ values is provided in Figure 5.4.
5.2.4 Discerning entrained air-voids from voids in aggregate

After application of $\overline{T_h_{opt}}$ to the full 3D data set, a final distinction between entrained air-voids in the paste and voids in the aggregate is still necessary to properly characterize the air-void system. Examples of µCT slices including entrained air-voids and voids in aggregate are provided in Figure 5.5. Detailed 3D renderings of a void in an aggregate particle and a cluster of entrained air-voids are shown Figure 5.6.
Figure 5.5 (a) Air-voids in aggregate and entrained air-voids as observed in a 2D slice from sample 044, and (b) sample 052.

Figure 5.6 (a) 3D renderings of a void in aggregate, and (b) entrained air-voids in close proximity to each other.
Due to overlap between the grayscale values of aggregate and paste, and the lack of uniform morphological boundaries between the aggregate and paste, attempts to discern the two phases through image processing proved unsuccessful. This was unfortunate, as this made the simple distinction of air-voids and voids in aggregate based on their respective locations impossible. However, due to their unique mechanism of formation, entrained air-voids tend to be more spherical than voids in aggregate. Possible exceptions to this rule include air-cooled blast furnace slag or vesicular basalt aggregates, both of which contain spherical voids. However, in most cases, entrained air-voids are more spherical, and this tendency was exploited here to filter out voids in aggregate. To accomplish this task, a minimum bounding sphere (Larsson 2008) was calculated for each object, where the term ‘object’ is used here to define a 3D set of connected void voxels. The volume ratio between the part of the original object that lies within its minimum bounding sphere and the volume of the minimum bounding sphere is defined as an object’s 3D spherical solidity. The higher the 3D spherical solidity value, the more likely that the object is an entrained air-void. Due to the large number of voxels in any given object, it is not practical to carry out a calculation for the bounding sphere using the entire object surface. Therefore, a reduced number of surface points was used as a reference to calculate the bounding sphere. To increase the reliability of the method, this process was repeated twenty times for each object, and the average 3D spherical solidity value determined. An illustration of the process for measuring the 3D spherical solidity of an air-void and the 3D spherical solidity of a void in aggregate is shown in Figure 5.7.

In general, air-voids tend to have a 3D spherical solidity closer to unity, and voids in aggregate tend to have a 3D spherical solidity closer to zero. Planar features, such as cracks, will also have a 3D spherical solidity closer to zero. The more complicated issue of voids in close proximity to each other, or clustered air-voids is discussed in Section 5.2.6.
Figure 5.7 Process for distinguishing entrained air-void (left hand side) from void in aggregate (right hand side) showing (a) original geometrical shape, (b) nine repetitions of the bounding sphere calculation based on ten randomly selected surface points, and (c) corresponding 3D spherical solidity values.
5.2.5 Test of 3D spherical solidity criteria for void type distinction

To better understand the limitations of a 3D spherical solidity-based criteria for void type distinction, an analysis was performed on simulated 3D voids within a range of 30 to 300 μm in diameter. The simulated voids were generated via a ‘random growth’ process, that is, first a seed voxel was generated, and the next voxel randomly grown from one of the surfaces of the seed at an equal possibility; and the process continued until a certain desired ‘spherical solidity’ was achieved. Examples of simulated 3D objects with different sizes and spherical solidities are shown in Figure 5.8. The average 3D spherical solidity results as calculated from twenty runs on simulated objects of different size and 3D spherical solidity are shown in Figure 5.9. Bounding spheres based on both five and ten surface reference points were also tested.

![Figure 5.8: Examples of simulated objects of varying size and 3D spherical solidity.](image-url)
Figure 5.9: Calculated 3D spherical solidity for the simulated objects with true 3D spherical solidity of 0.2 (a), 0.4 (b), 0.6 (c), 0.8 (d), and 1.0 (e).

In the simulation, 3D voids with a minimum diameter <3 pixels exhibited an instability in the calculated 3D spherical solidity values. Between a range of 3 to 6 pixels in diameter the measured 3D spherical solidity values begin to more closely approximate the true 3D spherical solidity values. However, the calculated 3D spherical solidity values were consistently overestimated in cases where the true 3D spherical solidity values were <0.6. In cases where the
true 3D spherical solidity was $\geq 0.6$, the calculated 3D spherical solidity values better approximated the computer-generated values.

Taking these results into account, it was decided to filter out (remove) any 3D voids with a minimum $x$, $y$, $z$ diameter $< 3$ pixels, since it is practically impossible to accurately assess the shape of such small objects. This minimum diameter cut-off ($T_{dmin}$) of 3 pixels effectively removed all voids $< 22.5 \, \mu m$ in diameter from the analysis. Furthermore, it was decided to treat 3D voids with a minimum diameter between 3 and 6 pixels differently than larger voids in terms of the 3D spherical solidity criteria used to distinguish air-voids from voids in aggregate. Voids with a minimum diameter between 3 and 6 pixels were assigned a 3D spherical solidity criteria ($T_{s1}$) that was slightly lower than the 3D spherical solidity criteria for larger voids ($T_{s2}$). This distinction was made to account for the consistent underestimation of true 3D spherical solidity for voids in the 3 to 6 pixel diameter range. For the purpose of computing efficiency, five surface points were selected on each object to calculate the bounding sphere. From qualitative observations of Figure 5.8, a value of 0.70 was initially selected for $T_{s1}$, and a value of 0.75 was initially selected for $T_{s2}$. As described earlier in Section 4.3.2, an uncertainty analysis was carried out to test the validity of the selected 3D spherical solidity criteria.

5.2.6 Voids in close proximity to each other

Although the difference in 3D spherical solidity values between entrained air-voids and voids in the aggregate were in most cases sufficient to distinguish the two, situations of entrained air-voids in close proximity to each other (clustered), or entrained air-voids in close proximity to a void in the aggregate significantly complicated the process. When voids are in close proximity to each other, the resolution of the $\mu$CT scan often causes them to appear connected. To solve this problem, all 3D voids that exceeded a minimum diameter criterion ($T_{dW}$) of 6 pixels were processed using a watershed algorithm (Russ and Neal, 2016), and the bounding sphere calculation was performed on each of the divided parts. The specific approach used was to first to sort the watershed segments according to their volume from highest to lowest. The entire object was considered as a potential cluster of air-voids if the largest watershed segment (Figure 5.10) had a 3D spherical solidity $> 0.8$, or if the average 3D spherical solidity of the three largest segments was $> 0.7$. If either criterion was satisfied, the entire object was treated as an air-void cluster, and kept in the analysis. If neither criterion was met, the entire object was categorized as
a cluster of voids in aggregate and excluded from the analysis. Two examples of the process, one for a coalesced grouping of entrained air-voids and another for a large void in aggregate, are provided in Figure 5.10.

Figure 5.10 (a) Coalesced entrained air-void object after division by watershed showing solidity values of the separated segments, and (b) a void in aggregate after division by watershed showing 3D spherical solidity values of the separated segments.

If the entire object was considered as a potential cluster of air-voids, each watershed segment was then treated as an individual void, and independently assessed as being either an air-void or as a void in aggregate according to the 3D spherical solidity criteria set forth in Section 5.2.5. As described later in Section 5.3.3, an uncertainty analysis was carried out to test the validity of the $T_{thw}$ criterion of 6 pixels.

5.2.7 Determination of paste content

The similar densities of the aggregates and cement paste in the concrete used in this study resulted in limited contrast in the μCT images. This prevented the use of an easy automated approach to distinguish paste from aggregate. As a result, manual point counts were performed on 2D image slices to determine the paste content in vol. %. To illustrate the general uncertainty of using this approach, an error matrix based on 12,000 points (2,000 from each sub-segment) from two different operators is listed in Table 5.2 and Table 5.3 for cores 044A and 052A respectively.
Table 5.2: Agreement of point counting from two operators (sample 044).

<table>
<thead>
<tr>
<th>Operator</th>
<th>Air</th>
<th>Paste</th>
<th>Aggregate</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Operator 1</td>
<td>188</td>
<td>68</td>
<td>16</td>
<td>272</td>
</tr>
<tr>
<td>Operator 2</td>
<td>27</td>
<td>2848</td>
<td>783</td>
<td>3658</td>
</tr>
<tr>
<td>Aggregate</td>
<td>7</td>
<td>468</td>
<td>7595</td>
<td>9070</td>
</tr>
<tr>
<td>Total</td>
<td>222</td>
<td>3384</td>
<td>8394</td>
<td>12000</td>
</tr>
</tbody>
</table>

Agreement Overall 89% \( \bar{K} \) 74%

Table 5.3: Agreement of point counting from two operators (sample 052)

<table>
<thead>
<tr>
<th>Operator</th>
<th>Air</th>
<th>Paste</th>
<th>Aggregate</th>
<th>Total</th>
</tr>
</thead>
<tbody>
<tr>
<td>Operator 1</td>
<td>347</td>
<td>140</td>
<td>43</td>
<td>530</td>
</tr>
<tr>
<td>Operator 2</td>
<td>71</td>
<td>1702</td>
<td>770</td>
<td>2543</td>
</tr>
<tr>
<td>Aggregate</td>
<td>23</td>
<td>323</td>
<td>8581</td>
<td>8927</td>
</tr>
<tr>
<td>Total</td>
<td>441</td>
<td>2165</td>
<td>9394</td>
<td>12000</td>
</tr>
</tbody>
</table>

Agreement Overall 89% \( \bar{K} \) 70%

Due to the relative insensitivity of paste content compared with other factors on the result of spacing factor (Pade, 2002), it is uneconomic to count too many points or have more than one person to carry out such an operation. However, considering the uncertainty reported in Tables 5.2 and 5.3, paste content based on the average from two operators with each counting the same 2000 points was used. The point counts were performed using 20 randomly generated points on 100 randomly selected and evenly spaced slices. The uncertainty in \( L \) caused by paste content values based on different numbers of points as well as different operators is discussed further in Section 5.3.3.

5.3 Results and discussion

5.3.1 Air-void parameters for mini core sub-segments

In addition to \( L \), a variety of more sophisticated air-void parameters have been proposed to describe air-void systems in concrete that more accurately describe the true distances between points in the paste and the edges of the nearest air voids (Snyder et al. 2001; Mayercsik et al 2014). The 3D data sets collected by \( \mu \)CT have the potential to be exploited in this manner, but \( L \) was chosen here as a basis to compare the effectiveness of different test methods. In spite of its deficiencies, \( L \) is a well-established parameter for the prediction of freeze-thaw performance of concrete materials, and data accumulated to date has made an uncertainty statement possible.
For the calculation of $\bar{L}$, air content and specific surface were derived from the processing of the 3D $\mu$CT images, while paste content was based on manual point counts of 2D slices from the $\mu$CT images. Specifically, the air content was determined by dividing the sum of the voxels categorized as air-void (excluding voids with diameters less than three pixels) by the total number of voxels contained in the sample volume. The specific surface was determined by dividing the sum of the air-void surface areas by the sum of the volume of the air-voids, after first converting pixels$^2$ to mm$^2$, and voxels to mm$^3$. All of the air-voids contained in the 3D ROI were included in the air content and surface area measurements. This also includes the air-voids clipped by the ROI boundary. Although this does introduce some error to the surface area measurement, it is negligible since the number of air-voids completely within the ROI (7,000 – 10,000 for sub-segments from sample 044, and 20,000 -25,000 for sub-segments from sample 055) greatly outnumber the clipped voids. Assuming the voids are evenly distributed throughout the ROI volume, only about 5% are clipped.

The suite of parameters used to identify entrained air-voids in the $\mu$CT images are provided in Table 5.4. The measured air-void parameters for each sub-sample are listed in Table 5.5. Paste content was the average of two operators counting 2,000 points.

The size of the coarse aggregate particles used in concrete pavements generally falls within a range that overlaps the size of the mini-core sub-samples used in this study. Therefore, any given sub-sample may or may not contain a representative portion of aggregate. As a result, variations in air-void content from sample to sample is well expected; segments with a relatively higher paste content (and correspondingly lower aggregate content) will tend to exhibit higher air contents than segments with a relatively lower paste content (and correspondingly higher aggregate content). If air content is normalized against aggregate content, the variation drops considerably; for sample 044 the COV drops from 40.95 % to 14.19 %, and for sample 055 the COV drops from 44.72 % to 12.28 %.
Table 5.4: Parameters for final air-void measurement.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sub-sample</th>
<th>$\overline{Th}_{opt}$ (pixel)</th>
<th>$Th_{dmin}$ (pixel)</th>
<th>$Th_{d1}$</th>
<th>$Th_{d2}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>044</td>
<td>1</td>
<td>0.7338</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>0.7573</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>0.7272</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>0.7487</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>0.7041</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>0.7273</td>
<td>&lt;3</td>
<td>≥6</td>
<td>0.70</td>
</tr>
<tr>
<td>052</td>
<td>1</td>
<td>0.7092</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>0.7449</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>0.7688</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>4</td>
<td>0.6990</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>0.7918</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>0.7053</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

$\overline{Th}_{opt}$ = Global threshold for detection of voids;
$Th_{dmin}$ = Cut-off diameter for removal of smallest voids;
$Th_{d1}$ = Cut-off diameter demanding watershed;
$Th_{d2}$ = First level cut-off 3D spherical solidity value;
$Th_{d2}$ = Second level cut-off 3D spherical solidity value;
Table 5.5: Results of air-void parameters for each sub-segment.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sub-sample (mm³)</th>
<th>V (%)</th>
<th>A₁ (%)</th>
<th>A₂ (%)</th>
<th>A (%)</th>
<th>p (%)</th>
<th>Vp (mm³)</th>
<th>α (1/mm)</th>
<th>L (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>044</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>113</td>
<td>4.14</td>
<td>4.01</td>
<td>1.84</td>
<td>32.77</td>
<td>37.04</td>
<td>42.15</td>
<td>0.1940</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>118</td>
<td>4.26</td>
<td>3.72</td>
<td>2.02</td>
<td>35.00</td>
<td>41.30</td>
<td>37.12</td>
<td>0.2176</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>127</td>
<td>2.51</td>
<td>2.44</td>
<td>2.16</td>
<td>42.60</td>
<td>54.10</td>
<td>34.81</td>
<td>0.2452</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>127</td>
<td>4.51</td>
<td>2.93</td>
<td>1.23</td>
<td>28.07</td>
<td>35.66</td>
<td>39.88</td>
<td>0.2281</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>102</td>
<td>6.09</td>
<td>3.21</td>
<td>1.21</td>
<td>28.63</td>
<td>29.20</td>
<td>42.49</td>
<td>0.2173</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>126</td>
<td>5.03</td>
<td>4.88</td>
<td>0.55</td>
<td>8.97</td>
<td>11.31</td>
<td>45.13</td>
<td>0.1743</td>
<td></td>
</tr>
<tr>
<td>average</td>
<td></td>
<td>1.50</td>
<td>29.34</td>
<td>34.77</td>
<td>40.26</td>
<td>0.2128</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>COV(%)</td>
<td>n/a</td>
<td>n/a</td>
<td>n/a</td>
<td>40.95</td>
<td>38.46</td>
<td>40.75</td>
<td>9.47</td>
<td>11.82</td>
<td></td>
</tr>
<tr>
<td>052</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>1</td>
<td>108</td>
<td>5.67</td>
<td>5.55</td>
<td>4.75</td>
<td>29.18</td>
<td>31.51</td>
<td>40.88</td>
<td>0.1245</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>125</td>
<td>7.45</td>
<td>6.49</td>
<td>5.34</td>
<td>38.25</td>
<td>47.81</td>
<td>45.05</td>
<td>0.1212</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>112</td>
<td>5.83</td>
<td>4.50</td>
<td>2.74</td>
<td>20.50</td>
<td>22.96</td>
<td>46.96</td>
<td>0.1185</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>121</td>
<td>6.68</td>
<td>2.96</td>
<td>1.48</td>
<td>7.83</td>
<td>9.47</td>
<td>47.19</td>
<td>0.1007</td>
<td></td>
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<tr>
<td>5</td>
<td>116</td>
<td>3.91</td>
<td>3.29</td>
<td>2.15</td>
<td>12.98</td>
<td>15.05</td>
<td>44.55</td>
<td>0.1133</td>
<td></td>
</tr>
<tr>
<td>6</td>
<td>121</td>
<td>5.10</td>
<td>4.94</td>
<td>3.98</td>
<td>29.50</td>
<td>35.70</td>
<td>47.69</td>
<td>0.1162</td>
<td></td>
</tr>
<tr>
<td>average</td>
<td></td>
<td>3.41</td>
<td>23.04</td>
<td>27.08</td>
<td>45.39</td>
<td>0.1157</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>COV(%)</td>
<td>n/a</td>
<td>n/a</td>
<td>n/a</td>
<td>44.72</td>
<td>49.48</td>
<td>52.10</td>
<td>5.59</td>
<td>7.19</td>
<td></td>
</tr>
</tbody>
</table>

\( V = \) Volume of the cropped ROI of sub-segment;  
\( A_1 = \) Total void content after void vs. non-void (solid) thresholding;  
\( A_2 = \) Total void content after the removal of voids with minimum x-y-z dimension smaller than three pixels;  
\( A = \) Air-void content after removal of voids in aggregate;  
\( \alpha = \) Specific surface;  
\( p = \) Paste content;  
\( \bar{L} = \) Spacing factor;  
\( V_p = \) Volume of paste phase;  

For both concrete samples the results in Table 5.5 show a reasonably satisfactory dispersion of values in terms of specific surface and \( \bar{L} \). One of the possible explanations for this is that \( \mu \)CT provides a complete interrogation of the large number of air-voids that exist within a small volume of cement paste, while test methods based on 2D inference only sample a small portion of the information that is contained within the physical range of the sample. Another factor that could contribute to these results is that air bubbles lying at the smaller end of the size spectrum tend to dictate specific surface and \( \bar{L} \) due to their sheer number. Thus, although there are good possibilities that some larger entrapped air-voids could occupy a considerable portion of the cm scale cross-section, still, their almost negligible frequency define them as rare events in a
statistical sense. That is to say these larger air-voids usually have quite a limited influence on specific surface and $\bar{L}$.

Since the smallest entrained air-voids have such a strong influence on specific surface and $\bar{L}$, it draws attention to the appropriateness of the $Th_{dmin}$ cut-off of 3 pixels (air-voids with diameters <22.5 μm) employed in this study. The limitations of the μCT for the detection of the smallest of air-voids are also shared by the commonly used stereological methods based on reflected light from a polished concrete surface. This is a somewhat contentious issue, as ASTM C457 recommends that circular air-void intercepts of 10 μm be clearly distinguishable. However, ASTM C457 also recommends a sample preparation regime that employs final polish with a >10 μm diameter abrasive. It is doubtful that this level of preparation is sufficient for the detection of air-voids in the size range of 10-20 μm in diameter. ASTM C457 also recommends a minimum magnification of 50×, which is arguably insufficient for the detection of such small air-voids. Whether or not air-voids in the size range of 10-20 μm in diameter are measured during an ASTM C457 traverse is debatable. While beyond the scope of this paper, a broad survey of collected ASTM C457 chord intercept data would be informative, and help to settle this issue. Certainly it is possible to carefully prepare and examine a polished specimen at a high enough magnification to observe 10-20 μm diameter air-voids, as well as hollow-shell (Hadley) cement grains of similar size, but this is not realistic within the practical application of ASTM C457, nor is it practical for the μCT method described here.

Although data points are limited, a clear trend can be found that sub-segments with smaller paste contents tend to have larger variations, for example this is the case for sub-segment 6 from core 044 and sub-segment 4 from core 052. Also as expected, the sample with the higher air content (052) shows less fluctuation in the air-void parameters. Measured by coefficient of variation (COV), for specific surface, the values are around 10% for 044A and 5% for 052A. For $\bar{L}$, the COV values are slightly larger with a roughly 2% increase in its absolute sense. This added uncertainly is mostly contributed by the errors that arise in the paste content estimation. It is important to note that these uncertainty estimations are based on samples with varying size. If the measurements were carried out on the same-sized samples, the precision would be expected to increase accordingly.
5.3.2 Estimation of uncertainty related to dimensional size of the sample

Local fluctuations in uniformity of materials exist in concrete. Therefore, the variation of calculated air-void parameters of sub-segments is also well expected. With the precision statement from other more common air-void parameter measuring methods as a reference (Saucier et al. 1996), a reasonable degree of stability of results among different sub-segments for both cores 044 and 052 can be found in Table 5. In order to estimate the required sample size for a reliable measurement of $\bar{L}$ using the proposed µCT procedure, it would still be instructive to have a quantitative estimation of the uncertainty associated with a certain effective sample size (volume of paste plus volume of entrained air-voids). This section centers on a discussion related to $\bar{L}$. Although a well-defined probability distribution function (pdf) of $\bar{L}$ within a unit volume of concrete is absent, considering the large number of air-voids within a small volume of concrete, each of the six $\bar{L}$ measurements corresponding to the concrete sample can be first assumed to follow a Gaussian distribution. In order to estimate the uncertainty of $\bar{L}$ associated with concrete samples with any volume, a statistical model was formulated as follows. If the distribution of $\bar{L}$ from paste of unit volume (1 mm$^3$) is expressed as $\bar{L}_0 \sim N(\mu, \sigma)$, the distribution of measurements from paste of $v_i$ volume can then be expressed as Equation 5.1:

$$\bar{L}_i \sim N(\mu, \sigma_i)$$

Equation 5.1

where $\sigma_i = \frac{\sigma}{\sqrt{v_i}}$ for $i=1, 2, 3, 4, 5, 6$. The jointed pdf of the six measurements can be expressed as in Equation 5.2:

$$f(\bar{L}_1, \bar{L}_2, \bar{L}_3, \bar{L}_4, \bar{L}_5, \bar{L}_6 \mid \mu, \sigma) = \prod_{i=1}^{6} \frac{1}{\sigma_i \sqrt{2\pi}} e^{-\frac{(\bar{L}_i - \mu)^2}{2\sigma_i^2}}$$

Equation 5.2

With limited prior information of the parameters $(\mu, \sigma)$ available, the parameters were inferred following the maximum likelihood method. Thus, the likelihood function can be further derived from Equation 5.2 as listed in Equation 5.3:

$$L(\mu, \sigma \mid \bar{L}_1, \bar{L}_2, \bar{L}_3, \bar{L}_4, \bar{L}_5, \bar{L}_6) = \frac{(\prod_{i=1}^{6} v_i)^{1/2}(2\pi)^{-3}}{\sigma^6} \exp[-\frac{\sum_{i=1}^{6} v_i (\bar{L}_i - \mu)^2}{2\sigma^2}]$$

Equation 5.3
Based on the maximum likelihood estimation, the estimated mean $\hat{\mu}$ and estimated standard deviation $\hat{\sigma}$ can be derived as in Equations 5.4 and 5.5:

$$\hat{\mu} = \left(\frac{\sum v_i L_i}{\sum v_i}\right)$$

Equation 5.4

$$\hat{\sigma} = \left(\frac{\sum v_i (L_i - \hat{\mu})^2}{6}\right)^{1/2}$$

Equation 5.5

From Equations 5.4 and 5.5, the expectation of $\bar{L}$ based on the six measurements and the standard variation of $\bar{L}$ when a specific volume of paste is selected for measurement can be estimated. For practical purposes to illustrate the number of cores needed for a measurement with a specific precision requirement, these paste volumes are then converted to the volume of concrete accordingly with the paste content assumed to be 30%. These results are listed in Table 5.6.

Table 5.6: Uncertainty of measurement of $\bar{L}$ associated with sample size.

<table>
<thead>
<tr>
<th>Sample</th>
<th>V(mm$^3$)</th>
<th>100</th>
<th>100</th>
<th>100</th>
<th>100</th>
<th>100</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>×1</td>
<td>×2</td>
<td>×3</td>
<td>×4</td>
<td>×5</td>
</tr>
<tr>
<td>044</td>
<td>$\hat{\mu}$ (mm)</td>
<td>0.2200</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>$\hat{\sigma}$ (mm)</td>
<td>0.0218</td>
<td>0.0154</td>
<td>0.0126</td>
<td>0.0109</td>
<td>0.0098</td>
</tr>
<tr>
<td>052</td>
<td>$\hat{\mu}$ (mm)</td>
<td>0.1184</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>$\hat{\sigma}$ (mm)</td>
<td>0.0053</td>
<td>0.0037</td>
<td>0.0031</td>
<td>0.0026</td>
<td>0.0024</td>
</tr>
</tbody>
</table>

* Each mini-core sub-segment has a volume ~100 mm$^3$

5.3.3 Estimation of uncertainty related to parameters in image processing

To evaluate the robustness of the proposed image processing procedure, sensitivity tests of the primary parameters involved in the image processing were also conducted. The values for $\bar{L}$ were used for this purpose of illustration. The related results due to the first and second level cut-off 3D spherical solidity ($Th_{s1}$ $Th_{s2}$) values are listed in Table 5.7 and that for the cut-off dimension demanding watershed ($Th_{dw}$) is listed in Table 5.8. For both Tables 5.7 and 5.8, the sensitivity test was run on one sub-sample from both mixes. To assess the uncertainty caused by paste content variation due to different operators as well as due to repetitions of one operator, the
results from two independent operators were observed with each counting 2000 points per sample. Five hundred and 1000 points were then randomly chosen from 2000 points to calculate the paste content. This process was repeated 100 times under both numbers of points and the average as well the COV based on them was then calculated. These results are listed in Table 5.9. For the results in Table 9, the sensitivity test was run on each sub-segment of both mixes with the awareness that the level of uncertainty is highly dependent on the volume of paste in a specific sub-segment.

Table 5.7: Sensitivity of first and second level cut-off solidity values ($Th_{s1}, Th_{s2}$) on $\bar{L}$ (mm).

<table>
<thead>
<tr>
<th>Sample</th>
<th>$Th_{s2}$</th>
<th>COV (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.60</td>
<td>0.65</td>
</tr>
<tr>
<td>044</td>
<td>0.60</td>
<td>0.2049</td>
</tr>
<tr>
<td></td>
<td>0.65</td>
<td>0.2095</td>
</tr>
<tr>
<td></td>
<td>0.70</td>
<td>0.2026</td>
</tr>
<tr>
<td></td>
<td>0.75</td>
<td>0.2113</td>
</tr>
<tr>
<td></td>
<td>0.80</td>
<td>0.2035</td>
</tr>
<tr>
<td>052</td>
<td>0.60</td>
<td>0.1178</td>
</tr>
<tr>
<td></td>
<td>0.65</td>
<td>0.1183</td>
</tr>
<tr>
<td></td>
<td>0.70</td>
<td>0.1188</td>
</tr>
<tr>
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<td>0.1196</td>
</tr>
<tr>
<td></td>
<td>0.80</td>
<td>0.1202</td>
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</table>

Table 5.8: Sensitivity of cut-off dimension ($Th_{d_w}$) demanding watershed on $\bar{L}$ (mm).

<table>
<thead>
<tr>
<th>Sample</th>
<th>$Th_{d_w}$</th>
<th>COV(%)</th>
</tr>
</thead>
<tbody>
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<td></td>
<td>5</td>
<td>6</td>
</tr>
<tr>
<td>044</td>
<td>0.2223</td>
<td>0.2161</td>
</tr>
<tr>
<td>052</td>
<td>0.1211</td>
<td>0.1216</td>
</tr>
</tbody>
</table>
Table 5.9: Sensitivity of paste content due to operator and # of points on $L$ (mm).

<table>
<thead>
<tr>
<th>Sample</th>
<th>Sub-sample</th>
<th>500 points</th>
<th>1000 points</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>$\bar{L}$</td>
<td>COV(%)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>OP$_1$</td>
<td>OP$_2$</td>
</tr>
<tr>
<td>044</td>
<td>1</td>
<td>0.1894</td>
<td>0.1997</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>0.2118</td>
<td>0.2236</td>
</tr>
<tr>
<td></td>
<td>3</td>
<td>0.2385</td>
<td>0.2520</td>
</tr>
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<td></td>
<td>4</td>
<td>0.2302</td>
<td>0.2263</td>
</tr>
<tr>
<td></td>
<td>5</td>
<td>0.2161</td>
<td>0.2167</td>
</tr>
<tr>
<td></td>
<td>6</td>
<td>0.1711</td>
<td>0.1770</td>
</tr>
<tr>
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<td>1</td>
<td>0.1181</td>
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<td>0.1215</td>
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<td>0.0940</td>
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</tr>
<tr>
<td></td>
<td>6</td>
<td>0.1124</td>
<td>0.1194</td>
</tr>
</tbody>
</table>

OP$_1$=Operator 1, OP$_2$=Operator 2

Unfortunately, there is no universal optimum parameter for image processing in practice, as any such parameter will necessarily be dependent on the specific concrete material under investigation. Still, due to the relative stability of the morphology of entrained air-voids and voids in aggregate among different materials, the results of the sensitivity tests conducted in this study should also be applicable to other similar designs, or at least, with the uncertainty bounds in place as listed in Tables 5.7-5.9, the parameters can be safely fine-tuned to suit other situations. In terms of paste content, the only quantity in this study that came from the traditional point counting method, it should be stressed that the concrete used in this study represents a ‘worst case scenario’ group of materials, whose limited contrast among different phases prevented an easy automated method. Although not attempted in the current study, efforts to find a more elaborate algorithm to automate the estimation of paste content should be pursued. For materials with a reasonable level of contrast, many classification algorithms are easily accessible such as a generalized linear model, a decision tree, or neural networks. At least if manual point counting is necessary, the number of points required can be assessed, and the time required kept to a minimum. In general, based on the stability of the processing parameters in this study, the
method as well as the parameters proposed in this study can be used as a practical approach to the measurement of $\bar{L}$ or other air-void parameters in general.

5.4 Conclusions

This chapter explored the potential of $\mu$CT to characterize air-void parameters in portland cement concrete. Aside from efforts made to look for solutions to issues in processing the X-ray images, such as void/solid thresholding, air type discernment and separation of connected air-voids, the uncertainty that could arise both in the selection of different image processing parameters, and from different sample sizes were also a focus of this study. The results of the study suggest that the proposed procedures can be followed to measure $\bar{L}$ and specific surface with a relatively small number of cm scale samples. As summarized in Table 5.10, the results for $\bar{L}$ listed for the two cores from the MTO QA program are in agreement with the average results from the $\mu$CT measurements from the mini-core sub-segments. However, based on the resolution limits of the $\mu$CT employed here, the minimum size of air bubbles that can be effectively measured falls in the range of 20 to 40 µm, which may prohibit its direct comparison with the results from other methods that might include smaller-sized air-voids.

Table 5.10: Summary of air void parameters as measured by different methods.

<table>
<thead>
<tr>
<th>ID</th>
<th>Test method</th>
<th>Air content, (%)</th>
<th>Specific surface, (mm$^{-1}$)</th>
<th>Spacing factor, (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>44</td>
<td>MTO QA</td>
<td>3.7</td>
<td>*</td>
<td>0.235</td>
</tr>
<tr>
<td></td>
<td>Flatbed scanner</td>
<td>3.58</td>
<td>32.40</td>
<td>0.250</td>
</tr>
<tr>
<td></td>
<td>$\mu$CT</td>
<td>1.5</td>
<td>40.26</td>
<td>0.213</td>
</tr>
<tr>
<td>52</td>
<td>MTO QA</td>
<td>6.5</td>
<td>*</td>
<td>0.130</td>
</tr>
<tr>
<td></td>
<td>Flatbed scanner</td>
<td>7.25</td>
<td>36.15</td>
<td>0.082</td>
</tr>
<tr>
<td></td>
<td>$\mu$CT</td>
<td>3.4</td>
<td>45.39</td>
<td>0.116</td>
</tr>
</tbody>
</table>

*Not reported.

Although potentially useful for determinations of $\bar{L}$ and specific surface, the $\mu$CT method explored here is not appropriate for the determination of bulk air content due to the cm scale sample size employed. The sample size necessarily precludes the inclusion of large entrapped air-voids, which exert a strong influence of bulk air content measurements. Furthermore, the observed variability in sub-segment bulk air content measurements can be attributed to differences in aggregate content from sub-segment to sub-segment, and drops dramatically when
air content was normalized against the aggregate content. If air content is normalized against aggregate content, for sample 044 the COV drops from 40.95 % to 14.19 %, and for sample 055 the COV drops from 44.72 % to 12.28 %.

To improve correlations with traditional bulk air concrete measurements, future research efforts could be focused on a hybrid approach where X-ray scans are also performed on larger samples to capture larger entrapped air-voids that are missed by the current cm scale sampling approach. In addition, with the true 3D spatial information of air-voids available, further efforts could be made to better characterize the air-void system. If detailed spatial information for fine aggregate could be obtained by μCT, or an informed simulation of fine aggregate distribution employed, more sophisticated 3D air-void parameters could be calculated and compared with current methods that neglect the influence of aggregate on air-void spacing.
6 X-ray CT measurement of air-void system in cement mortar

6.1 Introduction

In this chapter, the entrained air void systems generated by two different classes of air entraining admixtures (AEAs) are examined: a saponified rosin (SR) similar to traditional vinsol resin (VR), and a tall-oil (TO) based synthetic consisting of a blend of organic fatty acid salts. As summarized by Jeknavorian (2006), TO based synthetic AEAs are expected to generate air void systems that contain higher numbers of smaller diameter air voids as compared to the traditional VR AEAs. For both AEAs, low and high dosage mortars were produced, and the air void systems compared by flatbed scanner and by micro x-ray CT (µCT) methods.

6.2 Materials and method

6.2.1 Mortar mixtures

A series of mortar mixtures were produced with the constituents listed in Table 6.1, and cast into 50 mm cube specimens. All of the mixtures were based on a mass ratio of 0.45:1:2.07 water:cement:sand to achieve a 50/50 ratio of paste to aggregate, with varying AEA dosages as listed in Table 6.2. The mix designs in terms of kg/m$^3$ are listed in Table 6.2 as based on the average of three gravimetric air content tests. Oven dried sand was used, and water absorption by the sand neglected in the mix design process.

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>density (g/cc)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ASTM C109 Ottawa sand</td>
<td>2.67</td>
</tr>
<tr>
<td>portland cement</td>
<td>3.12</td>
</tr>
<tr>
<td>TO AEA</td>
<td>1.04</td>
</tr>
<tr>
<td>SR AEA</td>
<td>1.02</td>
</tr>
<tr>
<td>Water</td>
<td>1.00</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Mix ID</th>
<th>AEA dosage (ml/100 kg cement)</th>
<th>Air content</th>
<th>kg/m$^3$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>avg. (vol. %)</td>
<td>std. dev. (vol. %)</td>
<td>water</td>
</tr>
<tr>
<td>TO30</td>
<td>30</td>
<td>11.9</td>
<td>0.503</td>
</tr>
<tr>
<td>TO100</td>
<td>100</td>
<td>17.6</td>
<td>0.606</td>
</tr>
<tr>
<td>SR30</td>
<td>30</td>
<td>11.0</td>
<td>0.401</td>
</tr>
<tr>
<td>SR110</td>
<td>110</td>
<td>14.3</td>
<td>0.866</td>
</tr>
</tbody>
</table>
6.2.2 Hardened air void analysis by µCT
A 6 mm dia. core was extracted from each of the mortar mixture cube specimens. The µCT scans and reconstructions were performed in the same manner outlined in Section 5.2.1. The determination of the global optimal grayscale threshold ($T_{h_{opt}}$) between air and solid was performed in the same manner outlined in Section 5.2.3. The derivations of $T_{h_{opt}}$ from the distributions of individual air void optimum grayscale threshold ($T_{h_{opt}}$) for the mortar samples are shown in Figures 6.1 and 6.2. Figures 6.3 and 6.4 show example 2D slices before and after application of the grayscale threshold.

![Figure 6.1: Determination of $T_{h_{opt}}$ for sample TO30 (a) and TO100 (b).](image1.png)

![Figure 6.2: Determination of $T_{h_{opt}}$ for sample SR30 (a) and SR110 (b).](image2.png)
In the case of the mortars examined here, the occurrence of air voids in aggregate was negligible, so the only image processing employed for the determination of bulk air content \((A)\) was the removal of 3D air void objects with a minimum diameter \((T_{h_{\text{dmin}}}) < 3\) pixels (22.5 µm) as outlined in Section 5.2.5. Although voids in aggregate were not present, the problem of clustered air voids, or air voids in close proximity to each other, remained an issue, especially for the determination of specific surface \((\alpha)\).
In the case of air voids in close proximity to each other, the resolution of the scan (7.5 µm/pixel) interfered with the detection of the solid paste that separates air voids that are immediately adjacent to each other. In such instances, groupings of voxels that represent two or more air voids would be counted as a single air void, and the true air void surface area underestimated. However, with the application of an image processing watershed, such air voids can be effectively separated. Alternatively, for the case of clustered air voids (where the air voids are not separated by paste, but coalesced and physically connected) the application of a watershed algorithm (Russ and Neal 2016) has the undesired outcome of dividing clusters into segments.
when no such division truly exists, and would result in an overestimation of air void surface. To account for air voids in close proximity to each other, and coalesced air voids, air void objects with a $\theta_{dmin} > 6$ pixels (45 µm) were subjected to watershed based on the same criteria as outlined in Section 5.2.6.

6.2.3 Hardened air void analysis by flatbed scanner

Figure 6.5 shows a zoom stereo microscope overview of the cut and polished surfaces from the TO mortar cubes before performing the black and white contrast enhancement, along with flatbed scanner images after contrast enhancement. Figures 6.6 and 6.7 show close-up images of the areas outlined in Figure 6.5. Figures 6.8-6.10 show the same, but for the SR samples. The general procedures followed for the preparation and analysis are outlined in detail in Peterson et al. (2016). The surfaces were scanned with a V800 EPSON Perfection flatbed scanner at a resolution of 8 µm/pixel (125 dpm) in a $2^8$ grayscale format.

![Figure 6.5: Core sample for µCT and cut and polished surface from mortar cube prior to contrast enhancement (left) and post contrast enhancement (right) for samples TO30 (a), and TO100 (b). Regions outlined in pink show area of subsequent close-up images of Figures 6.6 and 6.7, all images to scale.](image-url)
Figure 6.6: Sample TO30 (a) zoom stereo microscope of polished surface, (b) flatbed scanner image of same area after black and white contrast treatment, (c) flatbed scanner image after grayscale threshold application.
Figure 6.7: Sample TO100 (a) zoom stereo microscope of polished surface, (b) flatbed scanner image of same area after black and white contrast treatment, (c) flatbed scanner image after grayscale threshold application.
Figure 6.8: Core sample for µCT and cut and polished surface from mortar cube prior to contrast enhancement (left) and post contrast enhancement (right) for sample SR30 (a), SR110 (b). Regions outlined in pink show area of subsequent close-up images of Figures 6.9 and 6.10, all images to scale.
Figure 6.9: Sample SR30 (a) zoom stereo microscope of polished surface, (b) flatbed scanner image of same area after black and white contrast treatment, (c) flatbed scanner image after grayscale threshold application.
Figure 6.10: Sample SR110 (a) zoom stereo microscope of polished surface, (b) flatbed scanner image of same area after black and white contrast treatment, (c) flatbed scanner image after grayscale threshold application.
6.3 Results

6.3.1 Flatbed scanner and µCT air void parameters

The air void parameters as measured by the flatbed scanner and µCT are reported in Tables 6.3 and 6.4 respectively. For the computation of spacing factor ($\bar{L}$) the paste volume was based on the volume ratios from the mix design as outlined in Equation 6.1.

$$ p = \frac{(100 - A)}{M + 1} $$

Equation 6.1

Where:

$p = \text{paste content (vol. %)}$

$A = \text{air content (vol. %)}$

$M = \text{aggregate to paste ratio by volume}$

Table 6.3: Air void parameters from flatbed scanner.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>total traverse length (mm)</th>
<th>specific surface, $\alpha$ (mm$^{-1}$)</th>
<th>void freq. (void/cm)</th>
<th>air content, $A$ (vol. %)</th>
<th>paste content, $p$ (vol. %)</th>
<th>spacing factor, $\bar{L}$ (mm)</th>
<th>paste to air ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>TO30</td>
<td>3763.2</td>
<td>26.8</td>
<td>8.6</td>
<td>12.8</td>
<td>43.4</td>
<td>0.126</td>
<td>3.39</td>
</tr>
<tr>
<td>TO100</td>
<td>3772.8</td>
<td>32.5</td>
<td>15.2</td>
<td>18.7</td>
<td>40.4</td>
<td>0.067</td>
<td>2.16</td>
</tr>
<tr>
<td>SR30</td>
<td>3859.2</td>
<td>18.7</td>
<td>7.7</td>
<td>16.5</td>
<td>41.7</td>
<td>0.136</td>
<td>2.53</td>
</tr>
<tr>
<td>SR110</td>
<td>3945.6</td>
<td>31.2</td>
<td>12.8</td>
<td>16.4</td>
<td>41.6</td>
<td>0.081</td>
<td>2.54</td>
</tr>
</tbody>
</table>

Table 6.4: Air void parameters from µCT.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>volume sampled (mm$^3$)</th>
<th>specific surface, $\alpha$ (mm$^{-1}$)</th>
<th>number density (voids/m$^3$)</th>
<th>air content, $A$ (vol. %)</th>
<th>paste content, $p$ (vol. %)</th>
<th>spacing factor, $\bar{L}$ (mm)</th>
<th>paste to air ratio</th>
</tr>
</thead>
<tbody>
<tr>
<td>TO30</td>
<td>200.44</td>
<td>29.3</td>
<td>$25.0 \times 10^{10}$</td>
<td>10.9</td>
<td>44.4</td>
<td>0.139</td>
<td>4.07</td>
</tr>
<tr>
<td>TO100</td>
<td>200.53</td>
<td>40.4</td>
<td>$31.6 \times 10^{10}$</td>
<td>19.1</td>
<td>40.5</td>
<td>0.052</td>
<td>2.12</td>
</tr>
<tr>
<td>SR30</td>
<td>182.51</td>
<td>23.2</td>
<td>$14.4 \times 10^{10}$</td>
<td>10.2</td>
<td>44.7</td>
<td>0.188</td>
<td>4.38</td>
</tr>
<tr>
<td>SR110</td>
<td>188.46</td>
<td>36.7</td>
<td>$32.9 \times 10^{10}$</td>
<td>11.6</td>
<td>44.0</td>
<td>0.103</td>
<td>3.79</td>
</tr>
</tbody>
</table>

6.3.2 Air void size distributions from different AEAs

The air void size distributions as determined by µCT are shown in Figures 6.11 as fit using a lognormal distribution. The application of the lognormal distribution to air void size distributions (Equation 6.2) was first suggested by Roberts and Scheiner (1981) and has been used by many subsequent researchers, as summarized in Snyder et al. (2001). Table 6.5 lists the parameters for the lognormal distribution fitted curves. From Figure 6.11 it can be seen that the synthetic AEA (TO) generated a finer air void system with larger numbers of small diameter air voids as compared to the vinsol resin AEA (SR).
\[ p(x) = \frac{1}{\sigma \sqrt{2\pi}} e^{-(\ln x - \mu)^2 / 2\sigma^2} \]

Equation 6.2

Where:
\( \mu \) = mean
\( \sigma^2 \) = variance

Table 6.5: Parameters for fitted lognormal distributions.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>( \mu )</th>
<th>( \sigma )</th>
<th>95% confidence interval for ( \mu )</th>
<th>95% confidence interval for ( \sigma )</th>
</tr>
</thead>
<tbody>
<tr>
<td>TO30</td>
<td>4.1439</td>
<td>0.3152</td>
<td>[4.1411, 4.1466]</td>
<td>[0.3133, 0.3172]</td>
</tr>
<tr>
<td>TO100</td>
<td>4.1161</td>
<td>0.2668</td>
<td>[4.1140, 4.1182]</td>
<td>[0.2653, 0.2682]</td>
</tr>
<tr>
<td>SR30</td>
<td>4.1655</td>
<td>0.3477</td>
<td>[4.1613, 4.1657]</td>
<td>[0.3448, 0.3507]</td>
</tr>
<tr>
<td>SR110</td>
<td>4.1305</td>
<td>0.3037</td>
<td>[4.1281, 4.1329]</td>
<td>[0.3020, 0.3054]</td>
</tr>
</tbody>
</table>

Figure 6.11: Air void size distributions for TO30 (a), TO100 (b), SR30 (c), and SR110 (d) fitted with lognormal distribution.
6.3.3 Flatbed scanner vs. µCT bulk air content determination

The most problematic air void parameter for the µCT, in terms of the duplication of results from the flatbed scanner stereological method, was the measurement of $A$. For this research, the area analyzed by the flatbed scanner covered the full 50 mm$^2$ cross-section of a mortar cube, and allowed for a representative sample of the larger (entrapped) air voids in the 5 mm diameter range. On the other hand, the limited dimensions of the samples retrieved for µCT, i.e. 6 mm diameter cores approximately 2 cm in length, precluded the acquisition of an adequate number of large air voids (Figures 6.5 and 6.8). The retrieval of a 6 mm dia. core containing the largest of air voids was not possible, as breaks in the cores occured whenever a large air void was encountered. Since the occurrence of large air voids exerts a strong control on the value obtained for $A$, and the sample size used in this µCT application necessarily excludes large air voids, discrepancies in the results for $A$ are inevitable. This problem could be addressed by performing an additional scan at a lower resolution covering the entire 50 mm$^3$ mortar cube to capture the larger air voids, and then unite the air void size distributions from the high resolution and low resolution scan data using a power law as described in Chapter 4. An alternative approach was implemented here using the power law to fit the distributions from the 6 mm dia. cores, and then obtaining an estimate for $A$ based on the power law distribution. In this manner, the occurrence of larger air voids not captured during the core sampling could be accounted for. Figure 6.12 shows the power law fitted air void size distributions, with the parameters summarized in Table 6.6.

In theory, the largest possible air void contained within a 50 mm cube specimen would be a 50 mm dia. air bubble. In practice, a more reasonable upper boundary for air void diameters in this case would be on the order of 1 cm. As such, estimates for $A$ based on the power law curve were extended over a maximum air void diameter range covering 2 to 10 mm (Table 6.7). For the TO100 sample, the µCT and flatbed scanner results for $A$ were already in general agreement to begin with, and the power law derived estimates for $A$ remained close to the initial values (Figure 6.13b). For the other samples, the initial µCT results for $A$ were significantly lower than those reported by the flatbed scanner. But, as the upper air void diameter limits were increased, the power law curve based estimates for $A$ began to approach the values reported for the flatbed scanner. In the case of sample TO30 (Figure 6.13a) the power law curve estimate for $A$ achieved the same value reported by the flatbed scanner when the upper air void diameter limit reached 10
mm. For samples SR30 and SR110, the power law curve values for $A$ remained lower than the values reported by the flatbed scanner, even at the upper air void diameter limit of 10 mm (Figures 6.13c and 6.13d).

Table 6.6: Parameters for fitted power law curves.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>$x_{\text{min}}$</th>
<th>$\alpha$</th>
<th>Standard deviation of $\alpha$</th>
<th>Log-likelihood of fit</th>
</tr>
</thead>
<tbody>
<tr>
<td>TO30</td>
<td>59.062</td>
<td>4.312</td>
<td>0.014795</td>
<td>-99693</td>
</tr>
<tr>
<td>TO100</td>
<td>89.844</td>
<td>4.8317</td>
<td>0.015229</td>
<td>-23103</td>
</tr>
<tr>
<td>SR30</td>
<td>53.182</td>
<td>3.963</td>
<td>0.018306</td>
<td>-72949</td>
</tr>
<tr>
<td>SR110</td>
<td>52.683</td>
<td>4.3743</td>
<td>0.013553</td>
<td>-1.64E+005</td>
</tr>
</tbody>
</table>

Table 6.7: Power law estimates of $A$ based on increasing upper air void diameter limit.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Air content $A$, vol %, with increasing upper air void dia. limit</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>2 mm</td>
</tr>
<tr>
<td>TO30</td>
<td>11.50</td>
</tr>
</tbody>
</table>
6.4 Discussion and conclusions

The air void parameters from the µCT compared favorably with those reported for the more conventional stereology-based approach of the flatbed scanner, with an average difference between the measured pairs of 17 % for $\alpha$, 25 % for $A$, and 23 % for $\bar{L}$. To place this within the context of the standard test method currently used for the microscopical determination of air void parameters, ASTM C457 (2012), COVs are reported in a range of 8 to 15 % for $A$, and 8 to 20 % for $\bar{L}$, with no statistics reported for $\alpha$. COVs from seven years of an ongoing Ministry of Transportation Ontario operator correlation program for the full population of operators that routinely perform air void testing are reported in the range of 11 to 28 % for $A$, 17 to 32 % for $\bar{L}$, and 3 to 12 % for $\alpha$ (Peterson et al. 2016).
Given the constraints of the mm scale sample size for µCT, obtaining an accurate measurement of \( A \) is not possible, as the sample size excludes the possibility of including the largest entrapped air voids. However, by fitting the measured air void size distribution with a power law, it is possible to extend the range of air void diameters beyond the limitations of the sample size, and obtain a more reasonable estimate for \( A \).
7 Significance of Findings and Recommendations for Future Work

Entrained and entrapped air voids in cement-based materials possess a variety of features that have direct implications on the physical properties of the bulk material. Their sheer numbers, mechanism of generation, and wide spectrum in size distribution all provide incentives to develop a more unified and systematic method for their measurement. In the context of µCT techniques applied for this purpose, efforts are increasing but still limited. This dissertation introduces several innovative approaches to the processing of µCT data for air void system characterization in cement-based materials. Demonstrations were carried out to varying degrees on lightweight concrete, structural and pavement concrete from the field, and laboratory mortars. The primary contributions include:

- This study represents the first application of active contour line methods to air void segmentation, and provides a more objective approach to the selection of a global grayscale threshold value to separate phases in materials. The active contour line method proved more robust when compared to other traditional methods for grayscale threshold determination in images complicated by signal overlap and noise. The active contour line based approach was demonstrated successfully on all three sets of materials covered in this study.

- This study introduces shape-based 3D spherical solidity as a parameter to distinguish entrained air voids in the paste from voids in aggregate. The 3D spherical solidity method proved practical in terms of its reasonable computation demand, and in terms of simplifying the task of selecting an appropriate threshold parameter value.

- This study provides guidance for the application of a 3D watershed algorithm to identify coalesced air void clusters, a not uncommon feature in concrete, as well as to distinguish air voids in close proximity to each other, yet not physically connected. The effective treatment of such air void objects allowed for a more reliable estimation of relevant air void parameters.

- This study introduces procedures that can be followed to measure $\bar{L}$ and $\alpha$ for concrete with a relatively small number of cm scale µCT samples. The results from field concretes with both acceptable and marginal air void parameters obtained from a routine MTO quality
assurance sampling and testing program compared favorably with traditional optical stereology based test results from cut and polished slabs. The reproducibility of these parameters among different cm-sized samples, expressed in terms of COV was improved as compared to COVs reported in ASTM C457 (2012).

- This study provides an estimate of the required sample volumes to reduce the corresponding levels of measurement uncertainty for measurements of air void parameters.

- This study provides a methodology to deal with discontinuities between air void size distributions collected at different spatial resolutions, a common problem for materials that contain features covering a wide spectrum of sizes. For the lightweight concrete examined in this study, the combined distribution was satisfactorily fitted by a power law distribution, and the modified bulk air contents based on the fitted curves were in general agreement with the computed mix design (9.0 ± 0.5 vs. 10.8 vol. %).

Aside from the primary contributions, a number of limitations of µCT as applied to air void characterization were identified, along with areas for potential improvement and development as follows:

- The shape-based parameters of the proposed processing method for µCT scans are tied to a specific resolution. The parameters have to be modified for the same object if scanned at a different resolution, e.g. the 3D spherical solidity threshold will tend to be smaller for the same object when scanned at a lower resolution. The proposed shape-based processing should be tested on samples scanned under a systematically varied series of resolutions, and the data analyzed to monitor changes in the parameters, to provide increased accuracy for similar analyses in the future.

- For concrete or mortar with a high degree of air void clustering, the geometric shape of the object consisting of connected bubbles can become extremely complicated, and limits the utility of mainstream 3D watershed algorithms. More work is needed in this area. To help with this effort, a visualization code is provided in Appendix G that can assist with the experimentation with, and the fine-tuning of, watershed parameters.
For the measurement of the entrained air void system parameters examined in this study, most of the errors could be attributed to the fraction of bubbles with the smallest diameters. The scanning resolution capacity of many of the current µCT machines is still a limiting factor, but constantly improving. Algorithms that can fit an objective boundary based on grayscale value transition or geometric shape should be further explored as economic alternatives for machines with increasing resolution.

Different from 2D characterization, air voids characterized using µCT have not only the size information but also the exact 3D spatial information of individual bubbles. The added information provides more opportunities not previously available to analyze and characterize the subtle differences in structures of the air void system. A concept in statistics called the point process may provide a natural tool to deal with these types of systems.

The procedures proposed in this study can be used to study many possible applications in the field of cement-based materials. For example, one promising area for future research could involve the investigation of the evolution of air void systems using X-ray transparent pressure and temperature vessels inside a µCT to simulate the in situ environments encountered during concrete pumping for general construction and for oil well cementation.
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Appendix

Appendix A - Code for cropping initial 3D ROI from reconstructed image

This code is for preprocessing of the raw µCT data, and has been tested using MATLAB® version R2016a(9.0.0.341360).

The input includes: grayscale image stacks in tiff format encoded at either 16 bit or 8 bit.

The output includes: grayscale image stacks with a cropped cylindrical shape.
%This is a code segment to pre-process raw X-ray CT data; It is set up for reading '.tiff' image stacks encoded either at unit16 or unit8 and the output is cylindrical stack images which are more amenable to systematic algorithmic analysis that followed. The code is configured for multiple cpu-core platform.

clc;
clf
clear
close all
Tfolder=uigetdir;% to locate the folder containing images
TdirListing=dir(Tfolder);% Get the info of file
for d=1:length(TdirListing);
    if ~TdirListing(d).isdir % there are usually two hidden files to get rid of
        fileName=fullfile(Tfolder,TdirListing(d).name);%since the file name is not usually in order, so need to get the names of the image
        A=imread(fileName);%load the image into matlab
        figure('Visible','Off')
        imshow(A)
        im_attr=imattributes;
        im_class=im_attr{3,2}; % the class of the image: uint16 uint8 etc.
        im_type=im_attr{4,2}; % the type of the image: intensity, RGB, binary etc
        break
    end
end
if strcmp(im_class,'uint16')
    bin=2^16;
    bit=16;
    grid=5;
end
elseif strcmp(im_class,'uint8')
    bin=2^8;
    bit=8;
    grid=0.005;
else
    disp('This code can only deal with unit16 or uint8 class')
end

[cx,cy]=size(A);

 falsely
[3:stp:(length(TdirListing))];

parfor d=1:n_d; % there are two hidden files that are not images in the fold
selected, so the first images actually starts from 3.
   fileName=fullfile(Tfolder,TdirListing(s_mar(d)).name);% Since the file
name is not usually in order, so need to get the names of the image
   A=imread(fileName);%load the image into matlab
   bw_act= activecontour(A,mask,1200,'Chan-Vese',1);
   bw_act=imfill(bw_act,'holes');
   CC=bwconncomp(bw_act);
   n=CC.NumObjects;
   if n>=2
       L = bwlabel(bw_act) ;
stats = regionprops(CC, 'Area');
stats_area = [stats.Area];
[max_area, index] = max(stats_area);
bw_act = ismember(L, index);
end
CC = bwconncomp(bw_act);
stats = regionprops(CC, 'MajorAxisLength', 'MinorAxisLength', 'Centroid');
stats_major(d) = max([stats.MajorAxisLength]);
stats_minor(d) = max([stats.MinorAxisLength]);
stats_centroid{d} = [stats.Centroid];
end
xy = stats_centroid{1};
r = stats_minor(1)/2;
[x, y] = meshgrid(-(xy(1)-1):(cx-xy(1)), -(xy(2)-1):(cy-xy(2)));
mask0 = ((x.^2+y.^2) <= r^2);
% locate a common crop across the image slices
for d = 2:n_d
    xy = stats_centroid{d};
r = stats_minor(d)/2;
    [x, y] = meshgrid(-(xy(1)-1):(cx-xy(1)), -(xy(2)-1):(cy-xy(2)));
    mask = ((x.^2+y.^2) <= r^2);
    mask0 = mask0 & mask;
end
CC = bwconncomp(mask0);
stats = regionprops(CC, 'MinorAxisLength', 'Centroid');
crop_radius = (stats.MinorAxisLength)/2;
crop_xy = stats.Centroid;
    [x, y] = meshgrid(-(crop_xy(1)-1):(cx-crop_xy(1)), -(crop_xy(2)-1):(cy-crop_xy(2)));
150
crop_mask0=((x.^2+y.^2)<=crop_radius^2);

disp('Common crop area decided')
disp('start cropping images....')

    n_effective=0;
    for d=1:length(TdirListing);
        if ~TdirListing(d).isdir % there are usually hidden files to get rid of
            n_effective=n_effective+1;
            fileName=fullfile(Tfolder,TdirListing(d).name);%since the file name is
            not usually in order, so need to get the names of the image
            A=imread(fileName);%load the image into matlab
            A=imcomplement(A);
            A(~crop_mask0)=NaN;
            A=imcrop(A,[crop_xy(1)-crop_radius-10,
            crop_xy(2)-crop_radius-10,2*crop_radius+20,2*crop_radius+20]);
            gray_scale_3d(:,:,n_effective)=A;
        end
    end

disp('images cropped')
disp('start calibrating images....')

%use histogram peak to calibrate an individual scan which will reduce the
errors inherent among different scans This part should be modified when
specific calibration material is installed.

    m=size(gray_scale_3d);
    I=reshape(gray_scale_3d,m(1)*m(2)*m(3),1);
    [counts,L] = imhist(I);
    [x,y]=findpeaks(counts);
    [x1,y1]=sort(x,'descend');
    gray_scale_cut_off=L(y(y1(1:2)))
    for i=1:size(gray_scale_3d,3)
a=gray_scale_3d(:,:,i);

a=imadjust(a,[gray_scale_cut_off(1)/(2^bit),gray_scale_cut_off(2)/(2^bit)],[0 ,1]);

gray_scale_3d_adjust(:,:,i)=a;

end

clearvars -except gray_scale_3d_adjust crop_radius gray_scale_cut_off
Tfolder

disp('done')

% Output includes (1)cylindrical grayscale image stacks(calibrated);(2)crop
% radius;(3)cut off grayscale values for calibration purposes.

out_put_name=strcat(Tfolder(~ismember(Tfolder,pwd)),'_calibrated_slices','.ma t')

save(out_put_name,'gray_scale_3d_adjust','crop_radius','gray_scale_cut_off')
Appendix B - Code for image calibration and active contour line extraction of features

This code aims to enhance the contrast of the images as well as determine the global grayscale threshold value to discern solid and air portion of the material, and has been tested using MATLAB® version R2016a(9.0.0.341360).

The input includes: image stacks in tiff format encoded at either 16 bit or 8 bit.

The output includes: the threshold intensity value between air and solid.
This code is used to calibrate the original images from Xray-ct scan and then get the threshold value of the calibrated images. For calibration, it is based on the alignment of two histogram peaks, that from image parts representing the control material (copper) and that of air. This processing can reduce the errors that could arise from random factors and varying setups from different scans. To get the threshold value of calibrated images, a comparison of air content between the images threshed based on varying threshold value and that segmented using active contour line is carried out.

The inputs of this code includes: (1) 16 bit grayscale image slices in a folder; (2) The name of the folder where the CT images are placed: "name" (a string); (3) The number of CPU cores that would like to be used: "n_cpu" (a value); (4) The resolution of the scan used: "n_reso" (a value); (5) The way that the original image slices are cropped for analysis: "crop_position" (a matlab file '.mat')

The outputs of this code includes: (1) The threshold value (between image representing solid and air): "name__input data" (a matlab file '.mat'); (2) Other parameters used in the code: "name__input data" (a matlab file '.mat')

The dependent matlab functions include: (1) imhist3 -- function that draw 3D image histogram; (2) extrema -- function that calculate the extreme values of a graph

Some assumptions: (1) The input images are 16 bit grayscale image slices; (2) The computers that this code is run have matlab "Image Processing Toolbox", "Parallel Computing Toolbox" modules; (3) This code is used as part of the code in a unix shell scripting language

Reasons for possible modification: (1) Change of input image format; (2) Use of a different control material. e.g. distilled water; (3) Running on a single cpu computer or using matlab packages without Parallel Computing Toolbox; (3) This code is used as part of the code in a unix shell scripting language

name='044A_2_3_0'; n_cpu=4; n_reso=7.5;
try
warning('off','all');
tic
% compile the 3-d volume from 2-d slices (for calibration)
Tfolder0=strcat(pwd,'/');
Tfolder=strcat(Tfolder0,name);
poolobj = gcp('nocreate'); % If no pool, do not create new one.
    if isempty(poolobj)
        poolsize = 0;
    else
        poolsize = poolobj.NumWorkers;
    end
    if poolsize~=n_cpu
        delete(poolobj)
        parpool(n_cpu)
    else
    end
    st=strcat('This program is running under : ', num2str(n_cpu), ' Cores');
    disp(st)

TdirListing=dir(Tfolder); % get the info of file, like how many images
reso=n_reso; % resolution of the image
load('crop_position');
namex=Tfolder((numel(Tfolder)-numel(name)+1): numel(Tfolder));
new_fold=strcat(namex,'_results');
mkdir(new_fold)
namex2=strcat('a',namex);
namex3=strcat(namex2,'_xyz');
namex3=eval(namex3);
    cx=namex3(3);cy=namex3(4);ix=namex3(5);iy=namex3(6);x=namex3(7); % since the images are not usually in the middle and the sample is not perfectly circle, so the crop is prepared based on its real location in order to capture as much as info as possible.
w=20; % small grid width in pixel
n_c=36; % slices used for calibration purpose
\[
\text{w_m} = \max(\min([\text{ix-cx-r}, \text{cx-r}, \text{iy-cy-r}, \text{cy-r}]) \times \frac{1}{10}, 2w + 1); \quad \text{~~~w_m is the distance from the edge of object to image border}
\]

\[
\text{w_m} = \text{round}(\text{w_m}); \quad \% \text{ preparing mask2 below, the parameter value need to be an integer}
\]

\[
\text{for } d = 1: (n_c+2);
\]

\[
\text{if ~} \neg \text{TdirListing(d).isdir ~ there are usually hided files, just get rid of them}
\]

\[
\text{fileName} = \text{fullfile(Tfolder, TdirListing(d).name)}; \quad \% \text{since the file name is not usually in order, so need to get the names of the image}
\]

\[
\text{a} = \text{imread(fileName)}; \quad \% \text{load the image into matlab}
\]

\[
\text{a} = \text{imcomplement(a)};
\]

\[
\text{C(:,:,d-2)} = \text{a};
\]

\[
\text{clear a;}
\]

\[
\text{end}
\]

\[
\text{end}
\]

\[
\text{figure('Visible','Off')}
\]

\[
\text{y} = \text{imhist3(C,65536-1)}; \quad \% \text{external function \text{imhist3}}
\]

\[
\text{clear C}
\]

\[
\text{x} = 1:1:65535;
\]

\[
\text{k} = 10;
\]

\[
\text{pp} = \text{pchip(x(1:k: numel(x)), y(1:k: numel(y)))};
\]

\[
\text{for } i = 1: \text{floor}((65535/k))
\]

\[
\text{x} = \text{linspace(pp.breaks(i), pp.breaks(i+1), 20); \% a 20 element vector}
\]

\[
\text{ys} = \text{polyval((pp.coefs(i,:)), xs-pp.breaks(i)); \% matrix of y for each x}
\]

\[
\text{s(i)} = ((pp.breaks(i+1)-pp.breaks(i))) / 20 \times \text{sum(ys)};
\]

\[
\text{end}
\]

\[
\text{s} = \text{smooth(s, 200)};
\]

\[
\text{t} = 1:k: (65536-k);
\]

\[
[y_{\text{max}}, y_{\text{im}}, y_{\text{min}}, y_{\text{min}}] = \text{extrema(s)}; \quad \% \text{external function}
\]
[x,y]=sort(ymax,'descend');

threshold_r=k*imax(y(1))

for i=1:50
    if k*imax(y(i))<20000;
        threshold_l=k*imax(y(i))
        break
    else
    end
end

if exist('threshold_l')==0
    threshold_l=threshold_r-50000
disp('threshold exception')
else
end

if name=='052A_1_1_0';
disp('052A_1_1_0 is an exception for calibration')
threshold_r=51650
threshold_l=7070
else
end

if name=='052A_1_6_1';
disp('052A_1_6_1 is an exception for calibration')
threshold_r=51650
threshold_l=7070
else
end

end
stp=30; % the step of taking slices
k1=0;
k_ma=[3:stp:length(TdirListing)];

parfor d=1:numel(k_ma)
    
    fileName=fullfile(Tfolder,TdirListing(k_ma(d)).name); % since the file
    name is not usually in order, so need to get the names of the image
    
    a=imread(fileName); % load the image into matlab
    a=imcomplement(a);
    [x,y]=meshgrid(-(cx-1):(ix-cx),-(cy-1):(iy-cy));
    mask=((x.^2+y.^2)<=r^2);
    a(~mask)=NaN; % do the mask
    a=imcrop(a,[cx-r_w_m,cy-r_w_m,2*r+2*w_m,2*r+2*w_m]);
    C(:,:,d)=a;
end

m=size(C);
Cl=reshape(C,m(1)*m(2)*m(3),1);
Cl=imadjust(Cl,[threshold_l/65535,threshold_r/65535],[0,1]);
Cl=imadjust(Cl,[60000/65535,1],[0,1]); %%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%

for i=1:m(3)
    Cl_1=Cl(((i-1)*m(1)+1):i*m(1)*m(2));
    for j=1:m(2)
        C2(:,:,i)=Cl_1(((j-1)*m(1)+1):j*m(1));
    end
end

clear Cl,Cl_1;
disp('3D image construct done')

C21=reshape(C2,m(1)*m(2)*m(3),1);
figure('Visible','off') % figure 2
imhist(C21)
%set(gca,'FontSize',15)
xlabel('Grayscale value','FontWeight','bold')
ylabel('Frequency','FontWeight','bold')

name2=Tfolder((numel(Tfolder)-numel(name)+1): numel(Tfolder));
dir_name=pwd;
name2f=strcat(dir_name,'/',new_fold,'/',name2,'_Histogram');
saveas(gcf,name2f,'png')
clear C21
n_effective=0;

for j=1:m(3)
a=C2(:,:,j);
b=im2bw(a,0.70); %preset threshold value
b1=imfill(b,'holes');
D=bwdist(~b1,'Quasi-Euclidean');
D=-D;
k2=watershed(D,8);

b1(k2 == 0)= 0; % the watershed part of the air bubble

b1=bwareaopen(b1,200);******
CC1=bwconncomp(b1);
STATS=regionprops(CC1, 'Eccentricity','Area','BoundingBox','Solidity');
ec=[STATS.Eccentricity];
ar=[STATS.Area];
bb=[STATS.BoundingBox];
so=[STATS.Solidity];

index_ec=find(ec<0.90);

index_soli=find(so>0.9);

index_t=find(ismember(index_ec,index_soli));

index_t=index_ec(index_t);

index_ar=find(ar<=5000);

index_t1=find(ismember(index_t,index_ar));

index_t=index_t(index_t1);

if numel(index_t)>0

bb_1=reshape(bb',4,numel(bb)/4);

bb_1=bb_1(:,index_t);

bb_1=[(bb_1(1,:)-5)',(bb_1(2,:)-5)',(bb_1(3,:)+10)',(bb_1(4,:)+10)'];

for il=1:numel(index_t)

    n_effective=n_effective+1;
    solid=imcrop(a,bb_1(il,:));
    range=[0.4:0.001:0.9];
    cx3=bb_1(il,3)/2;
    cy3=bb_1(il,4)/2;
    ix1=size(solid,2);
    iy1=size(solid,1);
    [x,y]=meshgrid(-(cx3-1):(ix1-cx3),-(cy3-1):(iy1-cy3));
    mask3=((x.^2+y.^2)<=(min(cx3,cy3)/2)^2);
    bw_act= activecontour(solid,mask3,200,'Chan-Vese',1);
    bw_act=imfill(bw_act,'holes');
    solid1=bw_act;
    solid_crop=solid;
    solid_crop(~bw_act)=NaN;
parfor i=1:numel(range)
    solid1=im2bw(solid,range(i));
    CS=bwconncomp(solid1);
    STATS1=regionprops(CS,'Area');
    air_t=[STATS1.Area];
    if numel(air_t)>0
        air(i)=max(air_t);
    else
        air(i)=NaN;
    end
end

air1=bwarea(solid1);
ab_value=abs(air-air1);
[ab_v_rank,ord]=sort(ab_value);
index=ord(1:2);
shape_1=ones(300,300); % this size is critical for the color of mixing
RA = imref2d(size(shape_1));
RB = imref2d(size(solid));
RB.XWorldLimits=RA.XWorldLimits;
RB.YWorldLimits=RA.YWorldLimits;
[solid_new,RC]=imfuse(shape_1,RA, solid, RB,'blend');
[ed_new,RC]=imfuse(shape_1,RA, solid_crop, RB,'blend');
    %solid_new=imfuse(shape_1,solid,'blend','Scaling','joint');
    %ed_new=imfuse(shape_1,ed,'blend','Scaling','joint');
C_show_soli(:,:,,:,n_effective)=solid_new;
C_show_ed(:,:,,:,n_effective)=ed_new;
thresh((2*(n_effective-1)+1):2*n_effective)=range(index);
end
end
disp(m(3)-j)
end

thresh(find(thresh>=0.87))=NaN;
thresh=thresh(find(thresh>0));
figure('Visible','off')
h=histfit(thresh,40,'kernel');
x=get(h(2),'XData');
y=get(h(2),'YData');
%[counts threshold1]=hist(thresh,50);
threshold=x(find(ismember(y,max(y))))
hold on
h1=stem(threshold,max(y));
set(h1(1),'Color','r','LineWidth',3) % Set marker properties
% xlabel('Threshold value','FontSize',15,'FontWeight','bold')
% ylabel('Frequency','FontSize',15,'FontWeight','bold')
% set(gca,'FontSize',15)
xlabel('Threshold value','FontWeight','bold')
ylabel('Frequency','FontWeight','bold')

name3=Tfolder((numel(Tfolder)-9): numel(Tfolder));

name3f=strcat(dir_name,'/',new_fold,'/',name3,'_Threshold value
determination');
saveas(gcf,name3f,'png')

%figure('Visible','off')
figure
h=gcf;
set(h,'Position', [0 0 1000 1000])

162
montage(C_show_soli)

name3f1=strcat(dir_name,'/',new_fold,'/',name3,'_Images for air bubbles');

saveas(gcf,name3f1,'png')

%figure('Visible','off')

figure

montage(C_show_ed)

name3f2=strcat(dir_name,'/',new_fold,'/',name3,'_Images for air bubble edges');

saveas(gcf,name3f2,'png')

namel=Tfolder((numel(Tfolder)-9): numel(Tfolder));

namelf=strcat(namel,'_input data');

clearvars -except name namelf threshold_l threshold_r threshold cx cy ix iy r n_reso w_m w Tfolder n_cpu ;

save(namelf)

toc

catch

disp('**error happens during getting the threshold**')

exit

disp
end
Appendix C - Code for isolation of air voids in the paste

This code aims to isolate air voids in the paste, and has been tested using MATLAB® version R2016a(9.0.0.341360).

The input includes: grayscale image stacks with a cylindrical shape and the threshold intensity value for air and solid.

The output includes: size and spatial info of air voids in the paste.
% This code is used to get the 3D entrained air

% The inputs of this code includes:

% (1) 16 bit grayscale image slices in a folder

% (2) The name of the folder where the CT images are placed: "name"(a string)

% (3) The number of CPU cores that would like to be used : "n_cpu"(a value)

% (4) The resolution of the scan used: "n_reso"(a value)

% (5) The way that the original image slices are cropped for analysis: "crop_position"(a matlab file '.mat')

% The outputs of this code includes:

% (1) The threshold value(between image representing solid and air): "name__input data"(a matlab file '.mat')

% (2) Other parameters used in the code: "name__input data"(a matlab file '.mat')

% The dependent matlab functions include:

% (1) imhist3--function that draw 3D image histogram

% (2) extrema--function that calculate the extreme values of a graph

% Some assumptions:

% (1) The input images are 16 bit grayscale image slices

% (2) The computers that this code is run have matlab "Image Processing Toolbox","Parallel Computing Toolbox" modules

% (3) This code is used as part of the code in a unix shell scripting language

% Reasons for possible modification:

% (1) Change of input image format

% (2) Use of a different control material. e.g. distilled water

% (3) Running on a single cpu computer or using matlab packages without Parallel Computing Toolbox"
name = '044A_4_2_0'; n_cpu = 8; re_a = 3; re_al = 10; point_n = 5; soli_v1 = 0.7; soli_v2 = 0.75;

parfor i = 1: size(C2, 3)
    a = C3(:, :, i);
    b = im2bw(a, threshold);
    C_w(:, :, i) = b;  % binary without watershed;
end

C_show{3} = C_w(:, :, n_show);
A_without = A_void(C_w, r);
fprintf('The air content without any processing is %.4f in percent\n', A_void(C_w, r))

clear C2
for i = 1:5
    C4_t = bwareaopen(C_w, i * 0);  % with noise removal;
    Air_c = A_void(C4_t, r);
    fprintf('The air content with %.2f pixel removal upon watershed image is %.4f in percent\n', i * 5, A_void(C4_t, r))
    C_show{3+i} = C4_t(:, :, n_show);
end

clear C_w;
dir_name = pwd;
% clear C2

name_C3 = strcat(name, '_3d_image');
save(name_C3, 'C3')
clear C3
name_C4 = strcat(name, '_3d_all_void_image');
save(name_C4, 'C4_t')
cx2 = cx - (cx - w_m); cy2 = cy - (cy - w_m); ix0 = r * 2 + w_m * 2 + 1; iy0 = r * 2 + w_m * 2 + 1; % be careful on the size of ix1, iy1
\[
\begin{align*}
\text{[x,y]=meshgrid((-cx2-1):(ix0-cx2),-(cy2-1):(iy0-cy2));} \\
\text{mask2=((x.^2+y.^2)<=r^2);} \\
\text{clearvars -except C4_t mask mask2 n_reso name C_show dir name new fold n_show r soli_v1 soli_v2 re_a re_a1 point_n A_without} \\
\text{toc} \\
\text{try} \\
\text{warning('off','all');} \\
\text{tic} \\
\text{re_a3=re_a;} \\
\text{name1=strcat(name,'_input data');} \\
\text{load(name1)} \\
\text{re_a=re_a3;} \\
\text{C_air=C4_t;} \\
\text{clear C4_t;} \\
\text{j_t=size(C_air);} \\
\text{n_slice=size(C_air,3);} \\
\text{%C_air_t=bwareaopen (C_air,re_a);} \\
\text{%A_without1=A_void(C_air_t,r);} \\
\text{%clear C_air_t} \\
\text{CC2=bwconncomp(C_air);} \\
\text{clear C_air} \\
\text{STATS=regionprops(CC2, 'Area','BoundingBox','PixelList');} \\
\text{ar=[STATS.Area];} \\
\text{index_t=find(ar<1000000);} \\
\text{arl=ar(index_t);} \\
\text{bb={STATS.BoundingBox};} \\
\text{bb_1=bb(index_t);} \\
\end{align*}
\]
bb_2 = cell2mat(bb_1');
bb_2 = round(bb_2);
PL = {STATS.PixelList};
clear STATS
PL1 = PL(index_t);
clear PL
xs = bb_2(:,1);
ys = bb_2(:,2);
zs = bb_2(:,3);
xx = bb_2(:,4);
yy = bb_2(:,5);
zz = bb_2(:,6);
clear bb_1 bb_2
n_im3d_index = 0;
for i = 1:numel(index_t);
    im3d = zeros(xx(i), yy(i), zz(i));
    PL2 = cell2mat(PL1(i));
    PL3 = [PL2(:,1) - xs(i) + 1, PL2(:,2) - ys(i) + 1, PL2(:,3) - zs(i) + 1];
    for j = 1:size(PL3,1)
        xyz = round(PL3(j,:));
        im3d(xyz(1), xyz(2), xyz(3)) = 1;
    end
    m1 = size(im3d);
    if numel(m1) == 3 & size(im3d,1) >= re_a & size(im3d,2) >= re_a & size(im3d,3) >= re_a
        n_im3d_index = n_im3d_index + 1;
        im3d_t{n_im3d_index} = im3d;
        index_t1(n_im3d_index) = index_t(i);
    else

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n_im3d_index=n_im3d_index+1;
im3d_t{n_im3d_index}=[];
index_t1(n_im3d_index)=index_t(i);
end
end
for ii=1:n_im3d_index
    ka=im3d_t{ii};
a_tv(ii)=sum(ka(:));
end
indec=find(a_tv==0);
n_small=size(indec,2);
im3d_tt=im3d_t(find(~ismember([1:n_im3d_index],indec)));

for ii=1:numel(im3d_tt)
    ka1=im3d_tt{ii};
a_tv1(ii)=sum(ka1(:));
end
air_rm=100*sum(a_tv1(:))/(pi*r*r*n_slice);% air content after romove small than 3*3*3

clear PL1 a_tv a_tv1 %CC2

% start
index_t=index_t1;
clear index_t1;
index_t_p=index_t;
clear index_t
im3d_t_p=im3d_t;
clear im3d_t
xs_p = xs;
y_p = ys;
zs_p = zs;
xx_p = xx;

yy_p = yy;
zz_p = zz;

name1 = strcat(name, '_small_3D');

save(name1, 'im3d_t_p', 'xs', 'ys', 'zs', 'xx', 'yy', 'zz', 'mask2', 'index_t_p', '-v7.3');

mm = numel(im3d_t_p);

clear xs ys zs xx yy zz

% starts

n = 2000; %%%%%%%%%%%%%%%%%%%

for ii = 1:ceil(mm/n)

disp(ceil(mm/n) - ii)

if ii > 1
load(name1)
else
end

if ii < ceil(mm/n)

im3d_t = im3d_t_p(((ii-1)*n+1):ii*n);
index_t = index_t_p(((ii-1)*n+1):ii*n);
xs = xs_p(((ii-1)*n+1):ii*n);
ys = ys_p(((ii-1)*n+1):ii*n);
zs = zs_p(((ii-1)*n+1):ii*n);

else

im3d_t = im3d_t_p(((ii-1)*n+1):mm);
index_t = index_t_p(((ii-1)*n+1):mm);

end
xs=xs_p(((ii-1)*n+1):mm);
ys=ys_p(((ii-1)*n+1):mm);
zs=zs_p(((ii-1)*n+1):mm);
end

clear im3d_t_p

poolobj = gcp('nocreate'); % If no pool, do not create new one.
if isempty(poolobj)
poolsize = 0;
else
poolsize = poolobj.NumWorkers;
end
if poolsize~=n_cpu
    delete(poolobj)
pause(30)
    parpool(n_cpu)
else
end

pctRunOnAll javaaddpath java
progressStepSize=round(0.01*n);
pro_t=num2str(ii);
pro_t1=num2str(ceil(mm/n));
pro_t2=strcat(pro_t,' out of ', strcat('_',pro_t1,'_'));
ppm = ParforProgMon(pro_t2, numel(index_t), progressStepSize, 300, 80);
parfor i=1:numel(index_t)  % parfor
if size(im3d_t{i},3)>=re_a

    a1=im3d_t{i};

    [ratio1, std_v, R, ce2] = sphere_or_no_1(im3d_t{i}, point_n);    %
    point_n=number of random point

    t_size=size(im3d_t{i},3);

    s_1=min(size(a1));

    if numel(size(a1))==3&s_1<=re_a1&s_1>=re_a & ratio1>=soli_v1

        index_final{ii,i}=index_t(i);

        sphere_not{ii,i}=1;

        n_b{ii,i}=1;

        kk=bwperim(im3d_t{i});

        s_a{ii,i}=sum(kk(:));

        a1=im3d_t{i};

        d_a{ii,i}=sum(a1(:));

        ratio_f{ii,i}=ratio1;

    elseif numel(size(a1))==3&s_1>=re_a1&ratio1>=soli_v2    %0.75

        index_final{ii,i}=index_t(i);

        sphere_not{ii,i}=1;

        n_b{ii,i}=1;

        kk=bwperim(im3d_t{i});

        s_a{ii,i}=sum(kk(:));

        a1=im3d_t{i};

        d_a{ii,i}=sum(a1(:));

        ratio_f{ii,i}=ratio1;

    elseif numel(size(a1))==3&max(size(a1))>=re_a1&ratio1<soli_v2
\[ [\text{sphere} \_\text{not}, \text{n} \_\text{bubble}, \text{s} \_\text{area}, \text{d} \_\text{area}, \text{ratio}2] = \text{water} \_\text{s1}(\text{im3d} \_\text{t}[i], \text{im3d} \_\text{f}[i]) \]

\[
\text{index} \_\text{final}[[ii,i]] = \text{index} \_\text{t}(i); \\
\text{sphere} \_\text{not}[[ii,i]] = \text{sphere} \_\text{not} \_\text{t}; \\
\text{n} \_\text{b}[[ii,i]] = \text{n} \_\text{bubble}; \\
\text{s} \_\text{a}[[ii,i]] = \text{s} \_\text{area}; \\
\text{d} \_\text{a}[[ii,i]] = \text{d} \_\text{area}; \\
\text{ratio} \_\text{f}[[ii,i]] = \text{ratio}2; \\
\]

\[ \text{else} \]

\[
\text{index} \_\text{final}[[ii,i]] = \text{index} \_\text{t}(i); \\
\text{sphere} \_\text{not}[[ii,i]] = 0; \\
\text{n} \_\text{b}[[ii,i]] = 0; \\
\text{s} \_\text{a}[[ii,i]] = []; \\
\text{d} \_\text{a}[[ii,i]] = []; \\
\text{ratio} \_\text{f}[[ii,i]] = []; \\
\]

\[ \text{end} \]

\[ \text{else} \]

\[
\text{index} \_\text{final}[[ii,i]] = \text{index} \_\text{t}(i); \\
\text{sphere} \_\text{not}[[ii,i]] = 0; \\
\text{n} \_\text{b}[[ii,i]] = 0; \\
\text{s} \_\text{a}[[ii,i]] = []; \\
\text{d} \_\text{a}[[ii,i]] = []; \\
\text{ratio} \_\text{f}[[ii,i]] = []; \\
\]

\[ \text{end} \]

\[ \text{if} \ \text{mod}(i, \text{progressStepSize}) == 0 \]

\[
\text{ppm}.\text{increment}(); \\
\text{else} \\
\text{end} \]
end% parfor

ppm.delete();
end% ii

parfor i=1:numel(sphere_not)
    if sphere_not{i}==1
        index_f(i)=index_final{i};
    else
        index_f(i)=0;
    end
end

clear im3d_t
index_f1=index_f(find(index_f));
L = labelmatrix(CC2);
clear CC2
n_b1=n_b(find(index_f));
n_b1=cell2mat(n_b1);
d_a1=d_a(find(index_f));
d_a1=cell2mat(d_a1);
s_a1=s_a(find(index_f));
s_a1=cell2mat(s_a1);
ratio_f1=ratio_f(find(index_f));
ratio_f1=cell2mat(ratio_f1);
size(ratio_f1)
entrain_3d=ismember3d(L,index_f1,5);
clear L
r_f=(d_a1./s_a1)*3*n_reso*2;
disp(r_f)
size(r_f);

name_bubble=strcat(name,'_entrained air bubbles');
save(name_bubble,'entrain_3d')

fprintf('The radius %.4f in micro
',r_f*n_reso)

fprintf('The air content without processing %.4f in percent
',A_without)

fprintf('The air content larger than 1*1*1 %.4f in percent
',air_rm)

fprintf('The # of air content smaller than than 1*1*1 %.4f in percent
',n_small)

fprintf('The air content with re_ta removal %.4f in percent
',A_without)

fprintf('The entrained air content %.4f in percent
',A_void(entrain_3d,r))

C_show{9}=entrain_3d(:,:,n_show);

fprintf('The total number of entrained air %.0f
',sum(n_b1))

fprintf('The total entrained air volume %.4f
',sum(d_a1))

fprintf('The total specific surface of entrained air %.4f in micro(-1)
',sum(s_a1)/(sum(d_a1)*n_reso*0.001))

fprintf('The final entrained air content %.4f in percent
',100*sum(d_a1)/(pi*r*r*size(entrain_3d,3)))

C_show{4}=C_show{9};

for i=1:numel(n_show)
    figure('visible','on')
    h=subplot(2,2,1);
    po=get(h,'pos');
    for j=1:4
        h=subplot(2,2,j);
        pol(1)=po(1)+mod(1+j,2)*0.3;
        pol(2)=po(2)-(ceil(j/2)-1)*0.35;
        pol(3)=po(3);
pol(4)=po(4);
C_show_1=C_show{j};
imshow(C_show_1(:,:,i))

    if j==1
        hold on
        b_cir=bwperim(mask);
        [x,y]=find(b_cir);
        plot(y,x,'b.');
        hold off
    else
    end

    set(h,'pos',pol);
end

namef1=strcat(dir_name,'/','new_fold','/','name','_','num2str(n_show(i))','th slice');

    saveas(gcf,namef1,'fig')
end

name1=strcat(name,'_coordinates of air bubbles','.mat');
save(name1)

name2=strcat(name,'_final_calculation','_',num2str(soli_v1),'_',num2str(soli_v2)
        ,'_',num2str(re_a),'_',num2str(re_a1),'.mat')

final{1}=r_f;% diameter in micro
final{2}=A_without;
final{3}=[air_rm,n_small]; % air content after removeal of 3*3*3
final{4}=A_void(entrain_3d,r);
final{5}=100*sum(d_a1)/(pi*r*r*size(entrain_3d,3));
final{6}=sum(n_b1);
final{7}=sum(s_al)/(sum(d_al)*n_reso*0.001);

final{8}=[n_slice,r]

save(name2,'final');

toc
Appendix D - Code for separation of connected air voids

This code aims to separate connected air voids and is included as a subroutine in the code listed in Appendix C. The code has been tested using MATLAB® version R2016a(9.0.0.341360).

The input includes: a 3D connected object.

The output includes: size information of separated objects included in the 3D connected object.
% this is function for extracting air bubble from clustered bubbles/voids

function [sphere_not_t,n_bubble,s_area,d_area,ratio2]=water_s(im3d,soli_v1,soli_v2,ratio_origin,re_a,re_a1,point_n);
    warning('off','all');

D=bwdist(~im3d,'Quasi-Euclidean');
D=-D;
L=watershed(D,26);
im3d(L == 0) = 0;
clear D;
clear L;

CC2_s=bwconncomp(im3d); %
    STATS=regionprops(CC2_s,'Area','BoundingBox','PixelList');
    clear CC2_s
    ar={STATS.BoundingBox};
    ar=cell2mat(ar');
    ar=ar(:,4:6);
    ar=min(ar');
    index_t=find(ar>=re_a);%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%%
    if numel(index_t)>0
        ar1=ar(index_t);
        bb_s={STATS.BoundingBox};
        bb_1s=bb_s(index_t);
        bb_2s=cell2mat(bb_1s');
        bb_2s=round(bb_2s);
        PLS={STATS.PixelList};
clear STATS bb_1s
PLls=PLs(index_t);
clear PLs
xss=bb_2s(:,1);
yss=bb_2s(:,2);
zss=bb_2s(:,3);
xxs=bb_2s(:,4);
yys=bb_2s(:,5);
zzs=bb_2s(:,6);
clear bb_1s bb_2s

n_im3d_index=0;
for i_s=1:numel(index_t);
   % clear im3d
   im3ds=zeros(xxs(i_s),yys(i_s),zzs(i_s));
   PL2s=cell2mat(PL1s(i_s));
   PL3s=[PL2s(:,1)-xss(i_s)+1,PL2s(:,2)-yss(i_s)+1,PL2s(:,3)-zss(i_s)+1];
   for j=1:size(PL3s,1)
      xyz=round(PL3s(j,:));
      im3ds(xyz(1),xyz(2),xyz(3))=1;
   end
   [x y z]=meshgrid(1:xxs(i_s),1:yys(i_s),1:zzs(i_s));
   xyz_t=[x(:),y(:),z(:)];
   ml=size(im3ds);

   if numel(ml)==3&size(im3ds,3)>=re_a&size(im3ds,1)>=re_a&size(im3ds,2)>=re_a
n_im3d_index=n_im3d_index+1;
im3ds_t{n_im3d_index}=im3ds;
area_s(n_im3d_index)=sum(im3ds(:));
h(n_im3d_index)=min(size(im3ds));
else
end
end

clear PL1s
if  n_im3d_index>0
    [a_v,n1_index]=sort(area_s,'descend');
else
end

if n_im3d_index>=1

    disp('Watershedded objects starts')

    for ii3=1:min(n_im3d_index,25) %%%%%%%
        a=im3ds_t{n1_index(ii3)};
        [ratio1, std_v, R, ce2]=sphere_or_no_1(a, point_n);
        ratio1_s(ii3)=ratio1;
        std_v_s(ii3)=std_v;
        R_s(ii3)=R;
        ce2_s(ii3)=ce2;
        area1(ii3)=sum(a(:));
        %h=min(size(a));
        kk=bwperim(a);
        sp_area(ii3)=sum(kk(:));
end

h=h(n1_index);

% disp('Watershedded objects ends')
else

% disp('No sign that it is an entrapped bubble after watershedding')

ratio1_s=[ ];
sphere_not_t=0;
n_bubble=0;
s_area=[ ];
d_area=[ ];
ratio2=[ ];
end


n_00=0 ;
ti=min(numel(ratio1_s)-1,3);%%%%%
if n_im3d_index>1&(ratio1_s(1)>0.8|mean(ratio1_s(1:ti))>0.7)%%%

for ii_t=1:min(n_im3d_index,25)

ratio1=ratio1_s(ii_t);
R11=R_s(ii_t);
area2=area1(ii_t);
h1=h(ii_t);

if ratio1>soli_v1&h1>=re_a&h1<re_a1  % h1>=3

n_00=n_00+1;
index_g(n_00)=ii_t;
elseif  ratio1>(soli_v2-0.1)&h1>=re_a1

n_00=n_00+1;
index_g(n_00)=ii_t;
else

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end
end

if n_00>=1
    sphere_not_t=1;
    n_bubble=n_00;
    s_area=sp_area(index_g);
    d_area=area1(index_g);
    ratio2=ratio1_s(index_g);
else
    sphere_not_t=0;
    n_bubble=0;
    s_area=[];
    d_area=[];
    ratio2=[ratio_origin];
end

elseif n_im3d_index==1 & ratio1_s(1)>=0.65
    sphere_not_t=1;
    n_bubble=1;
    kk=bwperim(im3ds);
    s_area=[sum(kk(:))];
    d_area=[sum(im3ds(:))];
    ratio2=[ratio1_s(1)];
else
    sphere_not_t=0;
    n_bubble=0;
s_area=[];

d_area=[];

ratio2=[ratio1_s(1)];

end

else

sphere_not_t=0;

n_bubble=0;

s_area=[];

d_area=[];

ratio2=[ratio_origin];

end
Appendix E - Code for assessment of 3D spherical solidity

This code aims to assess the 3D spherical solidity of a 3D object and is included as a subroutine in the code listed in Appendix C. The code has been tested using MATLAB® version R2016a(9.0.0.341360).

The input includes: a 3D object.

The output includes: the 3D spherical solidity of the 3D object.
function [ratio1, std_v, R, ce2] = sphere_or_no(im3d, point1);

warning('off', 'all');

m = size(im3d);

x11 = max(m); x12 = min(m);

if x11 > 60 & x12 > 10
    im3d = resize(im3d, [round(size(im3d)*(50/x11))]);
else
end

if x11 > 60 & x12 > 10
    coef = 50/x11;
else
    coef = 1;
end

parfor i1 = 1:size(im3d, 3)
    bw_cir = bwperim(im3d(:, :, i1));
    [x, y] = find(bw_cir == 1);
    xypool{i1} = [x, y, ones(numel(x), 1)*i1];
end

xypool1 = cell2mat(xypool');

clear xypool

if 0 <= size(xypool1, 1) & size(xypool1, 1) < 5
    [ce2, R] = minboundsphere(xypool1, im3d);
elseif size(xypool1, 1) >= 5
    parfor i2 = 1:20
        rand_n = randsample([1:1:size(xypool1, 1)], point1);
        point_xyz = xypool1(rand_n(:, :));
    end
end
\[ ce, radius \] = minboundsphere(point_xyz, im3d);

\[ V1(i2) = (4/3) \pi \cdot r^{3}; \]

\[ radius1(i2) = radius; \]

\[ c{i2} = ce; \]

end

clear V1
index_inf = find(radius1 < inf);

radius1 = radius1(index_inf);

% \[ \muhat, \sigmahat \] = normfit(radius1);

% index_m = find(radius1 < (\muhat + 2*\sigmahat) & radius1 > (\muhat - 2*\sigmahat));

% radius1 = radius1(index_m);

cel = cel(index_inf);

% cel = cel(index_m);

ce2 = cell2mat(cel');
clear cel

radius1 = radius1(find(radius1 < inf));

R = mean(radius1);

else
end

if 5 <= size(xypool1,1)

\[ [x, y, z] = \text{meshgrid([-R:R]);} \]

\[ V = \sqrt{x^2 + y^2 + z^2}; \]

\[ V(V \leq R) = 1; \ V(V > R) = 0; \]

ind = find(V);

\[ [x, y, z] = \text{ind2sub(size(V), ind);} \]

clear V
x = x + (mean(ce2(:,1)) - R - 1);
y = y + (mean(ce2(:,2)) - R - 1);
z = z + (mean(ce2(:,3)) - R - 1);
ind = find(im3d);
[x3d y3d z3d] = ind2sub(size(im3d), ind);
k0 = 0;
for i3 = 1: numel(x)
    if (x(i3) >= 0 & x(i3) <= max(x3d)) & (y(i3) > 0 & y(i3) <= max(y3d)) & (z(i3) > 0 & z(i3) <= max(z3d))
        k0 = k0 + 1;
        x1(k0) = x(i3);
        y1(k0) = y(i3);
        z1(k0) = z(i3);
    else
        end
    end

    if exist('x1', 'var') == 0
        x1 = x3d;
        y1 = y3d;
        z1 = z3d;
    end

n_sphere = zeros(max(1, round(max(x1))), max(1, round(max(y1))), max(1, round(max(z1))));
for i3 = 1: numel(x1)
    n_sphere(max(1, ceil(x1(i3))), max(1, ceil(y1(i3))), max(1, ceil(z1(i3)))) = 1;
end
m = size(n_sphere);
im3d1 = im3d(:, :, 1:m(3));
im3d1 = im3d1(1:m(1), :, :);
im3d1 = im3d1(:,1:m(2),:);
overlap_v = n_sphere & im3d1;
clear x1 y1 z1 im3d1;
if round(min(x3d))<m(1)
n_sphere = n_sphere(min(x3d):m(1),:,:);
else
end
if round(min(y3d))<m(2)
n_sphere = n_sphere(:,min(y3d):m(2),:);
else
end
if round(min(z3d))<m(3)
n_sphere = n_sphere(:,:,min(z3d):m(3));
else
end
% if exist('n_sphere','var')==0
% n_sphere=n_sphere;
% end
ratio1 = sum(overlap_v(:))/sum(n_sphere(:));
ratio2 = sum(im3d(:))/(pi*R^3*(4/3));
std_v = std(radius1)/mean(radius1);
R = R/coef;
ce2 = ce2/coef;
fprintf('SIZE=%5.4f 	 Standard error =%5.3f 	 Sodility=%5.3f 
', m(3), std_v, ratio1);
elseif 0<=size(xypool1,1)&size(xypool1,1)<5
[x,y,z]=meshgrid([-R:R]);
V=sqrt(x.^2+y.^2+z.^2);
V(V<=R)=1; V(V>R)=0;
ind = find(V);
[x y z] = ind2sub(size(V), ind);
clear V
x = x + (mean(ce2(:, 1)) - R - 1);
y = y + (mean(ce2(:, 2)) - R - 1);
z = z + (mean(ce2(:, 3)) - R - 1);
ind = find(im3d);
[x3d y3d z3d] = ind2sub(size(im3d), ind);
k0 = 0;
for i3 = 1:numel(x)
    if x(i3) >= 0 & x(i3) <= max(x3d) & y(i3) > 0 & y(i3) <= max(y3d) & z(i3) > 0 & z(i3) <= max(z3d)
        k0 = k0 + 1;
        x1(k0) = x(i3);
        y1(k0) = y(i3);
        z1(k0) = z(i3);
    else
    end
end
if exist('x1', 'var') == 0
    x1 = x3d;
    y1 = y3d;
    z1 = z3d;
end
n_sphere = zeros(round(max(x1)), round(max(y1)), round(max(z1))); 
for i3 = 1:numel(x1)
    n_sphere(ceil(x1(i3)), ceil(y1(i3)), ceil(z1(i3))) = 1;
end
m = size(n_sphere);
im3d1 = im3d(:,:,1:m(3));
im3d1 = im3d1(1:m(1),:,:);
im3d1 = im3d1(:,1:m(2),:);
overlap_v = n_sphere & im3d1;
clear x1 y1 z1 im3d1;
if round(min(x3d)) < m(1)
n_sphere = n_sphere(min(x3d):m(1),:,:);
else
end
if round(min(y3d)) < m(2)
n_sphere = n_sphere(:,min(y3d):m(2),:);
else
end
if round(min(z3d)) < m(3)
n_sphere = n_sphere(:,:,min(z3d):m(3));
else
end
% if exist('n_sphere','var')==0
% n_sphere = n_sphere;
% end
ratio1 = sum(overlap_v(:))/sum(n_sphere(:));

ratio2 = sum(im3d(:))/(pi*R^3*(4/3));

std_v = 0.05;
R = R/coef;
ce2 = ce2/coef;

fprintf('SIZE=%5.4f \t Standard error =%5.3f \t Sodility=%5.3f \n', m(3), std_v, ratio1);
else
ratio1 = 0;
std_v=1;

ce2=[0 0 0];

R=0.1;

disp('**********Too small to be considered**********')

end
Appendix F - Code for generation of simulated air voids

This code aims to generate simulated air voids with controlled 3D spherical solidity, and has been tested using MATLAB® version R2016a(9.0.0.341360).

The input includes: desired size and 3D spherical solidity of the simulated air voids.

The output includes: simulated air voids.
% This is the code for generating the simulated bubbles

n=10;
%ratio=1;
%R=4

n_cpu=8

poolobj = gcp('nocreate');

if isempty(poolobj)
    poolsize = 0;
else
    poolsize = poolobj.NumWorkers;
end

if poolsize~=n_cpu
    delete(poolobj)
    parpool(n_cpu)
else
end

if 1==2

figure1 = figure('PaperType','<custom>','PaperSize',[46.4036 66.1354],
                 'PaperOrientation','rotated');

% Create axes

axes1 = axes('Parent',figure1,'Projection','perspective',
              'PlotBoxAspectRatio',[1 1 1],
              'FontSize',16,
              'DataAspectRatio',[1.25 1.25 1],
              'CameraViewAngle',10.020904319919);

view(axes1,[-121.558255848365 13.2973352051746 10.020904319919]);

box(axes1,'on');

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grid(axes1,'on')
hold(axes1,'all')
end
for i=1:n^2
  % R=randsample([5:5],1);

  [x,y,z]=meshgrid([-R:R]);
  V=sqrt(x.^2+y.^2+z.^2);
  V(V<=R)=1; V(V>R)=0;

  %V0=zeros(randsample([2:n1],1),randsample([2:n2],1),randsample([2:n3],1));
  V0=zeros(size(V));
  size1=round(numel(find(V))*(ratio));
  m=size(V0);
  for j=1:100000000
    if sum(V0(:))>=size1;
      break
    else
      if j==1
        V0(randsample(find(V),1))=1;
      else
        end
      bw_cir=bwperim(V0);
      ind=find(bw_cir);
      ti=[m(1),m(2)]*[1,-1,1,-1,0,0;1,-1,0,0,1,-1];
      ti=[ti,-1,1];
      t1=repmat(ti,1,numel(ind));
      t_in=repmat(ind,1,8);
t_in2=reshape(t_in',1,numel(t_in));
t1=t1+t_in2;
t2=t1+m(1)*m(2);
t3=t1-m(1)*m(2);
ind1=[t1;t2;t3];
ind1=unique(ind1);
ind2=find(~V0);
ind2=ind2(find(ismember(ind2,find(V))));
ind3=ind2(find(ismember(ind2,ind1)));
V0(randsample(ind3,min(1,numel(ind3))))=1;%%%%%%%%%%%%%%%%%%%%%%
clear ind1
end
end
[ratio1,std_v,R1,ce2]=sphere_or_no_1(V0,point1);
ratio_f(i)=ratio1;
std_f(i)=std_v;
R_f(i)=R1;
if 1==2
subplot1=subplot(n,n,i);
view(subplot1,[-61.5 10]);
box(subplot1,'on');
grid(subplot1,'on');
hold(subplot1,'all');
axis(subplot1, 'vis3d')
set(gca,'Visible','off')
hold(subplot1,'all');
fv=isosurface(V0,0.5);%~~~~~~~~~~~~~~~~~~'verbose'
fvc=isocaps(V0,0.5);

    pl=patch(fv,...
    'FaceColor','red','EdgeColor','none');

    reducepatch(pl,1) %~~~~~~~~~~~~~~~,'verbose'

p2=patch(fvc,...
    'FaceColor','red','EdgeColor','none');

    reducepatch(p2,1) %~~~~~~~~~~~~~~~,'verbose'

    view(3), camlight, lighting gouraud

r1 = R1* ones(20, 20); % radius is 5
[th, phi] = meshgrid(linspace(0, 2*pi, 20), linspace(-pi, pi, 20));
[x,y,z] = sph2cart(th, phi, r1);
x = x + mean(ce2(:,1));  % center at 16 in x-direction
y = y + mean(ce2(:,2));  % center at 40 in y-direction
z = z + mean(ce2(:,3));   % center at 2 in z-direction

    surface(x,y,z,'FaceColor', 'none');

    % bwconncomp(V0)

end % 1==2

    clear V0

end

%figure;plot(ratio_f,'*')

fprintf('mean solidity=%5.3f \t std of solidity =%5.3f \n',mean(ratio_f),
std(ratio_f))

fprintf('mean ___std___=%5.3f \t std of ___std___=%5.3f \n',mean(std_f),
std(std_f))

fprintf('size=%5.3f \n',size1)

toc

catch
disp('error happens')
exit
end
Appendix G - Code for visualizing the effects of watershed parameters

This code aims to visualize the effect of different watershed parameters, and has been tested using MATLAB® version R2016a(9.0.0.341360).

The input includes: a 3D connected object.

The output includes: separated objects with their 3D spherical solidity stated.
% This code is for the visualization when fine tuning the parameters in the proposed procedure.
% options are available to decide whether to watershed an object and % the individual solidity value for each divided sub-objects;
clear
close all
reso=7.5;
re_a=10;  
re_al=30
n_cpu=3;
point_n=5;
uiopen(mfilename)
bulk_3d=gray2binary(gray_scale_3d_adjust,0.6798);%%%%%%%%%%%%
C_air=bulk_3d;
CC=bwconncomp(C_air);
clear C_air
    STATS=regionprops(CC, 'Area','BoundingBox','PixelList');
    ar=[STATS.Area];
    index_t=find(ar>re_a);%%%%%%%%%%%%%%%%%%%%%%%%%%%%
    ar1=ar(index_t);
    bb={STATS.BoundingBox};
    bb_1=bb(index_t);
    bb_2=cell2mat(bb_1');
    bb_2=round(bb_2);
    PL={STATS.PixelList};
clear STATS
    PL1=PL(index_t); clear PL
    xs=bb_2(:,1);
ys = bb_2(:, 2);
zs = bb_2(:, 3);
xx = bb_2(:, 4);
yy = bb_2(:, 5);
zz = bb_2(:, 6);
clear bb_1 bb_2
n_im3d_index = 0;
for i = 1:numel(index_t);
    % clear im3d
    im3d = zeros(xx(i), yy(i), zz(i));
    PL2 = cell2mat(PL1(i));
    PL3 = [PL2(:, 1) - xs(i) + 1, PL2(:, 2) - ys(i) + 1, PL2(:, 3) - zs(i) + 1];
    for j = 1:size(PL3, 1)
        xyz = round(PL3(j, :));
        im3d(xyz(1), xyz(2), xyz(3)) = 1;
    end
    [x y z] = meshgrid(1:(xx(i)), 1:(yy(i)), 1:(zz(i)));
    xyz_t = [x(:), y(:), z(:)];

    m1 = size(im3d);
    if numel(m1) == 3 & size(im3d, 3) >= re_a & size(im3d, 1) >= re_a & size(im3d, 2) >= re_a
        n_im3d_index = n_im3d_index + 1;
        im3d_t{n_im3d_index} = im3d;
    else
        n_im3d_index = n_im3d_index + 1;
        im3d_t{n_im3d_index} = [];
    end
clear PL1 CC2

for i=1:numel(im3d_t)
    im3d=im3d_t{i};
    s_tl=sum(im3d(:));
    x1=x(i); y1=y(i); z1=z(i); z2=zz(i); xw=xx(i); yw=yy(i); zw=zz(i);
    [x_c,y_c]=meshgrid([1:2:yw+1],[1:2:xw+1]);
    n1=numel(x_c); n2=numel(y_c);
    m=size(im3d);
    s_1=min(m);
    if numel(m)==3&s_1<re_a1
        [ratio1, std_v, R, ce2]=sphere_or_no_1(im3d, point_n);
    end

    if 1==2
        small_gray=small_3d(gray_scale_3d_adjust, [x1, y1, z1, xw+10, yw+10, zw+10]);
        small_gray1=small_3d(C_air, [x1, y1, z1, xw+10, yw+10, zw+10]);
        parfor i=1:size(small_gray,3)
            k1=small_gray(:,:,i);
            k2=small_gray1(:,:,i);
            k1(find(k2))=0;
            value_f{i}=k1;
        end
    end

    value_f1=cell2mat(value_f);
    value_f2=value_f1(:);
    value_f2=value_f2(find(value_f2));
clear value_f

mode(value_f2)

agg_value

air_value

end %1==2

figure1 =figure(1);

set(figure1,'Position',[10 280 700 700],'PaperSize',[50,50])

% Create axes

subplot1=subplot(1,2,1);

p=get(subplot1,'pos');

h=gca;

set(h,'Projection','perspective',...  
   'PlotBoxAspectRatio',[1 1 1],...  
   'FontSize',12,...  
   'DataAspectRatio',[1 1 1],...  
   'CameraViewAngle',10.020904319919);

grid(subplot1,'on');

fv=isosurface(im3d,0.7);%~~~~~~~~~~~~~~~~~~~'verbose'

fvc=isocaps(im3d,0.7);

hold(subplot1,'all');

p1=patch(fv,...  
   'FaceColor','green','EdgeColor','none');

reducepatch(p1,1) %~~~~~~~~~~~~~~~~~~~,'verbose'

p2=patch(fvc,...  
   'FaceColor','green','EdgeColor','none');

reducepatch(p2,1) %~~~~~~~~~~~~~~~~~~~,'verbose'

view(3), camlight, lighting gouraud
z_c=round((1/2)*size(im3d,3))*ones(size(x_c));
mesh(x_c,y_c,z_c,'FaceColor', 'none','EdgeColor','red')
set(get(gca,'title'),'Position',[0,0,2.5*zw]);
rl = R* ones(30, 30); % radius is 5
[th, phi] = meshgrid(linspace(0, 2*pi, 30), linspace(-pi, pi, 30));
[x,y,z] = sph2cart(th, phi, rl);
x = x + mean(ce2(:,1));  % center at 16 in x-direction
y = y + mean(ce2(:,2));  % center at 40 in y-direction
z = z + mean(ce2(:,3));   % center at 2 in z-direction
a=gray_scale_3d_adjust(:,:,round(z1+z2/2));
x_c=60;y_c=60;

a=imcrop(a,[max(x1-x_c,1),max(y1-y_c,1),min(xw+2*x_c,size(a,2)-max(x1-x_c,1)),min(yw+2*y_c,size(a,2)-max(y1-y_c,1))]);

subplot2=subplot(1,2,2);
p(1)=p(1)+0.35;
set(subplot2,'Position',p)
hold(subplot2,'all');

h=gca;

imshow(a)
x1=x_c;y1=y_c;
line([x1 x1],[y1 y1+yw],'Parent',h)
line([x1 x1+xw],[y1+yw y1+yw],'Parent',h)
line([x1+xw x1+xw],[y1 y1+yw],'Parent',h)
line([x1+xw],[y1 y1],'Parent',h)
%title('2D cross-section view')
hold off

prompt = '* Have a plot=1 ** watershed it=2 ** TEST_REAL=3 **other=ENTER
';
result = input(prompt);
n_plot=0;

if result==1
    n_plot=n_plot+1;
    plot_name=strcat(name,'_',num2str(n_plot));
    random_sphere_plot(im3d,plot_name,3,reso);
    pause
else
    end
if result==2;
    water_s(im3d,point_n)
    pause
else
    end
if result==3;
    test_real(im3d,point_n)
    pause
else
    end
end

close all
else
end
end