3D imaging and analysis of bouillon cubes using x-ray microtomography

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Aims

Unilever is a world-wide manufacturer of consumer goods. Although not always obvious, some of our products have complex and contradictory product properties. Bouillon cubes are a good example. These cubes (Figure 34) should be sufficiently strong to survive handling and transport but it must be easy to crumble them by hand.

Generally bouillon cubes contain sodium chloride (salt), fat, seasonings (including monosodium glutamate and flavoring).

These ingredients are mixed and compressed into a cubical shape. The mechanical behaviour, such as where cracks may occur or how many cracks are generated, strongly depends on the composition and the microstructure. Important microstructural parameters are the size and shape of the particles and the porosity of the matrix including the size and location of cracks.

In this study X-ray microtomography (µCT) was used for the non-invasive visualisation and measurement of the internal structure of bouillon cubes [1].

Method

Bouillon cubes contain salt particles which cause high absorption of the X-ray beam. Generally the attenuation depends on the atomic number of the components (low for fat and high for sodium chloride).

It requires experimentation to determine the optimal scanner settings. Higher voltage and electric current results in a larger penetration depth, which is required for the salt particles. But if it is set too high, then the fat matrix may become too bright. This is illustrated in Figure 35.

Figure 34 Bouillon Cube

Figure 35 Projection µCT image of a model bouillon cube acquired at X-ray power settings of 50kV/200µA (left) and 100kV/100µA (right).
The X-ray tube of the SkyScan 1172 system has a maximum peak voltage of 100 kV and maximum power of 10 Watt and in our case it is limited to 95 kV (for legislation issues).

To find optimal settings yielding the best quality images, a small set of comparative scans were carried out. Voltage output was set at 50 kV and 95 kV and also the effect of an aluminium filter (0.5 mm) was evaluated. Scan nr. 3 was carried out with full rotation, but it was reconstructed on both 180° and on 360°.

The size of the cubes is 14x14 mm and allows us to use a pixel size of 7.28 µm so that the whole cube still fits in the FOV. Frame averaging was set to 4.

The use of an aluminium filter increases the scan time considerably since the exposure time needs to be longer (from 1178 up to 3534 milliseconds causing total scan time to almost double from 2:20 hour to 4:30 hour). For reconstruction, we have used a smoothing factor of 4 (asymmetrical boxcar kernel).

Ring artefact correction was set to 20 and the beam hardening correction was set to 80%. Reconstructed cross-sectional images are compared in Figure 36.

<table>
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<th>Voltage/Current</th>
<th>Filter</th>
<th>Rotation</th>
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<tbody>
<tr>
<td>1</td>
<td>50 kV/196 µA</td>
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<tr>
<td>2</td>
<td>95 kV/104 µA</td>
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<td>Aluminium</td>
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Figure 36 Comparing different scan settings.
All images are scanned with a pixel size of 7.28 µm.
Horizontal cross sections of 2043*2043 pixels (14.9 mm²) on the left, and a zoomed-in area of 493*493 pixels (3.6 mm²) on the right.
The salt particles are clearly visible as dark objects in a light grey fatty matrix. At first sight the image quality at 95 kV/104 µA seems only marginally better than those obtained at 50 kV/196 µA. However, when zooming in, a better contrast is observed. The appliance of the aluminium filter of 0.5 mm improves the image quality significantly. Also a reconstruction of 360° further enhances the sharpness of the images.

A smaller sample was imaged to obtain more details and to further improve the contrast between the fatty matrix and air (pores and cracks). This was done by cutting a cylindrical piece (diameter 7.7 mm) from the bouillon cube (Figure 37). This smaller sample was imaged at X-ray power settings of 95 kV/196 µA, using an aluminium filter, a pixel size of 2.55 µm and 360° rotation (Figure 38).

For the determination of the size and shape parameters the original stack of reconstructed 2D µCT images was down sampled with a factor of 2 and cropped to 422 x 422 pixels (Figure 39a). The random noise of the µCT images was reduced using a 3D median filter with a voxel size of 3 pixels. For segmentation of the particles from the fatty matrix manual thresholding was used, resulting in a 3D binary image of the particles (Figure 39b). Particles touching each other were separated using a watershed transform of the Euclidean distance map of the inverted binary image of the particles (Figure 39d). The features in the binary image were labelled and measured (Figure 39e).

The size (volume), surface area, inertia and location of the particles were obtained using the 3D measure function of DIPlib [2]. The length, width and thickness of the particles were determined from the inertia of the particles which is calculated from the eigenvalues of the particle’s second order moments tensor [3]. This procedure was used because the length, width and depth calculated using the DIPlib measure function is not rotation invariant.
resulting in errors up to a factor of 3. The length is defined as the longest dimension and the thickness as the shortest dimension of the particle.

In addition to the size parameter the particle shape was analysed using the aspect ratio and the sphericity. The aspect ratios were calculated as both the ratio between the length and the thickness and the length and the width. The sphericity was calculated as the ratio of the surface area of a sphere having the same volume as the measured particle to the surface area of that particle \( (\psi = \pi^{1/3} (6V)^{2/3} / A) \), where \( \psi \), \( V \) and \( A \) are the sphericity, volume and surface area of one particle, respectively) [4]. The sphericity gives a measure how spherical or compact an object is. The sphericity is 1 for a true sphere and 0.81 for a cube. The size and shape parameters of some particles selected from bouillon cubes are presented in Figure 40.

For validation the measured length, breath and thickness measured by the image analysis procedure was compared to those obtained using manual measurement of the \( \mu \)CT images using the interactive 3D measurement tool in the Avizo [5] software.

The results were within 10% relative. Because salt particles are mainly cubical shaped the equivalent length of the particles was calculated as the edge of a cube with equal volume [6]. The volume fraction of the salt particles was calculated from the ratio between the total volume of salt particles and the total volume of salt and matrix. The mass fraction was calculated from the volume fraction by using a density of 2.1 g/cm\(^3\) and 1.5 g/cm\(^3\) for the fatty matrix.
### Results

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<th>manual analysis</th>
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Figure 40 Size and shape parameters measured for particles selected from bouillon cubes (comparison between manual and automatic measurement).
Conclusion
The salt particles are clearly visible in the µCT images as dark objects in a light grey fatty matrix. However sodium chloride cannot be distinguished from monosodium glutamate (MSG) by a difference in grey level. They can only be identified by a difference in morphology. Salt particles are commonly cubic shaped whereas MSG has a plate or needle structure (Figure 41).

µCT proved to be a very useful technique for the non-invasive 3D visualisation and quantitative analysis of the microstructure of bouillon cubes. The 3D particle size and shape of sodium chloride and monosodium glutamate crystals can be analysed from the 3D µCT images. These parameters are important factors that determine the behaviour of these systems.

References:

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3 Jahne, B (2004), Practical handbook on image processing for scientific applications, chapter 16, CRC Press
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5 Avizo from Visualization Sciences Group (VSG) http://www.vsg3d.com