A Study of Bubbles in Foods by X-Ray Microtomography and Image Analysis

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INTRODUCTION
Bubbles play a key role in many types of food products including bakery products, dairy foams, confectionary, breakfast cereals, snacks and ice cream [1,2]. Bubbles create an aerated structure resulting in novel textures, enhanced taste perception and an attractive appearance. They are cheap ingredients and will add no calories. Soft foams such as whipped cream, aerated desserts and mousses contain separate gas bubbles in medium viscosity liquids or semi solids. However many food products are like sponges, in which nearly all bubbles are interconnected to give a porous network with a continuous gas phase. Examples are bread, cake and biscuits.

Bubbles can be formed by a large variety of methods which may be specific to one or more food types, e.g. whipping, mixing, fermentation, chemical agents, freeze drying, puffing, extrusion, frying and gas injection. The microstructure will not only influence sensory properties but also the stability, shelf-life and preparation time of the products.

A detailed understanding is needed of the relation between microstructure, and product properties. This paper describes a study that uses X-ray micro-computed tomography (µCT) for the characterisation and visualisation of the 3D microstructure of aerated desserts, ice cream, chocolate bars, dried vegetables, puffed rice and bread [3-6]. 3D image analysis (IA) methods were used to obtain quantitative information about the cellular matrix.

RESULTS
Aerated Dairy Products
Dairy based products, such as whipped cream, dessert toppings and mousses, are manufactured in the form of an emulsion and subsequently aerated to a foam. With these products, the protein present provides emulsion stability while the emulsifiers promote fat crystal agglomeration which forms a matrix. This matrix provides structure and firmness to the foam. The product formulation and processing conditions directly impact the finished product properties (mouthfeel, texture, flavour delivery and appearance) and shelf stability.

The products should be able to stand up to transportation over long distances – and even altitudes. The amount of gas and the bubble size distribution of these products have to be followed in time. Representative µCT images of an aerated dairy sample during storage for 2 months are shown in Figure 2. The gas bubbles are clearly visible within the fat/protein/water matrix by their high grey value (low absorption coefficient).

For image analysis and 3D visualisation binary images were made by using thresholding (iso-data method). These images were generated after noise reduction using a median filter. The volume fraction of air bubbles was...
determined from the total stack of binary images by dividing the number of pixels identified as air bubbles by the number of pixels inside the sample holder. The size of the gas bubbles was measured using a 3D measurement function. Before measuring, the bubbles were separated in 3D by using a watershed transform of the Euclidean distance map of the inverted binary image (Figure 3). Bubbles touching the upper and lower edge were removed.

The results of the analysis of the images shown in Figure 2 are presented in Table 1. The gas bubbles were not stable during storage. The number of air bubbles per volume was reduced and larger air bubbles were formed in time. This can be due to coalescence. Also the total volume of gas bubbles decreased in time. The foam collapse will result in a negative consumer appreciation.

Ice Cream
A popular complex dairy product is ice cream, made by freezing and aeration of a pasteurised mixture of ingredients including milk, fat, sugars, emulsifiers, stabilisers, flavouring compounds and water [7]. Ice cream contains about 20-50% air. The sizes of ice crystals and air bubbles is critical to the ice cream’s quality and sensory attributes. Large crystals lead to a coarse, grainy and icy texture. Whereas large bubbles will decrease the smoothness and creaminess. The size of ice crystals and air bubbles have to remain below the threshold of perception. Ice cream is inherently unstable. If the temperature increases during shipping, storage at the grocery store, or even on exit from the consumer’s freezer, the small ice crystals can melt and recrystallise into larger structures with larger interconnected air bubbles. Also pressure fluctuation leads to coalescence of air bubbles.

Small pieces of ice cream (~5×5×5 mm³) were fixed on the cooling stage by using cryogenic glue. The sample handling was done on dry ice (CO₂). Representative µCT images of cross sections of a fresh ice cream sample before and after temperature abuse (cycling between -20 and -10°C) are shown in Figure 4. The air bubbles are clearly visible as light objects in the grey matrix. The boundaries of the ice crystals are faintly visible by dark grey lines. After temperature larger ice crystals and larger air bubbles are formed. The mean bubble size (D_{4,3}) increases from about 200 to 300 μm and the mean ice crystal size increases from 30 to 100 μm.

Water Ice
An ‘ice lolly’ is a frozen sugar syrup with flavour and colour. It can be made from real fruit juice and contains considerably less air than ice cream. Water ice melts very rapidly. During imaging a piece of water ice was surrounded by polystyrene foam as isolation and covered with a piece of dry ice. A µCT image of a piece of water ice in Figure 5 reveals the distribution of air bubbles and crystals. Only a few air bubbles are present in a matrix of large crystals surrounded by sugar. The ice crystals are clearly visible by their dark boundaries.
Bakery Products
Crispness is an important quality characteristic of dry porous cereal materials [8] such as crackers, cookies and bread crust. It is influenced by the morphology of the cellular matrix. The size, shape, connectivity and anisotropy of air bubbles and their cell walls are seen as critical factors influencing the moisture transport within the cellular structure and therefore having an impact on the crispness. The proving time of dough influences the cell size and porosity of the crumb of the final product (Figure 6) [9]. The proving time influences also the thickness of the total crust and the solid top layer of the crust of bread (Figure 7) [10].

Cake is a soft cellular food product with mainly open cells within a solid matrix of carbohydrate, protein, fat and water (Figure 8). Due to their less dense structure open foams show a tendency to fill with the fluid that surrounds it [21]. The micro-CT images show areas in the solid matrix with a low and higher X-ray absorption (low and high grey level). This may be attributed to areas high in water (higher absorption) and high in fat (lower absorption).

Dried Vegetables
Drying is one of the oldest preservation methods for foods. Dried vegetables and fruits are important ingredients in instant soups and meals. These products are rehydrated before final use. Changes in macro- and microstructure are very important factors for the rehydration properties and textural quality of these dried materials. Fast rehydration is needed without compromising final sensorial and nutritional quality. Freeze drying is based on the dehydration by sublimation of a frozen product. It results in a very open structure with large cavities promoting fast rehydration (Figures 9 and 10). These cavities are not the plant cells but are created by the ice crystals. The size of these cavities is therefore mainly influenced by ice crystallisation behaviour or the cooling rate [11].

With air drying the structure shrinks and collapses during drying. A very dense structure is created which will rehydrate very slowly (Figures 9 and, 10). The shrinkage of air dried carrots determined by IA of µCT images was 94% compared to 3% for freeze drying (Figure 10). After rehydration at 90°C for 5 min a shrinkage of 81% and 12% was found, respectively. Water gives a higher X-ray absorption than the dry material by which the penetration of water can be followed. This is validated by MRI in Figure 11.

Rice
Quick-cooking or instant rice can be made by controlled dry heating of rice in air resulting in partially gelatinised starch [12-14]. As a starting material extruded rice can be used. Figure 12 shows the influence of the temperature on the microstructure of the rice kernel. Increasing temperature results in a more homogeneous sponge-like structure with thinner walls. The expansion factors (1.7, 2.3 and 3.4) and porosity (41%, 57% and 71%) increased with the temperature (160°C, 180°C and 240°C, respectively). A more porous structure

Figure 5: µCT image of horizontal (left: 6.2x4.8 mm², right: 1.4x1.4 mm²) cross-sections of water ice (1 pixel = 2.6 μm).

Figure 6: µCT images of horizontal cross-sections (top: 15.4x15.4 mm²) of cracker samples produced using different proving times (10-130 min) with porosity and pore size distribution.

Figure 7: µCT images of the crust of crispy rolls obtained at increasing proving times (vertical cross sections of 2.3 x 4.3 mm² and 3D surface rendering with a box size of 1.8x1.8x2 mm³, 1 pixel = 4.6 μm).
resulted in a better water uptake but in a negative mouthfeel.

Expansion can also be obtained using extrusion. The cellular architecture of extruded cereal based foods can be elucidated using micro-CT and related to macrostructural properties and strength [22].

Chocolate bars
Aerated chocolate based confectionery products are a favourite treat for many people. Bubbles influence the creaminess and brittleness or crispness of these products. Aerated chocolate is also perceived to be lighter in terms of calories. The mechanisms of bubble formation and behaviour in aerated chocolate have been studied by several authors using µCT [3,15].

This paper shows examples for micro- and macro-aerated chocolate bars (Figure 13). The porous structure was surrounded with a dense chocolate coating. The results of the 3D image analysis of the bubble size, porosity and layer thickness are presented in Table 2. Sample B contained more air, larger bubbles and a thicker coating layer than sample A.

CONCLUSIONS
X-ray microtomography has proved to be a very useful technique for the 3D visualisation and quantitative analysis of bubbles in food products. µCT can probe the microstructure of samples non-invasively up to a few millimetres across with an axial and lateral resolution down to a few micrometres.

The 3D capabilities, the good contrast between air and solid and the minimum sample handling, provides an advantage over the current imaging methodologies. Techniques such as scanning electron microscopy (SEM) can only image a few bubbles in a single 2D scan and large bubbles will be partly outside the field of view [16]. 3D image analysis provides quantitative information such as volume fraction, size, shape and anisotropy of bubbles in soft foams such as aerated dairy products.
and pore size, connectivity, wall thickness of solid products like ice cream, chocolate, dried vegetables, bread and rice. The obtained quantitative information can be used as input for simulation models (e.g. for bubble growth or moisture transport) [6, 9]. In combination with other techniques (e.g. MRI) it will generate fundamental knowledge about the microstructure and its relation to product properties.

REFERENCES

Figure 12: Influence of the temperature on heat treated extruded rice. Vertical µCT images (top) with 3D surface rendering (box = 4.5x3.1x9.9 mm³).

Figure 13: µCT images of two aerated chocolate bars, A and B. 1: Surface rendering. 2: Cross-section at low magnification (11.2x16.4 mm², 1 pixel = 18.2 µm). 3: Cross-section inside bar at high magnification (4.9x6.9 mm², 1 pixel = 6.8 µm).

Table 2: 3D structural parameters of two aerated chocolate bars.

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<tr>
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<th>Volume fraction of bubbles (%)</th>
<th>240°C</th>
<th>180°C</th>
<th>160°C</th>
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<tr>
<td>A</td>
<td>39</td>
<td>54</td>
<td></td>
<td></td>
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<tr>
<td>B</td>
<td>54</td>
<td>39</td>
<td></td>
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<tr>
<td>Bubble size, D_{4,3} = volume weighted mean diameter (mm)</td>
<td>8.2</td>
<td>4.2</td>
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<td>Thickness of coating layer (mm)</td>
<td>0.57</td>
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