X-ray micro-tomography to quantify frozen ice cream structure

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ABSTRACT

Abstract

X-ray micro-tomography (X-ray μCT) provided high resolution 3D images of frozen ice cream samples for use in studying the structure and particle size distribution of air bubbles, fat and ice. Ice cream samples were prepared in a laboratory continuous freezer that allowed a feasibility study of microstructure of frozen samples, which were produced in the continuous freezer then stored in a laboratory freezer at -30°C. X-ray microfocus computed tomography (μCT) (Skyscan 1172, Bruker microCT, Kontich, BE) with a temperature-controlled cooling stage was applied to stored frozen samples to investigate the 3-D microstructure of ice cream produced in a continuous process conditions. This provided a unique insight of the fat destabilization and structure network obtained after production and storage. Morphological analysis revealed microstructural properties of the ice cream samples. Significant differences between the phases were observed. In addition, the particle size distribution of ice crystals, fat and air bubbles was obtained. Results showed that microstructure analysis is a powerful tool for quantifying frozen ice cream structure in relation to process, storage and formula changes.

1. INTRODUCTION

Ice cream processing governs a unique microstructure of fat, proteins, ice and air of the frozen product. One important industrial problem is the destabilisation of the fat

emulsion as a result of process conditions and composition. During ice cream freezing and whipping some of the fat in the mix (originally very small <1µm) are transformed into fat aggregates giving a certain three dimensional microstructure; this phenomena is called fat destabilisation (Goff, Hartel 2013). Fat aggregates play a very important role on the ice cream structure.

Ice cream microstructure influences not only sensory properties but also the stability, shelf-life and preparation time of the products. A complete understanding is needed of the relation between microstructure- process, and product properties.

A number of works in literature have been published (Goff D. & Hartel (2013) on the microstructure on ice cream samples, in particular using electronic microscopy or cryo-scanning electron micrograph or transmission electron micrograph. Sample preparation freeze drying of the sample could modify the microstructure.

X-ray microfocus computed tomography µCT has been recently used successfully for the non-destructive characterisation and visualisation of the 3D microstructure of aerated desserts, fruits, bread and aerated products to obtain highly precise and quantitative information about the porous matrix (Cantre et al., 2014; Herremans et al., 2013a, 2013b; Van Dyck et al., 2014; Verboven et al., 2008)

Microstructure of frozen foods such as ice cream have hardly been investigated. A unique insight of interconnected network of microstructure components Such as fat, ice crystals and air would improve considerably our understanding of microstructural changes in relation to product properties. This paper presents the 3D microstructure of ice cream by using X-ray µCT.

2. MATERIALS AND METHODS

2.1 Ice cream

Ice cream samples were prepared with mix of ice cream under typical process conditions of mixing, pasteurization, homogenization and cooling. The resulting ice cream was allowed to mature at 5°C for 24 h. The ingredients of the mix are: milk, dry milk, milk fat, sweetening agents, stabiliser and emulsifier. The mix density was

about 1130 kg m^{-3} with an initial freezing point of -2.5°C, a brix of 26° and containing 12% fat.

A continuous freezer (WCB® Model MF 50) was used to freeze and to incorporate air in the ice cream samples, as shown in Fig. 1.

The ice cream mix was stored in a refrigerated storage tank at 5 °C. A volumetric piston pump was used to supply into the freezer. The ice cream frozen flowed down through the outlet pipe of the freezer. Temperature of the product at the outlet was measured by a Pt100 probe.

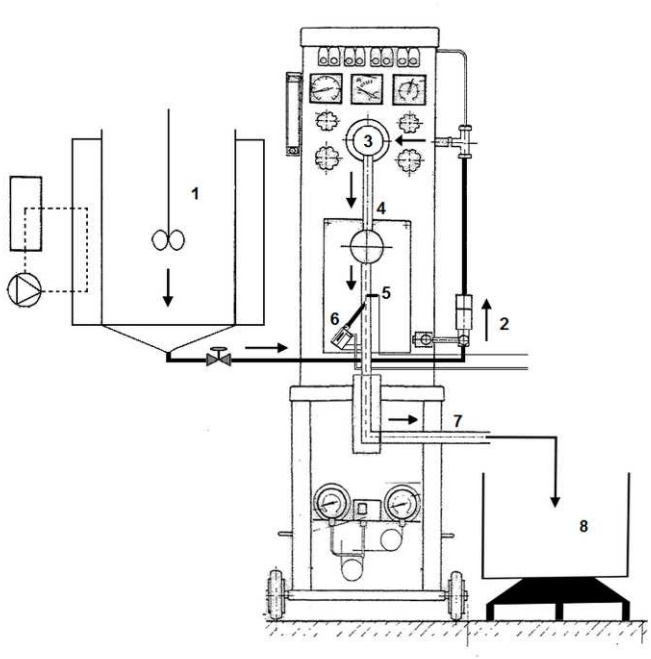


Figure. 1. Schematic representation of the experimental platform. 1. Refrigerated storage tank (200 litres capacity). 2. Volumetric piston pump. 3. Freezer. 4. Outlet pipe. 5. Pt100 probe inserted into the outlet pipe.

Process parameters in the freezer were adjusted: variable capacity from 25 to 75 kg h^{-1} . Ice cream samples with 75% overrun (volume of gas/ volume of mix) were prepared at a dasher speed 1000 rpm.

Samples of 500ml pins were obtained from the ice cream formula (fat content or 12%).

2.1 X-ray microtomography

X-ray μCT (Skyscan 1172, Bruker microCT, Kontich, BE) with a temperature-controlled cooling stage at -20°C was used to scan stored frozen samples and investigate the changes in the 3-D microstructure of ice cream samples. X-ray shadow projections were obtained at a pixel size of 5 μm with a power setting of 60 kV and 167 μA. The ice cream were imaged in a plastic cylindrical sample holder with an inner diameter of 5 mm. Ice cream sample and holder were isolated with 25 mm diameter polystyrene cylinder prior to scanning (Figure 2).

Shadow projection from each angular views was captured on a 2000 × 1048 pixels CCD X-ray camera and averaged from 3 frames as the object rotates on a high precision stage with 0.4° rotation step up to a rotation angle of 180°.

Cross section images (slices) were obtained using NRecon v.1.6.9 (Bruker microCT, Kontich, BE) tomography reconstruction software. Contrast of the images were enhanced using beam hardening correction, ring artefact reduction and smoothing values of 35%, 8, and 2 respectively. To standardize the greyscale range of the 8-bit bitmap output images, the linear attenuation coefficient (dynamic range) was set to 0 and 0.061.



Figure 2. The cooling stage of the Skyscan 1172 system.

2.2 Image processing

A volume size of 1 mm^3 (1 mm × 1 mm × 1 mm) with a resolution of 5 µm/pixel was virtually cropped and segmented using histogram and watershed segmentation operations to identify the air, fat and ice crystals. The segmented structure were then subjected to 3D analysis to obtain quantitative microstructural parameters. Image processing procedures were implemented in CTAn 1.13 (Bruker microCT, Kontich, BE) and Avizo Fire 8.1 (FEI, Oregon, USA). For 3-D visualisation, volume and surface rendering in Avizo Fire 8.1 (FEI, Oregon, USA) was used.

3. RESULTS AND DISCUSSION

A typical X ray µCT reconstructed slices obtained at a setting of 60 kV and 167 µA is presented in Figure 3.

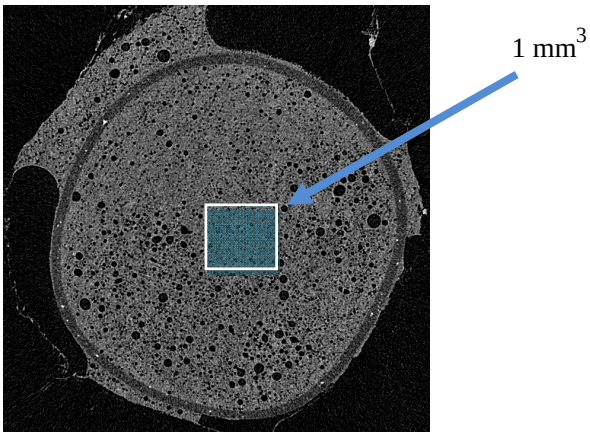
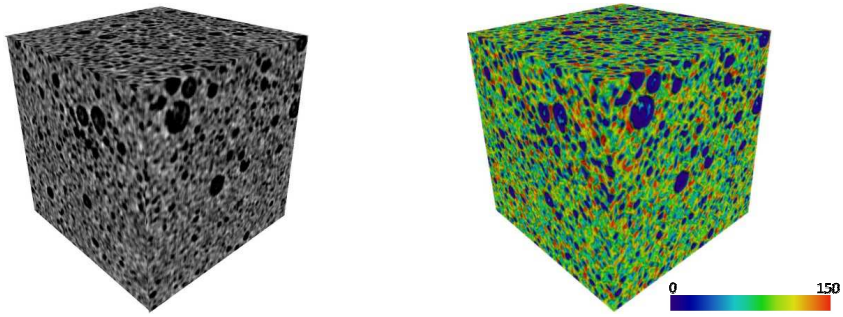


Figure 3. Typical X ray µCT reconstructed slices obtained at 60kV and 167µA.

A 3D reconstructed cube µof the ice cream sample in greyscale and its corresponding volume rendered image is shown in Figure 4. The gas bubbles are clearly visible because of their low grey value (dark regions) while the fat and ice matrix have medium and high grey values, respectively .

Histogram and watershed segmentation method was applied to separate the air bubbles, ice crystals, and fat globules in the ice cream mix. The resulting segmented structures from µCT images is presented in Figure 5.



a) b)

Figure 4. Reconstructed volume of 1 mm^3 (1 mm × 1 mm × 1 mm) in a) grey level image and b) volume rendered image showing colours based on pixel intensity.



a) air bubbles b)ice crystals c)fat globules

Figure 5. Segmented structures obtained after thresholding and watershed segmentation of the sample. Air bubbles in figure 5 a) showed an average size of 12 µm with a standard deviation of 10 µm and a sphericity of 0.83. The average size of the ice crystal is 24 with a standard deviation of 11 µm and a sphericity of 0.53. The average size of the fat crystal is 16 µm with a standard deviation of 13 µm and a sphericity of 0.71. A good agreement between experimental vs literature result are presented in table 1 .

Table 1. Comparison between experimental vs literature results .

Structural attributes	Present work	Literature Warren W. M., Hartel 2014
Ice Crystals	24µm ± 11	26-67µm
Air bubbles	12µm ± 10	17.1-39.5 µm
Fat Agglomerates	16µm ± 13	20-100µm

4. CONCLUSION

We can conclude that the use of X-ray µCT with a temperature-controlled cooling stage at -20°C is an adapted method to observe and to quantify microstructure of frozen samples. A good agreement between our method and literature results confirms that tomography will be a very powerful tool to investigate the changes in the 3-D microstructure of ice cream samples during process, storage and formula changes.

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