Benchtop and synchrotron X-ray micro-tomography to determine the cellular structure of cereal food foams

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Aims

The cellular structure of a cereal food is an essential feature that involves sensory aspects (appearance, texture) and carries information on the processing and composition of the recipe. Due to the interest of the computed X-ray micro-tomography technique and the information it provides, several results have been published on the cellular structure of cereal products, bread, biscuits and extruded cereals, either by laboratory devices[1,2,3,4,5,6] or synchrotron sources[7,8,9]. Besides the differences due to the composition of the products and their processing, the differences between µCT equipments and analysis procedures make the comparison of the results difficult, even for a property as essential as porosity, or density[10]. Therefore, there is a need to compare results obtained for same products with different equipments, and more precisely to compare the advantages and limitations of X-ray tube and synchrotron µCT in characterizing cellular cereal food. In this purpose, we have used two different µCT systems to study two types of cereals foods from commercial origin, exhibiting different cellular structure, at bare eye: an extruded breakfast cereal and a short dough biscuit. The results presented here have been published[11].

Method

Two different types of cereal products have been chosen to be studied by X-ray tube and synchrotron µCT: an extrudate (breakfast cereals) and a biscuit (fig 1).

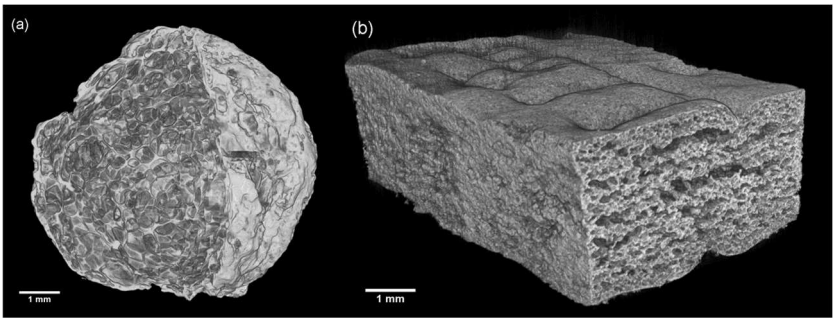

Fig. 1 *3D view of the outer shape and internal structure of the two samples from data acquired with the synchrotron µCT: (a) extrudate (breakfast cereals), (b) biscuit[11]*

Acquisitions were made by a compact desktop system Skyscan 1174 (Bruker µCT, Belgium) and at the European Synchrotron Radiation Facility (ESRF, beamline ID19, France) at different resolutions (voxel size of 6.5 µm, 7.5 µm, 16.2 µm and 25.8 µm). The acquisition parameters are reported in Table 1.

Table 1 *Experimental parameters for the µCT measurements*

Experimental parameters	synchrotron	X-ray tube	X-ray tube
X-ray source	17.6 keV	50 kV	50 kV
Voxel size (µm)	7.5	25.8 or 16.2	6.5
Resolution (# radiographs)	1200 to 5000	729	729
Acquisition time	2 to 15 min	75 min	75 min
Reconstructed volume (mm)			
Biscuit	13*7.7*14	10*22*24.5 (25.8 µm)	7*7*24
Extrudate	12.9*12.2*12.2	16*16*13.5 (16.2 µm)	8.5*8.5*6.5

The different steps of the image processing were: segmentation of grey level image, extraction of the volumes of interest in the images, and finally 3D analyses to calculate the density and the granulometry of cells and cell walls.

Results

3D images - Qualitative results

Fig. 1 depicts the 3D structure of the two samples and their reconstructed grey-level cross-sections. The internal structure of the material can clearly be seen in 3D, as well as the irregular aspect of the external surface, mainly in the case of the extrudate, which contrasts with its smooth appearance at bare eye.

Fig. 2 shows 2D cross-sections extracted from the middle of the 3D volume, in order to facilitate visual comparison. The two phases, the solid matrix, and the gaseous phase, have different values of attenuation coefficients; therefore the gaseous cells appear almost black, whereas solid cell walls appear in light grey, or white. At first sight, the biscuit appears denser than the extrudate, with thicker and more numerous walls. However, a zoom on the cross-section allows observing that the walls of the biscuit exhibit smaller internal pores (Fig. 2f) while no such pores are detected for the extrudate (Fig. 2c). The images also suggest that both phases, solid and gas, are co-continuous, or, said in other words, they are structured in an interconnected networks.

So only quantitative analysis brings the necessary information to understand the real differences in the microstructure of these two types of food products.

Size distributions

Cell walls

The distributions of the volume fraction of wall thickness are represented on Fig. 3. It appears clearly that the measurements depend on the resolution of the images, more than on the experimental device. For both products, when decreasing the voxel size, the distributions are shifted towards smaller values of the width. In the case of biscuit, for a voxel size of 25.8 µm, the distribution is characterized by a median value of 142 µm, a D10 of 83 µm and a D90 of 202 µm because walls thinner than 50 µm cannot be observed (Fig. 3b). Conversely, for a voxel size of 7.5 µm, more than half of the volume fraction exhibits a width lower than 50 µm, the median width is 49 µm and D10 and D90 equal 28 µm and 69 µm respectively. This fraction could not be detected with the 25.8 µm resolution. Even though the medians are really different, the spans of the distributions remain the same (0.8) meaning that the cellular structure has the same homogeneity but is quite coarser for the lowest resolution (voxel size of 25.8 µm). Same observations and rationale can be made for the extrudate. Like for the biscuit, measurements depend on the resolution and a shift in the distribution is observed toward lower thickness values when the voxel size was changed from 16.2 µm to 7.5 µm (Fig. 3a).

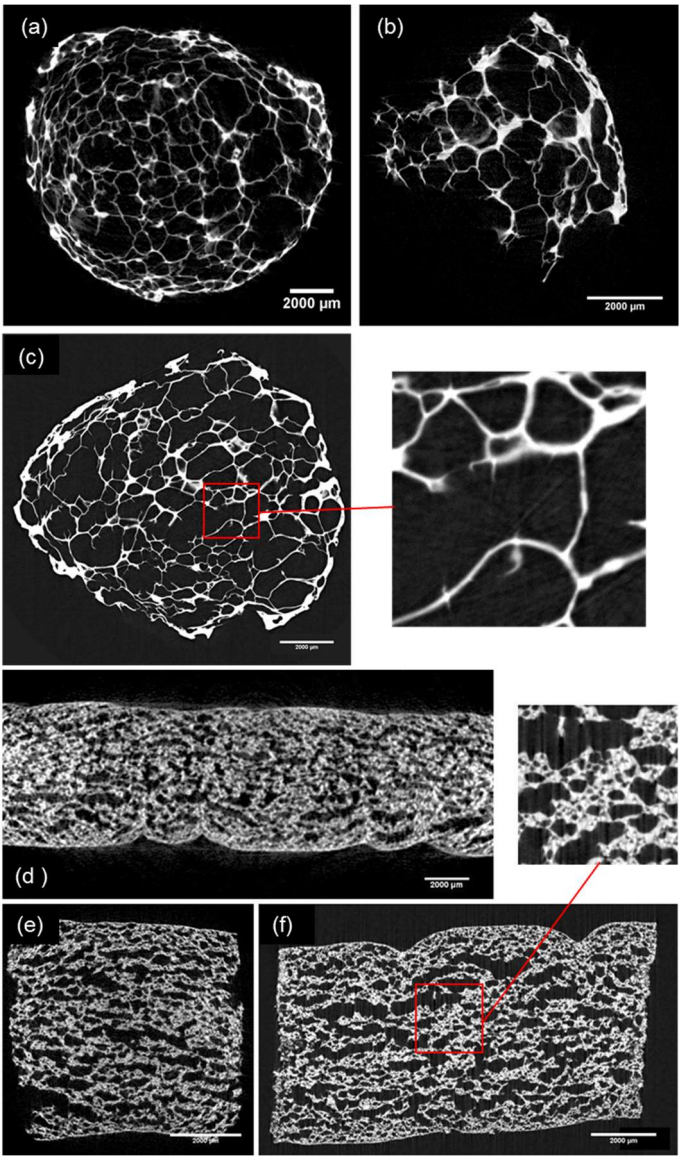

Fig. 2 *Cross sections (2D) of the samples at different resolutions and from the two tomographic systems. All scale bars represent 2 mm: extrudate by X-ray tube µCT at (a) 16.2 µm, (b) 6.5 µm, and (c) by synchrotron µCT at 7.5 µm with a zoom on the central area; and biscuit by X-ray tube µCT at (d) 25.8 µm, (e) 6.5 µm, and (f) by synchrotron µCT at 7.5 µm with a zoom on the central area*[11]

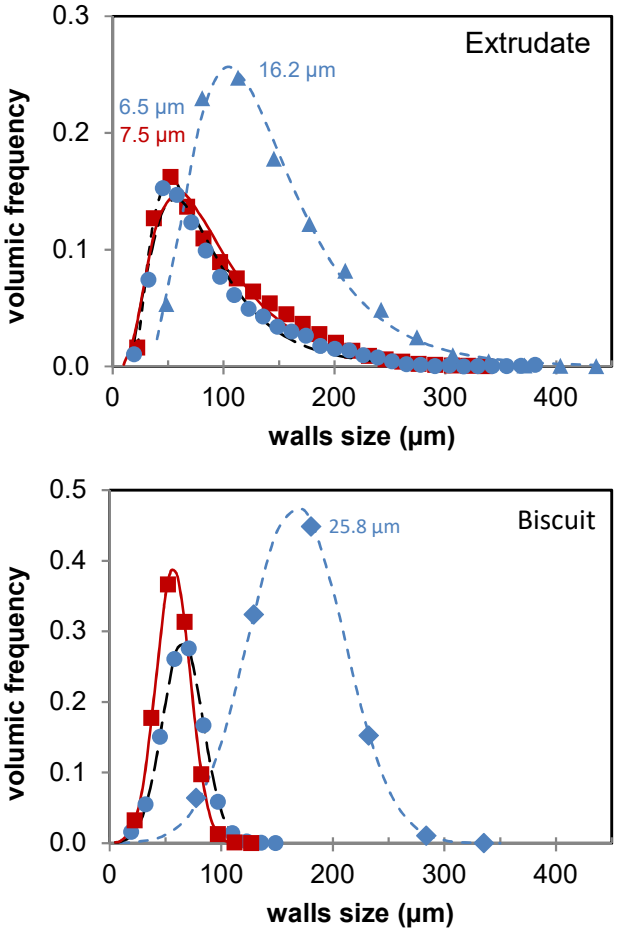

Fig. 3 *Distributions of simple volume fraction of wall thickness obtained by the analysis of images acquired from synchrotron (red) and X-ray tube (blue)*[11]

For both products, there is an agreement for the results obtained by the two µCT devices, when the resolution is close, since the distributions of the volume fraction of wall thickness did not exhibit significant differences. Although their volume fraction is very low (about 10^{-2} for the cell thinnest walls thinner than 13 µm), it should not be concluded that there are no walls having a thickness smaller than the space resolution (13 µm for the best resolution used in these experiments) contributing to the cellular structure.

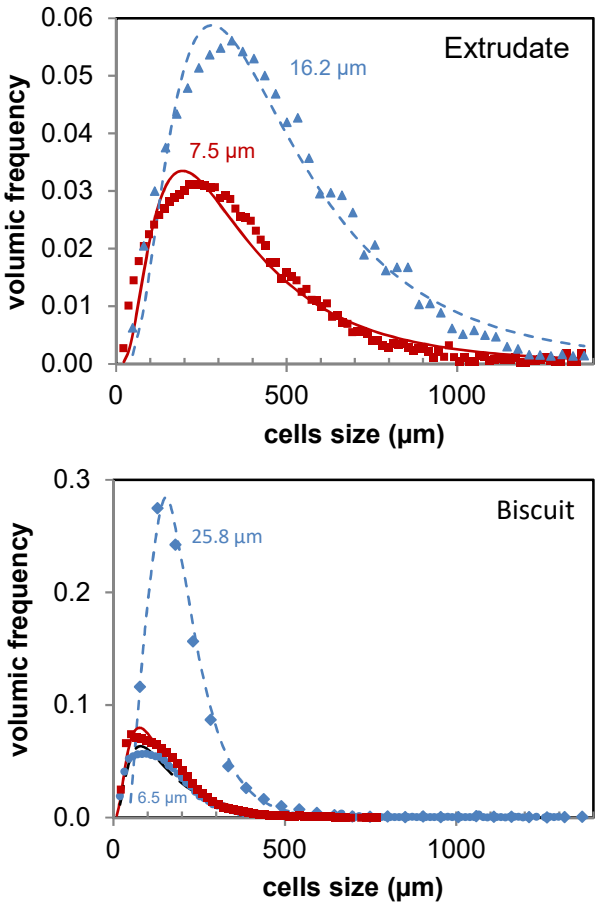

Fig. 4 *Distributions of simple volume fraction of cell width obtained by the analysis of images acquired from synchrotron (red) and X-ray tube (blue)*[11]

Cells

Like for the wall thickness distributions, the cell size distributions also depend on the resolution of the system, but they were much broader (Fig. 4).

Finally, these two cereal foods exhibited quite different cell structures. Compared with the extrudate, the biscuit had a lower porosity, with smaller cell sizes, but with thinner cell walls, resulting in a finer and more homogeneous cellular structure.

These differences may be attributed to the differences of processing kinetics. The cellular structure of the biscuit is created by the carbon dioxide released progressively by the leavening agents and relayed by the water vaporization as the temperature increases progressively in the oven, without exceeding 100°C inside the product at the end of the baking stage, during a few minutes before setting. This leads to an open-cell structure characterized by an interconnectivity index almost equal to 1. Conversely, the cellular structure of the extrudate results from a completely different process, which is not fully understood yet; this process involves the

application of high temperatures (above 100°C) and pressure. The sudden pressure drop encountered by the product at the die induces explosive sudden expansion and an instantaneous vaporization of water. Both phenomena create the cellular structure, which is afterwards stabilized upon immediate cooling and drying. Thus, cell walls, composed of a glassy blend of starch and protein are much larger (median value of 307 µm) than in the biscuit (median value of 125 µm). This comparison suggests that the quicker the expansion and setting processes, the more heterogeneous the cellular structure, as discussed by Mack et al.[12] who compared the time scales of the different mechanisms governing cereals based foam processing.

Conclusion

Using two different devices, this study has confirmed that X-ray micro-tomography is a powerful tool to describe the cellular structure of airy cereal foods. These experiments showed that, overall, similar images can be obtained from both devices, X-ray tube and synchrotron µCT, provided that they are operated at comparable pixel size. The processing of the images of two airy cereal foods well showed that, for both tools, a compromise between high resolution and describing large sample has to be found.

Images well showed that even though the food products had close values of porosity, 0.6 and 0.7 for biscuit and extruded breakfast cereal respectively, their cellular structures were very different. Beyond methodological issues, these differences could be clearly attributed to the differences of compositions and processes.

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