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Published in:
MC 2011 Kiel

Publication date:
2011

[Link back to DTU Orbit](#)

Citation (APA):

Jensen, L. H. S., Horn, A. F., Jacobsen, C., Nielsen, N. S., & Horsewell, A. (2011). Cryo-FIB SEM for Characterization of the Structure of Fish Oil Emulsions. In MC 2011 Kiel: Microscopy Conference 2011 (pp. M7-P633). DGE – German Society for Electron Microscopy.

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Cryo-FIB SEM for Characterization of the Structure of Fish Oil Emulsions

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Key words: cryo-SEM, cryo-FIB, FIB SEM, emulsions

The addition of fish oil to industrial food products is appealing both to the food industry and consumers for reasons such as health benefits and the extra commercial value. Fish oil is rich in long chain omega-3 fatty acids, which contain a large number of double bonds. This feature causes the omega-3 fatty acids to be highly susceptible to oxidation, thus their incorporation into foods is limited by the development of unpleasant off-flavours. Strategies for limiting oxidation which implies increasing the shelf-life of potential products are necessary for commercial production. One such strategy is to add the oil as an emulsion rather than as neat oil. Studies so far have indicated that emulsification of the fish oil changes the oxidative stability of the product but whether emulsification is an advantage seems to be dependent on the food matrix to which the emulsion is added [1, 2]. It is therefore of interest to look at the emulsions to assess what determines the oxidation. It has been proposed that oxidation is to some extent dependent on the structure of the emulsion; including oil droplet sizes, size distribution and the thickness of the interface between oil and water. This interface can be stabilized by food grade emulsifiers such as proteins and phospholipids from milk.

The main objective of this study is to characterize fish oil in water emulsions with respect to oil droplet size, size distribution, and ultimately to view the thickness, structure and morphology of the interface layers.

The emulsion fractures are random and impossible to control when using freeze-fracture cryo-SEM. We have previously shown that some types of emulsifiers tend to break along the interface layer, while others cause the fractures to be perpendicular to the interface layer [3]. To control the field of view more specifically and to ensure the access to the desired part of the sample, we propose now the use of cryo-FIB SEM. This method allows us to access the interface layers as needed, see figure 1.

Emulsions with high oil content, i.e. 70%, and relatively large oil droplets, i.e. μm range, have been frozen in slush nitrogen, fractured and ice has been sublimated from the surface in a Quorum Polar Prep 2000 Cryo Transfer System. Platinum has been sputtered onto the sample prior to sectioning. The sample is imaged in a Quanta 3D FEG (FEI) with a with ETD, 15 kV and WD 10 mm.

Figure 1 shows a sectioned oil droplet from an emulsion, which is emulsified with phospholipids from milk. On the micrograph it can be observed a contrast on the surface of the oil droplet that faces the interior below the original fracture plane, which has not been covered in platinum. The contrast is seen as a lighter line on the bottom side and the sliced oil droplet, visualised in figure 2. This could possibly be attributed to the phosphorous in the emulsifier.

1. Nielsen NS & Jacobsen C. *Int J Food Sci Tech*, 44, 1536-1546 (2009)
2. M. B. Let, C. Jacobsen and A. S. Meyer. *J Agric Food Chem*, 55, 7802-7809 (2007).
3. A. F. Horn, N. S. Nielsen, U. Andersen, L. H. Søgaaard, A. Horsewell and C. Jacobsen. Submitted to *European Journal of Lipid Science and Technology*.
4. The micrographs were recorded at CEN, DTU and CFIM, Panum, Copenhagen University.
5. This research was supported by the Danish Food Industry Agency under the Ministry of Food, Agriculture and Fisheries.

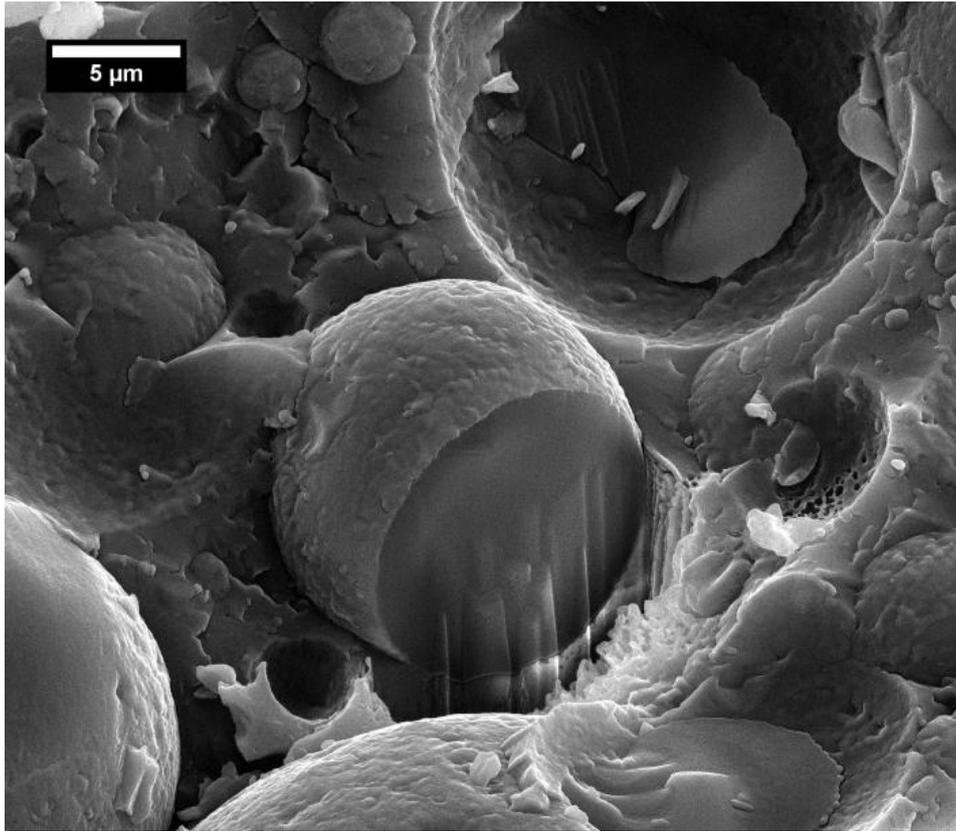


Figure 1. cryo-FIB sliced section of an oil droplet .

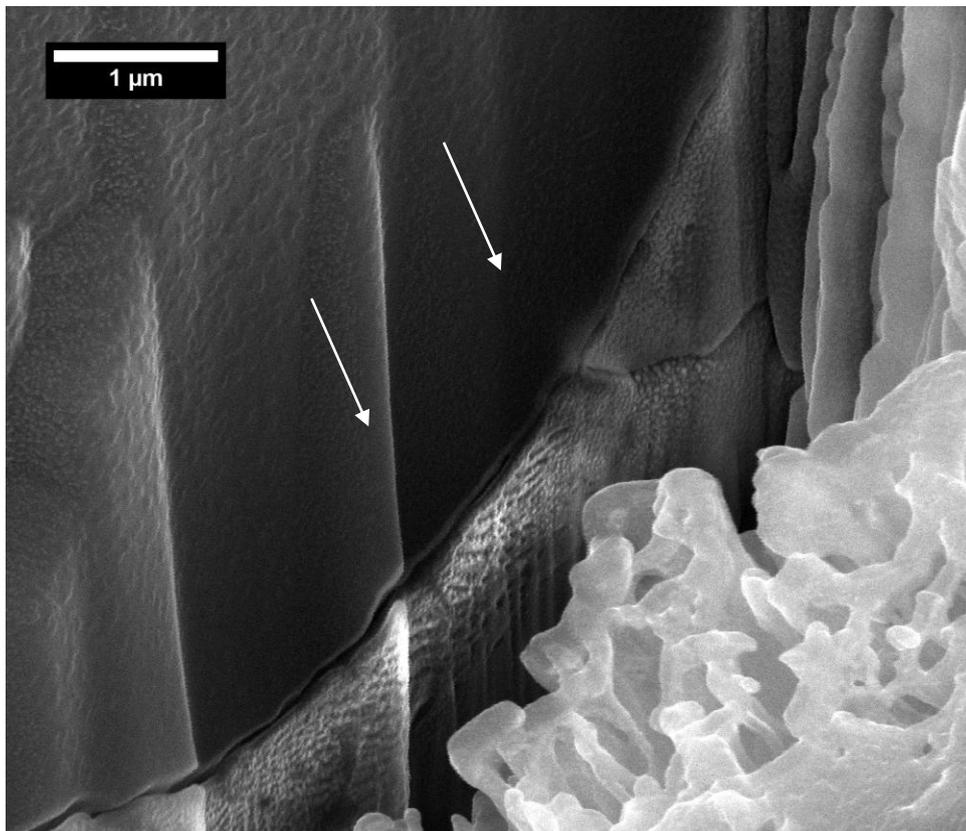


Figure 2. Closer view of the section. Contrast of the interface is visible in the micrograph (white arrows).