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A MOVING OPTICAL FIBRE TECHNIQUE FOR STRUCTURE ANALYSIS OF  
HETEROGENEOUS PRODUCTS: APPLICATION TO DIFFERENT FOODSTUFFS

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**Abstract**

The macro structure of many soft heterogeneous products can be studied by a moving optical fibre technique. The method is based upon an optical sensor that penetrates into the heterogeneous medium. The optical sensor discriminates between materials differing in refractive index or in scattering properties. Coupling of the signal of the optical sensor to its position reveals the spatial distribution of the materials in the product. Applications shown include bread, soft ice-cream, chocolate mousse and draining curd. The major advantages of the method are its speed and ease; moreover, the sample is hardly disturbed by the sensor. The limit of resolution is about 20  $\mu\text{m}$ .

**Introduction**

Heterogeneous foodstuffs, i.e. products that consist of two distinct materials such as bread or ice-cream, are on the daily menu. The spatial distribution of the two materials is often referred to in terms like bubble-size distribution or pore-size distribution. It is an important characteristic, not only with respect to the appearance of the product, but also in relation to other characteristics, for instance color, stability or permeability. The importance of the spatial distribution has led to the development of various determination methods. The common technique involves making cross-sections and photographing these; subsequently, the distances in the dispersed material are measured. This technique is hampered by the fact that making cross-sections may affect the materials in the cross-sectional area. Moreover, making cross-sections of unstable products, like foams (de Vries 1972), generates serious experimental difficulties. The interpretation of the micrographs is complicated by the finite depth of field of the picture. The interpretation is time consuming, when performed manually, or expensive, when image-analysis equipment is used. A disadvantage of the method may be destruction of the sample.

We present a new method that can offer additional information about the spatial distribution and may ultimately replace conventional techniques in the case of most soft products.

The method is based upon the application of an optical glass fibre sensor. The sensor, originally developed by Frijlink (1987), was used by Ronteltap (1989) to characterize beer foam. Since then, the method has been developed further, new applications have been found and part of its principle has been unraveled.

**Materials and Methods**

The method is based upon an optical sensor that penetrates into the heterogeneous medium and that distinguishes between materials. Coupling the position of the optical fibre sensor to its optical signal reveals the spatial distribution of the material. The optical fibre sensor is based upon detection of the amount of light "seen" by the tip of the fibre. The optical properties of the medium surrounding the fibre tip determine the amount of

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**Key Words:** Optical fibre, refraction, light scattering, structure, determination, heterogeneous media, pore size, bubble size.

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reflected light.

A beam of light ( $\lambda \approx 0.72 \mu\text{m}$ ) is sent into a glass fibre. The light is transmitted to the tip at the other end of the fibre. The tip is conically shaped, its outer end has either a hemispherical or a somewhat teardrop form, depending on which mechanism is being utilized. Its outer end has a diameter of approximately  $20 \mu\text{m}$ . Two distinct mechanisms cause reflection of light back into the fibre:

1) The refractive index of the quartz fibre tip ( $n = 1.4533371$ ) (Landolt-Börnstein 1962) is larger than the refractive index of the medium surrounding the tip. Total reflection begins at a certain critical angle  $\theta$  such that:

$$\sin \theta = \frac{n_{\text{medium}}}{n_{\text{quartz}}} \quad (1)$$

The angle, at which total reflection starts, depends on the refractive index of the medium surrounding the hemispherical tip and therefore the intensity of the reflected light also depends upon the refractive index of the medium. 2) The medium surrounding the somewhat teardrop shaped tip can cause considerable scattering of light. A part of the scattered light returns to the fibre tip. Light scattering is a complicated phenomenon (Orchard 1969) and its theory is beyond the scope of this article. The basic characteristic of this mechanism is that the intensity of the light seen by the tip is higher in more turbid media.

The returning light is transmitted back through the fibre. At the end, the light is splitted from the incoming light by means of a Y-splitter and projected onto a photo-electric cell. The voltage generated by the photo-electric cell depends on the intensity of the returned light. It should be noted that due to electronic circuitry used, the highest signal corresponds to the lowest light intensity. The optical fibre is held in a capillary, its tip protruding approximately 5 mm. As the capillary is moved forward into the medium (its total travel is 10 cm), the signal of the photo-electric cell is continuously sampled by means of a data-acquisition board in a computer. The position of the optical sensor is registered simultaneously in the computer. In this way series of chord lengths, i.e. a non-interrupted sequence of signals from the same material, is obtained.

Two different prototypes were built. The first was aimed at characterization of liquid foams. These highly unstable heterogenic media require standardized foam generation and fast penetration of the medium in combination with a very high resolution ( $0.1 \mu\text{m}$ ) in the direction of the moving probe. Bisperink et al. (1992) will discuss the technical details of this type. The second prototype was aimed at the determination of the spatial distribution in optical heterogeneous products. An important feature on this apparatus is the ability to measure in the vertical as well as in horizontal directions, so turning of the samples can be avoided. Statistics can not be applied to the results, as the products were heterogeneous and the shape of the pores c.q. bubbles was unknown. The

signals were collected by means of a MetraByte Dash-16 data-acquisition board in a Hewlett Packard ES/12 computer in the DMA (direct memory access) mode, and converted afterwards into a spatial distribution. The programming language used was Asyst 3.1. Both probe speed and sample rate were adjustable, the latter up to 20 kHz. The technique is thus suitable for studying rapid changes in the sample. Repeated measurements can be started within less than half a minute. The small dimensions of the optical sensor cause an additional advantage, namely that local variations can be traced. Prototype 2 is displayed in Figure 1.

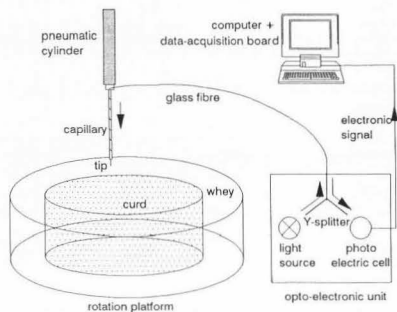


Figure 1. A schematic drawing of the apparatus (prototype 2) used to analyze the structure in heterogeneous foodstuffs, here set up for (draining) curd/whey mixtures. The pneumatic cylinder can also be mounted in horizontal direction. In that case the capillary is moved through tiny holes in the inner vessel.

The probe speed and the sample rate determine the axial resolution, i.e. in the direction of the movement of the fibre. The diameter of the fibre tip determines the lateral resolution of the method, i.e. perpendicular to the direction of the movement of the fibre. Isolated parts of one material inside the other material smaller than the fibre tip ( $\approx 20 \mu\text{m}$ ) will not be registered.

## Results

The technique was applied to various products, such as bread, ice-cream and chocolate mousse. The Figures 2, 3, and 4 display the patterns found. The structure of bread was clearly coarser than those

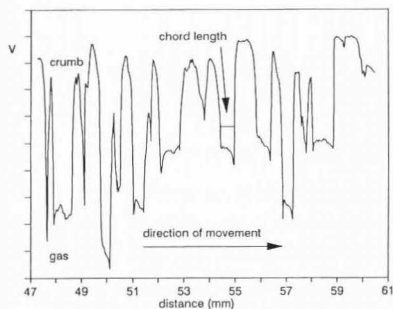


Figure 2. A part of the analogue signal of the optical fibre sensor penetrating into bread.

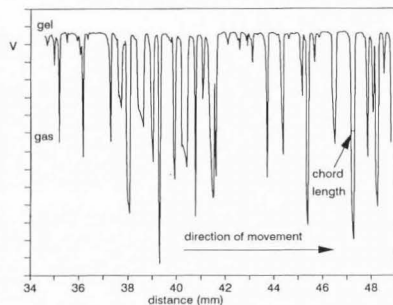


Figure 3. A part of the analogue signal of the optical fibre sensor penetrating into chocolate mousse.

of the other two products. Ice cream showed the smallest chord lengths and therefore average bubble-size. Actually, the displayed resolution of the signal of ice-cream is too low, but in order to ease comparing of the structure of the products, the graphs were shown at the same distance scale. The elastic properties of the medium may affect the signal. The penetration of the fibre in an elastic medium, e.g. bread, can cause sizeable mechanical indenting and stretching of the material, resulting in a gradually increasing and decreasing slope. However, as the fibre tip constitutes the most forward part of the probe, it may be expected that the net disturbance is only slight, and it may be taken into account when interpreting the signal. (The interpretation is based upon a screening upon negative and positive slopes.)

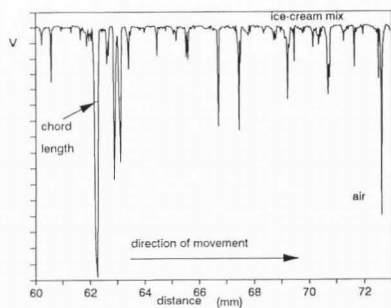


Figure 4. A part of the analogue signal of the optical fibre sensor penetrating into soft ice-cream.

In each of the graphs shown above, the two materials were primarily discriminated by this difference in refractive index. Examples of materials that can be discriminated by differences in scattering are scarcer. In cheesemaking, the separation of curd grains (basically contracted milk gel) and expelled liquid (whey) is an important step. The packing of curd grains before and after draining is schematically displayed in Figure 5.

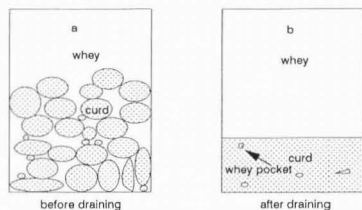


Figure 5. A schematic drawing of the situation before (a) and after (b) draining of a curd bed.

Whey has to percolate through the deformable mass of curd grains, mainly through connected openings between the curd grains. The size and location of these pores determine the permeability of the curd bed. Due to the rapid change in pore size, its estimation was, until recently, not feasible. It appeared that the optical fibre technique was applicable to the draining curd to measure both location and chord lengths of these pores; see Figure 6.

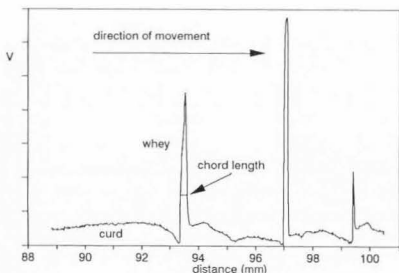


Figure 6. A part of the analogue signal of the optical fibre sensor penetrating into draining curd.

There are certain limits to this promising technique. In the case of discrimination by refractive index difference, the refractive index of at least one of the materials should be less than 1.44. In case of discrimination by scattering, the difference in the signal of the two materials is rather small and the difference in backscatter has therefore to be appreciable. The method can not be applied at rigid materials, because the fibre tip will snap. Fouling is remarkable absent in many products, but it happens in certain sticky materials, e.g. risen bread dough. It leads to a diminished signal, ultimately leading to blinding of the sensor. It has to be noted that the chemical resistance of the fibre is excellent and cleaning is therefore usually easy. The technique induces local destruction of the medium; therefore, it is not possible to measure twice at exactly the same spot. The diameter of the supporting capillary is slightly more than 1 mm.

An important feature of the optical fibre is its heat resistance. Application in heated media is therefore possible.

#### Conclusion

Applications of the moving optical fibre technique can be found in a wide range of heterogeneous products. Its speed, ease, and the limited disturbance of the sample challenge conventional techniques. Other important features are its heat and chemical resistance.

The method can not be applied to products with pockets of dispersed material smaller than the fibre tip. Application is limited to soft products. Fouling may diminish the intensity of the signal, but the discrimination of both materials often remains possible.

#### Acknowledgements

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#### Discussions with Reviewers

**M. Rosenberg:** As air cells in ice cream can be of less than 20  $\mu\text{m}$ , it seems that the results may not reflect the actual distribution?

**Authors:** The resolution of the method is determined by the fibre tip. Bubbles smaller than the fibre tip ( $\approx 20 \mu\text{m}$ ) will therefore be missed. Hence, the chord length distribution obtained will not include these bubbles. In the prototype 2 apparatus no statistics were applied to the obtained chord length distribution. Hence, a bubble size distribution is not calculated.

**M. Rosenberg:** Ice cream is not a two phase system. How do fat globules and ice crystals affect the results? Ice crystals and fat globules (which are attached to the lamella) may affect the interaction with the light in both directions and hence affect the results - both may affect light scattering.

**Authors:** Fat globules in ice cream are small ( $<2 \mu\text{m}$ ), as homogenization of the ice-cream mix is common practice, clusters of homogenized fat globules will generally not be larger than 10  $\mu\text{m}$ . Hence, the size of (clusters of) the fat globules is below the resolution of the method. It has to be remarked that the refractive index of fat is usually around 1.44, hence, much larger than those of air or ice-cream mix. Therefore, in case of very large fat clusters these can be distinguished. The signal will show three levels in that case. Ice crystals may be over 20  $\mu\text{m}$ , but will not be pierced by the optical fibre. The contribution of light scattered back toward the fibre tip is small in comparison to the total amount of light reflected.

**M. Rosenberg:** The effect of air cell piercing in the case of bread differ from the effect in ice cream. In the latter it may cause drainage due to mechanical damage to the "soft" lamella. The net disturbance caused by the moving apparatus is a

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function of the size of the air cells. How was this effect considered in data interpretation?

**Authors:** The sensitive part of the sensor is the most forward part of the fiber. The rapid movement and the small dimensions of the sensor limit the disturbance caused by the penetration drastically. The supporting capillary tube does disturb the structure, but the sensor is protruding approximately 5 mm, so its effect on the results is negligible.

**M. Rosenberg:** In case of ice cream, the tip of the probe is coated by various components of the product -solidified fat, etc. Is it affecting the light intensity and hence the results?

**Authors:** Fouling of the fiber tip is remarkably absent in many products. Apparently the movement of the fiber tip causes "self cleaning". Cleaning between subsequent measurements is usually redundant. There are certain products, e.g. risen bread dough, that stuck to the fiber tip, so it had to be cleaned between measurements. The interpretation of the signal is based upon a screening upon positive and negative slopes, hence, a reduction of the signal does not directly affect the results.

**B.E. Brooker:** In Figure 3,4 and 5, the authors present results obtained by application of an optical fibre technique to the study of the bubble size in bread, ice-cream and chocolate mousse. It is asserted that chord lengths in these traces give information about the bubble size, yet, in the absence of any attempt to compare these results with those obtained by other methods, it is difficult to assess the technique. Before this publication is formally accepted for publication, the authors should consider providing hard evidence that the technique gives reliable results that are comparable to, or better than, other techniques.

**Authors:** The technique was developed to study the porosity of draining curd. Up till now, there are no techniques available to measure this property. We tried hereto an extensive range of experimental techniques, but none of them gave satisfactory results. Using the optical fiber technique, we were able to make an overall balance; the amount of whey left in the curd grains (calculated by means of an expression model) and amount of whey enclosed between the curd grains (measured by means of the optical fibre technique) were compared to the overall whey content. The results were in accordance to each other.

**B.E. Brooker:** Does Fig. 4 contain information about ice crystal size as well as bubble size? If so, is it possible to obtain separate distributions for bubbles and ice crystals?

**Authors:** Applying the technique to measure the amount and/or size distribution of ice crystals is probably not feasible. Ice crystals are very rigid and will not be penetrated by the fiber. Using NMR is probably a better option.

**F. van Voorst Vader:** Which V-coordinate in the Figures 3,4 and 5 is used for evaluation of the chord size?

**Authors:** The V-coordinate is not given, as the evaluation of the chord length is based upon distance between rising and decreasing slopes. The difference between the signals of the two materials may be over 5 V in case of refractive index based distinction (between gas and "solid"), and as less as 0.5 V in case of distinction based upon differences in scattering properties.