



## Composition and structure of high temperature dairy fouling



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### ABSTRACT

The fouling structure and composition is dependent on the product, but also on the heating process applied to it. The structure will have profound effect on the cleaning process and the down time in the production plant. Here, the structure of high temperature (137 °C) milk fouling has been investigated, which so far has not been sufficiently studied in a systematic way. This particular fouling has a high content of the mineral calcium phosphate and a relatively low concentration of protein. Wide angle X-ray diffraction (WAXD) reveals a crystalline structure of calcium phosphate in agreement to the chemical analysis of the bulk layer. Microscopic investigations visualize the heterogeneous structure and energy dispersive X-ray spectroscopy (EDX) shows a spatial variation of the elements through the radius of the sample.

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### 1. Introduction

Dairy fouling is diverse in structure and composition and is heavily dependent on the product but also on the heating process (Visser & Jeurink, 1997). Fouling decreases the heat transfer that results in increased energy consumption, increased costs and environmental impact. The removal of these deposits presents a major challenge for the dairy industry as it is time consuming, causes a major environmental load (in the form of fresh water, sewage water, chemicals and heat), and could imply product hazards if not properly accomplished. For this purpose it is important to determine the fouling microstructure to be able to later optimize the cleaning procedure. Both the chemical composition and the fouling structure influence the cleaning process (Fryer & Asteriadou, 2009).

The spatial distribution of minerals and proteins, as well as the porosity and the thickness of the fouling layer could influence the penetration of the cleaning agent and the subsequent solubility/degradation and removal of the deposits. The chemical composition of the dairy fouling will be different depending on processing conditions, where the temperature can vary over a wide range

(Erabit, Ndoye, Alvarez, & Flick, 2015; Khaldi et al., 2015; Petit, Six, Moreau, Ronse, & Delaplace, 2013; Prakash, Kravchuk, & Deeth, 2015). Protein rich (~60%) fouling is produced at lower temperature with an onset around 85 °C. This structure has a soft and voluminous characteristic. When the processing temperature is increased the fouling composition changes and at the temperatures for ultra-high temperature (UHT) processing at 137–140 °C, the most abundant phase is instead a dense mineral phase (Burton, 1968; Jimenez et al., 2013; Lalande, Tissier, & Corrieu, 1984; Lyster, 1965; Schraml & Kessler, 1994).

Close to 100 °C it has been reported that the fouling layer from milk has a denser bottom layer and a more spongy layer on top, but that both layers contain protein as well as the mineral component (Tissier & Lalande, 1986). This structural difference between the top and the bottom fouling layer, close to the metal surface, has also been observed at 140 °C, where a higher concentration of protein was observed close to the bulk and the mineral content was higher close to the metal surface (Foster & Green, 1990). The mineral phase of dairy fouling is dominated by calcium phosphate. These early studies on the fouling composition are performed using relatively short production times (<2 h, (Foster & Green, 1990)) and therefore concerns thin layers of fouling and they also do not address the organization of the different components within the fouling layer.

The objective of this work is therefore to provide a fundamental understanding of the dairy fouling film microstructure, in terms of

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composition and micro and submicron structure as well as organization within the fouling layer. This will help us to understand processes by which the fouling layers are built up, and also how to remove the layer. For a cleaning process to be effective, the cleaning liquid needs to be able to penetrate into the fouling film and to dissolve the different parts of the fouling film. It is known that alkaline and acid treatment are expected to mainly dissolve proteins (organic material) and minerals; respectively. The spatial distribution of minerals and proteins, as well as the porosity and the thickness of fouling film are likely to influence the penetration of cleaning agent and the subsequent solubility/ degradation and removal of the deposits. However, few studies analyze the microstructure and chemical composition of the fouling with respect to cleaning of dairy processing equipment.

In this work, the detailed microstructure of dairy ultra-high temperature (UHT) fouling produced at close to real conditions (137 °C for 8 s over a period of 15 h) has been characterized for the first time to the best of our knowledge, using a combination of high resolution imaging techniques and specific staining of various components. The aims were to (i) determine the microstructure at different length scales and positions; (ii) analyze the overall chemical composition; (iii) reveal the local spatial dependence of the chemical composition as a function of distance from the metallic surface; (iv) reveal information about the crystalline structure in the UHT fouling film. The techniques used were confocal laser scanning microscopy (CLSM) and scanning electron microscopy (SEM) in combination with energy dispersive X-ray spectroscopy (EDX), chemical analysis and wide angle X-ray diffraction (WAXD).

SEM has previously been used in order to resolve questions concerning the fouling build-up and the structure of both the protein and mineral networks in the milk deposit (Bouman, Driessen, & Schmidt, 1981; Rosmaninho & Melo, 2006; Schraml & Kessler, 1994). However, most of these studies have been carried out using whey protein model fouling with varying concentration of minerals or pure mineral solutions such as simulated milk ultrafiltrate. The results therefore cannot be directly applied to explain the structure and behavior of a high temperature, mineral rich milk fouling. Two types of initial fouling structures have been identified from the topography of the fouling layer, namely mineral crystals and spherical protein aggregates (Tissier & Lalande, 1986). CLSM has been used to reveal the microstructure of the food products as a consequence of processing but also to link the microstructure and rheology to the sensory perception of food (Abhyankar, Mulvihill, & Auty, 2014). Long production times (in this study 15 h) under real processing conditions, producing a relatively thick fouling layer give the possibility to study the spatial variation in the cross section from the metal surface to the bulk phase. To our best knowledge this is the first time the technique has been used to reveal the cross section fouling structures using specific probes that target the key components of the deposit namely calcium and protein.

Wide angle X-ray diffraction is a structure analysis technique that can reveal the crystallinity and crystal structure of the calcium phosphate phase of the fouling layer (Mekmene et al., 2009; Visser & Jeurnink, 1997).

This work will provide a fundamental understanding of the structure of high temperature dairy fouling. One particular strength of this work is that the cross section of industrially produced, high temperature dairy fouling is used for the analysis. Results on the overall fouling microstructure at different length scales and the spatially dependent chemical composition will be presented. More specifically, the chemical composition and the microstructure as a function of the distance from the metal surface will be discussed.

## 2. Method and materials

It has been found that the combination of SEM, CLSM, EDX, WAXD and chemical analysis are powerful techniques to determine the high temperature fouling microstructure and its composition. The method of producing high temperature milk fouling and the techniques used to investigate the composition and structure is introduced below.

### 2.1. Online production of high temperature fouling

A normal aseptic production line does not allow collection of fouling samples; therefore a pilot plant that has been previously described by Hagsten et al. (2013) was used, that follow the time-temperature profile of a standard UHT. The pilot equipment is directly located in a dairy facility in order to process the milk as close to industrial settings as possible. The equipment for this study is run to mimic real process conditions and the milk is heated to 137 °C for 8 s during a production time of 15 h. The milk is not recirculated in the equipment but only a single pass through the heat exchangers is used (Hagsten et al., 2013).

The fouling samples collected for analysis are deposited on stainless steel coupons mounted in the process line of the pilot plant in such a way that the disturbance on the flow and temperature profiles are minimal. The fouled coupons are located in the high temperature test unit after the final heat exchanger and before the cooling section of the pilot plant. In order to study the cross section of the fouling layer, the deposit was removed from the steel surface. Since the fouling layer have a high mineral content and is rather rigid, this procedure is expected to have a low impact on the microstructure of the fouling layer.

Skim milk was used for this study, but the results are expected to be relevant also for whole milk processing since milk fat has not been found to be present in high concentration (max 4–8 w/w%) in fouling from full fat milk (Burton, 1968). Furthermore, whole milk has been shown not to produce a significantly different fouling from the skimmed milk with regards to chemical composition or the amount of produced fouling (Bansal & Chen, 2006; Foster & Green, 1990; Visser & Jeurnink, 1997).

### 2.2. Measurement of the chemical composition

#### 2.2.1. Overall chemical composition of the fouling film

The chemical composition of the high temperature fouling used in this study was analyzed in an external facility with regard to mineral and protein content. Before analysis the solid fouling sample were dissolved. For this purpose 0.5 g sample added into a solution of 7 ml HNO<sub>3</sub> and 3 ml of water and then heated in a closed Teflon vessel to 185 °C (CEM MARS 5, Buckingham, UK). Total carbon and nitrogen in the sample was determined in an elemental analyzer (Vario max CN, Elementar Analysensystem GmbH). The instrument was used in order to determine the protein content in the fouling, where the total nitrogen in the sample is multiplied with the Jones' factor for milk, 6.38 (Jones, 1941). The basic elements, Ca, P and Mg, which are the main mineral components in dairy fouling, were analyzed. The mineral content was determined with inductively coupled plasma optical emission spectroscopy (ICP-OES) (OPTIMA 8300, PerkinElmer). The water content of the fouling after production was determined by weighing the pieces of fouling before and after drying. Drying of the samples was performed in a vacuum oven at 80 °C for 17 h and then also tracking the water activity (AquaLab, Decagon) to make sure that the samples were dry at the end.

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