

# Melting characteristics of fat present on the surface of industrial spray-dried dairy powders

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## Abstract

The melting characteristics of the fat present on the surface (surface free-fat) of two industrial spray-dried dairy powders (cream powder and whole milk powder) were investigated in comparison with those of other milk fat fractions present in the powder, such as free-fat from the interior of the powder particle (inner free-fat) and encapsulated fat. The melting characteristics of the milk fat fractions were studied by fatty acid composition, melting profile and solid fat content profile. The results indicated that all milk fat fractions including surface free-fat contained various triglycerides with melting points ranging from  $-40$  to  $+40$  °C. However, some fractionation was observed among the different milk fat fractions. The free-fat fractions (surface free-fat and inner free-fat) had a greater proportion of high-melting triglyceride species than the encapsulated fat. Furthermore, the high-melting triglyceride species present in the free-fat fractions were slightly accumulated at the surface of powder. This phenomenon was observed in both cream powder and whole milk powder and its effect on wetting time was established. This indicates that manipulation of the surface fat content during drying operation may hold the key to functionality improvement. © 2005 Elsevier B.V. All rights reserved.

**Keywords:** Fatty acid composition; Melting behavior; GC–MS; DSC; Milk fat; Surface free-fat; Inner free-fat; Encapsulated fat

## 1. Introduction

Spray-dried dairy powders are common ingredients in many food and dairy industries. Many powder properties important in its storage, handling and final application (e.g. wettability, dispersibility, flowability and oxidative stability) are expected to be largely determined by the surface composition of the powder. If one of the milk components is preferentially present on the powder surface, these properties may be changed dramatically. Of special importance is the amount of fat on the powder surface. The presence of fat renders the powder surface hydrophobic decreasing wettability and dispersibility [1]. Fat on the surface acts as a bridge between the particles reducing flowability [2,3]. It is also readily susceptible to oxidation and the development of rancidity [4,5]. Understanding the mechanism behind the formation of pow-

der surface composition and the ability to control the surface composition will be, therefore, highly useful in product quality improvement and new product development.

In order to understand the mechanism behind the formation of powder surface composition, a detailed knowledge of the surface composition of spray-dried dairy powders is firstly needed. In our previous publication [6], the surface composition of various industrial spray-dried dairy powders was investigated by means of electron spectroscopy for chemical analysis (ESCA), a method providing direct chemical analysis of the outermost surface layer ( $\sim 10$  nm). It was found that the surface composition of powders is significantly different from their bulk composition. Particularly pronounced is the accumulation of fat on the surface of fat-containing dairy powders, such as cream powder and whole milk powder. The top 10 nm surface layer of these powders is mainly made of milk fat (99 wt.% for cream powder, 98 wt.% for whole milk powder) [6]. This observation indicates that the surface-related properties of these powders will be greatly

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determined by the melting characteristics of the fat present on the powder surface. Milk fat is a heterogeneous mixture of various triglycerides with a melting range from  $-40$  to  $40^{\circ}\text{C}$  [7]. In the spray-drying process, the milk fat is encapsulated by protein and lactose matrix (so called ‘encapsulated fat’), and only part of the fat can be extracted by organic solvents (so called ‘free-fat’). The free-fat is the fat that is extracted using solvent within a pre-fixed time and thus originates not only from the powder surface, but also comes from the interior of the powder particle [8]. Different triglycerides can have different encapsulation efficiency and the distribution of triglycerides in the powder particle may not be uniform. In order to further investigate the surface formation mechanism of fat-containing dairy powders, the melting characteristics of the fat present on both the powder surface and the bulk powder, therefore, are of great interest.

The effects of different fat phases on the encapsulation efficiency (level of surface fat) have been investigated using ESCA [4,9–11]. It was shown that the liquid oils and fully crystalline fats with high melting points were well encapsulated showing low surface fat coverage, while fats with intermediate melting points were poorly encapsulated with the highest surface fat coverage. The authors explained that the highest surface fat coverage in the latter is attributed to decreased emulsion stability of partially crystalline emulsions. A similar trend was observed when emulsions with mixtures of different fats were spray-dried, the pure fats are more efficiently encapsulated than any mixtures [12]. It is not possible to distinguish between solid fat and oil by ESCA since they do not differ in the C/O ratio. Due to this methodological limitation, the previous studies assumed that the different fats uniformly distributed in various fat fractions in the powder particle and no information could not be provided regarding the melting characteristics of the fat present on the powder surface.

The objective of the present study was to investigate the melting characteristics of the fat present on the surface of industrial spray-dried fat-containing dairy powders in comparison with other milk fat fractions present in the powder. To this end, the milk fat present in the powders was differentiated into three types; free-fat originated from the powder surface (defined as surface free-fat), free-fat originated from the interior of the powder particle (defined as inner free-fat) and encapsulated fat. Those various milk fat fractions were then analyzed for fatty acid composition and melting behavior. Furthermore, the melting point of fat present on the powder surface was directly estimated by testing the wettability of powders using various water temperatures.

## 2. Materials and methods

### 2.1. Materials

Two industrial spray-dried fat-containing dairy powders (cream powder (CP) and whole milk powder (WMP)) were

Table 1  
Bulk composition of the industrial spray-dried dairy powders used

Product	Composition (wt.%)				
	Lactose	Protein	Fat	Moisture	Ash
CP	12.3	11.5	71.5	2.7	2.0
WMP	36.6	27.9	26.6	3.0	5.9

obtained from a local dairy company. The powders were commercial products which had been freshly manufactured and packed for consumer use. The capacities of dryers where the powders were made are at least 4 t powder per h. The composition of the powders used is shown in Table 1. The hexane (95%) and iso-propanol (>99%) used in this study were purchased from Asia Pacific Specialty Chemicals Ltd. (Auckland, New Zealand). Boron trifluoride-methanol was purchased from Sigma Chemical Company (St. Louis, MO, USA). Deionised water was used throughout.

### 2.2. Methods

#### 2.2.1. Extraction of milk fat fractions

The extraction process of different milk fat fractions in spray-dried dairy powders is schematically shown in Fig. 1. The details of each extraction procedure are as follows. The procedures were repeated until a sufficient amount of fat was obtained for analyses. In all cases we are referring to the fat that is extractable by organic solvents.

**2.2.1.1. Extraction of surface free-fat.** The free-fat on the surface of a powder particle is quickly dissolved by very brief exposure to the organic solvents. The free-fat from the inner part of the particle dissolves much more slowly. In order to extract only surface free-fat and minimize the extraction of free-fat from the interior of powders, only a brief wash with organic solvent was carried out. Surface free-fat was extracted by a modification of the method described by Buchheim [13]. One gram of the fresh powder was accurately weighed on a filter paper (No. 4, Whatman, Maidstone, Kent, UK), and washed with  $4 \times 5$  ml of hexane. The powder residue was dried under vacuum at room temperature, and the filtrate solution containing the extracted fat was allowed to evaporate until the extracted fat residue achieved a constant weight. The extracted fat value was then recorded as g surface free-fat/g fresh powder.

**2.2.1.2. Extraction of inner free-fat.** The powder residue left after the extraction of surface free-fat was added to 40 ml of hexane/g powder, and shaken frequently by hand for 48 h. The powder residue and the solvent were first separated by filtration through filter paper (No. 4, Whatman, Maidstone, Kent, UK). The powder residue was further washed with  $2 \times 2$  ml hexane and then dried under vacuum at room temperature. The filtrate solution containing the extracted fat was allowed to evaporate until the extracted fat residue achieved a con-

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