



Physicochemical and structural properties of compound dairy fat blends

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ARTICLE INFO

Article history:

Received 23 November 2011

Accepted 9 March 2012

Keywords:

Anhydrous milk fat

Palm oil

Dry fractionation

Polymorphism

Hardness

Solid fat content

ABSTRACT

The physicochemical properties of fat model systems made of commercial samples of anhydrous milk fat (AMF) or a fraction blended with palm oil (PO) were studied. Physical properties such as solid fat content, melting curves by differential scanning calorimetry, textural properties, and polymorphism were investigated. The objective of the present work was to systematically map interactions (compatibility/incompatibility) that occur in such model of compound fat systems with respect to butyric-based shortening or butter-like spreads formulations.

For that purpose, iso-solid diagrams have been constructed from p-NMR data. Molecular interactions have been highlighted for all the blends, especially at low temperatures. Compositions at which molecular interactions were detected depend on the TAGs composition of the fractions involved in the blends. For example, under dynamic conditions, a minimum was observed (eutectic interaction) for all the blends. This minimum was shifted to higher PO content for blends made of AMF fractions with lower iodine value (IV) and to lower PO content for AMF with higher IV. After static crystallization followed by a tempering at 15 °C, interactions also existed for all the blends. It was shown that the deviations found in hardness after this tempering procedure can be explained by intersolubility, polymorphic and microstructure arguments.

Practical applications: Results reported here concern physical characteristics of several compound fat blends (butyric-vegetal). A comprehensive analysis of binary fat blends made of AMF, or its fractions, blended with a vegetal fat was conducted. This better understanding of crystallization phenomena occurring is required to further enhance the use of AMF in compound-margarines and shortenings. Indeed phase properties of fat blends have a significant influence on the sensorial characteristics of the final products (hardness, brittleness, grainy or smooth texture, ...). This study is relevant for the dairy food industry as it may contribute to the development of new mixed-fat products, also sometimes called dairy blends.

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1. Introduction

Milk fat is desired as a natural product that has excellent organoleptic properties in many food products and is used primarily for the butter flavor (Hartel, 1996). The main differences between butter and margarines are related to the sensory characteristics of milk fat. Milk fat makes butter superior to margarines in taste and mouth feel. However, simply adding butter flavor into margarine is often not enough. Blends of anhydrous milk fat (AMF) with vegetable oils can lower the costs relative to butter while having the preferred taste of butter. In this context, milk fat is increasingly blended with other edible fats or oils to obtain products with desired functional properties while

reducing the cost (Augustin & Versteeg, 2006). Examples of such products are the butter-like spreads and butter-like shortenings (dairy blends). The macroscopic properties and functionalities of fat-based products are closely related to their microscopic properties. Macroscopic properties depend not only on the solid fat content of the system; polymorphism and crystal network are also key factors (Brapson-Danthine & Deroanne, 2004; Marangoni & Narine, 2002). The sensory properties, especially texture and appearance, of edible fat-based products are largely dependent on the physical properties of the fats. In food systems that incorporate milk fat and other fats, the milk fat-fat interactions that occur are generally expressed by functionalities such as crystallization behavior. Milk fat has a more diversified, variable and complex composition than vegetal and other animal fats (Timms, 1994). According to the literature more than 400 fatty acids have been identified in milk fat (Jensen, Ferris, & Lammi-Keefe, 1991). Also the chemical composition of AMF is very different from other fats with similar melting point: its C4:0 content is unique. This butyric acid is a key fatty acid, which influences properties of AMF. This high level of short chain acids produce a softer fat that would otherwise be expected from the total saturated fatty acid content (more than 65%) (Timms, 1994).

Abbreviations: AMF, Anhydrous milk fat; DSC, Differential scanning calorimetry; HMP, High melting peak; HMA, High melting area; LMP, Low melting peak; LMA, Low melting area; MMP, Medium melting peak; MMA, Medium melting area; PO, Palm oil; RO, Rapeseed oil; SFC, Solid fat content; SFI, Solid fat index; TAG, Triacylglycerol.

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In order to increase the applications field milk fat can be modified through a variety of techniques. Kaylegian and Lindsay (1995) presented a review of milk fat fractionation technology. The most common technique is dry fractionation which involves melting of the fat, controlled cooling and crystallization followed by a separation of the crystals from the remaining liquid phase. The absence of organic solvents and the minimal loss of aroma components compared to other methods have made melt fractionation the most popular method in the AMF industry. As a result of the diversity of fatty acids, milk fat has a very wide melting range (-40 to 40 °C) and is a very suitable material for physical fractionation which relies primarily on the melting points of the different TAG. Dry fractionation of AMF can produce a range of products with different physical and chemical characteristics, which are interesting for use as food ingredients, suitable for a wide range of applications (De et al., 2007). To further enhance the use of AMF or its fractions blended with vegetal fats in new mixed-margarines or shortenings (also called dairy blends), a better understanding of crystallization phenomena occurring between those lipids is required. In this context, results reported here concern physical characteristics of several butyric–vegetal mixed-fat blends. More precisely, the objective of the present work is to systematically map interactions (compatibility/incompatibility) that occur in some model of compound fat systems involving PO and AMF (or one of its fractions) with respect to butyric-based shortening and butter-like spreads formulations.

This study is thus relevant for the dairy food industry as it could contribute to the development of new compound dairy fat products.

2. Experimental procedure

2.1. Material

Beside whole anhydrous milk fat (AMF), four different fractions all obtained by dry fractionation were used in this study: a Very Low Melting fraction (VLM-AMF) (IV 45.0), a Low Melting fraction (LM-AMF) (IV 42.0), a Medium Melting fraction (MM-AMF) (IV 37.5) and a High Melting fraction (HM-AMF) (IV 27.5). All of them were from industrial supply.

Palm oil (PO) and rapeseed oil (RO) were supplied by Loders Croklaan B.V. (Wormerveer, The Netherlands).

2.2. Methods

2.2.1. Sample preparation

Various blends were prepared according to the method proposed by Danthine and Deroanne (2003 & 2006) at composition intervals of $10 \pm 0.1\%$ (w/w). Appropriate weight compositions (250 g) of the melted fats were mixed at 80 °C (to erase crystal memory) in a clean, dry, and agitated glass vessel; samples of 50 g were put into a small plastic vessel and kept in liquid form at 50 °C with gentle stirring for 15 min. Then they were statically crystallized in a freezer at -20 °C for half an hour, in order to simulate on a laboratory scale an industrial scraped-surface heat exchanger. Samples were subsequently stored in a 15.0 ± 0.5 °C controlled room for 48 h.

2.2.2. FA determination

FAME were prepared from crude lipids by boron trifluoride-catalyzed transesterification according to AOCS Method Ce 2–66 (AOCS, 1997), which was slightly modified, and analyzed by GC on a Hewlett-Packard HP5880a apparatus fitted with a cold on-column injector and an FID (temperature 250 °C). The operating conditions were as follows: $25 \text{ m} \times 0.32 \text{ mm}$ CP-WAX-FFAP 58CB column from Chrompack (Middelburg, The Netherlands); film thickness, $0.2 \mu\text{m}$; temperature program, from 55 to 150 °C at 30 °C/min and from 150 to 240 °C at 5 °C/min. Helium at 70 kPa was used as carrier gas. FAME

were identified on the basis of their retention data compared with those of pure references.

2.2.3. Physical characterization

2.2.3.1. Melting properties by differential scanning calorimetry (DSC). DSC analyses were carried out using a Q1000 DSC (TA Instruments, New Castle, USA) with a refrigerated cooling system (TA Instruments, New Castle, USA) and aluminum SFI pans. Calibration was made with indium (m.p. 156.6 °C) and n-dodecane (m.p. -9.56 °C) standards. Nitrogen was used as purge gas in order to prevent condensation in the cells. An empty aluminum SFI pan was used as reference. Samples weighed between 2 and 4 mg. They were first heated in the DSC pan to 80 °C to ensure complete melting and held for 5 min at that temperature to erase thermal memory. Afterwards, they were quickly frozen at -60 °C (cooling rate -25 °C/min), and kept for 10 min at -60 °C, in order to ensure complete solidification. Melting profiles were recorded from -60 °C to 70 °C at a heating rate of 5 °C/min. All the DSC analyses were carried out in duplicate.

The integration and peak temperature measurements were performed using the Universal Analysis Software version 4.2 (TA Instruments, New Castle, USA). The melting peaks were integrated with a linear baseline.

2.2.3.2. Solid fat content determination. The SFC have been measured according to the standard serial non tempered IUPAC 2.150 method (IUPAC, 1987) using a pulsed NMR spectrometer (Minispec-mq20, BRUKER, Germany). Automatic calibration was made daily using 3 standards (supplied by Bruker, Germany) containing respectively 0.0, 31.3 and 74.8% of solids.

SFC were also measured directly on crystallized and tempered samples (see Section 2.2–Section 2.2.1) in order to compare data with results obtained for other measurements (i.e. textural measurements, powder X-ray diffraction analyses and microscopy). For that purpose, the NMR tubes were filled with crystallized material by means of a sampling device consisting of a glass tube with a tight fitting plunger. Each analysis was run at least twice.

2.2.3.3. Texture measurements. Texture measurements of the products were carried out after the tempering at 15 °C. Samples were first held at 20 °C for 1 h before measuring them in a controlled temperature cabinet using a SMS TA.XT2i/5 texturometer (Stable Micro System, Surrey, UK).

2.2.3.3.1. Constant speed penetration. The texturometer was used with a cone probe (P/45 C) in the penetration tests as previously described (Braipson-Danthine & Deroanne, 2004). At least four penetration tests were run on each sample; two samples of each blend were analyzed. Coefficients of variation (CV) were all lower than 10%.

2.2.3.4. Powder X-Ray Diffraction analyses. The polymorphic forms of the blends were determined by powder X-ray diffraction using a D8 advance diffractometer (Bruker, Germany) (λ Cu = 1.54178 Å, 40 kV, 30 mA) equipped with a Vantec (Bruker, Germany) detector, and a TTK450 low-temperature chamber and TCU 110 temperature control unit (Anton Paar, Graz, Austria) connected to a circulating water bath (Julabo, Germany). Diffraction pattern were recorded in the 2θ range 1.5° – 27° . d -Spacings were determined using the Bragg law. Analyses were performed isothermally after the tempering, as it was for the textural and p-NMR measurements. Each sample was run in triplicate.

2.2.3.5. Microscopy. Samples were imaged on a temperature-controlled microscope stage (Nikon Eclipse E400) after tempering at 15 °C, using a Linkam PE60 temperature controller and Peltier stage system (Linkam Scientific Instrument Ltd). All the images were recorded using a BASLER video camera with the gain switch in the auto position and digitized using the Lucia G software.

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