



## Ultra-stable self-foaming oils

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### ABSTRACT

This paper is concerned with the foaming of a range of fats in the absence of added foaming agent/emulsifier. By controlling the temperature on warming from the solid or cooling from the melt, crystals of high melting triglycerides form in a continuous phase of low melting triglycerides. Such crystal dispersions in oil can be aerated to produce whipped oils of high foamability and extremely high stability. The foams do not exhibit drainage and bubbles neither coarsen nor coalesce as they become coated with solid crystals. The majority of the findings relate to coconut oil but the same phenomenon occurs in shea butter, cocoa butter and palm kernel stearin. For each fat, there exists an optimum temperature for foaming at which the solid fat content reaches up to around 30%. We demonstrate that the oil foams are temperature-responsive and foam collapse can be controllably triggered by warming the foam to around the melting point of the crystals. Our hypothesis is given credence in the case of the pure system of tristearin crystals in liquid tricaprilyn.

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### 1. Introduction

Literature on the preparation and properties of oil foams (or gas-in-oil) is very scant compared with that on aqueous foams despite their importance in a number of industries. Due partly to the lower surface tension of most oils compared with water, common surfactants are not surface-active at the oil-air surface and hence do not stabilise oil foams. In a series of papers, Shrestha and co-workers however succeeded in stabilising foams of a range of oils using mono- and diglyceride fatty acid esters (Shrestha, Aramaki, Kato, Takase, & Kunieda, 2006; Kunieda et al., 2007; Shrestha, Shrestha, Sharma, & Aramaki, 2008; Shrestha, Shrestha, Solans, Gonzalez, & Aramaki, 2010). These surfactants formed either lamellar liquid crystals or surfactant particles in oil which were effective at preventing air bubbles from coalescence for a limited time. For a lubricating oil of poly(dec-1-ene), we showed likewise that various surfactants and polymers enabled foam stabilisation at conditions close to phase separation of the additive in oil (Binks, Davies, Fletcher, & Sharp, 2010). By contrast, different kinds of solid particle have recently been shown to be excellent foaming agents of many oils, provided the oil surface tension is not too low and that particles have the appropriate wettability (Murakami & Bismarck, 2010; Binks & Rocher, 2010; Binks, Rocher, & Kirkland, 2011; Binks & Tyowua, 2013; Binks, Sekine, & Tyowua, 2014; Binks, Johnston, Sekine, & Tyowua, 2015). These foams can be ultra-stable due to the irreversible adsorption of colloidal particles to the liquid surface (Binks & Horozov, 2006). For hydrocarbon-containing oils, particles

of low surface energy are required and these have included oligo- or polytetrafluoroethylene (Murakami & Bismarck, 2010; Binks & Rocher, 2010; Binks et al., 2011), fluorosilica (Binks & Tyowua, 2013) or fluoroclay (Binks et al., 2014; Binks et al., 2015) particles of a variety of shapes and sizes.

A strategy used recently to prepare stable oil foams has been to allow crystals of the additive to form within the oil phase on cooling which, after aeration, adsorb to air bubble surfaces endowing the foam with high stability to drainage, coalescence and disproportionation. These mixtures can be thought of as a high melting point additive (forms crystals) within a low melting point oil (continuous phase). In the four examples studied so far, the latter has been a vegetable oil with applications to aerated foods, but this is not a requirement (Garvey, 2014). The systems have been a commercial mono/diglyceride surfactant in rapeseed oil (Brun, Delample, Harte, Lecomte, & Leal-Calderon, 2015), long chain alcohols in sunflower oil (Fameau et al., 2015), a long chain carboxylic acid in high oleic sunflower oil and other vegetable oils (Binks, Garvey, & Vieira, 2016) and a saturated triglyceride in a soybean-rapeseed oil mixture (Mishima, Suzuki, Sato, & Ueno, 2016). The oils above are examples of triacylglycerides (TAGs) in which a high proportion of the long fatty acid (FA) chains are unsaturated such that the melting point of the oil is low (<0 °C) and hence the oils are liquid at room temperature.

Edible compositions in which fat is the continuous phase like butter, margarine and liquid oils are well known. They are commonly used as the fat ingredient in baking, as a frying medium or even consumed directly like the topping on pancakes or bread. The fat phase of the above is typically a mixture of liquid oil (fat that is liquid at room temperature) and fat (which is solid at room temperature). The solid fat

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(sometimes called the hardstock fat) structures the fat phase and, if water is present as droplets, aids in stabilising the emulsion by forming a fat crystal network in the continuous phase. It is ideal if the solid fat melts or dissolves around the temperature within the mouth otherwise there may be a waxy mouth feel. In addition to the ongoing need of manufacturers to reduce costs, health conscious consumers desire reduced fat-containing compositions these days. One way to achieve this is to replace part of the fat with an edible gas. Such aerated food compositions are known, e.g. ice cream, whipped cream and mousses, but these are complex colloidal formulations containing many other ingredients (Campbell & Mougeot, 1999). Normally, the gas is present as dispersed bubbles but, in the case of aerated fats/oils, their stability is frequently low. Thus, during storage, coarsening and coalescence of the bubbles can occur as well as drainage of liquid oil which reduces the aesthetic quality of the product. There is thus a need to develop aerated fat-continuous products exhibiting high stability. At the same time, some consumers deem it unhealthy to consume a product which contains synthetic ingredients like fatty acids, E-number emulsifiers and sugar. Unlike the studies referred to above, we put forward here a novel idea for stabilising aerated oils which does not require any additive. Our main objectives are: (a) to manipulate the temperature of a fat/oil such that oil foams stabilised by fat crystals can be formed in the absence of any additive and (b) to determine if these crystal-stabilised foams are sensitive to temperature approaching the melting point of the crystals. We select triglyceride oils/fats of medium chain length (mainly C<sub>8</sub>–C<sub>12</sub>) which contain predominantly saturated FA chains such that their melting points are around or above room temperature. Since such oils are composed of a mixture of TAGs of different chain length/composition, we make use of the selective crystallisation of those TAGs of higher melting point within liquid oil of those TAGs of lower melting point, i.e. an oil dispersion of crystals forms from one and the same oil. We acknowledge that TAGs can also exist in several polymorphic forms exhibiting different melting temperatures. The question is whether crystals of particular TAGs adsorb around air bubbles dispersed in liquid oil composed of other TAGs. The studies focus on the case of coconut oil, whose use is currently experiencing a renaissance (Cassiday, 2016), but we give examples for other oils containing saturated TAGs. We also demonstrate the thermo-responsive nature of these oil foams by destabilising them around the melting point of the crystals. In order to verify our hypothesis that crystals of certain TAGs enable foaming of liquid oil of the remaining TAGs, we show that stable foams in a pure short chain liquid TAG (tricaprylin, C<sub>8</sub>) can be prepared which contain adsorbed crystals of a pure long chain solid TAG (tristearin, C<sub>18</sub>).

## 2. Materials and methods

### 2.1. Materials

Refined, bleached and deodorized (RBD) coconut oil (*Cocos nucifera*) was a gift from AAK, Sweden (batch B0364E) with the free FA content being <0.02%. Coconut oil is an edible oil extracted from the kernel of mature coconuts harvested from the coconut palm. RBD oil is made from dried coconut kernel, which is placed in a hydraulic press with added heat, from which the oil is extracted. Because of its high saturated fat content, it is slow to oxidise (Moigradean, Poiana, & Gogoasa, 2012) and is thus resistant to rancidification. Coconut oil contains mainly saturated TAGs (>90%) and is an excellent source of medium chain fatty acids that can be easily burnt for energy. Typically, coconut oil contains around nine FAs of different melting point being caproic (Co, C<sub>6</sub>:0) 0.7%, caprylic (Cy, C<sub>8</sub>:0) 8.1%, capric (Ca, C<sub>10</sub>:0) 6.3%, lauric (La, C<sub>12</sub>:0) 48.3%, myristic (M, C<sub>14</sub>:0) 18.1%, palmitic (C<sub>16</sub>:0) 8.4%, stearic (C<sub>18</sub>:0) 2.4%, oleic (C<sub>18</sub>:1) 5.9% and linoleic (C<sub>18</sub>:2) 1.8%. Using HPLC-MS, the most abundant TAGs >5% were LaLaM, LaLaLa, LaLaCy, CyLaM and CaLaM accounting for over 43% of the TAGs detected (Neff, Byrdwell, & List, 2001). Palm kernel oil (*Elaeis guineensis*) Encore 100 was a gift from

Cargill Food Ingredients, USA and is in fact palm kernel stearin (Personal communication, 2016). It also contains over 90% saturated TAGs (mainly C<sub>12</sub>:0 and C<sub>14</sub>:0). Refined cocoa butter (*Butyrum theobroma cacao*) and refined shea butter (*Butyrospermum parkii*) were purchased from Naissance, UK and contained no added fragrances or colours. Cocoa butter contains around 60% saturated TAGs (mainly C<sub>16</sub>:0 and C<sub>18</sub>:0, Firestone, 2013) whereas shea butter contains only around 45% saturated TAGs (mainly C<sub>16</sub>:0 and C<sub>18</sub>:0, Firestone, 2013). Tricaprylin, a low melting triglyceride (99%, melting point 9.5 °C) and tristearin, a high melting triglyceride (80%, highest melting point 73 °C) were purchased from Sigma-Aldrich.

### 2.2. Melting and cooling of coconut oil

The solid fat content (SFC) of coconut oil was determined using a Bruker NMR minispec mq<sub>one</sub> SFC analyzer (AOCS Official Method, 2009). In these measurements, the oil was placed in an NMR tube and heated to 60 °C for 10 min. It was then cooled to 0 °C for 1 h before being placed in a water bath at either 10, 20 or 30 °C for 30 min before measurement of the SFC. Differential scanning calorimetry (DSC) measurements were carried out using a Perkin-Elmer DSC-7 instrument using nitrogen as the purge gas (see e.g. Chiavaro, 2015). Calibration was done using a sample of indium. Coconut oil (2.20 mg) was placed into a 20 µL aluminium pan which was sealed using a crimper. It was heated from –25 °C to 60 °C at 10 °C/min followed by cooling from 60 °C to –25 °C at 10 °C/min.

### 2.3. Aeration of oils

Apart from coconut oil, the other food oils were stored in the dark at 18 °C for various lengths of time before being warmed at a rate of approximately 0.1 °C/min. Coconut oil was stored in the fridge at 6 °C for up to 12 h and subsequently warmed at the same rate. A 250 mL polypropylene bowl containing 70 g of a solid vegetable oil (coconut oil, shea butter, cocoa butter or palm kernel stearin) was placed in a thermostated water bath. A Brannan England thermostat probe was also used to measure the temperature of the sample inside the bowl. For the majority of experiments, the initial temperature of the water bath was set at 18 °C and it was increased gradually at an approximate rate of 0.1 °C/min. This process continued until the desirable temperature was achieved between 19 and 42 °C depending on the type of vegetable oil. For coconut oil, experiments were also conducted in which a clear solution at 30 °C was cooled gradually to a set temperature before whipping. At the temperature of interest, the aeration of the sample was made with the use of a hand held double beater electric whisk (Argos value range, blade size 6 cm) on speed setting 1 (reaching 1100 rpm). The whipping technique included 5 min whipping following by a 5 min resting interval. The procedure was repeated for a total period of 45 min, i.e. actual whipping time of 25 min. At the resting intervals, digital photographs were taken to monitor the foamability of the samples. At the end of whipping, a homogeneous sample of the whipped oil was transferred into volumetric cylinders or glass beakers to monitor its foam stability under the same temperature conditions. The samples were photographed at fixed times to record the foam volume for at least one month.

### 2.4. Tristearin in tricaprylin dispersion and aeration

2.12 g of tristearin was added to 10 mL of tricaprylin and heated to 75 °C to ensure that the tristearin was fully dissolved. The transparent oil dispersion prepared at 75 °C was gradually left to cool to 15 °C at an approximate rate of 2 °C/min. At 15 °C, a semi-solid white opaque oil dispersion was formed. At this temperature, the aeration of the sample was achieved with the use of an IKA Ultra-Turrax T25 homogeniser equipped with a metal head of diameter of 18 mm (due to the low volume of this dispersion). This included 5 min at 13,000 rpm followed by a

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