



Microstructural basis for water release from glycerol monostearate structured emulsions upon transformation from the α -gel to the coagel phase



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ARTICLE INFO

Article history:

Received 13 September 2015
Received in revised form 31 October 2015
Accepted 11 November 2015
Available online 26 November 2015

Chemical compounds studied in this article:

Glycerol monostearate (PubChem CID: 24699)
Sodium stearoyl lactylate (PubChem CID: 23671849)
Xanthan gum (PubChem CID: 7107)

Keywords:

Monoglyceride structured emulsion
Polymorphic phase behaviour
SEM
Surface morphology

ABSTRACT

This work characterized changes in surface morphology of a glyceryl monostearate structured emulsion (MG-structured emulsion) during the polymorphic transformation from the α -gel phase to the coagel phase. The effects of using various concentrations of co-emulsifier and adding xanthan gum on emulsion structure and stability were also investigated. Microscopy methods, including light microscopy, cryogenic-scanning electron microscopy, and X-ray diffraction were employed in the examination of the emulsion microstructure. Results showed that the MG-structured emulsion has a smooth droplet surface in the α -gel phase and a porous surface in the coagel phase. The formation of a porous surface during the polymorphic transformation leads to water release upon emulsion destabilization. Formulating with higher content of co-emulsifier or adding xanthan gum enhanced the multi-lamellar structure and stability of this structured emulsion.

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1. Introduction

In recent years, many attempts have been made to develop food products that are free in partially hydrogenated oils (PHOs) and low in saturated fats. A monoglyceride (MG) structured oil-in-water emulsion has been developed as a low-fat shortening (Marangoni & Idziak, 2010; Marangoni et al., 2007) and was tailored to provide advantageous mechanical properties for applications in cookies (Goldstein & Seetharaman, 2011), breads (Calligaris, Manzocco, Valoppi, & Nicoli, 2013), and laminated puff pastries (Blake & Marangoni, 2015). These MG-structured emulsions have a fat-like gel network that is stable at room temperature for up to two months, after which water separation was observed, likely caused by changes in emulsion structure (Goldstein, Marangoni, & Seetharaman, 2012). The MG-structured emulsions consist of oil droplets surrounded by alternating MG bilayers and water (Batte, Wright, Rush, Idziak, & Marangoni, 2007; Marangoni et al., 2007). The change in emulsion structure is a result of the

polymorphic transformation of MGs. Therefore the polymorphic behaviour of MG-water system could help in predicting the mechanism of the emulsion destabilization, because the MG-water bilayers encapsulate the oil droplets in the emulsion structure.

The polymorphic properties of saturated MGs in water (Cassin, de Costa, van Duynhoven, & Agterof, 1998; Larsson, Fontell, & Krog, 1980; Larsson & Krog, 1973; Sein, Verheij, & Agterof, 2002; van Duynhoven et al., 2005; Wang & Marangoni, 2014; Wang & Marangoni, 2015a; Zetzel, Ollivon, & Marangoni, 2009), in oil (Chen, Damme, & Terentjev, 2009; Chen & Terentjev, 2009; Ojijo, Kesselman, et al., 2004; Ojijo, Neeman, Eger, & Shimoni, 2004), and MG-structured emulsions (Batte et al., 2007; Mao, Calligaris, Barba, & Miao, 2014; Mao, Roos, & Miao, 2013; Verstringe, Moens, De Clercq, & Dewettinck, 2015; Wang & Marangoni, 2015b) have been well characterized. In glyceryl monostearate (GMS) structured water systems, a hexagonally packed liquid crystalline lamellar phase (L_α) is formed when heating above the Krafft temperature (T_k) of GMS. Cooling below T_k leads to the formation of a structured L_β phase (α -gel phase), in which thick layers of water are retained between the lamellar structures formed by GMS molecules (Krog, 1997). Further cooling the α -gel phase below 13 °C leads to a thermally reversible phase transformation to the

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sub- α -gel phase (Wang & Marangoni, 2014). However the α -gel phase is thermodynamically unstable and will gradually undergo a polymorphic transformation into the more densely packed L_B , (coagel) phase, accompanied by a release of water (Cassin et al., 1998; Krog & Borup, 1973; Sein et al., 2002). The high water swelling capacity of the α -gel phase provides MG-structured emulsions their fat-like structure, while the formation of the coagel phase leads to destabilization of the structured emulsion and loss of their desirable physical properties.

Using powder X-ray diffraction (XRD), the lamellar packing and polymorphic form of MG-structured emulsions can be characterized. The α -gel phase can be characterized by a single d-spacing at ~ 4.2 Å in the wide angle scattering (WAXS) region and a spacing representing the (0 0 1) plane at ~ 55 Å in the small angle scattering region (SAXS) (Batte et al., 2007; Wang & Marangoni, 2015b). On the other hand, several diffraction spacings between 3.6 and 4.6 Å in the WAXS region and a spacing representing the (0 0 1) plane at 49 Å in the SAXS region represents that the system is in the coagel phase (Batte et al., 2007; Wang & Marangoni, 2015b).

Previous studies by our group investigated the kinetics of the polymorphic transformation of MG-structured emulsions, and have shown that the stability of this MG-structured emulsion could be improved by using co-emulsifiers such as sodium stearyl lactylate (SSL) and by adding xanthan gum (Wang & Marangoni, 2015b). Moreover, literature suggests that the polymorphic transformation from the α -gel phase to the coagel phase takes place through the formation of strong hydrogen bonds between the head groups of MG-molecules and by the separation of L and D isomers of MG-molecules (Cassin et al., 1998; Chen & Terentjev, 2009; Gehlert, Vollhardt, Brezesinski, & Mohwald, 1996; Heertje, Roijers, & Hendrickx, 1998; Sein et al., 2002). However, the mechanism of how changes in the structure and surface morphology in MG-structured emulsions cause the micro scale release of water during the polymorphic transformation is not well understood. The effect of the addition of co-emulsifiers and hydrocolloids on the structure and surface morphology of the emulsions in the micro scale is also not characterized.

The objective of this work, therefore, is to characterize the change in the microstructure of GMS-structured emulsions during the polymorphic transformation from the α -gel phase to the coagel phase using microscopic methods. Differences in the emulsion structure and stability using three formulations will also be examined.

2. Materials and methods

2.1. Sample preparation

The structured emulsion contained deionized water, oil, GMS, SSL, potassium sorbate and xanthan gum. The oil phase used in the emulsion was Neobee[®] M-5 oil from Stephan Company kindly supplied by Charles Tennant & Company (Canada) Limited (Weston, ON, Canada). This oil is a caprylic/capric triglyceride that contains 66% w/w C8:0 and 32% w/w C10:0. The distilled GMS used was Alphadim 90 SBK and the co-emulsifier SSL used was Emplex Sodium Stearyl Lactylate, both were provided by Caravan Ingredients (Lenexa, KS, USA). The xanthan gum was FASTir[®] Xanthan EC from TIC GUMS (White Marsh, St. Jos), and the potassium sorbate was from Sigma-Aldrich Canada Co. (Oakville, ON).

MG-structured emulsions were prepared following the methods used by Wang and Marangoni (Wang & Marangoni, 2015b). The emulsion samples contain 70% (w/w) water phase and 30% (w/w) oil phase. The oil phase contained 25% (w/w) of oil, 5% (w/w) of emulsifiers mixed of 19:1 (w/w) of MG: SSL or 9:1 (w/w) of MG: SSL. The water phase contain 0.1% (w/w) potassium sorbate. One

set of emulsion structured with 1:19 SSL: GMS contained 0.07% (w/w) of xanthan gum in the water phase. Both oil phase and water phase were heated to 75 °C to melt or dissolve GMS and SSL. The oil phase was then added to the water phase upon homogenization using a KitchenAid[®] two speed immersion blender (Whirlpool Corporation, St. Joseph, MI, USA) set at speed one and mixed for around one minute until achieving a desirable consistency. Emulsions were immediately transferred into glass jars and cooled on a bench top without shear. Samples were sealed and incubated at 45 °C for stimulated shelf life study.

Samples contain 19:1 (w/w) of MG: SSL, 9:1 (w/w) of MG: SSL, and 19:1 (w/w) of MG: SSL with xanthan gum will be called control, high SSL, and xanthan emulsion respectively in this work.

2.2. Transmitted light microscopy

MG-structured emulsions were observed using bright field transmitted light microscopy at room temperature weekly throughout the storage period. Emulsion sample were prepared on glass microscope slides at certain time points throughout the incubation period. Samples were observed with a Leica DMRXA2 microscope (Leica Microsystems Canada Inc., Richmond Hill, Canada), and images were taken with a CCD camera (RETIGA 1300i, Burnaby, BC, Canada) controlled by Volocity 6.2.1 software (PerkinElmer, Woodbridge, ON, Canada).

2.3. Cryogenic-scanning electron microscopy (cryo-SEM)

Emulsion samples were mounted on a copper holder designed for the Emitech K550 Cryo-preparation unit (Ashford, Kent, UK) using TissueTak[®]. The copper holder was plunged into liquid nitrogen slush (-207 °C) and then withdrawn from the freezing chamber through argon. Samples were transferred frozen and under vacuum into the preparation chamber of the cryo-unit for sublimation at -80 °C for 45 min. After sublimation, samples were then coated with 30 nm of gold (Emscope K1250x cryo-preparation system, Ashford, Kent, UK) and transferred frozen and under vacuum to the SEM (Hitachi S-570, Tokyo, Japan) cold stage held at -137 °C for image capturing. Images were captured digitally using the Quartz PCI imaging software (Quartz Imaging Corp., Vancouver, BC, Canada).

2.4. Powder X-ray diffraction

The lamellar spacing and polymorphic forms of the emulsions were characterized with a Rigaku Multiflex X-ray Diffractometer (RigakuMSC Inc., The Woodlands, TX, USA) following the method used by Wang and Marangoni (Wang & Marangoni, 2015b).

3. Results and discussion

The control, high SSL, and xanthan gum emulsions showed a similar creamy texture upon preparation when freshly made. Samples structured with high SSL and with xanthan gum preserved their original consistency after four weeks of incubation at 45 °C (Fig. 1a). After eight weeks of incubation at 45 °C, the control sample showed changes in its texture (Fig. 1b) as a sign of destabilization, wherein chunks were formed and the continuous phase became watery.

All MG-structured emulsions had an even distribution of droplet sizes when freshly prepared. Transmitted light microscopy images of emulsion samples taken throughout the incubation period are shown in Fig. 2a. After four weeks of storage at 45 °C, the control sample showed water exudation and phase separation (Fig. 2b). Even though a similar consistency was observed by eye after four weeks, emulsions containing xanthan gum (Fig. 2d) displayed bigger droplet sizes than emulsions containing high SSL

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