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Effect of oil viscosity on oil migration in a two-phase model system (cream-filled chocolate)

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ABSTRACT

Using tricaprylin, flaxseed oil, safflower oil, and peanut oil, we investigated the effect of oil viscosity on oil migration in a two-phase model system, to understand oil migration in cream-filled chocolate products. Non-tempered cocoa butter was used as the matrix, while interesterified hydrogenated palm oil mixed with each of the oils (60:40 w/w) was used as the cream phase. Oil migration was monitored using a flatbed scanner. Results of our study showed that tricaprylin, which had the lowest viscosity of about 37.2 mPa·s at 18 °C, exhibited the highest oil migration rate and diffusion coefficient; while flaxseed oil, safflower oil, and peanut oil with viscosities of about 60, 73.6, and 97.2 mPa·s at 18 °C, respectively, showed low oil migration rates and diffusion coefficients. These findings support the fact that as oil viscosity increases, oil migration rate decreases. Furthermore, a critical viscosity of above 60 mPa·s was found to significantly decrease oil migration rate, and therefore extend the shelf life of cream-filled chocolate products. The current study provides an experimental evidence and practical information to confirm existing knowledge, which is valuable for the understanding of oil migration in cream-filled chocolate products.

1. Introduction

Chocolate is a composite matrix comprised of fat (mainly cocoa butter or CB) as the continuous phase, with cocoa solids, sugar, and other ingredients as filler particles. The fat phase, CB, is composed of numerous saturated and unsaturated fatty acids like stearic acid (S), palmitic acid (P), and oleic acid (O) that make-up triacylglycerols (TAGs) such as POP, POS and SOS which are the most abundant TAGs in CB (Loisel, Keller, Lecq, Bourgaux, & Ollivon, 1998; Marty & Marangoni, 2009; Sato et al., 1989). In CB, liquid fat (mainly consisting of low-melting TAGs and some dissolved high-melting TAGs) co-exists with solid fat (primarily high-melting TAGs) at certain temperatures. This liquid fat is able to migrate to the surface of the chocolate product (possibly solubilizing some higher melting TAGs in the CB matrix) which can then recrystallize, resulting in bloom formation (Aguilera, Michel, & Mayor, 2004; Ghosh, Ziegler, & Anantheswaran, 2002; Lonchampt & Hartel, 2004; Timms, 1984). Fat bloom is the most common defect in chocolate and chocolate coated/filled products. Oil migration also accelerates other undesirable changes in chocolate such as softening and loss of snap (solubilization of TAGs in the CB matrix), and improper melt (transition from the β-V polymorph to the β-VI form) that then decrease the marketability of chocolate products. Several factors affect oil migration in chocolate, of which the most important are temperature fluctuation during handling and storage, and exposure to high temperatures. Other factors include physical properties of the matrix such as microstructure, presence of cracks or pores, particle size of the non-fat ingredients (cocoa solids and sugar), compatibility of CB with other fats and oils (*e.g.*, in cream filled chocolates), tempering, and CB origin (Afoakwa, Paterson, Fowler, & Vieira, 2009; Ali, Selamat, Che Man, & Suria, 2001; Ghosh et al., 2002; Maleky, Mccarthy, Mccarthy, & Marangoni, 2012; Marty & Marangoni, 2009; Miquel, Carli, Couzens, Wille, & Hall, 2001; Rousseau & Smith, 2008; Sonwai & Rousseau, 2010). Numerous techniques have also been utilized to monitor or explain the kinetics of oil migration in chocolate such as determining solid fat content using pulsed nuclear magnetic resonance (pNMR) and differential scanning calorimetry (DSC), monitoring polymorphism using X-ray diffraction (XRD), determining fatty acid composition using high performance liquid chromatography (HPLC) and gas liquid chromatography (GLC), and visualizing oil migration using magnetic resonance imaging (MRI)

Abbreviations used: CB, cocoa butter; POP, palmitic, oleic, palmitic; POS, palmitic, oleic, stearic; IHPO, interesterified hydrogenated palm oil.

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and a flatbed scanner (Khan & Rousseau, 2006; Marty, Baker, Dibildox-Alvarado, Rodrigues, & Marangoni, 2005; Miquel et al., 2001; Sonwai & Rousseau, 2010; Walter & Cornillon, 2002). The kinetics of oil migration in chocolate has been extensively characterized using Fick's law of diffusion (Aguilera et al., 2004; Delbaere, Van de Walle, Depypere, Gellynck, & Dewettinck, 2016; Galdámez, Szlachetka, Duda, & Ziegler, 2009; Khan & Rousseau, 2006; Lee, McCarthy, & McCarthy, 2010; Marty, Baker, & Marangoni, 2009).

In cream-filled chocolate products, besides compatibility and interaction with cocoa butter (*e.g.,* amount of contact at the interface), the composition of the oil that makes up the cream also affects oil migration (Delbaere et al., 2016; Ghosh et al., 2002; Khan & Rousseau, 2006; Lee et al., 2010; Marty et al., 2005; Miquel et al., 2001; Rousseau & Smith, 2008; Walter & Cornillon, 2002). In this study, the effect of oil viscosity on oil migration using a model cream-filled cocoa butter matrix will be investigated. Furthermore, the current investigation aims to provide experimental evidence to the established concepts about oil migration in cocoa butter matrices.

2. Materials and methods

2.1. Viscosity of oil samples

The viscosity of flaxseed oil (Nealanders International Inc., Oakville, ON, Canada), safflower oil, peanut oil (purchased at a local grocery store), and tricaprylin (Sigma, St Louis, MO, USA) was measured with a Brookfield Model RVF 100 Synchroletric viscometer (D.W. Brookfield Ltd., Cooksville, ON, Canada).

2.2. Sample preparation

The effect of oil viscosity on oil migration was studied in a two-phase model system described by Marty and Marangoni (2009). A layer of non-tempered cocoa butter (Masterfood, Slough, UK) was used as a matrix while chemically interesterified hydrogenated palm oil (IHPO) (Noblee & Thörl, Hamburg, Germany) mixed with each of the oils chosen above (60:40, w/w, IHPO:oil) was used as the cream phase. The fatty acid composition of the oils and CB used in the study are shown in Table A (Appendix A).

The cream phase was stained with Nile red at a final concentration of 0.025 g/100 g. A calibration curve of the pixel intensity as a function of Nile red concentration was constructed to quantify the extent of oil migration. Nile red was added at seven different concentrations (0.005–0.1 g/100 g) and sample mixtures of cocoa butter and cream were analyzed.

In the preparation of the mold for the study, two coverslips were attached on both ends of a microscope slide using a double-sided tape. A few drops (about 30 μL) of molten CB (80 °C for 30 min) were deposited in the middle of the slide, and a third coverslip was placed on top of the CB. This preparation was then stored for 11 d at 18 °C before carefully adding warm (37 °C) stained cream (IHPO with oil) at the interface of the CB matrix.

The polymorphism of the non-tempered CB over 11 d of storage at 18 °C prior to addition of cream was monitored using a Rigaku Multiplex Powder X-ray diffractometer (Rigaku Corporation, Japan) with a 0.5° divergence slit, a 0.5° scatter slit, and a 0.3 mm receiving slit. The instrument was operated at 40 kV and 44 mA. Scans were executed from 1° to 30° at 1°/min at 18 °C. Scattering intensity reported as a function of the inter-planar distance (d (Å)) was analyzed by MID's Jade 6.5 software (Rigaku Corporation, Japan). Results are shown in Fig. B (Appendix B).

Diffusion of the stained cream through the CB matrix was then monitored using a flatbed scanner. Images were acquired daily for 28 d.

2.3. Flatbed scanner imaging technique

The scanner imaging technique used in this study has been extensively described by Marty and co-workers (Marty & Marangoni, 2009; Marty et al., 2009, 2005).

2.3.1. Image acquisition and image analysis

A flatbed scanner (Hewlett-Packard Scanjet G 4010) was used for image acquisition with a resolution of 1200 pixels per inch. Images acquired in red green blue (RGB) color space were transformed into a hue saturation lightness (HSL) color space using Adobe Photoshop® 8.0 (Adobe Systems Inc., San Jose, USA) combined with Fovea Pro plugs-ins (Reindeer Graphics Inc., North Carolina, USA). The saturation channel was extracted and then converted to an 8-bit grey scale image. Image J (National Institutes of Health, USA) was used to measure the pixel intensity within a rectangular region on each image to obtain a graph of pixel intensity and distance. The distance was calibrated using the known dimensions of the moulds and the microscope slides. The areas containing the fluorescent dye appear lighter than the dye-free matrix, approximately 255 and 0 on the pixel intensity scale, respectively.

2.3.2. Determination of oil migration rate

Image intensity was normalized and analyzed using GraphPad Prism 4 (GraphPad Software, Inc., San Diego, USA). The reading of the 10% maximum (I_{10}) value was used to measure the oil moving front for each day. The I_{10} values were plotted against both time and square root of time and the slopes were used to define oil migration rates (m/s or m/$\sqrt{s}$).

2.3.3. Determination of the diffusion coefficient, D

From normalized curves of pixel intensity and distance, area under the curves were determined and reported in graphs as a function of square root of time. Assuming no oil migration at day 0, the day 0 values have been corrected to 0.

The Nile red concentration has been specifically chosen so that the ratio of intensities is comparable to a ratio of mass, as described by (Marty et al., 2005)

$$I_t/I_\infty \approx m_t/m_\infty \quad (1)$$

where I_t is the intensity measured at time t; I_∞ is the intensity at equilibrium, m_t is the mass of oil migrated at the same time t, and m_∞ is the mass migrated at equilibrium. Using this assumption and the simplified equation based on Fick's second law proposed by Ziegleder and co-workers (Ziegleder, Moser, & Geier-Greguska, 1996b, 1996a; Ziegleder, 1997), diffusion coefficients were determined by

$$\frac{m_t}{m_\infty} = \frac{\sqrt{D}}{l}\sqrt{t} \quad (2)$$

where l represents the maximum distance reached at day 28, D represents the coefficient of diffusion (m^2/s) and t represents time.

2.4. Differential scanning calorimetry (DSC)

The stained cream, and the interface and end regions of the CB matrix were analyzed using DSC after 0, 7, 14, 21 and 28 d. A DuPont Model 2910 differential scanning calorimeter (TA Instrument, Mississauga, ON, Canada) was used to determine the melting property of the samples. A small quantity of sample (6–12 mg) was placed in an aluminium DSC pan (TA Instruments, Mississauga, ON, Canada) which was hermetically sealed. An empty pan was used as reference. Temperature ramp tests were performed at a rate of 5 K/