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Development of an extraction protocol for the removal of the fat phase within chocolate

Dérick Rousseau*, Aliénor Coutouly, Patrick Hendricks, Shane Hodge, Nicole L. Green

Department of Chemistry and Biology, Ryerson University, 350 Victoria Street, Toronto, Ontario, M5B 2K3, Canada

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ABSTRACT

An extraction method to remove the cocoa butter fat phase from chocolate while leaving the dispersed particulate network intact was developed. Three parameters were evaluated: i) the method of addition of small amounts of water to the chocolate during its preparation; ii) the contact method of a solvent (petroleum ether) with the fat phase; and iii) the duration of fat phase extraction. The addition of water was necessary to retain the structure of the backbone particulate network during extraction. The absence of water resulted in dispersed particulates that fell apart, suggesting that the water aided in network preservation. Water in the form of an emulsifier-stabilized water-in-oil emulsion was the most efficient means of addition. Optimal fat extraction and structure retention were achieved with a combined capillary/vapor phase method. Extraction times upwards of 24 h were necessary to extract sufficient cocoa butter from the chocolate for particulate network analysis. Characterization with scanning electron microscopy and confocal microscopy confirmed the adequacy of the developed extraction method and demonstrated that the backbone structure consisting of dispersed particulates could be made into a self-supporting network.

1. Introduction

Plain chocolate consists of a mixture of micron-scale particulates (sugar crystals and cocoa powder) dispersed within a continuous fat phase. This cocoa butter (CB) phase is central to the enjoyment of chocolate, allowing chocolate to remain solid at ambient temperature and melt at body temperature (Beckett, 2000). Much like industrial concrete, the CB fat phase in chocolate is thought to act as the cement holding the dispersed particulates in place. Particulates are typically 5–30 μm in diameter, small enough to avoid a gritty mouthfeel when chocolate is eaten (Afoakwa, Paterson, & Fowler, 2007; Soulié, El Youssoufi, Cherblanc, & Saix, 2006). The sensory properties of chocolate such as a glossy surface, melting profile, and texture depend on the quality of the tempering process. Tempering is a highly controlled solidification protocol of the fat phase that promotes the crystallization of CB into the desired polymorphic form and morphology (Beckett, 2000).

The purpose of this study was to develop an effective extraction method to remove the CB fat phase from chocolate while leaving the dispersed particulate network intact. Such efforts would permit examination of the role of the dispersed particulates on the rheological and textural properties of heat resistant chocolate. Preliminary experiments indicated that the addition of moisture was necessary for successful fat phase extraction. The effect of moisture on granular materials has been studied extensively (Bika, Gentzler, & Michaels, 2001; Hornbaker, Albert, Albert, Barabasi, & Schiffer, 1997; Iveson, Litster, & Ennis, 1996; Kohonen, Geromichalos, Scheel, Schier, & Herminghaus, 2004; Willett, Adams, Johnson, & Seville, 2000), where it has been observed that increasing the moisture content will consolidate loose agglomerates through adhesive forces associated with interstitial liquid bridges between grains. These liquid bridges are responsible for capillary bonding between grains. As a result, when wet, the mechanical properties of the granular material change compared to its dry state. To illustrate, when sand becomes wet, it is able to form shapes and structure (i.e., sandcastles) that would otherwise be unstable in dry sand. These capillary bridges have been of recent interest with potential in engineering low fat or heat resistant chocolate (Hoffmann, Koos, & Willenbacher, 2014; Stortz & Marangoni, 2011).

In this case, we work in the reverse direction - removing fat from preexisting chocolates. Prior research has used a model chocolate system of just sugar and fat (Killian & Coupland, 2012), while we

* Corresponding author. Tel.: +1 416 979 5000x2155; fax: +1 416 979 5044.
E-mail address: rousseau@ryerson.ca (D. Rousseau).

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are interested in the effectiveness of our extraction method in virtually unadulterated chocolates. In developing an extraction method to remove the CB fat phase from chocolate, three factors were assessed: solvent contact method, experimental duration, and water addition technique. To extract the fat phase within the chocolate and expose the particulate network for subsequent microstructural characterization, petroleum ether (PET) was used. PET is a non-polar solvent often used for lipid extraction in food (Timms, 2003). More polar solvents were not as effective for fat phase extraction. The method of contact between the solvent and chocolate was examined, namely direct contact, vapor, and a combination of the two. The duration of CB extraction was determined as well, with the fat phase extracted for up to a week. Finally, the method of water addition (up to 4 wt% of the chocolate) during its preparation was considered, using either a direct spray method or incorporation via a water-in-oil (W/O) emulsion.

2. Materials and methods

2.1. Materials

The chocolate used for this study was provided by Cadbury Chocolates (Toronto, ON, Canada). Its ingredients included sugar, CB, unsweetened chocolate, and soy lecithin. The CB fat content for milk chocolate was 30%. Cadbury also provided raw CB. RO water was used throughout this work. Petroleum ether (PET) was used as received from Fisher Scientific (Toronto, ON, Canada). Polyglycerol polyricinoleate (PgPr) was provided by Nealanders (Mississauga, ON, Canada).

2.2. Sample preparation

By employing a lab-scale temperer (Tenon Engineering LTD, Leatherhead, UK), the chocolate was tempered to make the entire chocolate crystallize into the form V polymorph using the protocol shown in Fig. 1. For each experiment, 400 g of chocolate was melted at 50 °C for 1 h, then the temperature was reduced to 27 °C and held there under continuous stirring for 10 min to induce crystallization. The chocolate was then heated to 32 °C for 5 min to ensure that the unstable polymorphs melted and only form V seeds remained.

At this stage, water was added in one of two approaches: bulk addition or incorporation via a W/O emulsion. For the former, a spray bottle yielding finely-dispersed droplets was used to add water at the end of the tempering stage. The spray bottle was weighed regularly to determine the amount of water added. Water was also added to the chocolate as a W/O emulsion consisting of CB (47.5 wt%), water (47.5 wt%), and PgPr (5 wt%). Emulsification took place in a two-stage valve homogenizer (APV-1000, SPX Corporation, Brockville, ON, Canada) operating at 68.9 MPa, and the resulting emulsion was stored at 50 °C to prevent undesirable CB crystallization. Droplet size distribution of the emulsified water was determined at 25 °C using a Bruker Minispec Mq pulsed field gradient nuclear magnetic resonance (pfg-NMR) unit (Bruker Canada, Milton, ON, Canada) that allows unimodal characterization of emulsion droplet size distributions via restricted diffusion measurement.

The reported droplet size was averaged over three data sets. Amounts equivalent to 14 wt% water were added to the chocolate. For example, for a chocolate mass of 400 g to contain 4 wt% water, 34 g of emulsion were added. Beyond 4 wt% water, its negative impact on flavor and texture were too great. Dispersion of the emulsified water was ensured by pouring the post-tempered chocolate into a laboratory-scale blender and blended for 10 s.

The mixture was then poured onto a plastic kitchen cutting board and manually spread to remove air bubbles. Lastly, the chocolate mass was poured into cylindrical molds (h = 4 mm, D = 35 mm) followed by cooling at 4 °C for 30 min and storing at 17 °C until use (24 h).

2.3. Extraction protocol

For the fat phase extraction, chocolate disks were placed on a piece of circular Whatman filter paper with 47 mm diameter, 8 μm pore size. This was then placed on a second piece of Whatman filter paper with 150 mm diameter and 20–25 μm pore size. The larger filter paper was cut and folded to optimize extraction efficiency (Fig. 2). The chocolate and filter papers were set on a rectangular metal grid inside a 600 mL beaker. 200 mL of PET was poured into the beaker without contacting the filter paper atop the metal grid. The end of the cut section of the large filter paper was immersed in PET, leading to chocolate-solvent contact via capillarity. This allowed for more gentle fat extraction while retaining the

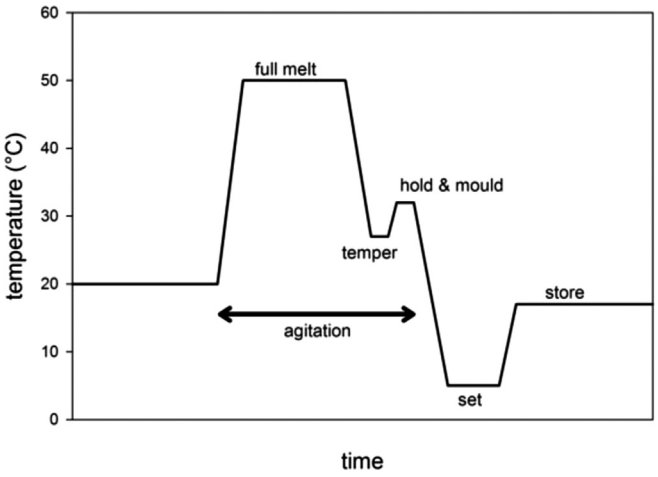

Fig. 1. Tempering procedure to generate the required fat phase crystal polymorph.

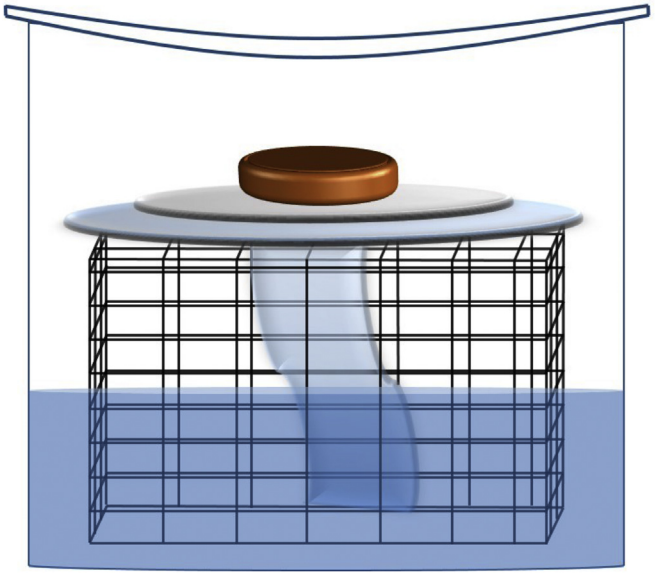

Fig. 2. Schematic of fat phase extraction method. Chocolate disk sits atop a smaller filter paper used to measure fat extraction and a larger filter paper cut such that a strip hangs into the PET solvent.