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Changes in the volatile profile of skim milk powder prepared under different processing conditions and the effect on the volatile flavor profile of model white chocolate

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ABSTRACT

The objective of this work is to determine the extent to which changes in the skim milk powder (SMP) manufacturing process alter the volatile profile of SMP, and whether these changes are carried through to a final product when the SMP is used as an ingredient and subjected to further processing. The manufacture of SMP is a multistage process involving a preliminary concentration step, heat treatment, and a drying stage. However, the methods and conditions used by the industry are not standardized, and the inherent variability in the production of SMP has consequences for the end-users, such as the confectionery industry, where the SMP is used as an ingredient during the production of milk chocolate, white chocolate, and caramel. This study investigates the effect of each stage of the manufacturing process on the concentration of reducing sugars and available amino groups (as precursors of the Maillard reaction) as well as on the volatile products of the Maillard reaction and lipid degradation. Eight types of SMP were produced using combinations of different processing conditions: concentration (by evaporation or reverse osmosis), heat treatment (low heat or high heat), and drying (spray-drying or freeze-drying). Maillard precursors were quantified after each processing stage and volatile compounds were extracted using solid-phase microextraction, and analyzed by gas chromatography-mass spectrometry. The resulting SMP were incorporated into a model white chocolate system, produced under varying conching conditions. We demonstrate not only that changes in the SMP manufacturing conditions affect the volatile profile of SMP, but also that these differences can be carried through to a final product when the SMP is used to prepare a model white chocolate. Understanding these

differences is important to the industry for controlling the flavor of the end product.

Key words: manufacture of skim milk powder, flavor, spray dry, freeze dry, chocolate

INTRODUCTION

The manufacture of skim milk powder (SMP) is a multistage process involving a preliminary concentration step; heat treatment, which is often included to control the functional properties of the final powder (Oldfield et al., 2005); and a drying stage. Because these all involve a rise in temperature, lipid degradation and the Maillard reaction can occur during any of these steps. The severity of the heat treatment applied to milk during milk powder production is classified by industry according to the levels of undenatured whey proteins present (i.e., whey protein nitrogen index). High heat powder, medium heat powder, and low heat powder have whey protein nitrogen index ranges of <1.5, 1.5–6.0, and >6.0 mg/g, respectively, and these can be achieved using a range of different time temperature combinations. Low-heat SMP is typically treated at 75°C for 20 s, whereas medium heat conditions range from 85 to 105°C for 1 to 2 min, and high heat up to 135°C for 2 to 3 min (Early, 1998). Given this range of conditions, the extent of the Maillard reaction in SMP is variable. An understanding of the critical control points during the manufacturing process is important to industries that require a consistent product.

The changes in milk powder during storage are well documented (Hurrell et al., 1983; Driscoll et al., 1985; Karagül-Yüceer et al., 2002, 2003; Drake et al., 2006). Most studies show that the formation of lipid-derived volatiles is prevalent during storage, contributing to the development of off-notes, but both formation and loss of Maillard reaction products were reported, depending on the conditions.

Research on high-temperature processes in milk tends to focus on UHT (Morales et al., 1992; Celestino et al., 1997; Romero et al., 2001; Valero et al., 2001;

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Tokuşoğlu et al., 2004) and sterilization (Contarini et al., 1997). The formation of Maillard intermediates and glycation products during manufacture of dairy products has been studied (Birlouez-Aragon et al., 2004; Erbersdobler and Somoza, 2007; Cattaneo et al., 2008), but the focus of these studies was the reduction in nutritional value as a result of lysine residues becoming unavailable (Mehta and Deeth, 2016). The development of volatile aroma compounds during the production of milk powder was studied by Drake et al. (2006) who showed that Maillard-derived compounds such as 2-acetylpyrrole, 2-acetylthiazole, and 2-acetyl-2-thiazoline increased, whereas little change occurred in the profile of the lipid degradation products. However, Li et al. (2012) monitored volatile lipid oxidation compounds during the production of milk powder, and demonstrated that all stages of the process could influence the formation and stability of these volatiles.

Recently, the role in flavor formation of the individual unit operations have been investigated. Falling-film evaporators are used extensively in the dairy industry, and evaporation (**EV**) under vacuum results in the milk being heated to a lower temperature. Other concentration methods include membrane separation techniques such as reverse osmosis (**RO**; Glover, 1985), which operates at high pressure and temperatures below those reached during EV. Comparison of RO, nanofiltration, and UF was discussed by Syrios et al. (2011) with regard to stability, pH, calcium content, and gel formation. Park and Drake (2016) showed that concentration by RO, compared with concentration by EV, retained far more of the sweet character of the milk, driven by a greater retention of most volatiles, particularly lactones and furaneol. Maltol, however, showed the reverse trend. Park et al. (2016) also showed significant changes in the volatile profile when different spray-drying parameters were employed. They showed that the sweet aromatic note increased as the inlet temperature increased, and this correlated with an increase in some lactones, maltol, and vanillin.

Given the significant changes in SMP brought about by different processing conditions, it is important to understand if these changes are reflected in the final products when SMP is used as an ingredient for the manufacture of more complex food products. Caudle et al. (2005) showed a decrease in consumer acceptability of SMP as the storage time increased up to 4 yr, and when these SMP were incorporated into ice cream, yogurt, and white chocolate (but not hot chocolate), a similar decrease in consumer acceptability was observed. Volatile analysis of the SMP showed an increase in dimethyl sulfide and dimethyl disulfide, and a decrease in maltol. Lloyd et al. (2009b) carried out a

similar experiment with stored WMP incorporated in white and milk chocolate and showed a similar decrease in consumer acceptance, which was attributed to an increase in lipid degradation products. Recently, Stewart et al. (2017) showed that the heat treatment applied during SMP manufacture leads to both changes in the aroma profile of the SMP, and flavor changes in white chocolate prepared from the resulting SMP.

The aim of this work was to investigate different processing conditions during the production of SMP to determine the key stages for flavor development, and to determine whether these changes are carried through to a final product. Eight types of skim milk powders were produced using combinations of different processing conditions. Maillard precursors (sugars and AA) and aroma compounds were quantified after each stage. The SMP were incorporated into a model white chocolate system and heated to mimic conching to determine the effect of milk processing methods on the flavor profile of a final confectionery product.

MATERIALS AND METHODS

Chemicals

Trehalose, glucose, galactose, lactose and lactulose, L-leucine, sodium hydroxide (50% solution in water; 1.515 g/mL), SDS, ethanol, *o*-phthaldialdehyde (**OPA**), 2-mercaptoethanol, sodium tetraborate buffer solution (pH 9), 1,2-dichlorobenzene, methanol, all aroma chemical, alkanes C₅–C₃₀, and diethyl ether were obtained from Sigma-Aldrich Co. (Dorset, UK). The EZ:Faast amino acid analysis kit was purchased from Phenomenex (Macclesfield, UK).

Preparation of Milk Powders

Raw whole bovine milk (**RWM_{RO}**; 40 kg) supplied by The University of Reading CEDAR Dairy Farm (CEDAR, Reading, UK) was pasteurized at 72°C for 15 s and separated using a disc bowl centrifuge. The skimmed pasteurized milk (**PM_{RO}**) was then concentrated to 20% TS using RO to produce concentrated milk (**CM_{RO}**). Half of the concentrated milk was then subjected to a heat treatment stage to give a heated concentrated milk (**HCM_{RO}**), and no heat treatment was applied to the other half. The concentrated milks were then spray-dried (**SD**) or freeze-dried (**FD**) to produce the following milk powders: **SDMP_{RO}**, **HSDMP_{RO}**, **FDMP_{RO}**, and **HFDMP_{RO}**. A second batch of raw whole milk (**RWM_{EV}**) was obtained one week later from the same herd, and the process was repeated concentrating to 20% solids using EV to produce a second

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