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#### Microwave vacuum drying of dairy cream: Processing, reconstitution, and whipping properties of a novel dairy product

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#### ABSTRACT

Traditional ways to preserve cream involve processing it into butter, butter oil, or frozen storage. These technologies do not preserve the unique functionality of cream with respect to whipping or processing into butter. In this work, microwave vacuum drying (MVD) was investigated as a method to manufacture dehydrated cream. Dehydrated cream microstructure, color, and free fat were evaluated using scanning electron microscopy, colorimetry, and solvent extraction, respectively. Effects of homogenization on reconstituted cream microstructure and functionality were investigated using confocal laser scanning microscopy, color, particle sizing, and texture analysis of whipped cream. Reconstituted MVD cream whipped faster, and the whipped cream was more cohesive and firmer when 2-step homogenization at 3.5/7 MPa was used. Fat globules in reconstituted MVD cream were covered by phospholipids, explaining MVD cream's similar functionality compared with pasteurized cream. These results may foster the development of novel shelf stable and highly functional dairy products using MVD.

**Key words:** microwave vacuum drying, cream, preservation, reconstitution, whipping cream

#### INTRODUCTION

Significant amounts of cream are produced in the dairy industry from the manufacture of products such as standardized milk, skim milk powder, or NDM. Most of the excess cream is converted into butter for prolonged storage. Although NDM production was projected to increase by almost 80% between 2010 and 2021 (FAPRI, 2011; USDA and ERS, 2022), butter production has seen an increase of only 33% in the same period, leading to an increasing gap between the

amount of cream available and its projected use. In addition, butter requires cold storage, and its production also results in buttermilk, a low-value byproduct that is mostly dried by energy-intensive spray drying. Additionally, the process of churning cream to make butter leads to the loss of the native milk fat globule membrane (**MFGM**) in the buttermilk, and subsequent re-emulsification of butter does not allow to recreate the native or a quasinative fat globule structure and functionality (Rombaut et al., 2006). Therefore, this cream processing pathway is a less than optimal solution to convert excess cream into a sustainable, functional, and high-value product.

Another way to preserve cream is dehydration and conversion into a shelf-stable powder. Manufacturing of dairy powders that contain fat, such as whole milk powder, require the starting raw materials (i.e., standardized whole milk) to be heat treated and homogenized at high pressures to stabilize the milk fat globules by size reduction and coverage with a dense layer of casein and whey proteins (Corredig and Dalgleish, 1996; Cano-Ruiz and Richter, 1997; Lee and Sherbon, 2002; Ye et al., 2008). Unhomogenized milk fat globules would lead to a sticky powder that can clog up the dryer and would lead to final powders with poor wetting and caking due to free fat on the powder particle surface (Sharma et al., 2012). However, homogenization results in irreversible physico-chemical changes to milk fat globules that alter their structure and compromise the techno-functionalities of cream such as whipping, foaming, or phase inversion by churning. Kováčová et al. (2010) showed that UHT heat treatment and homogenization increased cream viscosity and whipping time but decreased whipped cream firmness as compared with pasteurized cream. Therefore, preservation options for milk fat that increase its shelf life while retaining the functionality of milk fat globules better than current methods, ideally at ambient temperature, are needed.

Microwave vacuum drying  $(\mathbf{MVD})$  has recently been investigated as an innovative drying technology for dairy applications that not only provides an alternative

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to traditional drying technologies, but also offers opportunities for the creation of novel functionalities and structures (Ozcelik et al., 2019; Dumpler and Moraru, 2022a; McSweeney, 2022). In addition, MVD offers a range of advantages for cream drying over conventional drying processes such as spray, hot air, or freeze drying: (1) low drying pressure limits lipid oxidation (Yongsawatdigul and Gunasekaran, 1996); (2) drying is rapid due to electromagnetic heat generation (Ambros et al., 2019; Viji et al., 2019); (3) material properties such as stickiness of material are of little relevance compared with spray drying (Silalai and Roos, 2010); and (4) a dense material structure limits oxidation during storage (Ambros et al., 2018).

Dumpler and Moraru (2022b) investigated the effects of MVD on concentrated skim milk functionality. Suitable MVD conditions (including specific power input, drying pressure, and layer thickness) for this liquid dairy product were optimized to retain a high protein solubility in the final product while minimizing drying time and foaming. Dumpler and Moraru (2022a) further investigated the interaction between dielectric properties, drying kinetics, and energy demand for MVD of skim milk. However, little is known about the required pretreatments or optimal processing conditions for MVD for optimal MVD outcomes with respect to functionality for high-fat dairy products such as cream.

Therefore, the aim of this study was to determine the effects of preprocessing, drying, and reconstitution conditions on the functionality of reconstituted MVD cream. Physical properties and the microstructure of fat globules, before drying, after drying, and after reconstitution, were investigated and used to explain the impact of the various processing steps on the MVD cream functionality. Linking the effects of preprocessing and product microstructure of fat containing dairy products with their drying behavior under vacuum using microwaves as a heat source, dry product structure, reconstitution properties, and functionality will help unleash the potential of MVD for a broad range of dairy products, including cream and cheese.

#### MATERIALS AND METHODS

### Homogenization, Evaporation, and Determination of Evaporated Cream Viscosity

Pasteurized heavy cream was purchased from local grocery stores (Ithaca, NY). Cream was diluted to  $36 \pm 0.3\%$  TS using pasteurized skim milk to achieve a fat content of 30%. After heating to 65 to 67°C the cream was homogenized at 1.8, 3.5, 5.3, or 7 MPa, or 7 MPa in the first pass and 3.5 MPa in the second pass (hereaf-

ter: 7/3.5 MPa), using a FT-9 single-stage bench-top homogenizer (Armfield Ltd., Ringwood, UK). The latter 2 homogenization conditions were used for all cream subjected to MVD. After homogenization, the cream was rapidly cooled to  $<10^{\circ}$ C in ice water and stored under refrigeration at  $<5^{\circ}$ C until further processing.

Homogenized cream was then evaporated to about 43.5% TS at 65 to 70°C (20–25 kPa) using a rotary evaporator (Büchi, Flawil, Switzerland) and immediately cooled in ice water. Small amounts of deionized (**DI**) water were added as needed to adjust the solids content to 43.5% TS.

Viscosity of homogenized cream with and without further evaporation was determined as a function of temperature and TS content using a ViscoQC-300L (Anton Paar, Graz, Austria) equipped with a L1, L2, or L3 standard spindle, depending on the expected viscosity, and a 250-mL double-jacketed beaker (inner diameter: 54.5 mm) connected to a thermostatically controlled water bath.

#### Microwave Vacuum Drying of Cream

A small commercial-scale EnWave nutraReV 10-kW microwave vacuum drier (EnWave Corporation, Delta, BC, Canada) was used to carry out the drying experiments, as described by Dumpler and Moraru (2022b). A total of 40 ice cube trays was placed on the carousel in the drying chamber. Volumes of 2 or 3 mL of evaporated cream were pipetted using a Multipette M4 (Eppendorf AG, Hamburg, Germany) into each cavity of the 40 silicone ice cube trays, each of which had 15 cavities measuring  $3.2 \times 3.2 \times 3$  cm. The resulting cream layer thickness was 2 or 3 mm in each cavity. In total, 1,200 or 1,800 mL of evaporated cream were distributed in the 600 cavities. Drying conditions were determined in preliminary trials. A 3-step process was developed, based on the 3 drying stages of cream. It was intended to achieve a fast drying in the first and second drying stage while limiting the maximum product temperature in the last drying stage to  $<65^{\circ}$ C. An example of a programmable logic controller (**PLC**) recording is shown in Figure 1. Product temperatures exceeding 65°C led to leakage of butter oil from milk fat globules in the dried cream (i.e., a very oily, sticky product of dispersed milk solids in butter fat [not shown]). The drying conditions were 1.3 W  $g^{-1}$  for 20 min, 1.0 W  $g^{-1}$  for 10 min followed by 0.67 W  $g^{-1}$  for 30 min at 2.2 to 2.8 kPa. The pressure, microwave power, and time settings of the MVD unit were adjusted to the required values at the human machine interface of the PLC. The rotation speed of the carousel was set to 35% (~3 rpm,  $360^{\circ}$  rotation) in all experiments.

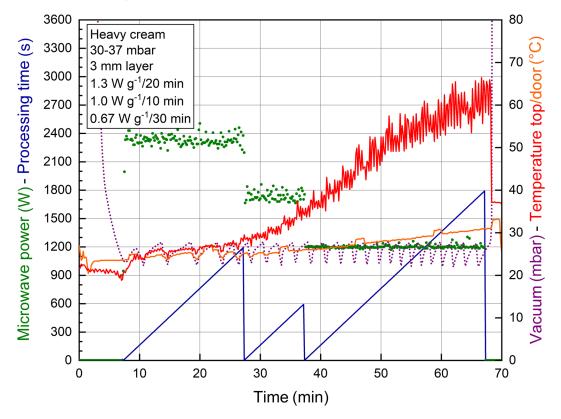


Figure 1. Programmable logic controller recording of a microwave vacuum drying run of cream in the EnWave nutraReV microwave vacuum drier (EnWave Corporation, Delta, BC, Canada). Colors of the axis labels correspond to the plots.

## Moisture, Water Activity, Color, Extractable Fat, and Fat Content

The moisture content of the initial cream, homogenized cream, evaporated cream, and dehydrated cream was determined using a CEM Smart Turbo 5 (CEM, Matthews, NC). Water activity of dehydrated cream was determined using an AquaLab 4TE (Meter Group, Pullman, WA). Color of dehydrated cream and reconstituted cream was determined using a CR-400 colorimeter (Konica Minolta Sensing Americas Inc.) equipped with a Granular Materials Attachment CR-A50 to shield sunlight. All measurements were performed at least in triplicate.

Extractable fat of dehydrated cream samples was determined by the solvent extraction method. In brief, 10 g of sample were weighed in a 125-mL Erlenmeyer flask, 50 mL of petroleum ether (boiling point 20–40°C) was added, and extraction was performed for 15 min at ambient temperature with a magnetic stirrer bar. After extraction, the extract was transferred to a 75mm glass funnel lined with a folded filter (597 1/2, 125 mm; Cytiva, Marlborough, MA) on top of a tared 100-mL flask. The extract and suspended solids were transferred quantitatively by rinsing the flask with another 25 mL of petroleum ether. After filtration, the

Journal of Dairy Science Vol. 107 No. 2, 2024

solvent was evaporated with slight vacuum in a gentle flow of air using the rotary evaporator described above. The extracted butter oil and flask were dried in oven at 104 to 106°C for 30 min. After cooling the flasks in a desiccator for at least 30 min, the flasks were weighed. The percentage of extractable butter fat was calculated from the amount of extractable fat divided by the total fat in the total sample.

Total fat after HCl digestion was determined by weighing 0.75 g of dehydrated cream in a XT4 filter bag (Ankom Technology, Macedon, NY) and adding 0.75 g of diatomaceous earth. Prepared samples were hydrolyzed in a sealed Teflon vessel in batches of 15 using 6 N HCl in an Ankom<sup>HCl</sup> Hydrolysis System (Ankom Technology, Macedon, NY) for 60 min. After acid hydrolysis, solvent extraction (45% petroleum ether, 45% diethyl ether, 10% ethanol) was performed with solvent at 90°C for 60 min using the Ankom<sup>XT15</sup> Extractor. Total fat content was determined by loss of weight. A commercial whole milk powder sample was used as a reference.

#### Scanning Electron Microscopy

Dehydrated cream was attached to a sample holder with silver conductive paint 503 (Electron Microscopy Sciences, Hatfield, PA) and carbon sputtered using a Desk V high vacuum magnetron sputter unit (Denton Vacuum, Moorestown, NJ) for 30 s. Scanning electron micrographs of dehydrated cream were obtained using a Zeiss Gemini 500 scanning electron microscope (Zeiss, Oberkochen, Germany) at 3 keV and low magnification mode.

#### Reconstitution of Dehydrated Cream

Dehydrated cream was reconstituted using a S25N-18G dispersing tool attached to an UltraTurrax T25 rotor-stator homogenizer (IKA Works, Inc., Wilmington, NC) by adding 224 g of DI water at 92°C to 172 g dehydrated cream. Reconstitution was performed in an insulated 600-mL beaker to yield a TS content of 43% TS in the reconstituted cream under conditions optimized in preliminary trials. Preliminary trials at different mixing temperatures ranging from 45 to 75°C, 12,000 to 24,000 rpm, and reconstitution times ranging from 0.5 to 6 min had shown that 18,000 rpm (calculated shear rate  $\dot{\gamma} = 4 \cdot 10^4 \text{ s}^{-1}$ ) for 4 min at 61 to 64°C were the minimum shear, time, and temperature needed to obtain a stable emulsion without any aggregates or lumps.

#### Particle Size Analysis

Particle size of pasteurized cream, homogenized cream, and reconstituted cream was determined by laser light diffraction/static light scattering using a Malvern Mastersizer 2000 equipped with a Malvern Hydro 2000G sample dispersion unit (Malvern Panalytical Ltd., Malvern, UK). Equal amounts of cream samples were dispersed in warm DI water to break up clusters bridged by free solid fat before adding the diluted cream to the sample dispersion unit until a laser obscuration between 10% and 20% was reached. The refractive index of the dispersant (DI water) was set at 1.33 and the refractive index for milk fat was set at 1.458 for the red laser (633 nm) and 1.460 for the blue laser (466 nm; Michalski et al., 2001). Particle absorption index was set to 0.0001. Pump speed was set to 1,000 rpm, stirrer speed to 850 rpm, and continuous ultrasonication at 20% tip displacement. Three independently dried and reconstituted samples were measured. Each sample was measured in duplicate at 25°C in DI water with two 12-s runs per aliquot.

#### **Confocal Laser Scanning Microcopy**

Stain solutions for confocal laser scanning microscopy (**CLSM**) were prepared by dissolving Nile Red (Sigma-Aldrich, Burlington, MA), a lipophilic fluorescent probe, at a concentration of 1 mg mL<sup>-1</sup> in 100% acetone. Fast Green FCF (Sigma-Aldrich, Burlington, MA) as dissolved at 1 mg mL<sup>-1</sup> in DI water to stain proteins. The fluorescent phospholipid analog 1,2-dioleoyl-sn-glycero-3-phosphoethanolamine-N-(lissamine rhodamine B sulfonyl) (**Rd-DOPE**) was purchased as a solution at 1 mg mL<sup>-1</sup> in chloroform (Avanti Polar Lipids, Croda Inc., Plainsboro, NJ).

An aliquot of 150 µL of cream, homogenized cream, or reconstituted microwave vacuum dehydrated heavy cream was mixed with 820 µL of warm DI water preheated to 70°C for dual-stained samples or 835  $\mu$ L for single-stained samples and vortexed in a 1.5-mL sample vial. Subsequently, samples were single-stained with Rd-DOPE (1:100 vol/vol) or dual-stained with Nile Red (1:100 vol/vol) and Fast Green FCF (1:100 vol/vol). After addition of 15 µL of Fast Green FCF, samples were vortexed, subsequently 15  $\mu$ L of Nile Red was added, and samples vortexed. For single-stained samples, 15 µL of Rd-DOPE was added to diluted cream and vortexed. Stained samples were incubated at least 30 min in the dark at room temperature and shaken from time to time to prevent phase separation. For fixation of stained fat globules in cream, 1%agarose solution was heated to 95°C in water bath to melt the gel. After solubilization, 500  $\mu$ L of 1% agarose was quickly added to the stained samples, shaken, and vortexed. An aliquot of 15  $\mu$ L of the liquid mixture was quickly transferred to a large cover glass (no. 1.5, 0.175-mm thickness,  $24 \times 50$  mm). A coverslip (no. 1.5, 0.175-mm thickness,  $18 \times 18$  mm) was immediately placed on top of the sample. The assembly was quickly transferred to a 12-mm aluminum plate on crushed ice for rapid cooling and gelling to prevent phase separation, evaporation, and thinning of the liquid layer between the slides due to capillary pressure that would squeeze the fat globules. Samples were checked under an AxioLab A1 light microscope (Carl Zeiss, Oberkochen, Germany) for homogeneity and stored in Petri dishes next to a wetted sponge to prevent drying of the gel layer. Confocal imaging was performed within 5 h after fixation.

An inverted confocal laser scanning microscope LSM 710 Axio Observer Z1 (Zeiss, Oberkochen, Germany) was used to image the milk fat globule samples with a plan-apochromat  $63 \times / 1.40$  Oil DIC objective. Excitation of Fast Green FCF was achieved with the 633 nm laser line and the emitted light was collected between 647 and 758 nm. Nile Red was excited with the 488 nm laser line and the filters were set to collect the emitted light between 555 and 646 nm. Samples stained with Rd-DOPE were excited with the 561 nm laser line and the emitted light was collected between 555 and 724 nm. Three-dimensional images were obtained by scan-

ning the sample in the z-axis in steps of  $0.4 \ \mu\text{m}$ . Images were analyzed using the Zeiss Zen blue software.

#### Whipping Properties of Reconstituted Cream

An aliquot of 200 g of cream was poured in a 600-mL insulated beaker, adjusted to 4.9 to  $5.2^{\circ}$ C, and whipped for defined times at level 4 using a Sunbeam hand mixer. Heavy cream from the store or reconstituted dehydrated cream were stored for at least 3 d at 3 to 4°C before use. About 100 mL of whipped cream was transferred to three 250-mL beakers. A P/CR cream probe developed by Pichert (1979) was attached to a TA-XT Plus Texture Analyzer (Stable Micro Systems, Surrey, UK). Texture analyzer settings were compression mode, pretest speed 1.0 mm s<sup>-1</sup>, test speed: 2.0 mm s<sup>-1</sup>, post-test speed: 10.0 mm s<sup>-1</sup>, distance 20 mm, trigger force 0.020 N. Penetration force in newtons was averaged between 6 and 10 s to determine whipped cream firmness. Measurements were performed in triplicate.

#### Statistical Analysis

A one-way ANOVA followed by a Tukey test was used to determine significant differences among lightness ( $\mathbf{L}^*$ ), red/green ( $\mathbf{a}^*$ ), and blue/yellow ( $\mathbf{b}^*$ ) color values of dehydrated cream and reconstituted dehydrated cream. Statistical data analysis and plotting was performed using OriginPro 2022b.

#### **RESULTS AND DISCUSSION**

#### Viscosity of Homogenized and Evaporated Cream

Pasteurized cream was diluted to 30% fat to limit the viscosity increase after homogenization. The protein-tofat ratio in higher-fat cream (<0.075 g protein/g butter fat) would be insufficient to cover the newly formed oilwater interface, especially at homogenization pressures higher than 7.5 MPa. A lower protein-to-fat ratio would result in excessive cluster formation after homogenization and a subsequent tremendous increase in viscosity. Figure 2 shows the effect of homogenization conditions and temperature on cream viscosity, concentrated from 36% to 43% TS (Figure 2A), and the effect of evaporation on viscosity at 50°C (Figure 2B). A temperature of 50°C was chosen to demonstrate viscosity trends, as this could be the temperature in the last stage of an evaporator. Cream viscosity decreased strongly with increasing temperature and increased with increasing homogenization pressure up to 7 MPa. A second pass at 3.5 MPa reduced the viscosity of cream due to the disruption of clusters. Increasing temperature reduced homogenized cream viscosity, which can help increase the maximum possible TS content of homogenized cream concentrate before MVD. Figure 2B shows that viscosity strongly increased with increasing TS content. Nevertheless, cream can be evaporated to >45% TS at  $50^{\circ}$ C, while remaining free flowing at a viscosity <100mPas. A high product viscosity can hamper the fast evaporation of water, which is necessary to reduce the energy demand for drying. In preliminary experiments, it was found that moderate homogenization of cream before MVD improves the stability of fat globules against coalescence during drying, caused by very close packing of the globules. Coverage of the small fat globules with a dense and thick layer of protein prevents the separation of liquid butter oil from the other milk solids. However, an increase in viscosity is inevitable when cream is homogenized due to cluster formation, the decrease in the interparticle distance between fat globules, and the rearrangement of phospholipids and protein at the oil-water interface. In cream, this increase in viscosity becomes more pronounced with increasing homogenization pressure (Kováčová et al., 2010). The limited availability of protein to cover the newly formed oil-water interface in high-fat emulsions such as cream leads to fat cluster formation (Ogden et al., 1976). Two-stage homogenization or a second pass at a lower homogenization pressure can reduce the viscosity of homogenized cream due to the disruption of the clusters formed during the first pass (Kessler, 2002).

#### **Desorption Isotherm of Dehydrated Cream**

Limiting chemical reactions such as Maillard browning and lipid oxidation to achieve a long shelf life is an important characteristic of dairy powders. To limit such reactions in dehydrated cream, a water activity of 0.2 to 0.3 is required (Kessler, 2002). In dehydrated cream containing more than 78% fat as obtained in this study, little water binding solids are available. According to the cream moisture sorption isotherm in Figure 3, a moisture content of 0.5% to 1.5% wt/wt, corresponding to water activity of 0.1 to 0.35, needs to be achieved to ensure shelf stability.

## Color, Structure, and Microstructure of Dehydrated Cream

Homogenization affected the visual appearance of dehydrated cream. Single-step 7-MPa homogenized cream yielded a brittle and slightly oily dry material after MVD, which formed small flakes when removed from the ice cube molds, as shown in Figure 4. Layer thickness had little effect on the visual appearance of the

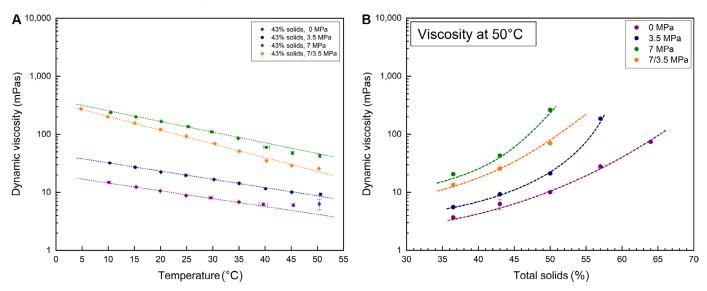


Figure 2. Apparent viscosity of evaporated cream (43% TS) homogenized at different homogenization pressures as a function of temperature (A). Viscosity of homogenized evaporated cream as a function of TS (B).

dry material. Two-step homogenized cream at 7/3.5 MPa yielded a cohesive material that appeared as chips when removed from the molds. Cream layers of 3 mm produced thicker dehydrated cream chips than 2-mm layers. Visual appearance was determined by cream homogenization conditions. Overall, the L\*, a\*, and b\* color values of dehydrated cream did not show major differences, whereas the L\* values were significantly different (Figure 4). However, this difference was neither correlated with layer thickness nor homogenization

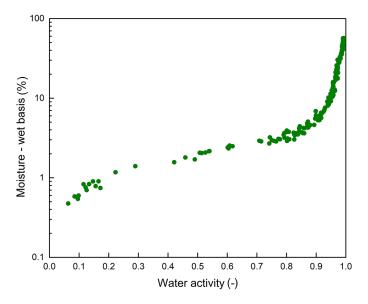


Figure 3. Desorption isotherm of dehydrated cream depicting the correlation of moisture content of cream and water activity.

Journal of Dairy Science Vol. 107 No. 2, 2024

pressure. Differences in L<sup>\*</sup> values may be related to the surface reflectivity of samples.

Scanning electron microscopy images of a crosssection of a chip of MVD dehydrated cream produced from cream homogenized at 7 MPa and 7/3.5 MPa are shown in Figure 5. These images revealed that the microstructure of MVD cream is very dense, individual fat globules could not be distinguished, and no micropores were visible. The dry material appeared to be a homogeneous matrix, indicating that fat globules partially or completely merged into a continuous matrix when water evaporated during drying. Some intact fat globules could be embedded in a continuous matrix of butter fat, although these images did not capture such instances. A more porous structure and larger number of large pores were observed in chips of dehydrated cream produced from cream homogenized at 7/3.5 MPa as shown in Figure 5B. This could be a result of stronger cohesion of the dehydrated cream and less free fat creating a less brittle material. Overall, homogenization conditions in terms of pressure and the number of passes had a strong effect on the appearance of dehydrated cream. Microwave vacuum drying of unhomogenized cream resulted in complete separation of butter oil and nonfat milk solids during drying (not shown). Increasing the homogenization pressure resulted in less visible free fat after MVD, especially when the dry material was dispersed in hot water. Two-step homogenization was therefore beneficial for the structure of the dry material.

To further investigate the dry cream microstructure and microstructural differences between dehydrated cream produced from cream homogenized at 7 MPa

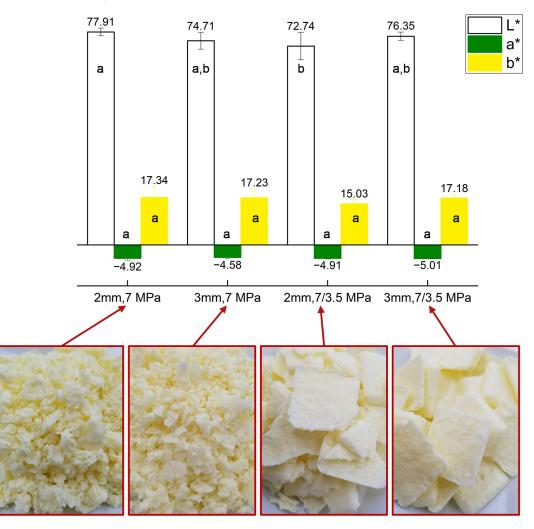


Figure 4. L\*, a\*, and b\* color parameters of dehydrated cream produced from cream homogenized at 7 MPa or at 7 MPa in the first pass and 3.5 MPa in the second pass (7/3.5 MPa) and dried with an initial product layer thickness of 2 or 3 mm. Pictures of the corresponding dehydrated cream are shown below for visual comparison. Different lowercase letters indicate significant differences ( $\alpha = 0.05$ ; n = 4). Error bars indicate SD ( $\pm$ ). L\* = lightness value; a\* = red/green value; b\* = blue/yellow value.

and 7/3.5 MPa, the amount of extractable fat was determined. Extractable fat is undesirable in fat containing milk powders, because it can compromise the keeping quality of milk powders due to the development of oxidized flavor (Písecký, 2012). Extractable fat can be categorized as (1) surface free fat, (2) capillary fat in fat globules located at the inner capillary surface, (3) dissolution fat located as fat globules at the surface of cracks, and (4) outer layer fat located as fat globules at the particle surface (Vega and Roos, 2006).

Considering observations in Figures 4 and 5 with respect to the microstructure of MVD cream pieces, it can be stated that MVD cream likely contains these 4 categories of extractable fat. However, the particle size (i.e., the size of the pieces of MVD cream) will be much larger than in spray-dried powders. It is worth noting that during extraction, particle size of dehydrated cream pieces decreased due to shear forces during solvent extraction. Table 1 shows the relative amounts of extractable fat, total fat based on TS, and extractable fat based on total fat. The data shows that homogenization of cream before MVD had no effect on extractable fat. The extractable fat based on total fat was around 53% for both samples. The extractable fat content of whole milk powder  $(27.6 \pm 0.3\%)$  total fat, 4.3% moisture) extracted under the same conditions was 48%, which means that milk fat in whole milk powder is less accessible to solvent than MVD cream. This higher stability of the fat globules was possibly due to more stable lipid droplets, due to a denser protein layer at the oil-water interface created by more intense homogenization and a higher-protein-to-fat ratio in whole

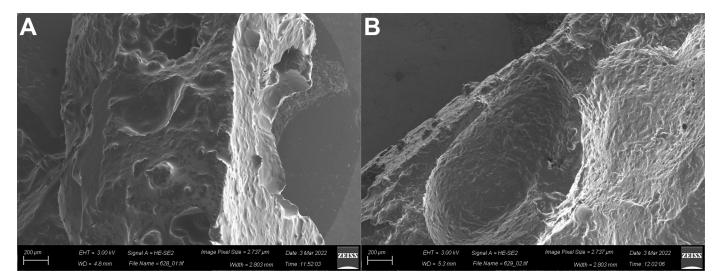


Figure 5. Scanning electron micrographs of the internal structure of dehydrated cream produced from cream homogenized at 7 MPa (A) or at 7 MPa in the first pass and 3.5 MPa in the second pass (7/3.5 MPa; B).

milk. The much smaller particle size of spray-dried whole milk powder appeared to have less of an effect on the accessibility of fat to solvent extraction.

#### Color and Microstructure of Reconstituted Dehydrated Cream

The effect of homogenization on appearance, fat globule size, and microstructure of MVD dehydrated cream reconstituted by a rotor-stator homogenizer at 61 to  $64^{\circ}$ C was investigated to understand the effect of cream homogenization and MVD conditions on reconstituted cream appearance, microstructure, and whipping functionality compared with pasteurized cream. Color measurements (Figure 6) revealed that homogenization of cream before MVD had a significant (P < 0.05) ef-

**Table 1.** Extractable fat based on total sample, total fat, and extractable fat based on total fat in dehydrated cream produced from cream homogenized at 7 MPa or at 7 MPa in the first pass and 3.5 MPa in the second pass (7/3.5 MPa)

Item	Extractable fat (% wt/wt)	Total fat (% wt/wt)	Extractable fat of total fat (% wt/wt)
7 MPa			
1	41.3	80.1	51.6
2	42.3	78.2	54.0
3	41.5	75.6	54.9
Average	41.7	78.0	53.5
SD	1.9	2.1	1.7
7/3.5 MPa			
1	41.7	80.1	52.0
2	46.1	79.8	57.7
3	38.4	76.7	50.1
Average	41.7	78.9	53.3
SD	4.0	3.1	4.0

Journal of Dairy Science Vol. 107 No. 2, 2024

fect on reconstituted cream color in comparison with pasteurized cream. L\* values were not different among samples. However, reconstituted cream produced from 7 MPa homogenized cream was more yellowish  $(a^*)$  and greenish (b<sup>\*</sup>) than pasteurized cream, although not significant in all cases. Reconstituted cream homogenized at 7/3.5 MPa showed significantly (P < 0.05) higher a<sup>\*</sup> and b<sup>\*</sup> values, both in comparison with pasteurized cream and reconstituted dehydrated cream produced from cream homogenized at 7 MPa. The reason for this difference in color can be found in the particle size distribution as shown in Figure 7. Single-stage 7/3.5 MPa homogenized reconstituted dehydrated cream had a smaller fraction of fat globules in the size range around  $1 \ \mu m$  (Figure 7, green and yellow lines) which can contribute to a more whiteish appearance of the reconstituted cream since particles in this size range strongly scatter visible light. Unhomogenized pasteurized cream (Figure 7, pink line) is shown for size comparison. It is not clear why reconstituted MVD cream produced from 2-pass homogenized cream (7/3.5 MPa) contains less of the small particle fraction than the single-pass homogenized cream (7 MPa). It is possible that the smaller particle fraction consists of MFGM fragments, which were reported before to be in this size range (Morin et al., 2007; Gassi et al., 2008). These fragments might adsorb better at the oil-water interface during reconstitution of dehydrated cream produced from cream homogenized at 7/3.5 MPa than in reconstituted cream homogenized at 7 MPa, possibly due to the smaller size of MFGM fragments in cream that was homogenized twice. Cream homogenized at 7 MPa (Figure 7, blue line) contains mostly larger clusters of disrupted fat

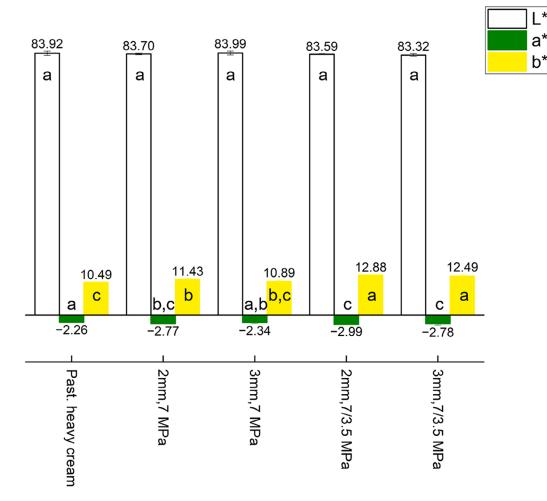


Figure 6. L\*, a\*, and b\* color parameters of pasteurized (Past.) and reconstituted dehydrated cream produced from cream homogenized at 7 MPa or at 7 MPa in the first pass and 3.5 MPa in the second pass (7/3.5 MPa) and dried with an initial product layer thickness of 2 or 3 mm. Different lowercase letters indicate significant differences ( $\alpha = 0.05$ ; n = 4). Error bars indicate SD (±). L\* = lightness value; a\* = red/ green value; b\* = blue/yellow value.

globules as also observed by Long et al. (2012) that might lead to larger MFGM fragments with a lower emulsifying activity. Small, dispersed fat globules constitute the major fraction in 7/3.5 MPa homogenized cream (Figure 7, brown line) that might be covered by smaller MFGM fragments in addition to milk proteins.

To better understand the reasons for differences in different samples of reconstituted cream appearance and functionality, the microstructure of the fat globules and the nature of the oil-water interface that covers them was investigated by CLSM. Confocal laser scanning microscopy images of pasteurized cream, homogenized cream at 3.5, 3.5/7 MPa, and reconstituted dehydrated cream produced from cream homogenized at 7 MPa and 3.5/7 MPa stained with Nile Red (lipids) and Fast Green FCF (protein), are shown in Figure 8. Pasteurized cream (Figure 8A) contains larger fat globules with little stained protein at the oil-water

interface. Increasing homogenization pressure from 3.5 MPa (Figure 8B) to 7 MPa (Figure 8B) resulted in smaller fat globules covered with protein, while cluster formation also increased, as expected. Two-step homogenization at 3.5/7 MPa resulted in the disruption of fat clusters and small individual fat globules covered to a large extent with protein (Figure 8D), which is similar to what would be expected from 2-stage homogenization. Figure 8E and Figure 8F show images of the corresponding reconstituted dehydrated cream produced from cream homogenized under the latter conditions. Most interestingly, the reconstituted fat globules are only covered to a small extent by protein, similar to fat globules in pasteurized cream, but fat globules are larger than in the pasteurized cream. Particle sizes of homogenized fat globules before MVD had no effect on particle size after reconstitution. However, reconstituted dehydrated cream produced from 7 MPa

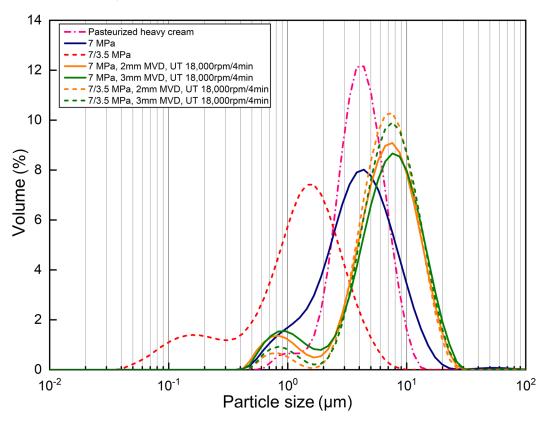


Figure 7. Particle size distribution of pasteurized cream, cream homogenized at 7 MPa or at 7 MPa in the first pass and 3.5 MPa in the second pass (7/3.5 MPa), and reconstituted dehydrated cream produced from cream homogenized at 7 MPa or 7/3.5 MPa and dried with an initial product layer thickness of 2 or 3 mm. MVD = microwave vacuum drying; UT = UltraTurrax T25 rotor-stator homogenizer (IKA Works Inc., Wilmington, NC).

homogenized cream contained more small particles in between the large red fat globules that could be MFGM fragments as can be observed in the particle size distribution results (Figure 7).

In addition to protein, the oil-water interface of native milk fat globules is covered by phospholipids. In its native state, the MFGM is a triple-layer of phospholipids and protein originating from the apical side of the secretory cell membrane in the mammary gland (Michalski et al., 2002). Because a dense layer of protein covering fat globules was not visible on the surface of fat globules in reconstituted dehydrated cream, coverage with phospholipids from the MFGM material was likely. This similar surface coverage might result in a functionality of reconstituted cream similar to pasteurized cream.

Figure 9 shows CLSM images (cross-section) of cream samples stained with Rd-DOPE to depict the spatial distribution of phospholipids in pasteurized cream (Figure 9A), homogenized cream (Figure 9B, C, and D), and reconstituted MVD cream (Figure 9E and 9F). These images show that mostly the larger fat globules are covered with phospholipids, and that larger fat globules in reconstituted dehydrated cream appear covered with phospholipids, similar to fat globules in pasteurized cream.

Corredig and Dalgleish (1998) found that oil-in-water emulsions prepared with MFGM material isolated from raw or pasteurized cream were more heat stable than emulsions stabilized by caseins or whey proteins. Moreover, MFGM material adsorbed at the oil-water interface could not be displaced by synthetic surfactants, indicating a low surface tension of MFGM-stabilized emulsions. The emulsification activity, microstructure, and functionality (whippability and foam stability) of lipid droplets coated with MFGM have been studied to some extent (Kanno, 1989; Kanno et al., 1991; Oehlmann et al., 1994; Lopez et al., 2017). However, the effect of mechanical actions (i.e., mechanical damage during pumping, churning, and whipping) on the properties of MFGM-stabilized quasinative emulsions has only been studied to a very limited extent to date. This work contributes to a better understanding of these effects.

To summarize, fat globules in MVD dehydrated cream reconstituted using a rotor-stator homogenizer

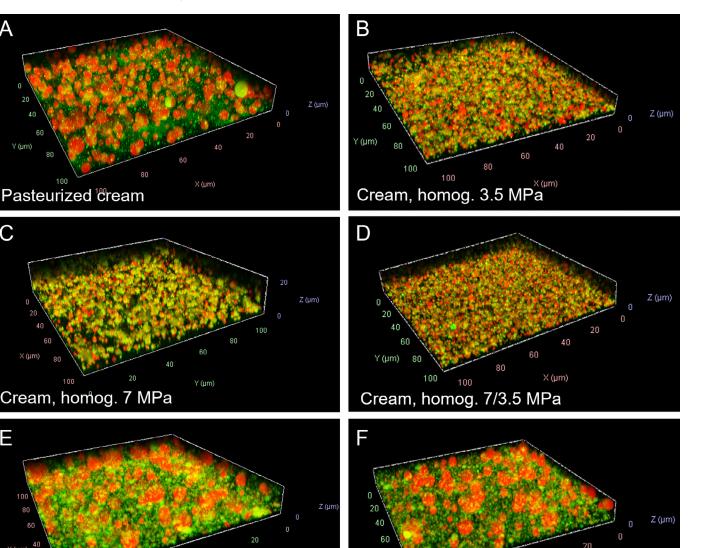


Figure 8. Confocal laser scanning microscopy z-scans (3-dimensional) of fat globules in pasteurized cream (A), cream homogenized (homog.) at 3.5 MPa (B), 7 MPa (C), or 7 MPa in the first pass and 3.5 MPa in the second pass (7/3.5 MPa; D), dehydrated cream reconstituted at 18,000 rpm for 4 min using the UltraTurrax (UT) T25 rotor-stator homogenizer (IKA Works Inc., Wilmington, NC) produced from cream homogenized

at 7 MPa (E) or 7/3.5 MPa (F). Milk fat was stained with Nile red (red) and protein was stained with Fast Green FCF (green).

Y (µm)

100

are larger in size but appear similar with respect to coverage with protein and phospholipids, under the reconstitution conditions used in this study. Homogenization before MVD was a necessary step to prevent separation of butter oil and nonfat milk solids during MVD but different homogenization conditions did not affect the size and surface coverage of fat globules in reconstituted cream. Changes induced by homogenization increased the emulsion stability, but also changed the functionality of reconstituted cream. Combining homogenization, MVD, and suitable reconstitution conditions allows to

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re-create fat globules with about twice the average size and similar surface coverage compared with fat globules in pasteurized cream. These similarities in structure are likely to lead to similarities in functionality, e.g., whipping properties, which will be discussed below.

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#### Whipping Properties of Reconstituted **Dehydrated Cream**

Reconstituted UT (7/3.5 MPa)

Figure 10A shows a visual comparison between pasteurized heavy cream and reconstituted dehydrated

Reconstitutëd UT (7 MPa)

С

Ε

X (µm)

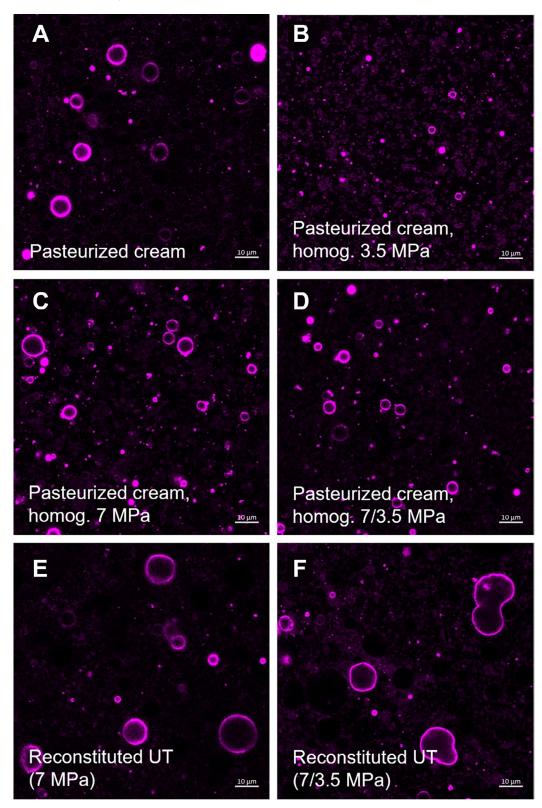
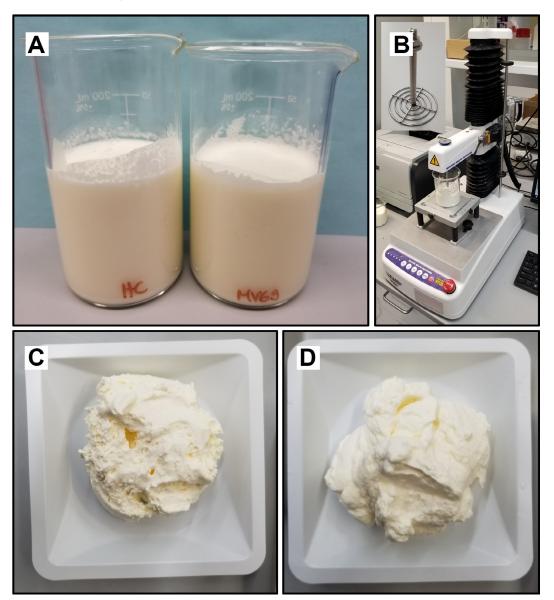


Figure 9. Confocal laser scanning microscopy images (2-dimensional) of fat globules in pasteurized cream (A), cream homogenized at 3.5 MPa (B), 7 MPa (C), and 7 MPa in the first pass and 3.5 MPa in the second pass (7/3.5 MPa; D), dehydrated cream reconstituted at 18,000 rpm/4 min using the UltraTurrax (UT) T25 rotor-stator homogenizer (IKA Works Inc., Wilmington, NC), produced from cream homogenized at 7 MPa (E) or 7/3.5 MPa (F). Phospholipids were stained with the fluorescent phospholipid analog 1,2-dioleoyl-sn-glycero-3-phosphoethanol-amine-N-(lissamine rhodamine B sulfonyl) (Rd-DOPE; purple). Homog. = homogenized.



**Figure 10.** Pasteurized heavy cream (left, labeled "HC") and reconstituted dehydrated cream (right, labeled "MV69") in beakers (A), TA-XT Plus Texture Analyzer (Stable Micro Systems, Surrey, UK) and cream probe in the top left corner (B), whipped cream from pasteurized cream (C), and whipped cream from reconstituted dehydrated cream (D).

cream in beakers. These 2 samples are very similar in appearance. The visual appearance of whipped cream obtained from pasteurized cream and whipped cream from MVD reconstituted cream, at peak firmness, was also very similar (Figure 10C). The whipped cream firmness over time was determined using a texture analyzer equipped with a cream probe (Figure 10B). Figure 11A shows whipped cream firmness at 30.5% fat after 90 s whipping as a function of homogenization pressure, and the insert shows a texture analyzer graph that depicts the force plot. Whipped cream firmness data correspond to the whipping properties of cream before evaporation and MVD. Whipped cream firmness data in Figure 11A serves as a control for the whipped cream produced from reconstituted dehydrated cream. Whipped cream firmness at 90 s whipping time decreased strongly with increasing homogenization intensity, with 0 MPa homogenized cream (passed through the homogenizer without applying pressure) showing the highest firmness, and the 2-step homogenized cream the lowest firmness. A direct comparison with the whipping behavior of spray-dried cream powders is important. Before spray drying, cream is typically subjected to more intense homogenization conditions

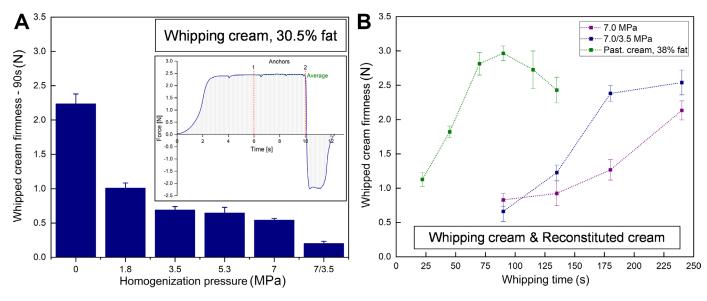


Figure 11. Whipped cream firmness from 30.5% fat cream depending on homogenization conditions of cream. The Texture Analyzer (Stable Micro Systems, Surrey, UK) graph with anchors at 4 and 6 s, as well as the average firmness, are also shown (A). Whipped cream firmness of pasteurized (past.) cream (38% fat) and reconstituted dehydrated cream produced from cream homogenized at 7 MPa or at 7 MPa in the first pass and 3.5 MPa in the second pass (7/3.5 MPa) as a function of whipping time (B). Error bars indicate SD ( $\pm$ ).

than those used in this work (Park and Drake, 2017). Preliminary experiments conducted with reconstituted spray-dried cream powder showed very poor whipping properties of this product (data not shown). Essentially, no whipped cream or butter formation could be obtained from such spray-dried cream products. This means that major functionalities of cream are lost when cream is homogenized and spray dried. After reconstitution, fat globules in reconstituted spray-dried cream powders remain very small and covered by a dense layer of protein after reconstitution. By contrast, fat globules in MVD cream appear similar in structure as those in pasteurized cream, resulting in similar whipping properties.

Figure 11B shows the whipped cream firmness of pasteurized cream (green squares) as a function of whipping time as compared with whipped reconstituted dehydrated cream produced from cream homogenized at 7 MPa (blue squares) and 3.5/7 MPa (purple squares). The fat content of all cream samples was 38%. Using the same percent fat was very important, as the fat content affects firming rate and peak whipped cream firmness (data not shown). Figure 11B shows that whipped cream firming rate was fastest for pasteurized whipping cream. Whipped cream firming rate for reconstituted dehydrated cream produced from 7/3.5MPa homogenized cream was slower than pasteurized cream, but faster than reconstituted cream prepared from 7 MPa homogenized cream. This faster firming rate of the 2-stage homogenized cream could be related

to differences observed in the number of MFGM particles, the minor fraction in the size distribution (Figure 7) and the higher intensity of proteinaceous material (in green) in the serum phase of 7 MPa homogenized cream (see Figures 8E and F). More MFGM material in the serum phase could indicate the presence of more protein (caseins and whey proteins) at the oil-water interface that stabilizes fat globules better against cluster formation during whipping than MFGM material. Also, the whipping rate was slower and the peak firmness for reconstituted dehydrated cream produced from 3.5/7 MPa homogenized cream was almost as high as that of pasteurized whipping cream. Therefore, MVD combined with appropriate homogenization before drying and reconstitution conditions represents a novel approach to preserve cream solids and retain full functionality of cream with respect to whipping properties.

#### **CONCLUSIONS**

This study demonstrated that a combination of an appropriate predrying treatment (i.e., homogenization) and optimal MVD parameters resulted in desirable dehydrated cream structure, reconstituted cream microstructure, and whipping properties. Additionally, insights into why the MVD cream retained good whipping functionality were obtained. Further optimization of this process could be achieved by conducting a more detailed analysis of the effect of homogenization and reconstitution conditions. In addition to whipping, other applications for reconstituted MVD cream, including use in cheese, butter, or ice cream, could be explored and may lead to unique opportunities in regions without a steady raw milk supply or reliable cold chain. It should also be noted that cream can be evaporated to TS levels higher than those of skim milk. Overall, MVD cream can be considered a highly functional shelf-stable product, characterized by high resource efficiency, and therefore this product and technology can improve the sustainability of dairy processing.

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