ABSTRACT

Microwave vacuum drying (MVD) of concentrated skim milk and its resulting powder properties have been studied to a very limited extent. To explore the potential of this technology for the manufacture of milk powder, MVD of concentrated skim milk (37.5% total solids) was evaluated with respect to product properties and drying efficiency. A custom factorial design was used to optimize drying parameters, which enabled us to find optimal drying conditions with a minimal number of drying experiments (16). Vacuum level (3.3–13.3 kPa), specific power input (0.86–1.72 W·g⁻¹), and product layer thickness (1–4 mm) were studied as factors. Total drying time, product foaming at the beginning of the process, product temperature in the last drying interval, browning, insolubility index, and calculated product yield were used as responses to identify optimal MVD processing parameters. Optimal drying of concentrated skim milk that maximized yield and minimized drying time while maintaining good product quality was achieved at a layer thickness of 2 mm, pressure of 6.0 kPa, and a specific power input of 1.29 W·g⁻¹. Under constant power output, layer thickness was found to be the most important processing parameter to control product temperature during the final drying stage. Maximum product temperatures below 55°C yielded powder with good solubility. The findings of this exploratory study for MVD of concentrated skim milk yield important information and guidelines for production of good quality milk powders or preservation of starter cultures in a dairy matrix such as infant formula.

Key words: microwave vacuum drying, concentrated skim milk, factorial design, process optimization

INTRODUCTION

Microwave vacuum drying (MVD) is a versatile drying technology because its ability to adjust a broad range of processing parameters—including power input, vacuum level, product shape, and load—allows for the drying of a wide range of products at higher drying rates compared with conventional conductive or convective drying. In addition to being versatile and fast, MVD is a very gentle drying technology that does not involve high shear, hot air, high product temperatures, freezing, or oxidative stress for the product. However, because MVD is based on heating in an electromagnetic field, it may result in uneven heating and drying due to the uneven field distribution in the drying chamber and within the product due to its shape, size, and dielectric properties. Uneven heating can result in local overheating (i.e., hot spots), leading to product damage or uneven moisture distribution in the dried product. Product shape, size, composition (especially water and mineral content), and distribution within the drying chamber can contribute to differences in local heat dissipation within the product, causing hot and cold areas (Datta et al., 2005). Foods high in salt content exhibit an increase in the dielectric loss factor with increasing temperature (Bengtsson and Risman, 1971; Wang et al., 2011). This leads to a self-propagating local overheating effect, often called “thermal runaway” (Datta et al., 2005), which is exacerbated by variations of electric field strength within the microwave drying chamber. In addition, differences in the heat conductivity and vapor permeability of a material may lead to local vapor pressure build-up and much higher internal temperatures, leading to brown spots. The formation of brown spots is of particular concern for the drying of products such as concentrated skim milk, which is high in soluble salts, lactose, and protein. However, this local pressure build-up can be desirable if the goal is to achieve a puffing effect; that is, the expansion of product structures in vacuum due to high energy transfer and a high vapor pressure gradient within the product (Rakesh and Datta, 2013). The “leveling effect” contributes to an eventually even moisture distribution in
the dried product. Dry areas absorb less microwaves even when the field distribution in the drying chamber is inhomogeneous.

Overall, MVD requires process optimization to achieve desirable results. To facilitate applications of MVD in dairy science and technology, significant pioneering research work is needed. To optimize the multitude of settings for MVD, a one-factor-at-a-time design could be used. Alternatively, researchers have successfully used cost-effective factorial designs or other reductive approaches to find optimal drying conditions for various food products with a limited number of drying runs compared with the numerous runs required by one-factor-at-a-time (Giri and Prasad, 2007; Mitra and Meda, 2009; Han et al., 2010; de Jesus and Filho, 2011; Ahmad et al., 2012; Pawlak et al., 2013).

Microwave heating has mostly been studied for the gentle heat treatment of milk and concentrated milk (Salazar-González et al., 2012; Graf et al., 2020). Some data on the frequency and temperature dependency of dielectric properties of milk and concentrated skim milk across a broad range of temperatures have been recently published (Muñoz et al., 2018), which could help our understanding of how milk is heated in an electromagnetic field. Microwave freeze-drying of milk has been investigated by Wang and Chen (2005), and theoretical models were developed to describe the microwave freeze-drying behavior of milk foams (Sochan-ski et al., 1990). An earlier study reported on the use of MVD milk as protectant and matrix for microbial cultures (Ahmad et al., 2012), but very little is known about the MVD behavior of milk. Microwave vacuum drying has potential for use in the dairy industry, but dairy applications of this technology have not yet been explored.

Therefore, the major aim of this study was to investigate the potential of MVD for drying of concentrated skim milk (CSM), and to optimize the process from both an economic perspective (i.e., drying efficiency and product output) and in terms of powder quality (i.e., the absence of brown particles and good solubility). Due to the complexities of MVD, this process requires optimization for each category of product, such as solid or liquid foods, to achieve desirable results. Therefore, we chose to use a factorial design approach in this work to optimize the MVD of CSM. Due to its high lactose content, skim milk powder could be used as a cost-effective protectant for microbial cultures in dried direct vat set cultures or infant formula. Studying the drying behavior of concentrated milk first, without, for example, starter culture, represents an efficient way to optimize the MVD process without additional restrictions.

MATERIALS AND METHODS

Reconstitution of Nonfat Dried Milk

Two different batches of low heat grade A nonfat dried milk (NDM; Dairy America) used for all drying experiments. The composition of the 2 batches was nearly identical and fulfilled the legal specifications for skim milk powder: 0.59 ± 0.17% and 0.52 ± 0.13% butter fat, 36.2 ± 0.13% and 36.5 ± 0.12% total protein, 34.7 ± 0.13% and 34.7 ± 0.19% true protein, 28.5 ± 0.17% and 28.1 ± 0.32% casein, 6.8 ± 0.5% and 6.7 ± 0.25% ash, 56.7 ± 0.28% and 56.3 ± 0.18% lactose, respectively, calculated as a percent of TS (n = 3). The free moisture content of the 2 NDM batches was 4.5 ± 0.05% and 4.4 ± 0.29%, and the whey protein nitrogen index (WPNI) was 6.5 and 5.8 g/kg of NDM (per 100% solids), respectively. The NDM was reconstituted to 37.5% TS using an Omni-mixer homogenizer (model no. 17105, Omni International) in deionized (DI) water heated to 80°C (175°F). The NDM was quickly pre-mixed with heated DI water to obtain a mixture of 55°C and then homogenized at 14,000 rpm for 3 min. The mixture was filtered through a strainer and kept at 54 ± 2°C for 30 min in a water bath before cooling in an ice-water mixture. After cooling to 10°C, total skin milk solids were determined on glass fiber pads in aluminum pans using an infrared moisture analyzer IR-30 (Denver Instruments), and the mixture was adjusted to 37.5 ± 0.8% TS by adding a small quantity of DI water, as needed. Subsequently, fine-ground lactose powder was added to the mixture, at a concentration of 2.8 g·L⁻¹ to facilitate controlled lactose crystallization. The reconstituted CSM was then stored at 2.6 ± 0.6°C for 44 to 50 h under gentle shaking at 140 rpm on a New Brunswick rotary shaker Excella E-5 (Eppendorf AG) to crystallize the lactose before MVD. This was done to prevent uncontrolled lactose crystallization during CSM storage at refrigerated temperatures, because the concentration of lactose in the CSM (37% of TS) exceeded the supersaturation limit for lactose at refrigerated temperatures.

MVD of CSM

A small commercial-scale EnWave nutraReV 10 kW MVD unit (EnWave Corp.) was used to carry out the drying experiments. The MVD unit’s internal rotating carousel can carry up to 8 rectangular polypropylene trays. Five silicone ice cube trays were placed in each of the 8 polypropylene trays, resulting in a total of 40 ice cube trays on the carousel in the drying chamber. Then, 1, 2, 3, or 4 mL of CSM was pipetted (Eppen-
dorf, Multipette M4, Eppendorf AG) into each cavity of the 40 silicone ice cube trays, which had 15 cavities each (3.2 × 3.2 × 3 cm, length × width × height). The use of the small cavities of ice cube trays allowed us to maintain an even film thickness while the carrousel was rotating, which was important when investigating the effect of film thickness on the dry product quality. The vacuum, power, and time settings of the MVD unit were then adjusted to the required values at the human–machine interface of the programmable logic controller (PLC) according to the custom factorial design. The rotation speed of the carousel was set to 35% (~3 rpm) in all experiments. Experiments were performed at constant power input and pressure. The chiller water temperature for the vapor condenser was set to 10 to 11.5°C.

**Process Optimization Using a Custom Factorial Design**

Based on preliminary drying experiments, advice from the manufacturer, and knowledge obtained from literature, the ranges for overall load, film thickness/layer thickness, drying pressures, specific power input, and other machine settings were established, which were then used to develop a custom, full-factorial design using JMP Pro 14 (SAS Institute Inc.). The model allowed us to identify optimal processing conditions for MVD of CSM based on 3 factors: CSM layer thickness, drying pressure, and specific power input, which resulted in a total of 16 experimental runs. Four levels of layer thickness were used: 1, 2, 3, and 4 mm, corresponding to 1, 2, 3, and 4 mL of CSM per cavity. The 4 levels of drying pressure used were in the ranges 3.3 to 3.7, 6.0 to 6.7, 9.3 to 10.3, and 13.3 to 14.7 kPa, and the 3 levels of specific power input were 1.0, 1.5, and 2.0 W·cm−3 (0.86, 1.29, and 1.72 W·g−1). Actual specific power inputs were calculated from PLC recordings transformed to watts per gram using the density of the concentrate at filling temperature ($\rho_{5^\circ C}$) = 1.1602 g·cm−3. Preliminary experiments and estimations had shown that a combination of very low pressure, thick layers, and high power input resulted in excessive foaming. The details of the full-factorial custom design for the drying runs are shown in Table 1. Responses were defined based on selected powder properties and process performance characteristics defined after preliminary trials.

**Characterizing and Monitoring the Drying Process**

The drying process was interrupted every 5 or 10 min, depending on the specific power input. The aim was to obtain a sample from at least 5 drying intervals for residual moisture analysis, to determine the drying rates of each run. After removal of 1 to 3 ice cube trays, the process was restarted at the same pressure but a reduced total power input. The overall power input was adjusted to maintain a constant specific power input by accounting for the reduction in load in the dryer chamber after sample removal. An example of the PLC recordings of a drying process is shown in Figure 1. Product samples were collected in plastic flip-top vials for further analysis. Based on the visual dryness of the material and the maximum product temperature recorded during processing, the process was either continued for another interval or terminated. The remaining dried material (see Figure 2, right) was collected in tightly sealed plastic bags and stored for grinding and further analysis. Figure 2 depicts, from left to right, examples of the liquid CSM, the dried material, and the final collected material obtained under 2 different processing conditions.

The samples taken during the drying process were analyzed for water activity at 25 ± 0.5°C using an AquaLab 4TE (Meter Group). The moisture content of the samples was determined using an IR-30 infrared moisture analyzer. Drying rates, $r$, were calculated from moisture differences of subsequent drying steps, using equation [1]:

$$r = \frac{X_1 - X_2}{100 - X_2} \cdot \frac{1}{t_2 - t_1} = \frac{\Delta X}{(100 - X_2) \cdot \Delta t},$$

where $r$ is the specific drying rate and $X_1$ and $X_2$ are the moisture contents (% wt/wt) of a sample at the beginning ($t_1$) and the end ($t_2$; h) of a drying interval. The drying rate was plotted over the average TS con-
tent within the drying interval as an approximation of the true average.

The degree of foaming during the drying process was linearly rated on a scale from 1 to 7 based on the maximum volume the dried material occupied in the ice cube tray cavity. The maximum foam volume could be detected on the walls of the cavity and was measured with a ruler. Overflowing of the trays was assigned a foaming index of 7 in all cases, regardless of the extent. To correlate this foaming index with drying kinetics, the drying rate in the first drying interval was used to calculate the water vapor flow rate \( \dot{V}_v \), depending on the drying pressure, using equation [2]:

\[
\dot{V}_v = r \cdot v''_p, \quad \text{[2]}
\]

where \( r \) is the specific drying rate and \( v''_p \) is the specific vapor volume \( (m^3\cdot kg^{-1}) \) at a specific pressure. Data for the properties of water vapor were taken from Wagner and Kruse (1998).

Product temperatures during drying were determined using a calibrated infrared thermometer (CT-SF15-C3, Micro-Epsilon, Optronic GmbH) attached at the top of the chamber. Maximum temperatures were taken from the PLC output. Pressure, run time, and power input were also recorded to calculate the specific power input, total energy, and drying efficiency.

Drying efficiency \( (\eta; \text{unitless}) \) of the microwave drying process was calculated using equation [3]:

\[
\eta = \frac{r \cdot \lambda_w \cdot \rho_0 \cdot (X_1 + X_2)}{P_{MW} \cdot 2 \cdot 100 \cdot 3,600}, \quad \text{[3]}
\]

where \( r \) is the drying rate (eq. [1]), \( \lambda_w \) is the latent heat of vaporization \( (kJ\cdot kg^{-1}) \) at the corresponding pressure, \( \rho_0 \) is the initial density \( (kg\cdot m^{-3}) \) of the concentrate, \( (X_1 + X_2)/2 \) is average moisture content in the drying interval, and \( P_{MW} \) \( (W\cdot m^{-3}) \) is the calculated specific power input during the drying interval. A browning index for the dried material was defined for the dried material based on simple visual observation on a scale: 1 = no brown spots, 2 = a few small spots, and 3 = clearly visible browning.

**Determination of Powder Properties**

**Grinding, Particle Size, and Bulk Density.** The dried material was ground into a powder in a high-speed...
grinder (HC-1000; Hanchen Instrument) at 13,000 rpm for 40 s. The ground material was characterized by sieving, and the mass fraction in each size class was determined to ensure homogeneous powders for further analysis. Sieves were stacked in the order 75, 150, 250, 425, and 850 μm (bottom to top) and mounted on a New Brunswick rotary shaker Excella E-5. Sieving was performed for 30 min at 300 rpm. Wooden cubes were used as sieve cleaners.

The poured and loose bulk densities of the ground powders were determined in duplicate and used to characterize and compare the different powders. Bulk density of the powders was determined according to IDF (2005a; Engelsmann Stampfvolumeter) with some modifications. In short, 40.0 ± 0.1 g of powder was slowly poured into a 250-mL graduated cylinder using a funnel, and the surface was leveled off with a spatula. The volume was recorded and used to calculate the poured bulk density. A rubber stopper was attached to the measuring flask to be able to tap the volumetric flask. The bottom of the stopper was fixed 10 mm above the table surface and the metering flask was tapped 100 times. The powder volume was then recorded and used to calculate the loose bulk density.

Dispersibility. Dispersibility of the powders was determined using a modified IDF (2005b) method. Due to the limited amount of powder available from the drying experiments, the sample size for this test was reduced. In brief, 13 g of powder was weighed into a small crystallizing dish (50 × 35 mm). Then, 125 mL of demineralized water at 24 or 50°C was poured into a 300-mL low-form beaker. The crystallizing dish was covered with a glass plate and flipped. After the powder was evenly distributed, both the glass plate and the crystallizing dish were attached concentrically above the beaker with a clamp attached to a stand. The glass plate was then removed to transfer the powder into the beaker. After 5 s, a spatula was used to stir gently for 20 s. The mixture was left for 30 s and then filtered through a 250-μm sieve for 30 s. The dry matter in the mixture was determined in triplicate using an infrared moisture analyzer and then used to calculate the dispersibility \( W \) according to equation [4]:

\[
W \text{ (\%)} = \frac{m_W \cdot w_1}{100 - (X_{H_2O} + w_1)} \cdot \frac{100}{m_P},
\]

where \( m_W \) is the volume of demineralized water (125 g), \( w_1 \) is the TS content of the filtrate, \( X_{H_2O} \) is the moisture content of the powder (both as mass percentage), and \( m_P \) is the amount of powder (13 g) used for the test.

Insolubility Index, Soluble Nitrogen Index, and WPNI. The milk powder solubility was evaluated quantitatively using the insolubility index, determined by a method adapted from IDF (2005c). Thirteen grams of powder was added to 125 mL of DI water at 24 or 50°C in a 400-mL stainless steel beaker with vortex breakers (Omni International). Six drops of silicone antifoam (Sigma-Aldrich) were added. A 2-inch-edge
sharp rotor knife was used to reconstitute the powder for 90 s at 3,600 ± 100 rpm. About 12 g of mixture was then transferred to four 15-mL conical-bottomed, graded centrifuge tubes and centrifuged at 3,300 × g for 10 min at room temperature in a Sorvall RC-5B superspeed centrifuge equipped with a GSA fixed-angle rotor (DuPont Company). About 6 g of the supernatant from each centrifuge tube was collected and used for the determination of the soluble nitrogen index (SNI), using the Kjeldahl method for total nitrogen analysis. The total nitrogen of the reconstituted samples was used to calculate the SNI according to equation [5]:

\[
\text{SNI} (\%) = \frac{w_s}{w_d} \cdot 100, \quad [5]
\]

where \(w_s\) and \(w_d\) represent the amount of nitrogen (mg-g\(^{-1}\)) in the supernatant and total sample, respectively.

The remaining liquid and sediment in the centrifuge tubes were diluted by adding about 6 g of demineralized water, and the sediment was completely resuspended with a spatula followed by vigorous shaking. The mixture was then centrifuged at 160 × g for 5 min. The volume of sediment in the 4 centrifuge tubes was determined using the graduation of the conical part of the tubes, and the average was reported as insolubility index in milliliters (\(n = 4\)).

The WPNI was determined by acidification to pH 4.6 ± 0.1 of 25 g of the noncentrifuged reconstituted skim milk by dropwise addition of 1 N or 0.1 N HCl to precipitate the caseins. The mixture was then centrifuged at 3,300 × g for 10 min at room temperature. The supernatant was carefully removed, collected, and analyzed for soluble nitrogen by the Kjeldahl method. The WPNI was then calculated according to equation [6]:

\[
\text{WPNI} \, (\text{mg} \cdot \text{g}^{-1} \text{ dry powder}) = w_d \cdot 6.38 \cdot \frac{100\%}{\bar{m}_1}, \quad [6]
\]

where \(w_d\) is the nitrogen content in the pH 4.6 supernatant corrected for NPN as 12% trichloroacetic acid (TCA)-soluble nitrogen (263 mg L\(^{-1}\)) and \(\bar{m}_1\) is the TS content (% wt/wt) of the reconstituted mixture as the average of triplicates. The WPNI was then converted to the relative WPNI of the MVD powder relative to the NDM powder, expressed as percent.

**Determination of the Glass Transition Temperature**

The MVD skim milk powders were placed in thin layers in Petri dishes in desiccators, above saturated salt solutions that created a controlled relative humidity (RH) environment, and left for 30 d at 21°C to equilibrate the moisture content. Saturated salt solutions of sodium hydroxide (7% RH), lithium chloride (11.3% RH), potassium acetate (22.2% RH), and magnesium chloride (32.7% RH) were used. The water activities of the samples were measured immediately before sealing into differential scanning calorimetry (DSC) pans and were 0.102, 0.126, 0.220, and 0.294, respectively. Small amounts of each of the powders (6–10 mg) were hermetically sealed in DSC aluminum pans. A DSC 2500 (TA Instruments) was used to determine their glass transition temperatures (\(T_g\), °C). The DSC procedure was as follows: heat to 63°C at 5°C/min, hold for 2 min, cool to −10°C at 25°C/min, hold for 2 min, and heat to 90°C at 5°C/min. The \(T_g\) was defined as the endpoint of the transition.

**Inactivation of Microorganisms During Drying**

*Escherichia coli* ATC 25922 and *Listeria innocua* C2-008 were cultivated for 14 h at 37°C on tryptic soy agar to obtain bacterial suspensions with a concentration >10\(^8\) cfu·mL\(^{-1}\). Ten mL of inoculum was added to 700 mL of 37% TS CSM to achieve a microbial count of 10\(^8\) to 10\(^9\) cfu·mL\(^{-1}\). Both inoculated concentrates were then subjected to MVD in the same drying run at 1.29 W·g\(^{-1}\), 6.0 to 6.7 kPa, and 2-mm layer thickness for 40 min, with and without a final drying step at 0.86 W·g\(^{-1}\) and 6.0 to 6.7 kPa for 20 min. Before microbiological analysis, the inoculated concentrate and MVD material were reconstituted to 9% TS by mixing 28.5 g of concentrate and 75 g of DI water, and 13 g of dry product and 125 g of DI water, respectively, to allow for the direct comparison of the plate counts. The reconstituted milk containing *E. coli* was plated on selective violet red bile agar and that with *L. innocua* on modified Oxford agar; both were incubated at 37°C for 24 h. Inactivation ratios were calculated as log(\(N_0/N\)), where \(N_0\) and \(N\) represent bacterial counts (in cfu·mL\(^{-1}\)) before and after drying, respectively. The drying process, with and without the modified final drying step, was performed in triplicate, and plating was performed in duplicate.

**Statistical Analysis and Optimization Criteria for the Drying Process**

The drying time to obtain a water content of <10%, browning index, foaming index, maximum product temperature detected during the last drying interval, insolubility index of the powders, and powder output per run were chosen as response variables for the optimization of the desirability functions of the MVD pro-
cess and the powder characteristics. Individual drying parameters were tested for their significance (α < 0.05) on responses using JMP Pro 14 (SAS Institute Inc.). All responses were weighed equally. The D-optimality criterion, which minimizes the determinant of the covariance matrix of the model coefficient estimates, was used for optimization. This criterion is most suitable for screening, estimating effects, testing for significance, and identification of active factors. Other statistical analyses and plotting of data were performed using OriginPro 2021b (https://www.originlab.com/2021).

RESULTS AND DISCUSSION

Drying Rate, Drying Efficiency, and Product Temperature

The effect of different processing parameters on the drying rate, as determined by the change in moisture content, was used to assess the overall process performance and efficiency for the MVD of CSM. The product temperature during MVD is an important parameter to monitor to prevent overheating and thereby irreversible physicochemical changes in the dried product, especially during the last drying stage. The importance of product layer thickness during drying of CSM became evident during preliminary trials and was therefore systematically investigated. The surface plots in Figure 3 show the results for the drying rate (top) and product temperature (bottom) depending on specific power input, layer thickness, and TS content of the concentrate. The drying rate showed a strong dependency on the TS content of the concentrate, whereas the maximum drying rate was dependent on the specific power input in its magnitude and location. At higher power input, a higher drying rate was observed, and the maximum drying rate occurred at a higher TS content. The highest drying rates were observed in a moisture range from 35 to 15%. When moisture levels fall below ~20%, the decreasing dielectric loss factor will reduce the drying speed (Figure 3, top).

Product layers >2 mm resulted in a much higher product temperature across the entire TS range during drying. Especially high temperatures were observed during the last drying interval, at >85% TS. Remarkably, the specific power input showed little effect on product temperature over the entire range of TS. Smaller variations in temperature at lower solids level were caused by the different drying pressures that result in a different boiling temperature of the concentrate. Figure 4 shows the correlation of the maximum product temperature in the last drying interval with the specific power input and the layer thickness, respectively. Most notably, the temperature in the last drying interval was uncorrelated with the specific power input. A high specific power input did not result in overheating of the dried material, as the dielectric loss factor ε", and therefore energy absorption, is likely to be very low at low moisture content.

In contrast, thicker product layers were correlated with higher temperatures in the last drying interval in Figure 4 (right), indicating that water vapor transport in the product layer was the limiting factor for drying. Despite a low surrounding chamber pressure, increased internal vapor pressure led to higher temperatures due to lower moisture diffusivity. A strong correlation of the maximum temperature (R² = 0.83) in the last drying interval with layer thickness was found. Underlying variation caused by differences in drying pressure and specific power input was not significant, as shown in Figure 4 (left) but might have contributed to data variation. The implications of higher product temperatures due to thicker product layers will be further discussed in the context of powder properties and process optimization. From these observations, we can conclude that layer thickness <2 mm will be a decisive parameter to avoid brown spots and degradation of nutrients in the product, while a high specific power input can be applied for rapid MVD. Nevertheless, additional process and product characteristics such as drying efficiency, foaming, and powder properties needed to be assessed as well.

Figure 5 shows the drying efficiency η as a function of the specific power input. The average efficiency was 0.28, 0.29, and 0.31 at 1.0, 1.5, and 2.0 W·cm⁻³, respectively. These values are somewhat lower than Ambros et al. (2018a) observed for MVD of microbial cultures at lower drying pressures (7 kPa), possibly due to the higher initial moisture content of microbial cultures and differences in composition. However, overall drying times were shorter in this study compared with those reported by Ambros et al. (2018a) as no pulsed power input was used to maintain product temperatures below a critical limit.

Mitigation of Foaming During Drying

Foaming and splashing of the liquid concentrate were identified as factors that limit the selection of specific power input and pressures during MVD of CSM. At first, foaming was attributed to dissolved gas in the reconstituted concentrate. However, foaming and splashing of CSM were observed with low pressure, thicker layers of CSM, higher power input (Figure 2) or all 3 factors combined and mostly in the first drying interval when CSM was still a liquid. This suggested that the reason for foaming was the water vapor that formed during drying, not the dissolved air. This negative ef-
fect needed to be addressed to avoid product loss in the drying chamber. The water vapor flow rate was calculated based on the drying rate (eq. [1], [2]) and plotted as a function of vapor pressure and specific power (Figure 6, left). A comparison of the data in Figure 6, left, to the foaming index as a function of pressure and specific power input (Figure 6, right) suggested that the vapor flow rate could be used as a reliable predictor for foaming of CSM in the first stage of the MVD process. Thicker layers of CSM and higher specific power input will result in a higher surface specific flow rate (vapor flux) at the same specific power input. Hence, the resulting effect of the water vapor flow rate on foaming can be estimated and used to predict foaming when one or more reference values are available.

For MVD of CSM, we found that a water vapor flow rate of ~20 m\(^3\)h\(^{-1}\)kg\(^{-1}\) at 37.5% CSM is critical and should not be exceeded at a layer thickness of 2 mm. Nevertheless, controlled foaming might be beneficial because it will lead to a porous structure of the drying product, and thus higher drying rates. Therefore, foaming was included in the optimization criteria for the drying process, and quantitative correlations with product parameters were investigated.

**MVD Skim Milk Powder Properties**

To enable a correlation of processing parameters with product properties, the physical properties of the drying material were determined. Figure 7 represents the moisture (de) sorption isotherm for the MVD skim milk powder, which shows the decrease in water activity of the drying material depending on the moisture content, covering the entire range from concentrate to the final powder. The moisture content and water activity of the 2 NDM powders used in this study are shown as a reference (red diamonds). The water activity of the powder is an important parameter for its storage stability. The final water activity of the powders obtained by MVD at constant pressure and energy input after 35 min of drying time was 0.33 to 0.55, corresponding to a final moisture content of 5.0 to 8.5%, which is higher than for the initial spray-dried NDM. However, a very low moisture content was not the primary target of these...
experiments; rather, the initial goal was to create a range of properties under constant drying conditions to find correlations and most suitable drying conditions. The higher moisture and consequently water activity would result in stronger Maillard browning, protein crosslinking, caking, and nutrient loss during storage if no further drying at low microwave power input or additional air drying is applied (Pereyra Gonzales et al., 2010; Le et al., 2011). Good storage stability of the MVD powder can only be achieved by sufficiently low moisture content and water activity and a corresponding high T_g. Minimal chemical degradation for dry foods occurs in a water activity range of 0.1 to 0.4 (Kessler, 2002; Barbosa-Canovas et al., 2007), which is lower than the value achieved for the MVD milk powder. Higher moisture and water activity could lead to significant Maillard browning, protein crosslinking, caking, and nutrient loss during storage if no further drying is applied. This could be either a final MVD step at low microwave power input or low temperature air drying in a fluidized bed (Pereyra Gonzales et al., 2010; Le et al., 2011). Keeping the powder temperature as low as possible in the final drying stages is critical for maintaining high solubility of the powder, because exposure of dairy powders to dry heat reduces the solubility of proteins.

Figure 7 shows the T_g of the powder as a function of water activity, both measured values (square data points) and values calculated using a model derived by Schmitz-Schug (2014), based on the Gordon and Taylor equation (green continuous line). When comparing the T_g curve with the correlation between product water activity and product temperature, shown in the inset in Figure 7, we found that the drying process occurred exclusively in the liquid and rubbery states, above the T_g. However, greater differences between the product temperature (T) and the glass transition temperature (T – T_g), which is an indicator of higher water mobility, did not appear to result in faster drying and lower final moisture content in samples. After processing, the MVD powder reached the glassy state as it cooled to room temperature.

After drying and grinding, the MVD powders were classified by sieving and the mass fractions in each size fraction. The powders contained the following particle size fractions: (1) <75 μm: 42.4 ± 5%; (2) 75–150 μm: 26.0 ± 2.5%; (3) 150–250 μm: 20.0 ± 3.5%; (4) 250–425 μm: 9.2 ± 2.7%; (5) 425–850 μm: 2.3 ± 1.2%; and (6) >850 μm: 0.1 ± 0.1%. The size distribution was broader than that of NDM, with more coarse particles present. For comparison, the 2 batches of NDM showed most particles in the size range <75 μm (22, 42%), 75–150 μm (74, 44%), and almost no larger particles. No correlation was found between the quantity of larger size particles in the ground milk powders and the insolubility index. The slope tested by ANOVA was not significantly different from zero (P > 0.05). Hence, we can state that the particle size distribution of MVD powder was different from NDM but did not affect the solubility tests. The dispersibility of the MVD powders was significantly (P < 0.05) lower at 24°C than at 50°C; mean values were 74.1 ± 7.0% and 78.6 ± 4.2% at 24°C and 50°C, respectively. This difference was more pronounced for NDM, which
showed a dispersibility of 60 to 64% at 24°C and 80 to 85% at 50°C. The repeatability of tests was ±5% in all cases. It can be stated that the dispersibility of MVD ground powders was higher than NDM at 24°C water temperature and comparable to that of NDM at 50°C, which could be explained by differences in particle size as large particles disperse more easily in water.

The average poured and loose bulk density values for the MVD skim milk powders were 0.71 ± 0.03 and 0.91 ± 0.03 g cm⁻³, respectively. The 2 NDM batches used in this study showed an average poured bulk density of 0.65 and 0.59 g cm⁻³ and loose bulk density of 0.78 and 0.73 g cm⁻³. Poured and loose bulk density were higher for the MVD powders than for NDM, possibly because of the broader particle size distribution, which led to a denser packing of particles.

**WPNI, Browning Index, SNI, and Insolubility Index**

The amount of undenatured whey protein, as determined by the WPNI, can be used as a temperature-time integrator to assess the effect of heat treatment of dairy products exposed to temperatures >60°C. The WPNI influences the functionality and suitability of dairy powders for different applications. Powders with a WPNI >6 are considered low-heat powders (Písecký, 1997). To quantify possible heat-induced denaturation in MVD skim milk powders, their WPNI was determined after reconstitution. The hypothesis was that powders that had reached >55°C during the last drying interval(s) might show a reduced WPNI. The WPNI values for the 2 NDM batches were 6.5 and 5.8 g/kg of NDM, respectively. The mean relative WPNI across all MVD powders compared with NDM based on 100% dry powder was 97.5 ± 2.6%. The mean was significantly lower than the value for NDM (100%; \( P < 0.01 \)). However, no major differences could be observed between MVD samples processed under different conditions, and no correlations with temperatures in the last drying interval were identified. It is most likely that the reconstitution of the NDM using hot water resulted in denaturation of a small proportion of whey proteins. Interestingly, WPNI was not different, even when a high number of large brown spots were detected in the dried materials due to local overheating, especially when thicker product layers had been dried. It is possible that the unfolding of whey proteins in the almost dried material was inhibited in the water activity range.
of 0.85 to 0.3 (~15–5% moisture), whereas browning still occurred at a high rate when the nearly dry material heated up in the last MVD interval. The browning index was weakly correlated with layer thickness (data not shown); the correlation was not as strong as expected, possibly due to the rough classification (1–3) and the visual inspection.

The insolubility index of the MVD material after grinding into powder, determined at both 24 and 50°C, is shown in Figure 8. The insolubility index, sometimes called the solubility index, is defined as the volume of insoluble material sedimented by centrifugation after reconstitution of skim milk powder at either 24 or 50°C. The insolubility index was found to be especially useful in identifying critical drying conditions for MVD of CSM. The bars in Figure 8 (left) show the insolubility index at 24°C and 50°C for all drying runs performed. The drying conditions are indicated on the x-axis. This figure also shows the maximum product temperature during the last drying interval (represented as diamonds) to enable visual correlation between the 2 parameters and attribute them to individual drying conditions.

The insolubility index for the 2 batches of NDM was 0.09 ± 0.03 and 0.05 ± 0.00 mL at 24°C and 0.06 ± 0.03 and 0.05 ± 0.00 at 50°C. For the MVD powders, the lowest insolubility index values were 0.20 ± 0.04 mL at 24°C and 0.04 mL at 50°C (at 0.67 W·g⁻¹ specific power, 3.3–3.7 kPa pressure, and 1-mm layer thickness). The fact that insolubility values were slightly higher for MVD powder than for the initial NDM powder is to be expected because of the additional exposure to processing (reconstitution, storage, drying, grinding). Heating of the powder to 55 to 60°C during grinding had no effect on solubility because the insolubility at 50°C at the lowest temperatures in the last drying stage was very low, close to the insolubility index of NDM.

Figure 8 (right) shows the linear correlation between the maximum drying temperature in the last drying interval and the insolubility index determined at 24 and 50°C. A good correlation was found for both the insolubility index determined at 24°C (R² = 0.78) and at 50°C (R² = 0.77). A strong correlation was also found between the independent insolubility values at 24 and 50°C (R² = 0.95) using an exponential model (data not shown). The variability of the data in this graph was mostly attributed to the fact that the maximum temperature represents a single point in time, and not an integrated temperature history, whereas the insolubility index represents a temperature-time integrator. An outlier (13.3 kPa, 4 mm, 1.72 W·g⁻¹; open symbol) was identified for both testing temperatures by regression analysis. This outlier was likely a result of excessive foaming, splashing, and subsequent product loss (Figure 2, bottom), which resulted in lower actual layer thickness. Either of the 2 insolubility indices at 24 or 50°C can be used to define the critical temperature in the last drying interval, and thus be used as an optimization tool. Process optimization to minimize the insolubility index is also used for spray drying of either NDM or skim milk or whole milk powder (Písecký, 1997). A low insolubility index is desirable for dairy
products because it is associated with a high colloidal stability in reconstituted concentrated or dry dairy products.

By comparing the results in Figure 4 and Figure 8, we can conclude that layer thickness is likely the most decisive factor that affects product temperature, and thus the insolubility index, in the last drying interval, rather than specific power input or pressure. The effect of dry heat on the solubility and WPNI of NDM has been investigated by Chen et al. (2013), who treated milk powders with radiofrequency dielectric heating for several minutes in the temperature range of 75 to 90°C to assess the potential of radiofrequency heating on microbial inactivation of pathogens in dry powders. Those authors found that solubility, assessed using the SNI, decreased significantly at temperatures ≥80°C. Their results also indicated that the effect on solubility was related to both heating temperature and time. In our study, we found that the SNI was less sensitive than the insolubility index for detecting small changes in protein solubility. Nonetheless, we observed a significant decrease in SNI with increasing temperature in the last drying interval across all MVD samples. The slope of the linear regression for the SNI versus temperature relationship was significantly different from zero ($P < 0.05$), although the correlation was weak ($R^2 = 0.37$). The high product temperatures in the last drying stage likely resulted in protein insolubility due to the exposure of dried milk to heat, but the SNI (as a less sensitive method compared with the solubility index) might only be able to detect more severe changes in protein solubility. Maximum product temperatures below 50 to 55°C in the last drying interval will, however, ensure high solubility of MVD milk powder.

**Selection of Optimal Drying Conditions for Skim Milk Concentrate**

As a last step, we combined all observations about the effects of processing parameters on product properties. A multivariate linear model for the optimization of MVD of CSM was created based on the custom factorial design, including the drying parameters and the multiple observations related to process efficiency (dry-
Figure 8. Top: insolubility index determined at 24 and 50°C (solid bars) overlaid with the maximum product temperature in the last drying interval (diamonds) for all drying runs. On the x-axis, * indicates drying runs that showed pronounced foaming (foaming index ≥6) and ** indicates drying runs that were not part of the factorial design. Bottom: Correlation between the maximum product temperature and the insolubility index determined at 24°C (black circles; $R^2 = 0.81$) and 50°C (black diamonds; $R^2 = 0.78$). Outliers (red circle and diamond) were excluded from the regression. Max. = maximum. Error bars indicate SD.
ing time, product temperature) and product properties (foaming, browning, insolubility). Drying parameters constitute factors, whereas processing efficiency and product properties constitute response variables. The responses that were significantly affected by changes in the factors (processing parameters) are summarized in Table 2. Layer thickness affected all responses significantly, whereas specific power input and pressure had a significant effect only on drying time and foaming.

In this study, we placed more focus on the general process efficiency and powder properties, but the theoretical amount of powder (95% TS) from skim milk concentrate (37.5% TS) per batch was included as an optimization criterion. The intent was to introduce a maximization criterion, while all other responses were set as minimization criteria. Figure 9 shows the desirability functions for the different factors and the corresponding responses. The desirability transformed values of the responses into 0 and 1, where 0 represents an unacceptable value of the response and 1 represents a highly desirable value. The overall desirability was maximized, and a maximum desirability of 0.86 was obtained for 2-mm layer thickness, drying pressure of 6.0 kPa, and 1.29 W·g⁻¹ specific power input when certain limits for drying time (<35 min), foaming (<4.5), maximum drying temperature (<50°C), and insolubility index at 24°C (<0.6) were allowed, except for the output of powder, which was maximized. This set of optimization criteria is different from minimizing all target functions (indicated in the column on the right in Figure 9 as downward-pointing functions). If, for example, all desirability functions were minimized and the overall output was maximized, an overall low desirability of 0.59 was obtained, leading to the following optimal MVD conditions: 1-mm layer thickness, 13.3 kPa maximum drying pressure, and 1.29 W·g⁻¹ specific power. The desirability of these conditions will be lower because the powder output per batch will be lower. If no maximization criterion, such as powder output, was added, the desirability function would be maximum at 1-mm layer thickness, the lowest pressure, and the highest power input to minimize all responses and obtain a desirability close to unity. The latter conditions might be best for a continuous process where the overall load in the system is not decisive as these conditions achieve the highest powder solubility. In continuous operation, no downtime for loading, unloading, and applying vacuum is required. In contrast, in a batch dryer, product load and downtime for loading and applying vacuum are highly relevant.

It should be noted that the overall desirability was not as sensitive to power input and pressure as it was to layer thickness. The value of this statistical approach is that it can show how much the overall desirability, calculated as the geometric mean of the individual desirability factors, will change when another optimization criterion is chosen. In sum, this shows how process optimization for MVD can be successfully performed by integration of processing parameters, product properties, and economic aspects.

### Inactivation of E. coli and L. innocua

A low microbial load of spoilage bacteria and the absence of pathogens in milk powders is a desired quality criterion. Microwave drying has been studied as a means to dry materials that contain bacteria as probiotics and achieve a high survival (Ambros et al., 2018a,b, 2019). In contrast, Chen et al. (2013) used targeted radiofrequency heating to achieve microbial inactivation in milk powders. The effect of MVD on microbial inactivation of bacteria in concentrated milk, under the optimal drying conditions identified in this study, was investigated using 2 pathogen surrogates, *Escherichia coli* ATC 25922 (gram-negative) and *L. innocua* C2-008 (gram-positive). The CSM was inoculated with the 2 microbial strains and subjected to MVD drying at 1.29 W·g⁻¹, 6.0 to 6.7 kPa, and 2-mm layer thickness for 40 min, followed by a second drying step at 0.86 W·g⁻¹ and 6.0 to 6.7 kPa, for 20 min. This second drying step was added to lower the moisture content and water activity further for good storage stability, while keeping the drying temperature at <55°C, the critical temperature for dried milk identified in this study, to achieve acceptable solubility of the powder. *Escherichia coli* was effectively inactivated during MVD. A >4 log reduction was obtained even when product temperatures did not exceed 50°C after 40 min of drying (Table 3). In contrast, *L. innocua* counts were reduced by 1.2 log. When a second drying step at lower specific energy input was added to reduce

### Table 2. Factors and responses significantly affected by changes in these factors and their corresponding significance

| Factor                  | Response                        | logWorth [log₁₀(P-value)] |
|-------------------------|---------------------------------|---------------------------|
| Specific power input (W·g⁻¹) | Drying time                     | 4.58                      |
| Drying pressure (kPa)   | Foaming                         | 8.15                      |
| Layer thickness (mm)    | Drying time                     | 4.41                      |
|                         | Browning index                  | 2.26                      |
|                         | Foaming                         | 7.18                      |
|                         | Maximum product temperature     | 6.21                      |
|                         | Insolubility index (24°C)       | 2.40                      |
the moisture content and water activity while keeping the product temperature <55°C, *L. innocua* counts were reduced by 2.1 log. The difference in microbial inactivation between the 2 strains might be partially explained by their gram-positive and gram-negative natures, which has been found in several studies on

Figure 9. Prediction profiler output of JMP14 Pro (SAS Institute Inc.) for the microwave vacuum drying experiments for co-optimization of all responses. Desirability for each response was set to maximum (Max.) acceptable value except for the powder output, which was maximized. Values shown for each parameter on the left of the y-axis are current (optimal) value, with the mean minus SD (lower end of whiskers) and mean plus SD (upper end of whiskers) shown in brackets.
drying (Steel and Ross, 1963; Hirai, 1991; Miyamoto-Shinohara et al., 2000). Escherichia coli is reported to be especially sensitive to drying (Gontijo Filho et al., 1985; Scott and Bloomfield, 1990). It is important to note that the microbial load of the powder was further reduced by the second drying step, which reduced moisture content to <5% and achieved a water activity <0.3. A 2-way ANOVA and Tukey test for multiple comparison of the means were used to test significance. The insolubility index at 24 and 50°C were not significantly different ($F_{1,19} = 2.08, P > 0.05$) between the MVD milk powder samples with and without the additional drying steps. These findings suggest that this drying technology improves the microbial quality of MVD skim milk powder and produces powder with good solubility.

### CONCLUSIONS

In this work, MVD was optimized for the drying of CSM in a nutraReV MVD unit. Conditions found in this study might be applied for other dairy products such as cream, Greek yogurt, other dairy concentrates, or cheese, with some parameter adjustments. Processing conditions determined in this study using a factorial design approach and the exploration of the effects of processing parameters on the overall desirability could help to transfer these findings to other dairy products. The product geometry or shape—in our case, product layer thickness—was a decisive factor for MVD behavior and a predominant factor for product quality. Therefore, layer thickness during drying of liquids or semi-solid foods as a shape or product geometry factor needs attention in future MVD studies. Microwave vacuum drying might also offer an alternative drying process for infant formula to retain high solubility of the final product and viability of probiotics because low drying temperatures are beneficial for both.

### ACKNOWLEDGMENTS

This project was funded by the New York State Milk Promotion Advisory Board (Albany, NY; grant ESDC 92067-2020). We thank Kyle Kriner and George Howick from Cornell AgriTech (Geneva, NY) for their assistance with the microwave vacuum drying experiments, and Hanyu Chen and Mario Cobo from Cornell University (Ithaca, NY) for assistance with microbiological analyses. This article does not contain any studies with human or animal subjects performed by any of the authors. The authors have not stated any conflicts of interest.

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### Table 3. Moisture, water activity, and microbial inactivation (means ± SD) microwave vacuum-dried concentrated skim milk dried under optimal conditions and optimal conditions plus a second drying step

| Microorganism and drying condition | Moisture (%) | Water activity (-) | Inactivation ($\log_{10} N_0/N$) | Insolubility index at 24°C | Insolubility index at 50°C |
|-----------------------------------|--------------|-------------------|-------------------------------|---------------------------|---------------------------|
| Escherichia coli ATC 25922         |              |                   |                               |                           |                           |
| Optimal2                          | 5.92 ± 0.37  | 0.376 ± 0.015     | 4.1 ± 0.4                     | 0.48 ± 0.04               | 0.31 ± 0.04               |
| Optimal plus additional drying3    | 4.75 ± 0.84  | 0.258 ± 0.019     | >4                            | 0.53 ± 0.04               | 0.29 ± 0.04               |
| Listeria innocua C2-008            |              |                   |                               |                           |                           |
| Optimal2                          | 5.57 ± 0.24  | 0.359 ± 0.012     | 1.2 ± 0.1                     | 0.41 ± 0.03               | 0.25 ± 0.06               |
| Optimal plus additional drying3    | 4.66 ± 0.54  | 0.256 ± 0.018     | 2.1 ± 0.5                     | 0.44 ± 0.05               | 0.28 ± 0.03               |

1Where $N_0$ and $N$ represent microbial counts (in cfu·mL$^{-1}$) before and after drying.

2$1.29$ W·g$^{-1}$, 6.0–6.7 kPa, 2-mm layer thickness, 40 min.

3$1.29$ W·g$^{-1}$, 6.0–6.7 kPa, 2-mm layer thickness, 40 min, followed by $86$ W·g$^{-1}$ at 6.0–6.7 kPa, 20 min.
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