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The effect of homogenization pressure on the flavor and flavor stability of whole milk powder

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ABSTRACT

Flavor is one of the key factors that can limit the application and shelf life of dried dairy ingredients. Many off-flavors are caused during ingredient manufacture that carry through into ingredient applications and decrease consumer acceptance. The objective of this research was to investigate the effect of homogenization pressure on the flavor and flavor stability of whole milk powder (WMP). Whole milk powder was produced from standardized pasteurized whole milk that was evaporated to 50% solids (wt/wt), homogenized in 2 stages with varying pressures (0/0, 5.5/1.4, 11.0/2.8, or 16.5/4.3 MPa), and spray dried. Whole milk powder was evaluated at 0, 3, and 6 mo of storage at 21°C. Sensory properties were evaluated by descriptive analysis. Volatile compounds were analyzed by sorptive stir bar extraction with gas chromatography-mass spectrometry. Fat globule size in condensed whole milk and particle size of powders were measured by laser diffraction. Surface free fat, inner free fat, and encapsulated fat of WMP were measured by solvent extractions. Phospholipid content was measured by ultra-high-performance liquid chromatography–evaporative light scattering. Furosine in WMP was analyzed by ultra-high-performance liquid chromatography–mass spectrometry. Increased homogenization pressure decreased cardboard and painty flavors, volatile lipid oxidation compound concentrations, fat globule size in condensed milk, surface free fat, and inner free fat in WMP. Encapsulated fat increased and phospholipid-to-encapsulated fat ratio decreased with higher homogenization pressure. Surface free fat in powders increased cardboard flavor and lipid oxidation. These results indicate that off-flavors were decreased with increased homogenization pressures in WMP due to the decrease in free fat. To decrease off-flavor intensities in WMP, manufacturers should carefully evaluate these parameters during ingredient manufacture.

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INTRODUCTION

Whole milk powder (**WMP**) is produced using fat standardization, pasteurization, evaporation, homogenization, and spray drying. The resulting powder must be between 26 and 40% fat and <5% moisture (USDEC, 2005). The shelf life of WMP is generally 6 to 9 mo when stored <27°C and <65% relative humidity (USDEC, 2005). Flavor of WMP is critical because it is the number one factor influencing consumer acceptance of WMP applications (Hough et al., 2002; Lloyd et al., 2009b). Typical flavors of fresh WMP include cooked or milky, milk fat, cooked, and caramelized, whereas off-flavors such as grassy, painty, and cardboard develop during storage due to lipid oxidation (Lloyd et al., 2009a,b). Due to its higher fat content, lipid oxidation in WMP occurs more readily than in nonfat dry milk. The flavor of WMP produced in the United States is highly variable, with lipid oxidation being the main source of off-flavors (Lloyd et al., 2009a). Sources of flavor variability among manufacturers of WMP are likely due to differences in how they are processed. Factors that affect lipid oxidation and shelf life of WMP include animal feed quality, raw milk storage, heat treatment, storage conditions, water activity, and packaging conditions (Hall and Lingnert, 1984; McCluskey et al., 1997; Stapelfeldt et al., 1997; Lloyd et al., 2009b).

During spray drying, fat migrates to the surface at the expense of protein and lactose due to its hydrophobic nature (Kim et al., 2009b). Free fat is defined as fat that is not entirely coated or stabilized by amphiphilic molecules (i.e., phospholipids or protein) or protected by amorphous carbohydrates or proteins during spray drying (Vignolles et al., 2007). Spray drying parameters significantly influence the amount of free fat on the surface of the whey protein ingredients, milk protein ingredients, and milk powders (Kim et al., 2009a; Vignolles et al., 2010; Park et al., 2014). Keogh and O'Kennedy (1999) observed that higher lactose-to-protein ratios decreased the aggregation of fat globules

and free fat in spray-dried whey protein stabilized milk fat emulsions. Crystallization of lactose during storage increases free fat by increasing solvent accessibility to the fat (Saito, 1985). During WMP manufacture, homogenization is performed to decrease the fat globule size. Because homogenization of condensed whole milk before spray drying decreases fat globule size, it decreases the migration of fat to the surface of WMP (Vignolles et al., 2007). During homogenization, the milk fat globule membrane ruptures and milk proteins and phospholipids interact with the newly formed smaller fat droplets (Ye et al., 2004, 2008; Vignolles et al., 2007). Increased homogenization pressures have been reported to increase the amount of protein on the surface of the milk fat droplets in condensed whole milk (Ye et al., 2008). The increased protein on the surface of the fat droplets, along with the smaller size due to homogenization, decreases the amount of free fat in the spray-dried WMP (Tamsma et al., 1959). Spray drying conditions also cause a change in the fat droplet size as well as amount of adsorbed proteins on the fat globule surface (Ye et al., 2007; Vignolles et al., 2010).

Increased free fat has been associated with increased lipid oxidation in dried dairy ingredients (Keogh and O'Kennedy, 1999; Park et al., 2014). Because off-flavors in dried dairy ingredients are directly related to lipid oxidation, it is hypothesized that off-flavors increase with increased free fat of dairy powders (Park and Drake, 2014; Park et al., 2014). Keogh and O'Kennedy (1999) related free fat and fat globule size to flavor in spray-dried whey protein stabilized dairy emulsions with decreased free fat in general, resulting in decreased off-flavor levels. To our knowledge, previous studies regarding WMP homogenization conditions have focused on lipid oxidation rates rather than overall flavor and flavor stability. The objective of our work was to determine the effect of homogenization pressure of condensed whole milk (50% solids) on the flavor of WMP throughout shelf life.

MATERIALS AND METHODS

WMP Manufacture

Raw bovine whole milk (310 kg) and raw skim milk (80 kg) were obtained from the North Carolina State University Dairy Research and Education Unit. The whole milk was standardized to a milk SNF-to-fat ratio of 2.48 using skim milk. The percent fat and solids were measured using the Smart Turbo moisture/solids analyzer and Smart Trac II (CEM, Matthews, NC), with the percent SNF being calculated as TS − fat. The standardized milk was pasteurized at 73°C for 20 s using a plate heat exchanger (model T4 RGS-16/2, SPX Flow Technology, Greensboro, NC). The milk was subsequently cooled to 4°C and stored in a bulk tank with constant stirring until introduction into the evaporator.

Evaporation was performed on a single effect pilot-scale falling film evaporator. The milk was preheated to 50°C upon introduction into the evaporator. The calandria temperature was 71°C with a vacuum of 74.5 kPa. The condensed whole milk exiting the evaporator was 50% solids and at a temperature of 60°C. The solids content was confirmed using the Smart Turbo moisture/solids analyzer (CEM). The condensed whole milk was then homogenized with a 2 stage homogenizer (model NS2006H, GEA Niro Soavi, Parma, Italy) at one of the following pressures: 0/0, 5.51/1.38, 11.0/2.76, or 16.5/4.14 MPa at 60°C. This made treatments of 0, 6.9, 13.8, and 20.8 MPa. The order of treatments was completely randomized. The condensed whole milk was then spray dried with a pilot scale spray dryer (model Lab 1, Anhydro Inc., Soeberg, Denmark) using a 2-fluid nozzle operated at 172 kPa, with an inlet temperature of 200°C and an outlet temperature of 95°C. The entire experiment was replicated 3 times. The treatments are hereafter named 0-, 6.9-, 13.8-, and 20.8-MPa WMP based on the homogenization pressure applied to the condensed milk before spray drying.

Storage and Sampling

The fresh WMP was placed in Mylar bags (TF-4000, Impak Corp., Central City, SD; ~1 kg per bag) and heat sealed. Storage conditions were 21°C and 50% relative humidity. Whole milk powder was sampled at 0, 3, and 6 mo postmanufacture. A new bag was used for each sampling time point.

Proximate Analysis

Percent fat and moisture of the fresh WMP were measured using ether extraction (AOAC International 2000; method 932.06) and a vacuum oven (AOAC International, 2000; method 990.20) respectively.

Descriptive Sensory Analysis

Flavor of the WMP at all time points was evaluated using descriptive sensory analysis using the Spectrum method (Drake et al., 2003). The WMP was rehydrated to 10% SNF with deionized water using a hand-held blender as described by Lloyd et al. (2009b). Whole milk powder solutions were dispensed into 60-mL souf-

flé cups (Solo Cup, Highland Park, IL), lidded, and tempered to 21°C before evaluation. A trained panel (n = 8; 6 females, 2 males, aged 22–47 yr) with >200 h of experience with descriptive analysis of dried dairy ingredients evaluated the samples using an established lexicon (Drake et al., 2003; Lloyd et al., 2009a). Each panelist evaluated each rehydrated WMP in duplicate at each time point. Compusense Cloud (Compusense, Guelph, ON, Canada) was used to collect the data.

Volatile Compound Analysis

Whole milk powder was sampled at each time point for volatile compound analysis. Volatile flavor compounds were extracted by sorptive stir bar extraction (Park and Drake, 2016). Prior to analysis, stir bars (polydimethyl siloxane, 10 × 0.5 mm; Gerstel Inc., Linthicum, MD) were cleaned by soaking 10 stir bars in 40 mL of a 1:1 mixture of methanol and methylene chloride for 4 h, air-drying in a fume hood for 2 h, and conditioning at 280°C for 1 h with 75 mL/min of nitrogen gas. Whole milk powder was rehydrated to 10% SNF with HPLC-grade water as performed for sensory analysis. Selected volatile compounds (Karagül-Yüceer et al., 2001; Lloyd et al., 2009b) were extracted as described by Park and Drake (2016). Five milliliters of rehydrated WMP was placed in a 10-mL amber screw top vial with a Teflon lined lid (Gerstel Inc.) along with 10 μL of internal standard (0.81 mg/L of 2-methyl-3-heptanone in water; Sigma Aldrich, St. Louis, MO). To more efficiently extract multiple classes of compounds, sequential stir bar extraction was employed. The first stir bar was placed into the vial, lidded, and then stirred for 1 h at 800 rpm at room temperature. The stir bar was removed, briefly rinsed with water, and placed in a thermal desorption unit (**TDU**) tube. Next, 1 g of NaCl was added to the vial and another stir bar was added and then stirred again for 1 h at 800 rpm at room temperature. After stirring, the second stir bar was removed, rinsed briefly with water, and placed in the same TDU tube as the first stir bar.

The TDU tubes with stir bars were injected using an autosampler (MPS Autosampler, Gerstel Inc.). The tubes were heated to 250°C for 10 min in a TDU (Gerstel Inc.) with cryogenic trapping of the compounds at −120°C (CIS 4, Gerstel Inc.). The volatile compounds were injected onto an Agilent 7890B GC (Agilent Technologies, Santa Clara, CA) and separated using a nonpolar column (30 m × 0.25 mm × 0.25μm; ZB-5MS, Phenomenex, Torrance, CA). Oven conditions were as described by Park and Drake (2016). Helium was used as the carrier gas, with a column flow rate of 1 mL/min and a purge time of 1.2 min. Compounds were detected with an inert mass selective detector (model 5970A, Agilent Technologies) using selective ion monitoring mode. Compounds were identified using the 2014 NIST mass spectral library (NIST, 2014), retention index, and retention time of authentic standards (Sigma Aldrich) injected under identical conditions.

Fat Globule Size and Particle Size

Particle size of the dry WMP at time 0 only was measured using a Mastersizer 3000 Particle Size Analyzer with the Aero S dry powder dispenser (Malvern Instruments, Malvern, UK). Fat globule size in the condensed whole milks (50% solids) before spray drying was measured with the Mastersizer 3000 with the Hydro EV attachment with water (40°C; refractive index 1.33) as the dispersant. The refractive index used for milk fat was 1.46 for both the red and blue lights. The size range was limited to 0.192 to 18.32 μm to exclude casein micelles from the measurement. The Mastersizer 3000 analysis method was used to generate the data fit; the obscuration range was 7 to 9%.

Milk Fat Fractions and Phospholipids

Surface free fat (**SFF**) was measured in WMP at every time point following the method described by GEA (2005; method No. 10a). Inner free fat (**IFF**) and encapsulated fat (**EF**) were measured as described by Kim et al. (2009b) with minor modifications. After extracting the SFF, 1 g of WMP was weighed and mixed with 40 mL of hexane. The solutions were shaken frequently for 48 h, filtered, and dried. The IFF was measured gravimetrically. The powder from the filter was dried in a fume hood and approximately 0.5 g were mixed for 15 min with 22.5 mL of 3:1 hexane:isopropanol (vol/vol). Afterward, they were centrifuged (385 × g, 10 min, room temperature) and the clear upper layer was extracted. This was repeated once more and the extracts were pooled together. The EF was measured gravimetrically.

To quantify phospholipids in the different fat fractions, the extracted fats were redissolved in 1 mL in a mixture of chloroform and methanol (2:1 vol/vol). Neutral lipids were removed by solid phase extraction as described by Donato et al. (2011). The final polar lipid residue was dissolved in chloroform and methanol (2:1, vol/vol). The polar lipid extracts were filtered with a 0.2 μm of polytetrafluoroethylene filter into amber HPLC vials. The polar lipids were separated by ultra-high-performance liquid chromatography using an evaporative light scattering detector with a drift tube temperature of 85°C and nitrogen gas flow rate of 1.75 L/min (25 psi; Acquity H-Class, Waters Corporation, Milford, MA). A hydrophilic interaction chromatogra-

phy column was used (BEH HILIC 1.7μm 150 × 2.1 mm; Waters Corporation) with a column temperature of 30°C. Compounds of interest were quantified using standard curves ranging from 2 μg/mL to 5 mg/mL. Gradient elution was used with mobile phase A as 100% acetonitrile, B as 100% water, and C as 200 m*M* ammonium acetate adjusted to pH 5.5 with acetic acid. The gradient conditions are shown in Table 1.

Table 1. Gradient conditions for ultra-high-performance liquid chromatography analysis of phospholipids[1]

Time (min)	Flow (mL/min)	%A	%B	%C
0	0.6	95	0	5
2	0.6	95	0	5
12	0.6	73	22	5
13	0.6	95	0	5

[1]Mobile phases: A = acetonitrile, B = water, C = 200 m*M* ammonium acetate in water pH 5.5.

Furosine Analysis

Furosine (**FUR**) was measured as described by Resmini et al. (1990) with minor modifications. Approximately 230 mg of WMP was dissolved in 2 mL of HPLC-grade water and placed in a glass tube with a Teflon-lined lid (Pyrex, Greencastle, PA). Next, 6 mL of 10.6 *N* HCl was added and nitrogen gas was bubbled through the sample for 1 min. Tubes were lidded and heated to 110°C for 23 h. Upon cooling, the samples were diluted 1:10 with HPLC-grade water, centrifuged at 18,000 × *g* for 10 min at room temperature, and placed into HPLC autosampler vials (Phenomenex). Furosine was detected using ultra-high-performance liquid chromatography-MS. A C18 column was used (HSS T3, 2.1 × 100 mm, 1.7 μm; Waters Corporation) with a mobile phase of 0.1% formic acid in water run at 40°C at a flow rate of 0.5 mL/min. Furosine was detected using a single quadrupole mass spectrometer (SQ Detector 2, Waters Corporation) in ES+ mode using a mass of 255.03 *m*/*z*. Capillary and cone voltages were 2.5 kV and 25 V, respectively. A standard curve was constructed in raw milk that was determined to have no detectable FUR. Concentrations ranged from 0 to 500 mg of FUR/100 g of protein.

Statistical Analysis

Two-way ANOVA with means separation was used to analyze the data (XL Stat, Addinsoft, New York, NY). Interaction effects between homogenization pressure and storage time were investigated. Fisher's least significant difference test was used to determine differences among samples.

RESULTS AND DISCUSSION

The percent moisture and fat in the WMP was 3.28 ± 0.18 and 27.3 ± 0.33%, respectively, and were not different among treatments ($P > 0.05$). The varying homogenization treatments affected the flavor and flavor stability of the WMP ($P < 0.05$). Interaction effects were significant between homogenization pressure and storage time ($P < 0.05$). The flavor profiles of the various homogenization treatments were distinct (Table 2; Figure 1). In fresh WMP, the 0-MPa homogenization treatment had decreased aroma intensity compared with all other treatments, and caramelized flavor decreased in the order of 20.8, 13.8, >6.9, and >0 MPa ($P < 0.05$). Cardboard flavor was only detected in the 0-MPa treatment in fresh WMP. At 3 mo of storage, 0-MPa homogenization had decreased cooked flavor and increased cardboard and grassy flavors compared with all other treatments ($P < 0.05$). The 13.8-MPa treatment resulted in decreased cardboard and grassy flavors compared with all other treatments ($P < 0.05$). Grassy flavor was detected at 3 mo of storage but was not detected in any of the treatments at 0 or 6 mo of storage. Lloyd et al. (2009a) documented grassy flavor in the United States before development of painty flavor, consistent with our results. At 6 mo of storage, painty flavor was detected in the 0- and 6.9-MPa treatments, with an increase observed in 0-MPa WMP ($P < 0.05$). Cardboard flavor intensity increased in the order of 0, >6.9, >20.8, and 138 MPa ($P < 0.05$). Caramelized and cooked flavors decreased at each time point across 6 mo of storage for all treatments, except caramelized flavor for the 0-MPa treatment ($P < 0.05$). These sensory changes are consistent with those reported by Lloyd et al. (2009a,b) for commercial US WMP. Whole milk powder in the United States is characterized by milk fat, cooked, and caramelized flavors initially, followed by the development of grassy and then painty flavors as the WMP is stored and lipid oxidation occurs (Lloyd et al., 2009a,b).

Volatile compound profiles in rehydrated WMP were also distinct and were consistent with sensory profiles (Table 3; Figure 2). Interaction effects between homogenization pressure and storage time were significant ($P < 0.05$). Initially, few differences were present in volatile compound concentrations. In fresh WMP, the 0-MPa homogenization treatment had increased maltol and furaneol concentrations compared with all other treatments, decanal compared with 13.8 or 20.8 MPa, and phenyl acetaldehyde compared with 6.9 MPa ($P < 0.05$). At subsequent time points, lipid oxidation compounds generally increased in WMP produced with lower homogenization pressures than higher pressures. After 3 mo, the 0-MPa treatment had increased

Table 2. Trained panel flavor profiles of rehydrated whole milk powder across 6 mo of storage[1]

Time (mo)	Treatment (MPa)	Aroma intensity	Cooked/milky	Caramelized	Cardboard	Grassy	Milkfat	Painty
0	0	1.6[c]	3.4[a]	1.2[cd]	0.7[e]	ND[2]	2.4[a]	ND
	6.9	1.8[b]	3.4[a]	2.0[bc]	ND	ND	2.5[a]	ND
	13.8	1.9[b]	3.5[a]	2.9[a]	ND	ND	2.5[a]	ND
	20.8	1.9[b]	3.5[a]	2.9[a]	ND	ND	2.5[a]	ND
3	0	1.8[b]	2.5[c]	1.3[cd]	1.8[c]	1.6[a]	2.5[a]	ND
	6.9	2.0[ab]	2.8[b]	1.5[c]	1.2[d]	1.2[b]	2.4[a]	ND
	13.8	1.8[b]	2.8[b]	2.4[b]	0.7[e]	0.5[c]	2.5[a]	ND
	20.8	1.9[b]	2.9[b]	2.3[b]	0.8[e]	0.6[c]	2.5[a]	ND
6	0	2.4[a]	2.0[e]	1.0[d]	3.3[a]	ND	2.0[b]	1.6[a]
	6.9	2.4[a]	2.1[de]	1.2[cd]	2.5[b]	ND	2.0[b]	0.7[b]
	13.8	2.2[a]	2.3[cd]	1.9[c]	1.7[c]	ND	2.0[b]	ND
	20.8	2.2[a]	2.2[d]	1.9[c]	1.6[c]	ND	2.1[b]	ND

[a–e]Means in the same column followed by a different superscript indicate significant difference ($P < 0.05$).

[1]Attributes were scored using a 0 to 15 point universal intensity scale (Lloyd et al., 2009a). Sweet taste intensity and astringency were not different among the whole milk powders at any time point ($P < 0.05$), x = 2.3 and 1.8, respectively.

[2]ND = not detected.

hexanal and heptanal compared with all other treatments, phenyl acetaldehyde compared with 13.8 MPa, and decanal compared with 138 or 20.8 MPa ($P < 0.05$). Vanillin concentration decreased at 3 mo of storage when condensed whole milk was homogenized at 0 or 6.9 MPa compared with 138 or 20.8 MPa ($P < 0.05$). At 6 mo of storage, the 0-MPa treatment had increased concentrations of octanal, nonanal, decanal, 2,4-nonadienal, E2-nonenal, E2-decenal, 1-octen-3-one, 3-octen-2-one, o-aminoacetophenone, gamma-decalactone, and gamma-dodecalactone compared with all other treatments ($P < 0.05$). Homogenization at 138 or 20.8 MPa

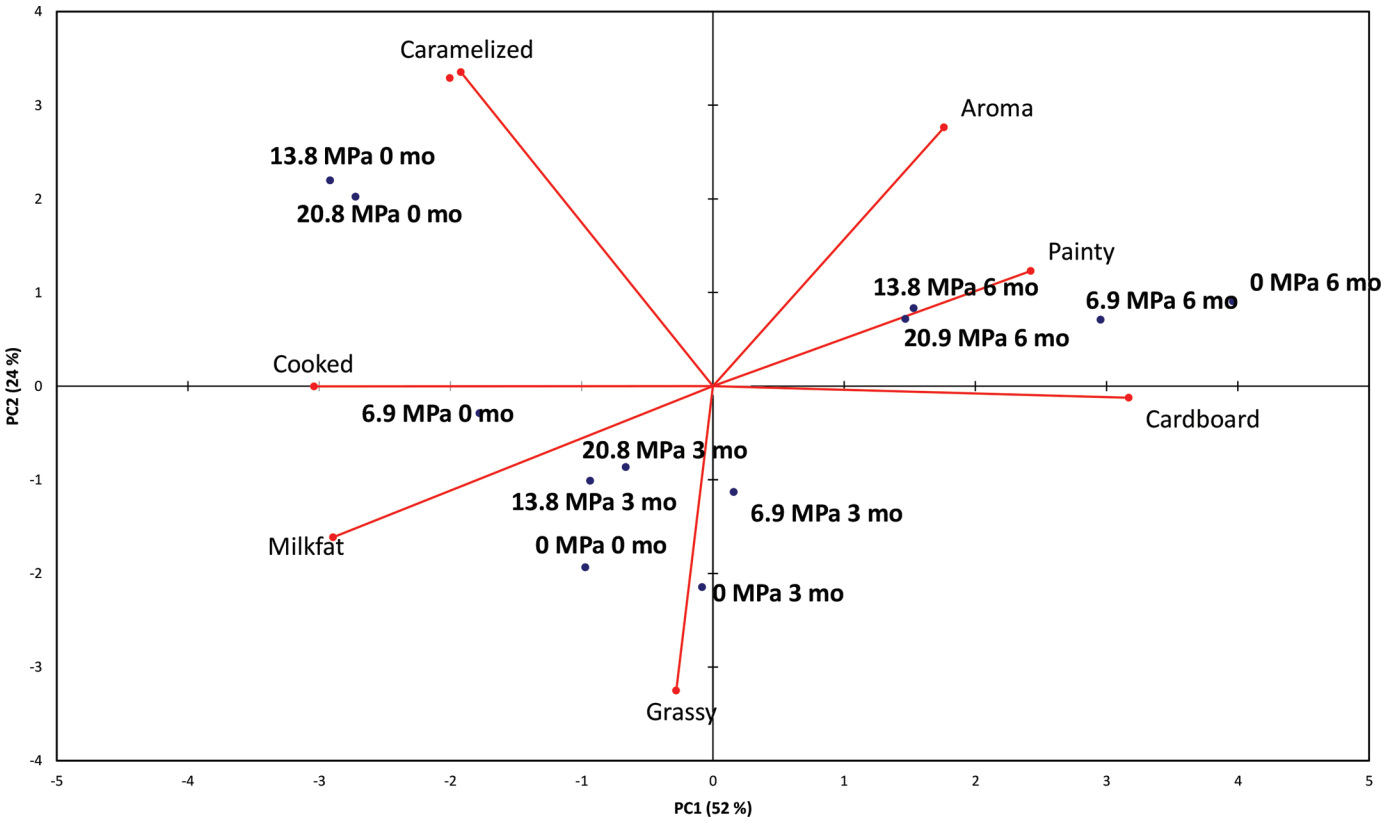

Figure 1. Principal component (PC) biplot of descriptive sensory analysis means of rehydrated whole milk powder across 6 mo of storage and 4 treatment pressures (0, 6.9, 13.8, and 20.8 MPa). Color version available online.

decreased concentrations of 2-heptanone compared with 0 or 6.9 MPa and delta-tetralactone compared with 0 MPa ($P < 0.05$). The concentrations of hexanal, heptanal, and 2,4-decadienal were in the order of 0, >6.9, >138, and 20.8 MPa ($P < 0.05$). Homogenization at 13.8 MPa decreased E,Z-3,5-octadien-2-one and E,E-3,5-octadien-2-one compared with 0 or 6.9 MPa, and homogenization at 20.8 MPa decreased E,Z-3,5-octadien-2-one and E,E-3,5-octadien-2-one compared with 0 MPa ($P < 0.05$). Collectively, these results demonstrate that although storage time plays the most critical role in flavor stability of WMP, improper or inadequate homogenization can also negatively affect shelf stability of WMP.

Lloyd et al. (2009a) reported that increased 3-methylbutanal, 2-methylbutanal, hexanal, octanal, and 3-octen-2-one concentrations were predictors of painty flavor development in WMP. In the current study, we observed that the 0-MPa treatment, the sample with the highest painty flavor intensity at 6 mo of storage, also had the highest concentrations of hexanal and octanal after 3 mo of storage and the highest 3-octen-2-one concentration at 6 mo of storage. Additionally, with the use of sorptive stir-bar extraction, we were able to extract more volatile compounds than Lloyd et al. (2009a), including heavier molecular weight compounds, because in the sorptive stir-bar extraction the stir bars are immersed into the liquid and more volatile compounds and a wider class of volatile compounds are recovered. With solid-phase microextraction, compounds must be in the headspace of the sample vial; therefore, heavier molecular weight compounds are extracted less efficiently. Examples of compounds we measured throughout storage that Lloyd et al. (2009b) did not include lactones, vanillin, and o-aminoacetophenone.

In general, lipid oxidation solid-phase microextraction increased with increasing storage time, similar to what was observed by Lloyd et al. (2009b), who used solid-phase microextraction. Specifically, hexanal, heptanal, octanal, o-aminoacetophenone, phenyl acetaldehyde, and lactones increased in all treatments at each subsequent time point ($P < 0.05$). Likewise, E,Z-3,5-octadien-2-one, E,E-3,5-octadien-2-one, 3-octen-2-one, and 2-heptanone were only detected after 6 mo of storage. In the 0-MPa treatment, decanal, 2,4-nonadienal, 2,4-decadienal, E2-nonenal, and E2-decenal increased at every time point, and nonanal and 1-octen-3-one increased after 6 mo of storage ($P < 0.05$). In the 6.9-MPa treatment, nonanal, 2,4-nonadienal, and 1-octen-3-one increased with increasing storage and 2,4-decadienal, E2-nonenal, and E2-decenal increased and vanillin decreased after 6 mo of storage ($P < 0.05$). When homogenized at either 138 or 20.8 MPa, 6 mo of storage increased nonanal, decanal, 2,4-nonadienal, 2,4-decadienal, E2-nonenal, E2-decenal, and 1-octen-3-one concentrations in WMP compared with 0 or 3 mo of storage ($P < 0.05$).

Furosine is an indicator of Maillard reactions in dairy products and can be used to measure heat treatment (Mehta and Deeth, 2015). When dairy products are heated, lysine residues from proteins react with lactose, a reducing sugar, to form lactulosyl-lysine, which is converted to FUR by acid hydrolysis (Mehta and Deeth, 2015). Both homogenization and storage time affected the FUR content in WMP, and interaction effects between homogenization and storage time were not significant ($P > 0.05$). Homogenization decreased FUR in 6.9- and 20.8-MPa treatments compared with no homogenization control (46.7 vs. 47.3 vs. 51.2 mg of FUR/100 g of protein, respectively; $P < 0.05$). As expected, FUR increased with 6 mo of storage compared with 0 or 3 mo (55.2 vs. 44.7 vs. 45.8 mg of FUR/100 g of protein, respectively; $P < 0.05$). Furosine has been reported to increase during storage of dried dairy ingredients, even at ambient temperatures (Le et al., 2011; Smith et al., 2016).

Particle size analysis indicated differences among treatments before and after spray drying (Table 4, Table 5). As expected, fat globule size decreased with increased homogenization pressure in the order of 0, >6.9, 13.8, and >20.8 MPa ($P < 0.05$), as observed for 90% of particles below that value [**D(90)**; 4.56 vs. 2.10 vs. 1.94 vs. 1.71 μm, respectively] and volumetric mean (1.71 vs. 0.85 vs. 0.83 vs. 0.77 μm, respectively). In the spray-dried WMP, the D(90) particle size decreased in the 6.9-MPa treatment compared with 0, 13.8, and 20.8 MPa (118 vs. 130 vs. 130 vs. 128 μm, respectively; $P < 0.05$).

Varying the homogenization pressure also affected the distribution of milk fat in the spray-dried WMP (Table 6). It should be noted that although our evaporator had only one effect and commercial evaporators have multiple effects, we were still able to achieve similar free fat levels to values reported in commercial WMP by Lloyd et al. (2009b). Interaction effects between homogenization pressure and storage time were not significant and storage time did not affect milk fat distribution ($P > 0.05$). The SFF decreased with increasing homogenization pressure in the order of 0, >6.9, >13.8, and 20.8 MPa (5.16 vs. 3.32 vs. 2.22 vs. 1.85 g of SFF/100 g of WMP, respectively; $P < 0.05$). The IFF decreased when WMP was homogenized at 13.8 or 20.8 MPa compared with 0 or 6.9 MPa (1.79 vs. 1.67 vs. 3.62 vs. 3.12 g of IFF/100 g of WMP, respectively; $P < 0.05$). Milk fat encapsulation increased with increasing homogenization pressure in the order of 13.8, 20.8, >6.9, and >0 MPa (23.5 vs. 24.1 vs. 20.2 vs. 17.1 g of EF/100 g of

Table 3. Relative abundance (μg/kg) of selected volatile compounds of whole milk powder throughout 6 mo of storage

	0 mo				3 mo				6 mo			
Compound	0 MPa	6.9 MPa	13.8 MPa	20.8 MPa	0 MPa	6.9 MPa	13.8 MPa	20.8 MPa	0 MPa	6.9 MPa	13.8 MPa	20.8 MPa
Hexanal	1.03^{f}	0.983^{f}	0.910^{f}	0.895^{f}	10.3^{d}	6.70^{e}	5.89^{e}	6.10^{e}	53.6^{a}	20.3^{b}	15.4^{c}	15.3^{c}
Heptanal	0.978^{f}	0.907^{f}	0.734^{f}	0.749^{f}	4.64^{d}	3.17^{e}	3.04^{e}	2.93^{e}	21.9^{a}	9.23^{b}	7.09^{c}	6.90^{c}
Octanal	0.638^{d}	0.595^{d}	0.596^{d}	0.599^{d}	1.83^{bc}	1.44^{c}	1.29^{c}	1.30^{c}	7.52^{a}	2.42^{b}	2.19^{b}	2.14^{b}
Nonanal	4.10^{cde}	3.10^{e}	3.57^{de}	3.17^{e}	5.48^{c}	4.96^{cd}	4.66^{cde}	3.99^{cde}	14.5^{a}	7.46^{b}	7.52^{b}	7.24^{b}
Decanal	0.410^{de}	0.305^{efg}	0.241^{fg}	0.236^{g}	0.546^{bc}	0.458^{cd}	0.371^{def}	0.347^{defg}	0.990^{a}	0.584^{bc}	0.610^{b}	0.601^{b}
2,4-Nonadienal	0.036^{e}	0.025^{e}	0.017^{e}	0.021^{e}	0.421^{cd}	0.312^{d}	0.244^{de}	0.252^{de}	1.99^{a}	0.922^{b}	0.658^{bc}	0.688^{b}
2,4-Decadienal	0.068^{e}	0.046^{e}	0.038^{e}	0.037^{e}	0.957^{cd}	0.558^{de}	0.490^{de}	0.468^{de}	4.16^{a}	2.29^{b}	1.47^{c}	1.47^{c}
E2-Nonenal	0.125^{d}	0.088^{d}	0.062^{d}	0.074^{d}	0.584^{c}	0.339^{cd}	0.326^{cd}	0.341^{cd}	4.41^{a}	1.15^{b}	0.967^{b}	1.04^{b}
E2-Decenal	0.073^{e}	0.043^{e}	0.031^{e}	0.037^{e}	0.277^{d}	0.156^{de}	0.160^{de}	0.165^{de}	2.86^{a}	0.819^{bc}	0.756^{c}	0.980^{b}
1-Octen-3-one	0.114^{cd}	0.062^{d}	0.021^{d}	0.027^{d}	0.267^{c}	0.231^{c}	0.171^{cd}	0.152^{cd}	3.65^{a}	0.549^{b}	0.480^{b}	0.519^{b}
EZ-3,5-Octadien-2-one	ND[1]	ND	ND	ND	ND	ND	ND	ND	24.3^{a}	21.9^{ab}	15.1^{c}	19.3^{bc}
EE-3,5-Octadien-2-one	ND	ND	ND	ND	ND	ND	ND	ND	16.9^{a}	13.2^{b}	8.95^{c}	11.9^{bc}
3-Octen-2-one	ND	ND	ND	ND	ND	ND	ND	ND	15.1^{a}	5.70^{b}	4.60^{b}	5.91^{b}
2-Heptanone	ND	ND	ND	ND	ND	ND	ND	ND	31.3^{a}	31.2^{a}	20.4^{b}	20.4^{b}
O-Aminoacetophenone	0.003^{d}	0.002^{d}	0.002^{d}	0.002^{d}	0.008^{c}	0.007^{c}	0.007^{c}	0.008^{c}	0.023^{a}	0.016^{b}	0.013^{b}	0.017^{b}
Phenyl acetaldehyde	0.206^{de}	0.158^{f}	0.172^{ef}	0.172^{ef}	0.253^{bc}	0.234^{bcd}	0.210^{de}	0.218^{cd}	0.316^{a}	0.317^{a}	0.278^{ab}	0.276^{ab}
Maltol	0.561^{a}	0.118^{b}	0.116^{b}	0.105^{b}	0.174^{b}	0.165^{b}	0.123^{b}	0.120^{b}	0.248^{b}	0.155^{b}	0.174^{b}	0.143^{b}
Furaneol	0.052^{ab}	0.021^{ef}	0.019^{f}	0.017^{f}	0.046^{abc}	0.044^{abcd}	0.037^{bcd}	0.032^{de}	0.053^{a}	0.033^{cde}	0.050^{ab}	0.040^{abcd}
Vanillin	0.014^{bcd}	0.012^{cd}	0.013^{bcd}	0.014^{abc}	0.011^{def}	0.012^{cde}	0.016^{a}	0.016^{ab}	0.011^{cdef}	0.007^{g}	0.009^{efg}	0.008^{fg}
Delta-nonalactone	0.674^{d}	0.567^{d}	0.621^{d}	0.647^{d}	5.12^{c}	5.62^{c}	5.74^{bc}	5.59^{c}	7.07^{a}	6.01^{bc}	5.57^{c}	6.65^{ab}
Delta-decalactone	9.10^{d}	7.48^{d}	8.87^{d}	9.14^{d}	62.3^{c}	65.7^{c}	64.8^{c}	66.5^{c}	93.3^{a}	81.2^{b}	80.8^{b}	88.0^{ab}
Delta-dodecalactone	8.19^{d}	6.83^{d}	7.76^{d}	7.82^{d}	65.2^{c}	68.8^{c}	67.1^{c}	69.1^{c}	99.3^{a}	87.6^{b}	88.5^{b}	92.5^{ab}
Delta-tetralactone	3.05^{d}	2.11^{d}	2.25^{d}	2.23^{d}	29.6^{c}	27.6^{c}	27.6^{c}	26.2^{c}	42.4^{a}	38.1^{ab}	36.5^{b}	36.8^{b}
Gamma-decalactone	0.132^{d}	0.150^{d}	0.130^{d}	0.124^{d}	0.901^{c}	0.895^{c}	1.12^{c}	1.03^{c}	3.68^{a}	1.91^{b}	1.94^{b}	1.95^{b}
Gamma-dodecalactone	0.923^{d}	0.598^{d}	0.651^{d}	0.674^{d}	11.8^{c}	10.4^{c}	11.8^{c}	11.1^{c}	21.3^{a}	17.8^{b}	17.8^{b}	19.0^{ab}

[a–g]Means in the same row followed by a different superscript indicate significant difference ($P < 0.05$).

[1]ND = not detected.

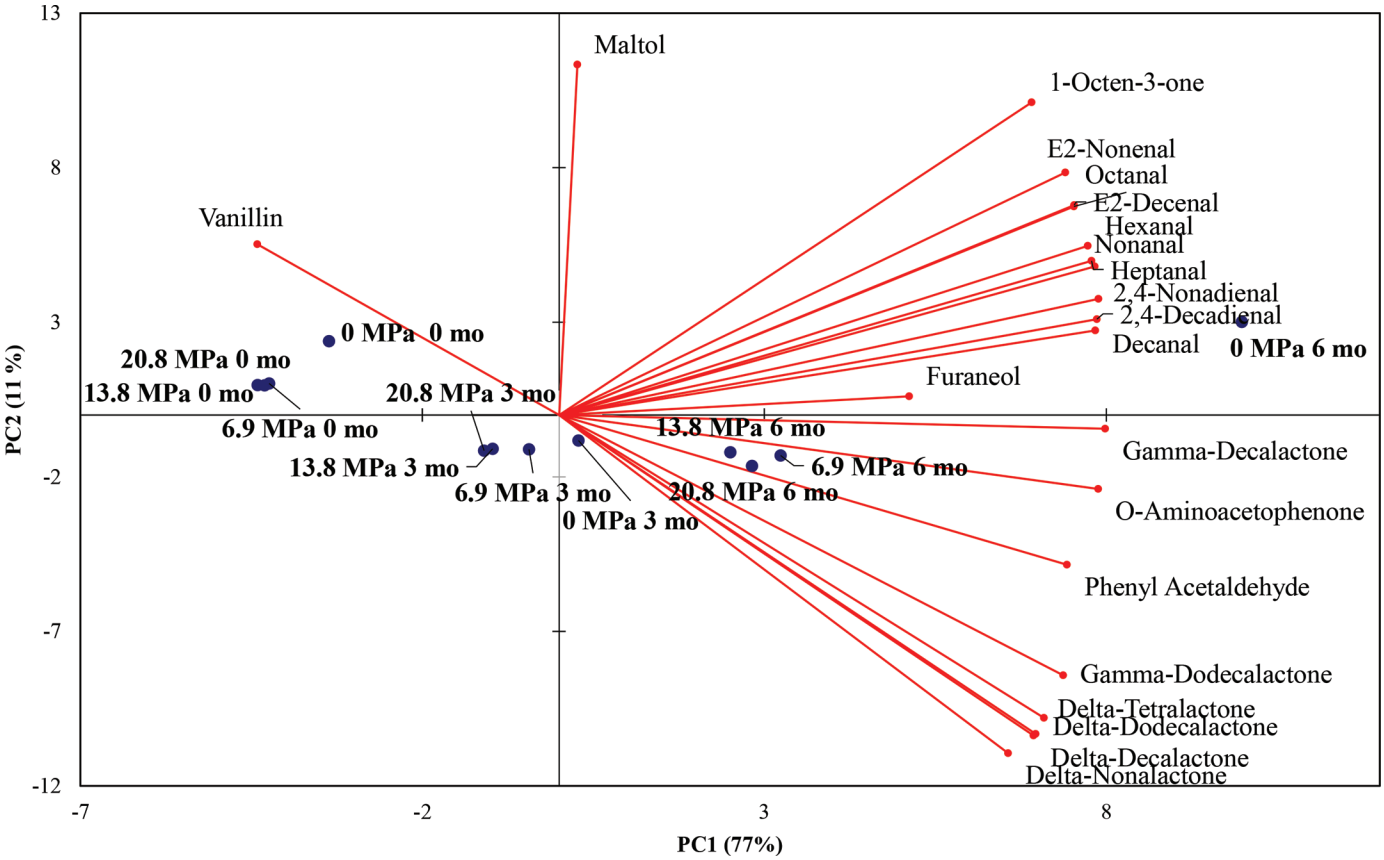

Figure 2. Principal component (PC) biplot of volatile compound analysis of whole milk powder through 6 mo of storage and 4 treatment pressures (0, 6.9, 13.8, and 20.8 MPa) at 21°C. Color version available online.

WMP, respectively; $P < 0.05$). Phospholipids were not detected in the SFF or IFF fractions. In the EF fraction, grams of phospholipids per 100 g of EF increased with decreasing homogenization pressure in the order of 20.8, 13.8, <6.9, and <0 MPa (0.77 vs. 0.88 vs. 1.25 vs. 1.75, respectively; $P < 0.05$).

Previous work has demonstrated that lipid oxidation is the primary source of off-flavors in WMP (Hall and Lingner, 1984; Lloyd et al., 2009a,b). Increases in lipid oxidation during storage of WMP, such as we observed, have also been documented extensively (Ulberth and Roubicek, 1995; Celestino et al., 1997; Stapelfeldt et al., 1997; Lloyd et al., 2009b). The off-flavors associated with lipid oxidation include cardboard, grassy, and painty (Lloyd et al., 2009a; Whitson et al., 2010). Lipid oxidation is slowed in WMP by several steps. First, extended storage of raw milk should be avoided (Celestino et al., 1997) to ensure high microbial quality. Increased heat treatment during the manufacture of WMP decreases lipid oxidation (McCluskey et al., 1997; Stapelfeldt et al., 1997). Calligaris et al. (2004) demonstrated that the antioxidant activity in milk decreased with short

Table 4. Particle size of fresh whole milk powder homogenized at varying pressures[1]

Treatment (MPa)	D(90) (μm)	D[4,3] (μm)
0	130[a]	65.5[a]
13.8	130[a]	66.6[a]
20.8	128[a]	65.6[a]
6.9	118[b]	60.0[b]

[a,b]Means in the same column followed by different superscripts indicate significant differences ($P < 0.05$).

[1]D(90) = 90% of particles below that value; D[4,3] = volumetric mean.

Table 5. Milk fat globule size in condensed whole milks (50% solids) before spray drying[1]

Treatment (MPa)	D(90) (μm)	D[4,3] (μm)
0	4.56[a]	1.71[a]
6.9	2.10[b]	0.85[b]
13.8	1.94[b]	0.83[b]
20.8	1.71[c]	0.77[c]

[a–c]Means in the same column followed by a different superscript indicate significant differences ($P < 0.05$).

[1]D(90) = 90% of particles below that value; D[4,3] = volumetric mean.

heat treatments and increased with more prolonged heat treatments. The increase in antioxidant activity with longer heat treatments is due to the unfolding of proteins and the exposure of sulfhydryl groups (Tong et al., 2000; Calligaris et al., 2004). Stapelfeldt et al. (1997) observed increased lipid oxidation with elevated temperatures (45 vs. 25°C) and water activity (0.31 vs. 0.11 vs. 0.13) in WMP. Storage conditions also play a large role in the flavor stability of WMP. Lloyd et al. (2009b) demonstrated that flushing the WMP packaging with nitrogen significantly improved the shelf life and reduced lipid oxidation throughout 1 yr of storage. The WMP in our study was given a mild heat treatment (73°C for 20 s), which thus limited the potential antioxidant activity. The pasteurization temperature that we used was similar to temperatures used by US WMP manufacturers. We observed a 6-mo shelf life, which is consistent with the shelf life of US WMP as reported by Lloyd et al. (2009a). Sensory and volatile compound profiles initially and across shelf life for the 13.8-MPa treatment were consistent with commercial US WMP (Lloyd et al., 2009a).

It has been hypothesized that the distribution of milk fat influences flavor and lipid oxidation in dried dairy particles (Park and Drake 2014; Park et al., 2014). Similar to what we observed, Vignolles et al. (2010) observed that homogenization of milk emulsions decreased the free fat content in the resulting spray-dried powders regardless of the total fat content. Also, similar to our study, Vignolles et al. (2010) observed decreased fat globule size with 20-MPa homogenization, but did not test multiple homogenization pressures as we did. The decreases in free fat that we observed with higher homogenization pressure may be due to the decrease in fat globule size and the encapsulation of the fat with proteins and phospholipid, as observed in the increased amount of encapsulated fat. The smaller fat globule size also decreased the SFF because it could potentially reduce the creaming velocity of the fat in the atomized droplets during spray drying.

The increase in milk fat encapsulation observed in higher homogenization pressures most likely decreased lipid oxidation due to the antioxidant activity of the proteins adsorbed onto the surface of the fat droplets (Lethuaut et al., 2002). Park et al. (2014) reported that in spray-dried whey protein concentrate 80% (**WPC80**), lipid oxidation products increased in powders with increased SFF. Lipid oxidation in PUFA is reduced when free fat content is reduced by means of microencapsulation (Gharsallaoui et al., 2007). Homogenization of condensed whole milk, as performed in the current study, could be seen as a microencapsulation step because increasing amounts of proteins are adsorbed onto the surface of the milk fat globules (Ye et al., 2004). We observed, similar to Park et al. (2014) in WPC80, that SFF increased lipid oxidation and cardboard flavor in WMP. Park et al. (2014) did not evaluate the spray-dried WPC80 throughout shelf life. We have demonstrated that not only is lipid oxidation elevated initially with increased SFF, but that it also increases lipid oxidation throughout shelf life.

Decreased particle size has been reported to increase SFF (Park et al., 2014). Whereas we observed decreased particle size in the 6.9-MPa treatment, it did not increase SFF compared with the 0-MPa treatment. This is most likely due to the homogenization that occurred and the relatively small difference in particle size (118 vs. 130 μm). These results indicate that spray drying parameters influence the particle size of the powders more than homogenization (Kim et al., 2009b; Park et al., 2014).

Native phospholipids in dairy products have been reported to have both pro- and antioxidant activity. The antioxidant activity is mainly due to their ability to chelate pro-oxidant metals (Chen and Nawar, 1991). The pro-oxidant activity of phospholipids is 2-fold. First, a high degree of unsaturation exists in the fatty acids associated with phospholipids (Sessa, 1985). Second, phospholipids reduce the surface tension of the fat droplets, which allows for greater oxygen uptake and, therefore, increased lipid oxidation (Choe and Minn, 2006). Whereas total phospholipids was not different among treatments, the ratio of phospholipid to encapsulated fat increased in the 0- and 6.9-MPa

Table 6. Fat fractions of whole milk powder (WMP) treatments[1]

Treatment (MPa)	g SFF/100 g of WMP	g IFF/100 g of WMP	g EF/100 g of WMP	g PL/100 g of EF
0	5.16[a]	3.62[a]	17.1[c]	1.75[a]
6.9	3.32[b]	3.12[a]	20.2[b]	1.25[b]
13.8	2.22[c]	1.79[b]	23.5[a]	0.90[c]
20.8	1.85[c]	1.67[b]	24.1[a]	0.93[c]

[a–c]Means in the same column followed by a different letter indicate significant differences ($P < 0.05$).

[1]Storage time had no effect on fat fractions ($P > 0.05$). SFF = surface free fat; IFF = inner free fat; EF = encapsulated fat; PL = phospholipids.

treatments due to decreased total encapsulated fat. The increased phospholipids around the encapsulated fat could partially explain the increased lipid oxidation and off-flavors in WMP homogenized at 0 or 6.9 MPa compared with 13.8 or 20.8 MPa. The increase in encapsulated fat in the 13.8- and 20.8-MPa treatments could have also decreased lipid oxidation because of the proteins adsorbed on the surface of the fat globules. Milk proteins have been reported to have a high antioxidant activity (Cervato et al., 1999; Tong et al., 2000).

Furosine is an indicator of early Maillard reactions and can be used as an indicator of heat treatment in dairy products. We observed that homogenization affected the FUR content in the spray-dried WMP very little. Whereas 0 MPa was statistically higher than 6.9 and 20.8 MPa ($P < 0.05$), the greatest difference was only 4.5 mg of FUR/100 g of protein. As expected, storage increased FUR levels in WMP, which has been reported in other dried dairy ingredients (Le et al., 2011; Smith et al., 2016). Whereas increased Maillard reaction products have been reported as having antioxidant activity, they are brown melanoidins, which are late Maillard products, as opposed to FUR, which is an indicator of early Maillard reactions (Calligaris et al., 2004). As such, the increased Maillard reactions observed in the 0-MPa treatment and after 6 mo of storage for all treatments would not be expected to affect the antioxidant activity and, therefore, flavor stability of the resulting WMP.

CONCLUSIONS

Homogenization pressure significantly affects the flavor and flavor stability of WMP. Our results indicate that WMP flavor stability was improved with increased homogenization pressure and decreased fat globule size in the condensed whole milk. This led to decreased free fat and an increase in fat encapsulation. Particle size of the resulting WMP was not affected by homogenization. Homogenization had little effect on Maillard reactions, as observed by FUR levels in the WMP. Manufacturers of WMP should continually evaluate the effectiveness of their homogenizer to improve the flavor and shelf life of their product with the goal of smaller fat globule size in their condensed whole milk.

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