DAIRY FOODS: **TECHNICAL ORAL SESSION: ANALYTICAL/PROCESSING**

0257 Modification of the functionality of micellar casein concentrates by changing the structure of casein micelles using high pressure processing. C. I. Moraru[*1], M. Walkling-Ribeiro[1], I. Aprodu[2], and M. V. Karwe[3], *[1]Cornell University, Ithaca, NY, [2]Dunarea de Jos University, Galati, Romania, [3]Rutgers University, New Brunswick, NJ.*

Growing interest in food products with high protein content has led to an increased demand for protein ingredients. Micellar casein concentrate (MCC) is an emerging dairy ingredient obtained by membrane filtration. MCC is characterized by a bland, clean taste and has potential for use in applications ranging from beverage fortification to manufacturing of soft gel products. High pressure processing (HPP) is a physical process that can be used to induce controlled changes in the structure of casein micelles and thus modify the properties and functionality of MCC. In this study, MCC suspensions with 2.5, 5, 7.5, and 10% protein content were processed at 150, 250, 350, and 450 MPa for 15 min, at cold (4-24°C) and warm (53−60°C) temperatures. Particle size, turbidity, and viscosity were assessed directly after HPP and during refrigerated storage. The study was replicated and data analyzed statistically. Under cold HPP conditions, casein micelle size decreased significantly with increasing pressure ($P < 0.05$) due to disruption of the casein micelle structure. The average micelle diameters ranged from 192 to 81 nm for 2.5% MCC and from 216 to 173 nm for 10% MCC after treatment at 150 and 450 MPa, respectively. These effects were concentration dependent, indicated by increased micelle size and turbidity in samples with higher casein concentration ($P < 0.05$). By contrast, HPP under warm conditions led to an increase in particle size, indicating a re-association of caseins. The increase in particle size was concentration dependent: for 2.5% MCC treated under warm conditions, particle sizes ranged between 175 and 216 nm, while for 10% MCC they ranged between 192 and 778 nm when treating the samples at 150 and 350 MPa, respectively. Particle size and turbidity did not change significantly during storage at 4°C of cold HPP treated samples, whereas samples treated under warm HPP conditions were less stable. Interestingly, cold HPP treated 10% MCC formed a weak gel above 250 MPa. The present study suggests that HPP is effective for modifying MCC functionality by inducing structural changes of the casein micelles. Most notably, cold HPP can induce shelf-stable size reduction of casein micelles and improved transparency at low casein concentrations, and gel-like structure at high casein concentrations. This data can be used as a basis for developing new food applications involving HPP treatment of MCC.

Key Words: micellar casein concentrate (MCC), high pressure processing (HPP), nonthermal processing

0258 Microfiltration of milk protein concentrate using ceramic membranes: Determination of limiting flux and serum protein removal at 8, 9, or 10% protein in the recirculation loop. E. E. Hurt[*1,2], M. C. Adams[1,2], and D. M. Barbano[1,2], *[1]Cornell University, Ithaca, NY, [2]Northeast Dairy Foods Research Center, Ithaca, NY.*

In designing a MF process to separate SP. from casein, both the amount of SP. removed and flux are likely to be a function of the protein concentration in the recirculation loop (RL). Our objective was to determine the limiting flux and SP. removal at 8, 9 or 10% protein in the RL using 0.1µm ceramic graded permeability membranes with 4 mm channel diameters. The MF feed was an 85% milk protein concentrate diluted to an average protein of 5.5 ± 0.1%. The concentration factor was chosen to achieve 8, 9 or 10% protein in the RL. The MF was operated with a longitudinal pressure drop of 220 kPa at 50°C. The MF was started at an average flux of 55 ± 2kg/m^2 per h and flushed until the protein concentration in the RL was near the target. Once the target protein was reached, the MF was run at 55 kg/m^2 per h for 1h, the flux was then increased in steps and run for 1h at each new flux. The flux was increased until the new flux could not be maintained. The limiting flux was the last flux the MF could run at for 1h. Transmembrane pressure averaged 66 kPa at 55kg/m^2 per h and 190 kPa at the limiting flux and transmembrane pressure did not vary with protein concentration in the RL ($P > 0.05$). Retentates and permeates at each flux were analyzed for true protein (TP) using Kjeldahl methods. SP. removal as a percentage of theoretical removal (SPR) was calculated as TP in the permeate divided by SP. in the permeate portion of the feed. TP concentrations in the RL averaged: 8.2 ± 0.1, 9.2 ± 0.1 and 10.1 ± 0.2%. Cross-flow velocities depended on the protein concentration in the RL ($P < 0.05$) and were: 7.09, 7.01 and 6.90m/s at 8, 9, and 10% protein, respectively. Limiting fluxes decreased with increasing protein in the RL ($P < 0.05$) and were: 154 ± 1, 133 ± 1 and 117 ± 6kg/m^2 per h. SPR was not a function of the protein concentration in the RL ($P > 0.05$), but SPR decreased ($P < 0.05$) from 80% at 55 kg/m^2 per h to 75% at the limiting flux, indicating fouling may have impacted passage of SP. through the membrane. The protein concentration in the RL that the MF operated at had an impact on the limiting flux, but not passage of SP. However, as flux increased to the limiting flux, there was reduced SP. passage through the membrane, likely caused by membrane fouling.

Key Words: limiting flux, microfiltration, milk protein concentrate

0259 Impact of membrane channel diameter on limiting flux and serum protein removal during milk protein concentrate microfiltration. M. C. Adams*, E. E. Hurt, and D. M. Barbano, *Cornell University, Ithaca, NY.*

The design of a ceramic microfiltration membrane will impact its ability to remove serum proteins (SP) from milk. Our objectives were to determine the limiting fluxes and SP. removal as a percentage of theoretical (SPR) of 0.1 μm ceramic graded permeability membranes with either 3 mm or 4 mm diameter flow channels. A microfiltration process was fed with an 85% milk protein concentrate that had been standardized to 5.62 ± 0.06% protein with reverse osmosis water. Retentate and permeate were continuously recycled to the feed tank. Limiting fluxes were determined by incrementally increasing flux once per h from 55 kg/m^2 per h until flux became independent of transmembrane pressure. Experiments were replicated 3 times and the Proc GLM procedure of SAS was used for statistical analysis. Temperature, longitudinal pressure drop (ΔP), and protein concentration in the retentate recirculation loop were maintained at 50°C, 220 kPa, and 9.16 ± 0.08%, respectively. Because the graded permeability membranes are designed to operate at ΔP between 200 and 220 kPa, ΔP was controlled. Consequently, 3mm cross-flow velocity was lower ($P < 0.001$) than 4 mm cross-flow velocity (5.48 vs. 7.00 m/s). In both membranes, cross-flow velocity decreased ($P < 0.05$) between the initial and limiting fluxes. The 3 mm membrane limiting flux was lower ($P < 0.001$) than the 4 mm membrane limiting flux (105 vs. 133 kg/m^2 per h). SPR was calculated by dividing true protein in the permeate by SP. in the permeate portion of the feed to describe the ease of SP. passage through the membrane. In the 4 mm membrane, SPR decreased ($P = 0.03$) between the initial and limiting fluxes due to fouling. No decrease in SPR was detected ($P > 0.10$) in the 3 mm membrane between the initial and limiting fluxes. Experimental variation and the fact that 3mm SPR was lower ($P = 0.07$) than 4 mm SPR at the initial flux contributed to this finding. Despite a lower limiting flux and a higher rejection of SP, the modular SP. removal rate (kg SP. removed/module per h) of 3mm membranes would be higher than that of 4mm membranes because 46% more membrane surface area can be housed in a 3mm membrane module. This relationship could change if the retentate protein concentration were different. A processor should consider both the increased performance of the 4 mm membrane and the reduced cost per module of the 3 mm membrane before proceeding with a purchasing decision.

Key Words: microfiltration, serum protein removal, limiting flux

0260 Using membrane filtration techniques to fractionate acid whey into value added ingredients. B. Chen*, K. E. Smith, J. A. Lucey, R. Kalscheuer, and M. Molitor, *University of Wisconsin, Madison.*

There has been a huge expansion in acid whey production due to the rapid growth in Greek yogurt manufacture; therefore it is critical to find an environmentally friendly and economically feasible way to process acid whey. Membrane filtration techniques have been used for many decades in the dairy industry to fractionate components into different streams. The objective of this study was to determine the suitability of nanofiltration (NF) membranes for fractionating acid whey into value added streams that could potentially be used in food products. Because of the relatively low protein content of Greek yogurt acid whey, our research focused on possible value added components in the UF permeate of acid whey. Potential end products include lactose, lactic acid, dairy minerals, peptides and oligosaccharides. Our initial focus was on reducing the calcium and lactic acid content of this UF permeate. Approximately 1000 L of acid whey was obtained from a Greek yogurt manufacturer for each trial. A 10,000 daltons UF membrane was used to fractionate the protein. The UF permeate was then processed by one of two different NF membranes. An experimental NF membrane was evaluated for divalent ion permeation and compared to a control NF membrane. Permeates were concentrated to 1× and 2×, and sampled at 1380, 2760, and 4140 kPa processing pressures, and at temperatures 4, 21, 43, and 54°C. Flux also was recorded. Total solids, lactose, galactose, lactic acid and calcium were determined. Higher temperatures and higher pressures yielded higher rates of component permeation for each membrane. The experimental NF membrane had higher permeation on all analyzed components compared to control NF membrane. Lactose and calcium were permeated on the experimental NF membrane, in contrast to the control NF membrane. By utilizing various membranes with very different permeation properties, fractionated products could potentially be achieved. We are exploring other membranes to evaluate the potential creation of purified value added fractions from acid whey.

Key Words: acid whey, membrane processing, nanofiltration

0261 Polymerization of lactose to polylactose by twin-screw extrusion. T. C. Schoenfuss*, C. E. Tyl, and E. M. Reid, *University of Minnesota, St. Paul.*

Our objective is to create a value-added product from lactose. We successfully polymerized lactose into polylactose, a mixture of oligosaccharides, via extrusion. Previously, we evaluated one extruder feed rate and two citric acid catalyst concentrations. We hypothesized that a lower residence time in the extruder would achieve similar fiber contents while reducing caramelization side-reactions. The objectives of this project

were to further evaluate acid catalyst concentrations and extruder conditions on fiber yield. We hypothesized that concentrations above 2% citric acid would yield more fiber. We tested the hypotheses by extruding lactose with 2, 4, and 6% citric acid and 20% glucose. Extruder feed rate was at 15 and 30kg/hour for all formulas. Product was extruded on a Buhler twin-screw 44 mm extruder and the screw configuration, rpms and temperature profile were kept constant between runs. Process (temperature, motor torque and specific mechanical energy (SME)) and product responses (color, dietary fiber, degree of polymerization (DP), and residual lactose) were measured in response to the formula and process changes. Color and brown pigments were determined by HunterLab (10° observer and D65 illuminant) and spectrophotometry (420 nm) of an aqueous extrudate solution. Liquid chromatography (LC)-evaporative light scattering detector analysis was used to quantify dietary fiber by the AOAC integrated dietary fiber method (2009.01). DP was determined by LC-Mass Spectrometry with positive electrospray ionization. Residual lactose was quantified enzymatically using a commercial test kit. The higher feed rate resulted in lower SME and higher motor torque, indicating the material had higher viscosity. The higher feed rate resulted in less brown pigmentation, lower b-values, higher *L*-values and more residual lactose and glucose. These results indicate that at the lower feed rate more lactose and glucose were converted to caramelization products. Citric acid concentration effects were more pronounced at the lower feed rate with higher concentrations leading to more browning and higher *b*-values. Residual lactose decreased with higher citric acid concentrations. Dietary fiber profiles were similar between formulas with DPs of generated oligosaccharides ranging from 3–5. In contrast to the higher feed rate, the HPLC profiles at the lower feed rate of 6% citric acid formulas had lower oligomer peak areas when compared to 2% citric acid. This was not the case for the high feed rate. Overall, the higher feed rate resulted in more favorable processing parameters, less caramelization, and maintained dietary fiber concentrations.

Key Words: extrusion, lactose, oligosaccharides, dietary fiber

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0262 A proficiency test system to improve laboratory and method performance and produce reference values for component calibration samples for infrared milk analysis.

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Our objectives were: 1) utilize data from 10 to 12 laboratories running duplicate chemical analyses of 14 milk samples with an orthogonal matrix of fat, protein, and lactose concentration to calculate all-lab mean reference values for fat, protein, lactose, and total solids for each sample, 2) monitor the analytical performance of the reference chemical analysis methods, and 3) evaluate and improve the analytical performance of individual laboratories conducting chemical reference methods. The chemical reference methods used were Mojonnier ether extraction for fat, Kjeldahl for true protein, spectrophotometric enzymatic assay for anhydrous lactose, and forced air oven drying for total solids. Statistical outliers were removed and all-lab mean reference values and within and between laboratory variation (i.e., Sr and SR) were calculated for each sample for each component. The set of 14 milk samples with all lab mean reference values was used to run diagnostic performance evaluation and calibrate infrared milk analyzers. The proficiency of each lab was evaluated utilizing Z-scores, Pareto diagrams, and Euclidian distance (ED) plots. Performance of the ether extraction, Kjeldahl true protein, and oven drying total solids methods were improved ($P < 0.05$) due to the improved ability to identify and trouble shoot between laboratory differences in results when using the modified milks with the orthogonal design of variation in composition. Residual plots of an individual lab's results minus the all-lab mean for each sample provides an evaluation of individual lab bias and proportional deviations from the all-lab mean over a wide range of concentration of each component. Residual and ED plots allow an experienced evaluator to identify the sources of poor performance by an individual lab and recommend corrective measures. Over a period of years, the feedback and method performance trouble shooting based on residual and ED plots has improved within (Sr) and between (SR) laboratory and method performance.

Key Words: proficiency testing, infrared milk analysis, method performance

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0263 A relatively rapid method for the estimation of the amount of exopolysaccharide produced by lactic acid bacteria during milk fermentation.

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The available methods for estimation of exopolysaccharide (EPS) produced in fermented milks are very lengthy (several days). A relatively shorter ($\leq$ 1 d) method has been investigated for the estimation of EPS produced during fermentation of non-fat milk at 40°C by two strains of *Streptococcus thermophilus* (St–143 and St–10255y). Milk samples were analyzed for EPS concentration every 30 min over a fermentation period of 300 min (final pH 4.6). Samples with pH > 5 were adjusted to pH ~5 before protein was removed by heat and acid precipitation. Excess ethanol was added to the neutralized supernatant and precipitated at -20°C for 3 h. The pellet was dissolved in water at 55°C and centrifuged before residual lactose in the supernatant was removed by repeated precipitation by ethanol. The EPS concentration in the final pellet was estimated by the phenol sulfuric acid method. In milk fermented by *S. thermophilus* St–143, the EPS content significantly increased

($P < 0.05$) from 24 to 68 mg/L during the fermentation period from 150 to 300 min. EPS concentration in samples fermented with *S. thermophilus* St–10255y also significantly varied ($P < 0.05$) from 16 to 52 mg/L during a similar fermentation period. Interestingly, both of the strains appeared to start producing significant amounts of EPS after 150 min of fermentation time, which corresponded to pH ~5.2 and was close to when milk gelation occurred. Thereafter, the EPS concentration continued to increase up to pH ~4.6 (end of fermentation). The total amounts of EPS obtained were comparable to the previously reported results in milks fermented by similar bacterial strains. To explore the recovery of EPS by this method, we added different concentrations of dextran (mol wt: 2×10^6 Da) to milk and found that up to ~70% of the added dextran could be recovered, suggesting that this method was reasonably effective in extracting most of the EPS produced in fermented milk.

Key Words: exopolysaccharides, yogurt, fermented milk

0264 Raw milk quality in the dairy industry: Compositional changes correlated with somatic cell counts. C. R. T. Júnior[1], G. C. Ribeiro[2], R. M. Longo[2], M. C. P. P. Oliveira[2], L. M. Fonseca[*3], M. O. Leite[2], and M. P. Cerqueira[2], *[1]Ministry of Agriculture, Poços de Caldas, Brazil, [2]Universidade Federal de Minas Gerais (School of Veterinary Medicine), Belo Horizonte, Brazil, [3]University of Wisconsin, Madison*.

The aim of this study was to evaluate the seasonal correlation of somatic cell count (SCC) and composition of raw milk. A total of 287,000 samples of raw milk were analyzed during twelve consecutive months by the UFMG Milk Quality Laboratory (UFMG/Veterinary School; LabUFMG) for composition and SCC. The data were analyzed using descriptive statistics, according to the months of production and SCC ranges: Range 1: SCC $\leq$ 200,000 cells/mL; 2: SCC from 201,000 to 400,000 cells/ml; 3: SCC from 401,000 to 750,000 cells/ml, and 4: SCC > 750,000 cells/mL. The least significant difference between treatments was evaluated using Tukey's test, with significance level of 5% in a randomized complete block design. SCC indexes (SCC Log/mL) were higher from January to April and from October to December, with peaks of SCC in February and March (rainy season). The lowest results for SCC ($p < 0.05$) were observed from May to September (dry season), with lower average results during the month of May. The highest fat, protein and total solids contents, and the lowest SCC were found during the dry season ($p < 0.05$), while the lowest concentrations were observed during the rainy season (spring and summer). The increase in SCC was correlated with reduction in lactose, solids, nonfat and protein concentrations ($p < 0.05$), except for SCC range higher than 750,000 cells/mL. In this range, protein content was higher if compared to the levels found in SCC range of 401,000 to 750,000 cells/mL ($p < 0.05$), but similar to the levels found in milk with SCC range from 201,000 to 400,000 ($p > 0.05$). This fact may be related to a reduction in milk secretion, and passage of serum proteins into the milk due to a more pronounced inflammatory process. Fat contents were higher ($p < 0.05$) for elevated SCC, which may be linked to lower milk secretion because of mastitis.

Key Words: milk quality, composition, somatic cells

0265 The effect of immunoglobulins and somatic cells on the gravity separation of fat, bacteria, and spores in pasteurized whole milk. D. M. Barbano[*1,2] and S. R. Geer[1,2], *[1]Northeast Dairy Foods Research Center, Ithaca, NY, [2]Cornell University, Ithaca, NY.*

Our objective was to determine the role that Ig and somatic cells (SC) play in the gravity separation of milk. There were 9 treatments: (1) low temperature pasteurized (LTP) (72°C for 17.31s) whole milk, (2) LTP (72°C for 17.31s) whole milk with added bacteria and spores, (3) recombined LTP (72°C for 17.31s) whole milk with added bacteria and spores, (4) high temperature pasteurized (HTP) (76°C for 7 min) whole milk with added bacteria and spores, (5) HTP (76°C for 7 min) whole milk with added bacteria and spores and added colostrum, (6) HTP (76°C for 7 min) centrifugal-separated gravity-separated (CS GS) skim milk with HTP (76°C for 7 min) low SC cream with added bacteria and spores, (7) HTP (76°C for 7 min) CS GS skim milk with HTP (76°C for 7 min) high SC cream with added bacteria and spores, (8) HTP (76°C for 7 min) CS GS skim milk with HTP (76°C for 7 min) low SC cream with added bacteria and spores and added colostrum, and (9) HTP (76°C for 7 min) CS GS skim milk with HTP (76°C for 7 min) high SC cream with added bacteria and spores and added colostrum. The milks in 9 treatments were gravity separated at 4°C for 23 h. Five fractions were collected by weight from each of the columns treatments starting from the bottom of the column: 0 to 5%, 5 to 90%, 90 to 96%, 96 to 98%, 98 to 100%. The SC, fat, bacteria, and spores were measured in each of the fractions. The experiment was replicated in 3 different weeks using a different batch of milk and colostrum. Portions of the same batch of the frozen bacteria and spore solutions were used for all 3 replicates. The presence of both SC and Ig were necessary for normal gravity separation (i.e., rising to the top) of fat, bacteria, and spores in whole milk. The presence of Ig without somatic cells was not sufficient to cause bacteria, fat and spores to rise to the top without SC. The interaction between SC and Ig was necessary to cause aggregates of fat, SC, bacteria, and spores to rise during gravity separation. The SC may provide the buoyancy required for the aggregates to rise to the top due to gas within the SC. More research is needed to understand the mechanism of the gravity separation process.

Key Words: immunoglobulin, somatic cells, gravity separation