533 Effect of microfiltration concentration factor on serum protein removal from skim milk using polymeric spiral-wound membranes. S. L. Beckman* and D. M. Barbano, *Cornell University, Ithaca, NY.*

The objective of this research was to determine the effect of concentration factor (CF) on the removal of serum protein (SP) from skim milk during 50°C microfiltration (MF) using a 0.3-µm polyvinylidene fluoride spiral-wound (SW) membrane. Pasteurized (72°C, 16 s) skim was MF at 3 CF, 1.50, 2.25, and 3.00×, each on a separate day of processing starting with skim milk. Two phases of MF were used at each CF, with an initial startup-stabilization phase (40 min, full recycle) to achieve desired CF, followed by a steady-state phase (90 min, feed-and-bleed) for data collection. The experiment was replicated 3 times, and SP removal from skim was quantified at each CF. System pressures, flow rates, and fluxes were monitored during the 90 min run. Permeate flux increased ($P < 0.05$) (12.8 15.3, and 19.0 kg/m^2 per h) with decreasing CF from 3.00 to 1.50×, while fouled water flux did not differ ($P > 0.05$) among CF indicating that the effect of membrane fouling on hydraulic resistance of the membrane was similar at all CF. However, the CF used during skim SW MF (0.3 µm) did affect the percentage of SP removed. As CF increased, from 1.50 to 3.00×, the percentage of SP removed increased ($P < 0.05$) from 10.6 to 35.6%, in a single stage feed-and-bleed MF system. Rejection of SP during polymeric SW MF of skim was caused by fouling of the membrane, not by the membrane itself and differences in the foulant characteristic among CF influenced SP rejection more than it influenced hydraulic resistance. We hypothesize that differences in the conditions near the surface of the membrane and within the pores during the first few minutes of processing, influenced the rejection of SP protein because more pore size narrowing and plugging occurred at low CF than at high CF due to a slower rate of gel layer formation at low CF. It is possible that SP removal from skim milk at 50°C could be improved by optimizing the membrane pore size, feed composition, and controlling the formation rate of concentration polarization derived gel layer at the membrane surface during the first few minutes of processing.

Key Words: microfiltration, concentration factor, spiral-wound

534 Modification of milk fat fatty acid profile by a combination of microfiltration and dry crystallization. K. E. Kaylegian*[1], J. Choi[1], K. Harvatine[2], J. N. Coupland[1], and R. J. Elias[1], *[1]Dept. of Food Science, Pennsylvania State University, University Park, [2]Dept. of Animal Science, Pennsylvania State University, University Park.*

A reduction in the saturated content of milk fat is desired to improve its healthfulness. Preserving the flavor and premium image of milk fat is paramount when evaluating technologies to change the fatty acid profile. The objective of this study was to combine microfiltration and dry crystallization to produce milk fat fractions with reduced saturated fat contents. Pasteurized milk was microfiltered through a 5 µ ceramic membrane using conditions that were optimized for the separation of large fat globules ($D_{32} > 4.5$ µm) and small fat globules ($D_{32} < 3.7$ µm) with minimal processing time, to protect the fat. The control milk, permeate and retentate streams were separated into cream, churned into butter, and clarified to produce anhydrous milkfat (AMF). Each AMF was fractionated using bench-scale dry crystallization with vacuum filtration and a 2-step process to produce solid (S) and liquid (L) fractions at 25 and 15°C. The experiment was repeated in triplicate. Fatty acid analysis (by GLC) showed no significant differences ($P > 0.05$) in total saturated fat between the control, permeate and retentate streams when comparing similar fractions (i.e., AMF, 25S, 25L, 15S, 15L). A shift in the fatty acid profile was observed when the liquid and solid fraction obtained at the same temperature were compared, and when the 15°C fractions were compared with the 25°C fractions. A decrease in long chain saturated fatty acids and an increase in short chain saturated and long chain unsaturated fatty acids were observed among these fractions. These changes counteracted each other, resulting in a 5% reduction of the total saturated fat content in the 15L fractions compared with the control. The 15L fraction showed a 16% increase in CLA 9–11 compared with the control AMF. The total saturated fat content was approximately 65.4% for the AMFs, 71.7% for 25S, 63.2% for 25L, 68.7% for 15S, and 62.3% for the 15L fractions. In conclusion, the use of dry crystallization produced milk fat fractions with small reductions in saturated fat content, and the addition of microfiltration did not result in additional benefits.

Key Words: milk fat, fatty acid, fractionation

535 Production of milk protein concentrate with a modified mineral content. C. Marella*, P. Salunke, A. Biswas, and L. E. Metzger, *Midwest Dairy Foods Research Center, Dairy Science Department, South Dakota State University, Brookings.*

Milk protein concentrates (MPC) are products obtained from ultrafiltration (UF) of skim milk. It is well established that MPC powders can have a loss of solubility during storage as a result of protein-protein interactions, and formation of insoluble complexes. Previous studies have shown that partial replacement of calcium with sodium improves MPC functionality and prevent the loss in solubility during storage. These studies have utilized pH adjustment with addition of acids, addition of monovalent salts or ion exchange treatment of UF retentate. The objective of this study was to utilize carbon dioxide to produce MPC with improved functionality. Preliminary studies were conducted with injection of CO_2 into skim milk that determined 1500, 1850 and 2200 ppm of dissolved CO_2 before UF for obtaining MPCs with 20, 30 and 40% reduced calcium content, respectively. From these preliminary results, a CO_2 injection level of 2200 ppm was selected for further study. In this study reduced-calcium MPC from 189.25 L of skim milk subjecting to injection of 2200 ppm of CO_2 before UF along with additional injection during UF. A control MPC was also produced from same lot of skim milk without injection of CO_2. The above processes were replicated 3 times, using different lots of skim milk for each replication. The reduced calcium UF retentates (17% TS) had a viscosity of 11 cP at 100/s shear rate, while the control UF retentates had a viscosity of 24 cP, a 2-fold higher in viscosity. Injection of CO_2 and the resultant solubilization of calcium phosphate had a significant ($P < 0.05$) effect on UF performance, resulting in a 10 and a 20% loss in initial and average flux, respectively. Processing of skim milk with injection of CO_2 resulted into higher irreversible fouling resistances (Rfi), a 48% increase in Rfi. As compared with the control, the reduced-calcium powders had 28 and 34% lower ash and calcium contents, respectively. This study demonstrates that MPCs with a modified mineral content can be produced with injection of CO_2 before and during UF of skim milk.

Key Words: MPC, reduced calcium, mineral modified

536 Direct capture membrane adsorption chromatography with crude whey at pilot scale. L. Voswinkel* and U. Kulozik, *Technische Universität München, Freising, Germany.*

This study aimed at the development of a chromatographic process for whey protein isolation from crude whey as unit operation in the dairy industry. There is a demand for purified protein fractions such as lactoferrin (Lf) or lactoperoxidase (LPO) as well as for a specific protein composition in whey; e.g., depletion of β-Lactoglobulin (β-LG) for baby food. During the last decade several attempts and solutions for whey protein isolation have been published, including large scale packed columns and membrane adsorption technology. These processes resulted in fractions with high purity and recovery, but still required pretreatment steps, such as crossflow filtration, to avoid blocking of the stationary phase. Sartorius Stedim Biotech (Göttingen, Germany) has developed a spiral wound membrane device for ion exchange chromatography for the handling of crude feedstocks. Some industrial applications with direct capture-membrane adsorption chromatography (MAC) are proposed in this work. Using unfiltered buffers and cheese whey without adjusting its pH (if it is between pH 6–7) β-LG binds to an anion exchanger (AEC). The bind and elute process takes 30 min and yields 40 g protein with 99.5% purity at 5 L bed volume. This method is useful for β-LG depleted fresh whey keeping it as native as possible. Another unit operation is as simple. Unfiltered whey with no pH-adjustment is loaded onto a cation exchanger. Within 90 min, 1 ton of whey can be processed to gain pure Lf and LPO. The flowthrough from the AEC-step can also be used as substrate. The third application is the isolation of CMP resulting in 92% purity. In this case adjustment of conductivity to 3 mS/cm and pH-value to 4.9 is necessary. The loss of binding capacity in all studies was <10% over 5 cycles. A cleaning procedure with acid and NaOH recovered the initial binding capacity. The experiments showed that the processes are easy to handle and technical equipment does not require high pressure systems and special chemical stability. Therefore, direct capture-MAC is a fast, simple and reliable method convenient as a unit operation, not least for dairy SMEs.

Key Words: direct capture-membrane adsorption chromatography (MAC), whey protein fractionation, crude feedstock

537 Effect of operating conditions on particle size of milk protein concentrates during ultrafiltration. X. Luo, L. Ramchandran, and T. Vasiljevic*, *Advanced Food Systems Research Unit, College of Health and Biomedicine, Victoria University, Melbourne, Victoria, Australia.*

Various processing conditions during concentration of milk are known to affect their physical functionalities. To be useful as functional ingredients, milk protein concentrates (MPC) should possess desirable physical properties. For example emulsifying properties of MPC are believed to be influenced by the particle size. The present work investigated the effect of processing temperature as well as varying pH on particle size and membrane performance during a 5-fold concentration of skim milk by ultrafiltration (UF) using polyethersulfone membrane (molecular cut-off 20 kDa) with the aim to establish foundation for optimizing a process regimen for production of MPC with targeted functionalities. The skim milk was concentrated by ultrafiltration operated first at 3 temperatures (15, 30 or 50°C) and then at a constant temperature of 15°C but with varying pH (6.3, 5.9 or 5.5). Particle size and zeta potential of skim milk, retentate and permeate samples were measured immediately after the collection of samples using a Malvern Zetasizer while calcium content was determined by AAS. Membrane performance was evaluated by measuring the permeate flux (every 30 min) and SEM examination of membrane surface and its cross section. The operation temperature of UF influenced the calcium removal and change in casein micelle size. More ($P < 0.05$) calcium was removed from skim milk into the permeate at lower temperature (15°C) of UF, resulting in smaller casein micelle size. However, pH did not ($P > 0.05$) apparently influence particle size; although calcium removal was significantly higher ($P < 0.05$) at pH 5.5. The absolute value of zeta potential decreased with increase in UF operation temperature and decreasing pH. Membrane fouling occurred more rapidly at 50°C than at 15 or 30°C. At 15°C, fouling occurred more rapidly at pH 5.5. UF of skim milk at 15°C and pH 6.3–5.9 favored the production of milk concentrate with smaller casein micelles and less membrane fouling and thus can be preferred during MPC production.

Key Words: milk protein concentrate, ultrafiltration, particle size