

## Dairy Foods: Filtration and Drying

**201 Impact of annatto color and bleaching of whey and micro-filtration permeate on ultrafiltration processing characteristics during production of 80% protein concentrates.** M. Adams<sup>1</sup>, J. Zulewska<sup>\*2</sup>, and D. M. Barbano<sup>1</sup>, <sup>1</sup>*Cornell University, Ithaca, NY*, <sup>2</sup>*University of Warmia and Mazury, Olsztyn, Poland*.

Our objective was to determine if annatto color or bleaching had any influence on ultrafiltration (UF) flux during production of 80% whey protein concentrate (WPC) or 80% serum protein concentrate (SPC). Cheddar cheese whey (18 vats using 900 kg of whole milk each) and microfiltration (MF) permeate of skim milk (18 processing runs using 1000 kg of skim milk each) were produced. The 18 runs were divided into 3 replicates with 6 different treatments within each replicate. The 6 treatments within either the whey or MF permeate replicates were: 1) no annatto (NA), 2) NA + benzoyl peroxide (BPO), 3) NA + H<sub>2</sub>O<sub>2</sub>, 4) annatto (A), 5) A + BPO, and 6) A + H<sub>2</sub>O<sub>2</sub>. Approximately 700 kg of separated, treated whey or treated MF permeate was heated to 50°C and processed with the UF system in batch recirculation mode using a polyethersulfone spiral wound UF membrane (Model 3838, GEA NIRO Inc., Hudson, WI) with a nominal pore size of 10,000 Da. Addition of annatto color had little or no effect ( $P > 0.05$ ) on UF flux. Bleaching separated Cheddar cheese whey or MF permeate with or without added color improved ( $P < 0.05$ ) UF flux during processing to produce 80% protein concentrates. Generally, H<sub>2</sub>O<sub>2</sub> produced higher fluxes than BPO treatments ( $P < 0.05$ ) and BPO increased SPC production fluxes to a lesser extent ( $P < 0.05$ ) than it did WPC production fluxes. Relative to the flux of unbleached whey (about 15 L/m<sup>2</sup>h), the bleached whey had a flux of about 20 to 23 L/m<sup>2</sup>h. Water flux before processing, after processing, and after final cleaning were measured at 50°C. The water flux after cleaning restored flux to the level before processing whey or permeate. The flux after processing (i.e., fouled water flux) demonstrated differences in fouling and loss of water permeability that were consistent with differences resulting from the bleaching treatments. Little, if any, effect of the annatto on fouled water flux was observed.

**Key words:** whey bleaching, ultrafiltration flux, annatto

**202 Functional properties of milk serum protein concentrates with varying levels of  $\beta$ -casein.** L. Coppola<sup>\*1</sup>, S. Rankin<sup>1</sup>, M. Molitor<sup>2</sup>, and J. Lucey<sup>1</sup>, <sup>1</sup>*University of Wisconsin-Madison, Madison*, <sup>2</sup>*Wisconsin Center for Dairy Research, Madison*.

Microfiltration (MF) can isolate serum proteins (MSP) from milk. The dissociation of  $\beta$ -casein from casein micelles at <8°C in combination with MF is a means to create MSP concentrates (MSPC) with varying levels of  $\beta$ -casein. MF permeates were produced at ~23°C (MSPC1) and ~5°C (MSPC2) using polymeric, cross-flow MF, concentrated and spray dried (~85% protein). Composition, browning, volatile profiles, and sensory characters of MSPC before and after accelerated storage (50°C for 28d) were compared with whey protein concentrate (WPC) and WPC samples. Significant effects were declared at  $P \leq 0.05$ . MSPC1 and MSPC2 contained different levels of  $\beta$ -casein (1.0 vs 20% of the total solids composition, respectively). WPC samples contained more fat than both MSPC. Browning was determined by colorimetry (CIELAB) of powders and optical density of rehydrated samples. Pre-storage L\* values were not different between samples, but a\* and b\* values of MSPC were lower than WPC. After storage, MSPC had higher L\* and lower a\* values than WPC. Rehydrated MSPC exhibited smaller changes in browning during storage than WPC. SPME/

GC-MS of rehydrated samples showed fewer types of volatiles were present pre-storage and that fewer types increased in intensity following storage for MSPC samples compared with WPC. Descriptive sensory analysis indicated that turbidity, odor, and astringency were not affected by storage and were higher in WPC. MSPC2 had lower sweetness than other samples pre-storage but not post-storage. Pre-storage, WPC samples had higher levels of cardboard and residual flavors. With storage, MSPC1 had a trend toward increased milkfat flavor and MSPC2 had an increase in cucumber flavor while WPC increased in salty, acid, sweet, milkfat, cooked, cabbage, and cucumber flavors. Principal component analysis (PCA) indicated marked differences between MSPC and WPC samples both pre-and post-storage, with WPC described by more off-flavor descriptors. MSPC differed in browning, volatiles, composition, and sensory properties compared with WPC samples, likely to make them attractive in food applications where fewer off-flavors and increased storage stability are desired.

**Key words:** milk serum protein,  $\beta$ -casein, whey

**203 Impact of microfiltration temperature on the composition and functionality of casein concentrates.** J. R. Koch<sup>\*1</sup>, J. A. Lucey<sup>1</sup>, K. J. Burrington<sup>2</sup>, and M. Molitor<sup>2</sup>, <sup>1</sup>*University of Wisconsin, Madison*, <sup>2</sup>*Wisconsin Center for Dairy Research, Madison*.

Microfiltration (MF) is used to separate serum proteins from caseins. There is a trend of performing membrane filtration at cold temperatures to reduce microbial growth. Our objective was to investigate if performing MF at cold or warm temperatures impacted the protein composition and functionality of casein concentrates. Five samples were produced using polymeric spiral wound MF (~0.08  $\mu$ m) - 2 samples of warm (24°C) temperature MF (WMF) and 3 of cold (5°C) temperature MF (CMF). At cold temperatures  $\beta$ -casein dissociates from casein micelles, which could result in more  $\beta$ -casein in the serum phase. Retentates were concentrated to attain powders with ~80% protein. A milk protein concentrate (MPC) was also produced using ultrafiltration. Reverse-phase HPLC was used to determine the protein composition. The casein to whey ratio was 17:1 and 11.5:1 in the WMF and CMF concentrates, respectively. The  $\beta$ -casein to  $\alpha$ s-casein ratio was 4:5 and 5:7 in the WMF and CMF concentrates, respectively. A low concentration of  $\kappa$ -casein was found in all MF permeates likely due to heat-induced complexation of  $\kappa$ -casein with  $\beta$ -lactoglobulin. Solubility of casein concentrates increased with an increase in the temperature of hydration and with the time of hydration. Foams were made from 5% protein solutions, and physical properties (over-run and stability) and rheological properties (vane geometry) were determined. There was no significant difference ( $P < 0.05$ ) in over-run for all samples. Both WMF and CMF concentrates had higher foam stability compared with foams made with MPC. Samples with a higher proportion of  $\beta$ -casein had greater foam stiffness. The foam made from WMF concentrate had significantly higher foam stability and foam stiffness compared with CMF concentrate. Producing casein concentrates at different separation temperatures alters the protein profile. Both separation temperatures reduced the whey (serum) protein content in casein concentrates, but at cold temperature the  $\beta$ -casein content was also reduced. Casein concentrates separated at various temperatures had different functional properties, which could be useful for specific food applications.

**Key words:** casein concentrate, microfiltration

**204 Spiral wound microfiltration process for production of micellar casein concentrate.** C. Marella\*, P. Salunke, and L. E. Metzger, *Midwest Dairy Foods Research Center, South Dakota State University, Brookings*

Micellar casein concentrate (MCC) is the retentate obtained from microfiltration (MF) of skim milk. Previous research has indicated that MF of skim milk with ceramic membranes result in improved membrane performance relative to spiral wound membranes. However, spiral wound polymeric units have an advantage in terms of capital and operating costs relative to ceramic units. Moreover spiral wound polymeric units are currently extensively used in the US dairy industry. The objective of this study was to evaluate impact of operating pressure and level of diafiltration on membrane performance during MF of skim milk using spiral wound polymeric membranes. Preliminary lab scale experiments were conducted using three levels of transmembrane pressure (TMP) and four levels of diafiltration (DF). Skim milk was microfiltered at 23.3 °C to a volume reduction of 4, using 0.5 µ Polyvinylidene fluoride membrane in a flat sheet configuration. During the experiments, process flux was maintained within 80% of the initial flux by addition of DF water at 6 intervals so as to control the viscosity of the retentate. The effect of TMP and effectiveness of DF were assessed by measuring overall flux, serum protein removal (SP removal), casein to total protein ratio (CN/TKN), casein to true protein ratio (CN/TP), and rejection of casein and serum protein. CN/TKN ratio of MCCs ranged from 0.87 to 0.96 while CN/TP ratio ranged from 0.89 – 0.96. The rejection of casein ranged from 0.97 to 1.0 while the rejection of SP ranged from 0.1 to 0.53. SP removal ranged from 35 – 81.45%. The highest ratio of CN/TKN, CN/TP, SP removal and the lowest rejection of SP were obtained with the use of 35.4 kPa TMP and 150% DF. These are the lowest TMP and highest DF used in the experiments. SP removal data were fitted into a model in which SP removal was expressed as a function of DF and square of TMP. The model predicted SP removal within 90-95% of actual SP removal obtained during pilot trials using various TMP and DF levels. With appropriate selection of TMP and DF level, the efficiency of polymeric MF membranes can be optimized for production of MCC.

**Key words:** micellar casein concentrate, spiral wound MF, serum protein

**205 Characterization of  $\alpha$ -lactalbumin and  $\beta$ -lactoglobulin powders obtained from serum whey.** C. Marella\*, P. Salunke, L. E. Metzger, and K. Muthukumarappan, *Midwest Dairy Foods Research Center, South Dakota State University, Brookings*.

The composition and functional properties of conventional whey protein products can vary widely due to variability in the cheese manufacturing processes. However, variations in the composition and functionality of whey protein products obtained from microfiltration (MF) of skim milk should be minimal since whey proteins are harvested before cheese manufacture. The objective of the present study was to evaluate the functional properties of  $\alpha$ -Lactalbumin ( $\alpha$ -La) and  $\beta$ -Lactoglobulin ( $\beta$ -Lg) enriched powders, serum protein concentrate (SPC) and a conventional WPI. The  $\alpha$ -La,  $\beta$ -Lg and SPC were obtained from MF of skim milk. All the powders were produced at SDSU dairy plant using a combination MF, ultrafiltration, reverse osmosis and spray drying. Functionality testing was conducted in triplicate. Solubility (1% protein solutions); thermal aggregation (heating 1% protein solutions at pH 3 and 7 at 80 °C for 5 min, with out and with added CaCl<sub>2</sub>, gel strength and syneresis (10% protein solutions heated at 80 °C for 30 min, with and without CaCl<sub>2</sub>, NaCl); and foam-

ing properties (5% protein solutions) were studied. All the powders were fully soluble at pH 3.0 and 7.0. Heating at pH 3 didn't cause any loss in solubility, whereas none of the solutions at pH 7 could withstand heating in the presence of added CaCl<sub>2</sub>. The  $\alpha$ -La,  $\beta$ -Lg and SPC powders exhibited much higher gel strength (3.5 to 7 fold higher) and lower syneresis when compared with WPI. Among these powders,  $\alpha$ -La powder had the lowest syneresis and  $\beta$ -Lg powder has the highest gel strength and moderate syneresis in the presence of added CaCl<sub>2</sub>. Foam over run (FOR) and foam stability of the  $\alpha$ -La,  $\beta$ -Lg and SPC powders were higher than those of conventional WPI. The  $\alpha$ -La powder has the highest foam over run of 1070%, which was 43% higher than WPI and about 30% higher than the  $\beta$ -Lg and SPC. The results of this study demonstrate that  $\alpha$ -La and  $\beta$ -Lg powders may have value in applications that require high gel strength, low syneresis and high foaming properties.

**Key words:**  $\alpha$ -lactalbumin,  $\beta$ -lactoglobulin, functional properties

**206 Effects of washing/diafiltration on milk protein concentrate (MPC) functionality.** J. Du\* and J. A. Lucey, *University of Wisconsin-Madison, Madison*.

The production of milk protein concentrate (MPC) has grown in recent years and MPC is produced by removing some of the lactose from skim milk (by ultrafiltration, UF) before drying. However, high protein MPC powders often have low solubility and they can exhibit a decrease in solubility during storage, especially at high storage temperatures. In high protein MPC products, washing or diafiltration (DF) is required to further reduce the lactose content. We were interested in understanding if extensive washing reduces the concentration of casein-bound calcium as this could be involved in the impaired functionality of MPC. The objectives of this study were to understand the changes that occur in soluble and bound calcium during UF/DF and the effects of the calcium status on the solubility of MPC products. Four MPC samples were produced from skim milk that was subjected to between one to 4 cycles of UF/DF. We wanted to keep the lactose content constant in all the powders, as that also impacts solubility, so water with 5.4% lactose was used as the washing solution during DF instead of water. Milks were concentrated to 1.67 fold with UF membranes (10k Da) and then back diluted (washed) to the original protein solids level with lactose solution. The retentate was then freeze-dried and the calcium equilibrium and structure of casein micelles were determined. The soluble calcium levels in these retentates were significantly impacted by the protein level used for rehydration ( $P < 0.05$ ); the lower the protein level used for rehydration, the higher the measured soluble calcium content. The ratio of bound calcium to casein decreased with the number of DF cycles but after 3 DF cycles the ratio became nearly constant. Scanning electron microscopy indicated that the casein micelles became much smaller in samples with 3 DF cycles compared with the less washed samples. Casein micelle size was determined by Malvern Zetasizer and the average size of the casein micelles was reduced with increased number of (DF) washing. Further research is investigating the impact of washing cycles on powder solubility.

**Key words:** milk protein concentrate, solubility, calcium

**207 Effect of adding NaCl or KCl during manufacture of MPC80 on its physico-chemical properties.** V. Sikand\*<sup>1</sup>, P. S. Tong<sup>1</sup>, S. Vink<sup>1</sup>, and J. Walker<sup>2</sup>, <sup>1</sup>*Dairy Products Technology Center, Cal Poly State University, San Luis Obispo*, <sup>2</sup>*Dept. of Statistics, Cal Poly State University, San Luis Obispo*.

Milk protein concentrate (MPC) powder with equal to or greater than 80% protein concentration has been shown to have poor solubility. The poor solubility of MPC limits its potential usage in the food industry. In a previous study in our lab, we have shown that the addition of 50–150mM NaCl into diafiltration water can improve the solubility of MPC80. However, adding NaCl into diafiltration water may impact other functional properties. The objective of this study was to determine the impact of addition of 150mM KCl and NaCl during MPC manufacture on solubility and other functional properties (foaming stability, turbidity and heat stability) on the resulting MPC powders. The powder samples were reconstituted at room temperature to contain 5% total solids. Percent solubility was tested after 3 h of mixing and was calculated as total solids in the supernatant to total solids in the original solution. Percent foaming stability, expressed as collapse of foam, was tested after 5 min to initial mass of foam. Turbidity was measured in Nephelos Turbidity Units (NTU) using a calibrated nephelometer. Heat stability was determined by measuring the heat coagulation time of 2 mL samples immersed in a oil bath set at 140°C. Our results indicate that higher solubility ( $P < 0.001$ ) was found in NaCl (100%) and KCl-treated MPC80 (98.8%) than in control MPC80 (90%). Foaming stability ( $P < 0.001$ ) was found to be highest in KCl-treated MPC (21.8%) followed by NaCl-treated MPC (10.6%) and then followed by control MPC (4.6%). Higher heat stability ( $P < 0.001$ ) was observed in control MPC80 (23.7 min) when compared with NaCl (16.5 min) and KCl-treated MPC80 (16.4 min). Lower turbidity ( $P < 0.001$ ) was observed in NaCl (129 NTU) and KCl-treated MPC (117 NTU) as compared with control MPC (564.2 NTU). Our results indicate that the functional properties of MPC80 powders are influenced by addition of KCl or NaCl in the manufacture of MPC. These results suggest that the functional properties of MPC80 powders can be modified by changing its mineral composition during its manufacture.

**Key words:** MPC80, functional properties

**208 Determination of the drying behavior of dairy products to improve the process, energy costs and the quality of the dairy powders.** P. Schuck<sup>1,2</sup>, A. Dolivet<sup>1,2</sup>, S. Mejean<sup>1,2</sup>, P. Zhu<sup>\*1,3</sup>, E. Blanchard<sup>3</sup>, and R. Jeantet<sup>2,1</sup>, <sup>1</sup>INRA, UMR1253, Rennes, France, <sup>2</sup>Agrocampus Rennes, UMR1253, Rennes, France, <sup>3</sup>Laiterie de Montaigu, Montaigu, France.

The most frequently used technique for dehydration of dairy products is spray drying. This is an effective method for preserving biological products as it does not involve severe heat treatment and it allows storage of powders at an ambient temperature. Due to the variety and complexity of the concentrates to be dried, a more rigorous understanding of spray-drying based on physico-chemical and thermodynamic properties have now become necessary. At the same time, the current state of the art and knowledge do not allow determination of the parameters of spray-drying of dairy products. The only way to determine these parameters is to perform several complex and expensive experiments with a pilot scale spray-dryer. The aims of this study were to propose a new indirect method that determines the ratio of bound to unbound water. The results, combined with thermodynamic and physico-chemical parameters (such as absolute and relative humidity of air, total solids and temperature of concentrate, air flow rate, etc.), provide more precise determination of certain spray-drying parameters such as inlet air temperature and mass flow rate. We performed more than 50 experiments to correlate calculated and measured parameters in a pilot plant dryer (Bionov, Rennes, France) using water, skim milk, caseinate, crystallized whey and maltodextrin. The results show that the difference between the calculated and measured inlet air temperature was below 5%, the determination coefficient being close to 0.96. The economic interest of this system is obvious, because it is easy to anticipate the spray-drying parameters by using a controller integrating the water availability of the concentrate and certain thermodynamic parameters. Software based on this step was developed (SD2P®, Spray Drying Parameters Simulation & Determination) and registered.

**Key words:** powder, prediction, software