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## **PREPARATION OF MILK PROTEIN CONCENTRATES BY ULTRAFILTRATION AND CONTINUOUS DIAFILTRATION: EFFECT OF PROCESS DESIGN ON OVERALL EFFICIENCY**

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The objective was to characterize the performance of the UF-DF process by evaluating permeate flux decline, fouling resistance, energy and water consumption, and retentate composition as a function of MWCO (10 and 50 kDa) and UF-DF sequence [3.5×–2 diavolumes (DV) and 5×–0.8DV]. The UF-DF experiments were performed on pasteurized skim milk using a pilot-scale filtration system operated at 50°C under a constant transmembrane pressure of 465 kPa. The results showed that MWCO had no effect on permeate flux for the same UF-DF sequence. Irreversible resistance was also similar for both sequences, whatever the MWCO, suggesting that soluble protein deposition within the pores is similar for all conditions. Despite lower permeate fluxes and greater reversible resistance for the 5×–0.8DV sequence, the overall energy consumption of the 2 UF-DF sequences was similar. However, the 3.5×–2DV sequence required more water for DF and generated larger volumes of permeate to be processed, which will require more membrane area and lead to greater environmental impact. A comparative life cycle assessment should however be performed to confirm which sequence has the lowest environmental impact.

## **INTERACTIONS BETWEEN WHEY PROTEIN OR POLYMERIZED WHEY PROTEIN AND SOYBEAN LECITHIN IN MODEL SYSTEM**

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The objective was to investigate the interactions between soybean lecithin (SL, 0–3%, wt/vol) and whey protein (WP, 10%, wt/vol) or polymerized whey protein (PWP, 10%, wt/vol) induced by heating WP solutions at 85°C for 0 to 20 min at pH 7.0. The samples were evaluated for zeta potential, particle size, morphology, rheological properties, thermal properties, secondary structure, and surface hydrophobicity. Zeta potential of WP increased linearly as SL level increased from 0 to 3%, whereas that of PWP changed with plateau at SL level of 1%, which may be due to the aggregation of SL. The addition of SL increased the particle size and apparent viscosity of both WP and PWP. All the samples exhibited different morphology depending on SL level and heating time according to transmission electron microscopy images. Whey protein showed obviously decreased gelation time and increased storage modulus in the presence of SL. Differential scanning calorimetry curves confirmed the effects of SL on the thermal

properties of both WP and PWP. Circular dichroism spectra indicated that SL had effects on the secondary structure of both WP and PWP. The changes in surface hydrophobicity indicated the hydrophobic interactions between WP/PWP and SL. Data indicate that the physicochemical and functional properties of WP and PWP can be altered by adding soybean lecithin.

### **INFLUENCE OF MILK PROTEIN CONCENTRATES WITH MODIFIED CALCIUM CONTENT ON ENTERAL DAIRY BEVERAGE FORMULATIONS: PHYSICO-CHEMICAL PROPERTIES**

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Because of their high protein and low lactose content, milk protein concentrates (MPC) are typically used in the formulation of ready-to-drink beverages. Calcium-mediated aggregation of proteins during storage is one of the main reasons for loss of storage stability of these beverages. Control and calcium-reduced MPC [20% calcium-reduced (MPC-20) and 30% calcium-reduced (MPC-30)] were used to evaluate the physicochemical properties in this study. This study was conducted in 2 phases. In phase I, 8% protein solutions were prepared by reconstituting the 3 MPC and adjusting the pH to 7. These solutions were divided into 3 equal parts, 0, 0.15, or 0.25% sodium hexametaphosphate (SHMP) was added, and the solutions were homogenized. In phase II, enteral dairy beverage formulations containing MPC and a mixture of gums, maltodextrin, and sugar were evaluated following the same procedure used in phase I. In both phases, heat stability, apparent viscosity, and particle size were compared before and after heat treatment at 140°C for 15 s. In the absence of SHMP, MPC-20 and MPC-30 exhibited the highest heat coagulation time at 30.9 and 32.8 min, respectively, compared with the control (20.9 min). In phase II, without any addition of SHMP, MPC-20 exhibited the highest heat coagulation time of 9.3 min compared with 7.1 min for control and 6.2 min for MPC-30. An increase in apparent viscosity and a decrease in particle size of reconstituted MPC solutions in phases I and II with an increase in SHMP concentration was attributed to casein micelle dissociation caused by calcium chelation. This study highlights the potential for application of calcium-reduced MPC in dairy-based ready-to-drink and enteral nutrition beverage formulations to improve their heat stability.

### **EFFECT OF PARTIAL WHEY PROTEIN DEPLETION DURING MEMBRANE FILTRATION ON THERMAL STABILITY OF MILK CONCENTRATES**

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The objective was to better understand the effect of partial removal of whey proteins by microfiltration (MF) on the heat stability of the fresh concentrates. The micellar casein concentrates were compared with control concentrates obtained using ultrafiltration (UF). Pasteurized milk was microfiltered (80 kDa polysulfone membrane) or ultrafiltered (30 kDa cellulose membrane) without diafiltration (i.e., no addition of water) to 2× and 4× concentration, based on volume reduction. The final concentrates showed no differences in pH, casein micelle size, or mineral concentration in the serum phase. The micellar casein retentates (obtained by MF) showed

a 20 and 40% decrease in whey protein concentration compared with the corresponding UF milk protein concentrates for 2× and 4× concentration, respectively. The heat coagulation time decreased with increasing protein concentration, regardless of the treatment; however, MF retentates showed a higher thermal stability than the corresponding UF controls. The average diameter for casein micelles increased after heating in UF but not MF concentrates. The turbidity (measured by light scattering) increased after heating, but to a higher extent for UF retentates than for MF retentates at the same protein concentration. It was concluded that the reduced amount of whey protein in the MF retentates caused a significant increase in the heat stability compared with the corresponding UF retentates. This difference was not due to ionic composition differences or pH, but to the type and amount of complexes formed in the serum phase.

### **INVITED REVIEW: USE OF INFRARED TECHNOLOGIES FOR THE ASSESSMENT OF DAIRY PRODUCTS—APPLICATIONS AND PERSPECTIVES**

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Dairy products are important sources of nutrients for human health and in recent years their consumption has increased worldwide. Therefore, the food industry is interested in applying analytical technologies that are more rapid and cost-effective than traditional laboratory analyses. Infrared spectroscopy accomplishes both criteria, making real-time determination feasible. However, it is crucial to ensure that prediction models are accurate before their implementation in the dairy industry. In the last 5 yr, several papers have investigated the feasibility of mid- and near-infrared spectroscopy to determine chemical composition and authenticity of dairy products. Most studies have dealt with cheese, and few with yogurt, butter, and milk powder. Also, the use of near-infrared (in reflectance or transmittance mode) has been more prevalent than mid-infrared spectroscopy. This review summarizes recent studies on infrared spectroscopy in dairy products focusing on difficult to determine chemical components such as fatty acids, minerals, and volatile compounds, as well as sensory attributes and ripening time. Promising equations have been developed despite the low concentration or the absence of specific absorption bands (or both) for these compounds.

### **SHORT COMMUNICATION: MULTI-COMPONENT INTERACTIONS CAUSING SOLIDIFICATION DURING INDUSTRIAL-SCALE MANUFACTURE OF PRE-CRYSTALLIZED ACID WHEY POWDERS**

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Acid whey (AW) is the liquid co-product arising from acid-induced precipitation of casein from skim milk. Further processing of AW is often challenging due to its high mineral content, which can promote aggregation of whey proteins, which contributes to high viscosity of the liquid concentrate during subsequent lactose crystallization and drying steps. This study focuses on

mineral precipitation, protein aggregation, and lactose crystallization in liquid AW concentrates (~ 55% total solids), and on the microstructure of the final powders from 2 independent industrial-scale trials. These AW concentrates were observed to solidify either during processing or during storage (24 h) of pre-crystallized concentrate. The more rapid solidification in the former was associated with a greater extent of lactose crystallization and a higher ash-to-protein ratio in that concentrate. Confocal laser scanning microscopy analysis indicated the presence of a loose network of protein aggregates ( $\leq 10 \mu\text{m}$ ) and lactose crystals (100–300  $\mu\text{m}$ ) distributed throughout the solidified AW concentrate. Mineral-based precipitate was also evident, using scanning electron microscopy, at the surface of AW powder particles, indicating the formation of insoluble calcium phosphate during processing. These results provide new information on the composition- and process-dependent physicochemical changes that are useful in designing and optimizing processes for AW.

#### **TECHNICAL NOTE: FOURIER TRANSFORM INFRARED SPECTRAL ANALYSIS IN TANDEM WITH $^{31}\text{P}$ NUCLEAR MAGNETIC RESONANCE SPECTROSCOPY ELABORATES DETAILED INSIGHTS INTO PHOSPHATE PARTITIONING DURING SKIMMED MILK MICROFILTRATION AND DIAFILTRATION**

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In this study, they investigated the same samples generated by the aforementioned separation using attenuated total reflectance (ATR) Fourier transform infrared (FTIR) spectroscopy analysis. The results confirmed that the technique was not only capable of differentiating between the mineral equilibrium of the casein phosphate nanocluster (CPN) and milk serum, but also complemented the application of  $^{31}\text{P}$  NMR. An ATR-FTIR broad band in the region of 1,055 to 1,036  $\text{cm}^{-1}$  and a specific band at 1,076  $\text{cm}^{-1}$  were identified as sensitive to the repartitioning of different phosphate species in milk in accordance with the  $^{31}\text{P}$  NMR signals representing casein-associated phosphate and inorganic phosphate in the serum. A third ATR-FTIR signal at 1,034  $\text{cm}^{-1}$  in milk, representing precipitated inorganic calcium phosphate, had not previously been detected by  $^{31}\text{P}$  NMR. Thus, the results indicate that a combination of ATR-FTIR and  $^{31}\text{P}$  NMR spectroscopies may be optimally used to follow mineral and protein phase changes in milk during membrane processing.

#### **PILOT-SCALE FORMATION OF WHEY PROTEIN AGGREGATES DETERMINE THE STABILITY OF HEAT-TREATED WHEY PROTEIN SOLUTIONS—EFFECT OF PH AND PROTEIN CONCENTRATION**

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Denaturation and consequent aggregation in whey protein solutions is critical to product functionality during processing. Solutions of whey protein isolate (WPI) prepared at 1, 4, 8, and 12% (wt/wt) and pH 6.2, 6.7, or 7.2 were subjected to heat treatment (85°C  $\times$  30 s) using a pilot-scale heat exchanger. The effects of heat treatment on whey protein denaturation and

aggregation were determined by chromatography, particle size, turbidity, and rheological analyses. The influence of pH and WPI concentration during heat treatment on the thermal stability of the resulting dispersions was also investigated. Whey protein isolate solutions heated at pH 6.2 were more extensively denatured, had a greater proportion of insoluble aggregates, higher particle size and turbidity, and were significantly less heat-stable than equivalent samples prepared at pH 6.7 and 7.2. The effects of WPI concentration on denaturation/aggregation behavior were more apparent at higher pH where the stabilizing effects of charge repulsion became increasingly influential. Solutions containing 12% (wt/wt) WPI had significantly higher apparent viscosities, at each pH, compared with lower protein concentrations, with solutions prepared at pH 6.2 forming a gel. Smaller average particle size and a higher proportion of soluble aggregates in WPI solutions, pre-heated at pH 6.7 and 7.2, resulted in improved thermal stability on subsequent heating. Higher pH during secondary heating also increased thermal stability. This study offers insight into the interactive effects of pH and whey protein concentration during pilot-scale processing and demonstrates how protein functionality can be controlled through manipulation of these factors.

#### **APPLICATION OF FRONT-FACE FLUORESCENCE SPECTROSCOPY AS A TOOL FOR MONITORING CHANGES IN MILK PROTEIN CONCENTRATE POWDERS DURING STORAGE**

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This study investigated the feasibility of front-face fluorescence spectroscopy (FFFS) to predict the solubility index and relative dissolution index (RDI) of milk protein concentrate (MPC) powders during storage. Twenty MPC powders with varying protein contents from 4 different commercial manufacturers were used in this study. The MPC powders were stored at 2 temperatures (25 and 40°C) for 0, 1, 2, 4, 8, and 12 wk. The front-face fluorescence spectra of tryptophan and Maillard products were recorded and analyzed with chemometrics to predict solubility of MPC powders. The similarity maps showed clear discrimination of the MPC samples stored at 25 and 40°C. Partial least squares regression models were developed using the fluorescence spectra of tryptophan and Maillard products to predict the solubility index and RDI measurements of MPC powders, and the prediction models were validated using an independent test set. Coefficients of determination ( $R^2$ ) of 0.76, 0.84, and 0.68 were obtained between fluorescence spectra (tryptophan emission, Maillard emission, and Maillard excitation, respectively) and solubility index. The  $R^2$  values for the RDI predictions were 0.58 and 0.60 for the data set of tryptophan emission and Maillard emission, respectively. The ratio of prediction error to standard deviation was  $>2$  for Maillard emission fluorescence spectra and solubility index measurements, indicating good practical utility of the partial least squares regression prediction models. The results indicated that the solubility and dissolution behavior of MPC powders were related to their protein content and storage conditions that could be measured using FFFS. Hence, FFFS can be used as a rapid nondestructive analytical technique to predict the solubility and dissolution characteristics of MPC powders.

**EFFECT OF PROCESSING METHODS AND PROTEIN CONTENT OF THE CONCENTRATE ON THE PROPERTIES OF MILK PROTEIN CONCENTRATE WITH 80% PROTEIN**

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In this study, MPC80 powders were manufactured by 2 types of concentration methods: membrane filtration with and without the inclusion of an evaporation step. Different concentration methods could affect the mineral content of MPC powders, as soluble salts can permeate the UF membrane, whereas no mineral loss occurs during evaporation, although a shift in calcium equilibrium toward insoluble forms may occur at high protein concentration levels. It is more desirable from an energy efficiency perspective to use higher total solids in concentrates before drying, but concerns exist about whether a higher protein content would negatively affect powder functionality. Thus, MPC80 powders were also manufactured from concentrates that had 3 different final protein concentrations (19, 21, and 23%; made from 1 UF retentate using batch recirculation evaporation, a similar concentration method). After manufacture, powders were stored for 6 mo at 30°C to help understand changes in MPC80 properties that might occur during shelf-life. Solubility and foaming properties were determined at various time points during high-temperature powder storage. Inclusion of an evaporation step, as a concentration method, resulted in MPC80 that had higher ash, total calcium, and bound calcium (of rehydrated powder) contents compared to concentration with only membrane filtration. Concentration method did not significantly affect the bulk (tapped) density, solubility, or foaming properties of the MPC powders. Powder produced from concentrate with 23% protein content exhibited a higher bulk density and powder particle size than powder produced from concentrate that had 19% protein. The solubility of MPC80 powder was not influenced by the protein content of the concentrate. The solubility of all powders significantly decreased during storage at 30°C. Higher protein concentrations in concentrates resulted in rehydrated powders that had higher viscosities (even when tested at a constant protein concentration). The protein content of the concentrate did not significantly affect foaming properties. Significant changes in the mineral content are used commercially to improve MPC80 solubility. However, although the concentration method did produce a small change in the total calcium content of experimental MPC80 samples, this modification was not sufficiently large enough (<7%) to influence powder solubility.