Effect of Soluble Calcium and Lactose on Limiting Flux and Serum Protein Removal During Skim Milk Microfiltration

M.C. Adams, E.E. Hurt & D.M. Barbano

The tendency of calcium to promote microfiltration (MF) membrane fouling is well documented, but the role of lactose has not been studied. Milk protein concentrate that is 85% protein on a dry basis (MPC85) contains less calcium and lactose than skim milk. Our objectives were to determine the effects of skim milk soluble calcium and lactose concentrations on the limiting fluxes (LF) and serum protein (SP) removal factors of 0.1-µm ceramic graded permeability membranes. The MF was fed with 3 different milks: skim milk, liquid MPC85 that had been standardized to the protein content of skim milk with reverse osmosis water (MPC), and liquid MPC85 that had been standardized to the protein and lactose contents of skim milk with reverse osmosis water and lactose monohydrate (MPC+L). Retentate and permeate were continuously recycled to the feed tank. The LF for each feed was determined by increasing flux once per hour from 55 kg·m$^{-2}$·h$^{-1}$ until flux did not increase with increasing transmembrane pressure. Temperature, pressure drop across the membrane length, and protein concentration in the retentate recirculation loop were maintained at 50°C, 220 kPa, and 8.77 ± 0.2%, respectively. Experiments were replicated 3 times and the Proc GLM procedure of SAS was used for statistical analysis. An increase in LF between skim milk (91 kg·m$^{-2}$·h$^{-1}$) and MPC+L (124 kg·m$^{-2}$·h$^{-1}$) was associated with a reduction in soluble calcium. The LF of MPC+L was lower than the LF of MPC (137 kg·m$^{-2}$·h$^{-1}$) due to the higher viscosity contributed by lactose. Permeates produced from the MPC and MPC+L contained more protein than the skim milk permeate due to the transfer of caseins from the micelles into the reduced-calcium sera of the MPC and MPC+L. A SP removal factor 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. No differences in SP removal factors were detected among the 3 feeds below the LF. As the fluxes approached the LF, SP removal factors decreased due to fouling. Feeding a MF system with MPC instead of skim milk will reduce the required membrane surface area, but the permeate protein composition will be slightly higher in casein content.
Effect of Ceramic Membrane Channel Geometry and Uniform Transmembrane Pressure on Limiting Flux and Serum Protein Removal During Skim Milk Microfiltration

M.C. Adams, E.E. Hurt & D.M. Barbano

The objectives were to determine the effects of a ceramic microfiltration (MF) membrane’s retentate flow channel geometry (round or diamond-shaped) and uniform transmembrane pressure (UTP) on limiting flux (LF) and serum protein (SP) removal during skim milk MF at a temperature of 50°C, a retentate protein concentration of 8.5%, and an average cross-flow velocity of 7 m·s⁻¹. Performance of membranes with round and diamond flow channels was compared in UTP mode. Performance of the membrane with round flow channels was compared with and without UTP. Using UTP with round flow channel MF membranes increased the LF by 5% when compared with not using UTP, but SP removal was not affected by the use of UTP. Using membranes with round channels instead of diamond-shaped channels in UTP mode increased the LF by 24%. This increase was associated with a 25% increase in Reynolds number and can be explained by lower shear at the vertices of the diamond-shaped channel’s surface. The SP removal factor of the diamond channel system was higher than the SP removal factor of the round channel system below the LF. However, the diamond channel system passed more casein into the MF permeate than the round channel system. Because only one batch of each membrane was tested in our study, it was not possible to determine if the differences in protein rejection between channel geometries were due to the membrane design or random manufacturing variation. Despite the lower LF of the diamond channel system, the 47% increase in membrane module surface area of the diamond channel system produced a modular permeate removal rate that was at least 19% higher than the round channel system. Consequently, using diamond channel membranes instead of round channel membranes could reduce some of the costs associated with ceramic MF of skim milk if fewer membrane modules could be used to attain the required membrane area.

Susceptibility of Whey Protein Isolate to Oxidation and Changes in Physicochemical, Structural, and Digestibility Characteristics

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Oxidation is an important factor for denaturing of whey protein isolate (WPI) during food processing. We studied the effects of chemical oxidation on physicochemical and structural changes along with in vitro digestibility of WPI in this work. Evaluation of physicochemical changes showed that carbonyl level and dityrosine content increased, whereas total and free thiol group levels decreased for oxidized WPI samples. For the structural changes, protein aggregation was measured by surface hydrophobicity, turbidity, and particle diameter, which was increased for oxidized WPI samples. The increase of the secondary structure β-sheets and antiparallel β-sheet
also supported the aggregation of oxidized WPI. A direct quantitative relationship between physicochemical and structural changes and protein digestibility indicated that oxidation-related damage restricts the susceptibility of WPI to proteases. In conclusion, WPI had high susceptibility to oxidative stress, and both physicochemical and structural changes caused by severe oxidative stress could decrease the rate of in vitro digestibility of WPI.

Effect of Temperature and Concentration on Benzoyl Peroxide Bleaching Efficacy and Benzoic Acid Levels in Whey Protein Concentrate

T.J. Smith, P.D. Gerard & M.A. Drake

The objective was to compare norbixin destruction, residual benzoic acid, and flavor differences between liquid whey and 80% whey protein concentrates (WPC80) bleached at different temperatures with 2 different benzoyl peroxides (soluble and insoluble). Two experiments were conducted in this study. For experiment 1, 3 factors (temperature, bleach type, bleach concentration) were evaluated for norbixin destruction using a response surface model–central composite design in liquid whey. For experiment 2, norbixin concentration, residual benzoic acid, and flavor differences were explored in WPC80 from whey bleached by the 2 commercially available BP (soluble and insoluble) at 5 mg/kg. In liquid whey, soluble BP bleached more norbixin than insoluble BP, especially at lower concentrations (5 and 10 mg/kg) at both cold (4°C) and hot (50°C) temperatures. The WPC80 from liquid whey bleached with BP at 50°C had lower norbixin concentration, benzoic acid levels, cardboard flavor, and aldehyde levels than WPC80 from liquid whey bleached with BP at 4°C. Regardless of temperature, soluble BP destroyed more norbixin at lower concentrations than insoluble BP. The WPC80 from soluble-BP-bleached wheys had lower cardboard flavor and lower aldehyde levels than WPC80 from insoluble-BP-bleached whey. This study suggests that new, soluble (liquid) BP can be used at lower concentrations than insoluble BP to achieve equivalent bleaching and that less residual benzoic acid remains in WPC80 powder from liquid whey bleached hot (50°C) than cold (4°C), which may provide opportunities to reduce benzoic acid residues in dried whey ingredients, expanding their marketability.

Low-Sodium Cheddar Cheese: Effect of Fortification of Cheese Milk with Ultrafiltration Retentate and High-Hydrostatic Pressure Treatment of Cheese

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Low-sodium cheeses often exhibit an acidic flavor due to excessive acid production during the manufacturing and the initial stage of ripening, which is caused by ongoing starter culture activity facilitated by the low salt-in-moisture levels. We proposed that this excessive starter-induced acidity could be prevented by the fortification
of cheese milk with ultrafiltration (UF) retentates (to increase curd buffering), and by decreasing microbial activity using the application of high-hydrostatic pressure (HHP) treatment (that is, to reduce residual starter numbers). Camel chymosin was also used as a coagulant to help reduce bitterness development (a common defect in low-sodium cheeses). Three types of low-Na (0.8% NaCl) Cheddar cheeses were manufactured: non-UF fortified, no HHP applied (L-Na); UF-fortified (cheese milk total solids = 17.2 ± 0.6%), no HHP applied (L-Na-UF); and UF-fortified, HHP-treated (L-Na-UF-HHP; 500 MPa for 3 min applied at 1 d post-cheese manufacture). Regular salt (2% NaCl) non-UF fortified, non-HHP treated (R-Na) cheese was also manufactured for comparison purposes. Analysis was performed at 4 d, 2 wk, and 1, 3, and 6 mo after cheese manufacture. Cheese functionality during ripening was assessed using texture profile analysis and dynamic low-amplitude oscillatory rheology. Sensory Spectrum and quantitative descriptive analysis was conducted with 9 trained panelists to evaluate texture and flavor attributes using a 15-point scale. At 4 d and 2 wk of ripening, L-Na-UF-HHP cheese had ~2 and ~4.5 log lower starter culture numbers, respectively, than all other cheeses. Retentate fortification of cheese milk and HHP treatment resulted in low-Na cheeses having similar insoluble calcium concentrations and pH values compared with R-Na cheese during ripening. The L-Na-UF cheese exhibited significantly higher hardness values (measured by texture profile analysis) compared with L-Na cheese until 1 mo of ripening; however, after 1 mo, all low-Na cheeses exhibited similar hardness values, which were significantly lower than R-Na cheese. Pressure treatment significantly increased maximum loss tangent (meltability) from rheology testing and decreased melt temperature. Sensory results indicated only very slight bitterness (<2.5 out of 15-point scale) was detected in all cheeses during the 6 mo of ripening. The L-Na-UF-HHP cheese did not significantly differ in bitterness and acidity from R-Na cheese during ripening. Pressures treatment of cheese at 500 MPa and cheese milk retentate fortification could be used to improve the quality of low-Na cheese.

The Composition and Functional Properties of Whey Protein Concentrates Produced From Buttermilk are Comparable with Those of Whey Protein Concentrates Produced From Skimmed Milk

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This study demonstrates that buttermilk (BM) can be a potential source for the production of a WPC with a comparable composition and functional properties to a WPC obtained by MF of SM. Through the production of WPC powder and a casein- and phospholipid (PL)-rich fraction by the MF of BM, sweet BM may be used in a more optimal and economical way. Sweet cream BM from industrial churning was skimmed before MF with 0.2-µm ceramic membranes at 55 to 58°C. The fractionations of BM and SM were performed under the same conditions using the same process, and the whey protein fractions from BM and SM were concentrated by ultrafiltration and diafiltration. The ultrafiltration and diafiltration was performed at 50°C using pasteurized tap water and
a membrane with a 20-kDa cut-off to retain as little lactose as possible in the final WPC powders. The ultrafiltrates were subsequently spray dried, and their functional properties and chemical compositions were compared. The amounts of whey protein and PL in the WPC powder from BM (BMWPC) were comparable to the amounts found in the WPC from SM (SMWPC); however, the composition of the PL classes differed. The BMWPC contained less total protein, casein, and lactose compared with SMWPC, as well as higher contents of fat and citric acid. No difference in protein solubility was observed at pH values of 4.6 and 7.0, and the overrun was the same for BMWPC and SMWPC; however, the BMWPC made less stable foam than SMWPC.

The Effect of Microfiltration on Color, Flavor, and Functionality of 80% Whey Protein Concentrate

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The objective was to evaluate the efficacy of microfiltration (MF) on norbixin removal and to compare flavor and functionality of 80% whey protein concentrate (WPC80) from MF whey to WPC80 from whey bleached with HP or lactoperoxidase (LP). Cheddar cheese whey was manufactured from colored, pasteurized milk. The fluid whey was pasteurized and fat separated. Liquid whey was subjected to 4 different treatments: control (no bleaching; 50°C, 1 h), HP (250 mg of HP/kg; 50°C, 1 h), and LP (20 mg of HP/kg; 50°C, 1 h), or MF (microfiltration; 50°C, 1 h). The treated whey was then ultrafiltered, diafiltered, and spray-dried to 80% concentrate. The entire experiment was replicated 3 times. Proximate analyses, color, functionality, descriptive sensory and instrumental volatile analysis were conducted on WPC80. The MF and HP- and LP-bleached WPC80 displayed a 39.5, 40.9, and 92.8% norbixin decrease, respectively. The HP and LP WPC80 had higher cardboard flavors and distinct cabbage flavor compared with the unbleached and MF WPC80. Volatile compound results were consistent with sensory results. The HP and LP WPC80 were higher in lipid oxidation compounds (especially heptanal, hexanal, pentanal, 1-hexen-3-one, 2-pentylfuran, and octanal) compared with unbleached and MF WPC80. All WPC80 had >85% solubility across the pH range of 3 to 7. The microstructure of MF gels determined by confocal laser scanning showed an increased protein particle size in the gel network. MF WPC80 also had larger storage modulus values, indicating higher gel firmness. Based on bleaching efficacy comparable to chemical bleaching with HP, flavor, and functionality results, MF is a viable alternative to chemical or enzymatic bleaching of fluid whey.

Properties of Acid Whey as a Function of pH and Temperature


The present study aimed to identify chemical and physical properties of acid whey (AW) solutions as a function of pH (3 to 10.5) at 4 different temperatures (15, 25,
To propose appropriate membrane-processing conditions for efficient use of AW streams, the concentration of minerals, mainly calcium and phosphate, and proteins in centrifuged supernatants was significantly lowered with increase in either pH or temperature. Lactic acid content decreased with pH decline and rose at higher temperatures. Calcium appeared to form complexes with phosphates and lactates mainly, which in turn may have induced molecular attractions with the proteins. An increase in pH led to more soluble protein aggregates with large particle sizes. Surface hydrophobicity of these particles increased significantly with temperature up to 40°C and decreased with further heating to 90°C. Surface charge was clearly pH dependent. High lactic acid concentrations appeared to hinder protein aggregation by hydrophobic interactions and may also indirectly influence protein denaturation. Processing conditions such as pH and temperature need to be optimized to manipulate composition, state, and surface characteristics of components of AW systems to achieve an efficient separation and concentration of lactic acid and lactose.

**Authentication of Whey Protein Powders by Portable Mid-Infrared Spectrometers Combined with Pattern Recognition Analysis**


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The objective was to develop a simple and rapid method to differentiate whey protein types (WPC, WPI, and WPH) used for beverage manufacturing by combining the spectral signature collected from portable mid-infrared spectrometers and pattern recognition analysis. Whey protein powders from different suppliers are produced using a large number of processing and compositional variables, resulting in variation in composition, concentration, protein structure, and thus functionality. Whey protein powders including whey protein isolates, whey protein concentrates and whey protein hydrolysates were obtained from different suppliers and their spectra collected using portable mid-infrared spectrometers (single and triple reflection) by pressing the powder onto an Attenuated Total Reflectance (ATR) diamond crystal with a pressure clamp. Spectra were analyzed by soft independent modeling of class analogy (SIMCA) generating a classification model showing the ability to differentiate whey protein types by forming tight clusters with interclass distance values of >3, considered to be significantly different from each other. The major bands centered at 1640 and 1580 cm\(^{-1}\) were responsible for separation and were associated with differences in amide I and amide II vibrations of proteins, respectively. Another important band in whey protein clustering was associated with carboxylate vibrations of acidic amino acids (\(\approx 1570\) cm\(^{-1}\)). The use of a portable mid-IR spectrometer combined with pattern recognition analysis showed potential for discriminating whey protein ingredients that can help to streamline the analytical procedure so that it is more applicable for field-based screening of ingredients.
Sensory and Functionality Differences of Whey Protein Isolate Bleached by Hydrogen or Benzoyl Peroxide

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The objective was to compare sensory and functionality differences between whey protein isolate (WPI) bleached by benzoyl peroxide (BP) or hydrogen peroxide (HP). HP and BP bleached WPI and unbleached controls were manufactured in triplicate. Descriptive sensory analysis and gas chromatography-mass spectrometry were conducted to determine flavor differences between treatments. Functionality differences were evaluated by measurement of foam stability, protein solubility, SDS-PAGE, and effect of NaCl concentration on gelation relative to an unbleached control. HP bleached WPI had higher concentrations of lipid oxidation and sulfur containing volatile compounds than both BP and unbleached WPI ($P < 0.05$). HP bleached WPI was characterized by high aroma intensity, cardboard, cabbage, and fatty flavors, while BP bleached WPI was differentiated by low bitter taste. Overrun and yield stress were not different among WPI ($P < 0.05$). Soluble protein loss at pH 4.6 of WPI decreased by bleaching with either hydrogen peroxide or benzoyl peroxide ($P < 0.05$), and the heat stability of WPI was also distinct among WPI ($P < 0.05$). SDS PAGE results suggested that bleaching of whey with either BP or HP resulted in protein degradation, which likely contributed to functionality differences. These results demonstrate that bleaching has flavor effects as well as effects on many of the functionality characteristics of whey proteins.