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Short communication: Effect of storage temperature on the solubility of milk protein concentrate 80 (MPC80) treated with NaCl or KCl

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ABSTRACT

A previous study in our laboratory showed that addition of 150 m*M* NaCl or KCl into diafiltration water improved the solubility of freshly made milk protein concentrate 80 (MPC80). In the present study, the objectives were (1) to evaluate the solubility of NaCl- or KCl-treated MPC80 samples kept at varying temperatures and then stored for extensive periods at room temperature (21°C ± 1°C); and (2) to determine if MPC80 samples stored at different temperatures and protein conformation can be grouped or categorized together. Freshly manufactured MPC80 samples were untreated (control), processed with NaCl, or processed with KCl. One set of sample bags was stored at 4°C; second and third sets of bags were kept at 25°C and 55°C for 1 mo (31 d) and then transferred to room temperature (21°C ± 1°C) storage conditions for 1 yr (365 d). Samples were tested for nitrogen solubility index (NSI) and for protein changes by Fourier-transform infrared (FTIR) spectroscopy. Analysis of variance results for NSI showed 2 significantly different groupings of MPC80 samples. The more soluble group contained samples treated with NaCl or KCl and stored at either 4°C or 25°C. These samples had mean NSI >97.5%. The less soluble groups contained all control samples, regardless of storage temperature, and NaCl- or KCl-treated samples stored at 55°C. These samples had mean NSI from 39.5 to 58%. Within each of these groups (more soluble and less soluble), no significant differences in solubility were detected. Pattern recognition analysis by soft independent modeling of class analogy (SIMCA) was used to assess protein changes during storage by monitoring the amide I and amide II (1,700^{-1} to 1,300 cm^{-1}) regions. Dominant bands were observed at 1,385 cm^{-1} for control, 1,551 cm^{-1} for KCl-treated samples, and 1,694 cm^{-1} for NaCl-treated samples. Moreover, SIMCA clustered the MPC80 samples stored at 4°C separately from samples stored at 25°C and 55°C. This study demonstrates that (1) the addition of NaCl or KCl during MPC80 manufacture reduces the deleterious changes in solubility upon prolonged storage at 4°C or 25°C, and (2) the solubility of samples stored at 55°C is poor irrespective of salt treatment.
Key words: milk protein concentrate, salt treatment, solubility

Short Communication

Milk protein concentrates (**MPC**) are manufactured by ultrafiltration (UF) and diafiltration, followed by spray drying of skim milk. High-protein milk powders such as MPC with protein content ranging from 80 to 86% have been reported to exhibit low solubility (De Castro-Morel and Harper, 2002; Havea, 2006; Sikand et al., 2013). Havea (2006) reported that MPC85 (i.e., with 85% protein) powders stored at 20°C had 53% solubility after only 2 d of storage. On the other hand, high solubility (98%) was reported even after 7 wk of storage of MPC85 when the calcium content of UF skim milk retentate was partially replaced by sodium ions via cation exchange process (Bhaskar et al., 2001). Thus, high solubility with low insoluble material in MPC80 and with low calcium was attributed to more electrostatic repulsive forces among the casein micelles. Similar effects of enhanced solubility were observed when 50 to 150 m*M* NaCl was added during the diafiltration step of MPC80 manufacturing (Mao et al., 2012). Those authors attributed the enhanced solubility to modification of hydrophobic sites and reduced disulfide contents.

Carr et al. (2002) reported significantly improved solubility for MPC85 samples treated with monovalent salt compared with that of untreated control samples. These samples were stored at 40°C for 0 to 4 wk. The improved solubility of salt-treated samples was observed upon powder reconstitution at temperatures ranging between 20°C and 40°C compared with the control samples.

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Several studies on poor solubility of MPC associated with increasing storage time, temperature, and humidity have been conducted (Anema et al., 2006; Le et al., 2011). However, there is no comprehensive study of solubility of MPC80 powders treated with monovalent salts (NaCl or KCl) during the diafiltration (**DF**) stage of MPC80 manufacturing and stored at different temperatures. Thus, a better understanding of age-related changes in MPC solubility is needed. The objectives of this study were (1) to evaluate the solubility of MPC80 powders treated with monovalent salts (150 m*M* NaCl or KCl), including one set of samples stored at 4°C for 1 yr; and 2 sets of samples at 25°C and 55°C for 1 mo followed by storage at room temperature (21°C ± 1°C) for a period of 1 yr (365 d); and (2) to group or categorize the solubility of MPC80 samples treated with monovalent salts and stored at various temperatures with protein conformations as indicated by Fourier-transform infrared (**FTIR**) spectroscopy.

Milk protein concentrate powders were manufactured in the pilot plant of Dairy Products Technology Center at California Polytechnic State University (San Luis Obispo) with an R12 cross-flow membrane pilot-plant unit (Niro Inc., Hudson, WI) equipped with dual 10-kDa cut-off spiral-wound polyethersulfone (PES) membranes (Synder Filtration, Vacaville, CA). In the MPC powder manufacture, pasteurized skim milk (140 kg) was concentrated up to 6×. The UF milk was diafiltrated (6×) before spray drying. During the DF stage, a premixed salt solution (150 m*M* NaCl or KCl) was used to wash the UF retentate. The DF process was repeated 3 times. The DF retentate was collected after the third washing of diafiltration and further spray dried by using a Niro Filterlab (Niro Inc.) unit. The manufacturing details for the MPC80 are described by Gualco (2010). The protein content of the MPC80 powders was approximately 80%.

Freshly manufactured MPC80 samples were untreated (control; **MPC80-C**) or treated with 150 m*M* NaCl or KCl (**MPC80-Na** or **MPC80-K**). Two replicates of each sample were produced. Each sample was further split into 3 small bags. A total of 9 types of MPC80 samples were generated in 3 sets. All 3 sets of samples consisted of 3 types, MPC80-C, MPC80-Na, and MPC80-K. The MPC80 samples were packed in vacuum sealed bags (18 × 7 × 4 cm). The packaging material used was a multiwall foil gusset bag (Stock Bag Depot, China, CA) construction consisting of polyethylene terephthalate, aluminum, and linear low density polyethylene (PET/AL/LLDPE). The manufacturer's reported water vapor transmission rate on the bag was 0.00001 g/100 in^2/day measured at 37.8°C. The packaging device used was a modified MVS 38 vacuum sealer (Minipack America Inc., Orange, CA). All of the MPC80 powders were stored in vacuum-sealed bags and further enclosed in a multiwall foil gusset sample bag. One set of sample was stored at 4°C and not treated further. Second and third sets of samples were stored at 25°C and 55°C, respectively, for 31 d and then both sets were transferred to room temperature (21°C ± 1°C) and stored for 1 yr.

For the present study, nitrogen solubility index (**NSI**) was used as measure of solubility, and FTIR spectroscopy analysis was used to determine any structural or conformational changes in the protein with respect to storage temperature of MPC80 samples.

The samples were prepared according to the method by Morr et al. (1985). The Vario Max analyzer (Hanau, Germany) was used to determine the nitrogen content in the samples. A conversion factor of 6.38 was used to convert nitrogen to protein content. One gram of sample was dissolved in 80 mL of deionized water and hydrated for 1 h. The pH of each sample was adjusted to pH 7.0 and the volume was made to 100 mL with deionized water. Samples were centrifuged at 20,000 × *g* for 30 min. The NSI was calculated as follows:

$$\text{NSI (\%)} = \frac{\text{N content in supernatant}}{\text{N content in MPC80 dispersion}} \times 100\%.$$

Spectral data were collected using an Excalibur Series 3100 FT-IR spectrometer (Varian, Walnut Creek, CA) equipped with a dynamically aligned Michelson interferometer, a potassium bromide beam splitter, and a broadband mercury-cadmium-telluride (MCT) detector. Aliquots (0.5 μL) of the MPC80 liquid samples (prepared per NSI test described in above section) were placed on a microscope slide and vacuum dried for 5 min to form thin films. The slide was placed directly onto the stage of a Varian 600 UMA FT-IR microscope (Varian, Randolph, MA), which was interfaced with the spectrometer and brought into contact with a slide-on attenuated total reflectance (ATR) germanium internal reflection element (Varian 600 UMA, Palo Alto, CA) for spectra collection. The spectra were collected using Resolution Pro Software (version 4.0, Varian) from 4,000 to 700 cm^{-1} with co-adding 128 scans to increase the signal to noise ratio. The absorbance spectrum was obtained by rationing the single beam spectrum against that of the air background. Soft independent modeling of class analogy (**SIMCA**), a multivariate technique based on principal component analysis, was used to discriminate among MPC80 samples treated and stored differently. Spectra were imported into Pirouette (version 4.0, Infometrix Inc., Bothell, WA), and chemometrics modeling software was used to perform the multivariate classification. Preprocessing methods such as normalization and second derivative were conducted