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Flavor and stability of milk proteins

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ABSTRACT

A greater understanding of the nature and source of dried milk protein ingredient flavor(s) is required to characterize flavor stability and identify the sources of flavors. The objective of this study was to characterize the flavor and flavor chemistry of milk protein concentrates (MPC 70, 80, 85), isolates (MPI), acid and rennet caseins, and micellar casein concentrate (MCC) and to determine the effect of storage on flavor and functionality of milk protein concentrates using instrumental and sensory techniques. Spray-dried milk protein ingredients (MPC, MPI, caseins, MCC) were collected in duplicate from 5 commercial suppliers or manufactured at North Carolina State University. Powders were rehydrated and evaluated in duplicate by descriptive sensory analysis. Volatile compounds were extracted by solid phase microextraction followed by gas chromatography-mass spectrometry (GC-MS) and gas chromatography-olfactometry. Compounds were identified by comparison of retention indices, odor properties, and mass spectra against reference standards. A subset of samples was selected for further analysis using direct solvent extraction with solvent-assisted flavor extraction, and aroma extract dilution analysis. External standard curves were created to quantify select volatile compounds. Pilot plant manufactured MPC were stored at 3, 25, and 40°C (44% relative humidity). Solubility, furosine, sensory properties, and volatile compound analyses were performed at 0, 1, 3, 6, and 12 mo. Milk proteins and caseins were diverse in flavor and exhibited sweet aromatic and cooked/milky flavors as well as cardboard, brothy, tortilla, soapy, and fatty flavors. Key aroma active compounds in milk proteins and caseins were 2-aminoacetophenone, nonanal, 1-octen-3-one, dimethyl trisulfide, 2-acetyl-1-pyrroline, heptanal, methional, 1-hexen-3-one, hexanal, dimethyl disulfide, butanoic acid, and acetic acid. Stored milk proteins developed animal and burnt sugar flavors over time. Solubility of MPC decreased and furosine concentration increased with storage time and temperature. Solubility of MPC 80 was reduced more than that of MPC 45, but time and temperature adversely affected solubility of both proteins, with storage temperature having the greatest effect. Flavor and shelf stability of milk proteins provide a foundation of knowledge to improve the flavor and shelf-life of milk proteins.
Key words: milk protein, flavor, shelf life

INTRODUCTION

Milk protein concentrates (**MPC**) and isolates (**MPI**) are a relatively new category of dried dairy ingredients. Milk protein concentrate is a complete dairy protein ingredient containing both caseins and whey proteins at a concentration ranging from 40 to 89%, whereas MPI contains a protein concentration of 90% or greater. Milk protein concentrates and isolates are manufactured by concentrating milk proteins (both whey proteins and caseins) from fluid skim milk by membrane filtration followed by spray drying. Globally, MPC production has quadrupled to over 164,000 t from 2000 to 2011 (USDEC, 2012). The United States is both the single largest user in the world and the single largest importer, and the US Dairy Export Council (**USDEC**) predicts significant growth through the end of the decade to satisfy rising demand. Micellar casein concentrate (**MCC**) is a product for which commercial production was initiated a few years ago so production data are not yet available. Micellar casein concentrate is produced by microfiltration of skim milk, followed by ultrafiltration to further concentrate proteins before spray drying. In the production of MCC, serum proteins are removed via microfiltration. In production of MPI, filtration is used to remove lactose from milk, but both casein and whey proteins are retained in their original proportions found in milk, without combining separately produced casein (caseinate) and whey proteins (ADPI, 2015). The flavor of these ingredients is an important quality parameter as ingredient flavor can influence consumer liking and acceptability of the finished product.

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Most of the research conducted on MPC, MCC, or caseins has been focused on functionality and very little research has been conducted on flavor or elucidating the compounds responsible for the characteristic flavors of these proteins. Functional characteristics such as solubility during storage (Le et al., 2011a), the effect of NaCl addition during diafiltration on solubility, hydrophobicity, and soluble protein composition (Mao et al., 2012; Sikand et al., 2013), change in protein structure upon oxidation (Dalsgaard et al., 2007), and the influence of the Maillard reaction on functionality (Le et al., 2011a; Thomas et al., 2004) have all been studied in recent years; however, the effects of low (45%) and high (80%) protein content, and low temperature storage (3°C) on solubility and flavor have not been investigated.

Rennet and acid casein have been described as having an animal/wet dog or gluey flavor and characteristic aroma components of rennet casein were 2-aminoacetophenone, hexanoic acid, indole, guaiacol, and *p*-cresol (Karagul-Yuceer et al., 2003). The sensory profiles of higher protein MPC (MPC 77, MPC 80, and MPI) were characterized by tortilla, brothy, cardboard, and animal flavors (Drake et al., 2009). The flavor of MCC has not been reported, and the flavor, flavor chemistry, and flavor stability of milk proteins have not been fully investigated. The objectives of this study were to characterize the flavor and flavor chemistry of milk protein concentrates (MPC 70, 80, 85), isolates (MPI), acid and rennet caseins, and micellar casein concentrate (**MCC**; experiment 1), and to determine and compare the effects of storage temperature and time on the sensory and functionality (shelf-life) of low (45%) and high (85%) protein MPC over an extended period (1 yr; experiment 2) using instrumental and sensory techniques.

MATERIALS AND METHODS

Experimental Overview

Two experiments (experiments 1 and 2) were included in this study. The purpose of experiment 1 was to determine the sensory profiles and key aroma active volatile compounds in milk proteins. To carry out this objective, 9 commercial milk protein products were collected and one manufactured in the North Carolina State University (**NCSU**) dairy pilot plant. Products were collected or manufactured in duplicate. Descriptive analysis, headspace extraction and quantification of volatile compounds by GC-MS and gas chromatography-olfactometry (**GC-O**), and solvent-assisted flavor evaporation (**SAFE**) followed by aroma extract dilution analysis (**AEDA**) were performed on the dried milk protein products. The purpose of experiment 2 was to determine the flavor and functional properties of milk protein concentrates across storage. To conduct this objective, MPC 45 and 80 were produced in the NCSU dairy pilot plant and stored at constant relative humidity (44%) at 3, 25, or 40°C. Samples were measured after 0, 1, 3, 6, and 12 mo. Descriptive analysis, furosine content, solubility, and flavor volatiles by GC-MS were measured on all samples at each time point.

Table 1. Commercial proteins and manufacturers[1]

Manufacturer	Protein type
A	Rennet casein
A	Acid casein
B	MPC 70
B	MPC 85
B	MPI
C	MCC
D	MPC 80
E	MPC 80
E	MPC 85
P	MPC 45
P	MPC 80

[1]Two lots of each protein were obtained from each supplier. MPC = milk protein concentrate; MCC = micellar casein concentrate; MPI = milk protein isolate. Letters A–E are commercial suppliers; P means manufactured in the North Carolina State University pilot plant.

Sample Collection

Commercial Samples and Chemical Standards. Nine different products were obtained in duplicate lots from 5 different manufacturers across the United States and Canada (Table 1). Upon arrival, samples were store at −80°C until analysis. Chemical standards were obtained from Sigma-Aldrich (Milwaukee, WI) and ChemStep (Montesquieu, France).

Pilot Plant Production of MPC. Raw skim milk was obtained from the North Carolina State University Dairy Research and Education Unit on 2 different occasions. The milk was HTST pasteurized (720 kg/h; hold time 15 s at 72°C) using a plate heat exchanger (model T4 RGS-16/2, SPX Flow Technology, Greensboro, NC). The milk was the ultrafiltered and diafiltered using a laboratory scale ultrafiltration unit (model Lab 46, Filtration Engineering, Champlin, MN) equipped with 4 spiral wound membranes (Snyder Filtration, Vacaville, CA; nominal cutoff: 10,000 Da; surface area 5.0 m^2/membrane). The temperature for ultrafiltration was 50°C. For the MPC 45, the skim milk was ultrafiltered and protein monitored by Sprint Rapid Protein Analyzer (CEM, Matthews, NC) and Smart System 5 moisture/solids analyzer (CEM) until 45% protein (wt/wt) was achieved. Total filtration time was approximately 25 min. Total solids before spray drying were approximately 10%. For MPC 80, diafiltration