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Physical and chemical changes in whey protein concentrate stored at elevated temperature and humidity

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ABSTRACT

In a case study, we monitored the physical properties of 2 batches of whey protein concentrate (WPC) under adverse storage conditions to provide information on shelf life in hot, humid areas. Whey protein concentrates with 34.9 g of protein/100 g (WPC34) and 76.8 g of protein/100 g (WPC80) were stored for up to 18 mo under ambient conditions and at elevated temperature and relative humidity. The samples became yellower with storage; those stored at 35°C were removed from the study by 12 mo because of their unsatisfactory appearance. Decreases in lysine and increases in water activity, volatile compound formation, and powder caking values were observed in many specimens. Levels of aerobic mesophilic bacteria, coliforms, yeast, and mold were <3.85 $\log_{10}$ cfu/g in all samples. Relative humidity was not a factor in most samples. When stored in sealed bags, these samples of WPC34 and WPC80 had a shelf life of 9 mo at 35°C but at least 18 mo at lower temperatures, which should extend the market for these products.
Key words: whey protein concentrate, color, powder flow, volatiles

INTRODUCTION

The liquid whey generated during cheesemaking may be ultrafiltered and dried to produce whey protein concentrate (**WPC**) powder with a recommended shelf life of 9 to 12 mo (Sithole et al., 2005; Javidipour and Qian, 2008), which may be extended to 24 mo under refrigeration. The United States exported over 115,000 t of WPC in 2014, with China, Indonesia, and Malaysia being major importing countries (Lagrange et al., 2015). A study by the US Agency for International Development and Tufts University recommended that emergency aid foods include WPC to prevent stunted growth in children (Webb et al., 2011), which should further expand the market. In all likelihood, the nutritional benefits provided by WPC will continue to increase the global demand for whey proteins (Lagrange et al., 2015). However, the bags of WPC sent overseas are usually stored without refrigeration, exposing the product to elevated temperature and humidity. The shelf life of WPC under these conditions must be known to prevent the product from being rejected.

Maillard reactions and color changes have been observed in shelf-life studies of sweet whey powder (Sithole et al., 2005), as have lipid oxidation (Wright et al., 2009), volatile compound formation (Lee et al., 1996), and reduction of lysine (Li-Chan, 1983). The WPC is typically transferred from the original bags to lidded bins (Wright et al., 2009), sealed bottles (Lee et al., 1996; Sithole et al., 2005), or polyethylene bags (Li-Chan, 1983) for laboratory evaluation of storage properties. These studies do not reflect the actual storage conditions of WPC bags, however, because water vapor permeability characteristics would differ. Shelf-life studies of full-size bags of WPC have not been conducted, primarily because the facilities available for such studies are limited. Therefore, walk-in environmental chambers were obtained for storing bags of WPC at temperatures of 25, 30, and 35°C at relative humidity (**RH**) levels of 70 and 90%. Another chamber at ambient temperature and RH (uncontrolled) was used. Because WPC bags may be subjected to rough handling, half of the samples had been sealed in standard (**ST**) bags and half in high-performance (**HP**) bags to determine if

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packaging affected the results. The physical properties and microbial quality of WPC containing 34 and 80% protein (**WPC34** and **WPC80**, respectively) were then determined over an 18-mo period.

MATERIALS AND METHODS

Sample Bags

A dairy processor supplied 78 bags of WPC34 (34.9 g of protein/100 g) and 78 bags of WPC80 (76.8 g of protein/100 g) derived from Mozzarella cheese manufacture. Half of the samples had been packaged in HP bags (with a plastic liner 0.16 mm thick) and half in ST bags (liner was 0.10 mm thick). All bags contained 3 layers of Kraft paper glued across the top, with the low-density polyethylene liner in contact with the WPC and heat sealed near the top. The bags were divided evenly and placed inside environmental chambers. One WPC34 bag and 1 WPC80 bag were removed from each chamber and opened at each analysis time (0, 3, 6, 9, 12, and 18 mo). Samples were transferred by plastic scoop or metal spoon into Whirl-Pak containers (Nasco, Ft. Atkinson, WI) for testing, which took place within a week. Some bags were not analyzed at 3 mo and others not at 6 mo because of personnel limitations. Unopened bags were always used and opened bags were discarded after sampling.

Environmental Chambers

The chambers (Bally Refrigerated Boxes, Bally, PA) were 1.57 m wide, 1.87 m deep, and 1.83 m high. Heating was supplied by model HD3D heaters (Chromolox, Pittsburgh, PA) and humidification by model MP-5 ultrasonic humidifiers (Humidifirst, Boynton Beach, FL). The conditions in the 7 chambers were ambient (about 21°C, 45–65% RH), 25°C/70% RH, 25°C/90% RH, 30°C/70% RH, 30°C/90% RH, 35°C/70% RH, and 35°C/90% RH.

pH and Water Activity

The pH was determined in triplicate by mixing 28 mL of deionized water with 12 g of whey protein and stirring for 1 h before measuring. Water activity ($\mathbf{a_w}$) of 1-g samples was measured in triplicate by AquaLab 4TE water activity meter (Decagon Devices, Pullman, WA).

Color

Color was measured by ColorQuest XE spectrophotometer (Hunter Associates Laboratory, Reston, VA) in the RSIN mode using a 20-mL cuvette and a 19-mm aperture. Three readings were taken for each sample and lightness (**L***), red-green color (**a***), and yellow-blue color (**b***) were recorded.

Lysine

A GC-EZ Faast Kit (Phenomenex, Torrance, CA) was used to determine lysine content. Samples were treated with proprietary wash, derivatization, and purification solutions in a tube at room temperature. The derivatized lysine was analyzed by GC with flame-ionization detection.

Volatile Compounds

The procedure for analyzing volatile compounds, based on Tunick et al. (2013), utilized solid-phase microextraction (SPME). After warming a sample to 40°C for 25 min, a divinylbenzene (DVB)/Carboxen/polydimethylsiloxane (PDMS) Stableflex SPME fiber (Supelco, Bellefonte, PA) was exposed to the headspace above the sample in a closed vial for 30 min. The fiber was injected into a Varian gas chromatograph (model 3380, Varian, Walnut Creek, CA) equipped with a DB-5 column (30 m, 0.25 mm i.d., 0.25 μm film thickness; Restek US, Bellefonte, PA). The volatiles were desorbed for 5 min at 250°C and the GC oven was heated from 40 to 225°C at 10°C/min and held for 3 min. The detector was a Varian Saturn 200 mass spectrometer and the internal standard was 2-methyl-3-heptanone (Sigma Aldrich, St. Louis, MO). Volatile compounds were identified by the National Institute of Standards and Technology (NIST) library in the instrument's software and by comparison with authentic standards (Sigma Aldrich). Samples were analyzed in triplicate.

Fatty Acids

Lipids were extracted with a Foss 2050 Soxtec Automatic Extractor (Foss North America, Eden Prairie, MN) using the procedure for powdered products described by Richardson (2001). Extracted lipids were stored at −20°C under nitrogen.

A KOH-catalyzed methanolysis method for preparation of FAME was followed (Ichihara et al., 1996). Samples were analyzed on a Varian 3400 CX gas chromatograph with a Supelco 2380 capillary column (30 m × 0.25 mm × 0.20 μm film thickness), a flame-ionization detector, and He as carrier gas. The injector and detector temperatures were 240°C and 250°C, respectively. The column temperature was held at 50°C for 3 min, raised to 150°C at 10°C/min, held 1 min, raised to 222°C at 3°C/min, held 1 min, and finally