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Differences in particle characteristics and oxidized flavor as affected by heat-related processes of milk powder

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ABSTRACT

Understanding the formation of oxidized flavor will be highly useful in the improvement of milk powder quality. Effects of preheating, concentration and spray-drying on the particle characteristics and the oxidized flavor stability of milk powder were investigated. The surface composition and free radicals were analyzed using x-ray photoelectron spectroscopy and electron spin resonance spectrometry, respectively. The concentrations of selected oxidized volatiles hexanal and 2-heptanone were determined using solid-phase microextraction gas chromatography-mass spectrometry. Levels of hexanal and 2-heptanone in fresh milk powder were higher than those in raw milk and heated milk, which drastically increased with increasing time of storage. Differences in the morphological observations, free fat, and surface composition of fresh milk powder were found among different heat-related processes. During storage, a radical (g value, a characteristic constant whose value serves to identify any given free radical, was 2.0054) was detected in milk powder. The specific population of the radical increased from 2.99×10^7 at 3 mo to 1.23×10^8 at 6 mo of storage. Addition of ascorbic acid in milk powder changed the type of radicals and reduced the oxidation off-flavor. According to the Pearson correlations, not the surface compositions but the morphological characteristics of milk powder particles should be considered in maintaining the stability of oxidized flavor in storage.
Key words: oxidized flavor, particle characteristics, milk powder, process

INTRODUCTION

The flavor quality of milk powder is highly associated with consumer acceptability. Milk powder is very susceptible to oxidation, resulting in oxidation off-flavor. Oxidized flavor is the total description of metallic, soapy, papery, fatty, mushroom, and fishy flavor in dairy products (Timmons et al., 2001; Hedegaard et al., 2006; Lloyd et al., 2009a). Oxidized flavor is the most critical factor in restricting flavor characteristics and shelf life of milk powder (Whetstine and Drake, 2007; Lloyd et al., 2009a). The milk powder industry is confronted by situations where powders demonstrate lower flavor stability than expected. Increasing the storage stability of this commodity is of great interest to producers and end users (Nielsen et al., 1997; Lloyd et al., 2009b).

Certain compounds contribute to the oxidized flavor, such as aldehydes, ketones, and lactones, which have been indicated to be formed via lipid oxidation (Nielsen et al., 1997; Cadwallader and Singh, 2009). Some factors that influence the oxidized volatiles and their stability in storage of milk powder have been conducted, including initial milk quality, processing variables, moisture content, packaging, oxygen exposure, light exposure, and storage temperature (Baldwin et al., 1991; Stapelfeldt et al., 1997; Whetstine and Drake, 2007; Lloyd et al., 2009b). However, these studies were focused on the preheating treatments of milk and the storage of milk powder. The effect of heat-related processing variables on the formation of oxidized flavor in milk powder was limited.

The fat fraction of milk powder is the most important for the development of oxidized flavor. Free radicals, peroxide value, and thiobarbituric acid-reactive substances have been used as indicators for evaluation of the lipid oxidation of dairy products (Stapelfeldt et al., 1997; Kristensen et al., 2002; Thomsen et al., 2005). Few recent studies on free fat and surface fat of powder particles (Kim et al., 2002, 2003, 2005, 2009a; Vignolles et al., 2010) reported that drying could affect the formation and presence of free fat at the surface of powder particles. Additional detailed studies are needed to understand the formation of oxidized flavor and milk powder particles as affected by different processes. The aims of the study were to (1) determine the changes in oxidized flavor of milk powder as affected by different processes, (2) investigate the difference in particle characteristics of milk powder due to different processes, and (3) evaluate the free radical in storage and the effect of antioxidants on oxidized flavor of milk powder.

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MATERIALS AND METHODS

Manufacture of Milk Powder

Raw milk (**RM**) obtained from a local dairy plant had 0.02% (wt/vol) NaN_3 added to it to prevent bacterial growth and was stored at 4 to 6°C. Heated milk (**HM**) was prepared according to the method of Lee and Sherbon (2002), with the intensity of 90°C for 30 s. The milk was cooled to ambient temperature (20 ± 1°C) in a stirred water bath at 0 to 4°C and prepared for concentration and drying. The RM (and HM) was concentrated to 4 times at 40°C and 3 times at 50°C under 0.1 MPa pressure. The concentrated milks were subclassified according to the temperature of concentration. Spray-drying for RM, HM, and the concentrated milk of RM and HM (**RMC** and **HMC**, respectively) was done using a laboratory-scale spray dryer (EYELA SD-1000; Tokyo Rikakikai Co. Ltd., Tokyo, Japan). The milk powder of RM, HM, RMC, and HMC (**RMP**, **HMP**, **RMCP**, and **HMCP**, respectively) was subclassified according to the inlet-air temperature (160 and 190°C). Other parameters were as follows: nozzle pressure = 20 MPa, feed flow rate = 400 to 600 mL/h, and flow rate of drying air = 40 to 60 m^3/h. The outlet-air temperature was controlled at 85°C by adjusting the speed of material and hot air. Milk powder was stored in a dry and air-tight container at ambient temperature for 3 and 6 mo for flavor analysis.

Component Analyses of Milk

Fat content was determined by the Röse-Gottlieb method and total protein by the Kjeldahl technique with a factor of 6.38, as described by Guinee et al. (2000). The free fat was extracted using the method described by Kim et al. (2005). The extracted fat value was then recorded as grams of free fat per gram of fresh powder. The concentrated milk and milk powder were reconstituted for analysis according to the TS by adding back with Milli-Q water (Millipore Corp., Billerica, MA). Total solids content of milk and moisture content (%) of milk powder were calculated according to the weight loss by drying the samples at 105 ± 1°C (Almeida et al., 2009; Kim et al., 2009a).

Morphological Observation of Particles

Milk powder (0.1 g) was dissolved in 10 mL of acetone. After homogeneously mixing, a drop was placed into a blood cell count plate and observed under an optical microscope (CX31; Olympus Corp., Tokyo, Japan). Five horizons were selected randomly from the digital camera imaging of milk powder particles. Particle size and morphological image were analyzed using Image-Pro Plus (**IPP**) software (Image-Pro Plus 6.0; Media Cybernetics Inc., Rockville, MD).

Surface Composition of Powder Particles

The elemental surface composition of different powders was determined using an X-ray photoelectron spectroscopy (**XPS**) system (K-Alpha; Thermo Fisher Scientific Co., Waltham, MA) according to the method of Kim et al. (2009a). The scan interval of the monochromator for full spectrum was 1.0 eV. The elements C, O, N, P, and S were scanned and the step size was 0.1 eV.

Determination of Volatiles

The volatiles in the headspace of the samples (RM, HM, concentrated milk, and reconstituted milk powder) were extracted and analyzed using solid-phase microextraction gas chromatography-mass spectrometry (**SPME-GC-MS**) according to a previous study (Li et al., 2012). Hexanal and 2-heptanone, as selected oxidized volatiles, were identified by the National Institute of Standards and Technology (Gaithersburg, MD) NIST-02L GC-MS spectrum library and the retention time of their standard chemicals (Sigma, St. Louis, MO).

Detection of Free Radicals in Milk Powder

The radicals were measured by an electron spin resonance (**ESR**) spectrometer (model A200S-95/12; Bruker BioSpin GmbH, Rheinstetten, Germany). Electron spin resonance analysis was based on the method described by Thomsen et al. (2005). Different instrument parameters used were as follows: sweep width = 100 Gauss (0.01 Tesla), microwave power = 3.10 mW, modulation amplitude = 1.00 Gauss (1.0×10^{-4} Tesla), receiver gain = 5.02×10^4, time constant = 40.96 ms, conversion time = 160 ms, and total sweep time = 163.84 s.

Effect of Antioxidants on Oxidized Volatiles

The parameters of $HMC_{50}P_{190}$ (the subscript numbers indicate that concentration was performed at 50°C and drying was performed at 190°C) were used in the production of milk powder for the experiment of antioxidants addition. Chemical-grade α-tocopherol (**TOC**) and L-ascorbic acid (**ASC**) were obtained (Sigma). Before drying, 0.05% (wt/vol) TOC, 0.05% (wt/vol) ASC, and 0.025% (wt/vol) TOC and ASC were added into the concentrated milk. The milk powder products were

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