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Short communication: Formation of oxidized flavor compounds in concentrated milk and distillate during milk concentration

Y. H. Li*¹ and W. J. Wang†

*College of Food Science and Biotechnology Engineering, Zhejiang Gongshang University, Hangzhou 310018, China
 †Yangshengtang Health Products Co. Ltd., Hangzhou 311106, China

ABSTRACT

Oxidized flavor stability of milk is highly associated with consumer acceptance. This study characterized oxidized flavor stability as affected by milk concentration. The selected volatiles in raw milk (RM), heated milk (HM), concentrated milk, and distillate were investigated using solid-phase microextraction–gas chromatography–mass spectrometry. Concentrated milk and distillate showed higher levels of heptanal (14.52–17.24 $\mu\text{g}/\text{kg}$), nonanal (5.08–8.64 $\mu\text{g}/\text{kg}$), 2-heptanone (25.22–73.25 $\mu\text{g}/\text{kg}$), and 2-nonanone (10.83–17.28 $\mu\text{g}/\text{kg}$) compared with RM and HM. Based on the odor activity values of different volatiles, oxidized volatiles may contribute to fatty, greasy, soapy, and dyestuff oxidized odors in concentrated milk. Oxidized volatiles of nonanal, 2-heptanone, and 2-nonanone were found to be higher in concentrated milk at 50°C than at 40°C. In addition, 18 and 24 volatiles were found in unconcentrated milk and distillate, respectively. Straight aldehydes and methyl ketones showed much higher levels in distillate than in RM and HM, which also had higher levels compared with their corresponding concentrated milk samples. These results indicated that oxidized volatiles were mainly evaporated into distillate as flavor loss. They also suggested that lipid oxidation could continue drastically even under the concentration processing at a low temperature and reduced pressure. **Key words:** oxidized flavor, concentrated milk, distillate

Short Communication

The flavor quality of dairy products is highly associated with consumer acceptance. Oxidized flavor is a common off-flavor in milk and is described as tasting metallic, soapy, papery, fatty, mushroom, and fishy in dairy products (Hedegaard et al., 2006; Lloyd et al.,

2009a). An oxidized flavor is the most critical factor in harming flavor characteristics and restricting the shelf life of milk powder (Carunchia Whetstine and Drake, 2007; Lloyd et al., 2009a,b).

Compounds that contribute to an oxidized flavor include aldehydes, ketones, and lactones, which form via lipid oxidation (Nielsen et al., 1997; Cadwallader and Singh, 2009). Oxidized flavor in dairy products could be caused by factors including initial milk quality, processing variables, oxygen exposure, light exposure, and storage conditions (Baldwin et al., 1991; Stapelfeldt et al., 1997; Carunchia Whetstine and Drake, 2007; Lloyd et al., 2009b). Previous studies on oxidized flavor in milk mainly focused on preheating treatments of milk and the storage of dairy products (Vazquez-Landaverde et al., 2005; Carunchia Whetstine and Drake, 2007; Lloyd et al., 2009a,b). Meanwhile, the parameters of concentration ratio, premature crystallization of lactose, viscosity, and denaturation of protein were the primary concern in the concentration processing.

Few studies have analyzed the flavor components in concentrated milk. Shimoda et al. (2001) indicated that volatile compounds, such as fatty acids, lactones, ketones, alkanes, alcohols, and aldehydes, are the main flavor compounds identified in commercial sweetened condensed milk by using steam distillation–gas chromatography–mass spectrometry. Patel et al. (1962) reported that a rubber smell occurred in pasteurized condensed milk during storage, which was attributed to ketones, sulfides, and fatty acid esters. Arnold and Lindsay (1969) found that methyl ketone ($\text{C}_3\text{--}\text{C}_{11}$) and *O*-aminoacetophenone appeared to increase during storage at 27°C for 26 wk, and they also indicated that *O*-aminoacetophenone was the main flavor substance associated with a stale smell.

The application of reduced pressure and heating is widely used to produce concentrated milk. Milk is very susceptible to oxidation due to thermal degradation reactions. However, the effect of concentration processing on the development of oxidized flavor in concentrated milk has been ignored, although the processing is an indispensable step in the manufacture of condensed milk,

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¹Corresponding author: liyanhua607@163.com

evaporated milk, and milk powder. Moreover, the distillate derived from milk evaporation is discarded directly or used as the heat source for additional concentration processing. So far, the distillate from concentration processing has never been examined. This study investigated oxidized flavor compounds in concentrated milk and changes of volatiles in the distillate during milk concentration. The results could have important theoretical and practical implications in the flavor stability of dairy products.

Raw milk (**RM**) was obtained from a local dairy plant (Xingfu Farm), and 0.02% (wt/vol) NaN_3 was added to it to prevent bacterial growth. The milk was stored at 4 to 6°C. According to the methods of Lee and Sherbin (2002) and Li et al. (2013), heated milk (**HM**) was prepared by heating to 90°C for 30 s. The milk was cooled to ambient temperature ($20 \pm 1^\circ\text{C}$) in a stirred water bath at 0 to 4°C. According to the methods of Drohan et al. (1997) and Li et al. (2013), RM and HM were concentrated using laboratory-scale evaporators (N-1000, Eyela, Tokyo, Japan). The RM and HM samples were concentrated under 0.1 MPa 4 times at 40°C and 3 times at 50°C, respectively. The distillate generated from concentration process was collected for the determination of oxidized flavor compounds.

Milk fat was determined by the Rose–Gottlieb method and total protein by the Kjeldahl technique with a factor of 6.38 as described by Guinee et al. (2000). Total solids in milk samples were calculated according to the weight loss by drying the samples at $105 \pm 1^\circ\text{C}$ (Almeida et al., 2009).

The volatiles were determined according to previous methods (Li et al., 2013). Duplicate analyses for GC-MS are expressed as mean values, and analyses replicated 3 times are expressed as mean \pm SD. The odor activity value (**OAV**) of different volatile compounds was calculated as the ratio of the concentration and corresponding flavor threshold value. A compound could contribute to flavor when its OAV was above 1.0, and could smell slightly when its OAV ranged from 0.5 to 1.0 (Qian and Reineccius, 2003). Analysis of variance was used to distinguish the difference of the treatments, and principal component analysis (**PCA**) was used for the difference in volatiles of distillate were carried out using PASWStatistics18.0 Software (SPSS Inc., Chicago, IL).

The average proportions of protein and fat in all the milk samples were $3.2 \pm 0.1\%$ and $3.2 \pm 0.2\%$, respectively. Total milk solids were $12.1 \pm 0.3\%$. No differences were found in the compositions of RM, HM, and reconstituted concentrated milk ($P > 0.05$).

Oxidized volatiles detected in the unconcentrated milk, concentrated milk, and distillate mainly included straight-chain aldehydes, methyl ketones, ketenes, and

olefinic aldehydes. These compounds have been previously documented in different milk products and could cause different oxidized flavors (Carunchia Whetstone and Drake, 2007; Lloyd et al., 2009a; Li et al., 2013). Heptanal, nonanal, 2-heptanone, and 2-nonanone were listed as typical volatiles in discussions of oxidized flavors among products after the processes of heating and concentration, which were responsible for fatty, greasy, soapy, and dyestuff oxidized odors in dairy products, respectively.

Table 1 shows the contents of the volatile compounds in unconcentrated milk (RM and HM) and in concentrated milk (**RMC** and **HMC**). Low levels of oxidized volatiles were found in liquid milk (RM and HM). The concentrations of heptanal, nonanal, 2-heptanone, and 2-nonanone were 1.92, 1.02, 5.44, and 1.73 $\mu\text{g}/\text{kg}$ in RM, and 2.32, 1.24, 7.22, and 2.11 $\mu\text{g}/\text{kg}$ in HM, respectively. Based on the flavor thresholds of these volatiles of 3.0, 1.0, 5.0, and 5.0 $\mu\text{g}/\text{kg}$ in milk (Bendall, 2001; Vazquez-Landaverde et al., 2005), the corresponding OAV of these compounds were 0.64, 1.02, 1.09, and 0.35 in RM, and 0.77, 1.24, 1.44, and 0.42 in HM, respectively. These compounds did not differ significantly between HM and RM ($P > 0.05$). The OAV results indicated that volatiles of nonanal and 2-heptanone contributed to off-flavors and could endow RM and HM samples with fatty and soapy odors associated with oxidized flavor. Heptanal could be detected and contributed to the oxidized flavor of HM ($0.5 < \text{OAV} < 1.0$).

Oxidized volatiles in concentrated milk occurred at higher levels than in the corresponding unconcentrated groups: heptanal (14.52–17.24 $\mu\text{g}/\text{kg}$), nonanal (5.08–8.64 $\mu\text{g}/\text{kg}$), 2-heptanone (25.22–73.25 $\mu\text{g}/\text{kg}$), and 2-nonanone (10.83–17.28 $\mu\text{g}/\text{kg}$). The OAV of these volatiles in concentrated milk were from 4.84 to 5.75, from 5.08 to 8.64, from 5.04 to 14.65, and from 2.17 to 3.46, respectively. Compared with RM and HM, the concentration treatment increased the formation of oxidized flavor significantly ($P < 0.05$). Meanwhile, the oxidized volatiles nonanal, 2-heptanone, and 2-nonanone in concentrated milk (except 2-nonanone in RMC) processed at 50°C had higher levels than milk processed at 40°C. This result also suggested that lipid oxidation continued under the concentration conditions (i.e., low temperature and reduced pressure).

Table 2 shows the volatiles of RM, HM, and their corresponding distillates during concentration. A total of 18 and 24 volatiles were found in unconcentrated milk and distillate, respectively. Among them, straight aldehydes and methyl ketones occurred at much higher levels in distillate than in RM and HM, which also had higher levels compared with the corresponding concentrated milk samples (Table 1). Alkanes were slightly increased because of the oxidation (Nursten, 1997);

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