2-Heptanone, 2-nonanone, and 2-undecanone confer oxidation off-flavor in cow milk storage

Yanmei Xi, Sana Ikram, Tong Zhao, Yiwei Shao, Ruirui Liu, Fuhang Song, Baoguo Sun, and Nasi Ai

ABSTRACT

Flavor sensation is one of the most prevalent characteristics of food industries and an important consumer preference regulator of dairy products. So far, many volatile compounds have been identified, and their molecular mechanisms conferring overall flavor formation have been reported extensively. However, little is known about the critical flavor compound of a specific sensory experience in terms of oxidized off-flavor perception. Therefore, the present study aimed to compare the variation in sensory qualities and volatile flavors in full-fat UHT milk (FFM) and low-fat UHT milk (LFM) samples under different natural storage conditions (0, 4, 18, 25, 30, or 37°C for 15 and 30 d) and determine the main component causing flavor deterioration in the FFM and LFM samples using sensory evaluation, electronic nose, and headspace solid-phase microextraction-gas chromatography-mass spectrometry (HS-SPME-GC-MS). In addition, the Pearson correlation between the volatile flavor components and oxidative off-flavors was analyzed and validated by sensory recombination studies. Compared with the LFM samples, the FFM samples showed a higher degree of quality deterioration with increased storage temperature. Methyl ketones of odd carbon chains (i.e., 2-heptanone, 2-nonanone, 2-undecanone, 2-tridecanone, and 2-pentadecanone) reached a maximum content in the FFM37 samples over 30 d storage. The combined results of the Pearson correlation and sensory recombination study indicated that 2-heptanone, 2-nonanone, and 2-undecanone conferred off-flavor perception. Overall, the present study results provide potential target components for detecting and developing high-quality dairy products and lay a foundation for specific sensory flavor compound exploration in the food industry.

Key words: UHT milk, storage temperature, sensory, oxidation flavor

INTRODUCTION

In recent years, milk has gained increasing popularity in China due to its excellent characteristics. Milk is processed in various ways and can be categorized as pasteurized milk, ultra-pasteurized milk, extended shelf life milk, and UHT milk (Schmidt et al., 2017). Of which, UHT processing involves quick heating of milk to a high temperature to produce a stable shelf life product, which can be stored at room temperature for about 4 to 6 mo (Vazquez-Landaverde et al., 2005; Rumbold et al., 2022). Therefore, UHT milk is prevalent in many cities due to its advantages of commercial value, long storage period, and compatibility with long-distance transportation.

A flavor profile is a comprehensive sensory impression that directly determines consumer preference and options (Xi et al., 2023). So far, the volatile flavor information of dairy products by feeding type, homogenization, and heat treatment has been reported extensively (Jo et al., 2018; Li et al., 2018; Manzocchi et al., 2021). Off-flavor has the potential to mask the fresh, delicate flavor of UHT milk, so determining the defective flavor properties, such as “cooked,” “sulfurous,” “fishy,” and components related to off-flavor in milk samples is highly imperative (Cadwallader and Singh, 2009; Zabbia et al., 2012). The long shelf life of liquid dairy products is often affected by physical and chemical changes, such as protein hydrolysis, lipid oxidation, lipolysis, and the Maillard process, causing sensory impairment in UHT milk (Lu et al., 2019; Ranvir et al., 2020). Notably, lipid oxidation is the primary cause of quality deterioration in dairy products (Park and Drake, 2014). Off-flavors from oxidation of unsaturated fatty acids make a poor impression on consumers, leading to consumer unacceptability (Jansson et al., 2019).

In a previous study, UHT milk was reported to have an oxidative off-flavor, being stale, and not being fresh
during the shelf life (Jensen et al., 2015), probably due to the increased concentrations of methyl ketone leading to sensory quality deterioration until full-fat UHT milk (FFM) is stored at 25 ± 2°C for 90 d (Valero et al., 2001). Recent studies have reported the effect of kinetic changes on the storage stability of UHT milk and the importance of maintaining temperatures under control during storage and transport (Sunds et al., 2018). Although many studies have proved that refrigeration at 4°C could reduce lipid oxidation in UHT milk, the variation in volatile components in fat oxidation has not been fully elucidated yet (Lu et al., 2019). Therefore, various natural temperatures are essential for product stability during storage.

Fewer studies exist that combine volatile flavor components with corresponding changes in consumer perception. The present study aimed to investigate the release characteristics of volatile aroma in FFM and low-fat UHT milk (LFM) samples stored under different conditions using headspace solid-phase microextraction-GC-MS (HS-SPME-GC-MS) and an electronic nose (E-Nose; Airsense Analytics GmbH, Schwerin, Germany). In addition, the primary components responsible for off-flavors in UHT milk were explored. Overall, determining the components conferring flavor defects could help monitor milk flavor during shelf life.

MATERIALS AND METHODS

Sample Overview

The FFM and LFM samples were purchased from the Beijing Yonghui supermarket. The FFM samples were from the same new batch of manufacture and package (packed with Tetra Pak bricks). The compositions of FFM and LFM were 3.6% (wt/wt) protein and 4.4% (wt/wt) fat, respectively.

Milk Sample Processing

The purchased samples were stored for 3 mo at room temperature (25°C) in the dairy flavor laboratory of Beijing Technology and Business University (Beijing, China). Subsequently, FFM and LFM samples were stored at 0, 4, 18, 25, 30, and 37°C for 15 and 30 d and labeled as FF15d, FF30d, LF15d, and LF30d. Meanwhile, samples stored at different temperatures were labeled as FFM0, FFM4, FFM18, FFM25, FFM30, FFM37, LFM0, LFM4, LFM18, LFM25, LFM30, and LFM37, respectively.

Chemical Standard

The reference chemicals used for identification were of high purity, over 95%. The n-alkane C7-C30 were purchased from Sigma-Aldrich (St. Louis, MO). The chemicals 2-heptanone (98%), 2-pentadecanone (95%), 2-nonanone (99%), dimethyl sulfone (99%), 5-octano-lide (98%), 5-decanolide (97%), 4-dodecanolide (97%), 5-dodecalactone (97%), and octanoic acid (99%) were purchased from Sigma-Aldrich. The chemicals decanoic acid (98%), 2-undecanone (98%), acetophenone (98.50%), 2-tridecanone (95%), hexanoic acid (98%), nonanoic acid (90%), and dodecanoic acid (98%) were purchased from TCI (Shanghai, China). The chemicals decyl ester (95%), 2-octanone (98%), 2-decanone (98%), octanal (98%), nonanal (95%), decanal (97%), 1-tridecanol (99%), decane (99%), undecane (99%), and styrene (99%) were purchased from Aladdin Reagents Co. Ltd. (Shanghai, China).

Sensory Evaluation

This study was reviewed and approved by the Beijing Technology and Business University Institutional Review Board, and informed consent was obtained from each panelist prior their participation in the study. The sensory panel consisted of 5 females and 1 male (20–35 yr of age) who voluntarily participated in sensory evaluation of milk powder samples and were selected and trained from the Beijing Technology and Business University. Each panelist had a routine milk-drinking habit and underwent daily training every morning for at least 2 h/d for 14 d. Based on their previous milk sensory experience, the trained panelists were first required to establish qualitative sensory descriptors for each UHT milk sample (stored at different temperatures for 15 and 30 d) during the blind tasting process. A total of 3 odor profiles, including milk flavor, creaminess, and oxidized off-flavor, were identified by the panelists to evaluate the samples. The sensory qualities were as follows: (1) oxidized off-flavor was referred to as a sensation of discomfort resembling metallic, harshness, or an unpleasant feeling; (2) milk flavor was defined as having a milky scent unique to milk; and (3) creamy flavor was defined as having a fat smell. The formal sensory evaluation was conducted in a brightly lit, odorless, clean, noiseless dairy flavor sensory evaluation laboratory with a dedicated space free of distractions. The samples from various storage conditions were equilibrated to room temperature and presented to the panel members labeled with 3 random numbers. An evaluation scale from 1 to 5 was established, where 1 indicated that the attribute was almost absent, 2 to 4 indicated moderate intensity, and 5 indicated that the attribute was extremely strong. The judges were required to rinse their mouths with mouthwash between tastings to remove any lingering flavors. A scheduled break of 5 to 10 min was provided to ensure that the
sensory panel were fresh for the sensory assessment. Each sample was assessed twice by all panelists.

**E-Nose Analysis**

Variation in aroma release was calculated using an E-nose coupled with 10 semiconductor metal oxide chemical sensor components to differentiate the aromas. The sensory components W1C, W5S, W3C, W6S, W5C, W1S, W1W, W2S, W2W, and W3S were allergic to aromatic compounds, nitrogen oxides, ammonia and aromatic compounds, hydrogen, alkanes and aromatic compounds, methane, sulfur compounds, ethanol, aromatic and organic sulfur compounds, and alkanes, respectively (Yang et al., 2018).

Briefly, 8 mL of milk sample was poured into a headspace vial equipped with 1.0 g of sodium chloride and magnetic rotor, equilibrated with water at 40°C for 20 min, stirred at 20 to 30 rpm, and then inserted into an E-nose luer-lock needle to adsorb the gas in the headspace. The specific parameters of the instrument were set as follows: measurement time 120 s, sample preparation time 5 s, zero return time 10 s, gas flow rate 200 mL/min, and injection flow rate 200 mL/min. Each sample was measured in triplicate at room temperature. After averaging the data from each sensor and extracting a steady value between 115 and 120 s, the principal component analysis (PCA) was performed.

**GC-MS Analysis**

For GC-MS analysis, 1.0 g of sodium chloride was weighed accurately, the internal standard 2-methyl-3-heptanone in acetone solution (concentration range 30–40 μg/L) was added to 8 mL of UHT milk samples in a 20 mL clean and odorless headspace vial, balanced at 40°C for 20 min at a speed of above 20 rpm, and then headspace volatiles were collected 30 min after inserting 65 μm of polydimethylsiloxane/divinylbenzene fiber assembly (Supelco Analytical, Bellefonte, PA). The solid-phase–microextraction fiber was transferred to the injection port and desorbed at 250°C for 5 min in a splitless mode. The extracted volatiles from milk samples were detected by a GC-MS system with a DB-WAX MS capillary column (30 m × 0.25 mm × 0.25 mm; Agilent Technologies, Palo Alto, CA). The temperature was set as follows: 3°C/min, increased to 90°C, maintained at 90°C for 1 min, increased to 210°C at 6°C/min, and maintained at 210°C for 5 min. Helium was used as a carrier gas at a constant flow rate of 1 mL/min. The temperature of ion source and MS quadrupole detector was 250°C, and the transfer line temperature was 230°C. The solvent delay was set at 3 min and the detection was performed in a full-scan mode in an mass-to-charge ratio range of 40 to 450 amu. Data were collected using Mass Hunter version B.07.00 software (Agilent Technologies).

We used 3 different qualitative approaches. (1) Mass spectra qualitative analysis. Each compound was identified using the standard spectra provided by the National Institute of Standards and Technology (NIST11) library (Agilent Technologies). (2) Calculation of the RI: Under conditions of constant temperature increase, the retention time of n-alkanes (C7–C30) was used to derive the RI of volatile chemicals (Equation 1). The calculated RI values of the sample compounds were compared with those reported in the database, and a successful characterization was achieved when the calculated values deviated from the literature values within ±100. (3) Standard qualitative: When the peak time that the chemical spiked into the sample agreed with the total ion flow diagram of the sample, the qualitative approach was effective:

\[
RI = 100 \times \frac{[n + (RT_x - RT_n)]/(RT_{n+1} - RT_n)]}{[1]
\]

where RI is the calculated retention index; \(n\) refers to carbon number of n-alkane; \(RT_x\) is the retention time of a certain unknown volatile compound to be measured, and the subscripts of \(n\) and \(n+1\) were the carbon-atom numbers of n-alkanes before and after the appearance of the certain unknown volatile compound in GC-MS.

For quantitative analysis methods, we used the semiquantitative internal standard method: the concentration of each volatile component was evaluated by determining the concentration of the internal standard (2-methyl-3-heptanone in acetone solution) and the concentration of the component to be measured based on the ratio of the peak areas of the component to be measured and the internal standard:

\[
Cx = \frac{Ax}{Ai} \times Ci
\]

where \(Cx\) and \(Ax\) are the concentrations and chromatographic peak areas of the identified volatile compounds, and \(Ci\) and \(Ai\) indicate the concentration and chromatographic peak area of the internal standard (2-methyl-3-heptanone in acetone solution), respectively.

**Sensory Recombination Study**

The sensory reorganization studies were conducted to determine the magnitude of the measured sensory perception changes and evaluate the relevant off-flavor compounds identified following the Potts and Peterson (2018) experiment method. The panelists were present-
ed with 6 different milk samples: a nonflavor defective skim milk (control), an FFM37 sample (stored for 30 d, group E), and 4 reconstituted milk samples of skim milk with added 2-heptanone (group A), 2-nonanone (group B), 2-undecanone (group C) separately, and a combination all of compounds (at equivalent concentrations of FFM37 sample for 30 d, group D). Afterward, the panelists were instructed to sniff the 6 randomly coded samples and evaluate the degree of difference in oxidative odor. A 5-point scale suitable for the evaluation of oxidation odor of different samples was employed, and the scoring criteria were aligned with the sensory evaluation. Finally, the sensory panelists were asked to depict the flavor differences in 6 samples.

Statistics

All experiments were performed in triplicate and the data were expressed as mean ± standard deviation. The Dunnett’s multiple comparisons test, R-value, and coefficient of variation were calculated by Pearson correlation analyses using SPSS Statistics 26 (IBM, Armonk, NY). After data standardization, PCA and partial least squares regression (PLSR) analysis were performed by the Unscrambler X 10.4 (CAMO Inc., Oslo, Norway). The radar chart and Venn diagram were generated using Origin 2019 (Origin Lab Inc., Northampton, MA), and other graphs were plotted by GraphPad Prism 8.0 (GraphPad Software Inc., La Jolla, CA).

RESULTS AND DISCUSSION

Sensory Evaluation

The sensory evaluation results of FFM samples stored at 0, 4, 18, 25, 30, and 37°C for 15 and 30 d are shown in Figure 1A and 1B, and LFM samples are shown in Figure 1C and 1D. Figure 1E and 1F showed the scores of milk flavor and off-flavor under various storage conditions. The milk flavor scores of FFM and LFM samples gradually decreased and the oxidative off-flavors increased with increased storage temperature. The FFM samples received the lowest ratings for milk flavor and the highest scores for oxidative off-flavor after being stored at higher temperatures (30, 37°C) for 30 d. The UHT milk with greater fat content is more vulnerable to oxidative off-flavor impressions. Overall, UHT milk maintained better flavor characteristics at refrigerated temperature (0, 4°C) compared with storage at higher temperatures (25, 30, and 37°C). Meanwhile, the FFM milk samples (stored for 15 and 30 d) were clearly dissimilar in the aroma release classification, indicating that both storage duration and temperatures affected the quality of UHT milk and the same sensory impressions of the assessors were aligned with the dynamic pattern. As shown in Figure 2C, LFM0, LFM4, LFM18, and LFM25 were on the positive half-axis of PC1; LFM30 and LFM37 were on the negative half-axis of PC1 for LF15d; and LFM stored at 25°C and even lower temperatures had similar aroma release. Compared with LFM25, the aroma profile of FFM25 had a larger degree of variation stored at 15 d. Therefore, the higher fat content was more prone to trigger aromatic deterioration, which has been validated in the study by Ai et al. (2015). All the clusters were positioned away from the sample stored at 0°C; refrigerated storage 0°C contributes to the freshness of UHT milk (Figure 2). The PCA results based on E-nose data revealed that the E-nose is sensitive enough to detect the evaporation of volatile scents at ambient storage temperatures, variations in the volatile composition and content of the sample might produce a distinct olfactory profile (Chi et al., 2021).

E-Nose Analysis

The sensory method for determining the flavor of UHT milk is subjective and easily affected by olfactory fatigue. In such cases, the application of the E-nose is notable as it can mimic human olfaction and quickly identify the volatile flavors. The E-nose uses a simple, nonspecific sensor array and a recognition software system to satisfy the characterization and discrimination of the aroma profile of different samples (Chung et al., 2017). The effect of different natural storage temperatures (0, 4, 18, 25, 30, and 37°C) on the volatility properties was evaluated by the PCA to obtain a low-dimensional visual and adaptable representation of the data. The PCA plots were generated when the trend of the response values of 10 sensors reached a stable equilibrium in the last 115 to 120 s. All samples showed marked separations, with more than 70% contribution variation of PC1 and PC2 (Figure 2). The first dimension separated FFM0 and FFM4 from FFM18, FFM25, FFM30, and FFM37 (Figure 2A); the second dimension differentiated the FFM25, FFM30, and FFM37 on the negative, compared with the other samples when FFM were stored for 15 d (Figure 2A). Overall, the aroma release profile of FFM milk stored at refrigerated temperature (0, 4°C) and 18°C was different from that of UHT milk stored at higher temperatures (25, 30, and 37°C). Meanwhile, the FFM milk samples (stored for 15 and 30 d) were clearly dissimilar in the aroma release classification, indicating that both storage duration and temperatures affected the quality of UHT milk and the same sensory impressions of the assessors were aligned with the dynamic pattern. As shown in Figure 2C, LFM0, LFM4, LFM18, and LFM25 were on the positive half-axis of PC1; LFM30 and LFM37 were on the negative half-axis of PC1 for LF15d; and LFM stored at 25°C and even lower temperatures had similar aroma release. Compared with LFM25, the aroma profile of FFM25 had a larger degree of variation stored at 15 d. Therefore, the higher fat content was more prone to trigger aromatic deterioration, which has been validated in the study by Ai et al. (2015). All the clusters were positioned away from the sample stored at 0°C; refrigerated storage 0°C contributes to the freshness of UHT milk (Figure 2). The PCA results based on E-nose data revealed that the E-nose is sensitive enough to detect the evaporation of volatile scents at ambient storage temperatures, variations in the volatile composition and content of the sample might produce a distinct olfactory profile (Chi et al., 2021).
Characterization and Quantification of Volatile Compounds

The qualitative and quantitative results of the volatile compounds in the FFM and LFM samples (stored at 0, 4, 18, 25, 30, and 37°C for 15 and 30 d) are shown in Supplemental Tables S1–S3 (https://osf.io/3qfv2/?view_only=63ac1b1b1bcc04f229dfc9a6d7f71234; Xi, 2023). A total of 48 and 47 volatile compounds were identified in the FFM and LFM samples, respectively, including ketones, acids, esters, aldehydes, alcohols, sulfides, phenols, alkanes, and other compounds. The concentration of total ketones exhibited a dynamic elevation with increasing storage temperature, accounting for the most percentage and maximum amount in both FFM and LFM samples (Figure 3). The highest concentrations of ketones in the FFM37 samples were 90.82% and 88.18% stored for 15 and 30 d, respectively. Meanwhile, the methyl ketones of odd carbon chains (C5, 7, 9, 11, 13, and 15; i.e., 2-pentanone, 2-heptanone, 2-nonanone, 2-undecanone, 2-tridecanone, and 2-pentadecanone) were detected, which reached the maximum percentage in FFM37 samples for 30 d (Figure 3). The UFA and their subsequent breakdown products from lipid oxidation might also yield methyl ketones. Hence, methyl ketones can be considered the tertiary products of lipid oxidation and the secondary degradation products of lipid oxidation can react in different ways to form methyl ketones (Grebenteuch et al., 2021). In a previous study on oxidative odors of dairy products, 2-heptanone and 2-nonanone were listed as the typical volatile components responsible for oxidative odors of soaps and dyestuff oxidized odors in dairy products (Cadwallader and Singh., 2009; Li et al., 2013). The total ketone content of FFM37 was almost 4 times higher than in LFM37 stored for 30 d (Figure 4; Supplemental Tables S2 and S3), probably because FFM samples produced more fat oxidation products. The C6, 8, 10, and 12 fatty acids are the precursors to methyl ketones (such as C5, 7, 9, and 11), which are essential for the generation of C6, 8, 9, and 10 aldehydes during thermally enhanced lipid self-oxidation (Jensen et al., 2015). Benzaldehyde was the sole Strecker aldehyde and reached the maximum concentration of 2.53 μg/L and 2.32 μg/L in the FFM37 and LFM37 samples after 30 d storage, respectively. The variation in benzaldehyde concentration indicated that the 37°C storage temperature was optimal for the onset of the Maillard reaction. Staleness-inducing Strecker aldehydes and other chemicals produced are stimulated by an increase...
in free amines at higher temperatures during the Maillard reaction (Nielsen et al., 2017). Notably, the higher storage temperature increased the formation of Strecker aldehydes and methyl ketones, a phenomenon that was in accordance with the conclusions of Bottiroli et al. (2021).

Four distinct lactones were identified: 5-octanolide, 5-decanolide, 4-dodecanolide, and 5-dodecanolide. The concentration of 5-decanolide and 5-dodecanolide reduced to varying degrees with increased storage temperature (Figure 4). The compound 5-decanolide smells and tastes similar to greasy peaches and contributes to the butter aroma in dairy products (Bendall, 2001). The lactones provide a richer, fuller fragrance, contributing to the overall flavor when stored at lower temperatures.

**Biplot by PCA and PLSR**

The biplot is a very useful method for representing multivariate data graphically (Chi et al., 2021). As shown in Figure 5A, the FFM25, FFM30, and FFM37 were all clustered together in the second quadrant, sharing a positive correlation with ketones. In the first main component of Figure 5B, FFM30 and FFM37 grouped with ketones and were clearly separated from FFM0, FFM4, FFM18, and FFM25. This phenomenon demonstrates that increased storage temperature and storage duration ketones are the main contributor to flavor deterioration of FFM samples. Higher storage temperatures (30 and 37°C) triggered a higher degree of fat oxidation and lipolysis compared with lower storage temperatures (0, 4, and 18°C), which was consistent with the findings in Lu et al. (2019). All clusters were separated from the samples LFM30 and LFM37 stored for 30 d, and both of them were associated with aldehydes, alcohols, and ketones on PCA (Figure 5). In contrast, the PCA categorization results of GC-MS and E-nose did not completely correspond to each other, which might be attributed to different contributions of various volatiles.

Currently, PLSR is used for modeling the correlation between 2 data matrixes (X and Y) using the latent variables (Seisonen et al., 2016). In this study, the concentration of volatile compounds of each sub-
stance identified in the FFM and LFM samples were selected as X variables and the scores of sensory attributes (milk flavor and off-flavor) as Y variables. All data were standardized for analysis. The 2 ellipses represented 50% and 100% variation, respectively (Figure 5E–H). A positive correlation between ketones and oxidized odor was observed on the scale from 50% to 100%. Except ketones, other compounds can improve the overall flavor of milk. Milk flavor is a combination and interaction of different volatile components (Zhang et al., 2016).

Correlation Between Sensory Attributes and Volatile Component

The correlations between sensory attributes and individual volatile organic compounds were analyzed by Pearson correlation further (Figure 6; Supplemental Figures S1–S4; https://osf.io/3qfv2/?view_only=63ac1b11bec04f229dfcffe6d7d71234; Xi, 2023). The results showed that in the FFM samples, most methyl ketones (i.e., 2-heptanone, 2-octanone, 2-nonanone, 2-undecanone, and 2-tridecanone) were significantly positively correlated with the oxidative odor after 30 d storage ($P < 0.05$). A Venn diagram can visually present the intersection between multiple samples and quickly capture the similarities and differences between samples to narrow the target (Feng et al., 2021). In the Venn diagram (Figure 7), the overlaps of the 3 circles represents the compounds co-associated with specific sensations in the corresponding FFM and LFM samples. The 2-heptanone and 2-undecanone were positively associated with the oxidative off-flavors ($P < 0.05$). No remarkable correlation ($P > 0.05$) was seen between oxidative off-flavors and methyl ketones in the LFM samples due to lower concentrations of oxidation products, which showed less variation in the LFM samples at different storage temperatures. The compound 2-ethylhexanol was significantly positively correlated with creaminess in the FF15d, FF30d, and LF30d samples (Figure 7B). Moreover, 5-dodecalactone was significantly ($P < 0.05$) positively correlated with milk flavor in the FFM sample stored for 30 d and LFM stored for 15 d (Figures 6 and 7A). The 5-dodeca-
lactone induces flavor changes in processed and stored dairy products due to its characteristic aroma and odor properties (Mounchili et al., 2005).

The odor activity value (OAV) refers to the key aroma active compounds influencing the flavor quality. Supplemental Table S4 (https://osf.io/3qfv2/?view_only=63ac1b11bcc04f229dfcffa6d7d71234; Xi, 2023) shows the OAV of compounds associated with 3 flavor attributes (milk flavor, creaminess, and oxidized odor). The OAV was calculated as the ratio of the concentration and corresponding flavor threshold value. If the OAV of a compound is more than 1.0, the compound may add flavor; if the OAV is between 0.5 and 1.0, the compound can smell slightly (Qian and Reineccius, 2003). The 2-heptanone, 2-nonanone, and 2-undecanone OAV was >1 (Supplemental Table S3) and triggered a change in flavor in the FFM37 sample stored for 30 d.

**Sensory Recombination Study**

Compared with the control group (Figure 8), the skim milk matrix separately spiked with 2-heptanone (group A) and 2-undecanone (at concentrations equivalent to the FFM37 sample when stored for 30 d, group...
Figure 5. Influence plot of full-fat UHT milk (FFM) and low-fat UHT milk (LFM) data with a 4-component principal component analysis (PCA) model. (A, B) Principle component analysis (PCA) score plots of volatile compound in FFM samples at different natural storage temperatures (0, 4, 18, 25, 30, or 37°C). (C, D) PCA score plots of volatile compound in LFM samples at different natural temperatures stored for 15 and 30 d. (E–H) Partial least squares regression (PLSR) models, the concentration of volatile compounds of each substance identified in the FFM and LFM samples were selected as X variables and the scores of sensory attributes as Y variables. FF_{15d} and FF_{30d}: FFM samples stored for 15 and 30 d, respectively; LF_{15d} and LF_{30d}: LFM samples stored for 15 and 30 d, respectively.
Figure 6. Results of GC-MS and sensory analysis of flavor compounds in UHT milk. Histogram is the average concentration of flavor compounds in UHT milk at different storage conditions (0, 4, 18, 25, 30, or 37°C), and the error bars represent their respective SE values. Scatter diagram is the linear regression analysis of quantitation of flavor compounds and the value of sensory evaluation. Red is milk flavor, purple is creamy, and green is oxidation odor. \( P < 0.05 \) indicates a significant positive correlation. Figure 6A, panels a–i and j–r represent ketones of the volatile flavor compounds produced by full-fat UHT milk (FFM) samples stored for 15 and 30 d, respectively (FF15d and FF30d). Figure 6B, panels a–i and j–r represent ketones of the volatile flavor compounds produced by low-fat UHT milk (LFM) samples stored for 15 and 30 d, respectively (LF15d and LF30d).
C) showed an increased score (score A = 2.2, score C = 2.6, \( P > 0.05 \)). In contrast, all 3 chemicals were added to the skim milk simultaneously (group D), the oxidative odor score was significantly higher than the control (score = 3.6, \( P < 0.05 \)). The panelists defined group D as “not fresh,” “oxidized odor,” and “old.” Descriptors such as old and oxidized odor were used to describe the FFM37 sample stored for 30 d (group E). Moreover, the samples in group D were alleged to have a bitter and strong pungent flavor. Overall, the combined effect of 2-heptanone, 2-nonanone, and 2-undecanone significantly contributed to oxidative odor.

**CONCLUSIONS**

Storage duration and temperature significantly affected lipid oxidation and lipolysis of FFM product during shelf life. The concentration of ketones, especially methyl ketones such as 2-heptanone, 2-nonanone, and 2-undecanone, increased in FFM37 stored for 30 d (OAV > 1). The increased storage temperatures accelerate the catalytic performance of fatty acids oxidation. Conversely, cooler temperature (0 and 4°C) is beneficial to the flavor quality of UHT milk. Moreover, the oxidized off-flavor attributes were associated with the increased concentrations of 2-heptanone, 2-nonanone, and 2-undecanone. The oxidized off-flavor was apparent in the FFM37 sample. The combined results of sensory reorganization studies revealed that 2-heptanone, 2-nonanone, and 2-undecanone significantly contributed to oxidative odor.

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8549


ORCIDS

Yanmei Xi https://orcid.org/0000-0001-7603-0327
Yiwei Shao https://orcid.org/0000-0003-2969-9604
Ruirui Liu https://orcid.org/0000-0003-5617-5299
Fuhang Song https://orcid.org/0000-0002-9162-3355
Nasi Ai https://orcid.org/0000-0003-3550-1668