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Changes of the volatile compounds and odors in one-stage and three-stage infant formulas during their secondary shelf-life

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ABSTRACT

The odor of infant formula changes due to alterations in its volatile composition during the shelf life. However, there is currently a lack of research on whether the odor changes in infant formula during the secondary shelf life, which refers to the period of repeated opening and usage in daily life. This study used headspace solid-phase microextraction (HS-SPME) coupled with gas chromatography-electrostatic orbitrap high-resolution mass spectrometry (GC-Orbitrap-MS) to investigate the volatile composition changes in one-stage and three-stage infant formulas during different stages (0 day, 3 days, and 7 days during the secondary shelf-life, i.e. simulated daily use). A total of 32 volatiles were identified, including nine aldehydes, seven ketones, four alcohols, three furans, two sulfur compounds, two esters, and five terpenoids. Of these, 16 compounds changed significantly in one-stage samples and 23 compounds in three-stage samples within 7 days of the secondary shelf-life. Further the odor of the three-stage infant formula samples was found changed substantially after 3 days of simulated use by using the triangle test. This study highlighted the considerable alterations in volatile compound composition and sensory changes during the simulated daily use and provided valuable insights for consumers in selecting and using infant formula products, as well as a new perspective for enterprises to improve the sensory quality of their products.

1. Introduction

Infant formula is the breastfeeding substitute for infants and can be sourced from goat's or cow's milk, processed by adding vitamins, minerals and other excipients to the raw material (Lönnerdal, 2014; Cama-Moncunill et al., 2017). The currently available commercial infant formula is a fourth-generation product using advanced standards, i.e., segmented formula. It is typically diverse regarding the type and quantity of nutrients and can satisfy the nutritional needs of different age groups via the subsection of each product, such as 0–6 months for stage one, 6–12 months for stage two, and 12–36 months for stage three. In recent years, the demand for infant formula has grown considerably, and the infant formula industry is developing rapidly. The sensory quality of infant formula, especially its odor, could affect consumers' preferences and even purchasing willingness.

Previous studies have shown that aldehydes, ketones, alcohols, lactones, furans, sulfides, and terpenoids represented the main volatile components in infant formula and are influenced by factors such as raw materials, processing methods, and storage conditions (Tang et al., 2022a; Zhang et al., 2022; Clarke et al., 2020; Borad et al., 2017). The processing (e.g., pasteurization, spray drying, and sterilization) of infant formula might destabilize the raw materials and final product. Furthermore, additional carbohydrates, proteins, or polyunsaturated fatty acids (PUFA) were added to maximize breast milk simulation, which could increase lipid oxidation (LO) (Lopez et al., 2015; MacLean et al., 2010), consequently affecting the shelf-life of the product (Wang et al., 2020). LO and the Maillard reaction were found the main causes of spoilage during the processing and storage of dairy powders (Park and Drake, 2014), which can lead to a loss of nutritional value and the formation of furans and furan derivatives (Nunes et al., 2019; Sabater et al.,

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2018). The aldehydes, ketones, and alcohols formed by the oxidation of unsaturated fatty acids (FA) could eventually lead to off-flavors in milk powders (Kilcawley et al., 2018) and nutritional loss and affect product stability during shelf life.

The sensory changes of milk powder after the package opened is rarely studied. It is highly likely that changes due to changes in atmospheric composition, increased oxygen and moisture, temperature changes, and loss of aseptic conditions. Therefore, the duration for which milk powder is organoleptically acceptable to consumers for daily use (i.e., the secondary shelf life) is also an important factor to consider. Currently, limited studies are available regarding the compositional changes during the secondary shelf life of milk powders. Chávez-Servín et al. (2015) found the slight increases of furfural compounds in commercial powdered infant formula after opening the packet for 70 days, which was attributed to the Maillard reaction. Aalaei et al. (2017) investigated the effect of different drying techniques and storage conditions on the formation of carboxymethyl lysine (CLM) as an advanced glycation end product (AGE) in skim milk powders, which demonstrated that AGEs might be formed during storage of skim milk powders after opening the packages by the consumers. Condurso et al. (2020) assessed the stability of infant formula and lactose-free milk powder during their secondary shelf-life and found that a large number of volatile compounds were identified and significant statistically differences resulted during second shelf-life that were correlated to the infant formula composition.

Numerous techniques used for the extraction and separation of volatile components have been reported, including static headspace (Maeztu et al., 2001), dynamic headspace (Choe and Min, 2006), direct thermal desorption (Cavalli et al., 2003), solvent-assisted flavor evaporation (SAFE), stir bar sorptive extraction (SBSE) (Tang et al., 2022a), and solid-phase microextraction (SPME) (Marsili, 1999). Headspace SPME (HS-SPME) is a commonly used technique for volatile compound extraction and enrichment, combined with gas chromatography-mass spectrometry (GC-MS) for identification and quantification. HS-SPME enriches volatiles in the upper part of the headspace vial via temperature control or magnetic stirring and then sorbs them through a fiber coating (Xue-lu et al., 2017; Mahdie Kamalabadi et al., 2015). This method is simple and minimizes sample handling. The gas chromatography-quadrupole mass spectrometry was commonly used but has limitations in infant formula powder because many volatiles were found in trace amounts (Wang et al., 2019). GC-Orbitrap-MS is a relatively new technique presenting advantages such as high resolution, sensitivity, and accuracy. It exhibits significant potential for analyzing and identifying trace compounds in foods (Liu et al., 2022).

The triangle test is usually used during sensory evaluation to determine the differences between two samples (Zhang et al., 2006). The method is practical, accurate, and reliable (Zhang et al., 2010) and is suitable for the sensory evaluation of dairy products (Lim et al., 2022). For example, Wei et al. (2011) used the triangle test to study the effect of four iron fortification agents on the sensory properties of milk powder, while Lim et al. (2022) examined the sensory differences between milk treated at different pressures and Bottiroli et al. (2020) assessed the impact of freezing on the sensory properties of ultra-high temperature hydrolyzed lactose milk.

This study used HS-SPME coupled with GC-Orbitrap-MS to analyze the evolution of volatile components in one- and three stage infant formula samples under the simulated daily use conditions, and investigated the sensory differences among the samples using the triangle test. This research provides a reference for establishing the sensory secondary shelf-life of infant formula in daily use conditions for consumers.

2. Materials and methods

2.1. Samples

Two segmented infant formula samples of the same brand, including one one-stage sample and one three-stage sample, were purchased at the same time from excellent supplier with strict quality control and used in this experiment. The samples are stored in a dry and dark environment before purchase. After opening the packet, the sampling cycle was performed according to the simulated infant milk powder feeding schedule. Sampling occurred 7 times daily at 2–3 h intervals. These samples used for the subsequent analysis were collected at 0 day, 3 days, and 7 days of simulated daily use, respectively. For each sample, about 12.9 g (three measuring spoons) were used for volatile component analysis, while those collected for sensory analysis (about 77.4 g) were stored at 4 °C in sealed oxygen insulation and dehumidification conditions. The detailed sample information is listed in Table 1.

2.2. Chemical and reagents

The C7-C40 n-alkane chemical reagents were obtained from Sigma-Aldrich (St. Louis, MO, USA). The internal standard (4-methyl-2-pentanol) with a purity of ≥98 % was purchased from Shanghai Ampoule Experimental Technology Co. Ltd. The standards used for qualitative as shown: 3-methylbutanal (≥99 %), pentanal (≥98 %), hexanal (≥99 %), heptanal (≥98 %), (E)-2-heptenal (≥95 %), nonanal (≥96 %), 2-butanone (≥98 %), 2-pentanone (≥98 %), methyl isobutyl ketone (≥99.5 %), 2-heptanone (≥98 %), 1-penten-3-ol (≥98 %), 2-ethylfuran (≥98 %), 2-pentylfuran (≥98 %), and β-Ocimene (≥90 %) were supplied by Shanghai Macklin Biochemical Co., Ltd., dimethyl disulfide (≥98 %), dimethyl trisulfide (≥98 %), β-Pinene (≥95 %) and (E)-2-hexenal (≥98 %) were obtained from Shanghai Aladdin Bio-Chem Technology Co., Ltd., and γ-Terpinene (≥95 %), α-Thujene (≥95 %) were supplied by Shanghai Acmec Biochemical Co., Ltd. (Table A.2)

2.3. HS-SPME/GC-Orbitrap-MS analysis

2.3.1. HS-SPME conditions

Automated HS-SPME was performed with a TriPlus RSH autosampler (Thermo Fisher Scientific, Bremerhaven, Germany). The extraction conditions were established according to a published method developed by Clarke et al. (2019). A powder sample (2.4 g) was mixed with 10 μL of 1.0018 mg/L 4-methyl-2-pentanol (internal standard) and 2.5 mL dH_2O in a 25 mL vial. The vial was capped with a PTFE-silicon septum and equilibrated at 43 °C for 10 min under agitation. Then, a DVB/CAR/PDMS fiber was inserted into the headspace of the vial to adsorb volatiles at 43 °C for 45 min. Afterwards, the fiber was immediately inserted into the GC injection port at 260 °C for 8 min to desorb the volatiles.

2.3.2. GC-Orbitrap-MS conditions

A Thermo Scientific Trace 1300 gas chromatograph coupled with a Thermo Scientific Q-Exactive Orbitrap mass spectrometer (GC-Orbitrap MS, Thermo Scientific, Bremen, Germany) was used to analyze the volatile compounds in the infant formula samples. A DB-5 capillary column (30 m × 0.25 mm × 0.25 μm, J&W Scientific, Folsom, CA, USA) was used to separate the volatiles under a carrier gas (helium, 99.999 % purity) at a flow rate of 1.2 mL/min. The chromatographic conditions were modified based on those described by Park et al. (2016). The oven temperature program was as follows: 40 °C held for 3 min, then

Table 1
The basic information of the experimental samples.

Sample name	Sample segment type	Production date	Sampling days	Product specification (g)
S1-0D	1	2022.12.18	0	700
S1-3D	1	2022.12.18	3	700
S1-7D	1	2022.12.18	7	700
S3-0D	3	2022.12.19	0	700
S3-3D	3	2022.12.19	3	700
S3-7D	3	2022.12.19	7	700

Table 2
The qualitative results of the volatile compounds in the infant formula samples.

Category	Number	Compounds	CAS	m/z	Calculated RI	NIST RI	Qualitative sources[a]	Fragrance description[b]
Aldehydes	AL-1	3-Methylbutanal	590-86-3	41.038342	636	650	MS, RI, STD	Chocolate, fatty
	AL-2	Pentanal	110-62-3	44.025551	669	700	MS, RI, STD	Coffee, nutty
	AL-3	Hexanal	66-25-1	41.038342	753	797	MS, RI, STD	Grass, fatty
	AL-4	*(E)*-2-Hexenal	6728-26-3	41.038342	797	854	MS, RI, STD	Fatty, green
	AL-5	Heptanal	111-71-7	41.038342	834	901	MS, RI, STD	Fatty, green
	AL-6	*(E)*-2-Heptenal	18829-55-5	41.038342	877	951	MS, RI, STD	Green, vegetables
	AL-7	Nonanal	124-19-6	41.038342	1004	1099	MS, RI, STD	Orange peel, fatty
	AL-8	*(E,E)*-2,4-Heptadienal	4313-03-5	81.033463	1027	1115	MS, RI	Fatty, green
	AL-9	*(E,E)*-2,4-Decadienal	25152-84-5	81.033463	1220	1277	MS, RI	Fatty, cucumber
Ketones	K-1	2-Butanone	78-93-3	43.017689	600	600	MS, RI, STD	Fruity, camphoraceous
	K-2	2-Pentanone	107-87-9	43.017689	659	668	MS, RI, STD	Sweet, fruity
	K-3	Methyl Isobutyl Ketone	108-10-1	43.017689	707	743	MS, RI, STD	Fruity, green
	K-4	2-Heptanone	110-43-0	43.017689	825	892	MS, RI, STD	Fruity, sweet
	K-5	3-Octen-2-one	1669-44-9	43.017689	945	1036	MS, RI	Earthy, mushroom
	K-6	2,5-Octanedione	3214-41-3	43.054073	964	983	MS, RI	Buttery, fatty
	K-7	3,5-Octadiene 2-one	38284-27-4	95.049149	973	1052	MS, RI	Fatty, mushroom
Alcohols	HA-1	Acetone alcohol	116-09-6	43.017689	571	666	MS, RI	Sweet, caramelized
	HA-2	Isobutyl alcohol	78-83-1	41.038342	572	625	MS, RI	Wine, leather
	HA-3	1-Penten-3-ol	616-25-1	57.033497	657	683	MS, RI, STD	Green
	HA-4	3-Hexen-1-ol	544-12-7	41.038342	914	856	MS, RI	Green, earthy
Furans	F-1	2-Ethylfuran	3208-16-0	81.033463	670	689	MS, RI, STD	Beany, fishy, cocoa
	F-2	2-Pentylfuran	3777-69-3	81.033463	903	999	MS, RI, STD	Beany, fishy, metallic
	F-3	2-n-Butyl furan	4466-24-4	81.033463	923	893	MS, RI	Fruity, spicy
Sulfides	S-1	Dimethyl disulfide	624-92-0	93.990517	711	738	MS, RI, STD	Sulfurous, oniony
	S-2	Dimethyl trisulfide	3658-80-8	125.962601	888	969	MS, RI, STD	Sulfurous, oniony
Esters	E-1	Ethyl acetate	141-78-6	43.017689	608	600	MS, RI	Fruity, sweet
	E-2	Allyl propionate	2408-20-0	57.033508	669	DB-1 776	MS, RI	Tropical fruits
Terpenoids	T-1	α-Thujene	2867-05-2	91.054214	859	910	MS, RI, STD	Herbal, woody
	T-2	*β*-Pinene	127-91-3	93.069862	903	971	MS, RI, STD	Woody, resin
	T-3	*(R)* -*α*-pinene	7785-70-8	91.054214	920	975	MS, RI	Woody, fresh
	T-4	*β*-Ocimene	13877-91-3	67.054192	938	1023	MS, RI, STD	Herbal, citrus
	T-5	*γ*-Terpinene	99-85-4	91.054222	963	1059	MS, RI, STD	Fresh, herbal

[a] MS is characterization based on matching mass spectral information; RI is characterization based on matching the actual RI to the NIST RI; O is the compound identified during GC-O; STD is characterization based on matching to a library of standard spectra.
[b] Volatile compound aroma descriptions from the Aroma Search website http://www.thegoodscentscompany.com.

increased to 90 °C at 10 °C/min, afterwards ramped from 90 °C to 200 °C at 5 °C/min, finally increased to 250 °C at 20 °C/min and held for 5 min. Positive ion electron ionization was set at 70 eV in the Orbitrap MS, and all scan mass from m/z 30–300 were recorded. All the samples were analyzed in triplicate.

2.3.3. Qualitative and quantification of volatile compounds

The data were collected and analyzed using Xcalibur (Thermo Fisher Scientific, Les Ulis, France) with a processing setup, Quan browser and Qual browser (Hu et al., 2022). The retention indices (RI) were determined using a C7-C40 n-alkane series (Supelco, Bellefonte, PA, USA) under the same chromatographic conditions. The deconvoluted mass spectral information was first matched to compounds in the established standards library. Those compounds that could not be determined were then matched to the NIST20 library. The calculated RI values of unknown compounds were compared to the RI values searched from NIST20 library to identify these compounds. Absolute RI difference values less than 100 were retained (Li et al., 2022). The quantitative results of the volatile compounds were expressed as the peak compound area to 4-methyl-2-pentanol (internal standard) ratio (Lin et al., 2021).

2.4. Sensory evaluation

The infant formula samples were prepared according to the product instructions. The weighed milk powder was added to warm water at 45–50 °C according to the preparation ratio specified in the package. Samples were then dispensed into 20-mL PET bottles (food-grade) and placed on a thermostatic heating pad at 45 °C for more than 40 min before sensory evaluation.

The triangle test was performed by 24 trained panelists, which included 18 females and six males aged between 20 and 25 years, from Beijing Forestry University (Beijing, China). Each panelist had practiced infant formula sensory evaluation skills for at least four months. In addition, each panelist was familiar with the sensory evaluation methods to ensure that they could implement the triangle test correctly. The sensory evaluation was carried out in a laboratory that complied with international standards. Here, 20 mL infant formula samples were presented in 30 mL disposable tasting cups. All samples were randomly coded with three digits and presented to the evaluator as triad samples, two of which were samples on 0 d of opening the package, while the other represented 3 d or 7 d of simulated daily use. To minimize experimental error, the three samples had to have an equal chance of occurring in the testing order. According to GB/T12311-2012, each panelist was asked to select one sample that differed from the other two. The evaluation form is shown in Table A.1. The panelists were required to take a break for 30 s between the different samples and 10 min between the different groups to minimize fatigue.

2.5. Statistical analysis of data

One-way analysis of variance (ANOVA) was used to compare the differences between the volatile compounds of the different samples using Turkey's multiple range test under R (3.6.3). A cluster heatmap analysis was performed using a web tool (https://hiplot-academic.com). The evaluation results of the panelists in the triangle test were calculated to compare the significant differences between different samples according to the triangle test table, after which a barplot was created using EXCEL 2019.

3. Results and discussion

3.1. Identification of volatile compounds based on GC-Orbitrap-MS

GC-Orbitrap-MS is a type of high-resolution mass analyzer. The ion fragments obtained can be accurate to five places after the decimal separator, which can exclude the influence of other non-target compounds on the qualitative results, thus making the qualitative results more accurate. Considering the characteristics of the GC-Orbitrap-MS, we collected 20 already-reported volatile compounds in infant formula powder beforehand including 7 aldehydes, 4 ketones, 1 alcohol, 4 terpenes, 2 furans, and 2 sulfides and obtained their high-resolution mass spectra (Fig. 1 & Figure A.1). The high-resolution raw data of infant formula samples were deconvoluted using the software TraceFinder Software (Thermo Fisher Scientific, Les Ulis, France), and then identified by comparing with the ion fragments in our high-resolution spectrum library and combining with the retention index; later, we will further identify the peaks that have not been identified in the spectrum after deconvolution by matching the mass spectrum with NIST spectrum library and retaining retention indices to qualitatively analyze, and a total of 12 new compounds were found (Table 2).

Comparing the high-resolution mass spectrometry obtained through GC-Orbitrap-MS in this study with the low-resolution mass spectrometry obtained through GC-quadrupole-MS in the NIST library, it was not difficult to notice that, there were significant differences between the two kinds of spectra for certain compounds. As shown in Fig. 1, the fragment with the highest abundance of aldehyde compounds such as hexanal in the high-resolution mass spectrometry was m/z 41.03835 (Fig. 1A), while the fragment with the highest abundance in the low-resolution mass spectrometry is m/z 44 (Fig. 1B). At the same time, the abundance of m/z 56 in the high-resolution and low-resolution mass spectrometry was also different. Similarly, the fragment with the highest abundance in the high-resolution mass spectrometry of heptanal was m/z 41.03835 (Fig. 1C), while the fragment with the highest abundance in the low-resolution mass spectrometry was m/z 70 (Fig. 1D). There were also significant differences in the abundance of other ion fragments. The fragments with higher abundance of (E) -2-hexenal in high-resolution mass spectrometry were m/z 39.02277, m/z 83.04909, and m/z 69.03348 (Fig. 1E), while in low-resolution mass spectrometry, they were m/z 41, m/z 42, and m/z 83 (Fig. 1F). The fragment with the highest abundance of sulfide, such as dimethyl trisulfide, in high-resolution mass spectrometry was m/z 91.05421 (Fig. 1G), while the fragment with the highest abundance in low-resolution mass spectrometry was m/z 126 (Fig. 1H). The fragments with the highest abundance in high and low-resolution mass spectrometry of terpene compounds such as β-ocimene were m/z 67.05424 (Fig. 1I) and m/z 93 (Fig. 1J), respectively. The phenomenon of differences between high and low-resolution mass spectrometry was consistent with previous research results in literature (Belarbi et al., 2021). The high-resolution mass spectrum and low-resolution mass spectrum of other 15 compounds were shown in Figure A.1 and it should be noted that for the other 15 compounds, the two spectra for each compound were found similar.

A total of 32 volatile compounds were identified, including nine aldehydes, seven ketones, four alcohols, three furans, two sulfides, two esters, and five terpenoids (Table 2) using SPME-GC-Orbitrap-MS. For each volatile compound, the CAS number, m/z, RI, qualitative sources, together with aroma description were detailed in Table 2.

3.2. Quantitative analysis

Table 3 presents the mean peak area ratios of all volatile compounds in the one-stage and three-stage samples at 0 d, 3 d, and 7 d of simulated daily use. For the one-stage samples, 16 compounds showed significant differences ($p < 0.05$) among samples at 0 d, 3 d, and 7 d of simulated daily use, including three aldehydes, four ketones, one alcohol, one furan, two sulfides, and five terpenoids. Moreover, 23 compounds displayed substantial differences ($p < 0.05$) among the three-stage samples, including six aldehydes, three ketones, two alcohols, three furans, two sulfides, two esters, and five terpenoids.

3.2.1. Aldehydes

Aldehydes are the most abundant volatile compounds in infant formulae and mainly result from unsaturated FA oxidation (Condurso et al., 2020). After 3 days of simulated daily use, the aldehydes displaying significantly increased in the one-stage samples (S1-3D) including nonanal (orange peel, fatty) and (E,E)-2,4-decadienal (fatty, cucumber), with nonanal increasing about three times and (E,E) −2,4-decadienal was not detected in S1-0D; while those in the three-stage samples (S3-3D) were pentanal (coffee, nutty), hexanal (grassy, fatty), heptanal (fatty, green), nonanal (orange peel, fatty), (E,E)-2,4-heptadienal (fatty, green), (E,E)-2,4-decadienal (fatty, cucumber), with heptanal increasing approximately 10 times, (E,E)-2,4-heptadienal and (E,E) −2,4-decadienal were not detected in S3-0D. After 7 days of simulated daily use, the peak area ratios of glutaraldehyde, nonanal, and (E,E)-2,4-decadienal in the one-stage samples (S1-7D) and pentanal, heptanal, nonanal, (E,E)-2,4-heptadienal, and (E,E)-2,4-decadienal in three-stage samples (S3-7D) were all significantly reduced, among which heptanal reduced about 10 times, (E,E)-2,4-heptadienal and (E,E) −2,4-decadienal were not detected.

3.2.2. Ketones

Studies have shown that ketones are important compounds related to LO and are vital indicators of infant formula stability (Li et al., 2013). After 3 days of simulated daily use, the peak area ratios of the methyl isobutyl ketone in the one-stage samples (S1-3D) decreased significantly and were mostly described as “fruity”. The peak area ratios of the 2-butanone in the three-stage samples (S3-3D) decreased significantly and were mostly described as “fruity”, while the 3-octen-2-one (earthy, mushroom) and 3,5-octadiene-2-one (fatty, mushroom) increased significantly, which were not detected in S3-0D. After 7 days of simulated daily use, the peak area ratios of the 2-butanone and 2-heptanone (fruity, sweet) decreased substantially in the one-stage samples (S1-7D), while a substantial decrease was evident in the methyl isobutyl ketone and 2,5-octanedione (buttery, fatty) levels. Only the peak area ratio of 3,5-octanedien-2-one decreased significantly in the three-stage samples (S3-7D), which was not detected.

3.2.3. Alcohols

Studies have shown that alcohols such as 1-penten-3-ol are oxidation products in infant formula (Clarke et al., 2020). After 3 days of simulated daily use, the peak area ratio of 3-hexen-1-ol increased significantly in both the one and three-stage samples, with it increasing about three times in S1-3D and four times in S3-3D, which was mostly described as “greenish” and “earthy”. While 1-penten-3-ol decreased considerably in the three-stage samples (S1-3D) and was mostly described as “green” and “radish”. After 7 days of simulated daily use, the peak area ratio of 3-hexen-1-ol was significantly reduced in both the one and three-stage samples (S1-7D and S3-7D), while 1-penten-3-ol was substantially higher in the three-stage samples (S3-7D).

3.2.4. Furans

Furans may be products of strecker, sugar, and protein degradation (Tang et al., 2022a). After 3 days of simulated daily use, the peak area ratios of 2-pentylfuran increased significantly in both the one and three-stage samples, with it increasing more than ten times in S1-3D and more than six times in S3-3D, which was mostly described as “beany” and “metallic”. While the peak area ratios of 2-n-butylfuran were substantially higher in the three-stage samples (S3-3D), and it was not detected in S3-0D, which was mostly described as “fruity”. After 7 days of simulated daily use, the peak area ratio of 2-pentylfuran decreased significantly in both the one and three-stage samples, with it decreasing

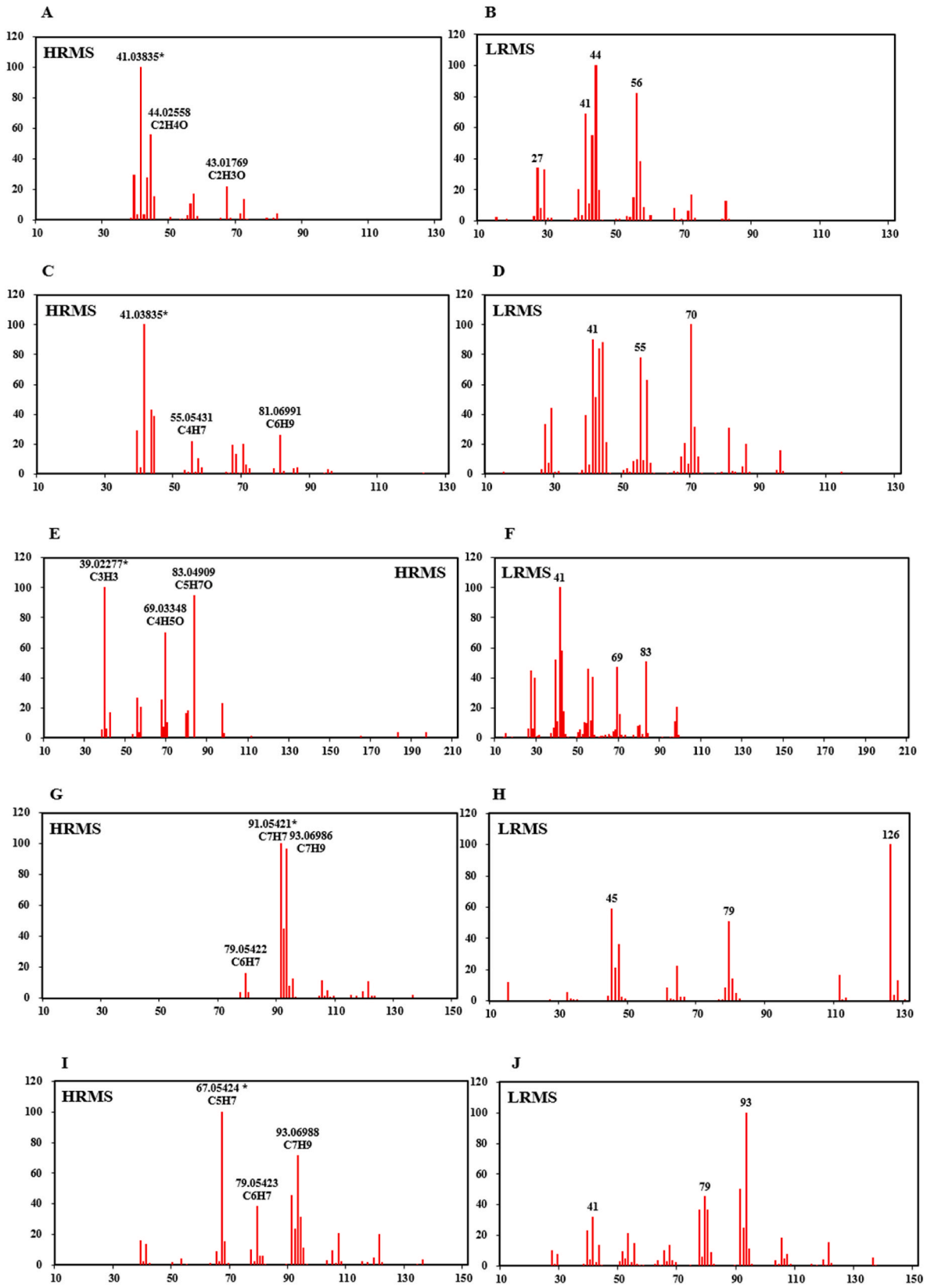

Fig. 1. The high-resolution mass spectrum and low-resolution mass spectrum of hexanal (A, HRMS; B, LRMS), heptanal (C, HRMS; D, LRMS), (e) -2-hexenal (E, HRMS; F, LRMS), dimethyl trisulfide (G, HRMS; H, LRMS) and β-ocimene (I, HRMS; J, LRMS). The low-resolution mass spectrum was cited from NIST library. The scan mass of high-resolution mass spectrum was recorded from m/z 30 to 300.

Table 3
The quantitative analysis of the volatile compounds in the infant formula samples on different days of uncovering.

Serial number	Compound	S1-0D	S1-3D	S1-7D	P	S3-0D	S3-3D	S3-7D	P
AL-1	3-Methylbutanal	5.72E-03 ± 2.30E-04	6.90E-03 ± 9.20E-04	6.20E-03 ± 6.60E-04	NS	1.88E-03 ± 1.80E-04	1.75E-03 ± 4.00E-04	2.03E-03 ± 2.40E-04	NS
AL-2	Pentanal	7.19E-02 ± 1.13E-02 [a]	8.58E-02 ± 3.39E-03 [a]	5.24E-02 ± 6.71E-03 [b]	**	5.59E-02 ± 3.10E-03 [b]	9.58E-02 ± 1.29E-02 [a]	4.98E-02 ± 3.84E-03 [b]	**
AL-3	Hexanal	3.39E-01 ± 2.07E-01	3.69E-01 ± 1.60E-02	3.13E-01 ± 5.63E-02	NS	2.90E-01 ± 2.41E-02 [b]	4.68E-01 ± 7.05E-02 [a]	4.27E-01 ± 5.03E-02 [a]	*
AL-4	(E)-2-Hexenal	ND	ND	ND	–	3.55E-03 ± 3.60E-04	6.98E-03 ± 2.71E-03	7.61E-03 ± 1.06E-03	NS
AL-5	Heptanal	9.28E-03 ± 7.20E-04	1.12E-02 ± 1.70E-03	1.07E-02 ± 2.32E-03	NS	1.27E-02 ± 2.21E-03 [b]	1.30E-01 ± 2.66E-02 [a]	1.25E-02 ± 1.69E-03 [b]	**
AL-6	(E)-2-Heptenal	ND	ND	ND	–	ND	ND	ND	NS
AL-7	Nonanal	3.64E-02 ± 1.45E-02 [b]	9.95E-02 ± 5.43E-03 [a]	3.46E-02 ± 5.14E-03 [b]	**	3.74E-02 ± 2.97E-03 [c]	9.10E-02 ± 3.73E-03 [a]	6.92E-02 ± 1.66E-02 [b]	**
AL-8	(E,E)-2,4-Heptadienal	ND	ND	ND	–	ND	3.15E-03 ± 2.10E-04 [a]	ND	**
AL-9	(E,E)-2,4-Decadienal	ND	1.73E-03 ± 4.60E-04 [a]	ND	**	ND	4.60E-04 ± 1.90E-04 [a]	ND	**
K-1	2-Butanone	5.44E-02 ± 2.88E-03 [a]	5.28E-02 ± 3.96E-03 [a]	3.99E-02 ± 5.76E-03 [b]	*	4.90E-02 ± 2.42E-03 [a]	4.21E-02 ± 2.56E-03 [b]	3.80E-02 ± 1.01E-03 [b]	**
K-2	2-Pentanone	7.83E-03 ± 5.70E-04	7.78E-03 ± 7.10E-04	7.23E-03 ± 8.90E-04	NS	1.07E-02 ± 5.30E-04	8.34E-03 ± 1.47E-03	1.05E-02 ± 8.00E-04	NS
K-3	Methyl Isobutyl Ketone	8.92E-02 ± 2.34E-03 [a]	7.23E-02 ± 4.57E-03 [b]	9.20E-02 ± 9.18E-03 [a]	*	9.30E-02 ± 7.52E-03	8.20E-02 ± 1.08E-02	8.25E-02 ± 1.03E-02	NS
K-4	2-Heptanone	1.95E-02 ± 3.22E-03 [a]	2.23E-02 ± 1.30E-03 [a]	1.41E-02 ± 1.67E-03 [b]	*	2.84E-02 ± 6.33E-03	2.33E-02 ± 3.10E-03	3.25E-02 ± 3.15E-03	NS
K-5	3-Octen-2-one	ND	ND	ND	–	ND	8.40E-04 ± 3.30E-04 [a]	ND	**
K-6	2,5-Octanedione	5.68E-03 ± 7.40E-04 [b]	5.84E-03 ± 3.30E-04 [b]	7.18E-03 ± 5.80E-04 [a]	*	5.00E-03 ± 1.05E-03	5.73E-03 ± 4.90E-04	5.04E-03 ± 6.10E-04	NS
K-7	3,5-Octadiene-2-one	ND	ND	ND	–	ND	2.78E-03 ± 6.40E-04 [a]	ND	**
HA-1	Acetone alcohol	3.63E-01 ± 5.46E-03	3.23E-01 ± 1.60E-02	3.69E-01 ± 5.86E-02	NS	2.82E-01 ± 2.03E-02	2.51E-01 ± 2.62E-02	2.51E-01 ± 1.85E-02	NS
HA-2	Isobutyl alcohol	1.60E-02 ± 1.09E-03	1.62E-02 ± 2.34E-03	1.39E-02 ± 1.91E-03	NS	9.43E-03 ± 1.39E-03	1.10E-02 ± 5.90E-04	9.02E-03 ± 1.34E-03	NS
HA-3	1-Penten-3-ol	9.85E-03 ± 2.30E-03	8.33E-03 ± 6.40E-04	1.18E-02 ± 3.78E-03	NS	1.96E-02 ± 2.14E-03 [a]	1.19E-02 ± 9.80E-04 [b]	2.28E-02 ± 2.02E-03 [a]	**
HA-4	3-Hexen-1-ol	1.10E-02 ± 4.90E-03 [b]	3.24E-02 ± 1.10E-04 [a]	1.20E-02 ± 1.68E-03 [b]	**	9.59E-03 ± 7.70E-04 [b]	4.47E-02 ± 1.53E-02 [a]	2.19E-02 ± 5.41E-03 [b]	*
F-1	2-Ethylfuran	5.29E-03 ± 1.58E-03	6.22E-03 ± 3.10E-04	6.71E-03 ± 1.79E-03	NS	1.32E-02 ± 1.12E-03 [b]	1.20E-02 ± 7.60E-04 [b]	1.74E-02 ± 1.13E-03 [a]	**
F-2	2-Pentylfuran	1.74E-02 ± 1.08E-02 [b]	1.88E-01 ± 9.42E-02 [a]	2.88E-02 ± 3.09E-03 [b]	*	1.20E-02 ± 2.30E-03 [b]	7.46E-02 ± 1.40E-02 [a]	2.38E-02 ± 2.84E-03 [b]	**
F-3	2-n-Butyl furan	ND	ND	0	–	ND	4.50E-03 ± 2.83E-03 [a]	ND	*
S-1	Dimethyl disulfide	1.14E-02 ± 1.59E-03 [a]	1.07E-02 ± 7.20E-04 [a]	5.11E-03 ± 1.03E-03 [b]	**	1.60E-02 ± 1.13E-03 [a]	1.38E-02 ± 1.41E-03 [a]	5.97E-03 ± 5.30E-04 [b]	**
S-2	Dimethyl trisulfide	2.86E-03 ± 4.30E-04 [a]	3.00E-03 ± 2.50E-04 [a]	2.00E-03 ± 3.00E-04 [b]	*	4.59E-03 ± 3.70E-04 [a]	4.36E-03 ± 4.00E-04 [a]	2.81E-03 ± 2.00E-04 [b]	**
E-1	Ethyl acetate	4.21E-03 ± 5.10E-04	2.57E-03 ± 7.90E-04	2.83E-03 ± 1.48E-03	NS	3.80E-03 ± 7.80E-04 [b]	5.55E-02 ± 2.67E-03 [a]	3.12E-03 ± 2.00E-04 [b]	**
E-2	Allyl propionate	2.31E-02 ± 5.78E-03	2.05E-02 ± 7.10E-04	2.31E-02 ± 5.61E-03	NS	3.11E-02 ± 2.27E-03 [a]	2.51E-02 ± 1.10E-03 [b]	3.14E-02 ± 1.76E-03 [a]	**
T-1	α-Thujene	9.52E-03 ± 1.83E-03 [a]	4.67E-03 ± 2.40E-04 [b]	1.19E-02 ± 2.04E-03 [a]	**	1.15E-02 ± 3.28E-03 [a]	4.64E-03 ± 7.80E-04 [b]	1.00E-02 ± 1.40E-03 [a]	*
T-2	β-Pinene	1.28E-02 ± 3.81E-03 [a]	5.28E-03 ± 1.69E-03 [b]	1.13E-03 ± 2.00E-05 [b]	**	1.83E-02 ± 6.78E-03 [a]	6.07E-03 ± 3.55E-03 [b]	1.06E-03 ± 1.00E-04 [b]	**
T-3	(R) -α-pinene	6.50E-03 ± 1.17E-03 [b]	3.70E-03 ± 1.20E-04 [c]	9.44E-03 ± 1.58E-03 [a]	**	8.71E-03 ± 2.62E-03 [a]	3.54E-03 ± 8.30E-04 [b]	7.54E-03 ± 1.50E-03 [a]	*
T-4	β-Ocimene	6.30E-01 ± 2.25E-01 [a]	1.57E-01 ± 1.39E-02 [b]	5.54E-02 ± 9.47E-03 [b]	**	1.13E+00 ± 4.79E-01 [a]	2.71E-01 ± 1.34E-01 [b]	5.33E-02 ± 7.20E-03 [b]	**
T-5	γ-Terpinene	2.08E-02 ± 7.58E-03 [a]	5.92E-03 ± 8.50E-04 [b]	ND	**	3.73E-02 ± 1.45E-02 [a]	9.63E-03 ± 4.15E-03 [b]	ND	**

* "ND" represents "Not Detected". Data are mean ± standard deviation of one-way ANOVA and Tukey's post hoc test. Different letters in each row indicate significant differences at a significant level of 0.05.
*Quantitative results are expressed as the ratio of peak area of GC-Orbitrap-MS.

about six times in S1-7D and about three times in S3-7D, 2-n-butylfuran decreased substantially in the three-stage samples (S3-7D), which was not detected. While 2-ethylfuran was considerably higher in the three-stage samples (S3-7D) and was mostly described as displaying mostly "beany" and "cocoa".

3.2.5. Other compounds

The sulfides in milk powder may originate from protein denaturation (Al-Attabi et al., 2008), resulting in oxidative off-flavors. The peak area ratio of the sulfides did not change significantly after 3 days of simulated daily use, while that of both the dimethyl disulfide and dimethyl

trisulfide decreased significantly after 7 days of simulated daily use and were mostly described as “sulfurous” and “oniony”.

After opening, the peak area ratios of the esters did not differ significantly in the one-stage samples. In the three-stage samples, the peak area ratio of ethyl acetate increased dramatically after 3 days of simulated daily use (S3-3D), with more than ten times, which was mostly described as “fruity” and “sweet”. While it decreased significantly after 7 days of simulated daily use (S3-7D), with more than ten times. Allyl propionate showed the opposite trend and was mostly described as displaying “tropical fruit”.

The terpenoids in infant formula may be derived from cow feed (Bayramoglu et al., 2008). Herbs such as oregano are reportedly often added to dairy cattle feed as oregano essential oil, oregano grass powder, and oregano infusion to promote growth and development (Tang et al., 2020). After 3 days of simulated daily use, the peak area ratios of all the terpenoids were significantly lower in both the one and three-stage samples (S1-3D and S3-3D) and were mostly described as “fresh”, “woody”, and “herbal”. Although the peak area ratios of all the terpenoids were significantly lower after 7 days of opening, the peak area ratios of the α-thujene and (R)-α-pinene increased dramatically in both the one and three-stage samples (S1-7D and S3-7D).

3.3. Heat map analysis

To visually compare the differences in the content of volatile compounds at 0 day, 3 days, and 7 days of simulated daily use, the semi-quantitative results of the 16 out of 32 volatile compounds with significant differences in one-stage samples and 23 out of 32 volatile compounds with significant differences in three-stage samples were plotted separately as cluster heat maps, respectively, as shown in Fig. 2.

The peak area ratios of β-pinene, β-ocimene, and γ-terpinene showed a decreasing trend overall as the number of days after opening increased in one-stage and three-stage samples, probably due to the escape of terpenoids associated with “freshness” into the environment. Except for 2-heptanone, which showed no significant difference in three-stage samples, the 2-butanone, 2-heptanone, dimethyl disulfide, and dimethyl trisulfide in both one-stage and three-stage samples decreased significantly after 7 days of opening, presumably due to the higher escape rate of the compounds as the number of days after opening increased.

In one-stage samples, four compounds, nonanal, (E,E)-2,4-decadienal, 3-hexen-1-ol and 2-pentylfuran showed an increasing and then decreasing trend at 0 day, 3 days and 7 days of simulated daily use, respectively, the ten compounds, including pentanal, heptanal, nonanal, (E,E)-2,4-heptadienal, (E,E)-2,4-decadienal, 3-octen-2-one, 3,5-octadien-2-one, 3-hexen-1-ol, 2-pentylfuran and 2-n-butylfuran, showed the same trend in three-stage samples. This might because when the milk powder was initially opened, the lipid oxidation rate accelerated and their content increased, and then as the days after opening the lid increased, the diffusion rate was greater than the lipid oxidation rate, leading to a decrease in their content. Hexanal, pentanal, heptanal, and octanal were good indicators of lipid oxidation (Clarke et al., 2020). (E,E)-2,4-Heptadienal and (E,E)-2,4-decadienal have been reported as intermediate products of lipid oxidation in research (Clarke et al., 2021). 3-Octen-2-one和3,5-octadien-2-one also originate from lipid oxidation (Clarke et al., 2021). Studies have shown that aldehydes, ketones, alcohols, and furans are the main compounds responsible for oxidative off-flavors in infant formula samples (Al-Attabi et al., 2008).

The peak area ratios of the methyl isobutyl ketone, 2,5-octanedione, α-thujene, and (R)-α-pinene in one-stage samples showed a decreasing trend primarily followed by an increasing trend at 0 day, 3 days and 7 days of simulated daily use. And the 1-penten-3-ol, allyl propionate, α-thujene, and (R)-α-pinene showed the same trend in three-stage samples. This presumably due to the rapid release rate of these compounds when the milk powder was initially opened, with the content decreased, and as time increased, their production rate was greater than the release rate.

3.4. The sensory difference among samples

The sensory differences in the infant formula samples at 0 d, 3 d, and 7 d of simulated daily use were investigated using the triangle test. The results provided by 24 panelists were statistically analyzed and checked against the triangle test method test sheet. The triangle test results for four sample groups were plotted as bar charts, as shown in Fig. 3. For the one-stage samples, eight panelists correctly selected the samples after 3 days of simulated use, and seven correctly identified the samples after 7 days of simulated use. For the three-stage samples, 13 panelists correctly selected the samples after 3 days of simulated use, and six accurately identified the samples 7 days of simulated use. According to checking against the triangle test method test sheet, the minimum number of correct answers required to cause a significant difference is 13 at the 5 %

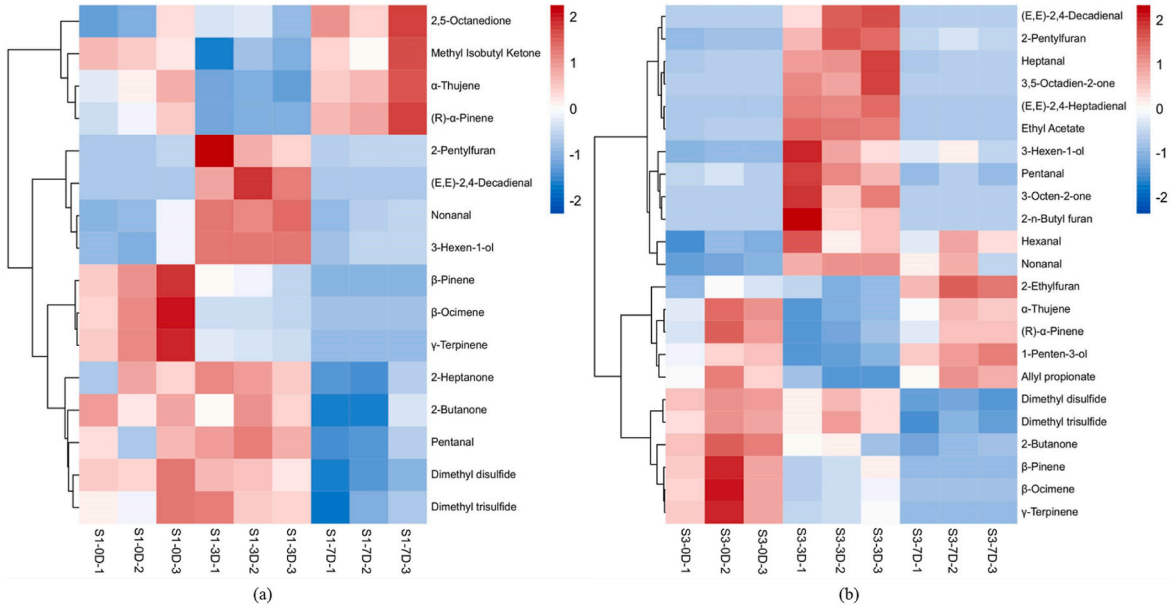

Fig. 2. The cluster heatmap of the volatile compounds in the infant formula samples. (A) One-stage. (B) Three-stage.

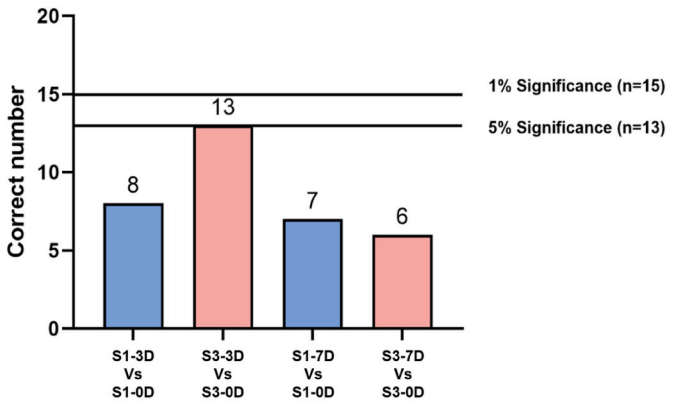

Fig. 3.

level, 15 at the 1 % level, and 16 at the 0.1 % level when n = 24.

The results showed no significant differences in sensory between the 0 d, 3 d, 7 d of simulated daily use in one-stage samples. In three-stage samples, there was a significant difference in sensory at the 5 % level on the 3 d of simulated daily use, while there was no significant change on the 7 d of simulated daily use. The experimental findings indicated no substantial changes in the sensory of the open-cap samples, except on the 3 d of simulated use in the three-stage samples. This could be due to the significant increase after 3 d of simulated use in the peak areas of aldehydes such as pentanal, (E,E)-2,4-heptadienal, 3-octen-2-one, 3,5-octadien-2-one, 3-hexen-1-ol, and 2-pentylfuran, which were identified as compounds that caused oxidative off-flavors in milk powder. It has been reported that the "fishy" odor in milk powder mainly comes from aldehydes, ketones, alcohols and furans (Tang et al., 2022a). Pentanal had been proven to be a marker volatile for lipid oxidation (Cheng et al., 2017). (E, E) −2,4-heptadienal were demonstrated to be the key aroma active compounds in infant formula (Zhang et al., 2022). It has been found that 3,5-octadiene-2-one (Marsili and Laskonis, 2014) was related to fishy odor in infant formula. However, their peak area ratios decreased significantly after 7 d of simulated use, resulting in no significant difference between this sample and the 0 d of simulated use.

4. Conclusion

This study employed HS-SPME/GC-Orbitrap-MS to analyze the volatile compounds in one and three-stage infant formula samples during the secondary shelf-life. A total of 32 compounds are identified, mainly consisting of aldehydes, ketones, alcohols, furans, sulfides, esters, and terpenoids. Within one week of simulated daily use, 50 % of the compounds in one-stage samples change considerably, while approximately 70 % of those in the three-stage samples are significantly different. The triangle test reveals significant changes in the sensory attributes of the three-stage samples at 3 d of simulated daily use, while minor changes are evident in the other samples. The significant odor changes in the three-stage samples at 3 d of simulated daily use may be related to the increased content of compounds such as pentanal, (E,E)-2,4-heptadienal, 3-octen-2-one, 3-hexen-1-ol, and 2-pentylfuran. Therefore, the volatile compounds during the different infant formula stages vary within 7 d during the secondary shelf-life, while the sensory attributes remain stable. From above all, in the composition of volatile compounds changed drastic and the odor might change during the secondary shelf-life of infant formula. This study might offer novel perspectives to enhance and refine infant formula, which could effectively cater to the requirements and concerns of customers.

According to our research, there were significant changes in the volatiles of infant formula during the second shelf life, and sensory changes were caused to a certain extent. The secondary shelf life is a noteworthy aspect in the study of infant formula quality, and further research is needed to clarify the impact of volatiles on sensory changes during the secondary shelf life, in order to determine whether the secondary shelf life should be used as an indicator for the development and production quality control of milk powder formulas.

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Ethical statements

The experiment posed no harm or risk to the participants. All participants signed informed written consent before the experiment. The Ethics Committee of the Department of Psychology, School of Humanities and Social Sciences, Beijing Forestry University, evaluated and approved the research protocol. The original file and its English version can be found in the Supplementary Material.

CRediT authorship contribution statement

Li Yilin: designed the study, was a major contributor in writing the manuscript, Writing – original draft. **Li Ruotong:** prepared the literature search, conducted the experiment, Formal analysis, Writing – original draft. **Hu Xinyu:** conducted the experiment. **Liu Jiani:** conducted the experiment. **Liu Guirong:** provided materials for the study and conceived, Supervision. **Gao Lipeng:** provided materials for the study and conceived, Supervision. **Zhang Yongjiu:** provided materials for the study and conceived, Supervision. **Baoqing Zhu:** Supervision, revised and approved the manuscript, All authors contributed to the article and approved the submitted version.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

I have shared the link to my data at the Attach File step.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.crfs.2024.100693.

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