



The role of packaging on the flavor of fluid milk

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ABSTRACT

Few studies have addressed the effects of package material in the absence of light on contributions to fluid milk flavor. The objective of this study was to compare the sensory and chemical properties of fluid milk packaged in paperboard cartons, low-density polyethylene, high-density polyethylene (HDPE), polyethylene terephthalate (PET), linear low-density polyethylene (LLDPE), and glass. Pasteurized (high temperature short time, 77°C for 25 s) skim and whole milk were filled (280 mL ± 10 mL) into paperboard cartons, low-density polyethylene, HDPE, PET, LLDPE, and glass (control). Milks were stored at 4°C in the dark and sampled at d 0, 5, 10, and 15. Descriptive analysis was applied to document sensory profiles at each time point, and volatile compounds were extracted and identified by solid-phase microextraction with gas chromatography mass spectrometry and gas chromatography-olfactometry. Tetrad tests with consumers were conducted at d 10. Both skim and whole milks packaged in cartons had noticeable paperboard flavor by d 5 and higher levels of hexanal than skim and whole milks in other package types at d 5. Skim milks packaged in paperboard cartons and LLDPE had distinct refrigerator/stale flavor compared with milks in the other package types, concurrent with increased levels of refrigerator/package-related compounds including styrene, acetophenone and 2-ethyl-1-hexanol. Milks packaged in glass, PET and HDPE were not distinguished by consumers at d 10 post-processing. Package type influences fluid milk flavor, and these effects are greater in skim milk compared with whole milk. Paperboard cartons do not preserve milk freshness, as well as PET, HDPE, or glass, due to flavor migration and scalping. Glass remains an ideal barrier to preserve fluid milk flavor, but in the absence of light, HDPE and PET provide additional benefits while also maintaining fluid milk flavor.

Key words: fluid milk, flavor, package

INTRODUCTION

The main concerns for the dairy industry for pasteurized fluid milk are preserving the quality and safety of the final product (Dainelli et al., 2008), while also preserving the characteristic pleasant flavor of milk (Alvarez, 2009). The characteristic flavor active compounds of pasteurized fluid milk are created during thermal processing (Shimamura and Uke, 2012). However, the flavor of milk has changed over time due, in part, to changes in packaging used to transport milk to the consumer (Azzara and Campbell, 1992; Dainelli et al., 2008). Light oxidation from transparent containers, as well as scalping and migration from different packaging types, affect the flavor of milk and have led to consumer dissatisfaction (Johnson et al., 2015; Potts et al., 2017; Schiano et al., 2019).

Pasteurized fluid milk is more susceptible to packaging-related off-flavors than many other beverages because of its relatively bland taste. The dairy industry, as such, has focused on light blocking options in regards to packaging. Light-oxidized flavor is a well-established and well-researched off-flavor in retail fluid milk in transparent containers, and is caused by photo oxidation of riboflavin and other naturally occurring photosensitive compounds in milk (Northrop-Clewes and Thurnham, 2012; Walsh et al., 2015; Brotherson et al., 2016). Paperboard cartons are generally considered to have good light barrier properties compared with other fluid milk package materials, such as polyethylene terephthalate (**PET**), high-density polyethylene (**HDPE**), or glass (Potts et al., 2016, 2017).

In addition to the light-oxidized flavor, off odors and flavors in milk can be created or enhanced, or both, by flavor active compounds from packaging itself via migration and scalping (Bassette et al., 1986). Food migration is the transfer of unwanted chemical contaminants such as trace amounts of solvents, by-products, additives, or plastic monomers, or all these contaminants, into a food from the surrounding environment or packaging (Kim-Kang, 1990). Food scalp-

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ing is the transfer of desirable volatile food flavors and aromas to the packaging through absorption or release of undesirable flavors and aromas into the food from the surrounding packaging (Roland and Hotchkiss, 1991). Migration and scalping of flavor active compounds are heavily affected by the volatility and polarity of both the food matrix and package, lipophilicity of the product, physical structure (solid or liquid), and temperature of the product and storage conditions (Sajilata et al., 2007).

Paperboard cartons and HDPE are the most widely used package types for fluid milk (Brody, 2009). Paperboard cartons are widespread because they are malleable and cheaper to produce than many other options (Brody, 2009). Paperboard cartons are made from wood chips that have been chemically treated to a pulped consistency. The pulped wood is then poured into molds and left to cure until set in the desired shape. Commonly, paperboard cartons are lined with millimeter-thin layers of low-density polyethylene (LDPE) as a barrier between the paperboard and the product. Low-density polyethylene acts as a fluid barrier because of its resistance to liquids, pH, and alcohol (Nielsen et al., 1992; Simon and Hansen, 2001). Paperboard cartons have potential disadvantages because paperboard is highly permeable to the surrounding environment and can allow volatile compound migration into the container contents (Ziegler, 1998). Paperboard may also be a direct source of off-flavors because the formation of wood lignin into paperboard is an oxidative process and these compounds can also migrate into product (Ziegler, 1998). High-density polyethylene, though costlier than paperboard, is commonly used because of its structural stability, moisture resistance, and barrier to the surrounding environment (Crosby, 1981; Nielsen et al., 1992). Polyethylene terephthalate is also an alternative for packaging of fluid milk (Brody, 2009). Polyethylene terephthalate, when compared with HDPE, is highly resistant to acids, oils, and alcohols, and is generally a good barrier against moisture (Sajilata et al., 2007). Polyethylene terephthalate is not as quite as rigid, durable, or resistant to heat when compared with HDPE, but is cheaper to produce and more malleable, making it easier to manipulate during production and recycling (Sajilata et al., 2007).

The role of flavors from packaging (scalping or migration) on the sensory quality of fluid milks have not been fully investigated. Previous research has shown that salted and unsalted butter absorbed flavors from packaging and the surrounding environment at 4°C storage (Lozano et al., 2007a). Leong et al. (1992) demonstrated that whole, low-fat, and skim milk packaged in polyethylene-coated half-pint paperboard cartons stored at 4°C had package off-flavor by d 1 (Leong et

al., 1992). Leong et al. (1992) focused on off-flavors contributed from polyethylene-coated paperboard cartons during fluid milk shelf life and did not conduct volatile compound analysis or compare with other packaging materials. Sipple et al. (2020) evaluated child consumer perception of extrinsic and intrinsic parameters of fluid milk in single-serve half pints of different package materials. They demonstrated that school children preferred skim or 1% unflavored milk packaged in HDPE or PET containers over traditional coated paperboard after approximately 10 d of cold storage. Volatile compound differences, multiple time points, and whole milk were not evaluated. Different package materials for fluid milk have not been directly compared in the absence of light, nor have volatile compounds been determined. The objective of this study was to determine the role of package on the chemical and sensory properties of milk in the absence of light exposure. The absence of light exposure allows control of the study parameters such that the actual role of package material on migration or scalping, or both, can be clearly identified and sourced.

MATERIALS AND METHODS

Experimental Overview

Whole and skim milk were high temperature short time (HTST) processed and filled into the following 6 different packages: amber glass (TraceClean Wide Mouth Packers VWR), LDPE (Wide Mouth, VWR), HDPE (Wide Mouth, VWR), PET (Storage Bottles, Corning), LLDPE (Gusset Bag, Elkay Plastics Co.), or paperboard carton (Evergreen Barrier Paperboard, Evergreen Packaging), and stored at 4°C. Sensory properties (descriptive sensory analysis) and volatile compound analysis [headspace solid-phase microextraction (HS-SPME) GC-MS] were conducted at time 0 and d 5, 10, and 15. Consumer difference testing (tetrad tests) were conducted for whole and skim fluid milk packaged in HDPE versus glass, PET versus glass, and paperboard carton versus glass at d 10. The entire experiment was repeated 3 times.

Milk Processing

Raw milk (<10,000 cfu/mL aerobic plate count, <100,000 SCC) was standardized (whole milk had $3.25 \pm 0.2\%$ fat and skim milk had $<0.10 \pm 0.05\%$ fat) and processed at the North Carolina State University Dairy Enterprise System (Raleigh, NC). Skim and whole milk were HTST processed (77°C for 25 s) and filled into the following 6 different 280-mL (± 10 mL) packaging types: amber glass, LDPE, HDPE, PET, LLDPE, or polyethylene-lined paperboard carton. All packag-

ing types were sanitized before filling in a 100-mg/kg (NaOCl) sanitizer/water solution (XY-12, Ecolab). Processing and filling were done with minimal light to reduce light exposure. Packages were filled to the same volume, as follows: 230 mL (± 10 mL) with 50 (± 10 mL) of headspace. Paperboard cartons were filled using an Evergreen EQ-2 gable top filler (Evergreen Packaging) system. All other packaging types were hand filled using a Micro Thermics Ultra-Clean Fill Hood (MicroThermics). Milks were stored at 4°C and covered with black laser cloth to shield from light. Milks were sampled for sensory and instrumental analysis on d 0, 5, 10, 15 post-processing. Total aerobic plate and coliform counts (coliform and aerobic plate count Petrifilms; 3M) were conducted on milks at d 0, 10, 15 to monitor microbial quality.

Dissolved Oxygen

The dissolved oxygen of skim and whole milk of each package type was measured using a benchtop dissolved oxygen analyzer (YSI Model 5100) at each time point (0, 5, 10, 15 d). The oxygen probe was placed in the sample, with both ends of the probe submerged, and stirred using the self-stirring probe BOD probe attachment. The reading was then allowed to stabilize for dissolved oxygen and a measurement was taken. All measurements were done at 21°C in triplicate for skim and whole milk on each package type and each experimental replication. Measurements were given in milligrams per liter of oxygen in sample. Calibration for dissolved oxygen was done initially before each time point by placing the probe in a known oxygen environment (250 mL of water in a bottle aerated for 15 min with pure O₂, the probe was stabilized for 15 min in the aerated water). The probe was adjusted if readings of known oxygen environment were out of specifications. Barometric pressure was adjusted to current elevation and temperature in the setting before each calibration

Headspace Oxygen

The headspace oxygen of skim and whole milk of each package type was measured using a benchtop oxygen analyzer (Quantek Model 905) at each time point (0, 5, 10, 15 d). To prevent oxygen leaking in or out of the packaging, the package was first fitted with a foam rubber septum (Foam Septum). The package was then punctured using a 20/2nd/5 leur needle (N720 Hamilton Needle) with a side-slotted opening. The oxygen analyzer was then turned on until a stable oxygen reading was established. Readings were conducted at 21°C. All measurements were done in triplicate for each package type and each experimental replication for skim

and whole milk. A calibration was done initially before each time point by filling a heat-sealed plastic bag (size 2" $\times$ 2", thickness 0.0009", Gusset Bag, Elkay Plastics Co.) with pure N₂ through a foam rubber septum, and then measuring the atmosphere in the bag. The reading was adjusted to 0% oxygen ($\pm 0.1\%$) with the ZERO potentiometer on the back of the analyzer as needed. The atmosphere of the room was then used to check calibration between each sample, and was re-adjusted or re-calibrated if necessary.

Descriptive Sensory Analysis

Samples were prepared with the overhead light off to minimize potential lights effects on milk. Sensory profiles of milks from each package type at each time point were evaluated by 7 experienced and trained panelists, using an established sensory language for fluid milk (Lozano et al., 2007b; Lee et al., 2017; McCarthy et al., 2017). Each panelist had a minimum of 90 h of previous experience evaluating the sensory profile of different fat levels of HTST fluid milk. Attributes were scored using a 0- to 15-point scaling consistent with the Spectrum Method (Meilgaard et al., 2007). Each milk was evaluated at each time point (0, 5, 10, 15 d) in duplicate by each panelist. Milks were poured (40 mL) into soufflé cups (65 mL) numbered with randomized 3-digit codes. Panelists evaluated milks from each experimental replication of each fat type (6 milks) in a randomized order in 1 session. A 3-min rest between samples was enforced. Paper ballots were used for data collection.

Volatile Compound Analysis of Fluid Milk

HS-SPME-GCMS. Volatile compounds were extracted from milks at each time point (0, 5, 10, 15 d) by HS-SPME, followed by GC-MS with a ZB-5ms (30 m $\times$ 0.25 mm i.d. $\times$ 0.25 μ m; Phenomenex) and a ZB-Wax (30 m $\times$ 0.25 mm i.d. $\times$ 0.25 μ m) column (Phenomenex) with MS (7820 GC 5975 MSD, Agilent Technologies Inc.). Samples were injected via a CTC Analytics CombiPal Autosampler (CTC Analytics). Extraction methods were modified from Jo et al. (2018). Five grams of milk was measured in triplicate in SPME vials (Microliter Analytical), with 20 μ L of internal standard added 81 mg/kg 2-methyl-3-heptanone (Sigma-Aldrich) in methanol and 167 mg/kg 2-methylpentanoic (Sigma-Aldrich) in methanol. Vials were equilibrated at 40°C for 20 min with 3 s of 350 rpm agitation. A single DVB/Carboxen/PDMS 1.0-cm fiber (Supelco) was used for all analysis. The SPME fiber was exposed to the samples for 25 min at depth 3.1 cm. The fiber was retracted and injected at 5.0 cm in

the GC inlet for 7.5 min. The GC oven was held at 40°C for 3 min with a gradient of 10°C/min to 90°C with no hold, and the gradient was increased at a rate of 5°C/min to 200°C and held for 10 min, and then increased to 20°C/min 250°C and held for 5 min at completion. Helium was pressurized at 7 psi in the column and maintained at a constant flow rate of 1 mL/min. A standard scan mode was used (30 to 350 m/z) to identify compounds of interest for initial evaluation. Selective ion monitoring mode was subsequently used for compound identification using authentic standards.

HS-SPME GC-Olfactometry. The HS-SPME GC-olfactometry was used to identify aroma active compounds. Gas chromatography-olfactometry was conducted on an Agilent 6850 GC (Agilent Technologies Inc.) with a ZB-5ms (30 m $\times$ 0.25 mm i.d. $\times$ 0.25 μ m) column (Phenomenex) or a ZB-Wax plus (30 m $\times$ 0.25 mm i.d. $\times$ 0.25 μ m) column (Phenomenex). Ten milliliters of milk was added to 40-mL vials in duplicate. Vials were equilibrated for 30 min at 40°C and SPME fibers were then exposed to the headspace of the vials for 30 min at 2 cm. Fibers were manually injected at 3-cm depth. The fiber was initially exposed to the inlet for 10 min at 250°C. The GC program was held at 40°C for 3 min and increased gradually to 150°C over 11 min (10°C/min). The temperature was then increased to 250°C over 3.5 min (30°C/min) and then held for 10 min. Carrier gas used was helium at a flow rate of 1.5 mL/min. The splitter was used at a 1:1 ratio between the flame ionization detector and sniffer port. Two experienced sniffers recorded aroma events, retention times, and perceived aroma intensities using a 0- to 5-point post-peak intensity scale.

Compound Identification and Quantitation. Compound identification was achieved by MS (NIST, 2017) and odor properties of authentic standards. Retention indices of volatile compounds were calculated (van Den Dool and Kratz, 1963) using a C_6 - C_{22} alkane series (Sigma-Aldrich) under identical GC olfactometry/MS conditions. External standard curves for 9 selected compounds (styrene, xylene, 2-ethyl-1-hexanol, hexanal, nonanal, p-cymene, acetophenone, toluene, octanal) were generated using skim or whole (HTST) milk packaged in glass as the matrix. Each stock solution was prepared using authentic standards (Sigma-Aldrich) and diluted (methanol, ethanol, or water based on solubility) at separate points for a 5-point standard curve. Each matrix was also spiked with 20 μ L of an internal standard (81 mg/kg 2-methyl-3-heptanone in methanol), with the ratio of response calculated along with the external standard in the matrix.

Direct Solvent Extraction Solvent-Assisted Flavor Extraction. Direct solvent extraction with solvent-assisted flavor extraction (DSE-SAFE) was

chosen to evaluate skim milk stored at 4°C for 15 d from paperboard cartons and glass (control) because of its more sensitive recovery of semi-volatile compounds and higher molecular weight compounds (Engel et al., 1999; Lozano et al., 2007b; Xie et al., 2013). Direct solvent extraction with solvent-assisted flavor extraction was used for the identification and determination of specific compounds related to paperboard cartons that were identified at the limits of quantification by SPME-GCMS.

Fluid milk was extracted using a modified method of direct solvent extraction, as described by Engel et al. (1999). Three hundred milliliters of fluid skim milk was separated into 3 equal parts into 3 250-mL Teflon bottles (Thermo Fisher Scientific). One hundred mL of ethyl ether (Sigma-Aldrich) and 40 μ L of each internal standard was added to each bottle (2-methyl-3-heptanone, 81 mg/kg, neutral/basic fraction, 2-methylpentanoic acid 167 mg/kg acid-fraction; Sigma-Aldrich), and 30 g of NaOH (Sigma-Aldrich) was added to break the emulsion. Each bottle was sealed and then shaken vigorously for 2 min, untightened to let out excess pressure, and then placed for 30 min on a platform mixer (Rotomix type 50800 Thermolyne) at the highest setting. Bottles were then centrifuged at 2,500 $\times g$ at 21°C (Thermo Lynx 6000, Thermo Fisher Scientific) for 30 min. The top aqueous layer (ethyl ether) was then removed and dried using anhydrous sodium sulfate (Na_2SO_4 ; Sigma-Aldrich) to remove excess water. All samples were concentrated to 100 mL under nitrogen gas.

Concentrated samples were then distilled using a SAFE apparatus, assembled as previously described (Engel et al., 1999; Lozano et al., 2007b). A round 1-L flask was attached to the main SAFE head apparatus and submerged in a 40°C water bath to maintain temperature. The SAFE head apparatus was then attached to 2 glass traps, in series, for collection of the sample. Both traps were submerged in liquid nitrogen-filled Nalgene buckets that were constantly filled to maintain temperature. The SAFE Apparatus (Ace Glassware) was then brought to 10^{-5} Torr vacuum, and attached to the end of the second trap in series. Sample was introduced slowly (over 15 min) via a stopcock into the main SAFE apparatus head into the 1-L flask, volatilizing the sample, and pulling it through the SAFE apparatus and condensing it in the first trap. After all sample was fully introduced, the SAFE apparatus was maintained at constant vacuum (10^{-5} Torr) and liquid nitrogen was maintained in Nalgene buckets for 2 h for maximum retention. The primary trap with condensed sample was collected and allowed to thaw at room temperature. Sample was then dried with anhydrous sodium sulfate (Na_2SO_4) in an amber glass jar (250 mL) and

then concentrated to 20 mL under a stream of nitrogen gas. The concentrated sample was then transferred to a screw-top glass tube for phase separation.

To aid in compound detection, all samples were separated into 2 separate fractions, neutral/basic and acidic. The concentrated sample was first washed with 3 mL of 0.5 M sodium bicarbonate (Thermo Fisher Scientific) and shaken for 10 min. The aqueous layer was removed (bottom layer) after each washing and collected. The sodium bicarbonate wash was repeated twice. The sample was then washed with 2 mL of saturated NaCl solution (23% wt/wt; VWR) and shaken for 10 min with the aqueous layer removed (bottom layer) and collected into the same glass tube. The ether phase remaining (upper layer) was the resultant neutral/basic fraction and was then concentrated to 2 mL under a stream of nitrogen gas, dried with anhydrous sodium sulfate (Na_2SO_4) into a new 2 mL glass vial, and concentrated to 0.5 mL under nitrogen gas.

The transferred aqueous layer (bottom layer) in the glass tube was acidified through the addition of 6 M hydrochloric acid (Sigma-Aldrich) to a pH of approximately 2.0. Five milliliters of diethyl ether was then added to the tube and shaken for 10 min and the ether phase removed. This process was repeated 3 times. The collected ether phase was the acidic fraction. The acidic fraction was then concentrated to 2 mL under a stream of nitrogen gas, dried with anhydrous sodium sulfate (Na_2SO_4), and concentrated to 0.5 mL under nitrogen gas.

Compound Identification for DSE-SAFE.

Three microliters of the acidic and neutral/basic fraction were injected in split-less mode in duplicate on a ZB-5ms (30 m $\times$ 0.25 mm i.d. $\times$ 0.25 μm) column and a ZB-Wax plus (30 m $\times$ 0.25 mm i.d. $\times$ 0.25 μm) column installed in an Agilent 7820-GC paired with a 5975-MSD. All injections had a 2.5-min solvent delay to protect the instrument. The GC oven was at held at 40°C for 3 min with a gradient of 10°C/min to 90°C with no hold, the gradient was increased to a rate of 5°C/min to 200°C and held for 10 min, and the gradient was then increased to 20°C/min 250°C and held for 5 min at completion. Selective ion mode and scan mode were used in tandem to identify and quantify compounds of interest. Compound identities were confirmed with authentic standards. Internal standards were used to calculate relative abundance for compounds of interest.

Consumer Difference Tests (Tetrads)

Sensory testing was conducted in accordance with the North Carolina State University Institutional Review Board for human subject regulations. Tetrad difference testing (ASTM International, 2015; E3009)

was conducted for both skim and whole milks with 4 packaging types, as follows: paperboard cartons, HDPE, PET, and glass (control) at d 10. Consumers were presented the following 3 separate tetrad tests for whole and skim milk (6 tetrads): HDPE versus glass, PET versus glass, paperboard carton versus glass at d 10 of shelf life. Package types for tetrads were selected based on descriptive analysis and realistic practical applications. High-density polyethylene and PET packages were selected because they make up more than 85% of fluid milk packaging sales (USDA, 2013). Paperboard cartons contribute close to 14% of fluid packaging sales with the majority of that being used for the school lunch programs (USDA, 2013). Consumers ($n = 50$) were recruited via emails from the North Carolina State University Sensory Service Center database with the qualifier being that they were self-reported regular consumers of fluid milk. During testing, each consumer was instructed both verbally and provided with an instruction guide describing how a tetrad test is performed. They were asked to pair the 4 coded samples based on which ones they believed to be the most similar to each other. Testing was replicated with 2 of the experimental replications for skim and whole milk for all 3 tetrad pairs ($n = 100$ consumers through each tetrad).

Milks (60 mL) were evaluated by consumers in 128-mL Styrofoam cups coded with 3-digit codes. Milks were served at 4°C. Each panelist was given 4 coded samples at once and instructed to sample them in the order presented left to right. A 3-min enforced rest occurred between each tetrad during which consumers were requested to rinse their mouths with water and to take a bite of unsalted cracker. Consumers were asked to complete 3 sets of tetrads (1 fat content level) in 1 seating, with the order of presentation of each tetrad test randomized between each consumer. Consumers were compensated at the completion of the test with a \$5 gift card. Compusense Cloud (Compusense) was used for data collection.

Statistical Analysis

Data analysis was conducted at a 95% confidence ($P < 0.05$). Analysis software used included XLSTAT version 2019.1.3 (Addinsoft) and SAS version 9.4 (SAS Institute). Two separate analyses were conducted, 1 with whole milk and 1 with skim. Each experiment was designed as a randomized complete block design with a time point by package type factorial arrangement of treatments and evaluated as a mixed model ANOVA (SAS). Principal component analysis was used to visualize differences among volatile compounds among package types within each fluid milkfat content (XL-

Table 1. Concentration of dissolved oxygen (mg/L) in skim milk in different packages across 4 time points (d 0, 5, 10, 15)

Package type ¹	Time point			
	d 0	d 5	d 10	d 15
Glass	10.31 ± 0.20 ^b	8.68 ± 0.50 ^c	5.71 ± 0.66 ^c	3.90 ± 1.20 ^c
PET	10.59 ± 0.08 ^{ab}	9.26 ± 0.40 ^b	7.77 ± 0.43 ^{ab}	5.12 ± 1.35 ^b
HDPE	10.66 ± 0.10 ^{ab}	9.37 ± 0.16 ^b	7.70 ± 0.35 ^{ab}	4.36 ± 0.50 ^{bc}
LDPE	10.61 ± 0.13 ^{ab}	9.00 ± 0.66 ^{bc}	7.04 ± 0.67 ^b	3.90 ± 0.32 ^c
LLDPE	10.46 ± 0.35 ^{ab}	7.08 ± 0.61 ^d	4.73 ± 0.96 ^d	0.96 ± 0.25 ^d
Carton	11.15 ± 0.13 ^a	10.06 ± 0.26 ^a	8.31 ± 0.59 ^a	7.02 ± 0.99 ^a

^{a-d}Means in the same column followed by a different superscript are significantly different ($P < 0.05$).

¹PET = polyethylene terephthalate; HDPE = high-density polyethylene; LDPE = low-density polyethylene; LLDPE = linear low-density polyethylene. Values shown ± SD.

STAT). Consumer difference data were evaluated using the minimum correct judgement for significance ($P < 0.05$; ASTM International, 2015; E3009–15).

RESULTS AND DISCUSSION

Milk Microbial Quality

No coliforms were detected in pasteurized whole or skim milks. All milks had an aerobic plate count of less than 10^2 cfu/mL initially, and less than 10^4 cfu/mL at d 15.

Dissolved and Headspace Oxygen Analysis

Dissolved oxygen concentration decreased over storage time for all packaging types, regardless of milkfat content ($P < 0.05$; Tables 1, 2). Dissolved oxygen decreases were consistent with previous studies found for fluid milk (Zygoura et al., 2004; Karatapanis et al., 2006). Dissolved oxygen for skim and whole milks packaged in LLDPE decreased faster compared with milks in other packages, presumably due to the greater permeability of the LLDPE ($P < 0.05$; Tables 1, 2, 3). Headspace oxygen (% O_2) concentration decreased

over storage time for glass, PET, HDPE, and LDPE, regardless of milkfat content ($P < 0.05$; Tables 4, 5). Headspace oxygen in milks packaged in cartons and LLDPE did not decrease over storage time due to the high permeability of both packaging types ($P > 0.05$; Tables 3, 4, 5).

Descriptive Analysis

Milks (whole and skim) packaged in different packages demonstrated distinct flavor differences initially and with storage time (Tables 6, 7). Skim milks packaged in paperboard cartons and LLDPE had lower cooked and sweet aromatic flavors with storage time and distinct refrigerator/stale flavor when compared with milks in the other packaging types ($P < 0.05$; Table 6). This flavor difference may be due to the high permeability of paperboard cartons and LLDPE (Table 3). High permeability of packaging has been shown to increase stale flavor and decrease overall flavor acceptability in UHT milks (Wadsworth and Bassette, 1985). Paperboard/cardboard flavor was only detected in milks packaged in cartons, and this flavor was detected within hours of packaging (d 0) and increased with storage time (Table 6). Previous studies have found that paperboard

Table 2. Concentration of dissolved oxygen (mg/L) in whole milk in different packages across 4 time points (d 0, 5, 10, 15)

Package type ¹	Time point			
	d 0	d 5	d 10	d 15
Glass	11.94 ± 0.08 ^b	10.63 ± 0.37 ^{bc}	7.89 ± 0.48 ^c	5.78 ± 0.36 ^b
PET	12.19 ± 0.11 ^a	10.09 ± 1.04 ^c	8.80 ± 1.01 ^b	4.72 ± 0.62 ^d
HDPE	12.18 ± 0.16 ^a	11.04 ± 0.44 ^{ab}	8.94 ± 0.56 ^b	5.37 ± 0.62 ^{bc}
LDPE	12.16 ± 0.14 ^a	10.57 ± 0.36 ^{bc}	7.75 ± 0.65 ^c	5.09 ± 0.59 ^{cd}
LLDPE	11.93 ± 0.07 ^b	7.57 ± 0.34 ^d	3.80 ± 0.43 ^d	1.16 ± 0.18 ^e
Carton	12.10 ± 0.06 ^a	11.47 ± 0.24 ^a	10.51 ± 0.26 ^a	8.38 ± 0.37 ^a

^{a-e}Means in the same column followed by a different superscript are significantly different ($P < 0.05$).

¹PET = polyethylene terephthalate; HDPE = high-density polyethylene; LDPE = low-density polyethylene; LLDPE = linear low-density polyethylene. Values shown ± SD.

Table 3. Oxygen transmission rates of packaging types over 24 h¹

Item	Package type ²					
	Glass	PET	LDPE	HDPE	LLDPE	Carton
Oxygen transmission rate ³	<0.5	5	195	72	1,009	801
Film/packaging wall thickness (mm)	1.1 ± 0.1	1.1 ± 0.1	1.3 ± 0.2	1.3 ± 0.2	0.019 ± 0.01	0.9 ± 0.2

¹Oxygen transmission rates were reported from package type manufacturer. Values shown ± SD.

²PET = polyethylene terephthalate; HDPE = high-density polyethylene; LDPE = low-density polyethylene; LLDPE = linear low-density polyethylene.

³Oxygen transmission rate (cc × mm/m² × 24 h × atm).

Table 4. Concentration of headspace oxygen (% O₂) in skim milk in different packages across 4 time points (d 0, 5, 10, 15)

Package type ¹	Time point			
	d 0	d 5	d 10	d 15
Glass	20.2 ± 0.3 ^b	18.6 ± 0.4 ^{cd}	18.5 ± 0.4 ^b	18.4 ± 0.3 ^b
PET	20.2 ± 0.2 ^b	18.4 ± 0.3 ^d	17.7 ± 0.3 ^c	17.5 ± 0.3 ^c
HDPE	19.7 ± 0.1 ^c	19.0 ± 0.4 ^c	17.8 ± 0.6 ^c	17.8 ± 0.7 ^c
LDPE	19.7 ± 0.1 ^c	18.6 ± 0.3 ^{cd}	17.7 ± 0.3 ^c	17.9 ± 0.4 ^c
LLDPE	20.8 ± 0.1 ^a	20.9 ± 0.2 ^a	20.9 ± 0.2 ^a	20.9 ± 0.3 ^a
Carton	20.6 ± 0.2 ^a	20.5 ± 0.3 ^b	20.6 ± 0.3 ^a	20.7 ± 0.3 ^a

^{a-d}Means in the same column followed by a different superscript are significantly different ($P < 0.05$).

¹PET = polyethylene terephthalate; HDPE = high-density polyethylene; LDPE = low-density polyethylene; LLDPE = linear low-density polyethylene. Values shown ± SD.

or cardboard off-flavors associated with paperboard cartons are detected by sensory panelists from d 1 to 5 of storage time (Leong et al., 1992; Karatapanis et al., 2006). Skim milk packaged in LLDPE had higher refrigerator/stale flavor than skim milk packaged in cartons across storage ($P < 0.05$; Table 6).

Whole milks packaged in paperboard carton and LLDPE had lower sweet aromatic flavor across storage time compared with milks filled into glass, PET, HDPE, or LDPE ($P < 0.05$; Table 7). Similar to skim milk, paperboard/cardboard flavor was only detected in milks packaged in paperboard cartons (Table 7). Unlike skim milk, this flavor was not detected until d 5, but also increased through d 15 (Table 7). This

effect has been previously documented in whole milk packaged in paperboard cartons by d 5 (Karatapanis et al., 2006). Refrigerator/stale flavor was detected in milks packaged in LLDPE at d 0 and in whole milk filled into paperboard carton milk by d 10, and this flavor increased with storage time (Table 7). Though skim and whole milk cannot be directly compared, paperboard/cardboard and refrigerator/stale flavors in milk packaged in paperboard cartons or LLDPE were generally more intense for skim milk at a specific time point compared with whole milk (Tables 6 and 7). This flavor difference between skim and whole milk is consistent with previous studies that off-flavors due to paperboard packaging were more pronounced in skim

Table 5. Concentration of headspace oxygen (% O₂) in whole milk in different packages across 4 time points (d 0, 5, 10, 15)

Package type ¹	Time point			
	d 0	d 5	d 10	d 15
Glass	20.2 ± 0.4 ^b	19.3 ± 0.3 ^b	19.3 ± 0.1 ^c	18.8 ± 0.5 ^c
PET	20.1 ± 0.4 ^{bc}	18.7 ± 0.1 ^c	18.6 ± 0.4 ^d	18.3 ± 0.3 ^d
HDPE	19.7 ± 0.7 ^c	19.0 ± 0.8 ^{bc}	18.5 ± 0.2 ^d	18.5 ± 0.3 ^{cd}
LDPE	19.7 ± 0.7 ^{bc}	18.8 ± 0.7 ^{bc}	19.0 ± 0.7 ^{cd}	18.5 ± 0.2 ^{cd}
LLDPE	20.9 ± 0.1 ^a	20.9 ± 0.2 ^a	21.0 ± 0.1 ^a	21.1 ± 0.1 ^a
Carton	20.2 ± 0.2 ^{bc}	20.5 ± 0.1 ^a	20.1 ± 0.3 ^b	20.3 ± 0.4 ^b

^{a-d}Means in the same column followed by a different superscript are significantly different ($P < 0.05$).

¹PET = polyethylene terephthalate; HDPE = high-density polyethylene; LDPE = low-density polyethylene; LLDPE = linear low-density polyethylene. Values shown ± SD.

Table 6. Trained panel profiles of skim milks filled into different packages with storage time (d 0, 5, 10, 15)

Time	Packaging type ¹	Sensory attribute ²							
		Overall aroma	Sweet aromatic	Cooked	Paperboard/ cardboard	Refrigerator/ stale	Sweet	Salty	Astringency
d 0	Glass	2.6 ^a	1.9 ^a	3.4 ^a	ND ³	ND	2.0 ^a	1.6 ^a	1.9 ^b
	PET	2.6 ^a	1.6 ^b	3.3 ^a	ND	ND	2.1 ^a	1.7 ^a	2.0 ^b
	HDPE	2.6 ^a	1.5 ^b	3.3 ^a	ND	ND	2.0 ^a	1.7 ^a	2.0 ^b
	LDPE	2.6 ^a	1.4 ^{bc}	3.3 ^a	ND	ND	2.0 ^a	1.5 ^a	2.0 ^b
	LLDPE	2.6 ^a	1.2 ^{cd}	3.2 ^{ab}	ND	0.5 ^d	2.1 ^a	1.7 ^a	2.0 ^b
	Carton	2.7 ^a	0.9 ^c	3.2 ^{ab}	1.3 ^c	ND	2.0 ^a	1.5 ^a	2.0 ^b
d 5	Glass	2.3 ^b	1.9 ^a	3.3 ^a	ND	ND	2.2 ^a	1.5 ^a	1.9 ^b
	PET	2.2 ^{bc}	1.5 ^b	3.3 ^a	ND	ND	2.0 ^a	1.7 ^a	1.9 ^b
	HDPE	2.0 ^{cd}	1.0 ^c	3.3 ^a	ND	ND	2.1 ^a	1.7 ^a	2.0 ^b
	LDPE	2.1 ^{bcd}	1.0 ^c	3.2 ^{ab}	ND	ND	2.1 ^a	1.6 ^a	2.0 ^b
	LLDPE	2.3 ^b	ND	3.0 ^b	ND	1.9 ^c	2.2 ^a	1.5 ^a	2.0 ^b
	Carton	2.2 ^{bc}	0.7 ^d	3.0 ^b	1.8 ^b	1.1 ^d	2.0 ^a	1.5 ^a	2.1 ^{ab}
d 10	Glass	2.1 ^{bcd}	1.5 ^b	3.3 ^a	ND	ND	2.0 ^a	1.7 ^a	1.8 ^b
	PET	2.1 ^{bcd}	1.0 ^c	3.2 ^{ab}	ND	ND	2.1 ^a	1.7 ^a	1.8 ^b
	HDPE	1.9 ^{cd}	0.8 ^{cd}	3.3 ^a	ND	ND	2.0 ^a	1.5 ^a	1.9 ^b
	LDPE	1.8 ^d	0.6 ^d	3.0 ^b	ND	ND	2.1 ^a	1.6 ^a	2.0 ^b
	LLDPE	2.3 ^b	ND	3.0 ^b	ND	2.4 ^b	2.0 ^a	1.5 ^a	2.2 ^{ab}
	Carton	2.2 ^{bc}	ND	3.0 ^b	2.0 ^b	1.9 ^c	2.0 ^a	1.5 ^a	2.2 ^{ab}
d 15	Glass	2.1 ^{bcd}	1.0 ^c	3.0 ^b	ND	ND	2.0 ^a	1.5 ^a	1.8 ^b
	PET	2.2 ^{bc}	0.9 ^{cd}	3.0 ^b	ND	ND	2.2 ^a	1.7 ^a	1.9 ^b
	HDPE	2.0 ^{cd}	ND	3.0 ^b	ND	ND	2.2 ^a	1.7 ^a	1.8 ^b
	LDPE	1.8 ^d	ND	3.0 ^b	ND	0.5 ^e	2.0 ^a	1.5 ^a	2.0 ^b
	LLDPE	2.6 ^a	ND	2.6 ^c	ND	3.4 ^a	2.0 ^a	1.5 ^a	2.1 ^{ab}
	Carton	2.6 ^a	ND	3.0 ^b	2.8 ^a	2.4 ^b	2.0 ^a	1.7 ^a	2.3 ^a

^{a-d}Means in the same column followed by a different superscript letter are significantly different ($P < 0.05$).

¹PET = polyethylene terephthalate; HDPE = high-density polyethylene; LDPE = low-density polyethylene; LLDPE = linear low-density polyethylene.

²Sensory attribute intensities were scored on a 0- to 15-point universal intensity scale (Meilgaard et al., 2007).

³ND = not detected.

milk compared with whole milk or low-fat milk (Leong et al., 1992; Sipple et al., 2020). Off-flavor problems in fluid milk due to package-related absorption or migration do increase with a decrease in container size due to the increase in the contact surface area with fluid milk. Comparisons have previously been made among various package sizes such as pint, quart, gallon, and liter (Bradley, 1980; Leong et al., 1992; van Aardt et al., 2001). Our packaging volume (230 mL) was selected to represent the largest contact area (surface area) or “worst-case” scenario. This volume is also the serving size for school lunch milk in the school lunch program in the United States (8 fl oz, 236 mL) and that milk is also typically served in a paperboard carton (Sipple et al., 2020).

Volatile Compound Analysis

Skim and whole milks packaged in paperboard cartons or LLDPE had higher concentrations of packaging/refrigerator taint volatile compounds styrene, acetophenone, and 2-ethyl-1-hexanol than milks filled into LDPE, HDPE, PET, or glass ($P < 0.05$; Tables 8, 9). Styrene concentrations increased with storage

across all packaging types except for the glass package. Styrene is a marker for degradation of plastic barriers over storage time (López et al., 2008), is present in refrigeration environments (Lozano et al., 2007a), and would be expected to migrate in from the environment with permeable containers such as cartons and LLDPE. Ethylbenzene is the main component in the production of styrene, and acetophenone is a byproduct of the oxidation of styrene and ethylbenzene (Azzara and Campbell, 1992; Ziegler, 1998; Vera et al., 2020). The more permeable barriers of paperboard cartons and LLDPE (Table 3) may be the cause of increased concentrations of styrene and acetophenone, which migrate into the milk from the refrigeration environment. The compound 2-ethyl-1-hexanol does occur naturally in some foods at low concentrations, but, industrially, it is used mainly used as a solvent for household cleaners and as a precursor to plasticizers (Vera et al., 2020). Plasticizers are substances that make materials (in this case plastic) more pliable or flexible (Vera et al., 2020). The compound 2-ethyl-1-hexanol is also known or considered to be an indoor air pollutant coming from the condensation of refrigerators or air-conditioners (Wakayama et al., 2019). Styrene is “generally rec-

Table 7. Trained panel profiles of whole milks filled into different packages with storage time (d 0, 5, 10, 15)

Time	Packaging type ¹	Sensory attribute ²								
		Overall aroma	Sweet aromatic	Milkfat	Cooked	Paperboard/ cardboard	Refrigerator/ stale	Sweet	Salty	Astringency
d 0	Glass	2.4 ^a	1.9 ^a	3.3 ^a	3.9 ^a	ND ³	ND	2.2 ^a	1.7 ^a	1.7 ^b
	PET	2.3 ^a	1.8 ^a	3.3 ^a	3.8 ^a	ND	ND	2.2 ^a	1.7 ^a	1.7 ^b
	HDPE	2.3 ^a	1.6 ^{ab}	3.2 ^a	3.8 ^a	ND	ND	2.0 ^a	1.5 ^a	1.6 ^b
	LDPE	2.2 ^{ab}	1.6 ^{ab}	3.2 ^a	3.8 ^a	ND	ND	2.2 ^a	1.7 ^a	1.7 ^b
	LLDPE	2.2 ^{ab}	1.2 ^{cd}	3.3 ^a	3.6 ^{ab}	ND	0.7 ^d	2.2 ^a	1.5 ^a	1.6 ^b
	Carton	2.3 ^a	1.4 ^{bc}	3.2 ^a	3.8 ^a	ND	ND	2.0 ^a	1.5 ^a	1.7 ^b
d 5	Glass	2.0 ^b	1.6 ^{ab}	3.2 ^a	3.7 ^{ab}	ND	ND	2.0 ^a	1.7 ^a	1.7 ^b
	PET	2.1 ^{ab}	1.5 ^{bc}	3.3 ^a	3.6 ^{ab}	ND	ND	2.2 ^a	1.7 ^a	1.7 ^b
	HDPE	2.0 ^b	1.1 ^{cd}	3.2 ^a	3.6 ^{ab}	ND	ND	2.3 ^a	1.5 ^a	1.8 ^b
	LDPE	2.0 ^b	1.1 ^{cd}	3.2 ^a	3.6 ^{ab}	ND	ND	2.2 ^a	1.7 ^a	1.8 ^b
	LLDPE	2.2 ^{ab}	0.6 ^e	3.0 ^a	3.0 ^e	ND	1.7 ^c	2.0 ^a	1.5 ^a	1.8 ^b
	Carton	2.0 ^b	0.9 ^{de}	3.2 ^a	3.4 ^b	1.2 ^c	ND	2.0 ^a	1.5 ^a	1.8 ^b
d 10	Glass	2.1 ^{ab}	1.6 ^{ab}	3.2 ^a	3.6 ^{ab}	ND	ND	2.0 ^a	1.5 ^a	1.8 ^b
	PET	2.1 ^{ab}	1.5 ^{bc}	3.3 ^a	3.5 ^b	ND	ND	2.2 ^a	1.7 ^a	1.8 ^b
	HDPE	2.1 ^{ab}	1.2 ^{cd}	3.0 ^a	3.4 ^b	ND	ND	2.2 ^a	1.7 ^a	1.8 ^b
	LDPE	2.1 ^{ab}	1.0 ^{de}	3.2 ^a	3.4 ^b	ND	ND	2.0 ^a	1.7 ^a	1.9 ^{ab}
	LLDPE	2.4 ^a	ND	3.3 ^a	3.0 ^e	ND	2.0 ^b	2.3 ^a	1.5 ^a	2.0 ^{ab}
	Carton	2.1 ^{ab}	0.6 ^e	3.0 ^a	3.4 ^b	1.7 ^b	0.5 ^d	2.0 ^a	1.5 ^a	2.1 ^{ab}
d 15	Glass	2.1 ^{ab}	1.5 ^{bc}	3.2 ^a	3.7 ^{ab}	ND	ND	2.2 ^a	1.7 ^a	1.7 ^b
	PET	2.0 ^b	1.3 ^{cd}	3.3 ^a	3.6 ^{ab}	ND	ND	2.2 ^a	1.5 ^a	1.8 ^{def}
	HDPE	2.0 ^b	1.0 ^{de}	3.2 ^a	3.4 ^b	ND	ND	2.2 ^a	1.7 ^a	1.8 ^{def}
	LDPE	2.0 ^b	1.6 ^{ab}	3.3 ^a	3.0 ^e	ND	ND	2.2 ^a	1.7 ^a	1.8 ^{de}
	LLDPE	2.3 ^a	ND	3.0 ^a	2.9 ^e	ND	2.5 ^a	2.0 ^a	1.5 ^a	2.0 ^{ab}
	Carton	2.1 ^{ab}	ND	3.2 ^a	2.9 ^e	2.2 ^a	1.5 ^c	2.0 ^a	1.5 ^a	2.2 ^a

^{a-f}Means in the same column followed by a different superscript letter are significantly different ($P < 0.05$).

¹PET = polyethylene terephthalate; HDPE = high-density polyethylene; LDPE = low-density polyethylene; LLDPE = linear low-density polyethylene.

²Sensory attribute intensities were scored on a 0- to 15-point universal intensity scale (Meilgaard et al., 2007).

³ND = not detected.

ognized as safe” by the Food and Drug Administration, though it is documented to cause a “chemical/plastic/stale” off-flavor in most food products when at detectable levels (Baner. 2000). Acetophenone is permitted by the Food and Drug Administration for human consumption (21CFR172, 2019) but is known to cause “stale” off-flavors in milk (Arnold et al., 1966) and 2-ethyl-1-hexanol has a “chemical/cleaning agent” aroma (Drake et al., 2014).

Limonene and p-cymene increased in skim milk packaged in LLDPE or paperboard cartons compared with LDPE, HDPE, PET, and glass. A similar increase was documented in whole milk packaged in LLDPE, but not paperboard carton (Tables 8, 9). Limonene is a compound found in fluid milk and is also used as a metric for assessment of environmental air quality (Subramanian et al., 2000). P-cymene is a degradation compound of limonene and correlates with the increase of limonene ($R^2 = 0.74$, $P < 0.05$). The increased concentrations of limonene and the further degradation into p-cymene may be due to the higher permeability of LLDPE and paperboard cartons (Tables 3, 8, 9). By d 5 of storage, toluene and p-xylene concentrations were higher in skim milks packaged in HDPE, LDPE,

LLDPE, and paperboard cartons compared with skim packaged in PET or glass (Table 8). Toluene and all 3 isomers of xylene (m-xylene, o-xylene, p-xylene) are used as solvents or intermediates in the production of polymers for plastics (Agency for Toxic Substances and Disease Registry, 2007, 2011). The degradation of plastics may be the cause for increased detection of toluene and p-xylene over time. These volatile compounds are likely contributors to stale/refrigerator flavor in skim and whole milks, consistent with Lozano et al. (2007a) who associated these compounds with refrigerator/stale flavor in stored butter.

Skim and whole milks filled into paperboard cartons had increased levels of hexanal compared with milks in other packaging types ($P < 0.05$; Tables 8, 9). This difference in hexanal concentration may signify the degradation of the surrounding paperboard packaging, which may increase hexanal concentrations in milk across time. Hexanal is a degradation compound formed from the oxidation of linoleic acid during the processing of wood extracts or paper pulp, and has been associated with off-flavor in food products packaged in paperboard and cardboard (Czerny and Buettner, 2009). Ziegler (1998) found that hexanal

Table 8. Selected volatile compounds in skim milk packaged in glass, HDPE, PET, LDPE, LLDPE, and paperboard carton at 4°C across 4 time points (d 0, 5, 10, 15)

Container ¹	Day of shelf life (time point)	Volatile compound ² (µg/kg)									
		Styrene	Toluene	P-xylene	P-cymene	Hexanal	Octanal	Nonanal	Limonene	Acetophenone	2-Ethyl-1-hexanol
Glass	0	ND ³	0.51 ^{ij} ± 0.10	ND	ND	1.49 ^j ± 0.41	ND	0.57 ^{lm} ± 0.37	ND	ND	3.09 ^j ± 1.16
	5	ND	1.11 ^h ± 0.03	ND	ND	3.26 ^{kl} ± 0.39	ND	1.96 ^{klm} ± 0.30	0.72 ^{hi} ± 0.03	ND	4.00 ⁱ ± 1.60
	10	ND	2.06 ^{ef} ± 0.31	0.46 ^h ± 0.02	ND	10.2 ^{gkl} ± 1.32	ND	2.42 ^{ijk} ± 0.36	0.98 ^{eg} ± 0.09	ND	4.11 ⁱ ± 1.05
	15	ND	1.26 ^{gh} ± 0.05	0.40 ^h ± 0.05	ND	22.1 ^e ± 1.99	0.92 ^e ± 0.49	3.86 ^{hjk} ± 0.10	0.88 ^{ghi} ± 0.04	0.51 ^h ± 0.03	4.16 ⁱ ± 1.35
PET	0	ND	0.44 ^j ± 0.07	ND	ND	1.35 ⁱ ± 0.10	ND	1.58 ^{lm} ± 0.10	ND	ND	1.05 ⁱ ± 0.29
	5	0.59 ^h ± 0.08	1.11 ^h ± 0.14	ND	ND	5.23 ^{ij} ± 0.52	ND	2.42 ^{ijk} ± 0.02	0.75 ^{ghi} ± 0.06	ND	4.70 ^{hi} ± 1.36
	10	ND	0.97 ^{hi} ± 0.07	0.44 ^h ± 0.01	0.41 ^h ± 0.01	29.7 ^d ± 0.83	0.79 ^{ef} ± 0.14	13.7 ^e ± 1.12	0.96 ^{gh} ± 0.04	ND	3.7 ^{fg} ± 0.16
	15	1.18 ^{gh} ± 0.11	1.68 ^{fg} ± 0.02	0.44 ^h ± 0.01	0.42 ^h ± 0.02	21.2 ^e ± 1.69	1.06 ^{de} ± 0.54	4.58 ^{lm} ± 0.26	0.87 ^{ghi} ± 0.04	0.72 ^{gh} ± 0.06	4.78 ^{hi} ± 0.24
HDPE	0	ND	0.97 ^{hi} ± 0.08	ND	ND	2.5 ^{kl} ± 0.63	0.78 ^{ef} ± 0.04	ND	ND	ND	3.72 ⁱ ± 0.25
	5	1.00 ^{gh} ± 0.10	2.87 ^d ± 0.19	0.89 ^f ± 0.06	0.85 ^f ± 0.02	12.8 ^{gh} ± 0.73	0.93 ^e ± 0.18	4.28 ^{lm} ± 0.63	0.48 ^k ± 0.12	0.81 ^{gh} ± 0.15	15.8 ^{efg} ± 0.35
	10	0.99 ^{gh} ± 0.10	2.42 ^{de} ± 0.20	1.15 ^e ± 0.08	0.96 ^f ± 0.03	8.48 ^{ghi} ± 0.24	0.87 ^e ± 0.21	7.12 ^g ± 0.34	1.30 ^d ± 0.10	1.07 ^g ± 0.20	15.4 ^{fg} ± 2.79
	15	1.40 ^{gh} ± 0.14	4.51 ^b ± 0.07	1.92 ^c ± 0.14	1.71 ^d ± 0.03	15.3 ^{deg} ± 1.29	1.36 ^d ± 0.39	4.69 ^h ± 0.13	1.02 ^{ef} ± 0.02	1.62 ^f ± 0.14	13.4 ^{gh} ± 2.70
LDPE	0	ND	1.62 ^{fg} ± 0.15	0.85 ^{fg} ± 0.09	ND	1.45 ⁱ ± 0.23	ND	1.52 ^{lm} ± 0.28	0.41 ^j ± 0.05	ND	1.24 ⁱ ± 1.02
	5	0.77 ^h ± 0.03	2.53 ^{de} ± 0.40	1.28 ^{de} ± 0.13	0.47 ^h ± 0.04	8.89 ^{ghi} ± 0.20	0.61 ^f ± 0.07	7.73 ^g ± 0.08	0.70 ⁱ ± 0.27	0.62 ^h ± 0.13	15.4 ^{fg} ± 1.01
	10	1.17 ^{gh} ± 0.07	1.91 ^f ± 0.03	1.39 ^d ± 0.15	0.68 ^g ± 0.02	1.61 ⁱ ± 0.32	1.44 ^d ± 0.19	2.05 ^{klm} ± 0.16	1.24 ^{de} ± 0.06	1.09 ^g ± 0.16	24.2 ^e ± 2.38
	15	2.21 ^f ± 0.14	4.91 ^{ab} ± 0.46	3.16 ^a ± 0.10	0.91 ⁱ ± 0.05	12.9 ^{gh} ± 1.12	1.38 ^d ± 0.17	10.8 ^f ± 0.56	1.01 ^{ef} ± 0.11	1.66 ^f ± 0.24	19.8 ^f ± 3.55
LLDPE	0	1.75 ^{fg} ± 0.39	0.90 ^{hij} ± 0.15	ND	ND	1.61 ⁱ ± 0.62	ND	4.44 ^{lm} ± 2.41	0.40 ^j ± 0.07	ND	1.06 ⁱ ± 0.12
	5	8.93 ^e ± 0.71	2.77 ^d ± 0.06	0.62 ^{gh} ± 0.09	1.29 ^c ± 0.12	9.86 ^{ghi} ± 0.43	0.44 ^g ± 0.06	19.1 ^d ± 1.97	0.89 ^{ghi} ± 0.15	3.20 ^d ± 0.30	36.4 ^d ± 4.52
	10	12.3 ^d ± 1.09	2.06 ^{ef} ± 0.21	1.38 ^{de} ± 0.18	1.90 ^c ± 0.09	10.9 ^{ghi} ± 2.24	ND	9.03 ^{fg} ± 0.89	2.19 ^c ± 0.22	5.65 ^c ± 0.35	59.1 ^c ± 11.36
	15	15.3 ^b ± 0.28	5.29 ^a ± 0.26	2.99 ^a ± 0.11	2.88 ^a ± 0.01	18.4 ^{ef} ± 3.24	0.52 ^g ± 0.01	34.6 ^a ± 1.98	4.48 ^a ± 0.11	20.0 ^a ± 0.68	119 ^a ± 12.34
Carton	0	0.74 ^h ± 0.09	0.53 ^{ij} ± 0.10	ND	ND	7.17 ^{hijk} ± 1.16	ND	0.70 ^{lm} ± 0.38	ND	ND	2.81 ⁱ ± 2.54
	5	11.7 ^d ± 0.99	3.42 ^c ± 0.60	0.78 ^{fg} ± 0.04	1.28 ^c ± 0.09	133 ^b ± 7.37	4.71 ^a ± 0.50	24.1 ^c ± 2.25	0.80 ^{ghi} ± 0.24	1.57 ^f ± 0.17	60.3 ^c ± 5.90
	10	13.9 ^c ± 1.09	3.67 ^c ± 0.25	1.27 ^{de} ± 0.11	1.87 ^c ± 0.08	115 ^c ± 9.25	2.78 ^b ± 0.21	17.2 ^e ± 1.41	1.28 ^d ± 0.12	2.24 ^e ± 0.04	70.7 ^b ± 5.28
	15	18.5 ^a ± 1.00	4.90 ^{ab} ± 0.43	2.37 ^b ± 0.41	2.72 ^b ± 0.16	159 ^a ± 12.9	2.22 ^c ± 0.15	28.8 ^b ± 2.48	3.78 ^b ± 0.32	6.54 ^b ± 0.34	58.3 ^c ± 1.36

^{a-m}Means in the same column followed by a different superscript are significantly different ($P < 0.05$).¹PET = polyethylene terephthalate; HDPE = high-density polyethylene; LDPE = low-density polyethylene; LLDPE = linear low-density polyethylene. Values shown ± SD.²Volatile compounds were quantified using 6-point standard curves ($R^2 > 0.95$).³ND = not detected.

Table 9. Volatile compounds in whole milk packaged in glass, HDPE, PET, LPDE, LLDPE, and paperboard carton at 4°C across 4 time points (d 0, 5, 10, 15)

Container ¹	Day of shelf life (time point)	Volatile compound ² (µg/kg)									
		Styrene	Toluene	P-xylene	P-cymene	Hexanal	Octanal	Nonanal	Limonene	Acetophenone	2-Ethyl-1-hexanol
Glass	0	ND ³	0.82 ^{kl} ± 0.04	ND	ND	0.84 ^l ± 0.13	ND	2.85 ^{bc} ± 0.93	7.74 ^b ± 0.51	ND	ND
	5	ND	0.98 ^{jik} ± 0.26	ND	ND	1.30 ^{ij} ± 0.17	ND	1.24 ^{efg} ± 0.47	3.04 ^{cde} ± 0.70	ND	0.71 ^{de} ± 0.28
	10	ND	1.26 ^{ghi} ± 0.14	ND	ND	1.38 ^f ± 0.46	ND	ND	2.67 ^{def} ± 0.41	ND	0.46 ^e ± 0.09
	15	ND	1.44 ^{eg} ± 0.23	ND	ND	3.26 ^{defgh} ± 0.89	ND	ND	2.32 ^{def} ± 0.09	ND	0.41 ^e ± 0.01
PET	0	ND	0.66 ^{lm} ± 0.04	ND	ND	2.98 ^{efgh} ± 0.30	ND	1.64 ^{de} ± 0.19	7.45 ^b ± 0.23	ND	ND
	5	ND	0.62 ^{lm} ± 0.14	ND	ND	3.17 ^{efgh} ± 1.65	ND	0.66 ^{gh} ± 0.21	1.69 ^{ef} ± 0.22	ND	ND
	10	ND	0.78 ^{kl} ± 0.03	ND	ND	4.11 ^{de} ± 0.54	0.51 ^a ± 0.16	0.75 ^{efgh} ± 0.34	2.08 ^{def} ± 0.04	ND	0.83 ^d ± 0.17
	15	ND	0.85 ^{kl} ± 0.09	ND	ND	2.28 ^{efgh} ± 1.06	ND	ND	1.65 ^{ef} ± 0.13	ND	ND
HDPE	0	ND	0.69 ^{lm} ± 0.05	ND	0.48 ^c ± 0.03	2.14 ^{ghij} ± 0.14	ND	0.80 ^{efgh} ± 0.05	8.44 ^b ± 0.83	ND	ND
	5	ND	ND	ND	ND	1.87 ^{hij} ± 0.36	ND	ND	1.48 ^{ef} ± 0.19	ND	ND
	10	0.69 ^f ± 0.11	1.04 ^{hij} ± 0.18	ND	ND	3.68 ^{defg} ± 0.57	ND	0.64 ^{gh} ± 0.34	2.11 ^{def} ± 0.32	ND	0.52 ^{de} ± 0.07
	15	ND	1.35 ^{efgh} ± 0.09	ND	ND	4.89 ^f ± 0.34	ND	0.57 ^{gh} ± 0.08	2.01 ^{def} ± 0.06	ND	1.32 ^e ± 0.09
LDPE	0	ND	1.67 ^c ± 0.12	ND	ND	2.92 ^{efgh} ± 0.17	ND	1.52 ^{def} ± 0.16	8.89 ^{ab} ± 0.32	ND	ND
	5	0.53 ^f ± 0.15	1.58 ^{ef} ± 0.22	ND	ND	3.34 ^{defgh} ± 0.60	ND	0.60 ^{gh} ± 0.04	2.56 ^{def} ± 0.39	ND	ND
	10	0.63 ^f ± 0.08	3.64 ^b ± 0.08	0.42 ^e ± 0.04	ND	3.94 ^{def} ± 1.22	ND	1.00 ^{efg} ± 0.13	3.01 ^{cde} ± 0.40	0.46 ^{de} ± 0.07	0.45 ^e ± 0.15
	15	0.47 ^f ± 0.25	5.09 ^a ± 0.31	0.95 ^c ± 0.19	ND	2.57 ^{efgh} ± 0.18	ND	0.85 ^{efgh} ± 0.35	3.12 ^{cde} ± 0.40	ND	ND
LLDPE	0	2.11 ^e ± 0.05	1.53 ^{ef} ± 0.09	ND	0.66 ^b ± 0.05	3.54 ^{defgh} ± 0.45	0.36 ^b ± 0.10	2.27 ^{cd} ± 0.26	9.41 ^{ab} ± 0.19	0.98 ^d ± 0.04	0.62 ^{de} ± 0.19
	5	3.39 ^c ± 0.46	1.18 ^{ghi} ± 0.12	0.40 ^e ± 0.08	0.51 ^c ± 0.08	2.78 ^{efgh} ± 0.47	ND	2.67 ^c ± 0.49	2.83 ^{def} ± 0.29	2.64 ^c ± 0.40	1.62 ^c ± 0.12
	10	7.45 ^b ± 0.74	2.05 ^d ± 0.33	1.59 ^b ± 0.28	0.94 ^a ± 0.10	4.09 ^{de} ± 0.42	ND	3.72 ^b ± 0.55	3.78 ^{cd} ± 0.80	6.63 ^b ± 0.83	8.06 ^a ± 0.22
	15	12.7 ^a ± 0.38	2.41 ^c ± 0.27	2.16 ^a ± 0.20	0.94 ^a ± 0.16	3.54 ^{efgh} ± 0.54	ND	7.88 ^a ± 1.63	4.83 ^c ± 0.58	8.95 ^a ± 1.61	7.67 ^b ± 0.60
Carton	0	0.71 ^f ± 0.02	0.69 ^{klm} ± 0.01	ND	ND	6.85 ^c ± 0.39	ND	1.28 ^{efg} ± 0.17	10.8 ^a ± 4.49	ND	ND
	5	0.88 ^f ± 0.01	0.45 ^m ± 0.04	ND	ND	7.07 ^c ± 0.23	ND	0.87 ^{efg} ± 0.28	0.96 ^f ± 0.06	ND	0.46 ^e ± 0.07
	10	2.66 ^d ± 0.32	1.40 ^{efg} ± 0.12	0.60 ^d ± 0.06	ND	13.2 ^b ± 1.77	ND	0.69 ^{efg} ± 0.29	1.86 ^{def} ± 0.15	1.05 ^d ± 0.11	0.69 ^{de} ± 0.11
	15	3.50 ^c ± 0.09	1.43 ^{efg} ± 0.06	0.62 ^d ± 0.05	ND	19.2 ^a ± 2.19	ND	0.99 ^{efg} ± 0.20	2.13 ^{def} ± 0.10	1.59 ^d ± 0.24	1.71 ^c ± 0.15

^{a-m}Means in the same column followed by a different superscript are significantly different ($P < 0.05$).¹PET = polyethylene terephthalate; HDPE = high-density polyethylene; LDPE = low-density polyethylene; LLDPE = linear low-density polyethylene. Values shown ± SD.²Volatile compounds were quantified using 6-point standard curves ($R^2 > 0.95$).³ND = not detected.

can be used as a guide for tracking lipid oxidation of paperboard across time. Both of these studies and others have found the association of increased concentrations of aldehydes such as hexanal, heptanal, octanal, nonanal, and decanal to an increase in cardboard/paperboard flavor (Czerny and Buettner, 2009; Whitson et al., 2010; Van Caelenberg et al., 2013). The elevated level of hexanal in milks in paperboard cartons compared with whole and skim milks in the other 5 packaging types may be attributed to hexanal being the largest source of paperboard or cardboard off-flavor (Tables 8, 9). Cardboard flavor in dairy products has also previously been sourced to lipid oxidation compounds including hexanal (Whitson et al., 2010). Skim milk packaged in paperboard cartons or LLDPE and whole milk packaged in LLDPE also had higher concentrations of nonanal compared with milks filled into other package types ($P < 0.05$; Table 8). This oxidation compound may indicate elevated lipid oxidation in milks packaged in these containers, indicative of increased oxygen and greater permeability of these package types compared with the others in this study. Dynamic headspace analysis conducted on commercial milks revealed that milks with higher concentrations of accessible oxygen were correlated with higher concentrations of lipid oxidation compounds (Kim and Morr, 1996). Increases in lipid oxidation were also documented in ultrapasteurized and HTST milks stored at 4°C for 14 d (Jo et al., 2018). Karatapanis et al. (2006) also documented lipid oxidation in HTST whole milk across 7 d of refrigerated storage (Karatapanis et al., 2006). Concentrations of volatile compounds were generally lower in whole milk compared with skim milk (Tables 8, 9). This result may signify that packaging-related compounds may be soluble in milkfat and thus less available for headspace extraction, as well as sensory perception, because refrigerator/stale and cardboard flavors by sensory analysis were also less prevalent in whole milks compared with skim milks. In a previous study, fluid milk with higher fat content had lower recoveries of most volatile compounds via headspace extraction compared with skim milk (Jo et al., 2018).

Skim milk packaged in paperboard carton or LLDPE were closely aligned across storage time with increasing concentrations of packaging-related volatile compounds styrene, toluene, p-xylene, p-cymene, limonene, acetophenone, 2-ethyl-1-hexanol, and hexanal (Figure 1). Skim milk packaged in LLDPE or paperboard carton after d 5 of storage were distinct from milks filled into glass, LDPE, HDPE, or PET (Figure 1). Whole milks were not as distinct in volatile compound differences compared with skim milks but whole milk packaged in LLDPE was closely aligned across storage time, with

increasing concentrations of packaging-related volatile compounds styrene, toluene, p-xylene, p-cymene, acetophenone, 2-ethyl-1-hexanol, and hexanal (Figure 2). Whole milk packaged in LLDPE after d 5 was distinct from milk filled into glass, LDPE, HDPE, and PET (Figure 2). Karatapanis et al. (2006) reported slight increases in packaging/storage volatiles in the low part per billion ($\mu\text{g}/\text{kg}$) for toluene and various benzene compounds (ethyl benzene, 1,4-dimethyl benzene, 1,3-dimethyl benzene, 1,3,5-trimethyl benzene) in whole HTST milk across 7 d of refrigerated storage time.

Relationship Between Volatile Compounds and Sensory Attributes

Principal component biplots provide a visualization of the changes in the volatiles and sensory attributes that characterize differences among the milks with storage time, along with correlations between sensory attributes and volatile compounds (Figures 1, 2). All milks decreased in cooked/milky and sweet aromatic flavors with storage time, with milks filled into paperboard cartons or LLDPE having the most distinct storage-related changes (Figure 1). Paperboard/cardboard flavor and the volatile compound hexanal were correlated ($R^2 = 0.84$, $P < 0.05$; Figure 1). Refrigerator stale flavor was correlated with the volatile compounds styrene ($R^2 = 0.88$, $P < 0.05$), p-cymene ($R^2 = 0.73$, $P < 0.05$), acetophenone ($R^2 = 0.71$, $P < 0.05$), and 2-ethyl-1-hexanol ($R^2 = 0.82$, $P < 0.05$). Styrene has also been attributed to “refrigerator/stale” flavor in stored butter (Lozano et al., 2007a). Similar to skim milk, whole milk filled into cartons was associated with paperboard/cardboard flavor and astringency, and the volatile compound hexanal (Figure 2). Whole milk packaged in LLDPE and paperboard cartons were also associated with higher overall aroma and refrigerator stale flavor compared with whole milk in other package materials (Figure 2).

DSE-SAFE

Skim and whole milk packaged in paperboard cartons had distinct paperboard and refrigerator stale flavors. Significant migration of volatile compounds associated with package or refrigerator taint compounds, or both, was documented at d 15 in whole and skim milk in paperboard cartons (whole and skim milk) by HS-SPME-GCMS (Tables 8, 9). Headspace volatile analysis of skim milk filled in paperboard cartons at d 15 revealed traces of plasticizers and ink-related compounds at detectable limits, but not above base line noise and, thus, not quantifiable (results not shown). Direct

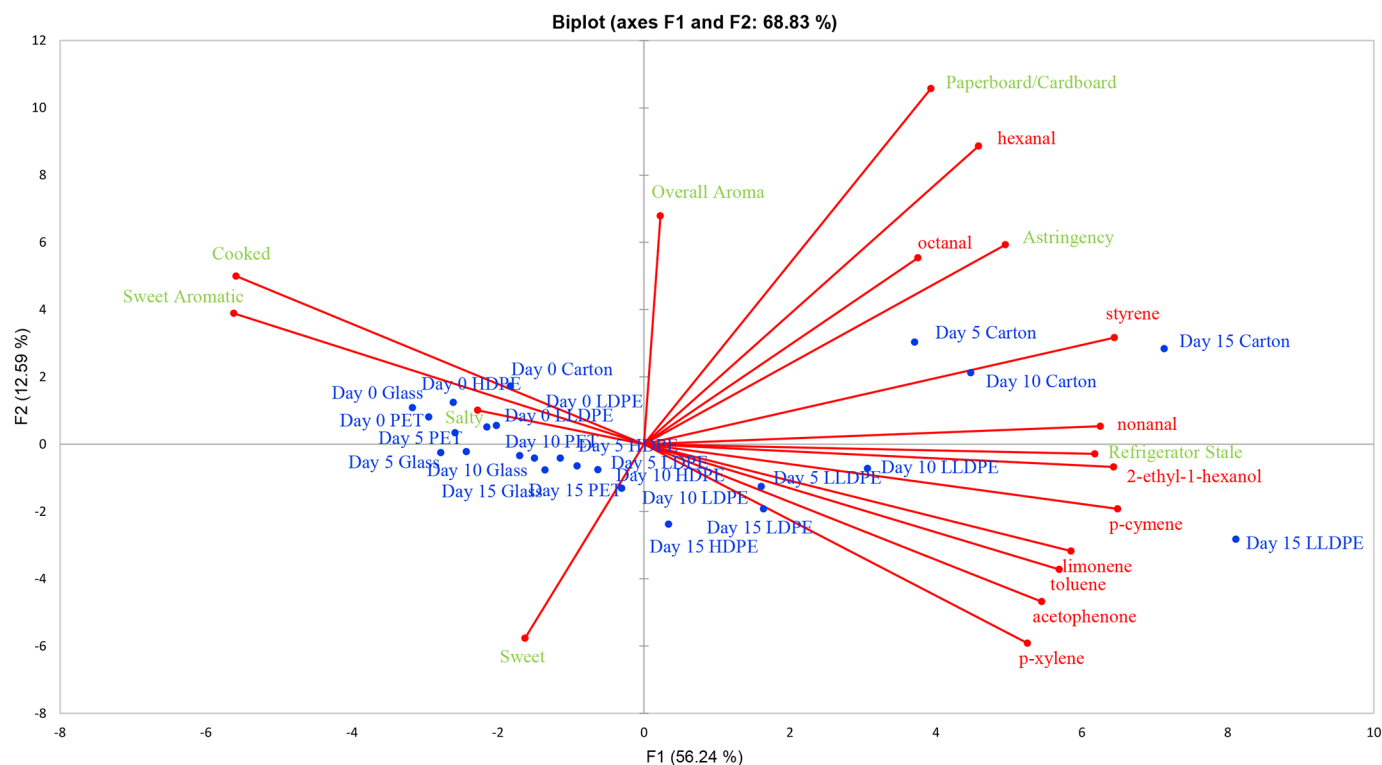


Figure 1. Principal component biplot of volatile compound concentrations and trained panel sensory attribute intensities of skim milk packaged in glass, high-density polyethylene (HDPE), polyethylene terephthalate (PET), low-density polyethylene (LDPE), linear low-density polyethylene (LLDPE), or paperboard cartons at 4°C across 4 time points (d 0, 5, 10, 15).

solvent extraction was subsequently applied to skim milks packaged in paperboard or glass to facilitate an increased recovery of these specific paperboard-related volatile compounds.

Taint compounds associated with paperboard packaging that were recovered from skim milks by HS-SPME-GCMS were also recovered by DSE-SAFE, as expected. Relative abundance of package/refrigeration-related compounds in skim milk stored in paperboard cartons versus glass for 15 d by DSE-SAFE were ($\mu\text{g}/\text{kg}$) styrene 283 ± 23.3 versus 1.7 ± 0.7 , toluene 930 ± 85.7 versus 8.6 ± 0.5 , p-xylene 46 ± 12.4 versus 3.6 ± 2.1 , acetophenone 38.7 ± 13.9 versus not detected, and 2-ethyl-1-hexanol 287.3 ± 8.19 versus 25.1 ± 0.90 ($P < 0.05$). These values were higher in skim milk

by SAFE extraction compared with headspace extraction (SPME-GCMS) milk from paperboard carton, as expected, but it is also important to note that these values from SAFE extraction were calculated based on internal standard recovery rather than actual quantitation with standard curves (SPME-GCMS) and, thus, do not represent absolute concentrations. More importantly, as expected, additional compounds were detected in skim milk by DSE-SAFE extraction (but not by SPME-GCMS) including n-propylbenzene (dye solvent), benzophenone (photoinitiators, plasticizer), dibutyl phthalate, and diethyl phthalate (plasticizers and softening agents; Frostling et al., 1984; Czerny and Buettner, 2009; Leja and Lewandowicz, 2010). The compounds n-propylbenzene, benzophenone, dibutyl

Table 10. Consumer tetrad difference tests of skim milk in 3 package types (paperboard, PET, and HDPE) compared with the control (glass) after 10 d at 4°C

Packaging comparison ¹	n	Fluid skim milk	
		No. correct	Significant at $\alpha = 0.05$
Paperboard carton vs. glass (control)	101	56	Yes
PET vs. glass (control)	103	28	No
HDPE vs. glass (control)	103	36	No

¹PET = polyethylene terephthalate; HDPE = high-density polyethylene.

Table 11. Consumer tetrad difference tests of whole milk in 3 package types (paperboard, PET, and HDPE) compared with the control (glass) after 10 d at 4°C

Packaging comparison ¹	n	Fluid whole milk	
		No. correct	Significant at $\alpha = 0.05$
Paperboard carton vs. glass (control)	104	47	Yes
PET vs. glass (control)	103	39	No
HDPE vs. glass (control)	103	37	No

¹PET = polyethylene terephthalate; HDPE = high-density polyethylene.

phthalate, and diethyl phthalate were detected in skim milk packaged in paperboard cartons at 210 ± 28.6 , 6 ± 0.3 , 56 ± 18 , and 300 ± 1.79 parts per billion ($\mu\text{g}/\text{kg}$), respectively, via relative abundance. Comparatively, these compounds were not detected in skim milk packaged in glass. The compound n-propylbenzene is a compound found in plastic packaging for milk (Abrantes, 1993), and benzophenone was previously used as a photoinitiator to cure ink on packaging materials (21CFR172: US FDA, 2019a; 21CFR177: US FDA, 2019b). Benzophenone and n-propylbenzene have been previously found in skim milk powders (Shiratsuchi

et al., 1994; Sanches-Silva et al., 2008), possibly due to these products being packaged in plastic and using photoinitiators to cure the ink on the packages. Diethyl phthalate is classified as a plasticizer by the FDA for specific use in food (21CFR181.27: US FDA, 2019c). Dibutyl phthalate and diethyl phthalate have been previously found in milk and other dairy products (Sørensen, 2006; Fierens et al., 2013). Determination of exact concentrations of these specific taint compounds in skim and whole milks filled into paperboard or other package types, or both, at larger package volumes merits its future work.

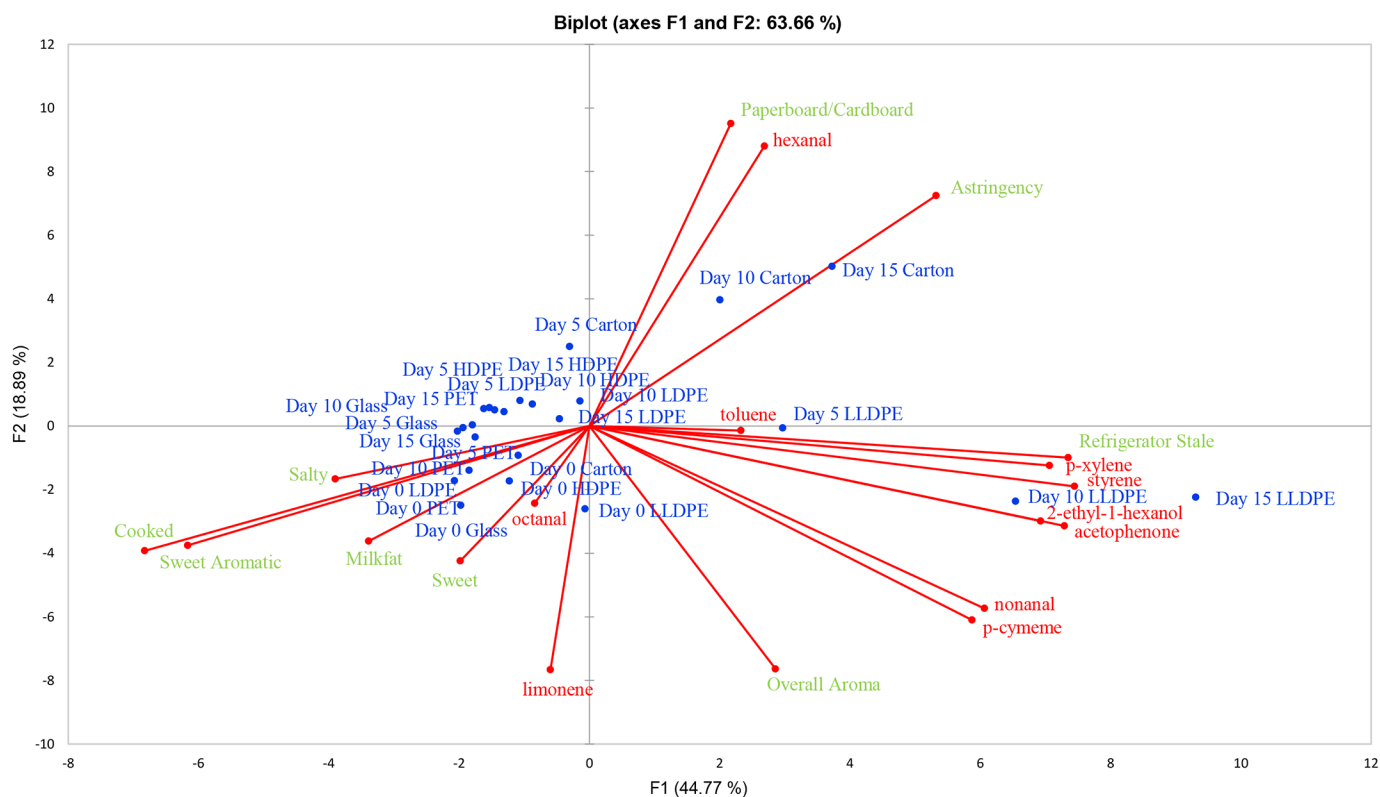


Figure 2. Principal component biplot of volatile compound concentrations and trained panel sensory attribute intensities of whole milk packaged in glass, high-density polyethylene (HDPE), polyethylene terephthalate (PET), low-density polyethylene (LDPE), linear low-density polyethylene (LLDPE), or paperboard cartons at 4°C across 4 time points (d 0, 5, 10, 15).

Consumer Difference Tests

Consumers detected differences between skim and whole milk filled into paperboard cartons and glass ($P < 0.05$; Tables 10, 11). Consistent with minimal to no differences by trained panels, consumers could not detect differences between the PET and glass pairings and HDPE and glass ($P > 0.05$; Tables 10, 11). These results are consistent with a previous study, where consumers reported similar flavor and acceptability of milks packaged in PET and HDPE (Potts et al., 2017). The results are also consistent with our trained descriptive panel and volatile compound results. Skim or whole milks filled into PET, HDPE or glass were similar in overall flavor profile with no paperboard/cardboard or refrigerator stale off-flavors present by d 15 (Tables 6, 7).

Paperboard cartons are the most widely used packaging type for school meal (breakfast and lunch) programs in the United States, and milks packaged in paperboard cartons showed distinct off-flavors and presence of specific migration volatile compounds. Our findings suggest that industry and policy makers should seek new package alternatives for school meal fluid milk. The consequences of using fluid milk packaging that contributes significant off-flavors, over time, may affect how young children, and those children as adults, perceive fluid milk.

CONCLUSIONS

Off-flavors due to packaging and refrigeration environment were detected in fluid skim and whole milk over refrigerated storage time. Off-flavors in milks detected by sensory analysis were correlated with increased specific migration volatile compounds. Milks packaged in paperboard cartons and LLDPE had the highest intensities of off-flavors, due to permeability and migration, with off-flavors present by day 0 in skim milk. Consumer difference tests were consistent with trained panel and volatile compound analysis, suggesting that HTST milks packaged in HDPE, PET, or glass in the absence of light exposure have no discernable sensory differences by day 10 post-processing. In contrast, milks filled into paperboard cartons were differentiated by consumers compared to milks filled into glass.

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