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QUANTIFICATION OF VOLATILE FLAVOR COMPOUNDS IN OFF-FLAVOR AND COMMERCIAL REDUCED-FAT MILK SAMPLES

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Summary

Various chemical compounds contribute to the naturally pleasant flavor of milk. Over time, however, and with unwanted chemical reactions, loss of flavor is inevitable. This study was conducted to identify and quantify volatile flavor compounds associated with off-flavored and commercial reduced-fat milk products. Fresh milk was used for the preparation of altered milk samples having off-flavors such as "light-oxidized" and "high-acid." Milk lacking freshness (i.e., milk produced two weeks before sampling and maintained at 40°F in the dark) also was compared with fresh unaltered milk and two commercial milk samples. For headspace analysis, milk samples were subjected to SPME-GC for volatile compound identification. In addition, the composition and aerobic and coliform microbial counts for all milk samples were analyzed. The milk samples did not differ in the concentrations of volatile flavor constituents. When comparing "light-oxidized" milk samples (200 lx exposure for 1 or 3 hr), 2-butanone and pentanal concentrations tended to increase as light exposure time increased. All milk samples had similar fat and total solids contents. "High-acid" milk had a greater total aerobic microbe count than the other milk samples. Fresh milk had a greater octanal concentration than the off-flavored reduced-fat milk samples did. This might indicate that octanal is an important contributor to fresh milk flavor and deserves further study.

(Key Words: Milk, Flavor, Compounds, GC Analysis.)

Introduction

Nutrition and flavor are the main reasons people consume milk and find milk acceptable. Although survey results show that milk consumption is increasing, children's milk consumption has dropped by 30% in the past 30 years. The beverages that children are preferentially consuming include soft drinks, juice, and other fruit drinks. Nutritionists indicate that, for adults, diets without milk and other dairy products generally are deficient in calcium. The flavor of milk is typically bland and, therefore, susceptible to flavor changes caused by enzymes, microbial contamination, and other catalysts. Off flavors found in commercial milk products include "high-acid," "light-oxidized," and "lacks freshness." Chemical compounds associated with these off flavors include: lactic acid for "high-acid"; heptanal, 2-heptanone, hexanal, nonanal, octanal, pentanal, and propanal for "light-oxidized"; and dimethyl sulfide, 1-butanol, 2-heptanone, hexanal, and pentanal for "lacks freshness." Hundreds of chemical compounds and their combinations are responsible for milk flavor perceived by consumers. Thus, the objectives of this study were to identify and quantify volatile flavor compounds associated with off-flavored and commercial reduced-fat milk products.

Experimental Procedures

Reduced-fat milk (3.776 L) was obtained within 2 d of production from the Kansas State University dairy plant, Manhattan, Kansas, during November and December of 2003. Milk samples were prepared to contain various off flavors, such as "light-oxidized" and "high-acid" as described in Table 1. A "lacks freshness" milk sample (Kansas State University dairy plant), two commercial milk samples (local supermarket brand purchase (Manhattan, KS), and school lunch milk packaged in a 250-mL paperboard carton) were included.

Milk samples were analyzed for headspace volatile compounds, total solids, fat content, total plate count, and apparent viscosity. For solid-phase microextraction (SPME) analysis, 75-µm Carboxen-PDMS fiber sampling at 140°F for 30 min was used to collect volatile headspace compounds from the milk. These compounds were subsequently quantified by gas chromatography, flame ion detection (GC-FID). For total solids, an atmospheric oven method was used. Fat was measured by the Babcock method and total aerobic and coliform microbial counts were done by standardized methods. Apparent viscosity was determined at a frequency of 2 Hz within a shear rate range 1 to 40 second^{-1} by using a rheometer at 40°F. Apparent viscosity values were compared at 10 second^{-1}.

An incomplete-block design was used for this experiment because sampling restrictions existed. Only four milk samples were compared per day. On one day, fresh unaltered milk, as well as the three induced off-flavored (light-oxidized at 1 hr, light-oxidized at 3 hr, and high-acid) milk samples were analyzed. On the other day, fresh unaltered milk, two commercial milk samples, and a "lacks freshness" milk sample were analyzed. The entire experiment was replicated twice during November-December, 2003. Data were subjected to analysis of variance, and significant differences among means were detected by Tukey's pair-wise comparison at a significance level of $\alpha = 0.05$.

Results and Discussion

Table 2 displays the results of the chemical and microbial analyses conducted on the seven milk samples. No significant differences were detected for fat or total-solids contents among the seven milk samples. Fat content ranged from 1.8 to 2.1%, and total solids ranged from 10.4 to 11.4%.

Total aerobic microbe counts were different ($P<0.05$) among samples. The "high-acid" milk had greater counts than the other milk samples did (Table 2). These results indicated that, despite different raw-milk sources, milk-distribution systems, and age of milk, six of the seven milk samples had similar aerobic microbe counts.

All coliform microbe counts were <1 CFU/mL for the seven milk samples. Apparent-viscosity results indicated that the various milk samples differed with respect to viscosity (range of 3.35 to 3.57 mPa·s). Mean differences are shown in Table 2; no apparent overall trend was observed.

Ten volatile flavor compounds (benzaldehyde, 2-butanone, ethyl caproate, heptanal, 2-heptanone, hexanal, octanal, 1-octen-3-ol, pentanal, and 2-pentylfuran) were quantified in all seven milk samples. Figure 1 shows the concentrations of indi-

vidual compounds in fresh unaltered milk, compared with those in the various altered milk samples. Figure 2 shows the concentrations of the individual compounds for fresh unaltered milk compared with those in the commercial and "lacks freshness" milks. Overall, mean compound concentrations ranged from 0 mg/kg (1-octen-3-ol in fresh unaltered, "light-oxidized" 1 hour and 3 hours, and commercial milk samples) to 1.962 mg/kg (2-butanone in "high-acid" milk). All ten of these compounds have been reported to contribute to milk flavor. Ranges for these compounds in the milk samples are shown in Table 3. The highest concentrations were associated with the "high-acid," commercial 1, or commercial 2 milk samples.

There are several possible reasons why no differences were observed for any of the ten compounds measured in the seven milk samples. Sampling constraints that included two commercial samples, as well as a milk sample close to its shelf-removal date, resulted in greater variation among samples, which, in turn, decreased the chances of detecting differences. Also, milk samples that were made to have "slight" to "definite" intensity changes seemed to have made fewer differences than initially hypothesized.

Stored milk has greater concentrations of heptanal, hexanal, and pentanal than fresh, non-aged milk does. We observed a similar trend in which the "lacks freshness" (14 days old) milk sample had slightly greater concentrations of heptanal, hexanal, and pentanal than the fresh unaltered milk did (2-days old; Figure 2). As a result, the rate of flavor change may not have been as quick in our study as in previously reported studies. Maintenance of a constant temperature or minimal light exposure, might have slowed the reaction rates. Greater heptanal, hexanal, and pentanal concentrations have been reported in "light-oxidized" milk compared with those in fresh, unaltered milk, but the induced light-oxidation conditions in those studies included long exposure times (18 hours) or unknown light intensities.

All seven milk samples were evaluated simultaneously by a trained descriptive panel for flavor characteristics. Panel results indicated that no differences existed for the "light-oxidized" trait among the fresh unaltered milk, milk "light-oxidized" for 1 hour, and milk "light-oxidized" for 3 hours, which agrees with our data for volatile compound concentration for these three samples. The two commercial and high-acid milk samples were rated as having some of the highest flavor-intensity scores. It is interesting to note that the greatest concentration of all of the ten compounds was associated with one of these three milk samples. This may suggest that a "good" milk flavor may be an optimum blend of specific compounds of this type.

Purposely altering milk samples did not significantly affect chemical compound concentration, nor did a trained panel detect differences among the purposely altered "light-oxidized" and the fresh, unaltered milk samples. Either the two commercial milk samples or the "high-acid" milk samples had the greatest concentration of the ten identified volatile compounds. This may suggest that high-quality milk flavor may be an optimal sum of a variety of compounds. Further work is needed to determine the importance of octanal concentration, as well as its relationship to other compounds that may contribute and influence fresh milk flavor.

Table 1. Milk Samples: Source and Preparation

Milk sample	Treatment	Holding time
Fresh unaltered[1]	None	None
High-acid[1]	Remove 100 mL reduced fat milk. Add 52 mL cultured lowfat buttermilk[2]	None
Lacks freshness[1]	None	14 d at 40°F
Light-oxidized at 1 h[1]	200 lx[3]	1 h at 40°F
Light-oxidized at 3 h[1]	200 lx[3]	3 h at 40°F
Commercial 1[4]	None	None
Commercial 2[5]	None	None

[1]Reduced-fat milk (3.776 L), plastic container (Kansas State Plastics, Inc., Hutchinson, KS).
[2]Hiland Dairy Co. (Springfield, MO).
[3]GE fluorescent light 3500K 15W, Sylvania, Inc. (Danvers, MA).
[4]Reduced-fat milk (3.776 L), plastic container, purchased at a supermarket in Manhattan, Kansas.
[5]Reduced-fat milk (250 mL), paperboard carton for school lunch program, donated by manufacturer.

Table 2. Milk-fat Contents, Total Solids Contents, Total Plate Counts, and Apparent Viscosities of Reduced-fat Milk Samples from Several Sources or Induced Off-flavors

Milk	Milk fat (%)	Total solids (%)	TPC[1] (log CFU/ml)	Apparent viscosity (mPa·s)
Fresh unaltered[2]	1.95 ± 0.04^{a}	11.36 ± 0.21^{a}	0.99 ± 1.08^{b}	$3.41 \pm 0.05^{b,c}$
High-acid[3]	2.05 ± 0.06^{a}	10.98 ± 0.29^{a}	6.67 ± 1.20^{a}	$3.35 \pm 0.05^{b,c}$
Light-oxidized 1 hr[3]	1.90 ± 0.06^{a}	10.99 ± 0.29^{a}	1.00 ± 1.20^{b}	$3.44 \pm 0.05^{a,b,c}$
Light-oxidized 3 hr[3]	1.95 ± 0.06^{a}	10.97 ± 0.29^{a}	0.98 ± 1.20^{b}	$3.36 \pm 0.05^{b,c}$
Commercial 1[3]	1.90 ± 0.06^{a}	11.06 ± 0.29^{a}	1.41 ± 1.20^{b}	3.57 ± 0.05^{a}
Commercial 2[3]	1.80 ± 0.06^{a}	10.73 ± 0.29^{a}	1.60 ± 1.20^{b}	$3.50 \pm 0.05^{a,b}$
Lacks freshness[3]	2.00 ± 0.06^{a}	10.43 ± 0.29^{a}	1.39 ± 1.20^{b}	$3.51 \pm 0.05^{a,b}$

[abc]Means within column having different superscript letters differ ($P<0.05$).
[1]TPC (Total Plate Count) = total aerobic microbial count.
[2]n = 4.
[3]n = 2.

Table 3. Range in Volatile Compound Concentrations and the Associated Milk Sample

Compound	Low conc., mg/kg	Milk sample	High conc., mg/kg	Milk sample
Benzaldehyde	0.878	Lacks freshness	0.923	High-acid
2-Butanone	0.503	Commercial 2	1.962	High-acid
Ethyl caproate	0.344	Light-oxidized 1 hour	0.775	Commercial 2
Heptanal	0.727	Fresh unaltered, Light-oxidized 1 hour, Light-oxidized 3 hours	0.737	Commercial 2
2-Heptanone	0.451	Fresh unaltered, Light-oxidized 1 hour, Light-oxidized 3 hours, Commercial 2	0.458	Commercial 1
Hexanal	0.423	Commercial 1	0.476	Commercial 2
Octanal	0.445	Light-oxidized 1 hour	1.062	Commercial 2
1-Octen-3-ol	0	Fresh unaltered, Light-oxidized 1 hour, Light-oxidized 3 hours, Commercial 1, Commercial 2	0.297	High-acid
Pentanal	0.456	High-acid	0.992	Commercial 1
2-Pentylfuran	0.478	Light-oxidized 1 hour	0.998	Commercial 1

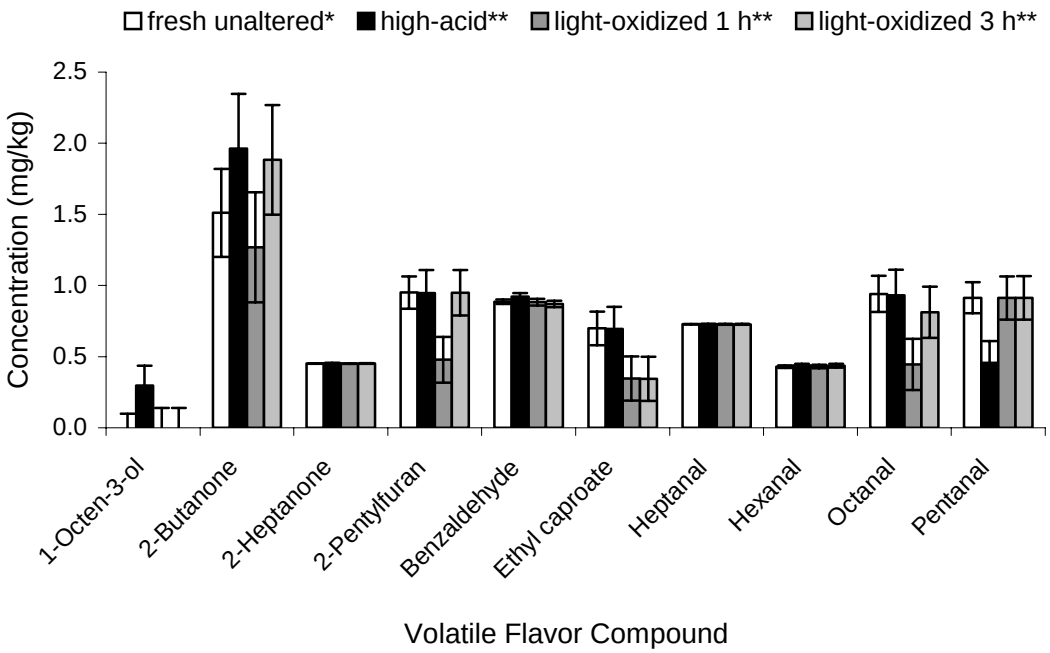

Figure 1. Mean Concentrations (mg/kg) of Volatile Flavor Compounds in Fresh Unaltered and Altered Milk Samples.

*n = 4.

**n = 2.

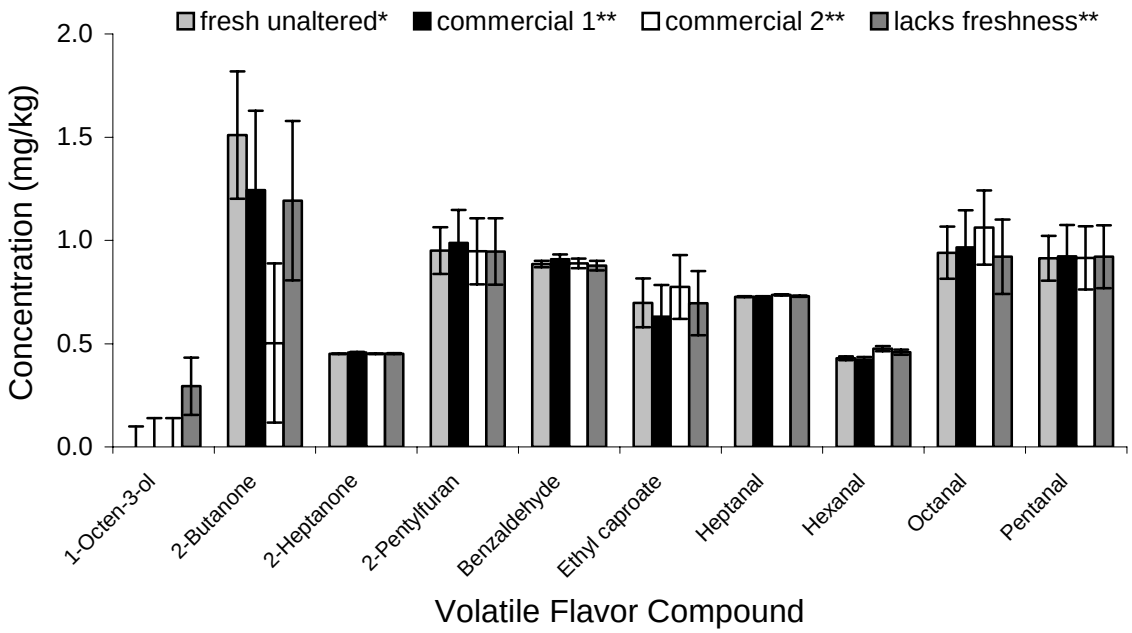

Figure 2. Mean Concentrations (mg/kg) for Volatile Compounds in Fresh Unaltered, Commercial, and Lacks Freshness Milk Samples.
*n = 4.
**n = 2.