Flavor Characteristics of Irradiated Apple Cider During Storage: Effect of Packaging Materials and Sorbate Addition

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ABSTRACT: Apple cider, with (0.1%) and without potassium sorbate, was packaged in 3 different materials to evaluate the effects of irradiation (2 kGy) and storage on flavor. Irradiated apple cider samples were compared with unirradiated samples stored in glass jars. Volatile flavor compounds, soluble solids, and titratable acidity were determined weekly throughout 3 wk of refrigerated storage. Oxygen permeability of the packaging materials was important in the retention of flavor during storage. Cider irradiated and stored in polystyrene containers or nylon-6 packaging materials (low oxygen permeability) had lower rates of loss of characteristic flavor compounds compared with unirradiated apple cider and cider irradiated and stored in low-density polyethylene (high oxygen permeability). The presence of sorbate, functioning as a yeast and mold inhibitor, reduced the rates of loss of the characteristic flavor compounds and the fermentation of sugars to acids. Principal component analysis resulted in several esters characteristic of apple flavor, soluble solids, and titratable acidity loading onto the 1st principal component (PC-1). Packaging material and sorbate treatment had the greatest effect on the compounds that loaded onto PC-1.

Keywords: apple cider, irradiation, flavor, packaging, sorbate

Introduction

Most of the food-borne illness outbreaks linked to unpasteurized, unfermented apple cider have been attributed to *Salmonella typhimurium*, *Cryptosporidium parvum*, and, most commonly, *Escherichia coli* O157:H7 (Ingham and Schoeller 2002). These microorganisms are resistant to low pH (3.3 to 4.1) and low storage temperatures (Jay 2000) and are able to survive the environment characteristic of apple cider. As a result of such outbreaks, the United States Food and Drug Administration mandated cider and juice processors to implement processing treatments to attain a 5 $\log_{10}$ reduction in the most resistant pathogen present. In apple cider, *E. coli* O157:H7 has been identified as the most resistant pathogen. If this 5 $\log_{10}$ reduction is not achieved, a special warning label must be present on containers of apple cider to be sold for human consumption (USFDA 1998).

Heat pasteurization, the current process used to reduce microbial load and considered to make apple cider safe for consumption, can cause undesirable changes in color, flavor, and viscosity (Poll and Flink 1983; Bettini and others 1998; Fischer and Golden 1998). Therefore, ionizing radiation has been investigated as a nonthermal method to inactivate food-borne pathogens and reduce spoilage. In apple cider, a 5 $\log_{10}$ reduction in microorganisms occurs after irradiation at between 1 to 3.55 kGy, although the precise dose necessary is dependent on pathogen species and strain (Fan and Thayer 2002). The addition of a preservative, such as potassium sorbate, extends the shelf-life of apple cider by inhibiting enzyme systems of yeasts and molds (Baroody and McLellan 1986).

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Previous research conducted at Iowa State Univ. (Ames, Iowa, U.S.A.) compared the effects of electron-beam irradiation and pasteurization on the quality attributes of apple cider (Boylston and others 2003; Wang and others 2003; Yulianti and others 2003, 2004). Irradiation and pasteurization treatments contribute to decreases in the color and turbidity of apple cider, compared with raw apple cider (Fan and Thayer 2002; Wang and others 2003). The flavor characteristics of apple cider irradiated at 2 and 4 kGy in low-density polyethylene (LDPE) packaging materials were similar to that of pasteurized apple cider, as determined by sensory and instrumental flavor analyses (Boylston and others 2003; Wang and others 2003). However, sensory evaluation panelists have also detected "cardboard" or "musty" off-flavors in irradiated apple cider containing potassium sorbate. Although the specific flavor compounds responsible for these undesirable flavor attributes have not been identified, they are believed to be breakdown products of the sorbate formed during irradiation (Boylston and others 2003; Yulianti and others 2003).

Absorption of flavor compounds by LDPE has been shown to be greater than other packaging materials (Ayhan and others 2001; Van Willige and others 2001). Interactions of fruit juices with packaging materials may result in direct migration of manufacturing compounds, absorption of flavor compounds, and transmission of light or oxygen (Askar 1999). The gas permeability or crystalline properties of LDPE, high-density polyethylene (HDPE), polypropylene, polyethylene terephthalate (PET), and poly(vinyl chloride) packaging materials are not affected by irradiation at doses of up to 8 kGy (Buchalla and others 1993). However, irradiation of these polymers can result in the release of hydrogen, carbon dioxide, carbon monoxide, and methane gases and the formation of volatile oxidation products, including peroxides, alcohols, aldehydes, ketones, and carboxylic acids. The extent of radiation-induced changes depends on many factors such as the type of polymer, processing ex-

posure, and irradiation conditions (Azuma and others 1983; Buchalla and others 1993).

The objective of this study was to determine the effects of packaging materials and sorbate addition on the retention of characteristic flavors in apple cider, treated by electron beam irradiation, during 3 wk of refrigerated storage. Because the penetration of the electron beams into the food is limited to 8 cm (Kilcast 1994; McLaughlin 1999), packaging materials were selected that had appropriate dimensions for complete penetration. Apple cider, packaged in 3 plastic polymers, polystyrene (PS), LDPE, or nylon-6 (N6), and irradiated at 2 kGy was compared with unirradiated cider packaged in glass containers. Flavor characteristics were monitored by the analysis of volatile flavor compounds and measurement of soluble solids and titratable acidity.

Materials and Methods

Sample preparation

Fresh cider consisting of dominant sweet and tart apple cultivars, with (0.1%) and without (0%) potassium sorbate, was obtained from a local cider producer in the spring of 2002. Cider was packaged and irradiated within 4 d of processing. The control, an untreated cider sample, was placed in 200-mL glass jars because this material is inert in terms of flavor migration, permeation, and absorption characteristics. Cider to be irradiated was placed into 3 different plastic packages, LDPE film (Nasco-Whirl Pak®, Fort Atkinson, Wis., U.S.A.), nylon-6 (N6) film (CleanFilm, Islandia, N.Y., U.S.A.), and PS flasks (Costar, Cambridge, Mass., U.S.A.) (Table 1). All of the packaging materials used in this experiment were presterilized. The LDPE and N6 bags were heat sealed to hold a cider sample of appropriate dimensions for electron beam penetration. The polystyrene containers were closed with plug seal caps. All containers were positioned on their side so the depth of the cider was less than 2.5 cm during irradiation. Cider was exposed to atmospheric conditions throughout the packaging process. After irradiation treatment, volatile flavor analysis, soluble solids, pH, and titratable acidity were measured initially and each week during 3 wk of refrigerated (4 °C) storage. Individual packages remained sealed until analyzed. Two replications were completed for each treatment.

Table 1—Specifications for packaging materials for apple cider samples

Packaging material	LDPE	Nylon-6	Polystyrene
Dimensions (cm)	11.5 × 23	15 × 20	2.5 × 9 × 9
Capacity (mL)	540	NA[a]	250
Thickness (mil)	2.5	2	NA
Oxygen permeability (cc-mil/100 in^2/24 h @25 °C)	276.5	2.6	0

[a]NA = information not available.

Electron beam irradiation

The cider was irradiated at the Iowa State Univ. Linear Accelerator Facility (Ames, Iowa) using electron beam irradiation at a target dose of 2.0 kGy at an energy level of 10 MeV and power level of 10.2 kW. Average absorbed dose was 2.22 ± 0.11 kGy, based on alanine dosimeter pellets attached to the top and bottom of the packages. The dose rate ranged from 78.9 to 81.9 kGy/min and the conveyor speed was set at 5.58 m/min. Electron beam irradiation at a dose of 2.17 kGy results in a 5 $\log_{10}$ reduction in *E. coli* O157:H7 (Wang and others 2001). Untreated (raw) cider was not exposed to irradiation but did follow similar transport to and from the facility. Irradiation was conducted at room temperature without temperature control. Cider samples were stored at 4 °C before and after irradiation treatment and deviated from these conditions only during irradiation treatment.

Volatile flavor analysis

Solid-phase microextraction (SPME) techniques were applied for the isolation and concentration of volatile flavor compounds (Boylston and others 2003). A representative cider sample (40 g) was transferred to a 100-mL headspace bottle and sealed with a Teflon septum. Samples were held in a 40 °C water bath with stirring during the isolation. Each sample was allowed to equilibrate and absorb onto the SPME fiber (2 cm-50/30 μm divinylbenzene/carboxen/polydimethylsiloxane; Supelco, Inc., Bellefonte, Pa., U.S.A.) for 45 min.

A gas chromatograph (GC; Model 6890; Hewlett-Packard, Inc., Wilmington, Del., U.S.A.) equipped with a splitless injection port and flame ionization detector was used for separation of volatile flavor compounds. Volatile flavor compounds were thermally desorbed (220 °C) for 3 min via the GC injection port onto a fused-silica capillary column (SPB-5, 30 m × 0.25 mm × 0.25-μm film thickness; Supelco, Inc.). The column pressure was set at 124.0 kPa with a helium flow rate of 1.9 mL/min. Oven temperature was initially 30 °C for 3 min, then increased to 80 °C at 5 °C/min, to 95 °C at 4 °C/min, to 115 °C at 5 °C/min, and to 200 °C at 10 °C/min. The detector temperature was constant at 220 °C. Flow rates of detector gases were air at 400 mL/min, hydrogen at 30 mL/min, and nitrogen (make-up gas) at 25 mL/min. Volatile flavor standards were identified using authentic standards (Sigma-Aldrich, Milwaukee, Wis., U.S.A.; AccuStandard, Inc., New Haven, Conn., U.S.A.). Analyses were conducted in duplicate and averaged for further statistical analysis.

Volatile flavor compounds were identified and confirmed with a GC-mass spectrometer (Trio 1000; Fisons Instruments, Danvers, Mass., U.S.A.) with a quadrupole mass analyzer. GC conditions were the same as those of the chromatographic analysis. The mass spectrometer conditions were as follows: source electron energy at 70 eV, source electron current at 150 μA, ion source temperature at 220 °C, interface temperature at 220 °C, source ion repeller at 3.4 V, electron multiplier voltage at 600 V and scan range between 41 and 250 m/z. Mass spectra of the volatile flavor compounds were compared with a spectral library (NBS Library) and a flavor and fragrance database (FlavorWORKS, ver. 2.0; Flavometrics, Anaheim Hills, Calif., U.S.A.) for identification and verification.

Soluble solids and titratable acidity

Soluble solids content was measured using a tabletop model 0 to 32°Brix refractometer (Milton Roy, Ivyland, Pa., U.S.A.) with an accuracy of ±0.05% dissolved solids (Brix) and reported as percent sucrose. Titratable acidity was determined by titrating a sample (20 mL apple cider + 80 g water) with 0.1 *N* NaOH to an endpoint of pH 8.2 using a digital pH meter (Accumet Model AB15; Fisher Scientific, Pittsburgh, Pa., U.S.A.). Titratable acidity was expressed as grams malic acid per 100 mL cider. Sample temperature was 20 °C for each analysis. Analyses were conducted in duplicate for each sample treatment.

Statistical analysis

The experiment was designed as a 3-way factorial with packaging material, sorbate addition, and storage time as the main factors. Analysis of variance and Fisher's least squares difference tests ($P < 0.05$) were conducted to determine the effects of the main factors

and interactions between main factors on the contents of volatile flavor compounds, soluble solids, and titratable acidity (SYSTAT, ver. 9.01; SPSS, Inc., Chicago, Ill., U.S.A.). Because of significant interactions between storage time and packaging material and/or sorbate addition, semi-log regression plots, as a function of storage time were plotted to determine rates of change for the contents of volatile flavor compounds, soluble solids, pH, and titratable acidity. Analysis of variance and Fisher's least squares difference tests ($P < 0.05$) were conducted on the linear regression slopes of the semi-log regression plots. These slopes were also analyzed using principal component analysis (PCA) with Varimax orthogonal rotation to examine relationships or groupings of flavor components based on treatment effects (SYSTAT). Two replications were completed for each of the treatments.

Results and Discussion

Soluble solids and titratable acidity

Soluble solids (SS) content and titratable acidity (TA) measure the sweetness and tartness intensity of apple cider. The SS and TA can also monitor the fermentation process, as sugars are converted into alcohols and organic acids (Stinson and others 1979). During the 3-wk storage period, the soluble solids content decreased and titratable acidity increased. Sorbate treatment and packaging material had a significant effect on the SS and TA of the apple cider with the greatest differences occurring after 3 wk of refrigerated storage (Table 2).

The addition of sorbate to the apple cider resulted in higher SS and lower TA following storage. Natural yeasts present in apple products convert sugars into alcohol and organic acids during the initial stages of fermentation (Stinson and others 1979). The sorbate plays a role in limiting fermentation or other degradation reactions of irradiated and unirradiated apple cider, as indicated by the higher soluble solids and lower acidity. Similar findings reported by Luedtke and Powell (2002) and Baroody and McLellan (1986) suggest that sorbate increases shelf-life by inhibiting yeasts and molds in a low pH environment.

The effect of packaging was dependent on the presence of sorbate in the apple cider. The presence of sorbate controlled the fermentation process and packaging had a minor effect on the soluble solids content and titratable acidity of the apple cider. In the absence of sorbate, the differences in the soluble solids content and titratable acidity of the irradiated apple cider samples were attributed to the differences in the oxygen permeability of the packaging materials (Table 1). Cider packaged in the LDPE film, which has a higher oxygen permeability than both the PS containers and N6 film, demonstrated the greatest decrease in soluble solids and increase in titratable acidity in comparison to the other irradiated samples following storage. Because LDPE was less effective than PS and N6 in terms of controlling oxygen permeability, the process of fermentation was most likely escalated in the cider packaged in LDPE film.

Table 2—Effect of packaging material[a] and sorbate addition on the soluble solids and titratable acidity of apple cider after 3 wk of refrigerated storage[b]

Treatment	0% Sorbate	0.1% Sorbate
Soluble solids[c]		
Glass – 0 kGy	10.5 dy	12.6 ax
PS – 2 kGy	11.9 by	12.4 ax
N6 – 2 kGy	12.3 ax	12.7 ax
LDPE – 2 kGy	11.0 cy	11.4 bx
Titratable acidity[d]		
Glass – 0 kGy	0.409 ax	0.211 ay
PS – 2 kGy	0.323 bx	0.246 ay
N6 – 2 kGy	0.251 cx	0.204 ax
LDPE – 2 kGy	0.456 ax	0.208 ay

[a]Glass = unirradiated apple cider; PS = polystyrene containers; N6 = nylon-6 polyamide pouches; LDPE = low-density polyethylene pouches.
[b]Means are duplicate analyses of 2 replications. Means followed by different letters (a–d) within the same column are significantly different from each other ($P < 0.05$), based on packaging treatment. Means followed by different letters (x–y) within the same row are significantly different from each other ($P < 0.05$), based on sorbate treatment.
[c]% sucrose at 20 °C.
[d]g malic acid/100 mL cider.

Volatile flavor analysis

Volatile ester compounds such as butyl acetate, 2-methylbutyl acetate, hexyl acetate, ethyl butanoate, ethyl 2-methylbutanoate, butyl butanoate, hexyl butanoate, ethyl hexanoate, and hexyl hexanoate were among the major compounds identified in this study. Acetates and butanoates and other esters are known to commonly occur in apples and apple products (Flath and others 1967; Dimick and Hoskin 1983; Cunningham and others 1986).

The initial content of most the volatile flavor compounds identified in the apple cider was not significantly affected by irradiation and packaging treatment (Table 3) or sorbate addition (Table 4). The irradiation and packaging treatments had a significant effect only on the contents of propyl butanoate and phenylacetaldehyde (Table 3). These differences were noted between the irradiated samples packaged in different materials. Irradiation treatment does not appear to have a significant effect on the content of the volatile flavor compounds present in apple cider. Sorbate addition had a significant effect on the initial content propyl acetate and butyl pentanoate (Table 4). The delay of 2 to 3 d between the addition of sorbate to the pressed cider and the irradiation treatment may have contributed to the differences in the content of these volatile flavor compounds. These 4 compounds are not among the volatile flavor compounds that are recognized as the predominant contributors to the flavor of apple cider, and are present at substantially lower levels than the other esters that contribute to characteristic apple flavor. Thus, it is believed that the irradiation treatment and sorbate addition did not have a significant impact on the overall flavor characteristics of the apple cider.

Initial statistical analysis of the data as a 3-way factorial (packaging material, sorbate, and storage week) for each flavor compound resulted in significant interactions between storage time and packaging material and/or sorbate addition. For this reason, GC peak areas were plotted against storage time to calculate the rate of loss in the content of the flavor compounds during storage. As a result, a treatment variable was removed and the effects of only packaging material and sorbate remained.

Volatile flavor compounds displayed the greatest decrease in intensity between week 0 and week 1 of storage and tended to level off by week 3. Figure 1a shows the effect of storage on the content of hexyl acetate, a major characteristic apple flavor compound. Most of the volatile flavor compounds showed a similar storage effect. The rate of loss of flavor compounds is best described by 1st-order kinetics, as shown by the plot of $\log_{10}$ GC peak area against storage time (Figure 1b). The slope for the rate of loss for each flavor compound was calculated to determine the effects of packaging material and sorbate addition on the stability of the flavor compounds during storage.

Because more than 50 volatile compounds were identified in the cider, the data were further analyzed using PCA techniques. PCA identifies patterns of interactions between variables to condense a large set of data into groups of similar characteristics and is more

effective in demonstrating relationships between variables than correlation or other statistical techniques. In this experiment, linear slopes of soluble solids and titratable acidity data plotted against week and slopes of the log GC area plotted against week were loaded onto the PCA function. Volatile flavor compounds were not exclusively grouped within the principal components based on the class of compound, such as alcohols, aldehydes, or esters. The first 4 principal components (PC) accounted for more than 71% of the total variability in the data set. PC-1 (38.5%) contained soluble solids, titratable acidity, and 27 volatile flavor compounds, including most the esters known to contribute to the characteristic apple flavor. PC-2 (12.5%) contained 7 volatile flavor compounds, many of which were alcohols. PC-3 (11.8%) contained 4 volatile flavor compounds, while PC-4 (8.8%) contained 3 volatile flavor compounds (Figure 2). Because of the grouping of the flavor compounds onto the principal components is influenced by their response to the packaging material and sorbate addition treatments,

Table 3—Effect of packaging materials on initial contents of apple cider flavor compounds[a]

	GC peak areas (wk 0)			
Irradiation dose:	**0 kGy**	**2 kGy**	**2 kGy**	**2 kGy**
Packaging material[b]:	**Glass**	**PS**	**N6**	**LDPE**
Ethyl propionate	31.0	43.7	32.1	65.7
Propyl acetate[c]	65.5	42.5	39.8	61.5
t-Butyl acetate	11.6	11.3	19.9	40.7
Isobutyl acetate	6.6	8.3	10.6	10.6
Methyl(ante)iso-pentanoate	22.2	10.4	10.9	83.3
Hexanal	317.0	245.5	194.0	425.3
Ethyl butanoate	491.0	380.7	273.0	480.4
1-Methylpropyl acetate	54.1	55.3	61.5	70.4
Butyl acetate	1869.7	1411.3	1694.5	1778.4
3-Pentyl acetate	74.0	72.4	81.7	94.4
Ethyl 2-methylbutanoate	1268.8	1238.3	1101.1	1263.0
c-3-Hexen-1-ol	ND	ND	ND	ND
Methyl 2-ethylbutanoate	ND	ND	31.4	ND
Methyl 2-methylpentanoate	125.8	52.8	54.2	22.3
Hexanol	898.7	827.8	1010.1	1268.2
2-Methylbutyl acetate	670.2	524.2	651.4	742.8
Methyl iso-hexanoate	138.2	85.3	150.4	104.9
Propyl butanoate	46.0 a	45.7 a	47.6 a	62.8 b
Ethyl pentanoate	35.6	43.2	40.1	63.4
Butyl propionate	64.2	96.8	67.8	88.1
Pentyl acetate	135.8	204.6	154.4	144.5
Methyl hexanoate	38.3	48.8	54.0	56.5
Isopropyl 2-methylbutanoate	17.4	15.6	14.9	33.0
Benzaldehyde	11.4	13.3	10.8	30.1
3-Methylbutyl propionate	9.1	9.6	10.5	25.3
1-Octene-3-ol	6.1	4.0	9.5	14.8
Butyl butanoate	341.0	265.0	259.5	267.1
Ethyl hexanoate	582.8	550.3	437.1	481.3
Octanal	ND	ND	ND	ND
3-c-Hexen-1-yl acetate	66.4	41.5	58.7	70.5
Hexyl acetate	5450.8	4932.2	4152.3	4545.7
Phenylacetaldehyde	6.5 b	ND a	13.6 b	17.5 b
Butyl 2-methylbutanoate	80.0	68.0	56.0	66.8
t-2-Octenal	28.3	18.1	22.0	37.5
1-Octanol	152.5	112.3	136.1	177.3
Butyl pentanoate[c]	139.0	85.5	142.1	132.2
Propyl hexanoate	35.1	2.8	18.3	76.8
Ethyl heptanoate	348.6	345.7	336.6	297.7
Nonanal	78.6	60.5	60.9	90.8
Hexyl propionate	92.0	ND	120.6	62.4
Heptyl acetate	232.1	213.6	161.9	171.5
3-Hepten-1-yl acetate	332.2	ND	426.8	659.5
Benzyl acetate	18.0	27.8	29.2	34.4
t-2-Nonenal	106.4	85.6	98.5	125.7
Hexyl butanoate	1525.9	1351.5	770.8	775.0
Estragole	126.2	104.2	50.6	79.8
Decanal	32.9	28.0	39.4	40.1
3-Octen-1-yl acetate	25.9	23.2	16.6	21.3
Hexyl 2-methylbutanoate	521.4	475.5	266.7	267.5
2-Phenylethyl acetate	17.8	12.2	10.3	6.2
2-Decenal	7.8	6.4	11.2	19.6
Pentyl hexanoate	37.5	31.7	19.9	32.1
Hexyl hexanoate	837.2	694.1	390.7	484.1
Terpene ester	45.6	37.0	59.7	166.5
β-Farnesene	59.8	66.9	100.3	58.3
α-Farnesene	1211.2	810.2	809.5	1029.1

[a]Compounds are presented in the order of elution from the gas chromatograph. Means are duplicate analyses of 2 replications (week 0) with data for sorbate addition pooled.
[b]Glass = unirradiated apple cider; LDPE = low-density polyethylene pouches; N6 = nylon-6 polyamide pouches; PS = polystyrene containers.
[c]Effect of sorbate significant ($P < 0.05$).

Table 4—Effect of sorbate on initial contents of propyl acetate and butyl pentanoate[a]

	Gas chromatography peak areas (week 0)	
	0% sorbate	**0.1% sorbate**
Propyl acetate	70.8 b	33.8 a
Butyl pentanoate	49.4 a	200.0 b

[a]Means are duplicate analyses of 2 replications with data for packaging material pooled.

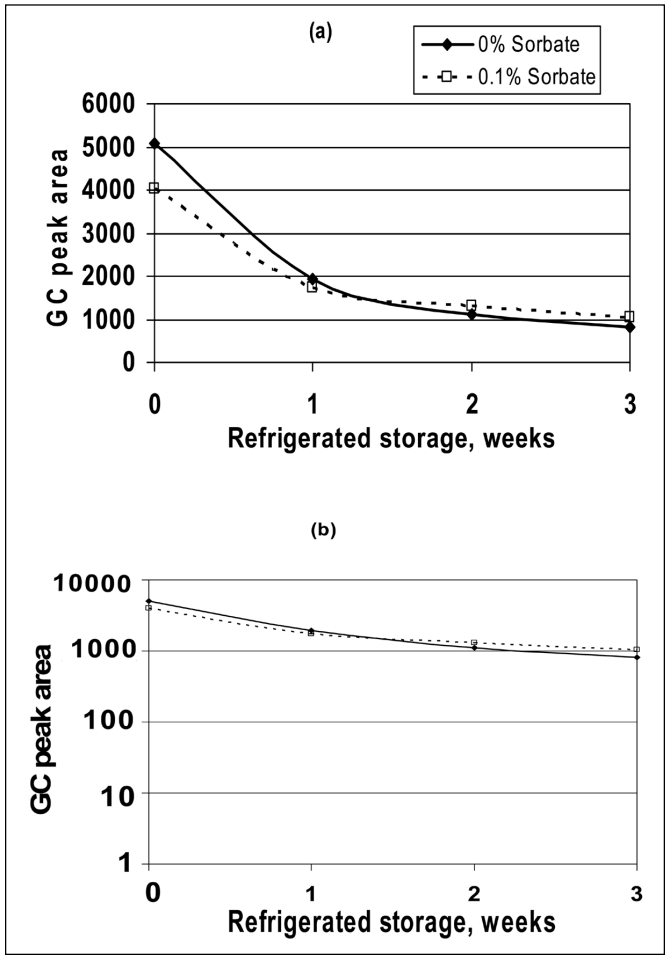

Figure 1—Effects of storage on content of hexyl acetate packaged in low-density polyethylene (LDPE) film using (a) linear gas chromatography (GC) peak area and (b) semi-$\log_{10}$ GC peak area plots versus storage time.

the effects of these treatments on the volatile flavor compounds will be discussed with respect to the 4 principal components.

Effect of sorbate. The addition of 0.1% potassium sorbate was effective in slowing the loss of volatile flavor compounds during storage compared with cider without sorbate. In all cases where a significant difference was noted between the presence and absence of sorbate, rates of loss were greater (more negative slope) in cider that did not contain sorbate (Table 5). All of the volatile flavor compounds that showed a significant effect of sorbate addition loaded onto PC-1 and included esters characteristic of apples. Therefore, the addition of sorbate decreased the rate of loss of the volatile flavor compounds that are important contributors to apple flavor. The decrease in the content of these volatile flavor compounds coupled with the decrease in soluble solids and increase in titratable acidity indicates that fermentation or other degradation reactions most likely diminish the apple or fruity compounds that give apple cider its desirable quality.

According to Thakur and Arya (1993), the characteristic fruit aroma of orange juice and mango pulp decreased as a result of irradiation at 10 kGy. However, off-flavor formation in these products was prevented in the presence of sorbate. Another study reported that the effects of processing treatment (irradiation and pasteurization) were related to the presence of sorbate. In irradiated apple cider, the presence of sorbate reduced the degradative effects of irradiation and resulted in a higher content of esters and other volatile flavor compounds that are characteristic of apple flavor (Boylston and others 2003). Irradiation generates hydrogen and hydroxyl radicals that can initiate oxidation and other degradation reactions. The sorbate is able to quench these free radicals that are formed during irradiation (Thakur and others 1990; Thakur and Arya 1993) and, thus, preserve the volatile flavor compounds that contribute to desirable apple-like flavors.

Effect of packaging material. A comparison of the slopes for each of the packaging materials illustrates the retention of flavor compounds in irradiated cider compared with untreated (raw) cider during 3 wk of storage following irradiation treatment (Table 6). The cider irradiated and stored in PS containers had a statistically less negative slope (lower rate of loss) than untreated cider or irradiated cider packaged in LDPE for volatile flavor compounds characteristic of apple or fruity taste and aroma. Examples of such trends can be noted for esters such as butyl acetate, 2-methylbutyl acetate, hexyl acetate, propyl butanoate, ethyl pentanoate, and

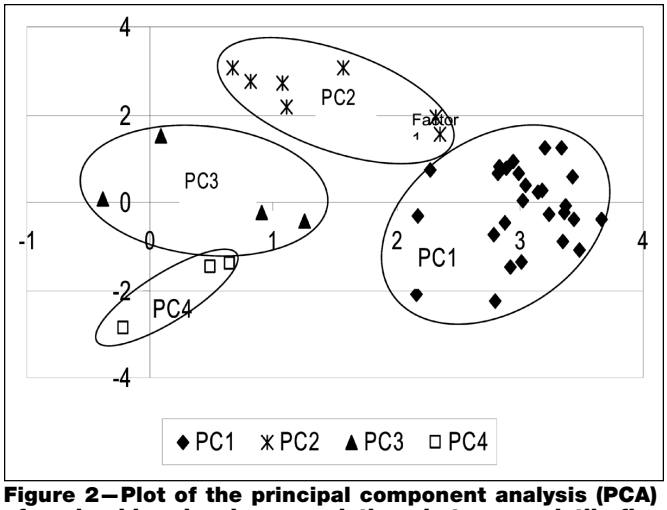

Figure 2—Plot of the principal component analysis (PCA) of apple cider showing associations between volatile flavor compounds and analytical data. Vector coordinates representing individual flavor compounds, soluble solids, or titratable acidity signify pooled responses for all processing treatments (irradiation, packaging material, and sorbate addition) using the rates of loss during 3 wk of refrigerated storage for 2 replications.

Table 5—Effect of sorbate addition on the retention of flavor compounds in apple cider during 3 wk of refrigerated storage[1]

	Slope (log GC peak area/wk)	
	0% sorbate	0.1% sorbate
Component 1 (PC-1)		
Soluble solids[2]	–0.361a	–0.122b
Titratable acidity[3]	0.061b	0.001a
1-Methylpropyl acetate	–0.119	0.009
Butyl acetate	–0.116a	–0.020b
2-Methylbutyl acetate	–0.062a	–0.005b
Pentyl acetate	–0.189a	–0.060b
Hexyl acetate	–0.164a	–0.098b
Heptyl acetate	–0.242a	0.028b
Benzyl acetate	–0.079	–0.014
Butyl propionate		
Glass – 0 kGy	–0.246a	–0.003b
PS – 2 kGy	–0.026a	–0.055a
N6 – 2 kGy	–0.122a	–0.009a
LDPE – 2 kGy	–0.311a	–0.120b
Ethyl 2-methylbutanoate	–0.250a	–0.044b
Propyl butanoate	–0.097a	–0.028b
Isopropyl 2-methylbutanoate	–0.140a	–0.046b
Butyl butanoate	–0.151a	–0.004b
Butyl 2-methylbutanoate	–0.053	–0.004
Hexyl butanoate	–0.288	–0.058
Hexyl 2-methylbutanoate	–0.078	–0.041
Ethyl pentanoate	–0.076a	0.003b
Methyl(ante)iso-pentanoate	–0.024	0.055
Methyl hexanoate	–0.254a	–0.089b
Ethyl hexanoate	–0.200a	–0.052b
Pentyl hexanoate	–0.260	–0.105
Ethyl heptanoate	–0.430a	–0.012b
Propyl hexanoate	0.370a	–0.144b
Hexanal	–0.641	–0.214
t-2-Octenal	–0.287a	–0.002b
Decanal	–0.065	0.006
Benzaldehyde	–0.292	–0.133
α-Farnesene	–0.191a	–0.086b
Component 2 (PC-2)		
3-Pentyl acetate	–0.668	–0.478
3-Methylbutyl propionate	–0.022	–0.032
1-Hexanol	–0.019	–0.012
1-Octene-3-ol	–0.151	–0.089
1-Octanol	–0.033	–0.010
t-2-Nonenal	0.043	0.057
Terpene ester	–0.120	–0.080
Component 3 (PC-3)		
Methyl iso-hexanoate	–0.235	–0.097
Octanal	–0.011	0.326
Estragole	–0.288	–0.058
β-Farnesene	–0.083	–0.084
Component 4 (PC-4)		
Isobutyl acetate	–0.047	0.017
2-Phenylethyl acetate	0.138	–0.015
3-Octen-1-yl acetate	0.018	–0.003

[1]Means are duplicate analyses of 2 replications with data for packaging material pooled, unless interactions between packaging material and sorbate addition were significant ($P < 0.05$). Means followed by different letters within the same row are significantly different from each other ($P < 0.05$).
[2] % sucrose at 20°C.
[3]g malic acid/100 mL cider.

t-2-octenal. In some cases, however, the raw, PS, and nylon-6 (N6) were more similar to each other and LDPE had the most negative slopes overall. Such trends, which were identified in isopropyl 2-methylbutanoate, butyl propionate, butyl 2-methylbutanoate, t-2-nonenal, and α-farnesene, suggest that LDPE demonstrates the poorest retention in characteristic apple flavors. Most of the compounds that displayed significant packaging effects loaded onto PC-1, which supports the grouping together of characteristic apple cider flavor compounds.

Table 6—Effect of packaging material and irradiation on the retention of flavor compounds in apple cider during 3 wk of refrigerated storage[a]

	Slope (log GC peak area/wk)			
Packaging material[b]:	**Glass**	**PS**	**N6**	**LDPE**
Irradiation dose:	**0 kGy**	**2 kGy**	**2 kGy**	**2 kGy**
Component 1 (PC-1)				
SS[c]	–0.295b	–0.092c	–0.012c	–0.480a
TA[d]	0.034b	0.031b	0.007a	0.039b
1-Methylpropyl acetate	–0.111	–0.040	0.065	–0.133
Butyl acetate	–0.092a	–0.011a	–0.069a	–0.100a
2-Methylbutyl acetate	–0.050a	0.018b	–0.029ab	–0.073a
Pentyl acetate	–0.116	–0.077	–0.135	–0.170
Hexyl acetate	–0.148ab	–0.057c	–0.094bc	–0.227a
Heptyl acetate	–0.139	–0.086	–0.044	–0.160
Benzyl acetate	–0.006	–0.020	–0.048	–0.112
Butyl propionate				
0% sorbate	–0.246a	–0.026b	–0.122b	–0.311a
0.1% sorbate	–0.003a	–0.055a	–0.009a	–0.120a
Ethyl 2-methylbutanoate	–0.213	–0.097	–0.103	–0.174
Propyl butanoate	–0.071b	0.027c	–0.050b	–0.156a
Isopropyl 2-methylbutanoate	–0.036b	0.018b	–0.014b	–0.339a
Butyl butanoate	–0.101ab	–0.009b	–0.037b	–0.163a
Butyl 2-methylbutanoate	–0.030b	0.036b	0.023b	–0.142a
Hexyl butanoate	–0.325	–0.131	0.064	–0.299
Hexyl 2-methylbutanoate	–0.063	–0.033	0.142	–0.284
Ethyl pentanoate	–0.041b	0.056c	–0.035b	–0.127a
Methyl(ante)iso-pentanoate	–0.099	0.100	0.167	–0.105
Methyl hexanoate	–0.181	–0.092	–0.139	–0.274
Ethyl hexanoate	–0.149	–0.076	–0.067	–0.212
Pentyl hexanoate	–0.323a	–0.129ab	0.022b	–0.300a
Ethyl heptanoate	–0.308	–0.080	–0.143	–0.352
Propyl hexanoate	0.339	0.264	–0.099	0.139
Hexanal	–0.607	–0.365	–0.323	–0.415
t-2-Octenal	–0.253a	0.005b	–0.022b	–0.308a
Decanal	–0.024	0.005	–0.019	–0.080
Benzaldehyde	–0.391	–0.121	–0.092	–0.247
α-Farnesene	–0.111b	–0.061b	–0.040b	–0.341a
Component 2 (PC-2)				
3-Pentyl acetate	–0.640	–0.464	–0.673	–0.516
3-Methylbutyl propionate	–0.009	0.025	–0.057	–0.067
1-Hexanol	–0.018	0.028	–0.045	–0.027
1-Octene-3-ol	–0.113	0.029	–0.257	–0.139
1-Octanol	–0.066	0.043	–0.020	–0.043
t-2-Nonenal	0.078b	0.090b	0.045b	–0.013a
Terpene ester	–0.103	–0.016	–0.177	–0.103
Component 3 (PC-3)				
Methyl iso-hexanoate	–0.310	–0.615	–0.169	0.300
Octanal	ND	ND	ND	0.157
Estragole	–0.325	–0.131	0.064	–0.299
β-Farnesene	–0.015	–0.035	–0.105	–0.180
Component 4 (PC-4)				
Isobutyl acetate	–0.103	0.037	–0.037	0.044
2-Phenylethyl acetate	–0.091	–0.070	0.300	0.146
3-Octen-1-yl acetate	–0.063	0.037	0.020	0.036

[a]Means are duplicate analyses of 2 replications with data for sorbate addition pooled, unless interactions between packaging material and sorbate addition were significant ($P < 0.05$). Means followed by different letters within the same row are significantly different from each other ($P < 0.05$).
[b]Glass = unirradiated apple cider; PS = polystyrene containers; N6 = nylon-6 polyamide pouches; LDPE = low-density polyethylene pouches.
[c]SS = soluble solids (% sucrose at 20 °C); slope = decrease in SS/wk in storage.
[d]TA = titratable acidity (g malic acid/100 mL cider); slope = decrease in TA/wk in storage.

The properties of the packaging material can influence the retention of the volatile flavor compounds during storage. The extent of flavor absorption by packaging materials is influenced by the polarity of the polymer and flavor compound, with plastic polymers having a greater affinity for compounds with similar polarity (Van Willige and others 2001). The retention of aldehydes, ethyl butanoate, and other flavor compounds in orange juice was found to be significantly higher in glass and PET than in HDPE and LDPE (Ayhan and others 2001). Matsui and others (1992) have also noted the LDPE has inferior gas barrier properties and greater sorption of flavor compounds than other films.

The raw cider, which was stored in glass jars, had a negative slope for the content of the volatile flavor compounds, even though volatile flavors cannot permeate through glass. Because glass is known to be inert in terms of flavor absorption, loss of flavor compounds in glass bottles is thought to be related to chemical degradation (Ayhan and others 2001). Because the cider that was stored in glass bottles was not irradiated, it is speculated that fermentation and other degradation reactions contributed to the loss of the volatile flavor compounds.

Conclusions

Packaging material and sorbate addition had a significant effect on the retention of characteristic flavor and quality attributes of irradiated apple cider during 3 wk of refrigerated storage. Storage of the apple cider resulted in loss of volatile flavor compounds and decreases in soluble solids content and increases in titratable acidity, attributed to the natural fermentation process. These undesirable changes were slowed when cider was irradiated and stored in PS and N6 packaging materials most likely because of the low oxygen permeability of these materials. Potassium sorbate, a yeast and mold inhibitor, also slowed the loss of volatile flavor compounds and the conversion of sugars to acids through the natural fermentation process.

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