

# Volatile Compounds in Five Starches<sup>1</sup>

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## ABSTRACT

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Volatile compounds in commercial wheat, corn, potato, waxy corn, and tapioca starches and in laboratory-prepared wheat, corn, and potato starches were collected, separated, and identified by a purge and trap concentrator (P&T) interfaced to a gas chromatograph (GC) equipped with a Fourier transform infrared detector (FTIR) and a mass selective detector (MSD). Hexanal was the most abundant compound in the corn and potato starches and in the laboratory-prepared wheat starch as determined by total ion chromatogram (TIC) peak areas. Hexanal was the third most abundant compound in commercial wheat starch after 2-ethyl-1-hexanol and benzaldehyde. Among the volatile organics, the level of aldehydes was the highest, followed by alcohols, ketones, benzenes, esters, and terpenes. Specific compounds identified, the majority of which appear

to be degradation products of lipid peroxidation, include hexanal, heptanal, octanal, nonanal, decanal, benzaldehyde, 2-propanone, 2-propanol, 1-butanol, 2-ethyl-1-hexanol, methylbenzene, and tetradecane. Waxy corn starch, which released a substantially higher level of total volatiles than all other starches, contained large amounts of pentyl, 2-methyl-1-butyl, benzyl, and isobornyl acetates; and citronella and 1,8-cineole. Tapioca starch contained few volatiles but did contain an increased level of 2-propanol. No alcohols occurred in the commercial corn starch. Terpene compounds were detected only in commercial potato, waxy corn, and tapioca starches. Many volatiles detected in wheat and corn starches also were detected in the kernels of their commercial samples.

Starch is added to food mainly to modify and control texture; it is expected to have a bland aroma and flavor. Pure starch is neither water soluble nor volatile, so it should be tasteless and odorless (Ramirez 1991). Yet odors are ascribed to starches and the descriptors for starch odor largely depend on the source. Corn, wheat, and sorghum starches are said to have a “cereal odor”, whereas potato starch has an “earthy” note. Drum-cooked and dried wheat and corn starches were described as having undesirable “woody” and “corny” odors (Seidel et al 1984). Tapioca starch is reported to have the least off-odor, waxy corn slightly more, and corn starch the most (Langan 1987). Therefore, tapioca starch has been preferred for use in delicately flavored puddings, pie and pastry fillings, and baby foods (Balagopalan et al 1988, Moore et al 1984, Rutenberg 1980).

Little is known about the nature, origin, and formation of off-odors in starches. Even though their levels are quite low, lipids associated with starch granules may be responsible for off-odors and off-flavors (Swinkels 1985a, Baldwin et al 1997). Intragranular starch lipids, which are mainly lysophospholipids and free fatty acids at a concentration of  $\leq 1\%$  in normal cereal starches (Morrison 1988), are reportedly stable to autoxidation (Morrison and Coventry 1989, Morrison 1995), probably because they occur as inclusion complexes with amylose (Morgan et al 1993). Upon even partial digestion of starch granules, however, starch lipids may be released and become susceptible to autoxidation. Also, surface lipids, which amount to  $<0.05$  and  $0.2\%$  in cereal and tuber starches, respectively (Swinkels 1985b), are composed of free fatty acids, phospholipids, and triglycerides (Vasanthan and Hoover 1992, Baldwin et al 1997). In cereal starches, the surface lipids contain  $\approx 50\%$  polyunsaturated fatty acids and they are susceptible to autoxidation during storage, leading to off-flavors (Radley 1976, Morrison 1988).

Volatile compounds in raw materials used to produce starch have been studied, including corn (Hougen et al 1971), wheat (McWilliams

and Mackey 1969, Maga 1978, Seitz 1995), and potato (Solms and Wyler 1979). During wet-milling, the volatiles in a raw material could be adsorbed onto the external surface of starch granules, which display protrusions of 10–50 nm (e.g., potato and wheat starches) (Baldwin et al 1998), as well as on the surfaces of pores leading into the granules (Fannon et al 1992). In addition, the amorphous phase inside granules is accessible to molecules with a size below  $\approx 1,000$  Da (French 1984). BeMiller and Pratt (1981) reported that various starches slurried in water with methanol, ethanol, 1-propanol, and 1-butanol concentrate the organics in their granules, perhaps because of formation of complexes with helical amylose (Godshall and Solms 1992). Hau et al (1996) investigated the binding of six low molecular weight, linear organics to wheat starch with a moisture content of 10–12% under equilibrium headspace conditions at 25°C for 1 hr. A major quantity of volatiles (60–80% of organics) was bound to the starch within 20 min, and the extent of binding upon 60 min of exposure at 25°C was greatest for propionic acid, followed by 1-hexanol, benzaldehyde, dodecane, diacetyl, and ethyl acetate.

Seidel et al (1984) described a procedure in which cereal starches were extracted at 25°C with aqueous sodium hydroxide at pH 10.4–12.5 for 30 min to 3 hr at a starch solids content of 35–43%. The extracted cereal starches were claimed to be free from off-tastes and off-odors. The authors suggested that the amorphous region of the granules was swollen sufficiently at the alkaline pH so that materials associated with off-taste and off-odor could be removed by washing with water. The benefits to starch flavor were measured by a sensory panel, but the compounds removed by the treatment were not reported. Matsunaga (1996) also reported that wheat starch extracted with sodium hydroxide at pH  $\approx 12$  was free of characteristic cereal odor. Harris (1963) described another patented process that removed off-flavors from cereal starches by washing starch with 28% aqueous ammonia in methanol at 60–65°C for 2–6 hr followed by washing with water.

The objective of this study was to collect, separate, and identify volatile compounds associated with commercial starches and from three starches isolated in the laboratory. Knowledge of the volatiles associated with starches could pinpoint their origin and lead to methods of remediation.

## MATERIALS AND METHODS

### General Methods

Moisture contents of starches were determined by AACC Method 44-15A (1995), and protein contents ( $N \times 5.7$ ) by combustion in a nitrogen determinator (FP-2000, Leco Corp., St. Joseph, MI).

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## Materials

Commercial native wheat, corn, and potato starches were purchased from Sigma Chemical Co. (St. Louis, MO), and tapioca and waxy corn starches were obtained from A. E. Staley Manufacturing Co. (Decatur, IL). Wheat, corn, and potato starches also were isolated in the laboratory using distilled water. Wheat starch was isolated and purified from flour (70% extraction) freshly milled from a blend of hard red winter wheats according to the method of Shogren et al (1969). The wheat starch contained 0.19% protein and 11.0% moisture. Corn starch was prepared from yellow dent corn according to the method described by Eckhoff et al (1996), except for modifications to the purification of the starch from the ground endosperm. The slurry was allowed to settle, and the yellowish top layer was scraped off and discarded. The sediment was resuspended in water, the mixture centrifuged at  $2,000 \times g$  for 10 min, and the top layer discarded. The washing step was repeated three times to give corn starch with 0.15% protein and 10.8% moisture. Potato starch was isolated from Idaho Russet potatoes obtained from a local market according to the method of Willigen (1964). The potato starch contained 0.11% protein and 12.9% moisture. The starches were stored at 5°C in glass containers with screw-caps lined with aluminum foil. All chemicals used were reagent-grade unless otherwise specified.

## Collection of Volatiles from Starch

Volatiles from starch were collected through dynamic headspace sampling using a purge and trap instrument (P&T) as previously described by Seitz (1995), with slight modifications to time and

temperature. Starch (10 g) was placed in a 25-mL Tekmar sparger tube mounted on a Tekmar P&T system (model LSC 2000, Tekmar Co., Cincinnati, OH) equipped with a sample heater (model 211005) and a capillary interface module (model 2530). Each starch sample was preheated at 80°C for 2 min, then the sample headspace was purged with helium at 80°C for 8 min, and the volatiles were trapped on a Tenax trap (0.29 g, 60–80 mesh,  $12 \times 1/8$ ", Tekmar). Following an 8-min drying purge with helium to remove water from the Tenax trap, it was preheated to 175°C and then to 225°C for 4 min to desorb the volatiles. With the capillary interface module, the desorbed volatiles were cryofocused at  $-120^\circ\text{C}$  by liquid nitrogen at the top of the gas chromatograph (GC) column and released into the GC column by the rapid heating of the cryofocused zone at 200°C for 45 sec.

## Analysis of Volatiles by Gas Chromatography and Fourier Transform Infrared Spectroscopy and Mass Spectrometry (GC-FTIRD-MSD)

The procedure described by Seitz (1995) was followed, with modifications to some parameters. A model 5890 Series II gas chromatograph (GC) coupled with a model 5965A Fourier transform infrared detector (FTIRD) and a model 5970 mass selective detector (MSD), all from Hewlett Packard Co. (Palo Alto, CA), were used to analyze starch volatiles. A capillary column of BP  $\times$  35 with polysilylphenylene-siloxane stationary phase (50 m  $\times$  0.32 mm i.d.  $\times$  0.25  $\mu\text{m}$  film thickness) from Scientific Glass Engineering, Inc. (Austin, TX) was used for the separation of volatiles. Column head pressure was  $\approx 89.5$  kPa (13.0 psi) at 50°C, and helium flow

TABLE I  
Aldehydes in Starches and Total Ion Count (TIC) Peak Areas

Compound	TIC Peak Area ( $\times 10^{-6}$ ) of Starches							
	Wheat		Corn		Potato		Waxy Corn	Tapioca
	Lab <sup>a</sup>	Com <sup>b</sup>	Lab	Com	Lab	Com	Com	Com
Butanal	0	0	0	0	2.9	0	0	0
Pentanal	trace <sup>c</sup>	0	6.4	0	9.9	8.1	41.3	0
Hexanal	28.4	47.1	59.3	138.8	112.6	45.6	410.3	13.0
2-Hexenal	trace	trace	trace	trace	2.3	0	0	0
Heptanal	3.2	12.6	3.5	6.7	5.7	6.3	0	0
(E)-2-Heptenal	0	0	0	0	0	0	25.5	0
Octanal	1.3	7.2	3.1	2.4	3.3	5.9	13.8	0
Benzaldehyde	0.8	58.5	trace	3.7	trace	10.6	16.7	trace
(Z)-2-Octenal	0	0	0	0	5.3	0	0	0
(E)-2-Octenal	3.1	5.0	7.0	0	18.5	18.4	19.8	0
Nonanal	17.8	27.9	21.2	19.3	11.9	13.6	55.5	5.9
(E)-2-Nonenal	10.7	15.0	14.0	8.0	1.6	11.2	20.0	0
Decanal	6.0	10.9	12.8	4.2	11.0	4.8	8.0	2.9
(E,E)-2,4-Nonadienal	0	0	0	0	2.4	0	0	0
Total TIC area	71.3	184.2	127.3	183.1	187.4	124.5	610.9	21.8

<sup>a</sup> Laboratory-isolated starch.

<sup>b</sup> Commercial starch sample.

<sup>c</sup> Compound was detected but its concentration was less than peak integrator threshold.

TABLE II  
Ketones in Starches and Total Ion Count (TIC) Peak Areas

Compound	TIC Peak Area ( $\times 10^{-6}$ ) of Starches							
	Wheat		Corn		Potato		Waxy Corn	Tapioca
	Lab <sup>a</sup>	Com <sup>b</sup>	Lab	Com	Lab	Com	Com	Com
2-Pentanone	trace <sup>c</sup>	0	0	0	0	0	0	0
2-Heptanone	trace	4.8	0	3.9	3.4	7.7	18.0	trace
2-Octanone	0	1.8	0	trace	0	0	24.2	0
6-Methyl-5-hepten-2-one	1.8	0	1.5	0	1.6	0	6.0	0
3-Octen-2-one	2.4	2.3	3.7	3.0	0	0	17.9	0
2-Nonanone	0	2.0	0	0	0	0	0	0
Total TIC area	4.2	10.9	5.2	6.9	5.0	7.7	66.1	0

<sup>a</sup> Laboratory-isolated starch.

<sup>b</sup> Commercial starch sample.

<sup>c</sup> Compound was detected but its concentration was less than peak integrator threshold.

rate was  $\approx 1.7 \text{ cm}^3/\text{min}$ . Flow rate was held constant by automatically increasing pressure as oven temperature was increased. Separation of volatiles was achieved by initially holding the oven temperature at  $50^\circ\text{C}$  for 2 min, increasing it to  $140^\circ\text{C}$  at a rate of  $7^\circ\text{C}/\text{min}$ , then increasing it to  $260^\circ\text{C}$  at  $17.5^\circ\text{C}/\text{min}$ , and finally holding it at  $260^\circ\text{C}$  for 1 min. The temperature of the GC injector zone under the capillary interface module was maintained at  $250^\circ\text{C}$ . The effluent from the column first passed through the FTIRD and then into the MSD, and mass and infrared spectral data were recorded simultaneously.

The FTIRD conditions included transfer lines and flow cell temperatures that were maintained at  $250^\circ\text{C}$ , a liquid nitrogen-cooled Hg-Cd-Te detector ( $750\text{--}4,000 \text{ cm}^{-1}$ ), and a spectral resolution of  $16 \text{ cm}^{-1}$  at a scan rate of 2.6 scans/sec. The MSD conditions were detector temperature,  $280^\circ\text{C}$ ; ionization voltage, 70 eV; mass range, mass/charge 33–250 amu; scan rate, 2.7 scans/sec; and electron multiplier voltage, 2,600V. The total time to trap the volatiles and run a chromatogram was 50–60 min per sample.

### Identification of Volatile Compounds

Identification of volatiles was based primarily on computer matching of the observed mass spectral data with the standard mass spectra in the HP Wiley 138 spectral database (Hewlett Packard Co., Palo Alto, CA). Selected ion chromatograms for specific ions or ion ranges also were used to identify specific compounds. The

identities of some volatiles that have strong infrared absorption spectra at  $750\text{--}4,000 \text{ cm}^{-1}$  were confirmed through computer matching of the experimental infrared spectra with the standard infrared spectra in the vapor-phase 1 and 2 libraries (Bio-Rad Laboratories, Sadtler Division, Cambridge, MA). The relative retention times of a homologous series of compounds also confirmed the identity of certain volatiles. The identity of some volatiles that are marked with an asterisk (\*) in the tables should be considered tentative.

The total ion chromatogram (TIC) peak areas obtained through the MSD provided an estimate of the relative abundances of volatile compounds and their levels in the starch samples. Replicates were limited to a few samples because 50–60 min were required to analyze each sample. Based on duplicate runs on two samples, the TIC were reproducible to at least  $\pm 19\%$ . Seitz (1995) found that the overall coefficient of variation among the concentrations of compounds identified in replicates of wheat samples in the same instrumental setup averaged 17.1% with a range of 13.0–20.5%.

## RESULTS AND DISCUSSION

Volatile aldehydes identified in the starches, along with their TIC peak areas, are compared in Table I. The rankings of the starches for total aldehyde levels as indicated by the sums of the TIC peak areas were waxy corn  $\gg$  laboratory-prepared potato  $\approx$  commercial wheat  $\approx$  commercial corn  $>$  laboratory-isolated corn  $\approx$  commercial

TABLE III  
Alcohols in Starches and Total Ion Count (TIC) Peak Areas

Compound	TIC Peak Area ( $\times 10^{-6}$ ) of Starches							
	Wheat		Corn		Potato		Waxy Corn	Tapioca
	Lab <sup>a</sup>	Com <sup>b</sup>	Lab	Com	Lab	Com	Com	Com
2-Propanol	0	2.8	0	0	0	0	0	96.0
Ethanol	2.7	0	5.4	0	0	0	0	0
3-Methyl-2-hexanol	0	0	4.1	0	0	0	0	0
1-Propanol	0	0	3.0	0	0	0	0	0
1-Butanol	12.0	9.3	21.8	0	9.5	0	0	0
1-Pentanol	1.2	13.6	4.2	0	0	3.5	21.1	2.3
1-Hexanol	10.4	12.6	1.3	0	0	0	0	0
2-Heptanol	0	0	0	0	0	2.6	0	0
1-Heptanol	0	6.9	0	0	0	19.5	0	0
1-Octen-3-ol	0	4.0	0	0	0	8.5	0	0
2-Ethyl-1-hexanol	2.7	68.2	2.7	0	1.9	17.3	10.6	10.1
1-Octanol	0	0	0	0	0	9.6	0	0
Total TIC area	29.0	117.4	42.5	0	11.4	61.0	31.7	108.4

<sup>a</sup> Laboratory-isolated starch.

<sup>b</sup> Commercial starch sample.

TABLE IV  
Esters in Starches and Total Ion Count (TIC) Peak Areas

Compound	TIC Peak Area ( $\times 10^{-6}$ ) of Starches							
	Wheat		Corn		Potato		Waxy Corn	Tapioca
	Lab <sup>a</sup>	Com <sup>b</sup>	Lab	Com	Lab	Com	Com	Com
Ethyl acetate	3.2	0	3.8	0	0	0	0	0
2-Methyl-1-butyl acetate	0	0	0	0	0	0	33.1	3.2
Ethyl butanoate	0	2.5	0	0	0	0	0	0
Pentyl acetate	0	0	0	0	0	0	247.7	8.3
6-Methylheptyl-2-propionate* <sup>c</sup>	0	7.9	0	0	0	0	0	0
Butyl benzoate	0	1.9	0	0	0	0	0	0
Benzyl acetate	0	0	0	0	0	0	40.2	3.9
Methyl-2-hydroxy benzoate	0	0	0	41.0	0	0	0	13.7
Isobornyl acetate	0	0	0	0	0	0	9.5	0
Acetic acid	2.8	0	0	0	0	3.0	0	0
Total TIC area	6.0	12.3	3.8	41.0	0	3.0	330.5	29.1

<sup>a</sup> Laboratory-isolated starch.

<sup>b</sup> Commercial starch sample.

<sup>c</sup> Asterisk (\*) indicates tentative identification.

potato > laboratory-isolated wheat > tapioca. Hexanal, which is the major product of oxidative degradation of linoleic acid (Frankel 1982, Kaced et al 1984, Belitz and Grosch 1987), was the most abundant compound detected in the starches. Three other aldehydes, nonanal, decanal, and benzaldehyde, also were common to all starches. All starches except tapioca contained unsaturated aldehydes, which have low odor threshold values (Belitz and Grosch 1987).

Potato and cassava tubers contain 24 and 38% solids with 0.1 and 0.8% lipids, respectively, which is lower than the ≈1–2% level of nonstarch lipids in corn and wheat endosperms (Codd et al 1975). In spite of the low level of potato lipids, aldehydes still were present in potato starch. Potato tubers are stored for months by farmers and two to three days by wet-millers (Bergthaller et al 1999), during which time lipids undergo oxidation in bruised tissue. On the other hand, cassava tubers are wet-processed into starch within 24 hr of harvest (Rakshit 1999). 2-Heptenal and 2-octenal are prominent autoxidation products of linoleic acid (Belitz and Grosch 1987), but 2-heptenal was found only in waxy corn starch, and 2-octenal was present at low levels (Table I). This may be because 2-alkenals and 2,4-alkadienals are oxidized substantially faster than the unsaturated fatty acids (Belitz and Grosch 1987).

Ketones and alcohols also are common products of the autoxidation of lipids. Waxy corn starch contained the highest level of total ketones (Table II), followed by commercial wheat starch. 2-Heptanone was the only ketone detected in almost all starches. Alcohols were absent or low in commercial corn and waxy corn starches and also in laboratory potato starch, but tapioca starch contained a relatively high level of 2-propanol (Table III). It is possible that the microorganisms present in commercial steep tanks absorb and digest the alcohols present in corn. Acetate and benzoate esters were present in the starches (Table IV). The only free organic acid identified was acetic acid.

Benzene compounds found in the starches are listed in Table V. Methylbenzene (toluene) was common to all starches at various levels. The total level of benzene compounds was low in laboratory-isolated starches. Listed in Table VI are the terpene compounds identified in the starches. In terms of terpene type and abundance, waxy corn and tapioca starches were quite similar. The level of total terpene compounds was the highest in waxy corn starch, followed by commercial tapioca and potato starches. However, no terpene compounds were detected in laboratory-isolated wheat, corn, and potato starches or in commercial wheat and corn starches. The terpenoids may originate from weed seeds or leaf fragments present in the starting materials of commercial starches. The hydrocarbons and other commonly detected compounds in starches are listed in Table VII. 2-Pentylfuran is another volatile product of autoxidation of linoleic and linolenic acids. Geosmin, which is well-known for its “earthy” odor in grains (Smith et al 1994) and

in rangia clam (Tanchotikul and Hsieh 1990), was not detected in potato starch or any other starch.

A summary of the types of compounds detected in the volatiles of the eight starch samples is given in Table VIII. Comparing the grand total of volatiles detected, the order of the starches in decreasing levels of volatiles was waxy corn, commercial wheat, potato, corn, and tapioca; and laboratory-prepared potato, corn, and wheat starches. The reason for the high level of volatiles in waxy corn starch may be due to an unusual sample. Alternatively, waxy corn kernels appear somewhat softer than dent corn kernels (Pomeranz et al 1984), which may result in increased mechanical damage to the germ region of waxy corn kernels during harvesting and handling. Also, waxy corn starch contains almost no amylose, so it may release volatiles readily.

### Sources of Volatiles

The volatiles detected in starches may be contaminants carried over from plant material during isolation of starch. Many of the volatiles in wheat grain (Seitz 1995) and corn grain (L. M. Seitz, *personal communication*) also were found in their starches. Unfortunately, no data are available on the volatiles in cassava and potato tubers. The most prominent volatiles in the starches are common products of lipid oxidation: pentanal, hexanal, heptanal, octanal, nonanal, and decanal; (E) or (Z) isomers of 2-hexenal, 2-heptenal, 2-octenal, 2-nonenal, 2-decenal, 2-undecenal, 2,4-nonadienal, and 2,4-decadienal (Belitz and Grosch 1987); and 1-pentanol, 1-heptanol, 1-octanol, 1-octen-3-ol, 2-pentylfuran, butanal, 2-butenal, and 3-methylketones (Frankel 1982). Those compounds may build up in damaged tissue in the sources and would be absorbed by starch during wet milling. In addition, lipids may be absorbed at the surface of starch, and be autoxidized during storage.

TABLE VI  
Terpene Compounds in Commercial Starch Samples  
and Total Ion Count (TIC) Peak Areas

Compound	TIC Peak Area ( $\times 10^{-6}$ ) of Commercial Starches		
	Potato	Waxy Corn	Tapioca
dl-Limonene / $\Delta$ -3-carene <sup>a</sup>	7.3	15.5	4.4
1,8-Cineole(eucalyptol)	0	20.3	0
<i>t</i> -Ocimene/ $\gamma$ -terpinene/linalool*	23.4	17.5	0
Citronella	0	34.2	9.1
(Z)- $\beta$ -Terpineol*	0	0	4.8
1- $\alpha$ -Terpineol*	0	10.8	13.4
<i>p</i> -Menth-1-en-8-yl acetate ( $\alpha$ -terpinyl acetate)*	0	4.5	9.9
Total TIC area	30.7	102.8	41.6

<sup>a</sup> An asterisk (\*) indicates tentative identification.

TABLE V  
Benzene Compounds in Starches and Total Ion Count (TIC) Peak Areas

Compound	TIC Peak Area ( $\times 10^{-6}$ ) of Starches							
	Wheat		Corn		Potato		Waxy Corn	Tapioca
	Lab <sup>a</sup>	Com <sup>b</sup>	Lab	Com	Lab	Com	Com	Com
Benzene	0	0	0	0	0	7.8	0	0
Methylbenzene	1.6	12.0	2.9	7.7	3.0	5.0	90.7	16.5
Ethylbenzene	0	3.5	0	0	0	14.3	8.4	trace <sup>c</sup>
1,2/3-Dimethylbenzene <sup>d</sup>	0	0	0	0	0	4.9	20.2	1.8
1,3,5-Trimethylbenzene*	0	0	0	0	0	0	3.7	0
Methoxybenzene*	0	2.2	0	0	0	0	0	0
1,3/4-Dichlorobenzene*	0	0	0	0	0	0	8.8	trace
Total TIC area	1.6	17.7	2.9	7.7	3.0	32.0	123.0	18.3

<sup>a</sup> Laboratory-isolated starch.

<sup>b</sup> Commercial starch sample.

<sup>c</sup> Compound was detected but its concentration was less than peak integrator threshold.

<sup>d</sup> Asterisk (\*) indicates tentative identification.

**TABLE VII**  
Some Hydrocarbon and Other Common Compounds in Starches and Total Ion Count (TIC) Peak Areas

Compound	TIC Peak Area ( $\times 10^{-6}$ ) of Starches							
	Wheat		Corn		Potato		Waxy Corn	Tapioca
	Lab <sup>a</sup>	Com <sup>b</sup>	Lab	Com	Lab	Com	Com	Com
Nitromethane	7.7	0	11.8	0	0	0	0	0
Styrene	trace <sup>c</sup>	10.5	0	0	0	5.5	0	0
Hexanenitrile	0	1.6	0	0	4.2	7.8	12.7	0
2-Pentylfuran	4.5	4.9	3.3	2.0	18.9	9.1	24.2	0
1-Nitrohexane	3.1	6.6	2.6	3.0	7.2	18.2	62.0	0
1-Methoxy-4-(2-propenyl)-benzene(Estragole)	0	0	0	0	0	35.3	trace	0
1-Methoxy-4-(1-propenyl)-benzene(Anethole)	0	0	0	0	0	0.3	1.7	4.4
Dodecane	0	4.3	0	0	0	0	0	12.2
Tetradecane	7.1	8.6	13.2	0	trace	0	0	6.1
Pentadecane	14.1	0	14.4	0	2.7	0	0	0
Hexadecane	trace	0	8.1	0	0	0	0	0
Total TIC area	36.5	36.5	53.4	5.0	33.0	76.2	100.6	22.7

<sup>a</sup> Laboratory-isolated starch.

<sup>b</sup> Commercial starch sample.

<sup>c</sup> Compound was detected but its concentration was less than peak integrator threshold.

**TABLE VIII**  
Classes of Volatile Compounds in Starches and Total Ion Count (TIC) Peak Areas

Compound	TIC Peak Area ( $\times 10^{-6}$ ) of Starches							
	Wheat		Corn		Potato		Waxy Corn	Tapioca
	Lab <sup>a</sup>	Com <sup>b</sup>	Lab	Com	Lab	Com	Com	Com
Aldehydes	71.3	184.2	127.3	183.1	187.4	124.5	610.9	21.8
Ketones	4.2	10.9	5.2	6.9	5.0	7.7	66.1	0
Alcohols	29.0	117.4	42.5	0	11.4	61.0	31.7	108.4
Esters	6.0	12.3	3.8	41.0	0	3.0	330.5	29.1
Benzenes	1.6	17.7	2.9	7.7	3.0	32.0	123.0	18.3
Terpenes	0	0	0	0	0	30.7	102.8	41.6
Hydrocarbons and others	36.5	36.5	53.4	5.0	33.0	76.2	100.6	22.7
Total TIC area	148.6	379.0	235.1	243.7	239.8	335.1	1365.6	241.9

<sup>a</sup> Laboratory-isolated starch.

<sup>b</sup> Commercial starch sample.

As mentioned before, water-slurried starch granules were shown to absorb methanol, ethanol, 1-propanol, and 1-butanol (BeMiller and Pratt 1981), whereas those in the semidry state adsorbed propionic acid, 1-hexanol, benzaldehyde, dodecane, diacetyl, and ethyl acetate (Hau et al 1996). The roles of the volatiles and their contributions to unique off-odors and flavors of starches were not assessed in this study. The odor-taste threshold values and quantitative analyses are needed to determine the contribution of individual volatiles to starch odor-taste.

Future work planned includes 1) a repeat of the experiments on new starch samples including hand-harvested samples, 2) identification of the volatiles that define the odor of a starch, and 3) finding methods to prevent the formation of volatiles or to remove them.

## CONCLUSIONS

The major volatiles in cereal and root starches were common breakdown products of autoxidation of lipids, especially linoleic acid. It may be speculated that bruised tissue in stored grain or tubers contributes to those volatiles. Tapioca starch contained few volatiles with the exception of 2-propanol, which agrees with it being a bland tasting starch. On the other hand, a sample of waxy corn starch released the highest level of volatiles, whereas wheat, corn, and potato starches contained intermediate levels.

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