Sensory and Chemical Studies of Lipid Oxidation in Raw and Heat-Treated Oat Flours

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ABSTRACT

The storage stability of oat flours (*Avena sativa* L.) was investigated by sensory and chemical methods. Raw and heat-treated flour samples of three cultivars of oats (Kapp, Mustang, and Svea) were stored at 23°C and 50% relative humidity for 0, 5, 18, and 42 weeks before analyses. Descriptive sensory analysis and analyses of total fatty acids (FA), free fatty acids (FFA) and volatile lipid oxidation products were performed after each storage period. Storage of raw flours for five weeks resulted in 66% FFA but stable levels of flavors and volatile compounds. After 18 weeks, the level of volatiles and FFA was higher, while the samples at 42 weeks had an intense paint flavor, high levels of several volatiles, and reduced levels of FA and FFA. The major volatiles in stored oat flours were hexanal and 2-pentyl-furan. The other carbonyls were mainly aldehydes. For raw oat flours, most correlation coefficients between volatiles and the attributes of paint odor, paint flavor, odor intensity, and flavor intensity were >0.90. The volatiles were negatively correlated to oat odor and flavor and sweetness (r = –0.80 to –0.90). Heat-treatment of oat flours reduced the levels of FA, FFA, and most flavors, particularly bitterness and astringency. The levels of hexanal and oat flavor increased, while most volatiles remained constant. Stability against lipid oxidation was greatly increased by heat treatment. The levels of volatiles in heat-treated samples were less well correlated to flavors. Thus, differently heat-treated samples should be assessed separately in future flavor prediction models.

Compared to wheat, the lipid content of oats is ≈5 times higher (Percheron and Löliger 1990) and the lipolytic enzymes are 10–15 times more active (Matlashewski et al 1982). Milling to flours allows lipids and enzymes to react and release free fatty acids (FFA), which are much more susceptible to lipid oxidation than the original lipids. Oats contain a variety of antioxidants, including tocopherols and phenolic acids (Kalbasi-Ashtari and Hammond 1977), and are thus considered to be fairly stable towards nonenzymatic oxidation (Percheron and Löliger 1990). However, high levels of unsaturated FFA and the presence of lipoxygenase favor lipid oxidation. High moisture levels may further enhance the enzymic oxidation, and exposure to heat, oxygen, catalysts, or light accelerates nonenzymic oxidation (Galliard 1994). While most oats for feed are enzyme-active, most commercially processed oats for human consumption are stabilized by heat-treatment. An adequate heat process inactivates lipolytic enzymes and develops the characteristic, pleasant flavor that is associated with high-quality commercial oat products. Optimum stability is expected after a heat treatment strong enough to inactivate lipolytic enzymes but mild enough to protect the natural antioxidants in oats and prevent excessive oxygen exposure due to drying (Galliard 1994).

The storage stability of cereals may be studied by sensory or chemical methods. Studies of milled, rolled and whole cereals by use of descriptive sensory analysis and chemical methods have been reported in rice (Paule and Powers 1989, Piggott et al 1991), pearl millet (Lai and Varriano-Marston 1980), and oats (Dahl et al 1989; Molteberg et al, *in press*). Oat oil stability has also been studied (Fors and Schlich 1989). Most of the studies investigated the effects of processing and storage conditions and related the sensory attributes related to various lipid oxidation products. In rice, removal of the bran and aleurone layer increased the storage stability and reduced the levels of FFA, hexanal, and carbonyls (Piggott et al 1991). For oats, Fors and Schlich (1989) found more green and rancid flavors and higher levels of aldehydes in heated oils from unprocessed oats than in oils from processed oats. Dahl and co-workers (1989) detected some differences in storage stabilities for differently processed rolled oats, even though most products remained fairly stable for 45 weeks. Molteberg et al (*in press*) reported that the sensory quality of heat-treated oat groats was influenced mainly by the type of heat process used but also by cultivar and storage time for raw groats. The results showed that even though increasing storage humidity and storage time increased the levels of FFA and hexanal, these characteristics did not correspond well with the sensory quality of whole groats.

In the present study, the storage stability of oat flours is investigated. The effects of cultivar and heat-treatment on flavor, fatty acid contents, and content of volatile lipid oxidation products are described, and their changes during storage discussed. Statistical methods are used to relate the volatiles to descriptive sensory analysis of raw and heat-treated oat flours.

MATERIALS AND METHODS

Oat Samples

The oat samples (*Avena sativa* L.) were of three cultivars (Kapp, Mustang, and Svea), grown in 1993 at Apelsvoll Research Station, Norway. A complete factorial design, with three processes (raw + two heat processes) and four storage periods, was used (Fig. 1). Each heat treatment was performed on 2.3 kg of hulled oats or 1.7 kg of groats. The heating procedures included soaking in water for 2 min, followed by steaming for 10 min at 100°C and drying in paper bags at 100°C, as described previously (Molteberg et al 1995). The two heating procedures (without hulls and with hulls) differed by the time of dehulling (before and after heat-treatment) and the duration of the drying (3.5 and 4 hr, respectively). Due to higher volumes of samples heat-treated with hulls, each replicate was also dried in two bags that were thoroughly mixed before milling. Raw and newly processed samples were milled on a Retsch Ultra centrifugal mill, 0.5-mm screen (Kurt Retsch GmbH & Co, Haan, Germany). The oat flours were stored in double, glued paper bags (greaseproof paper with gramage 42 g/m^2 inside and 70 g/m^2 craftpaper outside) under light at 23°C and 50%

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relative humidity. Prior to sensory and chemical analyses after 0, 5, 18, and 42 weeks of storage, one sample was withdrawn from each bag.

Sensory Analysis

Descriptive sensory analysis (ISO 1985) was performed by an accredited sensory laboratory employing a panel of nine part-time assessors. The assessors had 4–19 years of experience in descriptive analyses of food products. They had been selected and trained according to the guidelines of the International Organization for Standardization (ISO 1991). Sensory analyses were performed in a laboratory designed to ISO standards (ISO 1988), with individual booths, standardized lightning, charcoal filter, and positive pressure. Before profiling, two sessions were used to train the assessors in the definition of 16 selected odor, flavor, and texture attributes (Table I). Two very different samples were used for this. A 15-cm continuous unstructured scale was used for evaluation. The results were transferred onto a 9-point scale, where the left side of the scale (0 cm) corresponded to no intensity (value 1.0), and the right side (15 cm) to strong intensity (value 9.0) of each attribute. One unprocessed sample and one reference sample stored at –80°C was served before the evaluation at each storage period.

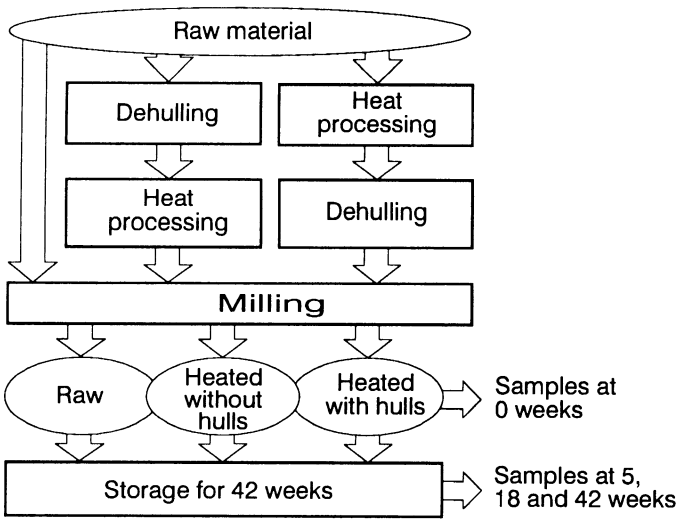

Fig. 1. Flow diagram for processing and storage of oat samples.

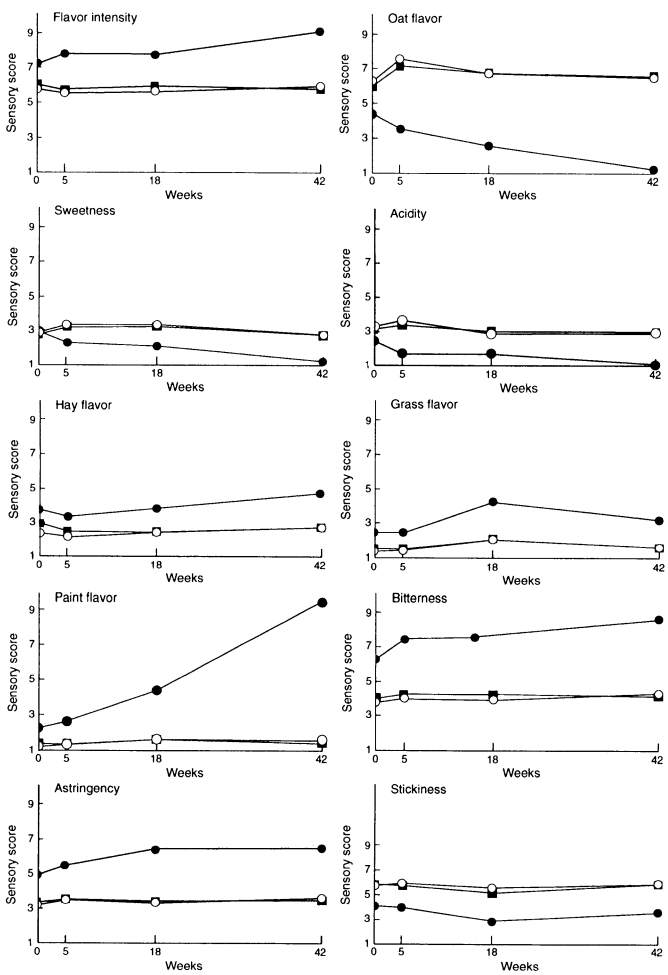

Fig. 2. Sensory scores from 10 selected flavor and texture attributes. Average scores adjusted relative to the reference sample. ● = raw, ■ = heat-treated with hulls, ○ = heat-treated without hulls.

The reference sample was included in all sensory analysis to control the drift of the panel. The assessors used water and bland crackers for palate cleansing between the samples.

The samples (60 g of oat flour and 210 ml of distilled water) were prepared in 300-ml beakers and heated in a waterbath at 65°C for 10 min. Stirring every minute ensured even temperatures and a smooth paste. One tablespoon (15 g) of each sample was served each assessor in a plastic tray. The serving temperature was ≈60°C. Each sample was served four times (two from each heat-treatment duplicate). The total sample order was randomized and each session of five samples was randomized for each assessor. Monadic evaluation was performed and the data was recorded directly on a computerized system (Compusense Inc., Guelph, ON, Canada).

To compare samples across storage periods, all results from sensory analyses were adjusted relative to the reference. This eliminates the effect of drift in the panel but is based on the assumption that the reference remains constant during storage at –80°C for 42 weeks. All results for a given attribute and storage period were adjusted by the constant:

$$K = \mathrm{ref}_0/\mathrm{ref}_x$$

where ref_x (x = 0, 5, 18, or 42) gives the intensity of the corresponding reference sample at each of the different storage periods.

Chemical Analysis

Moisture contents were analyzed by drying at 105°C (AOAC 1990). The contents and compositions of total fatty acids (FA) and

TABLE I
Sensory Attributes and Results from Analysis of Variance (ANOVA) of Freshly Processed Oat Flours at 0 Weeks

Attribute	Description	S_{tot}[a]	MSE[b]
Odors			
Intensity	Intensity of all odors	0.68	0.31
Oat	Odor of oats	0.92	0.56
Raw	Odor of raw, untreated grains	1.93	0.95
Hay	Odor of dry hay, dust	0.52	0.84
Paint	Odor of paint, turpentine, diesel	0.44	0.24
Flavors			
Intensity	Intensity of all flavors	0.70	0.28
Oat	Flavor of oats	0.96	0.58
Sweetness	Taste of sucrose	0.31	0.36
Acidity	Related to a mild flavor of acid	0.40	0.49
Raw	Flavor of raw, untreated grains	1.90	0.81
Hay	Dry hay, dust	0.55	1.06
Grass	Green grass	0.62	0.40
Paint	Paint, turpentine, diesel	0.52	0.31
Bitterness	Related to bitter taste (quinine, caffeine)	1.27	0.78
Texture			
Astringency	Contracting, astringent mouthfeel	0.89	0.45
Stickiness	Coherent, sticky, adhesive mouthfeel	0.82	0.38

[a] Total standard deviation.
[b] Mean square error from two-way ANOVA describing noise in sensory data.

free fatty acids (FFA) were determined by the gas chromatographic methods described by Molteberg et al (1995). FA were extracted by a mixture of chloroform, methanol, and water-saturated *n*-butanol while FFA were extracted by 96% ethanol and separated from other lipids by thin-layer chromatography. Trinonadecanoin and nonadecanoic acid, respectively, were used as internal standards. The results are expressed as grams of fatty acid in 100 g of dry groats. Volatile lipid oxidation compounds were analyzed by static head-space gas chromatography and mass spectroscopy (Molteberg et al 1995). The results are expressed relative to the amount of internal standard (IS, toluene) in the head-space gas (area of volatile compound × 100/area of IS). All chemical analyses were performed at least in duplicate.

Statistics

Analysis of variance (ANOVA) and Tukey's multiple comparison test were performed by the general linear model (GLM) procedure (version 6.04, SAS Institute, Cary, NC). Differences among samples are reported to be significant when $P < 0.05$. Mean square error (MSE) for the various sensory attributes was calculated by two-way analysis of variance (ANOVA) with samples and assessors as independent class-variables. MSE is related to the inconsistency between sensory replicates and describes the uncertainty of the sensory method. Total standard deviation (S_{tot}) and multivariate methods are based on average scores for two heating replicates, two sensory replicates, and nine assessors.

The multivariate methods principal component analysis (PCA) and partial least squares regression (PLS2) were performed by the Unscrambler-Extended version 5.5 software package (Camo a/s, Trondheim, Norway). PCA was performed to study the main structures of variation in sensory attributes. PCA calculates the linear combinations (principal components or factors) of the data that describes as much of the variance in the original data as possible. The factors are presented graphically in score plots (plots of samples) and loading plots (plots of variables). Score plots reveal the most important sample structures, with samples in the same area of the plot having important similarities. The samples are related to high levels of the variables that are located in the corresponding area of the loading plot. PLS2 (Martens and Næs 1989) was used to select volatile compounds with a major influence on flavor for use in further studies. From the content of these volatiles (X-data), PLS2 estimated the total predictability of the 16 sensory attributes (Y-data). PLS2 is a linear method that makes a model by extracting the information in the X-data that has the largest possible covariance with the Y-data. The method handles collinearity by compression of the data onto independent latent variables. All calibrations were performed on data standardized to equal variance and the models were validated by full cross validations.

RESULTS

Sensory Analysis

The sensory scores for raw and heat-treated samples from nine selected flavor attributes and stickiness are shown in Figure 2. The scores are mean values for the three cultivars. The odor attributes were generally highly correlated to their corresponding flavor attributes (results not shown). The largest effects of heat processing were found for the attributes raw odor and flavor, bitterness, and oat odor

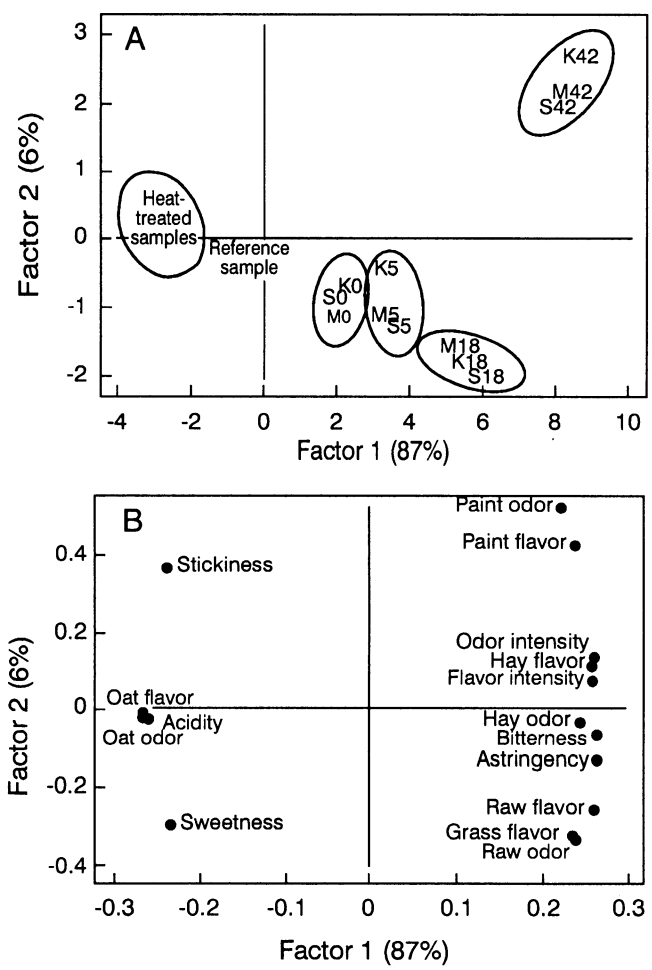

Fig. 3. Principal component analysis scores (**A**) and loadings (**B**) of sensory data for raw and heat-treated samples. K = Kapp, M = Mustang, S = Svea. Numbers indicate storage period (0, 5, 18, and 42 weeks)

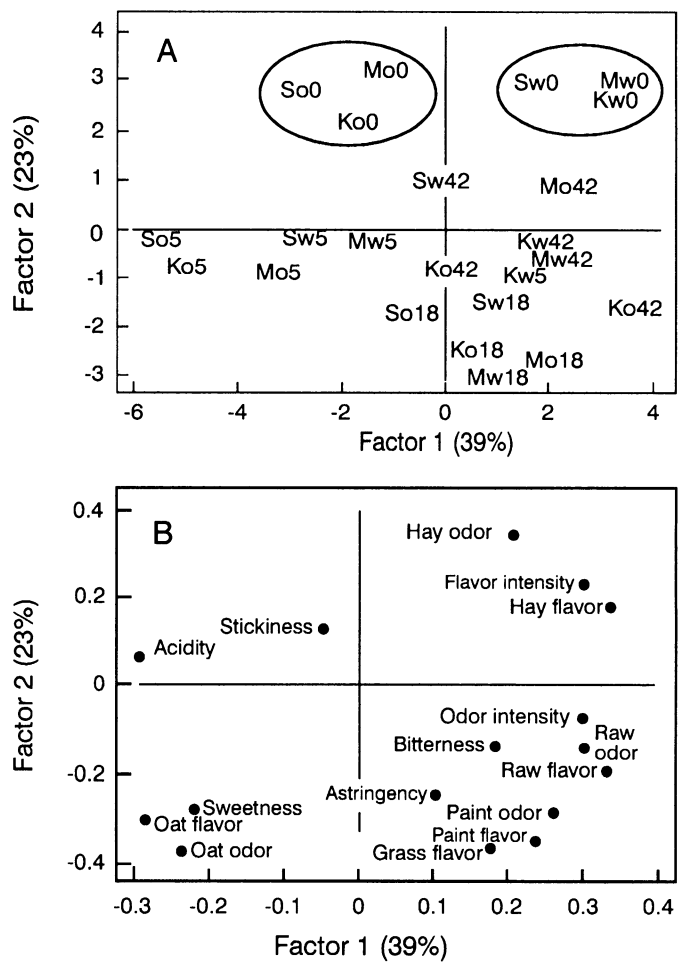

Fig. 4. Principal component analysis scores (**A**) and loadings (**B**) of sensory data for heat-treated samples. K = Kapp, M = Mustang, S = Svea, w = heat-treated with hulls, o = heat-treated without hulls. Numbers indicate storage period (0, 5, 18, and 42 weeks)

and flavor. This is suggested from the large total standard deviations (S_{tot}) at 0 weeks (Table I). Figure 2 shows the effects for bitterness and oat flavor. High errors (MSE) for raw odor and flavor, hay odor and flavor, and bitterness indicated that these attributes were difficult to evaluate consistently between sensory replicates. Heat processing affected significantly all sensory attributes except sweetness (results from ANOVA not shown).

The sensory quality changed during processing, but was fairly stable in processed flours (Fig. 2). There are major changes in some of the attributes during storage of raw oats. Storage of raw flours increased the varietal differences (results not shown). After 42 weeks of storage the cultivar Kapp had significantly more paint odor and flavor intensity than did Svea. In heat-treated oats, samples of Kapp were clearly less sticky than samples of Svea and Mustang at all storage periods.

The main structures in the sensory data are presented in the PCA plots in Figure 3. A total of 87% of the variation among samples is described in the first factor, while factor 2 explains 6%. Factor 1 in the score plot (Fig. 3A) separates all raw samples from the heat-treated samples. The different storage periods for raw samples are separated both by factor 1 and 2. The corresponding loading plot (Fig. 3B) shows that heat-treated samples had the highest levels of oat odor and flavor, acidity, sweetness, and stickiness, while the other attributes were characteristic for stored raw flours. Raw oats stored for 18 weeks had relatively high levels of grass flavor and raw odor and flavor, while paint odor and flavor increased during further storage.

When disregarding all raw samples, PCA also found some systematic differences among heat-treated samples (Fig. 4). Factor 1 and 2 explained 39 and 23%, respectively, of the variation among the heated samples. Factor 1 clearly separated the two heat processes when the samples were freshly processed (0 weeks), but this difference decreased and disappeared during storage. The difference between cultivars, with samples of Svea located to the left of comparable samples for Mustang and Kapp, was consistent during storage. Results from PCA indicate that samples of Svea, particularly when fresh and processed without hulls, had more oat odor, oat flavor, sweetness, and acidity than the others. Factor 2 shows how the adjusted sensory data differs between storage periods.

Moisture Content

There were no systematic moisture differences among processes for newly processed flours (Table II). For stored flour, however, raw flour had significantly higher moisture levels. The effect of groat processing was similar for all cultivars, and the average moisture loss due to milling was 0.7 percent points (results not shown). During storage of the flours for five weeks, the moisture contents in raw oats equilibrated from an average of 9.3 to 11.3% (Table II). Stored heat-treated oats had 10.1–10.5% moisture. Results from ANOVA (not shown) also showed that moisture content was significantly affected by storage time (generally 42 > 18 = 5 > 0). Heat treatment × storage time was the only significant interaction.

Total Fatty Acids

Raw samples of the cultivar Kapp had significantly higher contents of total fatty acids than did raw samples of the other two varieties (Table III). The difference between Svea and Mustang was small but significant. Samples of Kapp had the highest contents of palmitic, oleic, and linoleic acids. The proportion of linoleic acid was highest in Svea, while Kapp contained the highest proportion of oleic acid.

The changes in content of individual and total fatty acids during processing and storage are presented in Table IV. Average levels for the three cultivars are presented. ANOVA confirmed that the reduction in fatty acids during heat treatment was similar for the three cultivars, while the reduction during storage, particularly for raw oats, depended on the cultivar (results not shown). The changes were generally 20–50% greater in samples of Kapp than of Mustang and Svea (results not shown). Fresh, raw flours contained an average of 7.8 g/100 g of dry matter, of which oleic (C18:1) and linoleic (C18:2) acids consisted of ≈40% each; palmitic acid (C16:0) consisted of 15%; and stearic (C18:0) and linolenic (C18:3) acids consisted of 1.4% each. The levels of total fatty acids decreased significantly during storage of raw oat flours, particularly between 18 and 42 weeks. The most extensive reduction occurred in the two polyunsaturated fatty acids, but even the levels of the monounsaturated oleic acid were significantly affected.

Heat processing (at 0 weeks) reduced the average total content of

TABLE II
Moisture Content (%) in Raw and Heat-Treated Oat Flours[a,b]

Weeks	Raw	Heated	
		With Hulls	Without Hulls
0	9.3 a	9.1 a	9.2 a
5	11.3 a	10.3 b	10.1 c
18	11.3 a	10.2 b	10.3 b
42	11.8 a	10.5 b	10.4 b

[a] Values in the same row sharing the same letter are not significantly different ($P > 0.05$).
[b] Each value is based on three cultivars and four replicates ($n = 12$).

TABLE III
Individual and Total Fatty Acid Content in Raw Oat Flours Before Storage (g/100 g of dry matter)[a,b]

Fatty Acid	Cultivar		
	Kapp	Mustang	Svea
Total	8.32 a	7.45 b	7.33 c
Palmitic C16:0	1.22 a	1.13 b	1.05 c
Stearic C18:0	0.104 c	0.113 b	0.117 a
Oleic C18:1	3.42 a	2.95 b	2.87 c
Linoleic C18:2	3.47 a	3.15 b	3.19 b
Linolenic C18:3	0.112 a	0.112 a	0.107 b

[a] Values in the same row sharing the same letter are not significantly different ($P > 0.05$) ($n = 4$).
[b] Other fatty acids consist of a total of ≈0.08 g/100 g of dry matter.

TABLE IV
Individual and Total Fatty Acid (FA) Content in Raw and Heat-Treated Oat Flours During Storage (g/100 g dry matter)[a,b]

Flour	FA %[c]	Weeks of Storage			
		0	5	18	42
Raw					
Total FA		7.77 a	7.69 a	7.52 b	6.71 c
Palmitic C16:0	14.7 (0.4)	1.13 b	1.13 b	1.14 b	1.18 a
Stearic C18:0	1.4 (0.2)	0.111 a	0.107 b	0.109 b	0.111 a
Oleic C18:1	39.9 (0.9)	3.08 a	3.06 ab	3.01 b	2.80 c
Linoleic C18:2	42.5 (0.8)	3.27 a	3.21 b	3.09 c	2.48 d
Linolenic C18:3	1.4 (0.1)	0.110 a	0.106 b	0.100 c	0.072 d
Heated with hulls					
Total FA		7.38 a	7.10 b	7.08 b	7.11 b
Palmitic C16:0	14.5 (0.4)	1.06 b	1.03 c	1.06 b	1.09 a
Stearic C18:0	1.5 (0.2)	0.106 a	0.100 c	0.104 b	0.106 a
Oleic C18:1	40.2 (0.9)	2.94 a	2.85 b	2.85 b	2.88 b
Linoleic C18:2	42.4 (0.8)	3.10 a	2.95 b	2.90 bc	2.88 c
Linolenic C18:3	1.4 (0.1)	0.101 a	0.095 b	0.091 c	0.088 d
Heated without hulls					
Total FA		7.35 a	7.19 b	7.24 ab	7.12 b
Palmitic C16:0	14.5 (0.4)	1.06 b	1.06 b	1.09 a	1.10 a
Stearic C18:0	1.4 (0.2)	0.105 a	0.102 b	0.105 a	0.105 a
Oleic C18:1	40.1 (0.9)	2.93 a	2.88 b	2.91 ab	2.87 b
Linoleic C18:2	42.5 (0.8)	3.09 a	2.98 b	2.98 b	2.89 c
Linolenic C18:3	1.4 (0.1)	0.102 a	0.097 b	0.095 c	0.089 d

[a] Values in the same row sharing the same letter are not significantly different ($P > 0.05$).
[b] Results are means of three cultivars and four replicates ($n = 12$).
[c] In fresh flours. Standard deviations in parentheses.

fatty acids significantly (0.4 g/100g of dry matter), but had little influence on the relative composition of the fatty acids (Table IV). The oats heat-treated by the two different procedures were equally stable during storage. Both processes resulted in small but significant and consistent reductions in the levels of C18:2 and C18:3.

Free Fatty Acids

Storage of raw oat flours resulted in major changes in both the content and the composition of FFA (Table V). The major liberation of FFA occurred during the first five weeks, while the highest amount (6.4 g/100 g of dry matter) was found after 18 weeks. Further storage up to 42 weeks reduced the average content to 5.3 g/100 g of dry matter. The relative composition of FFA changed during storage, resulting in a decreased ratio of unsaturated versus saturated FFA. This is seen by fairly low net increases in the levels of C18:2 and C18:3 from 0 to 18 weeks, while the net reductions were greater from 18 to 42 weeks. The ratio of C18:2 to C16:0 and C18:3 to C16:0 were 3.1 and 0.11, respectively, at 0 weeks, and 1.6 and 0.04, respectively, at 42 weeks. The relative levels of C18:1 to C18:0 also decreased during storage. The ratios were 2.6 at 0 weeks and 2.1 at 42 weeks.

TABLE V
Individual and Total Free Fatty Acid (FFA) Content in Raw and Heat-Treated Oat Flours During Storage (g/100 g dry matter)[a,b]

Flour	FFA %[c]	Weeks of Storage 0	5	18	42
Raw					
Total FFA		0.45 c	5.07 b	6.41 a	5.28 b
Palmitic C16:0	14.3 (0.9)	0.066 c	0.796 b	1.171 a	1.125 a
Stearic C18:0	1.3 (0.2)	0.006 c	0.076 b	0.102 a	0.105 a
Oleic C18:1	37.8 (1.5)	0.173 d	2.155 c	2.686 a	2.289 b
Linoleic C18:2	44.9 (1.5)	0.202 d	1.990 b	2.383 a	1.723 c
Linolenic C18:3	1.6 (0.1)	0.007 d	0.056 b	0.065 a	0.041 c
Heated with hulls					
Total FFA		0.084 b	0.087 b	0.172 a	0.216 a
Palmitic C16:0	19.5 (1.1)	0.016 b	0.015 b	0.031 a	0.036 a
Stearic C18:0	2.4 (0.5)	0.0019 b	0.0016 b	0.0029 a	0.0033 a
Oleic C18:1	28.9 (2.5)	0.025 b	0.028 b	0.061 a	0.081 a
Linoleic C18:2	47.8 (1.2)	0.040 b	0.041 b	0.075 ab	0.093 a
Linolenic C18:3	1.5 (0.2)	0.0012 b	0.0012 b	0.0024 a	0.0028 a
Heated without hulls					
Total FFA		0.065d	0.091 c	0.140 b	0.167 a
Palmitic C16:0	18.7 (0.4)	0.012 D	0.016 c	0.025 b	0.029 a
Stearic C18:0	2.1 (0.3)	0.0014 b	0.0017 b	0.0025 a	0.0028 a
Oleic C18:1	30.5 (1.8)	0.020 d	0.030 c	0.048 b	0.061 a
Linoleic C18:2	46.9 (1.5)	0.031 c	0.041 b	0.062 a	0.072 a
Linolenic C18:3	1.9 (0.3)	0.0012 c	0.0015 b	0.0021 a	0.0023 a

[a] Values in the same row sharing the same letter are not significantly different ($P > 0.05$).
[b] Results are means of three cultivars and four replicates ($n = 12$).
[c] In fresh flours. Standard deviations in parentheses.

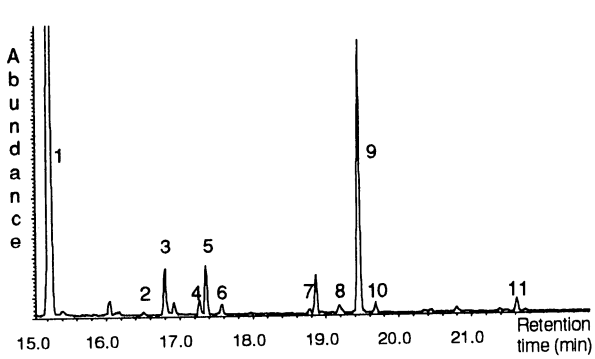

Fig. 5. Representative chromatogram of volatile lipid oxidation products in raw oat flours after 18 weeks of storage. Eleven selected chromatographic peaks are indicated (identity shown in Table VI)

Samples analyzed immediately after heat processing (at 0 weeks) had significantly lower contents of all FFA than did the raw oats sample, but particularly so for oleic acid (Table V). Storage of heat-treated flours resulted in low, but significant, increases in the amounts of FFA. The greatest increases were found for oleic acid. The cultivars and processes were very similar in levels and patterns of variation, and the difference between heat procedures in Table V is caused by higher levels of FFA in one replicate of Kapp heat-treated with hulls (up to 0.44 g/100 g of dry matter).

Volatile Lipid Oxidation Products

A representative chromatogram from analysis of volatile lipid oxidation products is given in Figure 5. The 11 chromatographic peaks with the strongest relationship to flavor were selected and subjected to identification and statistical analysis. Their identities and areas relative to the internal standard, averaged for the three cultivars, are given in Table VI. Hexanal (peak 1) was the predominant volatile in raw oats and had the greatest change in concentration during heating or storage. The other major volatile compound in stored, raw flours was peak 9, which is highly probable from MS-spectra and literature (Chang et al 1966) to be 2-pentyl-furan. Next comes peak 5 (unidentified). The volatiles with the medium-size

TABLE VI
Content and Tentative Identification of Selected Volatile Compounds in Raw and Heat-Treated Oat Flours[a,b,c]

Peak	Identity	Process[d]	Weeks of Storage 0	5	18	42
1	Hexanal	R	7.6 c	14.8 c	163 b	1524 a
		Hw	22 d	34 c	74 b	115 a
		Ho	21 d	39 c	84 b	138 a
2	Hexenal	R	0 b	0 b	0.7 b	8.4 a
		Hw	0.3 c	0.1 c	0.7 b	1.0 a
		Ho	0.2 b	0.1 b	0.6 a	0.7 a
3	1-Hexanol + shoulder	R	5.6 c	1.0 d	11.2 b	18.1 a
		Hw	0.5 b	0.3 b	0.9 a	0.9 a
		Ho	0.3 b	0.2 b	0.8 a	0.2 b
4	2-Heptanon	R	0.4 c	0.3 c	2.6 b	15.6 a
		Hw	0.3 c	0.2 d	0.8 b	1.0 a
		Ho	0.3 c	0.2 c	0.8 b	1.1 a
5	Unidentified	R	0.2 c	0.6 c	8.7 b	45.6 a
		Hw	0.1 c	0.2 c	0.6 b	1.0 a
		Ho	0.1 c	0.2 c	0.6 b	1.0 a
6	Heptanal	R	0.7 b	0.8 b	2.4 b	16.2 a
		Hw	1.0 b	1.0 b	2.5 a	2.2 a
		Ho	0.7 b	0.7 b	2.0 a	2.0 a
7	Heptenal	R	0 b	0 b	0.7 b	3.2 a
		Hw	0.2 b	0.1 b	0.6 a	0.7 a
		Ho	0.1 b	0.1 b	0.5 a	0.4 a
8	1-Octen-3-ol + shoulder	R	0.3 b	0 b	1.4 b	11.1 a
		Hw	0.4 b	0.2 b	0.9 a	0.2 b
		Ho	0.3 b	0.1 b	0.8 a	0.2 b
9	2-Pentyl-furan	R	1.3 c	5.4 c	48 b	254 a
		Hw	1.3 c	1.5 c	3.9 b	7.1 a
		Ho	1.1 c	1.4 c	3.5 b	6.3 a
10	Oktanal	R	0.1 b	0.3 b	1.8 b	5.2 a
		Hw	0.3 b	0.4 b	1.0 a	1.1 a
		Ho	0.3 b	0.4 b	1.0a	11.1 a
11	Nonanal	R	0.3 b	0.4 b	2.2 b	13.8 a
		Hw	0.8 b	0.9 b	1.8 a	1.8 a
		Ho	0.5 b	.5 b	1.5 a	1.6 a

[a] Values in the same row sharing the same letter are not significantly different ($P > 0.05$).
[b] Results are means of three cultivars and four replicates ($n = 12$).
[c] Expressed relative to the amount of internal standard (IS, toluene) in the headspace gas (area of volatile compound × 100/area of IS).
[d] R = raw, Hw = heat-treated with hulls, Ho = heat-treated without hulls.

peaks in raw flours included the saturated aldehydes heptanal, octanal, and nonanal (peaks 6, 10, and 11, respectively), the ketone 2-heptanon (peak 4) and two double peaks (3 and 8) that contained mainly alcohols (1-hexanol and 1-octen-3-ol, respectively). The smallest peaks in raw oats (2 and 7) were identified as hexenal and heptenal. Generally, the content of volatiles in raw flours changed little during the first five weeks of storage. The greatest changes occurred between 18 and 42 weeks storage.

Both heat processes increased the levels of hexanal (peak 1) substantially. Peaks 1 and 3 decreased in relative size, while the others showed minor changes. Storage of heat-treated flours resulted in a small but significant increases in most of the volatiles. The main increase occurred from 5 to 18 weeks of storage. Significant increases from 18 to 42 weeks were found only for peaks 1, 4, 5, and 9 . Hexanal (peak 1) had by far the largest increase during storage, followed by 2-pentyl-furan (peak 9) (Table VI).

Significant varietal differences for raw samples were found for hexanal and 2-pentyl-furan (results not shown). The varietal differences increased during storage, and only the levels of heptenal did not differ by cultivar after 42 weeks of storage. Generally, Kapp contained significantly higher levels of volatiles than Mustang and Svea. Varietal differences were much smaller in the heat-processed samples, but Svea mostly had lower levels of hexanal and 2-pentyl-furan than did Kapp and Mustang.

Relationship Between Chemical and Sensory Analysis

The individual correlation coefficients between selected volatiles and the various odors and flavors are presented separately for raw oats (Table VII) and heat-treated oats (Table VIII). The overall highest correlations were found for the raw samples, but the heat-treated samples generally had the highest correlations for grass flavor and raw odor and flavor. Sweetness, acidity and oat odor and flavor were negatively correlated to the volatiles, while the majority of attributes were positively correlated.

For raw samples, high correlations ($r > 0.90$) were found between most volatiles and odor and flavor intensity and between volatiles and paint odor and flavor. Peak 3, which contained 1-hexanol, had slightly lower coefficients for these attributes, but it had the overall highest correlation to raw odor and flavor and hay odor and flavor. Correlation coefficients >0.95 were achieved between combinations of odor intensity, paint odor and paint flavor, and the volatiles hexanal (peak 1), 2-heptanon (peak 4), the unknown peak 5, heptenal (peak 7), and 2-pentyl-furan (peak 9). For most other combinations of volatiles and attributes was $r > 0.7$, while astringency had r values between 0.61 and 0.72, and raw odor and flavor and grass flavor had coefficients mostly <0.4. For heat-treated samples, all correlation coefficients were <0.80. Nonanal was the volatile with the highest correlation to flavors of heat-treated flours. The sensory attributes with the highest coefficients were paint flavor, paint odor, acidity, and grass flavor ($r = 0.37$–0.80).

TABLE VII
Coefficient of Correlation (r)[a] Between Selected Volatiles and Sensory Attributes[b] for Raw Samples

		Odors					Flavors									Texture
Peak	Identity	Inten.	Oat	Raw	Hay	Paint	Inten.	Oat	Sweet	Acidity	Raw	Hay	Grass	Paint	Bitt.	Astrin.
1	Hexanal	0.97	–0.88	···[c]	0.73	0.98	0.95	–0.87	–0.83	–0.81	0.30	0.93	···	0.97	0.81	0.64
2	Hexenal	0.94	–0.83	···	0.71	0.95	0.93	–0.83	–0.80	–0.77	···	0.89	···	0.93	0.78	0.61
3	1-Hexanol + shoulder	0.80	–0.81	0.41	0.83	0.87	0.74	–0.82	–0.73	–0.63	0.56	0.93	0.59	0.88	0.64	0.76
4	2-Heptanon	0.95	–0.86	···	0.73	0.97	0.94	–0.86	–0.82	–0.78	0.32	0.91	···	0.96	0.81	0.66
5	Unidentified	0.98	–0.91	···	0.74	0.99	0.95	–0.90	–0.85	–0.82	0.37	0.93	···	0.99	0.83	0.70
6	Heptanal	0.94	–0.84	···	0.71	0.94	0.93	–0.83	–0.79	–0.77	···	0.88	···	0.93	0.79	0.62
7	Heptenal	0.97	–0.91	···	0.74	0.99	0.95	–0.91	–0.85	–0.81	0.40	0.93	···	0.99	0.83	0.72
8	1-Okten-3-ol + shoulder	0.93	–0.83	···	0.72	0.94	0.92	–0.82	–0.78	–0.75	···	0.88	···	0.93	0.78	0.62
9	2-Pentyl-furan	0.98	–0.91	···	0.73	0.996	0.96	–0.90	–0.85	–0.82	0.38	0.94	···	0.99	0.84	0.70
10	Oktanal	0.93	–0.83	···	–0.70	0.94	0.93	–0.83	–0.79	–0.76	···	0.87	···	0.93	0.79	0.62
11	Nonanal	0.91	–0.81	···	0.69	0.92	0.91	–0.81	–0.79	–0.75	···	0.85	···	0.91	0.78	0.63

[a] All given correlations $P < 0.05$ ($n = 24$).
[b] Inten. = intensity, Bitt = bitterness, Astrin. = astringency.
[c] ··· = $r < 0.30$.

TABLE VIII
Coefficients of Correlation (r)[a] Between Selected Volatiles and Sensory Attributes[b] for Heat-Treated Samples

		Odors					Flavors									Texture
Peak	Identity	Inten.	Oat	Raw	Hay	Paint	Inten.	Oat	Sweet	Acidity	Raw	Hay	Grass	Paint	Bitt.	Astrin.
1	Hexanal	···[c]	···	···	–0.38	0.60	···	···	–0.35	–0.58	0.49	0.31	0.46	0.66	0.47	0.58
2	Hexenal	0.36	···	0.44	···	0.54	···	···	–0.34	–0.66	0.60	0.31	0.59	0.67	0.38	0.34
3	1-Hexanol+ shoulder	0.46	···	0.46	···	0.37	···	···	···	–0.56	0.50	···	0.61	0.46	···	···
4	2-Heptanon	0.31	···	0.35	···	0.67	0.30	···	–0.37	–0.73	0.56	0.32	0.56	0.69	0.37	0.37
5	Unidentified	···	···	···	–0.37	0.48	···	···	–0.43	–0.66	0.48	0.33	0.48	0.60	0.41	0.47
6	Heptanal	0.52	···	0.65	···	0.70	···	···	···	–0.71	0.78	···	0.76	0.77	0.42	0.41
7	Heptenal	0.43	···	0.51	···	0.64	···	···	···	–0.64	0.64	···	0.68	0.69	···	···
8	1-Okten-3-ol + shoulder	0.44	···	0.59	···	0.50	···	···	0.36	–0.44	0.50	···	0.72	0.48	···	···
9	2-Pentyl-furan	0.30	···	···	–0.30	0.50	···	···	–0.50	–0.62	0.53	0.40	0.40	0.58	0.46	0.53
10	Oktanal	0.40	···	0.45	–0.39	0.68	···	···	···	–0.67	0.66	···	0.71	0.79	0.44	0.49
11	Nonanal	0.58	···	0.68	···	–0.73	0.33	···	···	–0.71	0.82	0.36	0.73	0.80	0.53	0.49

[a] All given correlations $P < 0.05$ ($n = 24$).
[b] Inten. = intensity, Bitt = bitterness, Astrin. = astringency.
[c] ··· = $r < 0.30$.

DISCUSSION

Changes During Storage of Raw Oats

Storage of raw oat flours strongly affected the flavor (Fig. 2), the FFA-levels (Table V), and the content of volatiles (Table VI). Lipolytic enzymes are present in all raw flours and contribute to the major increases in the levels of FFA. After five weeks of storage, the FFA constituted 66% of the total fatty acids, increasing to 85% after 18 weeks. These levels are considerably higher than the recommended maximum level of 5% in products for human consumption (Pomeranz 1992), as well as previously reported results (Ekstrand et al 1993, El Bayâ and Meyer 1978). However, even though polyunsaturated FFA are prone to autoxidation and attack by lipoxygenase, the levels of volatiles and the sensory attributes used for rancidity (grass, hay, and paint) remained quite low up to five weeks of storage. This may be ascribed to the high content of natural antioxidants in oats (Forsell et al 1990). In a previous study of drying processes in cereals, where the formation of pentan was studied, oats were found to be quite stable when compared to wheat, rice, or millet (Percheron and Löliger 1990). However in our study, some changes were found after five weeks of storage, such as increased bitterness and astringency, and reduced oat flavor, sweetness, and acidic flavor. These changes may be related directly to the high FFA levels, as free linoleic acid may possess a weak bitterness (Pokorný 1990), or to hydroxy monoglycerides, which are known as important contributors to bitterness (Biermann and Grosch 1979).

After 18 weeks of storage, oxidation of unsaturated FFA was evident. This is seen by increased levels of volatiles (Table VI), decreased levels of total FA (Table IV), and a decreased ratio of unsaturated to saturated FFA and FA (Tables IV and V). These changes were accompanied by a distinct increase in paint and grass flavors (Fig. 2). However, the major changes in all parameters occurred during the subsequent 24 weeks of storage. After 42 weeks of storage, paint flavor and odor dominated completely and seemed to cover the expected changes in grass and hay flavors.

Quality Changes During Heat Treatment and During Storage of Heat-Treated Flours

The sensory and chemical stability of oat flours during storage was improved greatly by heat treatment. Lipolytic enzymes were inactivated during both heating procedures (results not shown). This reduced the liberation of FFA during storage of heat-treated flours to a minimum, with levels <3% of total FA even after 42 weeks of storage (Table V). Only minor differences in chemical and sensory properties were found between oats heat-treated with hulls and oats heat-treated without hulls. However, a small flavor difference was found by PCA for freshly processed flours (Fig. 3). This is parallel to the results of Molteberg et al (*in press*) where groats heat-treated without hulls had higher scores for oat flavor and fresh flavor than did groats heated with hulls.

Despite inactivation of lipid-degrading enzymes through heat treatment and the relatively high content of antioxidants in oats, some lipid oxidation (nonenzymic) may still occur. Heat treatment may, in fact, promote the nonenzymatic oxidation, due to destruction of antioxidants and increased exposure to oxygen and catalysts (Martin 1958, Galliard 1994). In this study, small but significant chemical changes during storage of heat-treated samples were found. These included small decreases in ratios of unsaturated/saturated FFA (Table V) and some minor increases in the amounts of volatiles (Table VI). However, the chemical changes during storage of heat-treated flours were not reflected in the tested sensory attributes, as no consistent flavor changes in heat-treated flours appeared during the 42 weeks of storage. A similar observation was made by Dahl et al (1989), who found accelerating levels of hexanal in heat-treated rolled oats during 47 weeks of storage, but only small sensory differences between these samples and a reference stored at –40° for 47 weeks. These differences between sensory and chemical results may indicate that the flavor of frozen reference samples are not completely stable during storage, even though the levels of hexanal remained stable during freezing in this experiment (results not shown). Instability of flavors during freezing may contribute to making chemical methods more reproducible and reliable in storage studies than sensory analysis. However, the difference may also indicate that chemical methods detects indicators of future flavor compounds that cannot yet be perceived by humans.

In contrast to the changes during storage, the heat treatment of the grains had major influence on most sensory attributes (Fig. 2), but almost no effect on the levels of the analyzed volatile compounds (Table VI). Increased levels of perceived acidity and oat odor and flavor in the heat-treated oat flours, together with reduced levels of raw flavor and bitterness (Fig. 2), indicate overall more pleasant flavors. Heat treatment of oats was reported previously to change the flavor of oil from green, rancid, sourish, and chemical, to roasted, burnt, or popcorn (Fors and Schlich 1989).

Prediction of Lipid Oxidation by Volatile Compounds

Hexanal is commonly used as indicator of lipid oxidation in cereals (Dahl et al 1989, Ekstrand et al 1993, Piggott et al 1991). Results from this study show that hexanal, but also the other analyzed volatiles, may be useful in predicting changes during storage of raw oat samples. This is seen by the high coefficients of correlation between flavors and volatiles in Table VII. However, lower correlation coefficients for heat-treated samples (Table VIII) and a lack of predictive ability by PLS2 (results not shown) when all samples are included, indicates that raw and heat-treated samples must be considered separately. This is due to a major change in flavor caused by heat treatment that is not reflected in changing levels of volatiles. Only the amount of hexanal and the amount of the compounds comprising peak 3 (mainly 1-hexanol) showed major changes during heat treatment. An increase in the level of hexanal was also found by Molteberg et al (1995). The increasing level of hexanal is unlikely to explain the flavor changes during heating, as this volatile is mostly associated with green flavor (Guth and Grosch 1993, Fors and Schlich 1989). The reduced level of hexanol, which was previously associated with off-flavor in rice (Paule and Powers 1989) and plastic odor in beer (Fors and Nordløv 1987), may contribute to the improved flavor. The odor threshold for 1-hexanol in water is, however, 500 times higher than it is for hexanal (Buttery et al 1988). Most likely, the flavor changes during heat-treatment include compounds other than the carbonyls from lipid oxidation. Changing levels of hydroxy monoglycerides (Biermann and Grosch 1979) or free fatty acids (Pokorný 1990) may be involved in the changes in bitterness, while Maillard reactions and a complex mixture of pyrazines and other nitrogen heterocycles are important in the toasted, nutty oat flavor of heat-treated oats (Heydanek and McGorrin 1986). These nitrogen compounds were not analyzed in our gas chromatography system.

The amounts of volatiles and the sensory properties of the raw samples were generally in good agreement. A PLS2 prediction model with the 11 tabulated volatiles could explain 53.8% of the total variation in the sensory data for raw oats (results not shown). All individual volatiles were also strongly related to the odors and flavors (Table VII). The highest correlation coefficients, which were found between paint odor and flavor and the volatiles 2-pentyl-furan, heptanal, hexanal, and one unidentified compound (peak 5), were related to a major increase both in paint flavor and volatiles between 18 and 42 weeks of storage. However, the levels of most of the aldehydes (hexanal, heptanal, nonanal, hexenal, and heptenal) are not likely to be responsible for paint and grass flavors in raw samples. An evidence for this is the comparable levels of these volatiles in two groups of samples with very different flavors (Table VI): the paint-flavored raw samples stored for 18 weeks and the stable heat-treated samples stored for 42 weeks.

The rancid terms are more likely related to the much higher levels in raw oats of 2-pentyl-furan (peak 9), 1-hexanol (the main constituent of peak 3), or the unidentified peak 5. The somewhat higher levels of 2-heptanon (peak 4), octanal (peak 10), and the peak with mainly 1-octen-3-ol (peak 8) could also be involved. Of these compounds, 1-octen-3-ol and octanal have very low (<1 ppb) odor thresholds (Buttery et al 1988). Gas chromatography sniff tests indicate a prominent off-odor for 1-octen-3-ol (peak 8), having a mushroom or moldy odor (Börjesson et al 1993). It is also a key component in beany odor (Sugawara et al 1985). Octanal (peak 10) has been reported to possess an odor of soap or citrus (Guth and Grosch 1993). The flavor of 2-pentyl-furan itself is described as licorice-like (flavor threshold in oil 1 ppm), but in oil the compound imparts a strong beany or grassy off-odor reminiscent of those of a reverted soybean oil (Chang et al 1966). The 2-heptanon has an odor threshold of 140 ppb in water (Buttery et al 1988) and is described as having a flavor of soap (Schieberle and Grosch 1984).

Both lipoxygenase activities, photooxidation and autoxidation, may have contributed to the intense paint flavor in stored, raw flours. However, the mechanisms for oxidation are difficult to predict from the pattern of volatiles. Several of the detected volatiles with high levels may very well be products from the action of lipoxygenase, i.e., 2-pentyl-furan, 1-octen-3-ol, 2-heptenal, 1-hexanol, hexanal, and 2-heptanon (Heimann et al 1975, Heydanek and McGorrin 1981, Luning et al 1991, Mtebe and Gordon 1987). However, oat lipoxygenase and linoleic acid produce mainly hexanal and 2-nonenal (Heimann et al 1975), and compared to other cereals, the activity of lipoxygenase is very low in oats (Fretzdorff and Seiler 1987). The low moisture levels probably restricted the activity further (Mtebe and Gordon 1987). Even though the activity of lipoxygenase is expected to be low before chemical analyses, enzymatic activity may still contribute to flavor in the raw samples. Wet sample preparation, as used for sensory analysis, was previously related to bitterness formed from action of lipoxygenase (Biermann and Grosch 1979). In this case, however, nonenzymic mechanisms are the most likely cause of both paint flavor and high levels of volatiles. While Galliard and Gallagher (1988) found that the level of off-flavors was strongly related to both lipoxygenase activity and FFA-level in wheat, our study found the levels of flavors and volatiles to be very different at five and 42 weeks, whereas the levels of FFA were fairly constant. The levels of oleic acid in raw samples were also significantly reduced upon storage in this experiment, even though lipoxygenase only attacks polyunsaturated fatty acids. Photooxidation may be involved, as the samples were stored under light in paper bags. However, the observed induction period for the raw oat samples may indicate the presence of effective antioxidants, and that autoxidation was the predominant cause of lipid oxidation.

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