Differences Between Sensory Profiles and Development of Rancidity During Long-Term Storage of Native and Processed Oat

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ABSTRACT

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Changes in the sensory attributes, lipid composition, and amounts of volatile and phenolic compounds of native and processed (germinated and dried) crushed oat were followed during a 12-month storage period. The influence of the chemical attributes on the sensory profiles of oats was analyzed by statistical multivariate techniques (PLS regression). During the storage period, significant changes in the sensory profiles of the native and processed oat groats were observed. The stability of oat groats was significantly increased through germination and subsequent drying because the chemical changes causing rancidity and bitterness developed more slowly in the processed oat when compared with the native oat. In native oat, the most intensive changes due to deterioration had already occurred after one month of storage, whereas in processed oat, these changes were perceived considerably later. Stored oat that had deteriorated was evaluated as being musty and earthy in odor and bitter and rancid in flavor. The accumulation of free fatty acids and volatile compounds related to lipid oxidation were closely correlated with the development of the undesired sensory attributes described above. The total amount of phenolic compounds, as well as the volatile aromatic and branched chain compounds derived mainly from protein degradation, showed a significant relationship with favorable sensory attributes such as roasted odor and flavor. Lipid oxidation occurred during the storage and was observed both in the polar and in the nonpolar lipid classes of native oat, whereas in the processed oat, these changes were nonsignificant. Photo-oxidation of acylated fatty acids may significantly contribute to the development of volatile lipid oxidation products during storage.

Oat (*Avena sativa* L.) is perceived as palatable, it has a favorable lipid composition, and it has valuable technical properties, such as the capability to form highly viscous solutions (Ma 1983; Chang and Sosulski 1985). Oat has also gained a lot of attention due to its health-promoting properties. These factors have made oat appealing for new food applications and have developed a need for techniques that could expand the use of oat beyond conventional products. However, in several applications, the use of oat is limited because of its tendency to become rancid and form a bitter off-flavor during processing and storage.

One of the main advantages of oat is its tasty flavor, which, as well as its texture, can be tailored by processing. One possibility to enhance oat flavor is germination and subsequent drying, where the flavor can be modified by using different temperature and humidity parameters during the heat-treatment process (Molteberg et al 1996a; Liu et al 2000; Parker et al 2000; Heiniö et al 2001; Sides et al 2001). The most favorable results with regard to flavor are based mainly on a high processing temperature and a dry atmosphere. The use of oat malt is currently still very minor, although the chemical changes during the drying of germinated grains have enhanced the intensity of the favorable sensory attributes of oat. As a result, a product with a roasted, sweet, intense flavor, a crisp texture, and increased availability of some important micronutrients can be obtained by germination of oat (Heiniö et al 2001). However, in developing novel products from germinated oat, the stability of sensory properties during storage is of utmost importance and only a limited amount of information is currently available concerning this (Larsson and Sandberg 1995).

Flavor deterioration of cereals during storage is influenced both by reduction of the desired flavor attributes, due to physical processes such as volatilization, and by development of undesired off-flavors. Interactions of volatile compounds with other food constituents are important for the flavor characteristics, intensity, and stability. Hexanal, one of the most abundant volatile compounds in oat, is continuously formed as a secondary oxidation product of lipid oxidation. However, hexanal accumulates only partly in food products because it may evaporate or be quenched into nonvolatile compounds (Zhou and Decker 1999). In the presence of a proper enzyme system, the conversion of hexanal and other volatile aldehydes to relevant carboxyl acids or alcohols has also been observed (Maheswari et al 1997; Iersel et al 2000).

The most important off-flavor of oat is rancidity, which is generally caused either by volatile compounds such as aldehydes, ketones, and alcohols, or by high amounts of free fatty acids or phenolic compounds (Molteberg et al 1996a,b; Sjövall et al 1997; Peterson 1998; Zhou et al 1999). However, the mechanism of development of rancidity is dependent on the processing and storage conditions, and somewhat different volatile compounds cause the rancid perception at low temperatures and high moisture levels as compared with high processing temperatures and low humidity. In dried, rancid oat groats, hexanal, pentanal, 3-methyl-1-butanol, and pentyl furan comprise as much as 85–90% of the total area of the volatile compounds (Heydanek and McGorrin 1986; Molteberg et al 1996a). Volatile aldol condensation products such as 2-butyl-2-octenal and 2-propyl-2-octenal, and lipid oxidation products such as (E)- and (Z)-3-octene-2-one, (E, E)- and (E, Z)- 3, 5-octadien-2-one may also be responsible for the rancid flavor (Heydanek and McGorrin 1986). The bitter flavor of stored ground oat grains may be caused by a specific class of lipids, hydroxyoctadecadienoic monoglyserides (Biermann and Grosch 1979). Bitterness, in addition to rancidity, also correlates closely with phenolic compounds such as avenanthramides and oat cultivars processed with hulls (Molteberg et al 1996b). Unprocessed oat stored for one year had increased levels of phenolic acids and aldehydes, and the increase for phenolic acids was most pronounced after storage at high relative humidity (80% rh) (Dimberg et al 1996).

The development of rancidity is generally considered to be a consequence of the deteriorative reactions of lipids, although deterioration of proteins and reactions of phenolic acids should not be excluded. During storage, two distinct reactions of oat lipids take place: 1) the hydrolytic deterioration where triacylglycerols or phospholipids are converted to free fatty acids, and 2) the oxidative deterioration where polyunsaturated fatty acids are converted to hydroperoxides and further to secondary oxidation products. Oat is exceptionally high in lipase catalyzing the hydrolytic deterioration, whereas the lipoxygenase activity, catalyzing the oxidative deterioration is low (Ceumern and Hartfield 1984).

Linoleate hydroperoxides formed during the oxidation of linoleic acid, the most substantial oxidizable fatty acid, decompose readily to yield a wide spectrum of products that cause a rancid flavor. Depending on which of the hydroperoxide isomers are formed, the

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secondary oxidation products contain different short chain alkanes, aldehydes or alcohols, and furans (Grosch 1976). The most abundant volatile compounds found in moist (vacuum-steam distilled), rancid oat groats are hexanal, pentanal, 1-pentanol, and 3,5-octadien-2-one (Heydanek and McGorrin 1986). During the development of rancidity in food products that are susceptible to oxidative deterioration, a two- to 200-fold increase has been reported in the headspace concentrations of these secondary oxidation products (Fritsch and Gale 1977; Shin et al 1986; Lai et al 1995; Sjövall et al 2000). While the sensory effects of such changes are extensive and easily detectable by chemical analysis, the relevant changes in the lipid composition are very small, and a specific loss of polyunsaturated fatty acids can be expected only during excessive deterioration.

Currently, in conventional oat products, a satisfactory lipid stability is achieved by inactivating enzymatic activity, especially lipase, by heat treatment (Ekstrand et al 1993). The effect of such a heat treatment on the oxidative stability of processed oat products is, however, adverse, as many antioxidants that help to maintain oxidative stability during processing and storage are denatured by excessive heating. Oat is a source of many heat-labile compounds that exhibit antioxidant activity: vitamin E, phytic acid, sterols, and phenolic compounds such as avenanthramides and flavonoids (Dimberg et al 1996; Peterson 2001).

The present study is an extension to our earlier studies on oat, in which we studied the influence of the germination process on the chemical and microbiological quality of oats (Wilhelmson et al 2001) and specified factors affecting the sensory profile of germinated oat (Heiniö et al 2001). In this study, the sensory data collected during long-term storage of native and germinated oat are combined with the chemical data on lipid deterioration and appearance of certain volatile compounds during storage.

MATERIALS AND METHODS

Oat Samples

The hulled oat cultivar, Veli, harvested in summer 1997 in Finland was used in the present study in its native and processed (germinated and dried) forms. The germination conditions have been described elsewhere (Wilhelmson et al 2000). The germinated oat was dried using hot air to raise the temperature gradually from 65 to 93°C over 19 hr. Both the native and the processed oat grains were dehulled, coarse-ground (Wiley mill with a 2-mm sieve), and stored in the dark in closed, brown paper bags at 20°C for 12 months. Approximately 41% of crushed oat grains had a particle size of >2.50 mm, 23% had 1.25–2.50 mm, and 36% had <1.25 mm. Samples were exposed to changes in environment humidity, which caused some seasonal variation in moisture content during the storage period. The samples were analyzed for their sensory profile, lipid composition, certain volatile headspace compounds and phenolic compounds at the beginning of storage and after six, nine, and 12 months of storage. In addition, the volatile headspace compounds were followed during the first three weeks of storage; the sensory analysis was performed after one and three months; and the lipid composition was analyzed after one and two months of storage.

Sensory Analysis

The sensory quality of the oat groats was evaluated by a trained internal panel with proven skills using descriptive analysis (Stone et al 1974; Stone and Sidel 1993, 1998). All sensory work was conducted at the sensory laboratory of VTT Biotechnology, which fulfills requirements according to international ISO standards. All assessors of the internal sensory panel have passed the basic taste test (according to ISO), the odor test, and the color vision test. They have been trained in the sensory methods in numerous sessions over several years, and their evaluation ability is routinely checked by using individual control cards for each assessor. The 12-member multiproduct panel became particularly familiar with the sensory descriptors and the attribute intensities of native and processed oat in a few presessions before the evaluations with real samples, and by using verbal definitions describing the ends of intensity scales of the attributes. The same panel had been used frequently in our previous studies on oat and other cereals.

The vocabulary of the sensory attributes was developed by describing differences among 17 widely varying, extreme oat products by a six-member expert panel specialized in cereal products in a round-table session. Attribute intensities were rated on continuous

TABLE I
Effect of Storage Time on Sensory Profiles of Native and Processed Oat[a,b]

Sensory Attributes[c]	Months of Storage											
	0		1		3		6		9		12	
	N	P	N	P	N	P	N	P	N	P	N	P
O-cereal	4.4	5.9d	4.1	5.2cd	5.4	5.5c	5.5	5.0c	5.2	4.6c	4.7	4.4c
O-roasted	**0.8**	**5.7**	**1.2**	**4.8**	1.4	2.6	0.9	2.8	1.1	2.2	1.9	2.7
O-moist	**0.8**	**1.8**	0.6	1.1	1.5	1.4	0.4	1.3	1.3	1.7	1.1	1.3
O-musty	1.5	1.0c	1.5	1.8cd	2.0	2.0cd	2.3	3.9de	4.1	4.9e	4.0	4.6e
O-earthy	1.0ab	1.1	0.3a	0.7	0.7ab	1.2	0.6ab	1.2	2.3b	2.2	1.9ab	2.0
O-intense	**2.2**a	**5.9**d	**2.3**a	**4.0**cd	2.7ab	3.6c	**2.6**ab	**4.6**cd	4.3b	4.9cd	4.4b	5.4cd
F-cereal	5.3	6.1	6.1	6.3	5.8	5.8	6.2	4.9	6.5	6.3	5.4	5.5
F-roasted	**1.3**	**7.0**	**2.2**	**6.9**	**0.7**	**5.4**	**0.7**	**5.3**	**1.3**	**4.4**	**1.8**	**4.1**
F-nutty	**1.9**	**5.6**	**2.9**	**5.6**	**2.3**	**4.4**	3.7	5.4	4.2	4.5	2.6	3.6
F-sweet	**2.5**	**6.1**d	**1.6**	**5.1**cd	**1.5**	**5.1**cd	**2.0**	**4.8**cd	**1.6**	**4.0**cd	**2.0**	**3.4**c
F-bitter	2.6a	4.0cd	**5.0**ab	**2.8**c	5.5b	3.5cd	6.4b	5.8cd	6.7b	5.9cd	6.0b	6.3d
F-germlike	2.4	3.0	2.5	2.7	2.2	1.7	0.7	1.7	2.1	2.1	1.0	1.6
F-musty	2.3	2.2cd	3.1	2.2cd	3.0	1.9c	4.0	3.4cd	4.1	3.3cd	4.8	4.5d
F-rancid	0.5	1.1	0.8	0.8	1.9	1.2	2.8	2.2	2.7	1.8	2.8	3.1
F-intense	**2.9**a	**7.2**	**4.1**ab	**6.3**	**4.8**ab	**6.2**	4.8ab	6.0	5.9b	6.3	6.2b	6.7
Aftertaste	**3.0**a	**5.5**	4.8ab	5.0	4.3ab	4.0	5.7ab	5.9	5.3ab	5.7	6.2b	6.4
T-hard	4.6b	4.6	3.1ab	4.4	**2.2**a	**4.1**	3.7ab	5.2	3.3ab	4.3	4.1ab	4.6
T-tough	**5.8**b	**2.9**	3.9ab	2.9	**3.4**ab	**1.8**	**4.9**ab	**2.8**	3.3a	2.0	3.7ab	2.6
T-moist	3.3	1.8	2.0	0.9	1.9	1.0	1.5	1.2	1.2	0.9	1.3	1.2
T-crisp	**2.0**	**5.4**	**2.0**	**4.7**	**2.0**	**4.0**	**2.2**	**5.9**	**1.5**	**5.3**	**3.5**	**5.1**
T-brittle	**2.5**a	**5.4**	3.1a	3.1	2.8a	3.6	2.0a	4.0	3.3a	4.1	4.3b	4.9

[a] Mean descriptive analysis ratings of oat groats (0–10 scale), n = 10–20. N = native, P = processed.
[b] Values in bold indicate significant difference between native and processed oat for storage time vs. sensory attribute. Values followed by the same letter in the same row are not significantly different ($P < 0.05$).
[c] O = odor, F = flavor, and T = texture.

unstructured, graphic intensity scales. The scales were verbally anchored at each end: the left side of the scale corresponded to the lowest intensity (value 0) and the right side to the highest intensity (value 10) of the attribute. The crushed grain samples, ≈10 g of each, were presented to the assessors as processed or unprocessed oat groats as such from three-digit coded plastic containers covered by lids and in random order, and the closed containers were balanced 1–2 hr before the assessments. Each assessor was provided with two samples to be evaluated at each session. Water was provided for cleansing the palate between the samples. The panel was instructed to nose each sample before tasting them, and the samples were requested to be swallowed. Scores were recorded and collected using computerized data systems (Panel 5 v.4.00, Legolas Oy, Finland, 1995 or Compusense Five v.4.0, Guelph, ON, Canada, 2000).

Chemical Analysis

Lipids. For lipid extraction, the crushed oat samples were extracted twice in 19 volumes of dichloromethane-methanol (2:1) as described by Liukkonen et al (1992). After 2 hr of continuous shaking (250 rpm, 28°C), the mixtures were centrifuged (1,460 × *g*, 10 min), and the pellet was reextracted for another 2 hr. The extracts were combined and evaporated to dryness under N_2. These samples were used for lipid class separation and for fatty acid composition analysis. The lipid class separation of major lipid classes used thin-layer chromatography as described by Liukkonen et al (1992). The samples were supplemented with a standard mixture containing known amounts of dipentadecanoyl phospatidylcholine, heptadecanoic acid, dipentadecanoin, and triheptadecanoin. The mixture was applied to silica plates and the plates were developed. The identified spots were scraped off and used for fatty acid analysis. Fatty acid composition of extracts or separated lipid classes was analyzed by converting fatty acids to methyl esters and analyzing the latter by gas chromatography essentially as described by Suutari et al (1990).

Volatile compounds. The volatile compounds released into headspace of oat samples were studied by static headspace measurement. Only the data for compounds of which the headspace concentration changed during the storage is shown, as the detailed chromatogram of compounds detected in the headspace of the similar, freshly milled samples has been published previously (Heiniö et al 2001).

A 0.5-g crushed oat grain sample was weighed into a crimp-top vial and the sealed vial was equilibrated in the headspace injector oven (HP 7694) for 25 min at 100°C. The vial was pressurized with helium (15.8 psi) for 0.13 min after which the 1-mL loop was filled for 0.02 min. The headspace sample was introduced into a nonpolar capillary column (HP 5-MS) that was temperature-programed from 40 to 200°C. An EI-mass selective detector (HP 5971A) was used to identify and quantify the compounds. Compounds were identified based on their retention indices and mass spectrums (Heydanek and McGorrin 1986; SDBSweb http://www.aist.go.jp/RIODB/SDBS/). To compensate for the variation in the performances of the headspace injector and the MS-detector during the storage period, all detector responses of triplicate samples were proportioned to the response of an external standard (40 ppm of isobutanol in 0.4 mL of water). All the data are normalized so that for each compound, the maximum response during the whole 12-month storage period is set to unity.

Phenolic compounds. The total concentration of phenolic compounds was determined as gallic acid equivalents (GAE) using the

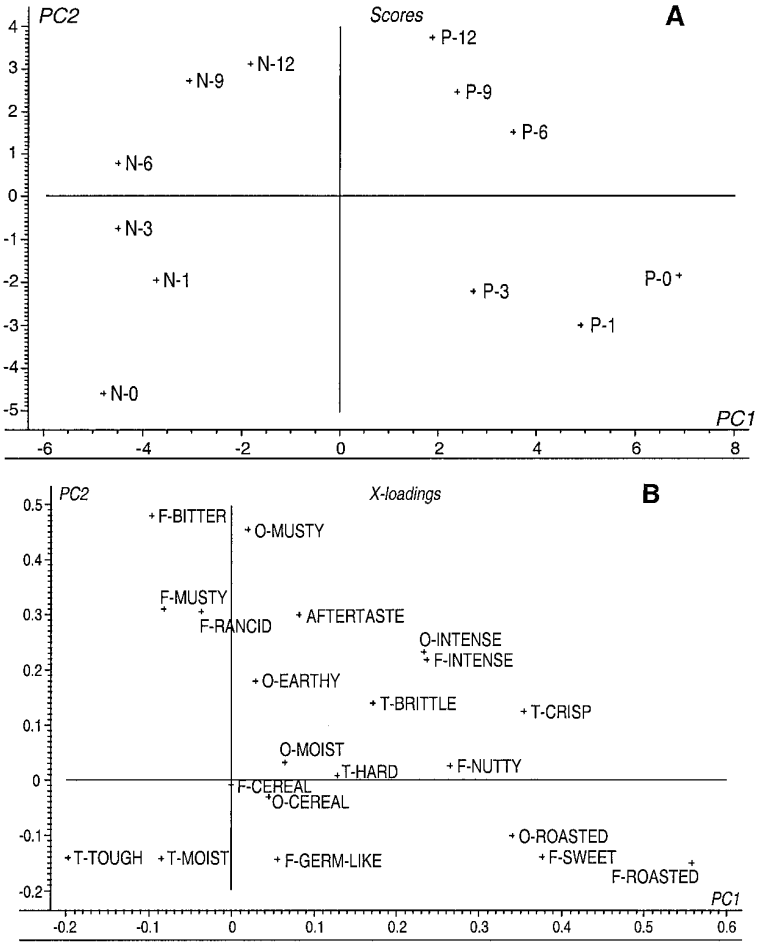

Fig. 1. Principal component analysis (PCA) scores (**A**) and loadings (**B**) of sensory data for the native (N) and processed (P) oat groats during 0, 1, 3, 6, 9, and 12 months of storage. Abbreviations of sensory attributes are defined in Table I.

Folin-Ciocalteu method (Singleton and Rossi 1965) with some minor modifications. Methanol was used as a solvent, and the total volume of the reaction was 1 mL. The hull was removed mechanically from the Veli cultivar before preparation of the extract.

Statistical Analysis

From each sensory session, raw data obtained were averaged across panelists. The significance of each descriptive attribute in discriminating between the storage periods and the samples was investigated using analysis of variance (ANOVA) and Tukey's honestly significant difference (HSD) test (significance of differences at $P < 0.05$) by the general linear model (GLM) procedure using statistical software (v. 8.0.2, SPSS). When the difference in ANOVA among the different storage times for each sample separately was statistically significant, pairwise comparisons of these attributes were analyzed by Tukey's test. The significance of the difference between native and processed oat groats at each storage time separately was executed by paired *t*-test ($P < 0.05$). The correlation coefficients between the scores of sensory odor and flavor attributes, and relative amounts of selected volatile compounds, lipid composition, and total amount of phenolic compounds were determined ($P < 0.05$). The amounts of lipids and volatile compounds of native oat and the corresponding processed oat were compared by paired *t*-test ($P < 0.05$).

The multivariate methods, principal component analysis (PCA) and partial least squares (PLS) regression were performed using a software package (Unscrambler v. 7.5, CAMO ASA). PCA was used to describe the variance among the whole sensory data obtained, and PLS regression was used to compare the sensory odor and flavor data with the selected volatile compounds. The sensory data used for the multivariate analysis were means calculated over all panelists.

RESULTS

Changes in sensory profiles. Significant changes in the sensory profiles of the native and processed oat groats occurred during the 12-month storage period (Table I, Fig. 1). The roasted odor and flavor, and nutty and sweet flavor were originally evaluated to be significantly more intense in the processed than in the native oat, which confirmed our earlier results (Heiniö et al 2001). In addition, processed oat groats had originally higher odor, flavor, and aftertaste intensities, moister odor, less tough, crispier, and more brittle texture as compared with native oat ($P < 0.05$).

When both oat samples at each storage time were considered, they were statistically different from each other with regard to some sensory attributes: roasted odor (F = 36.6), odor intensity (F = 34.1), sweet (F = 79.7), roasted (F = 131.7), and nutty (F = 23.7) flavor, and flavor intensity (F = 29.9) ($P < 0.001$), and all the texture attributes evaluated ($P < 0.05$). The most obvious changes in the stored, native oat were perceived as more bitter flavor, more intense odor and flavor and more brittle texture, whereas the processed oat became slowly less cereal, more musty and less sweet, and very slowly more bitter during the storage. The bitter flavor was perceived in the native oat groats already after one month of storage, whereas in the processed oat, the bitterness was observed considerably later. However, roasted and sweet flavor and crisp texture remained in the processed oat over the whole storage period. In general, the processed oat groats had better stability than the native oat.

It can be summarized from the PCA score and loading plots of the sensory data for the native, and the processed oat groats stored for 12 months, that the first two principal components together explained 83% of the variation among the samples (Fig. 1). The first factor (PC1) explained 58% of the variation and separated the two sample types (native and processed) based on existence of the heat treatment. The second factor (PC2) explained 25% of the variation and separated the samples on the basis of storage time. The most relevant sensory descriptors of the germinated and dried oat groats stored for less than six months were roasted odor and flavor, and sweet flavor. The corresponding native oat groats were described as tough and moist in texture. However, at the end of the storage period, the processed oat was perceived as being musty in odor, whereas the native oats were perceived as being bitter, rancid, and musty in flavor.

Changes in lipid composition. The most intensive lipid hydrolysis occurred during the first six months of storage, and during that time, less than half of the original triacylglycerols were hydrolyzed into free fatty acids (Table II). However, the amount of polar lipids remained unchanged during the whole storage period, indicating that the hydrolysis of membrane lipids was negligible. In our previous study, we reported that the lipolytic activity of oat, measured as a tendency to form free fatty acids upon soaking in water, was reduced during the germination and heat treatment of oat (Heiniö et al 2001). In accordance with this lower lipolytic activity, the accumulation of free fatty acids was slower in processed oat during the two first months of storage when compared with native the oat. However, we found that after two months of storage, the

TABLE II
Distribution of Fatty Acids in Different Lipid Classes During Oat Storage[a,b]

	Polar Lipids		Triglycerides		Free Fatty Acids	
Storage Time	**Native**	**Processed**	**Native**	**Processed**	**Native**	**Processed**
Unstored	9.2	9.2	67.8	60.4*	3.6	3.2
1 month	10.7	9.1	56.2	56.6	12.8	8.5*
2 months	9.9	9.6	52.5	48.5*	14.7	11.9*
6 months	10.3	10.0	44.8	33.5*	28.2	31.1*
9 months	10.2	9.8	41.2	32.4*	28.9	33.2*
12 months	10.1	9.9	36.8	29.7*	26.3	30.0*

[a] Determined as mg of fatty acids/mg of crushed oat dry weight.
[b] * = statistically significant difference ($P < 0.05$) between native and processed oat.

TABLE III
Changes in Degree of Unsaturation of Different Lipid Classes During Oat Storage[a,b]

	Polar Lipids		Triglycerides		Free Fatty Acids	
Storage Time	**Native**	**Processed**	**Native**	**Processed**	**Native**	**Processed**
Unstored	1.25‡	1.28	1.28‡	1.27	1.25†‡	1.19†
12 months	1.19†‡	1.25†	1.24‡	1.25	1.21‡	1.22

[a] † = Statistically significant difference ($P < 0.05$) within lipid class.
[b] ‡ = Statistically significant difference ($P < 0.05$) between storage times.

situation was reversed, and the accumulation of free fatty acids was actually greater in the processed oat than in the native oat. Despite the large amount of triacylglycerides hydrolyzed into free fatty acids, the fatty acid composition of triglycerides remained nearly unchanged throughout the whole storage period (data not shown), indicating that lipase did not discriminate between the acyl structures of fatty acids. However, at the end of the 12-month storage period, the degree of unsaturation (DUS) was reduced both in polar and nonpolar lipid classes in the native oat samples (Table III). In the processed oat, no changes in the DUS values of different lipid classes were noticed during storage.

Changes in volatile compounds. Changes in the volatile compounds released into headspace from native, and processed oat were measured periodically during 12 months of storage. As a general trend, the relative proportion of low-boiling substances diminished as a consequence of the increase of the volatile lipid oxidation products such as hexanal, pentanal, and 2-pentylfuran. The changes in amounts of selected volatile compounds are presented in the Tables IV–VIII. These compounds included dimethyl sulfide (DMS), other possible protein degradation products such as methylpropanal, methylbutanal, 3-pentanone, benzaldehyde, and phenylacetaldehyde, volatile secondary lipid oxidation products such as pentanal, hexanal, heptanal, and 2-heptanone, furans such as 2-ethylfuran, *n*-butylfuran, and pentylfuran, and volatile alcohols such as 1-pentanol and 1-hexanol.

In freshly milled, crushed oat, the amount of DMS, a known flavor perceived as roasted and produced by proteolytic activity during malting (Anness and Bamforth 1982; Heiniö et al 2001), was ≈100-fold in the headspace of processed oat when compared with native oat (Table IV). However, during storage, DMS decreased quickly in processed oat, and after four days of storage was about one-third of the initial amount. The remaining DMS continued to decrease during the course of the 12-month storage period, so that the final level in processed oat was still ≈10-fold compared with the native oat. Despite reduction in DMS levels, no other volatile sulfur-containing compounds were formed during the storage period, indicating that the decrease of DMS in germinated oat was due to evaporation or quenching into nonvolatile compounds.

TABLE IV
Changes in Dimethyl Sulfide (DMS) Concentrations During Oat Storage[a]

	DMS	
Storage Time	**Native**	**Processed**
Unstored	0.009	1.000*[b]
4 days	0.007	0.328*
7 days	0.008	0.301*
11 days	0.007	0.201*
21 days	0.006	0.084*
6 months	0.008	0.127*
9 months	0.007	0.105*
12 months	0.006	0.068*

[a] Normalized detector responses.
[b] * = statistically significant difference ($P < 0.05$) between native and processed oat.

TABLE VII
Changes in Concentrations of Volatile Alcohols During Oat Storage[a]

Storage	1-Pentanol		1-Hexanol	
Time	**Native**	**Processed**	**Native**	**Processed**
Unstored	0.095	0.055	0.100	nd[b]
4 days	0.087	0.067	0.115	0.050*[c]
7 days	0.156	0.062*	0.211	0.094*
11 days	0.193	0.144	0.165	0.099*
21 days	0.226	0.163*	0.157	0.103*
6 months	0.529	0.476	0.286	0.300
9 months	1.000	0.702*	1.000	0.575*
12 months	0.841	0.757	0.601	0.512*

[a] Normalized detector responses.
[b] Not detected.
[c] * = statistically significant difference ($P < 0.05$) between native and processed oat.

TABLE V
Changes in Concentrations of Volatile Protein Degradation Products During Oat Storage[a]

Storage	Methyl Propanal		2-Methyl Butanal		3-Pentanone		Benzaldehyde		Phenylacetaldehyde	
Time	**Native**	**Processed**	**Native**	**Processed**	**Native**	**Processed**	**Native**	**Processed**	**Native**	**Processed**
Unstored	0.073	0.538*[b]	0.087	0.359*	0.077	0.474*	0.190	0.101	0.037	0.309*
4 days	0.087	0.808*	0.033	0.641*	0.038	0.728*	0.074	0.455*	nd[c]	0.589
7 days	0.116	0.907*	0.047	0.750*	0.055	0.871*	0.096	0.456*	0.023	0.662*
11 days	0.124	0.841*	0.048	0.692*	0.063	0.778*	0.335	0.659*	0.076	0.734*
21 days	0.152	0.786*	0.056	0.659*	0.075	0.740*	0.423	1.000*	0.093	0.947*
6 months	0.022	0.780*	nd	0.671	nd	0.825	0.085	0.574*	nd	0.741
9 months	0.095	0.957*	0.050	0.895*	0.065	0.982*	0.255	0.728*	nd	0.945
12 months	0.108	1.000*	0.064	1.000*	0.068	1.000*	0.188	0.922*	nd	1.000

[a] Normalized detector responses.
[b] * = statistically significant difference ($P < 0.05$) between native and processed oat.
[c] Not detected.

TABLE VI
Changes in Concentrations of Volatile Aldehydes and Ketones During Oat Storage[a]

Storage	Pentanal		Hexanal		Heptanal		2-Heptanone	
Time	**Native**	**Processed**	**Native**	**Processed**	**Native**	**Processed**	**Native**	**Processed**
Unstored	nd[b]	nd	0.086	0.111	0.195	nd	nd	nd
4 days	nd	nd	0.108	0.088	0.185	nd	nd	nd
7 days	0.137	nd	0.235	0.120*[c]	0.400	0.068*	0.233	0.186
11 days	0.192	0.209	0.405	0.212*	0.448	0.258	0.448	0.237
21 days	0.235	0.184	0.492	0.292*	0.639	0.422*	0.162	0.221
6 months	0.564	0.522	0.734	0.643	1.000	0.981	0.638	0.515
9 months	0.841	0.707*	0.945	0.771*	0.930	0.840	0.989	0.785
12 months	1.000	0.909	1.000	0.941	0.841	0.795	1.000	0.864*

[a] Normalized detector responses.
[b] Not detected.
[c] * = statistically significant difference ($P < 0.05$) between native and processed oat.

Other compounds with a possible link to proteolysis were also released more efficiently from the processed oat than from the native oat. These compounds included 3-pentanone, benzaldehyde, phenylacetaldehyde, and branched chain aldehydes methyl propanal and 3-methyl butanal. During the 12-month storage period, the amount of these compounds in the headspace of the processed oat increased two- to ninefold, with most of the increment occurring during the first week of storage concurrently with the balancing of moisture content after the heat treatment. However, in the native oat, these compounds were either not present or they were unaffected by the storage time (Table V).

Aldehydes with five to seven carbon atoms together with a 2-heptanone are possible secondary oxidation products of unsaturated fatty acids (Grosch 1976; Keszler et al 2000) and formed another group of compounds that shared a similar behavior during storage (Table VI). The amount of these compounds in the headspace increased 10-fold during storage both in the processed oat and in the native oat. The same lipid oxidation products were formed regardless of whether the oat was processed or not. The increase in secondary oxidation products was larger in the native than in the processed oat, which can be linked to increased radical scavenging activity measured in the germinated oat after heat treatment (*unpublished results*). The reduced forms of pentanal and hexanal were also detected in similar amounts both in the processed oat and native oat (Table VII). The amount of these alcohols increased during the storage period and reached a maximum at nine months of storage. Such behavior may indicate a change in enzymatic activities or a shift in the reduction balance after nine months of storage. Another group of secondary fatty acid oxidation products includes three different furans (Table VIII). Consistent with the aldehydes presented in Table VI, the amount of furans increased during storage, the increase being larger in native oat. The final amount of furans after the 12-month storage period were 15- to 20-fold larger compared with the amounts released at the beginning of the storage period, except for the ethyl furan, where the level was unsteady during the first week of storage.

Changes in total amount of phenolic compounds. The total amount of phenolic compounds in unstored, processed oat was 465 mg of gallic acid equivalents (GAE)/kg. The total amount of phenolic compounds in unstored, native oat was only 63% of that in unstored, processed oat. In both the native and the processed oat, the amount of phenolic compounds decreased to 85% of the original level after six months of storage and stayed at that level for the rest of the 12-month storage period.

Correlation between sensory and chemical data. The individual correlation coefficients between the 16 sensory odor or flavor attributes and the lipids, 15 selected volatile compounds, and phenolic compounds are presented in Table IX. Some of the lipid-related parameters (free fatty acids and DUS of polar lipids), and volatile compounds (pentanal, 2-ethylfuran, 1-pentanol, hexanal, 1-hexanol,

TABLE VIII
Changes in Concentrations of Furan During Oat Storage[a,b]

Storage Time	2-Ethyl Furan		*n*-Butyl Furan		Pentyl Furan	
	Native	Processed	Native	Processed	Native	Processed
Unstored	0.301	0.058*	0.035	nd[c]	0.041	0.022
4 days	0.187	0.104	0.018	nd	0.066	0.031*
7 days	0.235	0.120*	0.050	0.007*	0.122	0.048*
11 days	0.168	0.189	0.075	0.041*	0.223	0.079*
21 days	0.366	0.214*	0.095	0.068*	0.420	0.154*
6 months	0.476	0.469	0.293	0.561	0.669	0.639
9 months	0.773	0.607*	0.844	0.664*	0.800	0.608*
12 months	1.000	0.819	1.000	0.708*	1.000	0.733*

[a] Normalized detector responses.
[b] * = statistically significant difference ($P < 0.05$) between native and processed oat.
[c] Not detected.

TABLE IX
Coefficients of Correlation (*r*) Between Sensory Attribute Scores[a,b] and Relative Amounts of Selected Volatile Compounds, Lipids, and Phenolic Compounds

	O-Cereal	O-Roast	O-Moist	O-Musty	O-Earthy	O-Intense	F-Cereal	F-Roast
Free fatty acids (mg/g)	–0.13	–0.39	0.10	**0.90**	0.60	0.26	–0.08	–0.26
Polar lipids DUS[c]	0.17	0.68	0.45	–0.34	–0.25	0.33	0.06	**0.79**
Triglycerides DUS	0.37	0.45	0.12	–0.53	–0.38	–0.11	0.34	0.48
Diglycerides DUS	0.13	0.33	0.59	0.57	0.58	0.69	0.49	0.34
Free fatty acids DUS	–0.39	–0.60	**–0.70**	–0.10	–0.46	**–0.79**	0.06	–0.41
Dimethyl sulphide	0.67	**0.92**	0.59	–0.56	–0.25	0.58	0.18	**0.76**
Methylpropanal	–0.26	0.50	0.66	0.48	0.35	**0.70**	–0.16	**0.75**
Methylbutanal	–0.37	0.39	0.58	0.54	0.38	0.62	–0.19	0.65
3-Pentanone	–0.29	0.46	0.63	0.50	0.34	0.65	–0.19	**0.73**
Benzaldehyde	–0.59	0.07	0.39	**0.74**	0.53	0.45	–0.22	0.37
Phenyl acetate	–0.36	0.36	0.55	0.55	0.34	0.59	–0.19	0.64
Pentanal	–0.31	–0.30	0.01	**0.87**	**0.70**	0.27	0.11	–0.26
2-Ethylfuran	–0.52	–0.47	–0.12	**0.81**	**0.70**	0.08	–0.04	–0.44
1-Pentanol	–0.28	–0.42	0.01	**0.86**	**0.76**	0.17	0.26	–0.37
Hexanal	–0.27	–0.37	–0.07	**0.86**	0.64	0.19	0.14	–0.32
1-Hexanol	–0.25	–0.44	0.10	**0.78**	**0.83**	0.14	0.37	–0.39
2-Heptanone	–0.42	–0.53	–0.12	**0.85**	**0.71**	0.04	0.15	–0.48
n-Butylfuran	–0.39	–0.30	0.12	**0.89**	**0.79**	0.28	0.01	–0.23
Heptanal	–0.20	–0.52	–0.28	**0.77**	0.33	–0.08	0.04	–0.37
Pentylfuran	–0.24	–0.40	–0.16	**0.80**	0.54	0.10	0.03	–0.36
Phenolic compounds[d]	0.35	**0.87**	**0.85**	–0.12	0.10	**0.77**	0.02	**0.96**

[a] Bold values indicate $r \geq \pm 0.70$.
[b] O = odor, F = flavor, and T = texture.
[c] DUS = degree of unsaturation.
[d] Gallic acid equivalent (GAE, mg/kg).

2-heptanone, *n*-butylfuran, heptanal, and pentylfuran), and phenolic compounds correlated closely with several sensory attributes ($r \geq \pm 0.70$, $P < 0.05$). Free fatty acids showed a close correlation with the sensory attributes perceived in the long-term stored oats, including musty odor and flavor, bitter, germ-like, rancid flavor, and intense aftertaste. Furthermore, the small change in the DUS of triglycerides had a similar correlation to the sensory attributes. Musty, earthy odor, and bitter, musty, rancid flavor had statistically significant correlations with several volatile compounds related to reactions of lipids (pentanal, 2-ethylfuran, 1-pentanol, hexanal, 1-hexanol, 2-heptanone, *n*-butylfuran, heptanal, and pentylfuran). The total phenolic content correlated with the roasted, moist, and intense odor and with the roasted, nutty, sweet, and germ-like flavor when both oat samples at all storage times were considered. Roasted odor and flavor and sweet flavor correlated closely with DMS when the selected volatile compounds were followed during storage.

In addition to rancidity, the bitter flavor of oat is also a restrictive factor for its use. In the present study, rancidity and bitterness were highly positively correlated with each other and with the amount of free fatty acids ($r = 0.85$ and 0.90, respectively) and certain volatile compounds such as pentanal, 2-ethylfuran, 1-pentanol, hexanal, 1-hexanol, 2-heptanone, *n*-butylfuran, heptanal, and pentylfuran ($r = 0.65$ to 0.91 for rancid, and 0.70 to 0.92 for bitter), and negatively with the DUS of triglycerides ($r = -0.71$ for rancid, and –0.76 for bitter, respectively). The influence of nonvolatile compounds on the rancidity and the bitterness was not extensively investigated here.

The relationship of the sensory odor and flavor attributes and the selected volatile compounds during storage was studied by PLS regression (Fig. 2). The first factor of the PLS regression model explained 71% of the variation of the chemical results and 52% of the variation of the sensory data within the two oat samples analyzed during the storage period, and the second factor 12 and 31%, respectively. The unstored, native oat was mainly described by 2-ethylfuran, and the unstored, processed, and dried oat was mainly described by DMS and sweet, roasted odor and flavor. The storage produced significantly different perceptions of the native oat groats: bitter, rancid, and musty sensations, described by pentanal, *n*-butylfuran, pentylfuran, and hexanal. However, in the stored processed oat groats, intense odor, flavor, and aftertaste and nutty flavor were perceived. Of the analyzed volatile compounds, phenyl acetate described these attributes best.

DISCUSSION

Oat flavor can be modified and improved by the germination process as shown previously (Heiniö et al 2001). The limited storage stability typical for oat and oat products was significantly improved by germination and subsequent heat treatment. During the storage, significant sensory changes were noticed both in native oat and processed oat, although the onset of deterioration occurred considerably later in the processed oat than in native oat. While these changes in native oat were already perceived to occur after just one month of storage, in processed oat they did not take place until considerably later.

Statistical multicomparison methods (PCA and PLS regression) are effective for comparing sensory profiles of oat with different chemical variables or even with different processing parameters (Dimberg et al 1996; Molteberg et al 1996a,b; Liu et al 2000; Heiniö et al 2001). According to the PLS regression in the present study, rancidity, bitterness, and musty perceptions were explained by certain volatile compounds related to lipid degradation (hexanal, pentanal, *n*-butylfuran, and pentylfuran). This finding is in good concordance with an earlier study, where the major volatile compounds in stored oat flours were hexanal and 2-pentyl furan (Molteberg et al 1996a). According to Molteberg, both the sensory and chemical stability of oat flour during storage were greatly improved by heat treatment (soaking in water, steaming, and drying). The levels of volatiles and the sensory attributes used to describe rancidity such as grass, hay, and paint, remained rather low up to five weeks of storage. The heat treatment used in the present study maintained the original sensory profile of the processed oat groats effectively for a storage period of at least three months, whereas significant deterioration of the native oat groats was observed within one month.

Deterioration of oat was perceived as a musty, earthy odor and bitter, rancid flavor. These storage-induced perceptions correlated with the hydrolysis products of storage lipids (highly positive with total amount of free fatty acids and negative with the DUS of triglycerides) and with volatile compounds derived from oxidation of unsaturated fatty acids (highly positive with aldehydes, furans,

TABLE IX (continued)
Coefficients of Correlation (*r*) Between Sensory Attribute Scores[a,b] and Relative Amounts of Selected Volatile Compounds, Lipids, and Phenolic Compounds

	F-Nutty	F-Sweet	F-Bitter	F-Germ	F-Musty	F-Rancid	F-Intense	Aftertaste
Free fatty acids (mg/g)	0.04	–0.28	**0.90**	**–0.71**	**0.81**	**0.85**	0.31	**0.70**
Polar lipids DUS[c]	0.57	**0.81**	–0.54	**0.73**	**–0.75**	–0.57	0.22	–0.19
Triglycerides DUS	0.45	0.54	**–0.76**	0.64	**–0.87**	**–0.71**	–0.09	–0.56
Diglycerides DUS	0.46	0.18	0.52	0.01	0.37	0.42	**0.80**	**0.73**
Free fatty acids DUS	–0.22	–0.46	0.00	–0.11	–0.08	–0.20	–0.68	–0.39
Dimethyl sulphide	0.61	**0.81**	–0.39	0.66	–0.60	–0.45	0.49	0.07
Methylpropanal	0.52	0.66	0.17	0.24	–0.04	0.07	0.58	0.45
Methylbutanal	0.41	0.55	0.19	0.17	0.02	0.11	0.50	0.42
3-Pentanone	0.50	0.63	0.17	0.22	–0.04	0.07	0.53	0.43
Benzaldehyde	0.18	0.23	0.35	–0.02	0.27	0.30	0.36	0.40
Phenyl acetate	0.43	0.55	0.21	0.14	0.02	0.12	0.48	0.43
Pentanal	–0.08	–0.46	**0.88**	–0.63	**0.95**	**0.90**	0.44	**0.72**
2-Ethylfuran	–0.37	–0.63	**0.70**	–0.62	**0.92**	**0.78**	0.20	0.49
1-Pentanol	–0.08	–0.56	**0.87**	–0.53	**0.88**	**0.83**	0.33	0.57
Hexanal	–0.06	–0.50	**0.92**	–0.68	**0.95**	**0.92**	0.38	**0.71**
1-Hexanol	–0.08	–0.57	**0.73**	–0.29	**0.71**	0.65	0.25	0.36
2-Heptanone	–0.26	–0.65	**0.81**	–0.63	**0.91**	**0.82**	0.20	0.51
n-Butylfuran	–0.09	–0.43	**0.77**	–0.49	**0.86**	**0.76**	0.40	0.61
Heptanal	0.04	–0.47	**0.89**	**–0.77**	**0.79**	**0.83**	0.13	0.57
Pentylfuran	–0.10	–0.53	**0.89**	**–0.76**	**0.95**	**0.91**	0.32	0.69
Phenolic compounds[d]	**0.78**	**0.95**	–0.16	**0.72**	–0.45	–0.24	0.60	0.19

[a] Bold values indicate $r \geq \pm 0.70$.
[b] O = odor, F = flavor, and T = texture.
[c] DUS = degree of unsaturation.
[d] Gallic acid equivalent (GAE, mg/kg).

and alcohols). The high temperature used in the drying process of the germinated oat proved to be insufficient to denature oat lipase. Consequently, the free fatty acids accumulated in oat samples, regardless of the germination and drying. Despite this, the bitterness of processed oat was considered to be less pronounced than that of native oat with an equal amount of free fatty acids present. This may be explained by the smaller amount of volatile lipid oxidation products in the former and the stronger total flavor of processed oat. However, accumulation of both free fatty acids and the volatile lipid oxidation products appears to affect the development of undesired sensory properties such as rancidity and bitterness.

On the other hand, none of the studied volatile compounds related to protein degradation, nor did the total amount of phenolic compounds explain the perceived deterioration but showed high positive correlations with the desired sensory attributes such as roasted odor and flavor. The perceived odor is always changing according to the changes in the amounts of the volatile compounds (disappearance of the original compounds and development of new ones). However, the compounds identified by the GC/MS technique may not fully explain the perceived odor and flavor as some of the identified compounds may have higher odor thresholds compared with their content in the studied material, or there may be unidentified compounds with odor thresholds below the instrumental detection limit. Also, the very polar volatile compounds may have been ignored due to poor separation in the gas chromatography.

Although free fatty acids are considered to be more susceptible to enzymatic oxidation than acylated fatty acids (Piazza et al 1996), it appears that, in the present study, the hydrolytic deterioration of storage lipids was not related to the development of oxidative deterioration, as the latter was most intensive in the polar lipid fraction, while the hydrolysis occurred exclusively in the triacylglycerols of storage lipids. Qualitative information about the relevance of different oxidation mechanisms in the progress of observed lipid oxidation could be interpreted from the kinetics of oxidation of unsaturated fatty acids. In the present study, this was followed by measuring the rate of accumulation of volatile oxidation products produced through decomposition of fatty acids hydroperoxides. No evidence of autocatalytical oxidation with induction period was noticed, as the accumulation of oxidation products was, after an initial burst, linear with respect to time. Therefore, also the conversion of unsaturated fatty acids into relevant hydroperoxides and the initial formation of free radicals or single oxygen during storage of oat are likely to happen with constant rate. This constant formation of reactive oxygen species could be explained by the presence of photosensitizers that are closely associated with oat lipid phases. Enzymatic oxidation could be expected to be of minor importance compared with other cereals as oat has been reported to have a very small lipoxygenase activity (Ceumern and Hartfield 1984). However, as oat lipids are not present as a continuous phase, but dispersed in different structures such as oil bodies, membranes, and starch structures, the oxidation may simultaneously proceed by a different mechanism in different locations of groat particles.

The improved sensory and chemical stability of processed oat groats could be attributed to the reduction in formation of free fatty

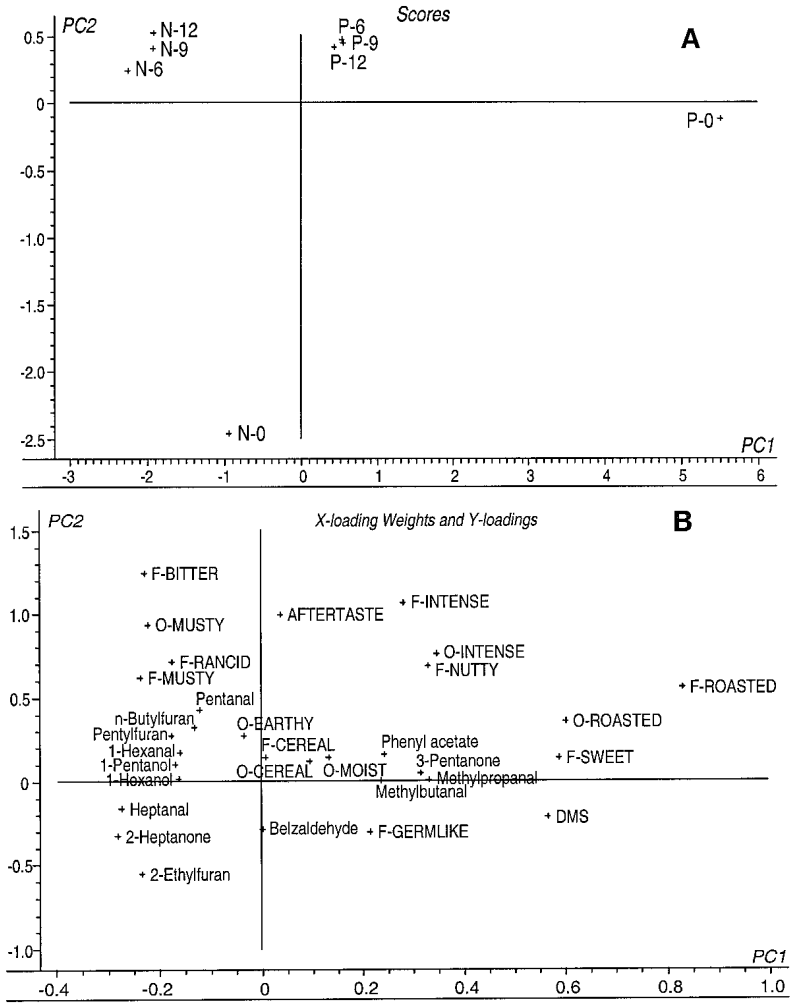

Fig. 2. Partial least squares (PLS) regression scores (**A**) and loadings (**B**) of sensory and headspace data for native and processed oat groats during 0, 1, 3, 6, 9, and 12 months of storage. Abbreviations of sensory attributes are defined in Table I.

acids at the beginning of storage and to the reduced formation of volatile lipid oxidation products over the whole storage period. Compared with a heat treatment normally used to inactivate lipase, the process where oat is germinated and dried appears tempting as it improves the oxidative stability, whereas the heat treatment used for lipase inactivation may promote oxidative deterioration (Ekstrand et al 1993). However, a more intensive lipase inactivation would be needed to ensure the storage stability of processed oat. Otherwise, the free fatty acids generated by the residual lipase activity will not only produce unpleasant lipid degradation products, but may also inhibit the formation of the more pleasant Maillard reaction derived flavors (Parker et al 2000).

CONCLUSIONS

The factors influencing the perceived off-flavors of oat and the importance of processing in extending the shelf-life of oat was demonstrated in the present study. The stability of oat was significantly increased through combined germination and heat treatment. The lipolytic enzyme activity causing rancidity was lower in the processed oat groats than in the native oat. During the storage period, an increase in stability was clearly obtained because formation of the perceived rancid and bitter flavor, degradation of lipids, and production of volatile oxidation products was slower in the processed oat than in the native oat. According to the present study, photosensitivity might significantly influence the oxidation of oat lipids. The volatile compounds related to protein degradation or the total amount of phenolic compounds showed correlations only with the desired sensory attributes such as roasted odor and flavor.

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