JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

Investigation of Packaging Systems for Shelled Walnuts Based on Oxygen Absorbers

PERNILLE N. JENSEN,*,† GITTE SØRENSEN,† PER BROCKHOFF,‡ AND GRETE BERTELSEN†

Department of Dairy and Food Science, and Department of Mathematics and Physics, The Royal Veterinary and Agricultural University, Rolighedsvej 30, DK-1958 Frederiksberg C, Denmark

Storage of nuts at a high oxygen concentration results in rancid nuts whereas storage at a low oxygen concentration results in fine-tasting nuts. During a 13 month experiment, packaging of walnuts with an oxygen absorber was compared to packaging in nitrogen or atmospheric air. At the same time, the effects of oxygen permeability of the packaging material and storage temperature (11 and 21 °C) were investigated by determination of hexanal and rancid taste of the walnuts. The optimal storage condition for walnuts is at 11 °C or lower, eventually combined with an oxygen absorber. However, without chilled storage and use of an oxygen absorber, it is possible to obtain an acceptable quality of walnuts with a packaging material having a very low oxygen permeability (e.g., laminate with EVOH) combined with nitrogen flushing. The results also revealed that the development of hexanal during time can be described by a second-order polynomial regression model.

KEYWORDS: Walnut (*Juglans regia* L.); sensory evaluation; lipid oxidation; hexanal; temperature; permeability; packaging; oxygen absorber

INTRODUCTION

Lipid oxidation is the most important quality parameter in walnuts resulting in oxidation products with an undesirable rancid taste. During harvest, handling, and storage, walnut kernels decline in quality. Factors such as storage temperature and gas atmosphere have a great influence on this quality deterioration. Because walnut kernels contain about 65% lipid of which approximately 70% is unsaturated fatty acids, the walnuts are very susceptible to oxidative deterioration. Until shelling, walnuts are protected by the presence of an intact pellicle and oxidative deterioration is hereby inhibited (*1*). During shelling, the walnuts are affected by mechanical injury and exposed to oxygen and light (*2*), which initiate lipid oxidation.

Extrinsic factors such as oxygen, light, temperature, and relative humidity (RH) all affect the rate of lipid oxidation and thereby the overall quality. The influence of these parameters on the oxidative and sensory changes in walnuts and other nuts is compared in the following.

Several studies have examined the influence of packaging with different oxygen levels on lipid oxidation of nuts by, e.g., nitrogen and carbon dioxide (CO_2) flushing, vacuum packaging, and use of an oxygen absorber. Comparison of storage at 21% and <2.5% oxygen (nitrogen flushing) revealed lower peroxide values (PV) and hexanal content for shelled peanuts and walnuts (*3*) and a better sensory quality of walnuts (*4*) when packed with nitrogen flushing. Exchanging the atmospheric air with CO_2 extended the shelf life of pecans from 2 to 27 weeks and raw peanuts obtained a shelf life of 12 months in which the peanuts obtained the same sensory quality as peanuts stored at 1.7 °C (*5*). This is in agreement with another study where pecans obtained a slightly better sensory quality by nitrogen flushing as compared to vacuum packaging and both packaging methods resulted in pecans that were sensory acceptable after 6 months of storage at 24 °C and 60% RH (*6*). On the other hand, oxygen concentrations of no more than 2.1% were found to be sufficient to initiate lipid oxidation and thereby rancid taste (*4*). To exclude oxygen from the packages during the entire storage period, the use of oxygen absorbers has been investigated, and it has been shown that oxygen absorbers prevented lipid oxidation in Brazilian nuts and kept the PV at a lower level than nuts packed with atmospheric air or nitrogen flushing (6% oxygen). This was especially obvious after 2 months of storage where nuts packed in atmospheric air were unacceptable (*7*).

The availability of oxygen in the package can to a certain extent be controlled by the oxygen permeability of the packaging material. As expected, a high oxygen permeability is not satisfactory for packaging of nuts, as the nuts oxidize and develop rancid taste (*4*, *6*). Interestingly, packaging materials with a low oxygen permeability or use of vacuum packaging did not result in the expected improved quality of pecans (*6*).

The previously described investigations illustrate that storage of nuts at high oxygen concentration or in materials with high oxygen permeability results in pronounced lipid oxidation and

* To whom correspondence should be addressed. Tel: +45 35 28 32 68. Fax: +45 35 28 33 44. E-mail: pnj@kvl.dk.
† Department of Dairy and Food Science.
‡ Department of Mathematics and Physics.

10.1021/jf021206h CCC: $25.00
Published on Web 07/17/2003

thereby lower sensory quality. Parallel to this, the use of an oxygen absorber or flushing with N_2 or CO_2 resulted in nuts with very good sensory quality and an almost inhibited lipid oxidation. However, one investigation showed that the packaging material could be too efficient an oxygen barrier as a low oxygen permeability resulted in lower sensory quality, probably due to fermentation of the nuts (*6*).

The effect of light and RH on the oxidative rancidity of nuts is not as extensively investigated as the effect of oxygen even though these have a great influence on the rate of lipid oxidation. Storage of almonds, peanuts, pinenuts (*8*, *9*), and walnuts (*8*, *9*) in light gave rise to lower sensory quality as compared to storage in the dark. Walnuts and peanuts stored at 53% RH have a much greater increase in PV than nuts stored at 21% RH (*3*). The same tendency was seen for the water content in walnuts, where storage at 60% RH resulted in a faster loss of water as compared to 40%. This can be explained by the fact that the water content of the nuts tends to equilibrate with the RH in air (*10*).

The rate of lipid oxidation is also affected by temperature, and the optimum storage conditions for shelled walnuts are 0−3.5 °C and 55−65% RH. Under these conditions, walnuts generally have a shelf life of 12 months (*2*, *11*). Walnuts are reported to maintain their quality for 4−12 months even when stored at 10 °C (*4*, *9*, *10*). Storage at higher temperatures such as 21 and 40 °C results in very rancid walnuts within a few months (*4*, *9*), and this temperature effect is especially seen when walnuts are stored under light exposure (*9*). In addition, several studies have shown an increased oxidative rancidity in nuts with prolonged storage (*4*, *5*, *8*, *9*).

The primary aim of the present study is to investigate how oxygen absorbers, different gas atmospheres, and storage temperatures affect the oxidative and sensory quality of walnut kernels. To determine the effect of oxygen absorbers, an experiment comparing the use of oxygen absorbers with nitrogen flushing and atmospheric air was performed. Simultaneously, the effect of oxygen permeability by using different plastic packaging materials and storage temperatures (11 and 21 °C) was evaluated.

In a 13 month experiment, the oxidative changes were followed by determination of the hexanal content and the sensory quality was evaluated by the rancid taste. The concentration of hexanal in headspace has in several studies shown to correlate well with the sensory evaluation and has found applicability in, e.g., walnuts (*9*) and pecans (*12*). A minor aim is to investigate if the development of hexanal during time can be described mathematically, and the hexanal content was therefore subjected to a polynomial regression model. In addition, walnuts were analyzed by vis/near infrared (NIR) spectroscopy, which is a good general purpose technique for monitoring quality deterioration of different foodstuffs (*9*, *13*−*16*).

MATERIALS AND METHODS

Walnut Kernels and Bulk Packaging. The walnuts (*Juglans regia* L.) were harvested in 1997 in France and stored in shell at 5 °C for 2−3 months until they were cracked and shelled mechanically. Immediately hereafter, they were bulk-packed in paperboard cartons with an inner plastic bag of PE containing 12.5 kg of walnuts and transported to Denmark. Upon arrival, the walnut kernels were packed in plastic bags with a low oxygen transmission rate (OTR), nitrogen flushed, and stored at −18 °C for 2 months until the beginning of the experiment. Each bag contained 1 kg of walnut kernels.

Experimental Design. The walnut kernels were packed in three different laminated packaging materials: 70 μm LDPE (PE) with an OTR of 2900 cm^3/m^2/24 h/atm (23 °C); 12 μm PET/50 μm LDPE (PET) with an OTR of 100 cm^3/m^2/24 h/atm (23 °C) and OTR of 56 cm^3/m^2/24 h/atm (10 °C); and 15 μm OPA (EVOH)/50 μm LDPE (EVOH) with an OTR of 2 cm^3/m^2/24 h/atm (23 °C); see **Table 1**.

Table 1. Experimental Design for the Packaging and Storage Experiment Material

packaging material	packed with	temperature (°C)	treatment
PE	atmospheric air	21	1
PET	atmospheric air	11	2
		21	3
	nitrogen flushing	11	4
		21	5
	oxygen absorber	11	6
		21	7
EVOH	atmospheric air	21	8
	nitrogen flushing	21	9
	oxygen absorber	21	10

The samples were stored in darkness at either 11 or 21 °C, and after 0, 1, 3, 5, 7, 9, and 13 months of storage, the samples were withdrawn for chemical and sensory analyses. At each measurement, two identical bags were analyzed and duplicates were made of the chemical analyses. One assessment was performed by each judge at the sensory evaluation. The results from the chemical analyses are reported as an average of four determinations, whereas the sensory evaluation is a sum of the ranking values for the seven judges. Between the different chemical analyses, the samples were vacuum-packed and stored at 5 °C for maximum 14 days, as all the analyses could not be performed on the same day. Control reference samples used for sensory evaluation were vacuum-packed and stored at −18 °C.

Chemical Analyses. *Gas Composition.* Before the bags were opened for analysis, the gas composition was determined using a calibrated Gaspace 2 gas analyzer (Systech Instruments Ltd., Thame, U.K.). The gas composition was used to identify leaking bags.

Water Content. The water content of the products was determined by placing approximately 2.0 g of the homogenized (10 s in a coffee mill (Braun, Germany)) sample on a dry aluminum tray. The sample was dried for 16−18 h at 105 °C and tempered in a desiccator for 30 min before weighing to get the water content. The results were expressed as g water/g dry matter.

Lipid Content. The lipid was extracted with a chloroform/methanol (2:1) mixture according to Erickson (*17*) with minor modifications. The homogenized (10 s in a coffee mill) walnuts and the solvent were homogenized with an Ultra Turrax for 15 s at 8000 rpm; the solvent was evaporated using vacuum. The result was expressed as % fat.

Fatty Acid Composition. The fatty acid composition was determined according to Jart (*18*) with the following modifications. Ten milligrams of extracted oil was used; the samples were kept at 60 °C (water bath) for 40 min; prior to analysis on the gas chromatograph, the samples were evaporated and dissolved in 1.0 mL of pentane.

The oven temperature program and conditions were as follows: 50 °C for 1 min; from 50 to 180 °C at 15 °C/min; from 180 to 220 °C at 5 °C/min; at 220 °C for 10 min. Column pressure at 136 kPa; 1 μL was injected, the split ratio was 1:10, and the flow rate was 1 mL/min. The results were expressed as % fatty acid of the total content of fatty acids.

Hexanal Content. Hexanal content was measured by headspace gas chromatography according to Jensen et al. (*9*).

Visible/NIR Spectroscopy. Visible/NIR spectroscopic data were measured in the range from 400 to 2498 nm using a NIRSystems spectrophotometer (model 6500, NIRSystems Inc., Silver Spring, MD) according to Jensen et al. (*9*).

Sensory Evaluation. The sensory evaluation was performed by the same seven trained panelists throughout the storage period after the samples were withdrawn for analysis. Prior to the sensory evaluation, the walnut kernels were homogenized in a blender in order to ensure that each panelist was served a representative sample of walnuts. At each session, the panelists were served a control reference sample (stored at −18 °C) and samples from the actual storage time. Homogenized walnuts (20 g) were placed on small cardboard plates

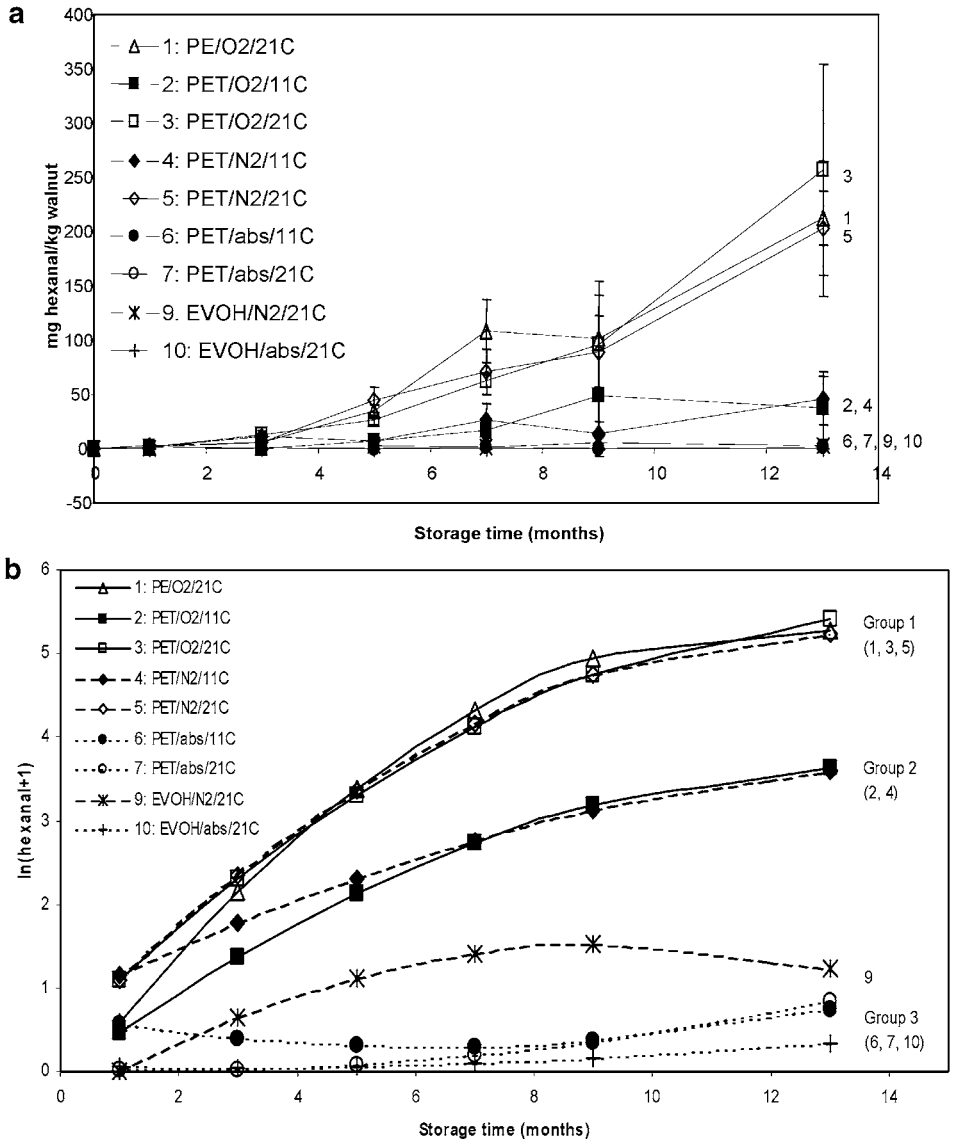

Figure 1. Lipid oxidation in walnut kernels measured as hexanal content (mg/kg) shown as (**a**) raw data with a loosely fitted curve and (**b**) regression models (ln hexanal + 1).

for each assessor 15 min before the session. In each session, all of the samples were randomly distributed to each panelist. All sensory evaluations took place in individual taste panel booths. The descriptor term rancid taste was used to describe the sensory quality, which was evaluated using ranking, where a high ranking value corresponded to rancid taste. In addition, the panelists were instructed to comment on the different samples. Each time the rancid taste was evaluated in two sessions. In the first sensory session, walnuts stored at 21 °C were evaluated and conclusions about the importance of the packaging materials were made. The second session accessed the packaging material PET stored at both 11 and 21 °C, and the effects of storage temperature on the rancid taste were evaluated. The samples were served at a temperature of 20 °C. Between each serving, the panelists rinsed their mouths with sparkling water (room temperature), cucumber, and neutral tasting crackers.

Data Handling. Results from measurement of hexanal content were subjected to analysis of covariance using storage time as a covariate (SAS version 6.12 software; SAS Institute Inc., Cary, NC). Response was used as the natural logarithm of the hexanal content with a one added, ln(hexanal + 1). This ensured that the assumptions of normality and variance homogeneity in the analysis were reasonable. A second-order orthogonal polynomial model with coefficients depending on the treatments was used to describe the development of ln(hexanal + 1) over time. Post hoc pairwise comparisons, by *t*-tests, between treatments were carried out for the intercepts (average levels), the slopes, and the curvature estimates. The vis/NIR spectra were evaluated by multivariate data analysis using principal component analysis (PCA). The data were both mean centered and autoscaled (divided by the standard deviation), and the PCA models were evaluated using segmented cross-validation. Each segment contains data from one storage time only, except one segment that contains data from weeks 0 to 4 (months 0 and 1). Multivariate data analysis was performed using The Unscrambler version 7.5 (CAMO, Trondheim, Norway). The ranking values from the sensory evaluation were compared by simple ranking test (Friedman analysis) (*19*).

RESULTS

Walnuts are prone to lipid oxidation because of a high content of unsaturated fatty acids. In this study, the walnut kernels had an initial fat content of 55.4% and fatty acids composed of 7.3% palmitic acid, 2.2% stearic acid, 16.4% oleic acid, 59.6% linoleic acid, and 12.0% linolenic acid.

The oxidative stability of the walnuts was determined by measurement of hexanal content, and as can be seen from **Figure 1a**, the hexanal content increased during storage except for walnut kernels packed in EVOH with nitrogen flushing (*9*)

or with an oxygen absorber (*6*, *7*, *10*). For these packaging systems, the hexanal content was constantly low during the entire storage period. Walnuts stored at 21 °C and packed in PE/atmospheric air, PET/atmospheric air, and PET/N_2 (*1*, *3*, *5*) achieved the highest hexanal content, whereas storage of walnuts packed in PET/atmospheric air and PET/N_2 at 11 °C (*2*, *4*) resulted in a significant reduction in the formation of hexanal. At the beginning of the experiment, hexanal content in the walnuts was actually below detection level of the method, indicating that shelling and transport conditions of the walnuts from harvest to packaging do not result in development of secondary oxidation products in the kernels. However, the lipid oxidation could already be initiated.

The results from gas composition analysis (data not shown) overall correlated well with the degree of hexanal formation. In packages using an oxygen absorber (treatments 6, 7, 10), oxygen content was kept at 0.00% throughout the storage period, just as in the packaging material with a low oxygen permeability (EVOH, treatments 8 and 9) where oxygen content was reduced to 1.5% or lower. The latter indicates that walnuts consume almost the total amount of oxygen available in headspace as minimal oxygen is transported into the plastic bag.

Figure 1a illustrates some variation in the degree of oxidation when the availability of oxygen is high, especially for treatments 3 and 5. The variation is observed between two bags, which have been stored under exactly the same conditions and assumed to be identical. This difference could be explained by biological variation between walnut kernels in oxygen consumption rates. Another possibility is of course the existence of pinholes in the packaging material, which on the other hand is not supported by gas composition analysis.

A thorough examination of raw data revealed lack of systematic in the results from EVOH packed with atmospheric air and stored at 21 °C (treatment 8). A large variation in the hexanal content from time to time was observed that could not be accounted for by availability of oxygen but might be explained by biological variation or pinholes as described above. Because of these uncertainties, it was decided to exclude this treatment from the data analysis.

According to the analysis on the transformed data (ln(hexanal + 1)), the development of hexanal during storage for a given packaging system can be described by a second-order polynomial regression model. The estimated regression models for the nine packaging systems are shown in **Figure 1b**. The comparison of the regression models clearly shows three groups: The first group (PE/atm air/21 °C, PET/atm air/21 °C, PET/N_2/21 °C (treatments 1, 3, and 5)) has the highest level of hexanal and the highest availability of oxygen as they are packed in either PET with atmospheric air or nitrogen flushing or in PE and stored at 21 °C. The second group (PET/atm air/11 °C, PET/N_2/11 °C (treatments 2 and 4)) is stored at 11 °C, and the temperature shift significantly lowers the hexanal formation. The third group (PET/abs/11 °C, PET/abs/21 °C, EVOH/abs/21 °C (treatments 6, 7, and 10)) has no significant development of hexanal, and oxygen is kept at approximately 0% due to the use of oxygen absorbers. When using oxygen absorbers, the hexanal content for PET stored at 11 and 21 °C is the same and the storage temperature is therefore insignificant. The curvature of EVOH/N_2 (*9*) is very similar to group two, but the level corresponds more to group three.

By performing *t*-tests, the differences between estimates of intercepts, slopes, and curvatures can be compared for the nine regression models. From **Table 2**, it can be seen that the grouping into three is mainly explained by the intercept, which is the overall start level of ln(hexanal + 1), and by the slope, which can be perceived as the rate of hexanal formation. Group 1 (PE/atm air/21 °C, PET/atm air/21 °C, PET/N_2/21 °C (treatments 1, 3, and 5)) has the highest hexanal content (intercept) and the fastest development of hexanal (slope). Group 3 (PET/abs/11 °C, PET/abs/21 °C, EVOH/abs/21 °C (treatments 6, 7, and 10)) has the lowest content of hexanal and the slowest rate of hexanal formation. Between these two groups are EVOH/N_2 and group 2 (PET/atm air/11 °C, PET/N_2/11 °C (treatments 2 and 4)). The interpretation of the differences between curvatures for the nine treatments is more complex, but the tendency is that groups 2 and 3 and EVOH/N_2 have similar curvatures, which are different from group 1.

Table 2. Comparison of Intercepts, Slopes, and Curvatures[a]

treatment	intercept[b]	slope[c]	curvature[d]
1	3.44[a]	0.391[a]	–0.0387[a]
2	2.26[b]	0.264[b]	–0.0194[ab]
3	3.49[a]	0.359[ab]	–0.0240[ab]
4	2.45[b]	0.203[bc]	–0.0108[abc]
5	3.49[a]	0.344[ab]	–0.0277[ad]
6	0.446[d]	0.0156[d]	0.00993[ce]
7	0.196[d]	0.0622[d]	0.00642[be]
9	0.948[c]	0.0982[cd]	–0.0221[ab]
10	0.102[d]	0.0202[d]	0.00285[bde]

[a] Within intercept, slope, or curvature treatments 1–10 with different letters (a, b, c, or d) are significantly different ($P < 0.05$). [b] Estimates from model ln(hexanal + 1) = *f*(treat). [c] Estimates from model ln(hexanal + 1) = *f*(treat, treat × time). [d] Estimates from model ln(hexanal + 1) = *f*(treat, treat × time, treat × time^2).

The oxidative changes caused by oxygen content in headspace and temperature also affected rancid taste of the walnuts. The sensory evaluation was performed as ranking and compares the different treatments at a given time. In addition, the panelists have commented on the rancidity of the walnuts. In the following, comments are used to support the comparison of hexanal content of different treatments. In both the first and the second session, the statistical analysis of ranking values ($p \leq 0.05$; data not shown) supports the comments from the panelists.

Figure 2a shows the panelists' comments for walnuts stored at 21 °C. In the figure, samples within the same circle are judged similar. Walnuts packed in PE/atm air, PET/atm air, and PET/N_2 were already rated very rancid already after 5 months and became inedible after 8 months. Opposite these rancid walnuts, the reference sample (stored at −18 °C) is ranked as having the best obtainable quality in the experiment. During the entire storage experiment, walnuts packed with an oxygen absorber achieved the same good quality as the reference. The packaging system EVOH/N_2 was almost as good as the reference, except after 7 months at which time the walnuts were slightly rancid.

The rancid taste for walnuts stored in PET at 11 and 21 °C is shown in **Figure 2b**. Walnuts packed in atmospheric air or nitrogen and stored at 21 °C became much more rancid than walnuts stored at 11 °C, i.e., lowering the temperature markedly reduced the formation of rancid taste. However, the walnuts packed with oxygen absorber and stored at 11 or 21 °C maintained the same quality as the reference. Thus, when using oxygen absorbers, the storage temperature is in this study found to be insignificant.

The initial water content in the walnut kernel was 4.3%, and during the first 3 months of storage, water content decreased independent of storage conditions by approximately 50% (data not shown). After 7 and 11 months, the water loss was more pronounced for walnuts stored at 21 °C rather than at 11 °C.

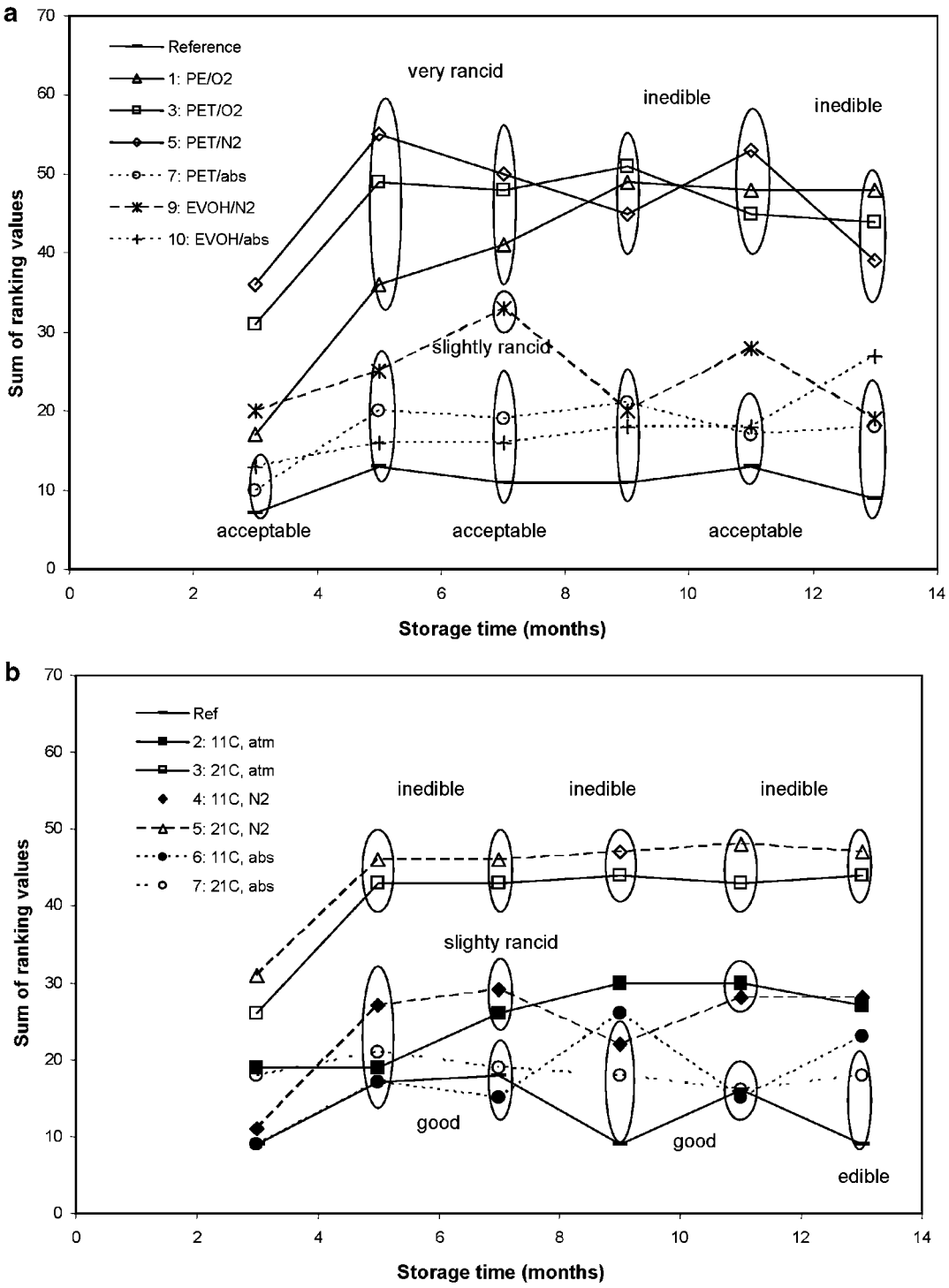

Figure 2. Sensory evaluation of walnut lipid oxidation expressed as rancid taste for (**a**) walnuts stored at 21 °C and (**b**) walnuts packed in the reference material PET. Samples within the same circle are sensory evaluated similar.

The loss of water affects the texture as the nuts get drier and hence reduces the quality. However, this water loss was not commented by the panelists as they were only evaluating the rancid taste.

The vis/NIR data were evaluated using PCA, and a score plot for the PCA performed on the packaging material PET is shown in **Figure 3**. The score plot shows the two first principal components (PCs), which describe 96% (85 + 11%) of the total variance in the data material. In the plot, the effect of temperature is evident as walnuts stored at 11 °C are positioned at the upper side of the plot and walnuts stored at 21 °C are situated at the lower side (separated by line). This corresponds to the trend in rancidity, where samples at the left (21 °C) are more rancid than samples at the right (11 °C) of the plot. A closer study of this plot shows that walnuts packed in PET/atm air or PET/N_2 and stored at 21 °C are placed alike in the plot. The same accounts for walnuts stored at 11 °C using the same packaging conditions. It is also seen that walnuts packed with an oxygen absorber and stored at 11 °C are placed near the reference (PET) (at right) during the whole storage period.

The score plot for PCA performed on walnuts stored at 21 °C is seen in **Figure 4**. PC1 and PC2 describe 95% (80 + 15%)

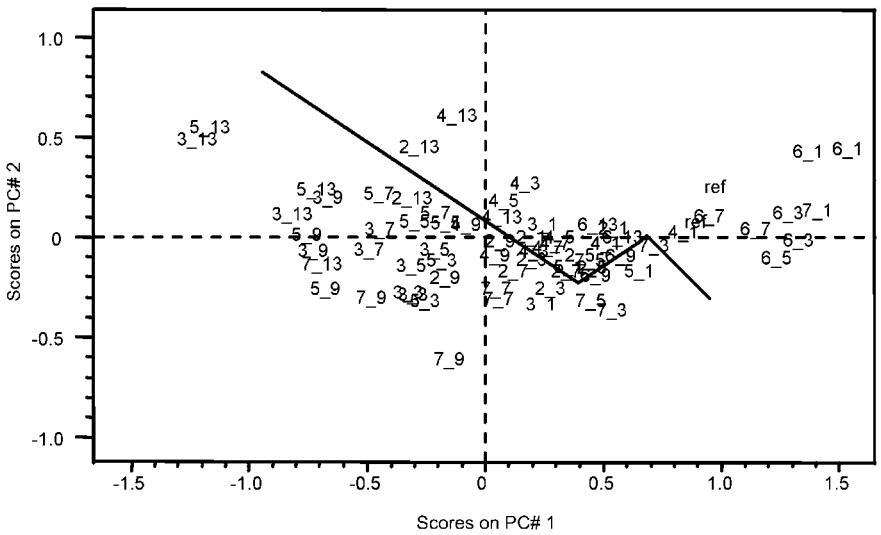

Figure 3. Score plot from PCA on multiplicative scatter corrected (MSC) vis/NIR spectra of walnuts packed in PET. Legends: first number corresponds to treatment (see **Table 1**) and second number to storage time in months.

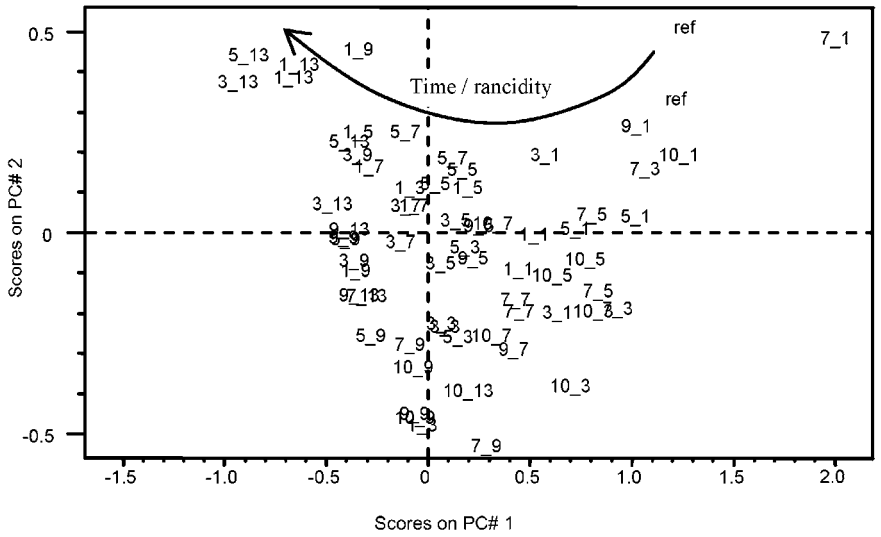

Figure 4. Score plot from PCA on MSC vis/NIR spectra of walnuts stored at 21 °C. Legends: first number corresponds to treatment (see **Table 1**) and second number to storage time in months.

of the total variation. A time and/or rancidity effect is observed in the model, as storage time and/or rancidity increase from right to left. Near the reference are walnuts stored for 1 month and opposite these walnuts stored for 9 and 13 months.

DISCUSSION

Initially, the walnut kernels contained no hexanal, indicating that shelling and transport of the walnuts until packaging does not cause oxidation of the kernels (**Figure 1b**). To maintain this good quality, the walnuts have to be packed in a material with a low oxygen permeability and removal of oxygen in headspace.

Second-order polynomial regression models can describe the development in hexanal content and organize the packaging systems in three groups. During the entire storage period, walnuts packed in PE/atm air, PET/atm air, and PET/N_2 (treatments 1, 3, and 5) and stored at 21 °C contained the highest hexanal content (**Figure 1b**) and were evaluated as very rancid and inedible (**Figure 2b**). This is in agreement with Dull and Kays (*6*) and Jan et al. (*4*) who also described that exposure of nuts to a high amount of oxygen results in pronounced lipid oxidation and the nuts develop a rancid taste. The correlation between hexanal content and rancid taste observed here has also been found in walnuts (*9*), pecans (*12*), and potato chips (*20*). For the reference material, PET storage at 11 °C results in walnut kernels with an improved quality as compared to storage at 21 °C as the development in hexanal and rancid taste are reduced significantly. Previous studies have shown that in order to retard oxidation completely it is necessary to store walnuts below 10 °C (*4*, *9*, *10*). On the other hand, exchange of the atmospheric air with N_2 or CO_2 (*3*−*7*) or even better, use of an oxygen absorber (*7*), prevented lipid oxidation in different nuts. Therefore, not surprisingly, we found that the initial quality of the walnuts did not change during 13 months of storage independent of storage temperature, when an oxygen absorber was used.

The water content was already after 3 months of storage reduced to 2.5%. According to Rockland et al. (*21*), only small sensory changes were observed at a water content of 3.1−4.0%, but 3.1% exhibited the greatest oxidative stability and the water content should therefore not be reduced below 3.1% to ensure optimal storage. López et al. (*10*) found that because of the weight loss it is not advisable to store walnuts at 40% RH as compared to 60% RH. In our experiment, the RH at 21 and 11

°C was 35 and 54%, respectively. Here, the water loss was, not surprisingly, more pronounced at 21 °C than at 11 °C. The effect of water loss on the oxidative stability is not clear from our experiment as the content of oxygen in the packages is dominating and seems to "control" the degree of oxidation.

Using PCA on vis/NIR spectra demonstrates the effect of temperature as walnuts packed in PET and stored at 11 or 21 °C are clearly separated in the score plot (**Figure 3**). This split-up demonstrates the rancidity of the walnuts, as walnuts stored at 21 °C are more rancid (higher hexanal content and inedible) than walnuts stored at 11 °C. As for the rancid taste, the score plot also shows that walnuts packed in PET/atm air or PET/N_2 and stored at 21 °C are placed opposite the reference indicating deterioration of the walnuts. As expected, the walnuts stored at 11 °C with an oxygen absorber achieved the best quality shown by the samples being placed near the reference.

The vis/NIR data were also able to illustrate the effect of storage time or rancidity of walnuts stored at 21 °C. In the score plot (**Figure 4**), walnuts stored for 13 months are placed opposite walnuts stored for 1 month and the reference, and between these, walnuts stored for 3−9 months are located. This is in agreement with several investigations, where prolonged storage resulted in increased rancidity (*4*, *5*, *8*, *9*).

The experiment described in this paper suggests that the optimal storage condition for walnuts is at 11 °C or lower, eventually combined with an oxygen absorber. Unfortunately, storage of walnuts at 11 °C is not a good solution for the retail market as it requires too much refrigerated space in the store and when using oxygen absorbers the consumer has to become accustomed to it. On the other hand, the price of cooling facilities for the supermarket and the additional cost of the oxygen absorber for the manufacturer also have to be take into account. However, without cooling and use of an oxygen absorber, it is possible to obtain walnuts with high quality with a packaging material having low oxygen permeability like EVOH combined with nitrogen flushing.

ACKNOWLEDGMENT

This work was performed within the Centre for Food Packaging, which is sponsored by the Danish Agency for Trade and Industry. We are grateful to participants from Coop Danmark, AGA A/S, Amcor Flexible, Scan Pack I/S, Ehrno Plast A/S, PBI-Dansensor A/S, Standa Industrie, and The Danish Technological Institute (Packaging and Transport) who provided technical assistance.

LITERATURE CITED

(1) Jurd, L. Plant polyphenols. I. The polyphenolic constituents of the pellicle of the walnut (*Juglans regia*). *JAOCS* **1956**, *78*, 3445−3448.

(2) Anonymous. Nutty know-how: Food processing reports. *Food Proc.* **1990**, *59*, 23−24, 27.

(3) Maté, J. I.; Saltveit, M. E.; Krochta, J. M. Peanut and walnut rancidity: Effects of oxygen concentration and relative humidity. *J. Food Sci.* **1996**, *61*, 465−468, 472.

(4) Jan, M.; Langerak, D. I.; Wolters, T. G.; Farkas, J.; Kamp, H. J. V. D.; Muuse, B. G. The effect of packaging and storage conditions on the keeping quality of walnuts treated with disinfestation doses of gamma rays. *Acta Aliment.* **1988**, *17*, 13−31.

(5) Holaday, C. E.; Pearson, J. L.; Slay, W. O. A new packaging method for peanuts and pecans. *J. Food Sci.* **1979**, *44*, 1530−1533.

(6) Dull, G. G.; Kays, S. J. Quality and mechanical stability of pecan kernels with different packaging protocols. *J. Food Sci.* **1988**, *53*, 565−567.

(7) Ribeiro, M. A. A.; Regitano-d'Arce, M. A. B.; Lima, U. A.; Nogueira, M. C. S. Storage of canned shelled brazil nuts (*Bertholletia excelsa*): effects on the quality. *Acta Aliment.* **1993**, *22*, 295−303.

(8) Sattar, A.; Mohammad, J.; Saleem, A.; Jan, M.; Ahmad, A. Effect of fluorescent light, gamma radiation and packages on oxidative deterioration of dry nuts. *Sarhad J. Agric.* **1990**, *6*, 235−240.

(9) Jensen, P. N.; Sørensen, G.; Engelsen, S. B.; Bertelsen, G. Evaluation of quality changes in walnut kernels (Juglans regia L.) by VIS/NIR spectroscopy. *J. Agric. Food Chem.* **2001**, *49*, 5790−5796.

(10) López, A.; Pique, M. T.; Romero, A.; Aleta, N. Influence of cold-storage conditions on the quality of unshelled walnuts. *Int. J. Refrig.* **1995**, *18*, 544−549.

(11) Ravai, M. Quality characteristics of california walnuts. *Cereal Foods World* **1992**, *37*, 362, 365−366.

(12) Kanamangala, R. V.; Maness, N. O.; Smith, M. W.; Brusewitz, G. H.; Knight, S.; Chinta, B. Reduced lipid pecans: chemical alterations and implications for quality maintenance during storage. *J. Am. Soc. Hortic. Sci.* **1999**, *124*, 389−398.

(13) Brøndum, J.; Byrne, D. V.; Bak, L. S.; Bertelsen, G.; Engelsen, S. B. Warmed-over flavour in porcine meat—a combined spectroscopic, sensory and chemometric study. *Meat Sci.* **2000**, *54*, 83−95.

(14) Engelsen, S. B. Explorative spectrometric evaluation of frying oil deterioration. *JAOCS* **1997**, *74*, 1495−1508.

(15) Indahl, U. G.; Sahni, N. S.; Kirkhus, B.; Næs T. Multivariate strategies for classification based on NIR-spectra—with application to mayonnaise. *Chemom. Intell. Lab. Syst.* **1999**, *49*, 19−31.

(16) Yildiz, G.; Wehling, R. L.; Cuppett, S. L. Method for determining oxidation of vegetable oils by near-infrared spectroscopy. *JAOCS* **2001**, *78*, 495−502.

(17) Erickson, M. C. Methods for measurement of pecan quality. In *Pecan Technology*; Santerne, C. R., Ed.; Chapman & Hall: New York, 1994.

(18) Jart, A. The magnetic field as an additional selectivity parameter in fat hydrogenation. *JAOCS* **1997**, *74*, 615−617.

(19) Meilgaard, M.; Civille, G. V.; Carr, B. T. *Sensory Evaluation Techniques*, 3rd ed.; CRC Press: USA, 1999.

(20) Goode, J. E.; Soutar, A. M. Rancidity in packaged nuts: end-use technical service. *J. Plast. Film Sheeting* **1995**, *11*, 235−247.

(21) Rockland, L. B.; Swarthout, D. M.; Johnson, R. A. Studies on English (Persian) walnuts, *Juglans regia*. III.: stabilization of kernels. *Food Technol.* **1961**, *15*, 112−115.

Received for review December 12, 2002. Revised manuscript received May 19, 2003. Accepted June 5, 2003.

JF021206H