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Storage stabilities of pork scratchings, peanuts, oatmeal and muesli: Comparison of ESR spectroscopy, headspace-GC and sensory evaluation for detection of oxidation in dry foods

Pernille N. Jensen, Bente Danielsen, Grete Bertelsen, Leif H. Skibsted, Mogens L. Andersen *

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

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Abstract

Peanuts, pork scratchings, oatmeal and two types of muesli were stored in two experiments, where external factors (light, oxygen concentrations, product-headspace ratios) were varied, and where packaging materials with different properties (light transmission and oxygen permeability) were used. The oxidative changes in the products were followed by the formation of hexanal as detected by headspace gas chromatography (headspace-GC), free radicals as detected by electron spin resonance (ESR) spectroscopy and sensory evaluations. Generally, increased oxygen availability and exposure to light resulted in increased lipid oxidation. Statistical analysis of the results revealed that light accounted for the greatest systematic variation of the relative levels of free radicals in peanuts, oatmeal and muesli, whereas the oxygen availability had the largest influence on the formation of hexanal. The opposite was observed for pork scratchings, where oxygen had the most significant effect on the formation of radicals. It is concluded that ESR and headspace-GC complement each other in detecting the oxidative changes.

Keywords: Electron spin resonance (ESR); Hexanal; Peanuts; Pork scratchings; Oatmeal; Muesli; Oxidation; Light; Oxygen

1. Introduction

Peanuts, pork scratchings, oatmeal, and muesli are dried foods that are susceptible to lipid autoxidation due to a high content of unsaturated fatty acids. Free radicals are important chain-carrying intermediates in lipid autoxidation, which lead to the formation of hydroperoxides which further are precursors of secondary oxidation products, such as ketones and aldehydes. The appearance of secondary oxidation products is associated with changes in the odour and flavour of the products, resulting in rancidity. Measurements of hexanal or other volatile compounds, using headspace gas chromatography (GC), have been used to monitor the oxidative deterioration of e.g. peanuts, oats and potato chips (Erickson, 1993; Jensen, Sørensen, Engelsen, & Bertelsen, 2001; Jeon & Bassette, 1984; Lennersten & Lingnert, 1998; Molteberg, Magnus, Bjørge, & Nilsson, 1996a; Molteberg, Solheim, Dimberg, & Frølich, 1996b; Molteberg, Vogt, Nilsson, & Frolich, 1995; Reed, Sims, Gorbet, & O'Keefe, 2002). In addition, the evaluation of oxidative status is occasionally combined with sensory evaluation, as for roasted peanuts, for which formation of secondary oxidation products (including pentanal, hexanal and octanal) results in loss of peanutty flavour and development of a painty flavour instead (Reed et al., 2002). Oxidative deterioration of oats, which is accelerated by storage under an atmosphere with

* Corresponding author. Tel.: +45-3528-3262; fax: +45-3528-3344.
E-mail address: mola@kvl.dk (M.L. Andersen).

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increased relative humidity, leads to the formation of hexanal and is accompanied by a reduction in oat odour and flavour, sweetness and pleasant aftertaste (Molteberg et al., 1996b).

Radicals that are formed during oxidation of food can often be detected and quantified by electron spin resonance (ESR) spectroscopy and correlations between the tendency toward formation of radicals in fresh products and the oxidative stability (shelf life) have been demonstrated for several products (Andersen & Skibsted, 2002). In dehydrated chicken meat, milk powder and potato flakes, the content of free radicals has already proved to be a good indicator of early stages of oxidation (Nissen, Huynh-Ba, Petersen, Bertelsen, & Skibsted, 2002; Nissen, Månson, Bertelsen, Huynh-Ba, & Skibsted, 2000; Stapelfeldt, Mortensen, & Skibsted, 1997; Stapelfeldt, Nielsen, & Skibsted, 1997). In addition, the concentration of free radicals in dehydrated chicken meat correlates with the hexanal content and the sensory descriptor ''rancidity'' (Nissen et al., 2000) while, for milk powder, the amount of free radicals correlates both with the concentration of thiobarbituric acid-reactive substances, TBARS, and with the sensory impression of oxidation (Stapelfeldt et al., 1997). Furthermore, both the radical concentration (Szocs, 2002) and the hexanal content (Jensen et al., 2001) have proven to be good indicators of lipid oxidation in walnuts.

The use of ESR and headspace-GC as a tool for evaluating the oxidative status of peanuts, pork scratchings, oatmeal and two types of muesli has been examined, in the present study, by comparison of the amount of radicals, as detected by ESR spectroscopy, and the formation of hexanal affecting the sensory quality, during storage of the products under different conditions. Another objective has been to investigate the effects of external factors, such as oxygen availability (oxygen concentration and product-to-headspace ratio) and light on oxidation. The effect of oxygen transmission rate (OTR) of the packaging material, along with different oxygen concentrations in the package, on the oxidative stability was also studied since knowledge of the influence of these parameters on the stability of the actual products is lacking in the literature. A comparison of the stability of oatmeal and a mixture of coated rolled oats and wheat flakes from muesli was also included in the study, in order to evaluate the influence of coating on the oxidative stability.

2. Materials and methods

2.1. Products

Roasted, salted peanuts and pork scratchings were obtained from KiMs A/S, Denmark. Pork scratchings are a Danish snack product, which are produced by frying the pig skin with approximately 1 cm layer of fat. The cracklings are finally flavoured with salt and packed. Muesli I (Nutana A/S, Denmark) was made from rolled oats, wheat flakes, extruded rice, raisins, almonds and desiccated coconut. Refined vegetable oil (4%) and honey were added to the mixed ingredients before baking. Muesli II (Cerealia Foods A/S, Denmark) contained rolled oats, extruded rice and corn flakes that were coated with a mixture of sugar, vegetable oil, glucose syrup and honey. Oatmeal was obtained from Cerealia Foods A/S, Denmark. All the products were produced immediately before packaging in order to ensure that no oxidation took place before starting the experiment.

From muesli I, only the rolled oats and wheat flakes were analysed. The water content of rolled oats and wheat flakes in this muesli was approximately 2%, and the oatmeal was accordingly dried to a similar water content before the experiment in order to be able to compare the stability of the two cereal products.

2.2. Design of experiment

2.2.1. General

The effects of the external factors, oxygen availability (oxygen concentration and the volume ratio between product and headspace (PH ratio)) and light, and the influence of oxygen permeability combined with different oxygen concentration (oxygen availability) on the oxidative stability were investigated in two experiments. The designs of both experiments are summarized in Table 1.

2.2.2. Storage experiment I: effects of external factors

Pork scratchings, peanuts, oatmeal, and muesli I were packed in a transparent plastic laminates (15 μm OPA-barrier/60 μm PE spec.; Ehrno Flexible A/S, Denmark) with an oxygen permeability of <1.5 ml/m^2/24 h/atm and a light transmission of 100% in the visible region. The volumes of the bags were 2000, 500, 800, and 2000 ml for pork scratchings, peanuts, oatmeal, and muesli I, respectively. Different amounts of products were added in order to obtain different volume ratios between product and headspace (PH ratio). Pork scratchings were packed in portions of 200 and 120 g, peanuts in portions of 244 and 163 g, oatmeal in portions of 223 and 120 g, and muesli I in portions of 842 and 433 g. The bags were packed on a Multivac C500 double-chamber machine (Multivac, Denmark). Oxygen was removed from the packed products by including oxygen absorbers. For pork scratchings and muesli I, absorbers with capacities of 100 ml (ZPT-100, AGELESS®, Japan) were used whereas peanuts and rolled oats only required 50 ml absorbers (ZPT-50, AGELESS®, Japan). Oxygen concentrations of 2% and 4% were obtained by using premixed gasses. The extent of oxidation during storage was monitored by following the contents of free radicals

Table 1
Design of experiments I and II

	Pork scratchings	Peanuts	Oatmeal	Muesli I	Muesli II
Exp I: external factors					
Oxygen concentration	0%, 4%, 21%	0%, 2%, 4%	0%, 4%, 21%	0%, 4%, 21%	
Product:headspace	1:2, 1:4	1:1, 1:2	1:1.7, 1:4	1:0.5, 1:2	
Light	0, 2900 lux	0, 2900 lux	0, 2900 lux	0, 2900 lux	
Time of analysis	2, 5, 8, 11, 15, 17, 19 and 23 weeks	3, 7, 11, 15, 19, 23 and 27 weeks	2, 5, 8, 12, 16, 19 and 24 weeks	3, 7, 12, 16, 19, 24, 36 and 52 weeks	
Exp II: packaging					
Oxygen permeability (ml/m^2/24 h/atm)	<2 (Pf, Ff), 100 (Sf)	<2 (Pf, EVOH)		<2 (OPA), 100 (PET)	<2 (OPA, OPET/PP), 100 (PET)
Oxygen concentration	<1%, 21%	<1%, 2%, 8%, 21%		<1%, 5%, 21%	<1%, 5%, 21%
Product:headspace	1:2	1:1		1:1	1:1.2
Light	0 lux	0 lux		2900 lux	0 lux
Time of analysis	3, 11, 13, 17, 18, 21 and 24 weeks	3, 11, 17, 18, 21, 24 and 26 weeks		4, 17 and 26 weeks	4, 17, 26, 34, 39, 42 and 52 weeks

[a] Further information of the abbreviations Pf, Ff, Sf, EVOH, OPA, PET and OPET/PP are given in Table 2.

and hexanal. The experiment was performed as (3×2×2) full factorial design-cubes at time points 1, 4, and 7 (plus 8 for muesli), whereas intermediate time points 2 and 3 plus 5 and 6 were as a mirrored set-up as fractional factorial designs.

2.2.3. Storage experiment II: effects of packaging

Pork scratchings, peanuts, and muesli I and II (with recommended general shelf lives by the producers of 16, 24, 36, and 36 weeks, respectively) were packed in flexible plastic materials with different oxygen permeabilities and the oxygen concentration was varied between <1% (nitrogen flushing) and 21% at the time of packaging by using premixed gasses, cf. Tables 1 and 2. For comparison, the products were also packed in their conventional packaging materials which, depending on the wavelength, allows between 0% and 60% transmission of visible light while the other materials transmit 100%. To identify leaky bags, all bags contained at the time of packaging, 15% CO_2. However, bags with a permeability of 100 ml/m^2/24 h/atm (Sf, PET) did not retain the CO_2 concentration over the whole storage period, due to a too high permeability. Even though Ff (OPP/M-OPP-Y) had a low oxygen permeability (<2 ml/m^2/24 h/atm), the bags could not be "sealed" completely as heat sealing of OPP with OPP resulted in peelable bags. The same accounted for pork scratchings packed in Sf and muesli II in OPET/PP. For each product, a sample packed with 0% O_2 in Pf (OPP/M-PET) or OPA and stored at 5 °C were intended for use as reference for the sensory evaluation. However, this control reference sample was compared to a freshly produced sample and if the stored reference had changed it was discarded.

Table 2
Packaging material and oxygen concentration applied in the packaging experiment (exp. II)[a]

Pork scratchings	Ff (OPP/M-OPP) <1%, 21%	Sf (OPP/M-OPP) <1%, 21%	Pf (OPP/M-OPET/PE) <1% (Ref), 21%
Peanuts	Pf (OPP/M-OPET/PE) <1% (Ref), 2%, 8%, 21%	EVOH PE/EVOH/PE <1%, 21%	
Muesli I	OPA (OPA/EVOH/OPA/PE) <1% (Ref), 5%, 21%	PET (PET/PE) 5%, 21%	CONV (PET/PE) 21%
Muesli II	OPA (OPA/EVOH/OPA/PE) <1 (Ref), 5, 21%	PET (PET/PE) 5, 21%	CONV (OPET/PP) 21%

[a] *Abbreviations*: Ff, pork scratching foil; Pf, peanut foil; Sf, snack foil; Ref, control reference sample for sensory evaluation stored at 5 °C; OPP: oriented polypropylene; M: metallised; OPET: oriented poly(ethylene terephthalate); OPA: oriented polyamide; EVOH: ethylene-vinyl alcohol; PET: poly(ethylene terephthalate); CONV: conventional packaging material.

The oxidative stability was evaluated by measuring the content of free radicals and hexanal and by a sensory evaluation.

2.3. Storage

All the samples were stored at 27 °C, and the light-exposed samples were stored under fluorescent light (Philips 'TL'D 36W/830, Holland) with a radian flux of 2900 lx. Throughout the storage periods the samples were randomly interchanged with two to four week intervals in order to minimize unequal light exposure. The products were withdrawn for analyses frequently during the storage period, (cf. Table 1). The samples were vacuum-packed and stored at 5 °C in darkness for a maximum of three days before analysis. Two identical bags were analyzed at each chemical/spectroscopic measurement, and two replicates were made. Four identical bags were used for the sensory evaluation and one assessment of each panellist was performed.

2.4. Gas composition

The gas composition of the headspace above the packed products was determined by using a calibrated Gaspace 2 gas analyzer (Systech Instruments Ltd., Thame, UK).

2.5. Sample preparation

Prior to chemical/spectroscopic analysis the products were homogenized for 10 s in a household coffee mill (Braun, Germany).

2.6. Lipid content

Lipids were extracted from the products with a chloroform/methanol (2:1) mixture according to Erickson (1994) with the following modifications: The homogenized product and the solvent were mixed with an Ultra Turrax for 15 s at 8000 rpm, and the solvent was evaporated using a vacuum.

2.7. Fatty acid composition

The fatty acid composition was determined according to Jart (1997) with the following modifications: 10 mg extracted oil was used; the samples where kept at 60 °C (water bath) for 40 min; prior to analysis on the gas chromatograph (5890 II from Hewlett–Packard Co., San Fernando, CA with a 25 m×0.20 mm×0.33 μm HP-FFAP 19091F-102 column from Agilent Technologies Inc., USA) the samples were evaporated and dissolved in 1.0 ml of pentane. The oven temperature programme and conditions were: 50 °C for 1 min; from 50 to 180 at 15 °C/min; from 180 to 220 at 5 °C/min; at 220 °C for 10 min. Column pressure was 136 kPa; 1 μl was injected, the split ratio was 1:10 and the flow was 1 ml/min. The results were reported as % fatty acid of the total content of detected fatty acids.

2.8. Tocopherol concentration

The concentrations of α-, δ-, and γ-tocopherols in muesli were determined according to Buttriss and Diplock (1994). The analysis was slightly modified as follows: 4.0 g (accurately weighed) muesli were homogenised with 20 ml 1.15% KCl using an Ultra Turrax (T-25, Janke and Kunkel) for 30 s at 13,500 rpm. 2.00 ml of the homogenate (accurately weighed) was transferred to a tube containing 200 μl saturated KOH and 2.00 ml 0.5% pyrogallol (Sigma–Aldrich, USA) in ethanol and mixed using a vortex mixer. After saponification and extraction, the samples were re-dissolved in 0.25 ml ethanol with 0.001% BHT and quantified by HPLC. For oatmeal, peanuts, and pork scratchings, 50 mg lipid (extracted according to Erickson (1994)) were diluted in 5.00 ml ethanol with 0.001% BHT, centrifuged at 2500 rpm for 5 min and quantified by HPLC. 20 μl was injected into the HPLC system and quantification was performed as described by Haugaard, Danielsen, and Bertelsen (2003). The tocopherol content was measured in duplicate and quantified by external standard curves for α-, δ-, and γ- tocopherol.

2.9. ESR spectroscopy

The relative free radical content was measured by electron spin resonance (ESR) spectroscopy. Approximately 0.9, 0.8, 0.7, 0.7 and 0.7 g (accurately weighed) of homogenized peanuts, pork scratchings, oatmeal, muesli I and muesli II, respectively, were transferred to cylindrical, thin-walled 702-PQ-7 clear-fused quartz (CFQ) tubes (o.d., 5 mm; Wilmad Glass Company Inc., Buena, NJ), which were gently tapped against the table in order to establish a dense and uniform packing. The column height was 6.3–6.8 cm for all the products and the density approximately 0.1 g/cm for all the products. The ESR measurements were performed using a JES FR30 Free Radical Monitor (JEOL, Tokyo, Japan) with the following parameters: sweep time 4 min, sweep width 7.5 mT, microwave power 4 mW, modulation width 0.1 mT and time constant 0.3 s. The amount of radicals was quantified as the height of the ESR signals relative to the signals from a built-in manganese standard and normalized by the density (g/cm) of sample in the ESR tube.

2.10. Hexanal content

The content of hexanal was measured by static headspace-GC analysis according to Jensen et al. (2001). An internal standard (2-heptanon, Sigma–Aldrich, USA) was added in a glass liner in order to avoid humidification of the sample.

2.11. Sensory evaluation

The sensory evaluation was performed each time samples from the packaging experiment were withdrawn for analyses by an 11-member trained sensory panel. Before each session, the control reference sample was compared to a freshly produced sample and, if the stored reference had changed, it was discarded. For each session approximately 50 g of pork scratchings, muesli I and II and 75 g of peanuts were placed in plastic containers (425 ml for pork scratchings and 200 ml for peanuts, muesli I and II) approved for sensory evaluation and the containers were tightly capped. To allow equilibration of volatiles in the headspace, the samples stood for 1 h prior to the evaluation. In order to calibrate the panel on odour and flavour descriptors a reference sample was served at each session. In each session, five samples were randomly distributed to each panellist. All the evaluations took place in individual taste panel booths. Between servings the panellists cleansed their palates with non-carbonated (tap) water (room temperature) and cucumber. Furthermore, they compensated for adaptation in their olfactory pathway by sniffing ground coffee beans.

Different odour and flavour descriptors were used for each product. The overall impression was evaluated for each product with the purpose of determining when a sample became unacceptable. This was evaluated on a 5-point scale where score 1 was very good, 3 acceptable and 5 completely unacceptable. Pork scratchings were evaluated by the rancid odour and flavour, peanuts by nutty and rancid odour and flavour, muesli I by sweet, honey, and rancid odour and flavour, and muesli II by sweet, vanilla, and rancid odour and flavour. The odour and flavour descriptors were evaluated on a 10-point scale where score 1 corresponded to a fresh product (like the control reference sample) and 10 to a very rancid product; e.g., for peanuts the score 1 corresponded to a strong nutty but not rancid odour/flavour, whereas score 10 corresponded to very rancid peanuts without any nutty odour/flavour.

2.12. Statistical analysis of data

Results from measurement of free radicals and hexanal were subjected to analysis of covariance using storage time as covariate (SAS version 6.12 software; SAS Institute Inc., Cary, NC, USA). Significant treatment effects were further classified by LSD ($p \leqslant 0.05$). The influence of panellists was analysed as a random effect in analyses of sensory results.

3. Results

3.1. General

Pork scratchings, peanuts, oatmeal, and muesli I and II are all products with low water activity, $a_w < 0.4$ (Table 3). The products have varying contents of fat, but the fatty acid composition of all the products are dominated by unsaturated fatty acids, with oleic acid as the most abundant. The concentrations of tocopherols (α-, δ- and γ-tocopherol) were quantified in order to evaluate the amount of potential antioxidants in the products that could influence the oxidative stability (Table 3). The contents of tocopherols in the five products were very different, with oatmeal having the lowest amount of tocopherols. Pork scratchings contained a high amount of α-tocopherol, whereas δ-tocopherol was predominant in peanuts.

The oxidative stability of pork scratchings, peanuts, oatmeal, muesli I and II were studied in two storage experiments. In the first experiment, the effects of oxygen availability (oxygen concentration and PH ratio) and light exposure on the stability were investigated, whereas the second storage experiment examined the effects of packaging with different materials and gas compositions (Table 1). The amounts of free radicals and hexanal formed in the products were determined in both

Table 3
Composition of pork scratchings, peanuts, oatmeal, muesli I and muesli II

	Water activity	Fat content (g/100 g)	Palmitic acid (%)	Stearic acid (%)	Oleic acid (%)	Linoleic acid (%)	Linolenic acid (%)	α-tocopherol (μg/g)	δ-tocopherol (μg/g)	γ-tocopherol (μg/g)
Pork scratchings	0.11	45	24	12	46	11	1	280	16	0
Peanuts	0.14	50	12	3	51	27	0	25	133	20
Oatmeal	<0.03	7	15	1	42	38	1	3	0	0
Muesli I	0.38	14	11	2	48	31	5	28	25	1
Muesli II	0.23	15	24	3	52	21	0	16	1	1

Table 4
Minimum, maximum and mean values of the chemical analyses during storage of dried products

	Experiment I			Experiment II		
	Minimum	Maximum	Mean	Minimum	Maximum	Mean
Pork scratchings						
Free radicals (arbitrary unit)	4.4	15.0	10.9	6.1	12.2	9.5
Hexanal (mg/kg)	0.0	394.3	36.6	0.0	43.8	3.7
Peanuts						
Free radicals (arbitrary unit)	8.2	13.1	10.5	13.1	19.3	15.3
Hexanal (mg/kg)	0.0	1.3	0.2	0.0	7.3	1.3
Oatmeal						
Free radicals (arbitrary unit)	5.5	43.2	19.6			
Hexanal (mg/kg)	0.0	134.0	22.6			
Muesli I						
Free radicals (arbitrary unit)	4.8	16.3	8.7	9.2	75.7	47.1
Hexanal (mg/kg)	0.0	55.5	5.2	0.9	141.9	20.5
Muesli II						
Free radicals (arbitrary unit)				6.0	8.7	7.2
Hexanal (mg/kg)				0.0	1.5	0.7

experiments in order to examine the degree of oxidation. The second storage experiment also combined evaluation of the sensory quality with studies of the oxidative stability. The range of contents of free radicals and hexanal is shown in Table 4. The experiment investigating the effect of packaging of muesli I in different materials was stopped after 26 weeks due to development of unacceptable sensory quality of the products. The descriptors used in the sensory evaluation included the characteristic odours and flavours of the products, e.g., sweet and honey odour and flavour in muesli I. In addition, the rancid odour and flavour were evaluated, along with the overall impression. Since the profiles of the different descriptors were similar to the results of the overall impression, the latter was used to describe the overall sensory changes during storage.

It was possible, in experiment I (experiment II for peanuts and muesli II), to assess the relative oxidative stabilities of the five products by following the oxygen concentration in the headspace during storage in darkness (Fig. 1). The atmosphere of packaging of the product was 21% oxygen at the time of packaging. Oatmeal and peanuts consumed all available oxygen in 8 and 11 weeks, respectively, whereas pork scratchings consumed negligible amounts of oxygen during the storage period of 23 weeks. Steady decreases in the oxygen concentrations were observed for muesli I and II, and there was still approximately 15% oxygen left after 52 weeks. The rates of oxygen consumption suggest that the oxidative stabilities of the products increase in the following order: Oatmeal < peanuts $\ll$ muesli I $\approx$ muesli II < pork scratchings.

The dry products were stored in packaging materials with different properties (Table 2). A steady increase in the oxygen concentration during storage, due to oxygen permeation, was observed when pork scratchings were packed with 1% oxygen in the headspace and in a gas-permeable material (OTR of 100 ml/m^2/24 h/atm), while the oxygen concentration was constant during the storage period when packaged with 21% oxygen in materials with both high and low gas permeabilities and stored in darkness (Fig. 2).

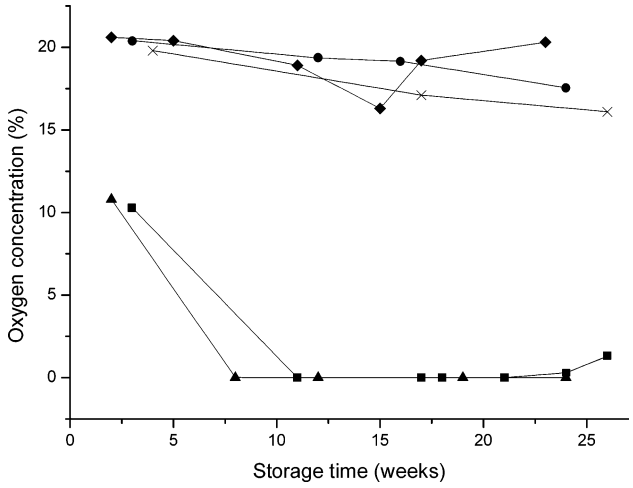

Fig. 1. Oxygen concentration in headspace for pork scratchings: (♦), peanuts; (■), oatmeal; (▲), muesli I; (●), muesli II; (×), packed with 21% oxygen and stored in darkness.

The tocopherol concentration decreased generally during storage of the products (data not shown). Muesli I, packed with 21% oxygen, decreased more than when packaged with 0% or 4% oxygen. Also, a clear effect of oxygen concentration on the tocopherol content was observed for peanuts, where the rate of disappearance of tocopherol increased with the oxygen concentration in the headspace.

Exposure of the dry products to light accelerated the consumption of oxygen considerably and hence the oxidation rate was concluded to increase as illustrated for pork scratchings in Fig. 2. Storage of pork scratchings,

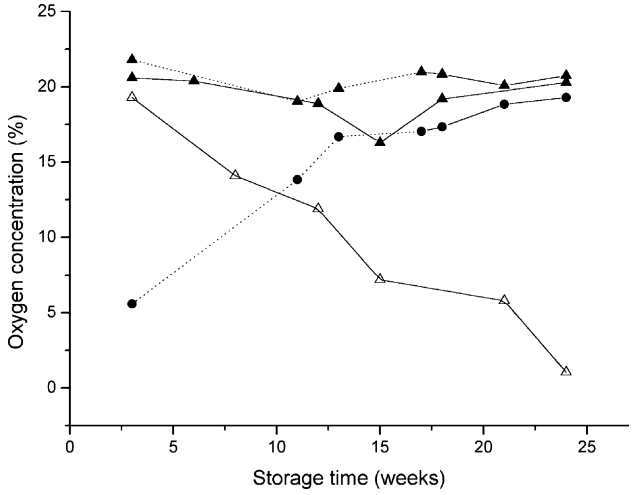

Fig. 2. Oxygen concentration in headspace for pork scratchings packed with <1 (●) or 21% oxygen (▲). Open and solid symbols correspond to storage in light and darkness, respectively. Solid (—) and dotted (·····) lines indicate packaging in material with low (<1.5 ml/m^2/24 h/atm) and high OTR (100 ml/m^2/24 h/atm; Sf), respectively.

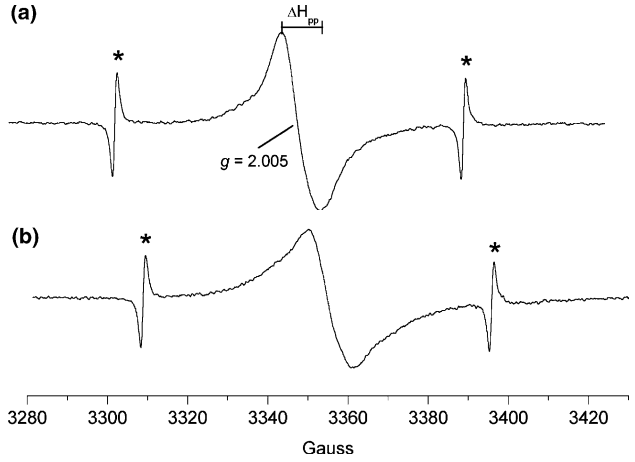

Fig. 3. ESR spectra for pork scratchings: (a); peanuts (b). The g value and the peak-to-peak width (ΔH_{pp}) are marked. The signals marked by asterisks (*) are the built-in manganese standard.

oatmeal, and muesli I under light also led to a decrease in the concentrations of tocopherols (especially α-tocopherol) as compared to samples stored in darkness (results not shown).

All products gave nearly identical ESR spectra, consisting of a single broad line with $g = 2.005$ (Fig. 3 and Table 5). The width of the ESR signals, measured as the peak-to-peak line width (ΔH_{pp}) was dependent on the type of product, and the width decreased slightly during the storage periods for all products (Table 5).

The amounts of radicals in the products were measured as the intensity of the ESR signals and quantified as the peak-to-peak heights. At the beginning of the storage experiments, all products contained radicals, whereas no hexanal was detected in the products, except

Table 5
ΔH_{pp} and g values for electron resonance spectra of the different dried products

	g	$\Delta H_{pp,\ day\ 0}$ (gauss)	$\Delta H_{pp,\ end}$ (gauss)
Pork scratchings	2.005	10.64	10.30
Peanuts	2.005	11.68	11.44
Oatmeal	2.005	8.65	7.43
Muesli I	2.005	8.19	7.81
Muesli II	2.005	10.67	9.26

for oatmeal, which apparently developed hexanal during the initial drying process.

3.2. Peanuts

Generally, the amount of both free radicals and hexanal increased with storage time (Fig. 4 and Tables 6 and 7). Peanuts packed with 4% oxygen obtained a higher content of free radicals than after packaging with 0%

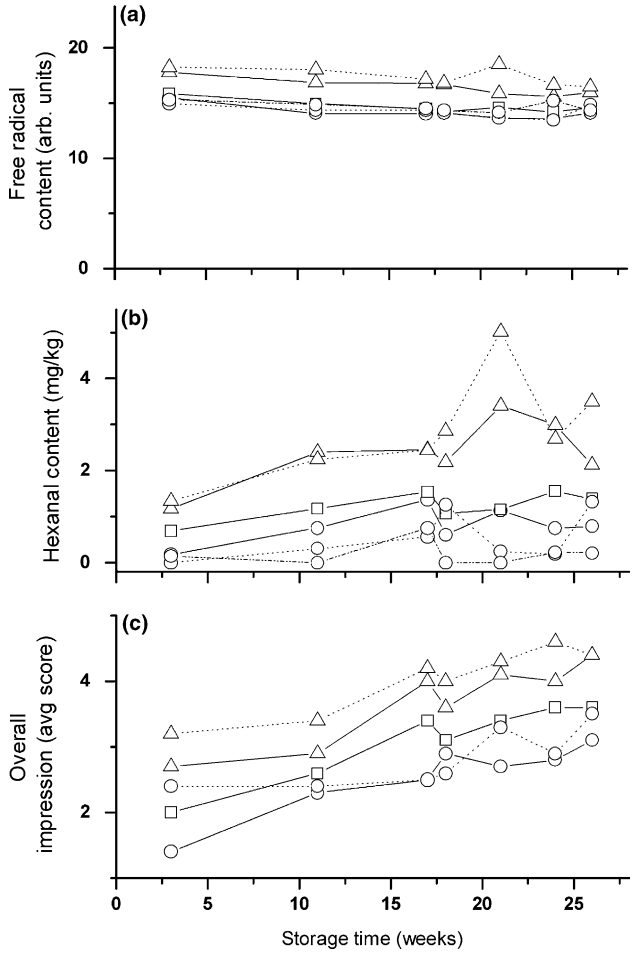

Fig. 4. Amount of: (a) free radicals (relative); (b) hexanal (mg/kg); (c) overall sensory impression (average score) in peanuts packed in Pf (–), Ref (-·-·-) or EVOH (·····) with <1 (○); 8 (□) or 21% (△) oxygen.

Table 6
Significant effects observed by the statistical analysis of the free radical content in dry products during storage[a]

Product	Experiment I				Experiment II
	Systematic variation (%)				
	Oxygen availability	Light	Oxygen availability	Light	Oxygen availability
Pork scratchings	85	15	• Increased oxygen conc. resulted in increased free radical conc.	• Increased free radical content in light exposed vs. darks stored samples (except for 0% oxygen)	• Packaging in high OTR materials or with 21% oxygen resulted in higher free radical content of pork scratchings
Peanuts	36	64	• Generally highest free radical content for peanuts packed with 4% compared to 0% oxygen • Peanuts packed with 0% oxygen and PH of 1:1 obtained a lower free radical content than packaging with 2% and 4% oxygen (when comparing light and dark samples separately)	• Increased free radical content in light exposed vs. dark stored peanuts	• Packaging with 21% oxygen resulted in higher free radical content than packaging with $\leqslant$8% oxygen (no difference in free radical content between $\leqslant$8% oxygen and reference was observed)
Oatmeal	22	78	• No clear effect of oxygen availability	• Increased free radical content in light exposed vs. dark stored oatmeal	
Muesli I	12	88	• Increased free radical content for muesli packed with PH ratio 1:2 compared to 1:0.5 (in light after 16 weeks) • Increased oxygen content resulted in increased free radical conc. (in darkness; after 16 weeks)	• Increased free radical content in light exposed vs. dark stored muesli	• Muesli packed in PET obtained a higher free radical content than packaging in CONV or reference (also higher than OPA after 26 weeks) • Higher free radical content in muesli packed with 21% than 5% oxygen (after 17 weeks for PET, and 26 weeks for OPA)
Muesli II					• Minor difference between muesli packed in OPA with 5% oxygen and the other packaging conditions • Lowest free radical content in reference

[a] Abbreviations: OTR, oxygen transmission rate (ml/m^2/24 h/atm); PH ratio, product-to-headspace ratio; OPA, oriented polyamide; PET, poly(ethylene terephthalate); CONV, conventional material (PET with light barrier).

oxygen (experiment I), whereas no difference was observed between packaging of peanuts with $\leqslant$8% oxygen and the reference sample stored at 5 °C, either for free radicals or hexanal content (experiment II, Fig. 4).

The oxidative stabilities of the products were quantified sensorially as the time up to which the score of the products was acceptable, i.e. an overall impression score below or equal to 3 (Table 8). The sensory quality (Fig. 4c) was acceptable for 24–26 weeks for peanuts packed with <1% or 2% oxygen, whereas packaging of peanuts with 8% oxygen reduced the time of acceptance to 18 weeks, even though the hexanal content in these samples did not differ (Table 8). The results suggest that peanuts with free radical contents of 13–14 on the relative scale and hexanal contents above 1 mg/kg are sensorially unacceptable, but further investigations are required.

Exposure of peanuts to light resulted in accelerated formation of both free radicals and hexanal. The significant effects obtained by statistical analysis of all the data in experiment I for the content of free radicals and hexanal are outlined in Tables 6 and 7. The greatest systematic variation in the formation of radicals in peanuts was ascribed to the light exposure, whereas the development of hexanal was mainly affected by the oxygen availability. Lowering the storage temperature from 21 °C to 5 °C did not affect either the oxidative stability or the sensory quality of peanuts.

3.3. Pork scratchings

The amount of radicals and hexanal increased during the storage period for pork scratchings. An

Table 7
Significant effects observed by the statistical analysis of the hexanal content in dry products during storage[a]

Product	Experiment I				Experiment II
	Systematic variation (%)				
	Oxygen availability	Light	Oxygen availability	Light	Oxygen availability
Pork scratchings	28	72	• Increased oxygen conc. resulted in increased hexanal content (for samples stored in darkness)	• Increased hexanal content in light exposed vs. darks stored samples (very pronounced)	• No clear effect of oxygen due to large biological variation but tendency to higher hexanal content in pork scratchings packed with 21% oxygen
Peanuts[b]	84	16	• For each oxygen conc. a higher content of hexanal was observed for peanuts packed with high than low PH ratio	• Increased hexanal content in light exposed vs. dark stored peanuts (after week 15)	• Highest hexanal content in peanuts packed with 21% oxygen • No difference between $\leqslant$ 8% oxygen and reference sample
Oatmeal	100	0	• Increased hexanal content in oatmeal with 21% compared to 0% oxygen	• No significant effect of light	
Muesli I	61	39	• Increased hexanal content in muesli packed with 21% oxygen and stored in light	• Increased hexanal content in light exposed vs. dark stored muesli (only for 21% oxygen)	• Muesli packed in PET obtain higher hexanal content than packaging in OPA
Muesli II					• Higher hexanal content in muesli packages with 21% than 5% oxygen (after week 17 for OPA and 26 weeks for PET) • Minor oxidation in reference (stored at 5 °C) • Light effect as higher hexanal content was observed for muesli packed in PET than in CONV • Hexanal content significantly lowest in reference sample • Muesli packed in OPA with 21% oxygen obtained the same hexanal content as muesli packed in PET (both 5 and 21% oxygen) and CONV

[a] *Abbreviations*: PH ratio, product-to-headspace ratio; OPA, oriented polyamide; PET, poly(ethylene terephthalate); CONV, conventional material (PET with light barrier).
[b] Due to minor development of hexanal in peanuts in experiment I the mentioned effects are only trends.

Table 8
Acceptability time determined by sensory evaluation and the content of free radicals and hexanal at the end of the acceptability time[a]

	OTR ml/(m^2 24 h atm)	Oxygen concentration (%)	Acceptability time (weeks)	Free radical content (arbitary units)	Hexanal content (mg/kg)
Pork scratchings					
Ff-<1	<2	<1	17	9	5
Ff-21	<2	21	11-13	10-11	2-7
Sf-<1	100	<1	21	11	1
Sf-21	100	21	3-11	7-11	1-12
Ref-21	<2	21	13	11	8
Peanuts					
Pf-2	<2	2	26	14	0.8
Pf-8	<2	8	18	14	1.1
Pf-21	<2	21	11	17	2.4
EVOH-<1	<2	<1	24	13	0.2
EVOH-21	<2	21	3	18	1.3
Muesli II					
OPA-5	<2	5	34	7	1
OPA-21	<2	21	34	8	1
PET-5	100	5	34	8	1
PET-21	100	21	34	8	1
CONV-21	100	21	34	8	1

[a] Please refer to Table 2 for abbreviations.

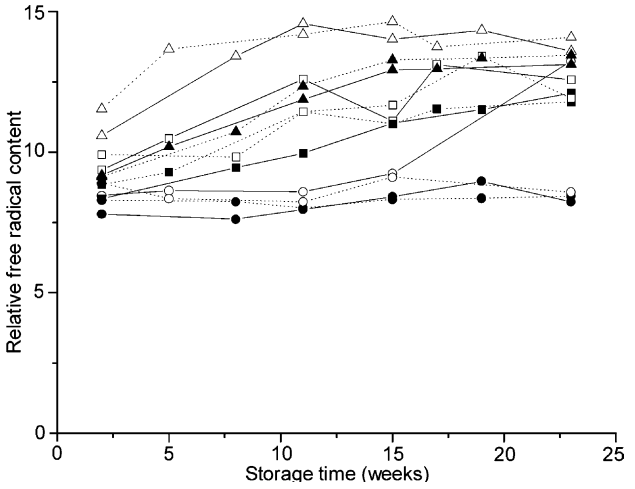

Fig. 5. Amount of free radicals in pork scratchings determined by electron spin resonance (ESR) spectroscopy. Open and solid symbols correspond to storage in light and darkness, respectively. Solid (—) and dotted (. . .. . .) lines indicate product to headspace ratio of 1:2 and 1:4, respectively, of pork scratchings packed with 0% (○), 4% (□) and 21% (△) oxygen.

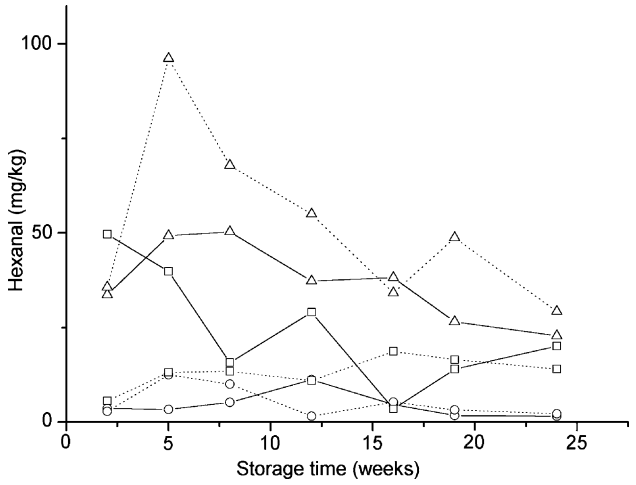

Fig. 6. Amount of hexanal (mg/kg; average of light and darkness) in oatmeal determined by headspace-GC. Solid (—) and dotted (· · · · · ·) lines indicate product to headspace ratios of 1:2 and 1:4, respectively, of oatmeal packed with 0 (○), 4 (□) and 21% (△) oxygen.

increase in the content of free radicals was observed during the first 10 weeks of storage, followed by an almost constant level during the rest of the storage period for most of the samples (Fig. 5). An increased formation of hexanal with increasing oxygen concentration was noted for pork scratchings stored in darkness (experiment I). Large variation in the hexanal content was observed for pork scratchings stored in light, which, however, most likely was caused by biological variations between the analyzed samples. Light had a strong influence on the formation of hexanal in pork scratchings (72%), which was more important than the influence of oxygen (Table 7). The opposite was the case for the content of free radicals (Table 6). In addition, the formation of free radicals was inhibited by lowering the storage temperature to 5 °C (reference sample).

Pork scratchings packed with <1% oxygen had an acceptable quality until week 17 or 21, depending on the packaging material (Table 8). The sensory quality of pork scratchings packed with 21% oxygen in high OTR material (100 ml/m^2/24 h/atm) became unacceptable between weeks 3 and 11 (no evaluation was performed between these weeks). Packaging with <1 oxygen in the same material prolonged the acceptability time to 21 weeks. On the contrary, pork scratchings packed in a material with lower OTR (<2 ml/m^2/24 h/atm) containing <1% oxygen were only acceptable until week 17. These contrasting results made it difficult to define a limiting content of free radicals or hexanal, at which the sensory quality was still acceptable.

3.4. Oatmeal

Generally the amount of hexanal that was detected in the headspace above the products increased with storage time, but for oatmeal a decrease was observed (Fig. 6). This was probable due to the fact that the oatmeal was strongly oxidized after 2 weeks, as was indicated by the development of a rancid smell after only 2 weeks of storage. The observed decrease could be caused by decomposition of hexanal to other volatiles. The formation of free radicals in oatmeal already reached a maximum after 2 weeks of storage, which was followed by a constant level during the rest of the storage period.

No clear effect of oxygen availability was observed for the content of free radicals and the statistical analysis revealed that the greatest systematic variation in the amount of free radicals in oatmeal could be ascribed to light (Table 6). This was in contrast to the results of the hexanal content, where light had no influence on the amount of hexanal (Table 7).

It has been reported that the ratio between unsaturated and saturated fatty acids in oat flours decreases during storage, indicating decomposition of the unsaturated fatty acids due to oxidation (Molteberg et al., 1996a). This was not confirmed in the present study, where the fatty acid compositions of oatmeal and the other dry products were constant in the two storage experiments, which is in agreement with results reported by Bekbölet (1990) and Sattar, deMan, and Alexander (1976).

3.5. Muesli

An increased degree of oxidation with increased oxygen availability was generally observed for both mueslis

I and II (Tables 6 and 7). Additionally, exposure of muesli to light also resulted in increased formation of radicals and hexanal. Packaging of muesli I in the transparent material PET revealed a higher content of hexanal than the conventional packaging material (CONV), which only allows 0–60% transmission of visible light, depending on the wavelength. The effect of light was estabilished for the greatest systematic variation on the formation of radicals, whereas the oxygen availability mainly affected the development of hexanal. No difference in the sensory quality was observed for muesli I and II packed under different conditions. Muesli I packed in OPA with 5% oxygen and in the conventional packaging material gave the best quality and was not evaluated as rancid, but the quality was still not acceptable. Lowering the temperature decreased the formation of free radicals and hexanal in the two muesli products. A large difference in the formation of free radicals and hexanal was observed between the two muesli products, as muesli I in contrast to muesli II was more oxidation-labile and hence developed higher amounts of free radicals and hexanal (Table 4). The minor formation of free radicals and hexanal in muesli II was also reflected by the long shelf life of 34 weeks. It was not possible to define the levels of hexanal and radicals that corresponded to the sensorially acceptable muesli II, due to the low levels of radicals and hexanal formed.

Especially in muesli, but also in pork scratchings, the combination of light and high oxygen availability resulted in marked oxidation. This effect was confirmed by storage of extruded oats as the effect of light was found to be most predominant when combined with high levels of oxygen and high temperature (Larsen, Magnus, & Wicklund, 2003).

The reference samples used for sensory evaluation and stored at 5 °C gave a lower content of free radicals and hexanal for the two muesli products, and a lower free radical content in pork scratchings. In peanuts, no difference between storage at 5 and 21 °C was observed.

4. Discussion

The ability of free radicals, hexanal and sensory evaluation to describe the influence of light and oxygen availability (including oxygen concentration, product-to-headspace ratio and OTR) on the oxidative stability depends to some extent on the individual product. In the investigated products, enzymes are inactivated by heat treatment (e.g. steaming, blanching) during manufacture and hence the primary modes of chemical deterioration are autoxidation and photosensitised oxidation.

The oxygen consumption of the products during storage revealed that pork scratchings were the most and oatmeal was the least oxidatively stable product, even though oatmeal only contained 7% fat compared to 45% fat in pork scratchings. However, oatmeal had the highest percentage of linoleic acid and lowest content of tocopherols, whereas the opposite was the case for pork scratchings. These observed differences were only partly reflected in the free radical and hexanal contents. Pork scratchings developed almost three times as much hexanal as oatmeal, whereas the content of free radicals was three times higher in oatmeal (Table 4). Contrasting results were obtained for pork scratchings, as the formation of hexanal was nine times less in experiment II than in experiment I, presumable due to a large biological variation, as pork scratchings in two bags, stored identically, were found to be significantly different. A high oxygen consumption was also noted in peanuts, but the development of hexanal and free radicals was only small. These observations indicate that several factors affect lipid oxidation in dry products; some examples are contents of fat, fatty acids, water and anti- and pro-oxidants and the surface structure and porosity of the products.

Statistical analysis of the data from experiment I revealed that the greatest systematic variation in the amount of free radicals in peanuts, oatmeal and muesli, could be ascribed to light (Table 6), whereas the oxygen availability had the largest influence on the formation of hexanal (Table 7). The opposite was the case for pork scratchings. These results indicate that ESR primarily determines oxidation that takes place at the surface of the product, whereas hexanal, which is developed later in the lipid oxidation processes, depends on the presence of oxygen.

The influence of oxygen availability and light on pork scratchings was best reflected by the free radicals as a large variation between the individual scratchings was observed in the hexanal content. In peanuts, only little hexanal was formed in experiment I and hence, the radical concentration gave a better description of the oxidative stability. However, in experiment II, both the free radicals and the hexanal content could describe the oxidative changes. In mueslis I and II, the development of both radicals and hexanal revealed separation of the samples according to oxygen availability and light exposure, as was expected from the experimental design. The free radicals separated oatmeal stored in light and darkness, whereas hexanal showed an effect of oxygen availability. The observed differences indicate that the contents of free radicals and hexanal complement each other in describing the impact of oxygen and light on the stabilities of the different products.

The sensory evaluations to some extent supported the oxidative changes noted by measurement of free radicals and hexanal and reflected the oxidative stability, especially in peanuts and muesli I. No clear defined levels of radicals and hexanal that corresponded to a sensorially acceptable product could be defined for pork scratchings or mueslis I and II. In peanuts, a hexanal

content of 1 mg/kg and a relative radical content of 13-14 may be the limits for acceptability, but further investigations are required.

Generally, increased oxygen availability resulted in increased lipid oxidation. In peanuts and oatmeal, the highest degree of oxidation was observed in packages containing 21% oxygen and only minor differences were noted at lower oxygen concentrations. These results are in accordance with the literature as vacuum packaging, flushing with nitrogen or carbon dioxide or use of oxygen absorbers in combination with a material with low OTR inhibited the oxidation of peanuts and maintained the initial good quality for a longer period (Holaday, Pearson, & Slay, 1979; Sheikh, Hirata, & Ishitani, 1985). This improvement of packaging with carbon dioxide was also seen for pecans, and the shelf life was extended from 2–4 to 27 weeks. The nuts absorbed the carbon dioxide, which resulted in a package with vacuum (Holaday et al., 1979). Investigations performed in the 1980s showed, as expected, that a material with a high barrier towards oxygen was necessary in order to obtain high quality peanuts. Using a packaging material with a low OTR (<2 ml/m^2/24 h/atm) resulted in a better quality of peanuts (Sheikh et al., 1985), walnuts (Jensen, Sørensen, Brockhoff, & Bertelsen, 2003) or cashew nuts (Lima, da Silva, & Goncalves, 1999) than using a material with a high OTR (>100 ml/m^2/24 h/atm).

One aim of this investigation was to compare the stability of oatmeal and muesli I. According to the relative free radical content, both products were affected by storage in light but the hexanal content revealed that only muesli was influenced by light, probable due to the fact that the lipid in oatmeal was completely oxidized after 2 weeks. In addition, the hexanal content for oatmeal decreased with time, whereas it increased for the other products. For both oatmeal and muesli (stored in darkness), the influence of the variation in oxygen availability on oxidative stability was depicted by the hexanal content, as a higher availability resulted in higher hexanal content, i.e. the hexanal content seemed to be a good indicator of the degree of oxidation. Packaging of muesli in different materials showed, as before, an effect of storage in light and oxygen availability, as a low availability resulted in a better quality. Comparison of the level of oxidation in experiment I revealed a higher content of both free radicals and hexanal in oatmeal than muesli.

According to Burri (1994) and Labuza (1982) it is not common practice to pack dried foods in materials with low OTR and without oxygen, as the effect is insignificant and the expenses too high. One of the advantages noted of packaging rolled oats in packaging materials with permeability is that the rancid odours can escape as soon as they are formed (Labuza, 1982). This was further confirmed by Matz et al. (1955), who found that rancid odours accumulated if wheat flakes were stored in an air-tight container. Therefore this product was usually sold in breather boxes without inner or outer linings. When packaged as described, the product was just as stable as any other prepared cereal, except that moisture absorption could take place much more readily, resulting in a reduction in shelf life of these products (Matz, 1991). However, extruded oats stored in PE bags developed rancid odours within 5 weeks at 22 °C, but by reducing the temperature to 4 °C, the aroma did not change within 1 year (Guth & Grosch, 1994). Another investigation on extruded oats showed a high degree of rancidity within 3 months when packed in medium (PA/PE laminate) or high OTR packages and stored at 23 °C in light. On the other hand, no rancidity was detected in extruded oats packed in a material with low OTR (EVOH laminate) with or without oxygen absorber (Larsen et al., 2003). In addition, an improved storage stability was found for flaked oat cereals packed in materials with low oxygen permeability with an oxygen absorber compared to packaging without an oxygen absorber (Sakamaki, Gray, & Harte, 1988; Robertson, 1993). This is in accordance with our present results, where high oxygen availability resulted in high degree of oxidation.

A clear effect of light exposure was observed for muesli in the first experiment and in the second experiment. Storage in the conventional material (PET with 0–60% transmission of visible light) resulted in a better quality than storage in the more transparent PET, demonstrating that exclusion of some wavelengths was sufficient to reduce oxidation. Storage of peanuts, pork scratchings and oatmeal in light also accelerated the rate of oxidation. Improvement of the light barrier of the packaging material could, accordingly, protect the product against light and thereby delay the lipid oxidation. However, the effect of the light barrier will depend on the colour of the pigment added to the packaging material (Firman, 1973). Incorporation of UV absorbers is also a possibility, but those in common use normally absorb below 400 nm and allow visible light to be transmitted. As visible light also had an effect on the lipid oxidation in the actual products, the packaging materials will have to exclude both ultraviolet radiation and visible light (Lennersten & Lingnert, 1998). Incorporation of pigments as light barrier was found to extend the shelf life of potato chips from 7 days to 10 weeks (Kubiak, Austin, & Lindsay, 1982), a result which supports the previous finding of Lennersten and Lingnert (1998). Packaging material with a high light barrier (e.g., metallized foil) was thus found to extend the shelf life of potato chips from 7 days to 5 months compared to a low light barrier (Lennersten & Lingnert, 1998). Light exposure of various nuts (almonds, peanuts, pinenuts and walnuts) stored in amber-coloured glass bottles did not affect the storage stability and has been found

comparable to dark storage, in contrast to storage in clear polyethylene (PE), which did not protect the nuts from light-induced lipid oxidation measured as peroxide value (Sattar, Mohammad, Saleem, Jan, & Ahmad, 1990). Walnuts were similarly found to be light sensitive, as exposure to light increased the degree of oxidation, thereby resulting in a significantly reduced sensory quality (Jensen et al., 2001). Comparison of packaging materials with different light transmissions (3%, 47%, and 87% transmittance) showed, for pecan nuts, a dose-response relationship, where a higher transmission results in larger colour changes (Heaton & Shewfelt, 1976).

In conclusion, different dried food products show different sensitivities to oxygen and light. Both ESR spectroscopy and headspace-GC have been shown to hold the potential of being used for prediction of shelf-life of this type of product but should in each case be calibrated against a sensory evaluation depending on the product.

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