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Impact of storage of oat bran flour and dispersion on lipid oxidation, odour profile, and oxidative degradation of beta-glucan

Outi Mattila [a], Natalia Rosa-Sibakov [a], Hanna Ahola [a,b], Anna-Maija Lampi [b], Oskar Laaksonen [c], Martina Lille [a], Sari A. Hakanen [c], Vieno Piironen [b], Kaisa M. Linderborg [c], Emilia Nordlund [a,*]

[a] VTT Technical Research Centre of Finland Ltd, P.O. Box 1000, VTT, FI-02044, Finland
[b] University of Helsinki, Department of Food and Nutrition, P.O. Box 66, University of Helsinki, FI-00014, Finland
[c] Food Sciences, Department of Life Technologies, University of Turku, Itäinen pitkäkatu 4 C, 20520, Turku, Finland

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ABSTRACT

Lipid oxidation induced oxidative degradation of beta-glucan could lead to technological, sensory, and nutritional challenges during food storage. This study aimed to explore the impact of storage of oat bran concentrate (OBC) flours and aqueous oat dispersions on lipid, beta-glucan, and odour properties. Formation of free fatty acids was observed in non-heat treated OBC flour during storage (12 weeks at 4–40 °C), indicating the activity of endogenous hydrolytic enzymes, while lipid oxidation was highest in heat-treated (HT) OBC flour. The molecular weight of beta-glucan remained the same, indicating that lipid oxidation did not induce oxidative degradation of beta-glucan in the flours. Fresh and 8 weeks at 40 °C stored HT OBC flours were used for the preparation of aqueous oat dispersions with and without 3 % rapeseed oil. No difference in the odour characteristics of dispersions prepared from fresh and stored OBC flours was found. However, 4 weeks at 22 °C storage of HT OBC dispersions had a significant effect on the odour in comparison to the freshly prepared dispersions. During the storage of the dispersions, oxidation of lipids was mild and degradation of beta-glucan was not detected. This study provides new insights into the impact of storage on dry and aqueous oat products.

1. Introduction

Palatable flavour and nutritionally beneficial composition make oats an appealing ingredient for the food industry. However, their use is limited by their tendency to become rancid and form a bitter off-flavour during processing and storage (Heiniö et al., 2002). Oxidation and hydrolysis of oat lipids are considered to have a major role in the development of rancidity. Oats are high in lipase that catalyses the hydrolysis of lipids liberating free fatty acids (FFA). Thus, heat treatment is routine in oat processing to inactivate the endogenous enzymes. Heat treatment is also required for the formation of the typical oat flavour, since oats lack flavour as collected from the field (Zhou et al., 1999). Yet, heat in general is known to promote oxidation. Oxidation of unsaturated oat lipids initiates a complex series of reactions leading to the formation of secondary oxidation products, such as hexanal, which is commonly used as an indicator of oxidative rancidity (Lehto et al., 2003; Puganen et al., 2024). From lipids, oxidation extends to other compound classes.

The effects of lipid degradation also seem to cause collateral damage. Wang et al. (2016) discovered that lipid oxidation induced oxidative degradation of beta-glucan in an oil-in-water emulsion system, leading to decreased molecular weight (MW) of beta-glucan and reduced viscosity of the emulsion. The physiological and technological functionality of oat beta-glucan has been linked to its ability to form viscous solutions, which is affected by the solubility, concentration, and MW of beta-glucan (Hakkola et al., 2021). The possible degradation of beta-glucan by oxidized lipids might pose a problem in foods with oat ingredients high in beta-glucan, as both the food matrix and the oat ingredients in themselves contain potentially degradable lipids. In aqueous foods, the degradation of beta-glucans can lead to technological challenges, changes in the sensory properties as well as nutritional

Abbreviations: OBC, oat bran concentrate; HT, heat-treated; NHT, non-heat-treated; HTD, heat-treated defatted; F-HT, fresh heat-treated; S-HT, stored heat-treated; Mw, molecular weight.

* Corresponding author.
E-mail address: emilia.nordlund@vtt.fi (E. Nordlund).

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losses. In the literature, the effect of beta-glucan on sensory properties in liquid products has been mainly focused on textural properties or flavour (Lyly et al., 2003, 2004; Chakraborty et al., 2019). In studies focusing on odour, beta-glucan has often been mixed with juices (Lyly et al., 2003) or other fruit concentrates (Mielby et al., 2016), whereas the potential sensory changes in beta-glucan dispersion alone have not been studied.

The previous studies regarding lipid oxidation induced oxidative degradation of beta-glucan were made in oil-in-water emulsion model containing tocopherol-stripped rapeseed oil as fresh or oxidized together with beta-glucan and iron catalyst (Wang et al., 2016). Lipid oxidation level was thus quite high. Therefore, it is important to investigate whether the oxidative degradation of beta-glucan happens at lower oxidation level and in more complex food systems such as dry oat materials or in aqueous foods containing oat. The aim of the current work was to study the impact of the storage of dry oat bran ingredients and aqueous oat dispersions on changes in their lipids, possible oxidative degradation of beta-glucan, and the potential effect of these changes on the perceived odour. To our understanding this is the first study investigating odour differences in oat beta-glucan beverage models (aqueous dispersions) during storage. The hypothesis was that in the presence of oxidized lipids, beta-glucan would undergo oxidative degradation leading to decreased MW and reduced viscosity, and that the storage-induced changes could be detected from the odour by a sensory panel. To investigate the hypothesis, different types of oat bran concentrate (OBC) flours (non-heat treated, heat-treated or heat-treated and defatted) were prepared, aiming to obtain different types of lipid degradation (oxidation and enzymatic hydrolysis) during storage.

2. Materials and methods

The schematic presentation of the study set up is presented in Fig. 1.

2.1. Preparation and storage of oat bran concentrate (OBC) flours

Dehulled oat kernels (produced without heat treatment) and oat bran (produced from industrially heat treated dehulled oat kernels), were obtained from Raisio Plc, Finland. Three different OBC flours were prepared: Non-heat treated (NHT) OBC, heat treated (HT) OBC and heat treated defatted (HTD) OBC flour. The production procedures were designed so that all the OBC flours would have approximately the same median particle size and beta-glucan content.

To prepare the NHT OBC flour, dehulled oat kernels (produced without heat treatment) were milled twice with a Hosokawa Alpine 100UPZ pin disc mill (17800 rpm) and air-classified using a Minisplit Air Classifier (British Rema Manufacturing Company Ltd., UK) with classifier wheel speed of 2900 rpm and air flow of 220 m3/h to separate a fine and a coarse (bran) fraction. The coarse fraction was further milled twice as above and air classified (4500 rpm, 220 m3/h) to obtain NHT OBC in the coarse fraction. To prepare the HT OBC flour, regular oat bran produced from heat-treated oat kernels was milled twice as above and air-classified (3500 rpm, 220 m3/h) to obtain HT OBC in the coarse fraction. To prepare the HTD OBC flour, regular oat bran produced from heat-treated oat kernels was defatted with supercritical carbon dioxide using a Nova Swiss extraction vessel (Nova Werke AG, Effretikon, Switzerland) with a compressor Chematur Ecoplanning (Chematur Engineering Ltd., Pori, Finland), extraction pressure was 295–305 bar and the temperature in extraction container was 4 °C and in separation container 48 °C. Milling or air classification was not required for the HTD OBC flour because it already had similar beta-glucan content and median particle size as the two other OBC flours.

For the storage study, the three OBC flours were stored at 4, 22 and 40 °C in dark in closed glass bottles up to 12 weeks. All samples (freshly prepared OBC flours and the OBC flours after their storage at different temperatures) were stored airtight at −20 °C until their use. However, volatile compounds were analysed from the samples immediately.

2.2. Preparation and storage of OBC dispersions

Fresh (F) and stored (S) (8 weeks at 40 °C) HT OBC flours were used for the preparation of oat dispersions with 1 % beta-glucan and with or without 3 % rapeseed oil. Oil content of 3 % was used to mimic commercial dairy alternatives. HT OBC flour was mixed with water (1 g beta-glucan/97 g dispersion based on the beta-glucan content of the HT OBC flour; it was assumed that the total beta-glucan content of the flour did not change during the storage) by Ultra Turrax (1 min, 10 000 rpm), followed by blade mixing (100 rpm) at 85 °C for 1 h in a mashing bath (Bender & Hobein, Munich, Germany). After that, water or rapeseed oil (Bunge, Finland) was added to a concentration of 3 % followed by homogenization with Ultra Turrax (3 min, 16 000 rpm). Dispersions were autoclaved (121 °C, 5 min) (to prevent their microbiological spoilage during the storage) and stored in closed glass bottles in dark at 22 °C for 0, 1 and 4 weeks. After that the bottles were stored at −20 °C until the analyses except for the viscosity measurement and volatile analysis, which were done right after the preparation/storage of the sample.

2.3. Microbiological quality analysis

The microbiological quality of the oat dispersions was analysed by colony count technique by plating 1 ml of the sample to appropriate

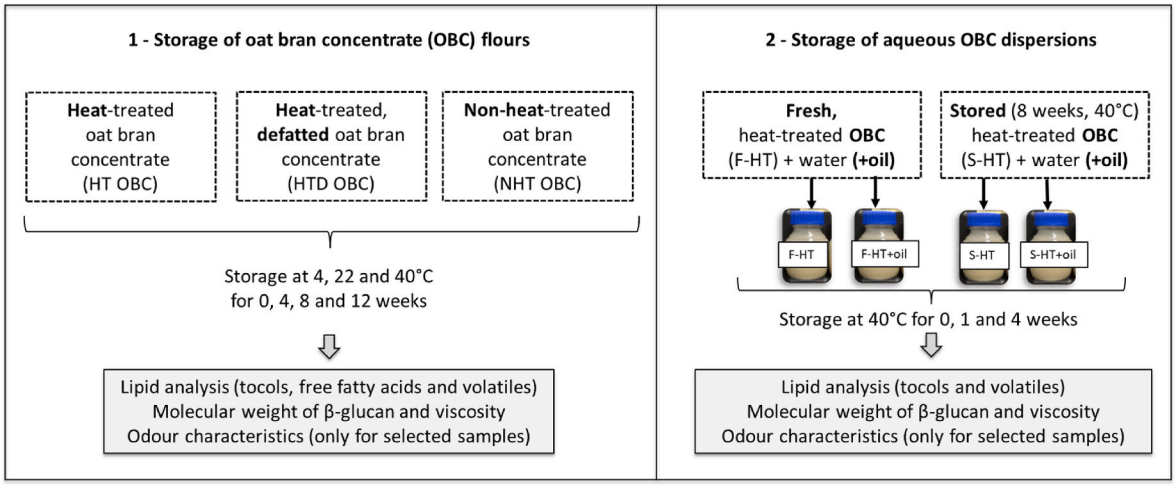

Fig. 1. Schematic presentation of the oat samples studied and analyses performed.

growth media. In addition, 1 ml of the sample was homogenized with 9 ml of peptone saline and plated on the agar.

Plate count agar (PCA, Difco, Bordeaux, France) was used to determine viable counts of aerobic heterotrophic bacteria and spore-forming bacteria (heat treatment 80 °C, 10 min) and incubated at 30 °C for 3 days. Preliminary *Bacillus cereus* was detected with Mannitol Egg Yolk Polymyxin (MYP) agar (Oxoid, Basingstoke, England) and incubated at 37 °C for 24 h. Coliformic bacteria were detected with Chromocult coliform agar (Merck Millipore, Darmstadt, Germany) incubated at 37 °C for 24 h. Sulphite reducing clostridia were determined with Sulphite Iron agar in conditions anaerobic (BioLab, Tampere, Finland, Anaerobic conditions (N_2 85 %, CO_2 5 %, and H_2 10 %) were generated with Anoxomat (Mart Microbiology BV, the Netherlands). Three replicate samples were analysed in each time point.

2.4. Particle size and moisture content analyses

The volume-based particle size distributions of the OBC flours were analysed based on Silventoinen et al., (2018) by laser light diffraction (750 nm) by Beckman Coulter LS 230 (Beckman Coulter Inc., CA, USA) using the liquid module and ethanol as a carrier. Each sample was analysed in duplicate. The moisture content of the OBC flours was determined gravimetrically by oven drying overnight at 105 °C.

2.5. Beta-glucan content and molecular weight analysis

Beta-glucan content of the OBC flours was analysed using a Megazyme β-glucan assay kit following AACC method 32–23.01 (AACC Approved Methods of Analysis). The molar mass of beta-glucan of OBC flours and dispersions was determined in triplicate by size exclusion chromatography (SEC) using calcofluor staining and fluorescence detection, as described in Rosa-Sibakov et al. (2022). Samples were dispersed (0.1 % concentration) into 10 mL of 0.05 M NaOH and diluted and filtered before analysis (0.45 μm). The device had a Waters Alliance 2690 separation module with Calcofluor white (Fluorescent Brightener 28, Aldrich, Germany) and a Scanning Fluorescence 474 detector (Waters Inc., Milford, MA, USA). The calibration curve was prepared with β-glucan standards from Megazyme ranging from 33.6 to 667 kDa.

2.6. Free fatty acid, tocol and volatile analyses

The following analyses were conducted to examine changes in lipids of OBC flours and dispersion samples. Lipids of OBC flours were extracted with acetone using accelerated solvent extraction (100 °C, 1000 psi) (Lampi et al., 2015). Extractions were done with 1.0 g sample sizes and in triplicate. After extraction, lipids were dissolved in heptane and used for tocol (tocopherol and tocotrienol), free fatty acid (FFA) and total fatty acid analyses. Tocopherols and tocotrienols were analysed using normal-phase liquid chromatography (NP-HPLC) with a fluorescence detector (Lampi et al., 2008). Neutral lipids were separated into lipid classes using NP-HPLC with evaporative light scattering detection, and from these chromatograms contents of FFA were determined (Yang et al., 2019). Total fatty acids were analysed after methylation using an internal standard method by gas chromatography with flame-ionization detection (GC-FID), and the sum of fatty acid methyl esters were reported as total fatty acids (Damerau et al., 2014). Tocols of dispersion samples were extracted and analysed similarly after the dispersion samples were freeze-dried.

Volatile compounds were analysed using head space solid-phase micro-extraction gas chromatography with mass spectroscopic detection (HS-SPME-GC-MS) as presented earlier (Damerau et al., 2014) except for using a CAR/PDMS fiber (85 μm film thickness; Supelco, Bellefonte, PA, USA) to collect the volatiles at 50 °C. Analyses were done with 1.0 g of OBC flours and dispersion samples both from freshly prepared materials and from storage experiment in 20-ml amber vials. Analyses were conducted in three replicates.

2.7. Viscosity measurements

Viscosity measurements were carried out during two separate phases of this study. In the first phase, the effect of storing OBC flours in dry form was studied. NHT OBC, HT OBC, and HTD OBC flours were stored at temperatures of 4, 22, or 40 °C for various durations, after which they were mixed with deionised water to a fixed beta-glucan content of 0.65 % (w/w). This meant that the OBC flour content in the dispersions was 3.0–3.4 % depending on the beta-glucan content of the flours. The mixing (100 rpm) was carried out in a mashing bath (Bender & Hobein, Munich, Germany) at 50 °C for 30 min in a, followed by cooling to 22 °C in an ice-water bath. The viscosity of the flour-water dispersions was measured right after cooling.

In the second phase, the effect of storing aqueous dispersions of OBC flours was studied. Dispersions were prepared from fresh or stored HT OBC flour with or without oil as described in section 2.2. The viscosity of the dispersions was measured immediately after preparation and after 1 week and 4 weeks storage at 22 °C.

All viscosity measurements were carried out with a stress-controlled rheometer (AR-G2, TA Instruments, UK), using a narrow gap vane as measuring geometry (diameter of vane and cup 28 and 30 mm, respectively, height of vane 42 mm). Measurements were conducted at 22 °C. After pouring the sample to the measurement system, it was allowed to rest for 2 min before starting the measurement. The flow behaviour was measured over a logarithmically increasing range of shear rates (from 1 to 100 s^{-1}, 10 points/decade). Measurements were carried out in triplicate as steady state measurements with a maximum point time of 1 min. From the viscosity results of the first phase, the consistency index (K) and flow behaviour index (n) were determined by fitting a Power law model ($shear\ stress = K * shear\ rate^{n}$) to the viscosity data.

2.8. Sensory evaluation

Triangle tests and descriptive analysis were performed to detect and describe differences in odour characteristics of selected fresh and stored OBC flours and dispersions. Voluntary panellists (n = 9) were not required to have any prior experience in participating to sensory evaluations. Panellists were informed about the study goals, usage of the collected data and their rights to withdraw their participation at any time. Before the evaluation, the panellists underwent three 1-h training sessions to familiarise themselves with the test methods and to reach consensus on odour attributes to be selected for the descriptive tests.

The panellists evaluated the samples with triangle test during four sessions (6–9 triangle tests in each session). Each panellist evaluated the sample sets in duplicate. The samples were presented in a randomized order within each sample set, whereas the sets were in the same fixed order for each panellist.

For the descriptive analysis, each panellist was given a set of four samples in each session to evaluate the intensities of five attributes; rancidity, pungency, mustiness, oat-like, and sweetness of the odour using line scale (0–10; 0 = very weak, 10 = very strong). The samples were presented in balanced randomised order in each session. The flour samples were evaluated in duplicate, and the dispersions were evaluated only once by each panellist.

The flour samples were presented in brown glass bottles with plastic screw caps (5 ml of sample in a 30 ml bottle) and dispersion samples in transparent plastic containers with glass lids (5 ml of sample in a 40 ml container). All samples were coded with random three-digit numbers and the evaluations were organised in controlled sensory laboratory conditions. All data was collected using Compusense Cloud software (version 19.0, Compusense Inc., Guelph, Canada).

2.9. Statistical analysis

The results of the descriptive sensory methods were analysed by *t*-

test or one-way ANOVA, and the results of the triangle test were analysed by a binomial test using the exact Clopper-Pearson 95 % confidence intervals (CI). The calculations were carried out with SPSS software version 29.0 (IBM Corporation, Armonk, NY). The limit of statistical significance was set at $p < 0.05$. For all other analyses, the results are presented as means of three replicates and the standard deviations are shown as error bars.

3. Results and discussion

3.1. Characteristics of the OBC flours and dispersions

The prepared OBC flour samples had approximately the same median particle size (314 ± 5, 253 ± 6, 306 ± 6 μm in NHT, HT and HTD OBC flour, respectively) and beta-glucan content (18.1 ± 0.4, 23.2 ± 0.3, 19.9 ± 0.2 % of dry matter, in NHT, HT and HTD OBC flour, respectively). Beta-glucan MW was >1000 kDa in all samples. The total fatty acid content of the samples was 9.0 ± 1.2, 7.8 ± 1.0 and 1.3 ± 0.2 % fresh weight (fw), total tocol content 83.4 ± 1.1, 57.3 ± 0.8 and 3.9 ± 0.2 μg/g fw, and moisture content 5.7, 4.7 and 5.2 % in NHT, HT and HTD OBC flour, respectively. No free fatty acids were present. Total tocols included α-tocopherol and α-tocotrienol, β-tocopherol and β-trienol, of which α-tocotrienol was the major one contributing to approximately 80 % and was thus reported in the results. The microbiological quality of the oat dispersions was good and stable during the whole study (below 10 colony forming units/ml).

3.2. Effect of storage on OBC flours

3.2.1. Lipid degradation of OBC flours

Stability of lipids in OBC flours was followed by measuring hydrolysis of lipids using analysis of FFAs, by consumption of primary antioxidants to indicate early phases of lipid oxidation using analysis of tocols, and by formation of secondary lipid oxidation products using analysis of volatile products. Lipids in HT OBC flour oxidized mildly during the storage as also previously observed in oat flours (Lampi et al., 2015): Hexanal as a degradation product of linoleic acid was formed (Fig. 2a) and α-tocotrienol, as a representative of tocols, was degraded due to scavenging lipid radicals (Fig. 2b). As expected, the degradation reactions were accelerated at higher storage temperatures. Hexanal is known to first fill the airspace fairly quickly and then to dissipate and/or be absorbed into the oat matrix (Zhou and Decker, 1999; Lehto et al., 2003), which is one explanation for why hexanal contents started to decrease in HT OBC flour after 8 weeks of storage at all storage temperatures (Fig. 2a). Alternatively, hexanal might be subjected to further reactions such as oxidation. No changes in content of FFA were observed (data not shown). HT OBC flour had very little lipids (1.3 ± 0.2 %) to begin with, and only traces of any volatile compounds and no FFAs were found, as expected (data not shown). Only losses of antioxidative tocols were observed indicating that lipid oxidation of HT OBC had started during storage. Otherwise, no indications of degradation of lipids in HT OBC were observed.

In the NHT OBC flour, the most notable change during the storage was the increase in the level of FFA (Fig. 2c), which refers to enzymatic degradation of lipids. The final FFA content was 1.5, 3.2 and 3.8 mg/g in the flour samples stored at 4 °C, 22 °C and 40 °C, respectively. There were no detectable volatile compounds typical for lipid oxidation in the

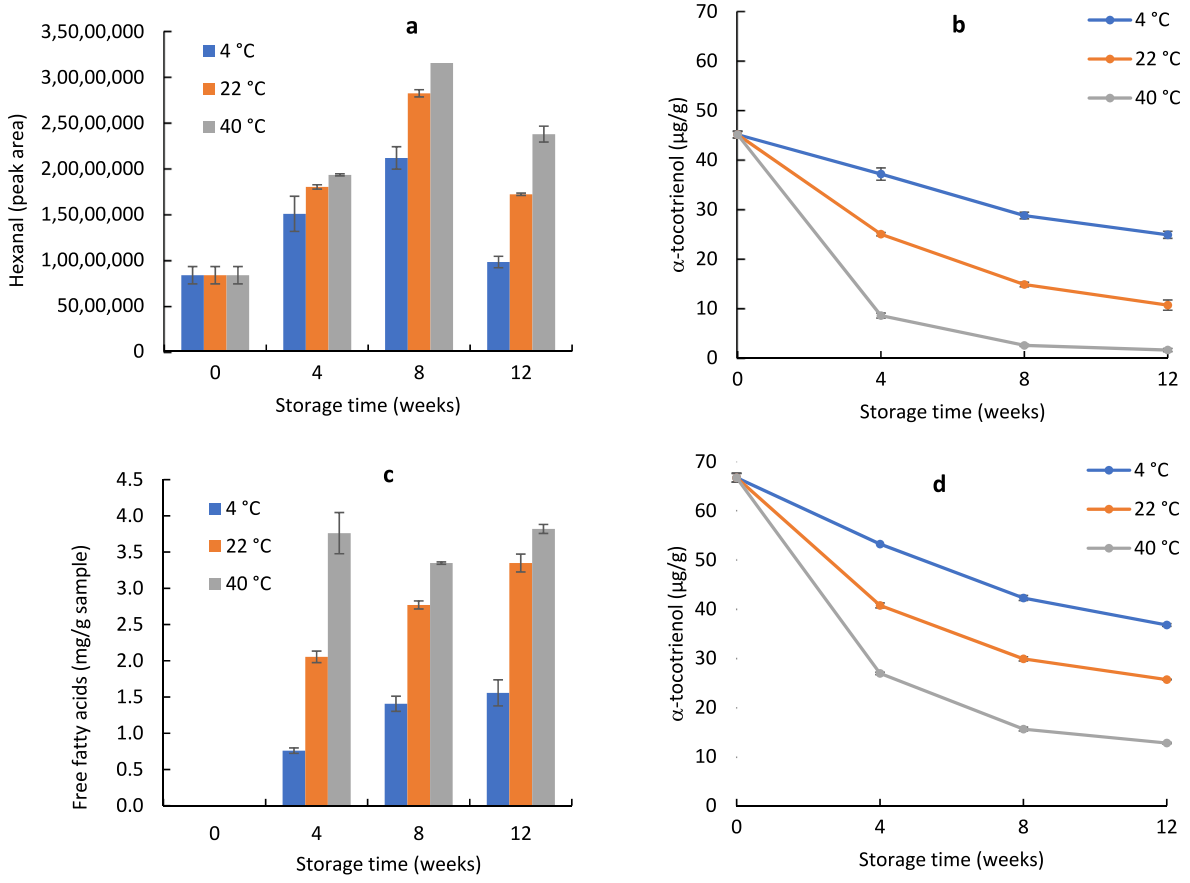

Fig. 2. The level of hexanal in heat treated (HT) OBC (a), alpha-tocotrienol in HT OBC (b), free fatty acids in non-heat-treated (NHT) OBC (c), and alpha-tocotrienol in NHT OBC (d) flours during storage.

samples stored at 4 °C or 22 °C for 12 weeks. Only in the flours stored at 40 °C some 2-pentylfuran, typically formed in NHT oats indicating lipid oxidation (Lampi et al., 2015), could be detected (data not shown).

NHT OBC flour had considerably higher concentrations of total tocols (45 %) and alpha-tocotrienol (49 %) compared to HT OBC flour at the beginning of the storage period. Depending on the storage temperature, total tocol levels decreased to 23−59 % in NHT OBC flour and to 8−60 % in HT OBC flour, which are in line with decreases in α-tocotrienol (Fig. 2b and d), indicating high antioxidant activity that could have protected lipids from degradation in both NHT and HT flours. Oat contains many antioxidants that are decomposed by excessive heating (Heiniö et al., 2002; Bryngelsson et al., 2002; Pöysä et al., 2024), which could also explain the lower level of tocols and higher level of oxidation in the HT flour. Previous studies on the effect of heat treatment and storage of whole grain oats and oat bran also indicated that intensive heat treatment, such as the treatment normally used for inactivation of lipase, may promote oxidative deterioration, while the formation of FFAs is reduced (Lehtinen et al., 2003; Lampi et al., 2015). In addition to inactivation of heat-labile antioxidants, Lehtinen et al. (2003) suggested that disintegration of membranous structures by heat treatment might increase the susceptibility to oxidation.

3.2.2. *Beta-glucan molecular weight and viscosity of OBC flours*

The MW of beta-glucan was the same in all the fresh OBC flours (MW > 1000 kDa), and a notable change was not observed in any of the OBC flours during the storage (data not shown). This indicates that lipid oxidation did not induce oxidative degradation of beta-glucan in the flours, even though lipid oxidation was observed especially in the HT OBC flour. Wang et al. (2016) studied the effects of lipid oxidation on beta-glucan in an oil-in-water emulsion system. They discovered that lipid oxidation induced oxidative degradation of beta-glucan, leading to decreased MW of beta-glucan and reduced viscosity of the emulsion. The experiment of Wang et al. (2016) was done with highly oxidizable tocopherol-stripped rapeseed oil. In the current study, in addition to size exclusion chromatography, possible degradation of beta-glucan during storage was also studied by measuring the viscosity of aqueous dispersions prepared from OBC flours stored for different periods of time. Beta-glucan degradation was assumed to reduce viscosity, as demonstrated by Tosh et al. (2004). The storage time of the flours had, however, a minimal impact on the viscosity of the OBC dispersions in this study (Fig. 3 and Supplementary Fig. S1). A slight change in viscosity was only observed for the NHT and HT OBC flours stored for 12 weeks at 40 °C. The viscosity of the NHT flour dispersion increased slightly, whereas the viscosity of the HT flour dispersion decreased a bit, especially at smaller shear rates. Degradation of beta-glucan is expected to decrease the viscosity of an aqueous solution, but the viscosity of the OBC flour dispersions in our study was not only determined by the properties of the beta-glucan present in the matrix. The OBC materials are not fully soluble and therefore also the properties of the insoluble particles in the system have to be taken into account. An important factor is the extractability of soluble compounds (also other than beta-glucan) from the solid particles to the surrounding aqueous phase. It is possible that the extractability is affected by some chemical or enzymatic processes taking place in the OBC flours during storage.

The viscosity measurements also revealed that there were differences in the viscosity of the dispersions prepared from fresh OBC flours (Fig. 3). The viscosity of the NHT dispersion was found to be lower than that of the HT dispersion, especially at lower shear rates. NHT flour had lower viscosity than the HT flour. Since the dispersions were prepared based on the beta-glucan content of the samples, there were also small differences in the dry matter content of the dispersions (NHT 3.2 %, HT 3.0 %, and HTD 3.4 %), but that cannot explain the low viscosity of the NHT dispersion. It is more likely that the higher viscosity of the HT flour was caused by the heat treatment step in the production of the HT flour, which has been reported previously for oat flours (Jokinen et al., 2023). However, the results of the previous studies regarding the effect of heat treatment on the pasting behaviour of oats are somewhat controversial, which might be related to the differences in the heating and milling processes and variations in the composition of the oat samples (Bai et al., 2021; Jokinen et al., 2023; Nguyen et al., 2019).

3.2.3. *Odour characteristics of OBC flours*

Differences in odour characteristics were detected in a triangle test when comparing NHT, HT, and HTD OBC flour samples with each other, both in the case of fresh and stored (8 weeks at 40 °C) samples (Table 1). This is in line with the known literature where differences in sensory properties, both in flavour and in odour, are found due to different treatments of oats (Salmenkallio-Marttila et al., 2004; McGorrin, 2019). Lipids are precursors to several aroma compounds, so due to the low lipid content of HTD OBC flour compared to the other samples, HTD OBC was understandably different from other OBC flours in terms of odour characteristics. Especially, for the comparison of stored HTD and HT OBC flours, the estimate value was 72.2 % and the 95 % confidence interval (CI) was 46.5 %–90.3 % ($p < 0.001$), while for HT and NHT

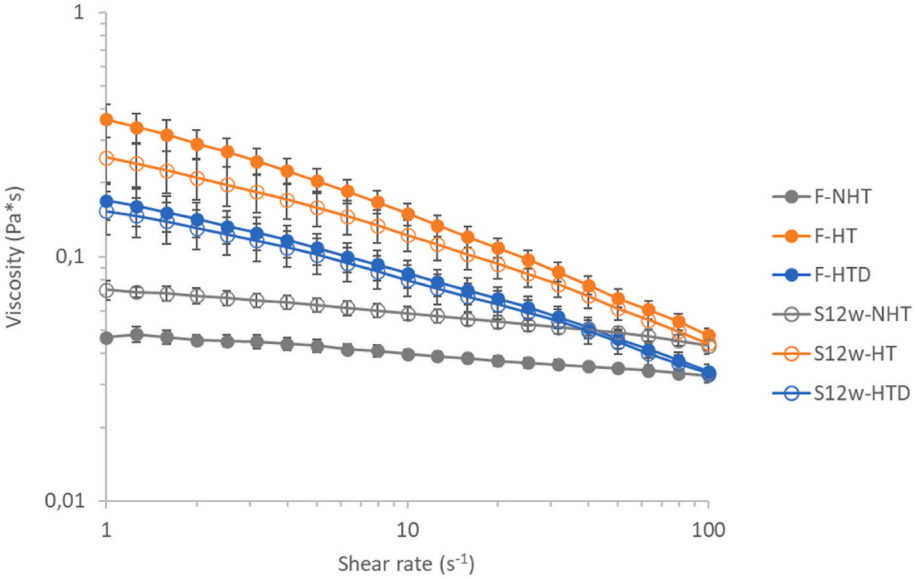

Fig. 3. Viscosity profiles of aqueous dispersions prepared from fresh (F) and 12 weeks (40 °C) stored (S12w) OBC flours.

Table 1
Triangle tests of the OBC flour and dispersions based on odour (p-values, the estimate proportion of correct judgements in the population, and a measure of uncertainty in the estimation using the exact Clopper-Pearson 95 % confidence intervals).

Sample pair in triangle test	p-value	Estimate (%)	Confidence interval (%: CI)	
			lower	upper
OBC flours				
F-HTD & F-HT	0.014	61.1	35.7	82.7
F-HT & F-NHT	0.043	55.6	30.8	78.5
F-NHT & F-HTD	0.043	55.6	30.8	78.5
S-HTD & S-HT	<0.001	72.2	46.5	90.3
S-HT & S-NHT	0.043	55.6	30.8	78.5
S-NHT & S-HTD	0.043	55.6	30.8	78.5
S-NHT & F-NHT	ns	11.1	1.4	34.7
S-HTD & F-HTD	ns	22.2	6.4	47.6
S-HT & F-HT	ns	33.3	13.3	59.0
OBC dispersions stored for 0 or 4 weeks made from F-HT and S-HT flours with or without oil				
S-HT + oil 0w & F-HT + oil 0w	ns	22.2	6.4	47.6
S-HT 4w & F-HT 4w	ns	27.8	9.7	53.5
S-HT + oil 4w & F-HT + oil 4w	ns	22.2	6.4	47.6
S-HT 0w & S-HT 4w	0.014	61.1	35.7	82.7
S-HT + oil 0w & S-HT + oil 4w	0.043	55.6	30.8	78.5
F-HT 0w & F-HT 4w	0.004	66.7	41.0	86.7
F-HT + oil 0w & F-HT + oil 4w	ns	50.0	26.0	74.0

Evaluations in each sample pair 2*9 = 18; F = fresh, S = stored (8 weeks at 40 °C), HT = heat treated, HTD = heat treated defatted, NHT = non-heat-treated; ns: the number of correct judgements of triangle test not reached above the critical value of probability level of 0.05 (Lawless and Heymann, 2010).

flours, the differences were likely due to heat treatment and lack of it (Molteberg et al., 1996). However, a note of caution is due to the interpretation of odour difference significance since the lower CIs were quite low.

In descriptive analysis, oat odour was significantly ($p = 0.026$) higher in stored HT compared to fresh HT OBC flour, 4.8 ± 1.89 and 3.3 ± 2.01, respectively (Supplementary Table S1). However, the effect size (Cohen's $d = 0.08$) remains small, so it is questionable whether a true difference exists between the samples. This could also be supported by the fact that the number of correct judgements was below the critical value of 5 % probability level of triangle tests when comparing stored OBC flours to the corresponding fresh OBC flours (Table 1). It seems that neither the formation of hexanal in HT OBC flour or the increase of FFA in NHT OBC flour developed different odours in the flours. A possible explanation is that the storage time was too short, during which the samples did not have time to develop significant odour differences to be observed (Molteberg et al., 1996). According to Heiniö et al. (2002), some odour differences were not noticeable until after several months of storage.

3.3. Effect of storage on OBC dispersions

3.3.1. Lipid degradation of OBC dispersions

The storage stability of OBC flour dispersions was studied with the HT OBC flours, which were used as fresh (F) or stored (S). For preparing the stored OBC flour, an 8-week storage at 40 °C was chosen because lipid oxidation had progressed to the level where most of the anti-oxidative tocols had been consumed and hexanal contents has started to decline (Fig. 2). The OBC dispersions were stored at 22 °C for up to 4 weeks. In all the dispersion samples, the level of volatile compounds was relatively low. The largest signals were obtained from degradation of linoleic acid, being hexanal, 2-pentylfuran, nonanal, and 2,4-decadienal (data not shown). As expected, the dispersions prepared with stored HT OBC flour had higher levels of hexanal than the dispersions prepared with fresh HT OBC flour (Fig. 4a). During the storage of the dispersions (4 weeks at 22 °C), no significant changes occurred in the hexanal contents. Oil-containing dispersions released less hexanal as compared to the corresponding samples without oil (Fig. 4a). This is likely due to the high solubility of hexanal in oil, which resulted in low release during the HS-SPME analysis.

In the dispersions without oil, the level of total tocols was approximately 70 % lower in the dispersions prepared with S-HT OBC (7 μg/g dm) as compared to the dispersion with F-HT OBC flour (23 μg/g dm) (Fig. 4b). During the storage of the dispersions, the tocol content decreased only mildly (16 %) in both F-HT and S-HT dispersions, which is in line with the finding of minor changes in hexanal during storage. As assumed, the tocol contents were significantly higher in the oil-containing samples (249 and 247 μg/g dm) (Fig. 4c), as the rapeseed oil contains high amounts of α-, γ-, and δ-tocopherols, and only a minor loss occurred (6 %) during storage. Thus, the oxidative status of the OBC flour did not have a clear effect on the storage stability of the dispersions.

3.3.2. Beta-glucan molecular weight and viscosity of OBC dispersions

The dispersions prepared with stored HT OBC showed higher viscosity than the corresponding dispersions prepared with fresh OBC (Fig. 5a). Concerning the storage of the dispersions, the viscosity increased between 0 and 1 week (Fig. 5a). Similarly, there was a small increase in MW of beta-glucan between 0 and 1 week. The increase of viscosity might be related to increased solubility or aggregation of beta-glucan and other compounds during the first week of storage. The dispersions with oil had a higher viscosity than the corresponding dispersions without oil, which is expected as the viscosity of a fluid typically increases with the addition of dispersed particles (in this case oil droplets) to the system (Barnes, 1994).

The MW analysis indicated that degradation of beta-glucan was not occurring during the storage of the aqueous dispersions (Fig. 5b), which is similar to the results concerning the storage of the dry flours. It should, however, be noted that the beta-glucan MW was already initially lower in the dispersions than in the dry flours, possibly due to the heat treatment (121 °C) applied to the dispersions. Regardless of that, no significant decrease in the beta-glucan MW was detected as a function of the dispersion storage time. In the study of Wang et al. (2016) lipid oxidation induced oxidative degradation of pure beta-glucan (MW decrease up to 21–46 %) in an oil-in-water emulsion system with tocopherol-stripped oil, barley beta-glucan, and an iron catalyst. The results of the current study indicate that the same phenomenon did not happen in OBC dispersions. However, there were no significant changes in the hexanal content during the 4-week storage of the dispersions, and probably the level of oxidative compounds was too low to induce oxidative degradation of beta-glucan. However, an increase in the content of hexanal was observed when OBC was stored as flour (Fig. 2a), which might be related to the better availability of oxygen in the flours as compared to the dispersions.

3.3.3. Odour characteristics of OBC dispersions

Oat dispersions prepared with either F-HT or S-HT OBC flour and having a similar storage period (0 or 4 weeks) did not differ from each other in odour, which is in line with the observations made on the fresh and stored OBC flours (Table 1). Nevertheless, the 4-week storage of the dispersions had a significant effect on the odour when compared to the freshly prepared dispersions (Table 1). The estimated value of correct judgements varied between 55.6 % and 66.7 %, though the lower 95 % CI levels were observed to be relatively low, suggesting caution in interpreting the results regarding their reliability and the existence of a true difference. Chemically the samples did not differ much after storage (Figs. 3 and 4), so the observed differences in odour might be due to other chemical, enzymatic, or physical reactions that were not analysed in this study. Additionally, no significant differences were observed in the odour rancidity, pungency, mustiness, oat-like, or sweetness evaluated by the descriptive analysis (Supplementary Table S1). Thus, even though the panel was able to detect some differences among the freshly

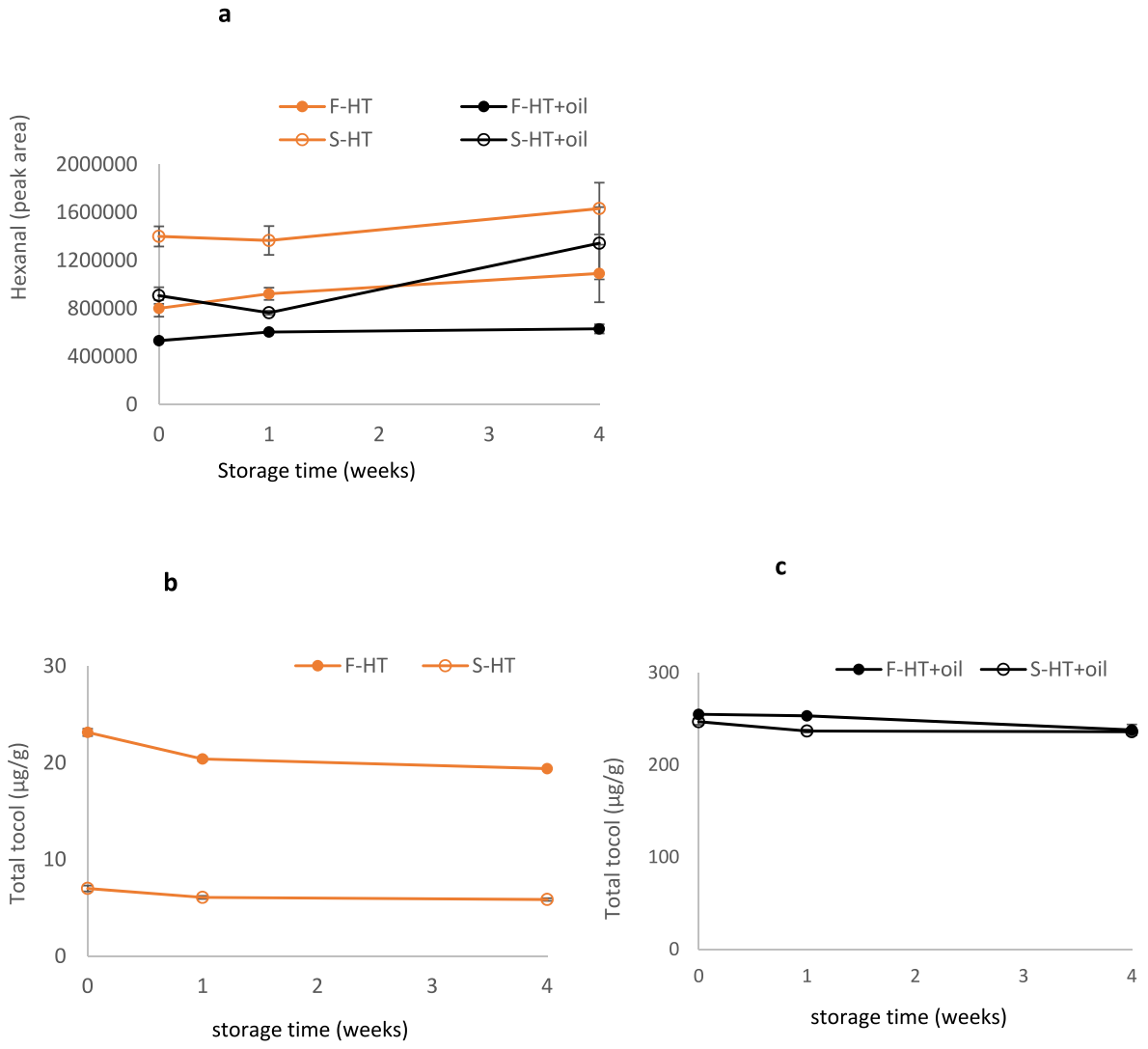

Fig. 4. The level of hexanal in oat dispersions during storage (a), total tocol contents in oat dispersions during storage of F-HT and S-HT samples (b) and samples with oil (c). F-HT = dispersion prepared from fresh, heat-treated OBC, S-HT = dispersion prepared from stored (8 weeks at 40 °C) heat-treated OBC.

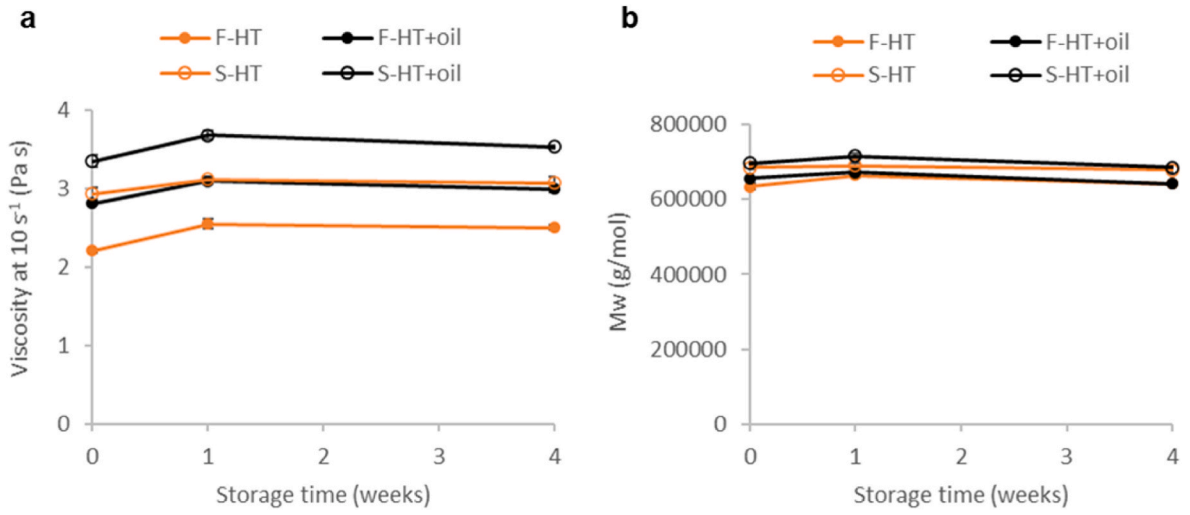

Fig. 5. Viscosity (A) and beta-glucan molecular weight (Mw) (B) of OBC dispersions during storage. F-HT = dispersion prepared from fresh, heat-treated OBC, S-HT = dispersion prepared from stored (8 weeks at 40 °C) heat-treated OBC.

prepared and 4-week stored dispersions, the odour changes were so minor that the panel was not able to identify the odour characteristic causing the differences. The ingredients and properties of food emulsions affect the release of volatile aroma compounds, as reviewed by Mao et al. (2017). The authors reviewed that high oil content and viscosity might increase the retention of aroma compounds, which could also be the factors explaining the minor odour differences in this study.

4. Conclusions

. Oxidative degradation of beta-glucan in the presence of easily oxidising lipids has recently been observed in a model system with purified compounds, and thus the current study explored whether a similar oxidative degradation reaction would also happen in a more complex food system containing oat bran. Lipid oxidation occurred in the heat-treated flours during the storage, but the MW of beta-glucan remained unchanged, indicating that under the conditions applied, lipid oxidation did not induce oxidative degradation of beta-glucan in the flours. Degradation of beta-glucan was neither detected during the storage of the aqueous oat bran dispersions. However, since there were no significant changes in the hexanal content during the 4-week storage of the dispersions, possibly the level of oxidative compounds was too low to induce oxidative degradation of beta-glucan. Further studies are required to elucidate the factors affecting oxidative degradation of beta-glucan and the relevancy of this phenomenon to food processing and products.

When the odour profiles were investigated, heat treatment and defatting had a detectable effect on the sensory properties of the OBC flours. During the storage, the amounts of volatile compounds changed, but this did not affect the odour characteristics of the samples. On the contrary, the storage of the aqueous dispersions had a detectable effect on the odour properties as compared to the freshly prepared dispersions, even though the changes in the level of volatile compounds were only minor. Thus, no clear relation between the amounts of volatile compounds and sensory properties of OBC flours or dispersions was observed in this study, indicating the importance of sensory evaluations to compliment volatile analysis. The observed changes in odour properties during storage need to be attributed to other factors than the analysed volatile compounds originating from lipid oxidation, or the odour may be heavily influenced by minor volatile compounds with low odour threshold.

CRediT authorship contribution statement

Outi Mattila: Writing – original draft, Visualization. **Natalia Rosa-Sibakov:** Writing – review & editing, Visualization, Investigation. **Hanna Ahola:** Investigation. **Anna-Maija Lampi:** Writing – review & editing, Methodology, Investigation. **Oskar Laaksonen:** Methodology, Investigation. **Martina Lille:** Writing – review & editing, Visualization, Methodology, Investigation. **Sari A. Hakanen:** Writing – review & editing, Writing – original draft, Investigation. **Vieno Piironen:** Writing – review & editing, Conceptualization. **Kaisa M. Linderborg:** Writing – review & editing, Funding acquisition, Conceptualization. **Emilia Nordlund:** Writing – review & editing, Supervision, Funding acquisition, Conceptualization.

The use of human subjects

The sensory evaluations were performed in compliance with relevant laws and institutional guidelines in force at the time of the research. Oral informed consent was obtained from the panelists of the sensory evaluations. The privacy rights of human subjects have been observed.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jcs.2025.104238.

Data availability

Data will be made available on request.

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