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Reutilization of berry press residues in minced Baltic herring (*Clupea harengus membras*) – Effect on lipid oxidation and sensory characteristics during cold storage

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ABSTRACT

Lipid oxidation is one of the main causes of quality deterioration in dark-muscled fish. This study investigated the effect of berry side streams of juice pressing or oil extraction at different concentrations on lipid oxidation and sensory quality of minced Baltic herring (*Clupea harengus membras*) stored at 1 °C. Lipid oxidation was assessed by determining the peroxide value and volatile secondary oxidation products. Generic descriptive analysis and gas chromatography-olfactometry were used to investigate the odor, flavor, and odor-active compounds of minces. Based on the peroxide value and formation of hexanal, 1-penten-3-ol, and 3,5-octadien-2-one, a concentration of 1.5 g/100 g of lingonberry-bilberry juice press residue and sea buckthorn juice press and oil extraction residue was sufficient to retard and reduce the extent of oxidation. Furthermore, the addition of these residues inhibited the formation of fishy odors during 3 days of storage. The control mince stored for 3 days showed the highest number and intensities of oxidation products such as (*Z*)-4-heptenal, 2,3-pentanedione, and (*E,Z*)-2,6-nonadienal, while their formation was retarded in the mince containing press residue from sea buckthorn. Berry press residues were efficient in controlling lipid oxidation of minced Baltic herring during storage and have the potential to mask potentially unpleasant fishy odor and flavor.

1. Introduction

Lipid oxidation is a main cause of quality deterioration in fish due to the high content of pro-oxidants, such as heme proteins and polyunsaturated fatty acids (PUFAs), especially in dark-muscled fish. Although high PUFA content is desirable in terms of nutritional quality, the oxidation of PUFAs may give rise to potentially harmful compounds (Rundblad et al., 2017; Vieira et al., 2017) and produce aroma compounds that negatively impact the sensory quality (Fu et al., 2009; Hammer & Schieberle, 2013; Jiarpinijnun et al., 2022). Most processing, such as mincing, further accelerates oxidation in fish. Mincing increases the oxygen to surface ratio and exposes the lipids to pro-oxidants and enzymes, such as lipoxygenases. While low storage temperature delays the development of lipid oxidation, even freezing at temperatures close to −20 °C is not sufficient to fully control it (Aydin & Gokoglu, 2014; Baron et al., 2007; Damerau et al., 2020).

Baltic herring (*Clupea harengus membras*), a subspecies of Atlantic herring (*Clupea harengus*), is a small dark-muscled fish. PUFAs account for approximately one-third of the total fatty acids in Baltic herring (Aitta et al., 2021; Aro et al., 2000; Damerau et al., 2020). Baltic herring is commercially the most important catch species in Finland (based on quantity and value), but most of the catch is directed to low-value uses, such as feed (Sarkki & Pihlajamäki, 2019). One of the factors limiting the use of Baltic herring is its high susceptibility to oxidation and development of its distinctive odor and flavor that many consumers find unappealing (Pihlajamäki et al., 2019). Previously, several secondary lipid oxidation compounds were observed contributing to the odor of minced Baltic herring (Kakko, Aitta, et al., 2022). Retarding lipid oxidation of Baltic herring could therefore control the formation of potentially unpleasant odor and flavor.

While there are several synthetic antioxidants available for controlling lipid oxidation, rising consumer demand for natural products is driving the search for alternatives. For instance, synthetic phenolic antioxidants, such as butyl hydroxyanisole and dibutyl hydroxytoluene, have raised concerns due to their toxicity and spread in the environment (Wang et al., 2021). The antioxidative potential of extracts, fractions, and residues of different botanical origins has been widely studied in fish and meat (Bellucci et al., 2022; Cropotova et al., 2019; Mattje et al.,

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2019; Silveira Alexandre et al., 2022). The berry-processing industry produces high amounts of berry biomass with bioactive properties, such as antioxidative (Puganen et al., 2018), antimicrobial (Puupponen-Pimiä et al., 2021), and antiviral (Granato et al., 2022) effects *in vitro*, but currently, the biomasses remain underutilized. Berries and berry byproducts have also been investigated as potential natural antioxidants in meat and fish (Dai et al., 2021; Damerau et al., 2020; Püssa et al., 2008; Sampels et al., 2010). For example, sea buckthorn press residue was observed to retard lipid oxidation in mechanically separated chicken and turkey meat (Püssa et al., 2008). In a previous study conducted by our research group, both lingonberry-bilberry and sea buckthorn press residues were found to be as or even more effective than conventional antioxidants in retarding lipid oxidation of Baltic herring mince stored at −20 °C for 10 months (Damerau et al., 2020).

However, employing antioxidants of natural origin may present challenges concerning sensory quality and consumer acceptability. In the study by Damerau et al. (2020), berry additions had a negative impact on consumer perception, which emphasizes the need to optimize the dosage to minimize the effect on sensory properties. In another study, supercritical CO_2 ginger extract and ginger essential oil reduced the pleasantness of fish burgers due to a strong ginger flavor (Mattje et al., 2019). While assessing the potential of antioxidants, investigation of sensory quality is important since the antioxidative effects based on instrumentally measured chemical oxidation indicators do not always correlate with sensory quality. For example, a combination of δ-tocopherol, ascorbyl palmitate, and lecithin was seen to inhibit hydroperoxide formation in refined fish oil, but despite extremely low peroxide values, development of off-flavors was not inhibited (Hamilton et al., 1998). Conversely, Sveinsdóttir et al. (2020) reported that dipping Atlantic mackerel (*Scomber scombrus*) fillets into a sodium erythorbate solution significantly prolonged the shelf life of frozen fillets based on sensory evaluation, although no clear inhibition of hydroperoxide or thiobarbituric acid reactive substance (TBARS) formation was observed.

The aim of this study was to investigate whether the addition of lingonberry-bilberry juice press residue, sea buckthorn juice press residue, and sea buckthorn juice and oil extraction residue added at different concentrations (1, 1.5, and 3 g/100 g) retarded the formation of primary and secondary lipid oxidation products in minced Baltic herring stored at 1 °C for 0–21 days. A further aim was to study the effect of the additions on the sensory quality of the mince and to examine connections between lipid oxidation, flavor, odor, and odor-active compounds of the minces during the first three days of storage.

2. Materials and methods

2.1. Materials

For the 21-day storage test, fresh Baltic herring fillets (with skin) (Martin Kala Oy, Turku, Finland) were delivered to the laboratory, packed in ice, immediately portioned in sealable bags, and frozen at −80 °C until the beginning of the storage test. For the sensory evaluation and investigation of odor-active compounds, Baltic herring fillets were kindly provided by Kalaset Oy (Uusikaupunki, Finland). The fillets used for sensory evaluation and gas chromatography–olfactometry (GC-O) (3-day storage test) were brought to the laboratory packed in ice within 24 h of being caught. Upon arrival, they were immediately processed and subjected to sensory analysis. The fillets provided by the two companies (Martin Kala Oy and Kalaset Oy) were caught in the same area and processed similarly and hence were comparable.

Lingonberry-bilberry press residue after juice pressing ("LR") was provided by Kiantama Oy (Suomussalmi, Finland). The press residue is mostly composed of lingonberry, but due to minor "contamination" with bilberry during commercial harvesting of lingonberry, as indicated by the anthocyanin profile of the press residue in our previous study (Damerau et al., 2020), the press residue also contains small amounts of bilberry and is referred to as lingonberry-bilberry juice press residue. Two sea buckthorn press residues, obtained after juice pressing ("SR") and after juice pressing and supercritical CO_2 oil extraction ("SRO"), were provided by Aromtech Oy Ltd. (Tornio, Finland). SR and SRO were from the same berry batch. All berry press residues were brought to the laboratory as dried and stored at −20 °C prior to use. L-Ascorbic acid and α-tocopherol were purchased from Sigma (Sigma–Aldrich Co, St. Louis, MO, USA).

2.2. Storage test

2.2.1. Sample preparation and storage

A storage test of processed Baltic herring mince was conducted at 1 °C for up to 21 days. The fish (Martin Kala Oy) were defrosted overnight at 4 °C and processed into mince. Fish were minced using a Kenwood food processor (Chef Titanium, Kenwood Limited, Havant, United Kingdom) with a meat grinder attachment (with holes 4 mm in diameter), and the antioxidant additions were mixed into the mince immediately. Mixing was conducted manually, the distribution of press residues was confirmed visually, and the control mince was mixed for an equivalent duration. Berry press residues were added at concentrations of 1 g/100 g, 1.5 g/100 g, and 3 g/100 g. These concentrations were chosen based on our earlier observations on 3 g/100 g LR and SR being effective (Damerau et al., 2020), but to reduce costs and effect on sensory quality, the aim was to determine whether a lower concentration (1 or 1.5 g/100 g) would suffice. In addition to LR, SR, and SRO, a combination of α-tocopherol and ascorbic acid (αT + AA, 0.01 g/100 g and 0.2 g/100 g, respectively) was studied for comparison. This combination was chosen due to the synergistic effect of α-tocopherol and ascorbic acid, particularly the ability of ascorbic acid to regenerate α-tocopherol (Packer et al., 1979). Their concentrations were the same as in our previous study (Damerau et al., 2020) and within the same range as previous reports by others (EFSA ANS Panel, 2015; Hamilton et al., 1998; Wang et al., 2017; Yanishlieva & Marinova, 2001). Mince without any additions was used as a control (C).

The fish minces subjected to lipid extraction and PV measurement were stored in plastic containers (30 g in 350 mL container). Minces used for volatile analyses were stored in SPME vials (3 g in a 20 mL vial) to capture the volatiles formed during storage. Both containers and vials were stored at a temperature of 1 °C. At each designated time point during storage (0, 3, 7, 10, and 14 d for volatiles, 0, 3, 7, 10, 14 d, and 21 d for PV measurement), the vials and containers were transferred to −80 °C until analysis.

2.2.2. Lipid extraction, PV measurement, and volatile analysis

Lipids were extracted using 2:1 (mL/mL) chloroform-methanol with 0.5 g/L BHT (2,6-di-tert-butyl-4-methylphenol) according to the method by Cavonius and Undeland (2017) with minor modifications. A 2-g aliquot of mince was first extracted by homogenizing the sample for 2 min in 15 mL chloroform-methanol, followed by another extraction with 6 mL chloroform-methanol. A 3 mL volume of 20 g/L aqueous NaCl was added to the pooled extracts to promote phase separation, followed by vortexing for 30 s and centrifugation at 1000×*g* for 2 min. The organic phase was collected and used for PV analyses. Samples were kept on ice during the extraction, and extraction was performed in duplicate for each sample. Peroxide values were measured in duplicate from each lipid extract according to (Lehtonen et al., 2011).

Volatile compounds were extracted by headspace solid phase microextraction (HS-SPME) and analyzed by gas chromatography–mass spectrometry (GC-MS) as previously reported by our group (Kakko, Damerau, et al., 2022). Briefly, HS-SPME extraction of volatiles was achieved by 20 min incubation and 30 min extraction at 40 °C using a 1 cm divinylbenzene/carboxen/polydimethylsiloxane (DVB/CAR/PDMS) fiber (50/30 μm film thickness; Supelco, St. Louis, MO, USA). Volatiles were desorbed in a 240 °C injector for 6 min and separated on a Supelco SPB-624 column (60 m × 0.25 mm i.d., 1.4 μm film thickness; Supelco,

U.S.A.). MS was operated in electron ionization mode. 1-Penten-3-ol, hexanal, and 3,5-octadien-2-one (*E,Z* or *E,E*) were identified using commercial reference compounds and/or the NIST library (version 2.3, National Institute of Standards and Technology, Gaithersburg, Maryland, USA) and used as indicators of secondary lipid oxidation.

2.3. Sensory analysis of minces with and without LR and SRO

2.3.1. Panelists and training

Nine panelists, consisting of staff and students of the Food Sciences unit at the University of Turku, Finland, were recruited for the generic descriptive analysis (GDA). All panelists had prior experience in sensory analysis. National ethical guidelines (the Finnish National Board on Research Integrity) involving humans were adhered to, but no formal documentation process is available. The panelists were informed about the study protocols and their right to withdraw from the study at any time, and their informed consent to participate and allow storage of personal data during the study was obtained. Microbial analyses of minces were conducted at a certified laboratory (Eurofins Scientific Finland Oy, Raisio, Finland) to ensure that there were no risks for consumption. The panelists were trained to evaluate the odor and flavor characteristics of Baltic herring minces during four sessions. The first three sessions included building the vocabulary, selecting reference samples (Supplemental Table S1), determining their intensities, and placing samples on the scale (0–10, 0 = not at all, 10 = very intense). In the final training session, the panelists evaluated six samples, after which they received feedback on their performance.

2.3.2. Sample preparation and sensory evaluation

Sensory analysis was conducted on minces stored for 0 or 3 days at 1 °C, and minces were not frozen at any point. To limit the number of samples for sensory analyses, two antioxidant additions, LR (1.5 g/100 g) and SRO (1.5 g/100 g), were selected for odor and flavor profiling, while SR was excluded due to having less pronounced antioxidative effects in the 21-day storage test. Samples were evaluated both raw and after *sous vide* cooking, but raw samples were assessed only for their odor.

To prepare the *sous vide* cooked samples, raw minces (stored for 0 or 3 days, with or without LR and SRO) were mixed with NaCl (0.65 g/100 g), vacuum-packed in vacuum seal bags in portions of 25 g and cooked in a 70 °C water bath for 20 min. Cooked samples weighing approximately 12 g were served in 50 mL glass bowls with lids and were tempered on a 65 °C hot plate for 15–20 min prior to serving to the panelists. Raw samples were served in brown glass bottles (6 g in 30 mL bottle).

Odor and flavor/taste attributes were evaluated on a scale of 0–10 (0 = not at all, 10 = very intense), and data were collected using Compusense Cloud 19 (Compusense Inc., Guelph, Ontario, Canada). Evaluations were held in 5 sessions (cooked samples in duplicate, raw samples in triplicate in separate sessions), and 6 samples coded with three-digit codes were served monadically in a randomized order on each session. Cooked samples were evaluated separately from raw samples. Due to the availability of the fish, in most sessions, the panelists evaluated only fresh or only stored samples. Assessors were provided with a low-salt cracker and active charcoal filtered water to cleanse their palate between samples.

2.4. Gas chromatography-olfactometry (GC-O) of raw minces with and without SRO

2.4.1. Panelists and training

The odors of raw C and raw SRO (1.5 g/100 g) minces after 0 d and 3 d of cold storage were further studied by GC-O. Six panelists (4 women, 2 men, age 25–33), 4 of whom had also taken part in the GDA, were recruited and trained in three sessions to describe and evaluate the intensity of odor compounds. The first session was group training that included dilutions of standard compounds pipetted on 1 cm × 1 cm paper in a brown glass bottle (30 mL). The intensity of odors was evaluated on a scale of 0–4 (1 = very mild, barely noticeable, 2 = mild, 3 = fairly strong, and 4 = strong; 0 = not detected). The second and third training sessions included evaluation of a standard compound mix and one of the four Baltic herring mince samples, respectively, on the GC-O.

2.4.2. HS-SPME-GC-O

Volatile compounds for GC-O were extracted using HS-SPME. For the extraction, 20 ± 0.1 g of mince was weighed in a 90 mL Erlenmeyer flask and incubated at 35 °C for 20 min prior to 35 min of extraction at the same temperature. A 2 cm DVB/CAR/PDMS fiber was used to extract the volatiles, and desorption of volatiles occurred at 240 °C for 5 min. GC-O evaluations were conducted using an HP 6890 Series GC with a flame ionization detector (FID) (Hewlett Packard, Palo Alto, CA, U.S.A.). All four samples were assessed twice by the 6 panelists: once on a DB-WAX column (60 m, 0.25 mm, 0.25 μm; Agilent Technologies, Santa Clara, CA, U.S.A.) and once on an SPB-624 column (30 m, 0.25 mm, 1.40 μm, Supelco Inc., Bellefonte, PA, U.S.A.). The oven temperatures for both columns were the same, 40 °C held for 3 min, followed by a ramp of 10 °C/min until 220 °C was reached, and then held for 10 min. The sniffing time was approximately 20 min. Panelists evaluated the samples in a randomized order. Recorded audio files were processed using Audacity 3.0.2 (The Audacity Team). The average odor intensity for each compound was calculated by including only the intensities given by the assessors who detected the odor (i.e., null values were not included). Compounds detected by at least 2 out of 6 panelists (Nasal Impact Frequency, NIF ≥33%) were considered odor-contributing compounds.

Odor-active compounds were identified using external standards or analysis using HS-SPME-GC-MS. External volatile standards included ethyl isovalerate, 3-methylbutanal, 2,3-butanedione, (*Z*)-2-penten-1-ol, 2-methylpropanal, 1-octen-3-one, 2,3-pentanedione, (*E,E*)-2,4-hexadienal, 2,4-heptadienal, (*Z*)-4-heptenal, heptanal, and hexanal (all from Sigma–Aldrich, St. Louis, MO, USA), 1-penten-3-ol (Fluka Chemicals, Neu Ulm, Switzerland), 2-methylbutanal, propanal (Acros Organics, Geel, Belgium), 1,5-octadien-3-one, and 1,5-octadien-3-ol (Eptes Sàrl, Vevey, Switzerland). TRACE 1310 gas chromatograph coupled with an ISQ7000 single quadrupole mass spectrometer and TriPlus RSH autosampler (Thermo Fisher Scientific, Waltham, Massachusetts, U.S.A.) were used for the identification of volatiles by HS-SPME-GC-MS. The MS was operated in electron ionization mode. The transfer line and ion source temperatures were 220 °C and 250 °C, respectively, and mass spectra were collected in the range of *m/z* 40–300 in scan mode. GC-MS data were processed using Chromeleon 7.0 (Thermo Scientific™), and identification was performed by comparison to the NIST library (version 2.3, National Institute of Standards and Technology, Gaithersburg, Maryland, U.S.A.)

2.5. Statistical analyses

The PVs, volatile peak areas, and intensities of sensory attributes of minces with different additions, within the same time point, were compared using one-way analysis of variance (ANOVA) and Tukey's test in SPSS Statistics 27 (IBM, Armonk, New York, U.S.A.). The intensities of odor and flavor attributes after 0 or 3 days of storage within the same type of mince were compared using paired samples t tests. A 95% confidence level was used. Partial least squares (PLS) regression using Unscrambler (version 10.4.1, Camo Process AS, Oslo, Norway) was used to model how odor-active compounds explain differences in observed odor attribute intensities in GDA (X variables were weighed intensities, i.e. intensity*NIF, of odor-active compounds; Y variables were observed odor intensities in GDA). PanelCheck software (version 1.4.2, Nofima, Tromsø, Norway) was used to evaluate the agreement, sensitivity, and reproducibility of the panel in GDA (Tomic et al., 2009).

3. Results and discussion

3.1. Lipid oxidation during storage of minced Baltic herring

3.1.1. Changes in peroxide value during 0–21 days of storage

PVs (Fig. 1) after 0, 3, 7, 10, 14 and 21 d of storage were analyzed as markers of primary lipid oxidation. The *p* values of differences between samples within each time point are presented in Supplemental Table S2. The PV of the control mince reached its peak after 10 d, after which it declined over the remaining storage period. In the berry press residue-containing minces, however, the highest PV was observed after 14 d or later. In addition to delaying hydroperoxide formation, most berry press residues also decreased the total extent of the formation. SR1.5 and SR3, as well as αT + AA, however, had a PV similar to that of C at the beginning of storage, but at 14 days, the PV was significantly ($p < 0.05$) higher. The higher PVs observed in SR and αT + AA could be caused by their possible pro-oxidativity effects. Another explanation might be the stabilization of hydroperoxides, which would lead to their accumulation. In a previous study, chub mackerel (*Scomber japonicus*) mince with pomegranate seed extract showed higher levels of hydroperoxides during frozen storage compared to the control, but the TBARS levels were considerably lower (Özalp Özen et al., 2011), indicating that the higher PV observed in the mince with pomegranate seed extract was not due to pro-oxidativity of the extract.

Compared to SR, SRO minces had lower PVs, especially when comparing SR3 and SRO3 (8.4 vs. 4.3, respectively, on Day 14). SR has been reported to contain a significant amount of lipids but also lipid soluble antioxidants, such as tocopherols (Damerau et al., 2020). The SRO was from the same berry batch as SR but had been further subjected to supercritical CO_2 extraction of oil and therefore contained less lipids and lipid soluble antioxidants than SR. In LR-containing minces, the PV increase was slow during the storage period and did not reach its peak during the storage period of 21 days.

LR, SR, and SRO affected the PV differently depending on the concentration. In the case of SR, the difference between SR3 and SR1.5 was significant only on Days 3, 10, and 21 (Supplemental Table S2), and in most cases, the former had a similar or higher PV compared to C. In the case of SRO, SRO3 had a significantly lower PV compared to SRO1 and SRO1.5 at most time points. However, SRO1.5 had significantly lower PV than the control at Days 0, 3, and 10. The PVs of LR3 and LR1.5 did not differ statistically significantly at any point. However, due to the rapid degradation of hydroperoxides to form secondary oxidation products, PV by itself is not a sufficient measure of lipid oxidation and should be considered together with secondary oxidation indicators (Ross & Smith, 2006).

3.1.2. Formation of volatile secondary oxidation products during 0–14 days of storage

1-Penten-3-ol, hexanal, and 3,5-octadien-2-one were chosen as secondary oxidation indicators (Fig. 2) since they have been previously found to be suitable indicators for lipid oxidation produced from different precursors and were among the most abundant volatiles in the samples. 1-Penten-3-ol is reported to be formed from n-3 fatty acids through scission of the C17 alkoxy radical, which is formed from 17-OOH through O–O cleavage (Hammer & Schieberle, 2013; Lee et al., 2003). In previous studies, 1-penten-3-ol was shown to be one of the most abundant volatiles formed during lipid oxidation of herring (Sampels et al., 2010) and Baltic herring (Damerau et al., 2020). Hexanal, which can be formed by scission of the fatty acid chain on either side of the radical and is mainly produced from oxidation reactions of n-6 fatty acids (Gómez-Cortés et al., 2015), has been widely used as a lipid oxidation indicator and was identified as an odor-active compound in Baltic herring (Kakko, Aitta, et al., 2022). 3,5-Octadien-2-one can be produced during docosahexaenoic acid (DHA) autoxidation via 14-OOH-13,16-epidioxide (Noble & Nawar, 1975).

At most time points, volatile formation was highest in the control sample, whereas berry press residue and αT + AA addition had an inhibitory effect. At all concentrations, LR significantly inhibited the formation of 1-penten-3-ol and 3,5-octadien-2-one after 3–14 days of storage when compared to the control ($p < 0.05$) (Fig. 2 and Supplemental Tables S3–S5). On the other hand, SR1 showed the least inhibition of the formation of these compounds among the berry press residue additions. Similar inhibition of volatile secondary oxidation product formation in fish by other natural antioxidants has also been reported in previous studies (Farvin et al., 2012; Joaquin et al., 2008; Sampels et al., 2010). For example, milk protein concentrate added to herring (Joaquin et al., 2008) and ethanol extracts of potato peel added to horse mackerel (Farvin et al., 2012) decreased the formation of 1-penten-3-ol during storage.

In general, based on the PVs and the content of the volatile secondary oxidation products, LR and SRO additions showed the most pronounced antioxidative effects in minced Baltic herring (Figs. 1 and 2).

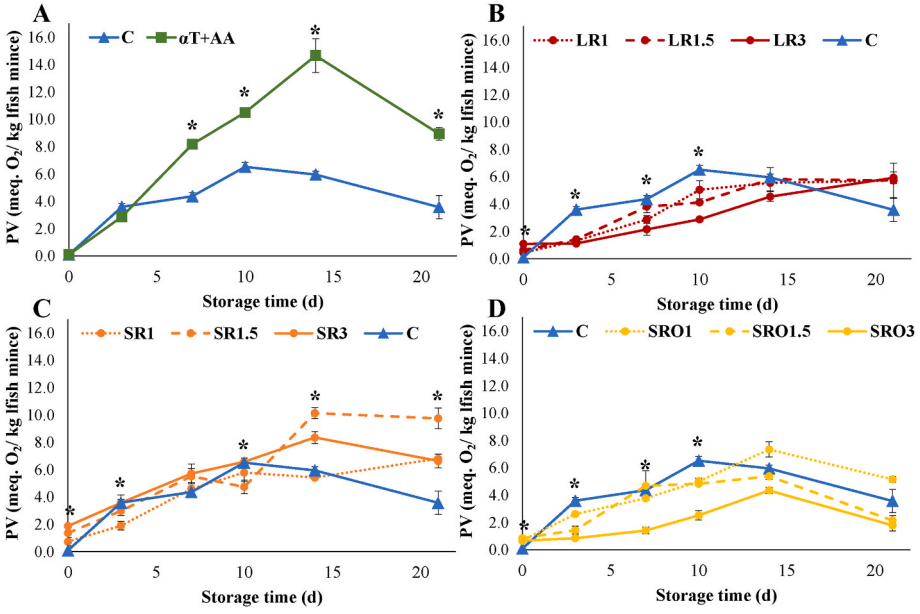

Fig. 1. Peroxide values (PV, meq/kg fish, n = 4) of Baltic herring minces containing no additions (control, C) (A–D), α-tocopherol and ascorbic acid (αT + AA) (A), 1.0, 1.5, or 3.0 g/100 g lingonberry-bilberry juice press residue (LR) (B), 1.0, 1.5, or 3.0 g/100 g sea buckthorn juice press residue (SR) (C), or 1.0, 1.5, or 3.0 g/100 g sea buckthorn juice press and oil extraction residue (SRO) (D), after 0, 3, 7, 10, 14, and 21 days at 1 °C. An asterisk (*) indicates a statistically significant ($p < 0.05$) difference between the samples at the given time point.

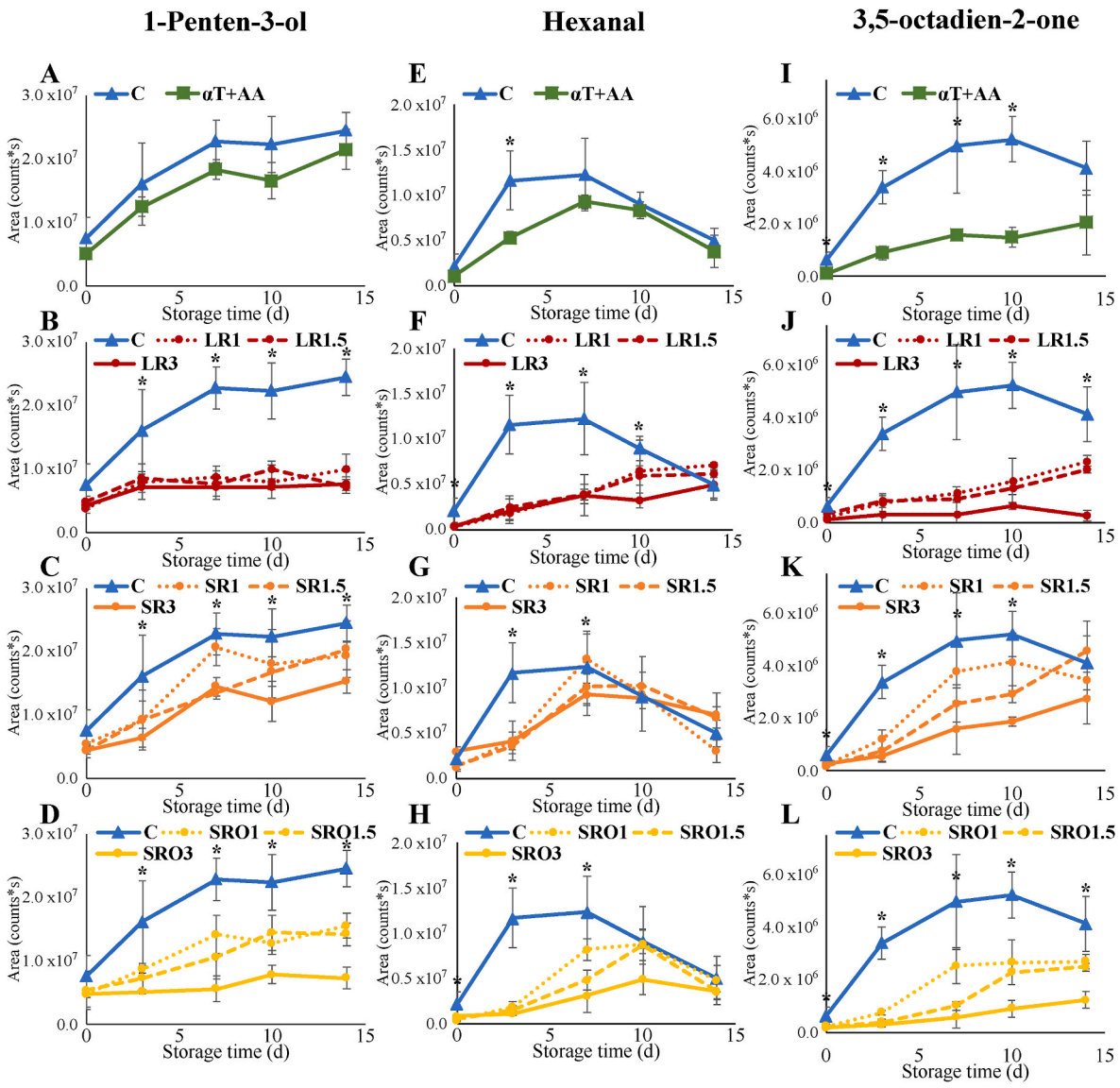

Fig. 2. Changes in selected secondary volatile oxidation compounds, 1-penten-3-ol (A–D), hexanal (E–H), and 3,5-octadien-2-one (I–L) as peak areas (n = 3) in Baltic herring minces without any additions (control, C) (A–L) and with α-tocopherol and ascorbic acid (αT + AA) (A, E, I), 1–3 g/100 g lingonberry-bilberry juice press residue (LR) (B, F, J), 1–3 g/100 g sea buckthorn juice press residue (SR) (C, G, K), or 1–3 g/100 g sea buckthorn juice press and oil extraction residue (SRO) (D, H, L) during the 14-day storage period. An asterisk (*) indicates a statistically significant ($p < 0.05$) difference between the samples at the given time point.

Interestingly, SRO was more effective than SR, despite the removal of lipids and lipid soluble antioxidants by supercritical CO_2 extraction. This may be attributed to the removal of lipids leading to the concentration of water-soluble antioxidants, such as flavonol glycosides, which are not removed during supercritical CO_2 extraction (Linderborg et al., 2012). Püssa et al. (2008) suggested that flavonol glycosides were significant contributors to the antioxidative effect of SR in mechanically deboned chicken and turkey. In addition, the high content of oil in the SR may interfere with the dispersion of phenolic compounds in the fish mince and reduce their access to reactive radical species. Lingonberry press residue was previously shown to be a potent antioxidant during pH-shift processing of herring and salmon based on reduced levels of malondialdehyde and 4-hydroxy-(*E*)-2-hexenal (Zhang et al., 2022).

Regarding different concentrations of berry press residues in the present study, based on their inhibitory effect on the formation of secondary volatile compounds, the addition of 3 g/100 g and 1.5 g/100 g LR, SR, and SRO performed similarly. Especially during the first seven days of storage at 1 °C, and in the case of LR and SRO, there were only a few statistically significant ($p < 0.05$) differences between the 1.5 g/100 g and 3 g/100 g concentrations (Supplemental Tables S3–S5). Reducing the concentration of added press residues is desirable for minimizing the changes in sensory properties and for limiting the costs of the addition.

3.2. Flavor and odor profile of raw and cooked Baltic herring minces

3.2.1. Panel performance

The performance of the GDA panel was evaluated using PanelCheck software. According to 3-way ANOVA, Tucker-1 PCA, and *p*-MSE plots (data not shown), out of the attributes in which significant differences were observed, most disagreement was found in fish oil odor. The disagreement was, nevertheless, less significant than the sample effect. Marine odor and berry/fruit-like odor were most agreed upon. In the case of cooked minces, the berry-/fruit-like odor was most difficult for the panel, whereas fishy flavor and marine odor and flavor were among the best understood attributes due to low and nonsignificant panelist*sample interactions. Although some panelists had difficulties in repeatability or sensitivity of individual attributes, no one performed systematically weakly, and therefore, the results of all panelists were

included for other statistical analyses.

3.2.2. Effect of berry press residue addition on the odor and flavor of Baltic herring mince during storage

The odor profiles of raw and cooked minces and flavor/taste profiles of cooked minces (Fig. 3, Supplemental Table S6) were assessed to investigate the effect of LR (1.5 g/100 g) and SRO (1.5 g/100 g) additions on the sensory quality of the minces during storage. In the case of raw samples, the C, LR1.5, and SRO1.5 at Day 0 differed statistically significantly ($p < 0.05$) only in terms of berry-/fruit-like odor. During the 3-day storage, however, the raw control mince exhibited significant increases in total intensity of odor, fishy odor, marine odor, and fish oil odor along with a decrease in berry-like/fruit-like odor intensity. LR1.5 showed a slight increase in fishy (3.3 vs. 4.0) and marine odor (2.0 vs. 3.5), and SRO1.5 did not show any significant ($p < 0.05$) differences on Day 0 vs. Day 3. A similar improvement in sensory quality by antioxidant treatment was reported by Sveinsdóttir et al. (2020). In their study, dipping Atlantic mackerel fillets into sodium erythorbate was seen to increase the shelf life of fillets during frozen storage due to inhibition of rancid flavor development. In another study, tannic acid was seen to efficiently reduce the formation of TBARS, hydroperoxides, and fishy odor during storage of fish emulsion sausages (Maqsood et al., 2012).

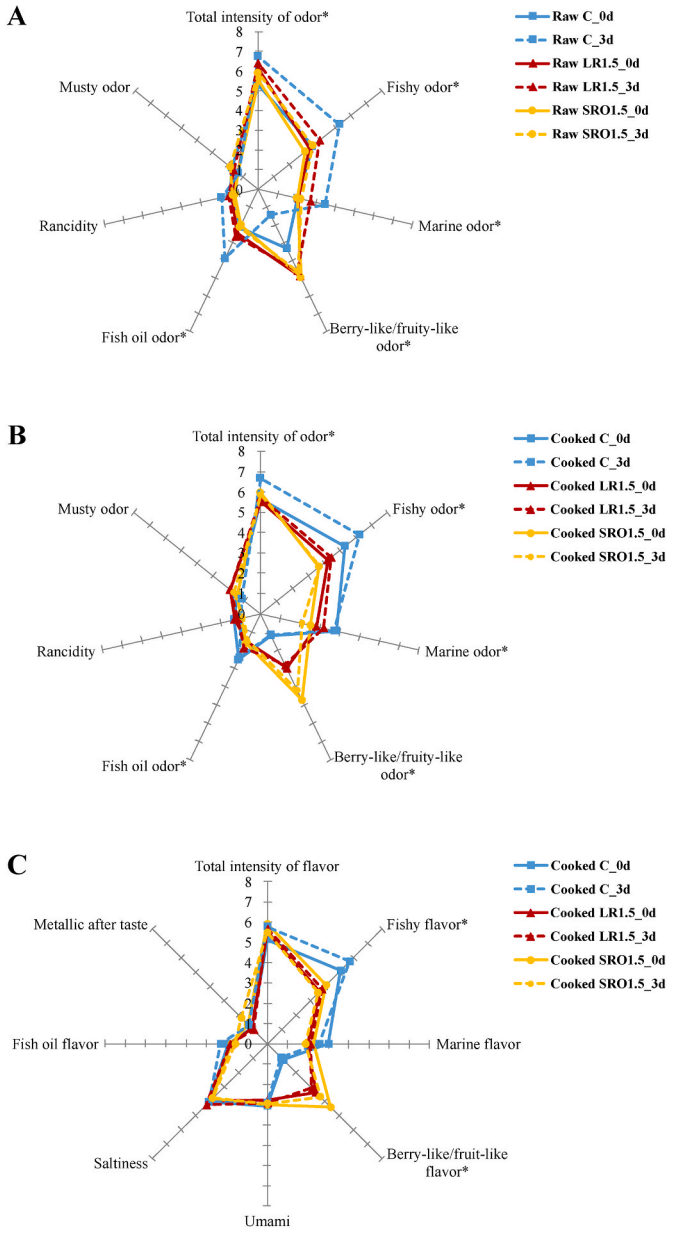

Fig. 3. Odor profiles of raw (A) and cooked (B) and flavor/taste profile of cooked (C) Baltic herring minces based on the generic descriptive analysis (n = 9*3 for raw samples, n = 9*2 for cooked samples). Sample abbreviations: C=Control, SRO1.5 = mince with 1.5 g/100 g sea buckthorn juice press and oil extraction residue, LR1.5 = mince with 1.5 g/100 g lingonberry juice press residue, 0d = after 0 days of storage, 3d = after 3 days of storage at 1 °C. Attributes marked with an asterisk (*) differed statistically significantly ($p < 0.05$) between minces.

A previous study by Fu et al. (2009) reported that hemoglobin-induced oxidation caused an oxidized oil odor in silver carp (*Hypophthalmichthys molitrix*) mince. In the present study, fish oil odor increased in raw minces during the 3-day storage, but after cooking, there was no difference in fish oil odor between C-0d and C-3d. This could be due to the masking effect of other odors formed during *sous vide* cooking. Previously, *sous vide* cooking was seen to significantly alter the volatile profile of European sea bass to a higher extent than steaming and boiling (Nieva-Echevarría et al., 2017).

Unlike the raw minces at Day 0, the cooked minces at Day 0 differed significantly in several odor attributes, indicating that cooking influenced the odor of the minces differently. For instance, cooked SRO1.5 at 0 d had a lower intensity of marine odor (2.5 vs. 3.8) and fish oil odor (1.6 vs. 2.5) compared to cooked C at 0 d. Cooked LR1.5 at 0 d had a lower fishy flavor (3.6 vs. 5.1) compared to C at 0 d. In the case of cooked minces at Day 3, significant differences in fishy odor were observed between C vs. both LR1.5 and SRO1.5. In a study by Cropotova et al. (2019), the addition of an antioxidant (commercial preparation containing rosemary extract) was seen to promote the oxidative stability of Atlantic mackerel during *sous vide* cooking, although sensory quality was not evaluated.

The rancidity of samples was considered low (<2) in all samples (Fig. 3). The reference for rancid odor was fish oil oxidized by microwave heating, and hence, the rancidity was perhaps different from the rancid odors formed during cold storage of the fish minces. In general, the term "rancid" may refer to different sensory perceptions in different products since oxidation may produce different odor- and flavor-active compounds depending on the mechanism and precursor fatty acids (Fu et al., 2009; Jacobsen, 1999). In the raw control mince, a significant increase in fishy odor was observed after 3 days of storage at a low temperature (1 °C), which demonstrates the impact of lipid oxidation on the sensory quality of Baltic herring and fish in general. The present study showed that in addition to delaying lipid oxidation, measured by PV and secondary oxidation-derived volatiles, the added berry press residues inhibited oxidative deterioration from a sensory perspective and thus helped to maintain the sensory quality during the storage period.

3.3. Odor-active compounds in raw minces with or without added sea buckthorn press residue

As the most significant differences in odor, based on GDA, were found between raw control mince and raw mince with 1.5 g/100 g SRO, their odor-active compounds at 0 d and 3 d were investigated using GC-O (Table 1 and Table 2). Considerably more odor-contributing compounds (NIF ≥33%) were detected in the stored control mince (C-3d) than in SRO1.5-0d, SRO1.5-3d, and C-0d (22 vs. 12, 11, and 8 odor-active compounds, respectively, on the semipolar column). 3-Methylbutanal (described as musty, chocolate, solvent) and (*Z*)-1,5-octadien-3-one (green, raw, metal, pelargonium) were detected in all samples by all panelists and on both columns, but the NIFs and intensities of most odor-active compounds differed between minces. Most odor-active compounds, such as 2,3-pentanedione (butter, caramel, fatty), hexanal (grass, green, fresh), (*Z*)-4-heptenal (fish, rancid, green), (*E,E*)-2,4-hexadienal (musty, potato, green, wax), and (*E,Z*)-3,5-octadien-2-one (fruity, musty), had the highest NIFs and intensities in C-3d. This is in

Table 1
NIFs (Nasal Impact Frequencies), intensities, and weighed intensities (NIF*intensity)[a] of odor-contributing (NIF ≥33%) compounds detected on the semipolar column (SPB-624) in Baltic herring mince with (SRO1.5-0d, SRO1.5-3d) and without (C-0d, C-3d) sea buckthorn press residue, after 0 days (0d) or 3 days (3d) storage at 1 °C.

Code	Compound	Identification[b]	RI SPB-624	Description	NIF				Intensity				NIF*Intensity			
					C_0d	C_3d	SRO1.5_0d	SRO1.5_3d	C_0d	C_3d	SRO1.5_0d	SRO1.5_3d	C_0d	C_3d	SRO1.5_0d	SRO1.5_3d
A1	unknown		505	stale, musty	17%	33%	33%	17%	1.0	1.0	1.0	2.0	0.2	0.3	0.3	0.3
A2	unknown		526	citrus, bilberry, flowery	0%	0%	17%	33%	0.0	0.0	1.0	1.8	0.0	0.0	0.2	0.6
A3	2-methylpropanal	std, ms, O, RI	600	solvent, chocolate, musty, green	50%	67%	50%	50%	1.7	2.6	1.5	2.0	0.8	1.8	0.8	1.0
A4	2,3-butanedione	std, ms, O, RI	640	butter, caramel, fatty	67%	50%	67%	67%	2.0	1.7	1.5	1.1	1.3	0.8	1.0	0.8
A5	unknown		683	sweet, fruity, creamy	17%	50%	33%	33%	1.0	2.2	1.3	1.0	0.2	1.1	0.4	0.3
A6	3-methylbutanal + 2-methylbutanal	std, ms, O, RI	701	musty, green, chocolate, solvent	100%	100%	100%	100%	2.6	3.1	2.1	2.7	2.6	3.1	2.1	2.7
A7	2,3-pentanedione	std, ms, O, RI	741	caramel, butter, fatty	17%	100%	17%	0%	1.0	2.3	1.0	0.0	0.2	2.3	0.2	0.0
A8	unknown		765	fruity	0%	17%	33%	17%	0.0	1.0	1.0	1.0	0.0	0.2	0.3	0.2
A9	unknown		781	rancid, wax, soap	17%	83%	0%	0%	1.0	2.5	0.0	0.0	0.2	2.1	0.0	0.0
A10	(*Z*)-2-penten-1-ol	std, ms, O, RI	834	no common descriptor	0%	33%	0%	0%	0.0	1.3	0.0	1.0	0.0	0.4	0.0	0.0
A11	hexanal	std, ms, O, RI	849	grass, leaf, fresh	33%	50%	17%	33%	1.0	1.7	1.0	1.3	0.3	0.8	0.2	0.4
A12	ethyl-3-methylbutanoate	std, ms, O, RI	882	sweet, fruity, candy, citrus	17%	33%	100%	83%	1.5	2.3	2.0	2.2	0.3	0.8	2.0	1.8
A13	Heptanal	std, ms, O, RI	942	potato-like	17%	33%	17%	17%	1.5	1.0	1.0	2.0	0.3	0.3	0.2	0.3
A14	(*Z*)-4-heptenal	std, ms, O, RI	954	fish, rancid, green, oil	33%	100%	33%	33%	1.0	3.1	1.0	2.0	0.3	3.1	0.3	0.7
A15	(*E,E*)-2,4-hexadienal	std, O, RI	986	musty, potato, green, wax	0%	83%	33%	50%	0.0	1.8	1.0	1.3	0.0	1.5	0.3	0.7
A16	(*Z*)-1,5octadien-3-ol or (*E*)-1,5-octadien-3-one	std, O, RI	1031	stale/musty, mushroom, green	67%	50%	67%	33%	1.7	2.7	1.5	1.5	1.1	1.3	1.0	0.5
A17	(*Z*)-1,5-octadien-3-one	std, O, RI	1042	green, waxy, soil, metal, pelargonium	100%	100%	100%	100%	2.9	3.3	2.3	2.7	2.9	3.3	2.3	2.7
A18	(*E,Z*)-2,4-heptadienal	ms, O, RI	1070	green, cucumber	17%	33%	0%	0%	2.0	2.5	0.0	0.0	0.3	0.8	0.0	0.0
A19	unknown		1097	no common descriptor	0%	0%	33%	17%	0.0	0.0	1.0	1.0	0.0	0.0	0.3	0.2
A20	(*E,Z*)-3,5-octadien-2-one	ms, RI	1136	fruity, musty	17%	67%	17%	0%	1.0	1.3	1.0	0.0	0.2	0.8	0.2	0.0
A21	*E,E*)-3,5-octadien-2-one	ms, RI	1156	soap, cucumber, cooked rice	17%	50%	17%	0%	1.0	1.3	2.0	0.0	0.2	0.7	0.3	0.0
A22	unknown		1208	no common descriptor	0%	33%	0%	0%	0.0	1.0	0.0	0.0	0.0	0.3	0.0	0.0
A23	(E,Z)-2,6-nonadienal	std, ms, O, RI	1228	cucumber, fatty	0%	33%	0%	17%	0.0	2.5	0.0	1.0	0.0	0.8	0.0	0.2
A24	unknown		1260	no common descriptor	0%	33%	0%	17%	0.0	1.0	0.0	1.0	0.0	0.3	0.0	0.2
A25	unknown		1361	musty, grain-like/ cereal-like	0%	50%	0%	0%	0.0	1.3	0.0	0.0	0.0	0.7	0.0	0.0
A26	unknown		1384	no common descriptor	33%	33%	0%	0%	1.5	2.0	0.0	0.0	0.5	0.7	0.0	0.0

Literature and databases for comparison included Ahonen et al. (2022); Aitta et al. (2021); An et al. (2020); Selli et al. (2006); Sérot et al. (2002), NIST Chemistry WebBook, https://webbook.nist.gov, and Flavornet database, https://www.flavornet.org.

[a] n = 6.

[b] Identification was based on comparison to a commercial reference compound (std), comparison of ms spectra match (recorded by HS-SPME-GC-MS analysis) to the NIST library (ms), similarity of odor description to previous literature (O), and/or observed LRI close to LRI reported by previous literature or NIST.

Table 2
NIFs (Nasal Impact Frequencies), intensities, and weighed intensities (NIF*intensity)[a] of odor-contributing (NIF ≥33%) compounds detected on the polar column (DB-WAX) in Baltic herring mince with (SRO1.5) and without (C) sea buckthorn press residue, after 0 days (0d) or 3 days (3d) storage at 1 °C.

Code	Compound	Identification[b]	RI DB-WAX	Description	NIF				Intensity				NIF*Intensity			
					C-0d	C-3d	SRO1.5-0d	SRO1.5-3d	C-0d	C-3d	SRO1.5-0d	SRO1.5-3d	C-0d	C-3d	SRO1.5-0d	SRO1.5-3d
B1	propanal	std, ms, O, RI	801	no common descriptor	0%	33%	0%	0%	0.00	1.00	0.00	0.00	0.0	0.3	0.0	0.0
B2	2-methylpropanal	std, ms, O, RI	817	chocolate, solvent, coffee	50%	67%	50%	67%	2.50	2.00	2.00	2.13	1.3	1.3	1.0	1.4
B3	3-methylbutanal+2-methylbutanal	std, ms, O, RI	912	solvent, cacao, stale	100%	100%	100%	100%	2.17	3.00	2.50	2.58	2.2	3.0	2.5	2.6
B4	unknown		926	stale	0%	50%	0%	0%	0.00	2.00	0.00	0.00	0.0	1.0	0.0	0.0
B5	unknown		958	fresh, sweet	0%	0%	0%	50%	0.00	0.00	0.00	1.33	0.0	0.0	0.0	0.7
B6	2,3-butanedione	std, ms, O, RI	974	caramel, sweet, butter	100%	83%	67%	0%	1.83	2.00	1.50	0.00	1.8	1.7	1.0	0.0
B7	unknown		1011	solvent, rancid oil, wax	0%	100%	17%	33%	0.00	2.33	1.50	2.00	0.0	2.3	0.3	0.7
B8	2,3-pentanedione + unknown	std, ms, O, RI	1051	caramel, butter, sweet, fresh, fruity	33%	100%	100%	100%	1.00	2.83	1.67	1.92	0.3	2.8	1.7	1.9
B9	ethyl-3-methylbutanoate	std, ms, O, RI	1066	pineapple, fresh, fruit	33%	0%	33%	67%	1.00	0.00	2.50	2.13	0.3	0.0	0.8	1.4
B10	hexanal	std, ms, O, RI	1078	green, fresh	0%	33%	0%	0%	0.00	2.25	0.00	0.00	0.0	0.8	0.0	0.0
B11	(Z)-3-hexenal	O, RI	1131	green, grass	17%	33%	17%	33%	2.00	1.50	2.00	1.00	0.3	0.5	0.3	0.3
B12	heptanal	std, ms, RI	1172	no common descriptor	17%	33%	0%	17%	1.00	1.25	0.00	2.00	0.2	0.4	0.0	0.3
B13	unknown		1219	pungent, musty/stuffy	50%	0%	17%	33%	2.00	0.00	2.00	2.25	1.0	0.0	0.3	0.8
B14	(Z)-4-heptenal	std, ms, O, RI	1237	fish, stale, pungent, sea	50%	100%	17%	33%	2.33	3.33	2.00	2.50	1.2	3.3	0.3	0.8
B15	(Z)-2-penten-1-ol + 1-octen-3-one or 2-octanone	std, ms, O, RI	1300	mushroom, stale	50%	100%	100%	100%	2.67	2.33	1.67	2.50	1.3	2.3	1.7	2.5
B16	unknown		1349	bergamot, pelargonium	0%	67%	0%	0%	0.00	1.50	0.00	0.00	0.0	1.0	0.0	0.0
B17	(Z)-1,5-octadien-3-one	std, O, RI	1377	green, raw, metal, pelargonium	100%	100%	100%	100%	2.83	3.08	2.83	3.33	2.8	3.1	2.8	3.3
B18	(E)-1,5-octadien-3-ol	std, RI	1453	no common descriptor	0%	33%	0%	17%	0.00	2.00	0.00	1.00	0.0	0.7	0.0	0.2
B19	2,4-heptadienal (E,Z or E,E)	ms, O, RI	1473	potato	0%	33%	33%	50%	0.00	2.00	1.50	1.83	0.0	0.7	0.5	0.9
B20	(Z)-1,5-octadien-3-ol	std, O, RI	1485	green, mushroom	33%	83%	0%	0%	1.00	1.70	0.00	0.00	0.3	1.4	0.0	0.0
B21	(E)-2-octenol	ms, O, RI	1621	green, grass, cucumber	0%	67%	0%	17%	0.00	2.13	0.00	1.00	0.0	1.4	0.0	0.2
B22	unknown		1643	no common descriptor	0%	33%	0%	0%	0.00	2.50	0.00	0.00	0.0	0.8	0.0	0.0
B23	unknown		1740	no common descriptor	0%	33%	0%	0%	0.00	2.00	0.00	0.00	0.0	0.7	0.0	0.0
B24	unknown		1770	no common descriptor	33%	17%	17%	50%	1.00	2.00	2.00	1.67	0.3	0.3	0.3	0.8

Literature and databases for comparison included Ahonen et al. (2022); Aitta et al. (2021); An et al. (2020); Selli et al. (2006); Sérot et al. (2002), NIST Chemistry WebBook, https://webbook.nist.gov, and Flavornet database (Acree and Arn), https://www.flavornet.org.

[a] n = 6.

[b] Identification was based on comparison to a commercial reference compound (std), comparison of ms spectra match (recorded by HS-SPME-GC-MS analysis) to the NIST library (ms), similarity of odor description to previous literature (O), and/or observed LRI close to LRI reported by previous literature or NIST.

accordance with the higher total intensity of odor observed in the raw stored control (Fig. 3) compared to the other samples.

The weighed intensities (intensity*NIF) of the detected odor-active compounds on the semipolar and polar columns were used as predictors of the intensity of the odor attributes of the minces using partial least squares regression (PLS) (Fig. 4). Fishy odor, fish oil odor, marine odor, total intensity of odor, rancidity, and most odorants were associated with C-3d. Many of these compounds are derived from lipid oxidation, such as propanal, hexanal, (*E,Z*)-2,6-nonadienal, (*Z*)-4-heptenal, (*Z*)-1,5-octadien-3-ol, 2,3-pentanedione, and (*E,Z*)-3,5-octadien-2-one, the formation of which was retarded by the addition of SRO (1.5 g/100 g) in the mince. Ethyl-3-methylbutanoate (fruity, fresh, sweet) and two unknown compounds, A2 and A19 (Table 1), detected on the semipolar column correlated with the fruit-like/berry-like odor.

Fishy odor has been acknowledged to be caused by a combination of lipid oxidation-derived aldehydes with low odor thresholds. Many of the odor-active compounds identified in the present study (Tables 1 and 2) have been previously associated with fishy odor (Sae-leaw & Benjakul, 2014; Triqui & Bouchriti, 2003; Venkateshwarlu et al., 2004). (*Z*)-4-heptenal, (*Z*)-1,5-octadien-3-one, and methional were suggested to result in fishy odor to sardine stored on ice for 2 days (Triqui & Bouchriti, 2003). They further reported that due to its low threshold, (*Z*)-1,5-octadien-3-one was also one of the most potent odorants in fresh sardine (stored for 0 d), and during the first two days of storage, 2,3-pentanedione, (*E,Z*)-3,5-octadien-2-one, and (*E,E*)-2,4-decadienal, among other secondary lipid oxidation products, also became more pronounced. Similarly, in a study by Sae-leaw and Benjakul (2014), lipid oxidation of sea bass skin during ice storage led to the development of a fishy odor, which was associated with several volatiles, including hexanal, heptanal, and 1,5-octadien-3-ol. In the present study, (*Z*)-1,5-octadien-3-one, which can be produced during autoxidation of EPA (Hammer & Schieberle, 2013), was detected on both columns by all panelists in all samples. (*Z*)-4-heptenal, known to impart a fishy odor in combination with other volatiles such as (*E,Z*)-2,6-nonadienal (Venkateshwarlu et al., 2004), was detected at the highest frequency and intensity in the stored control mince on both columns.

4. Conclusions

Side streams of berry processing were potential antioxidants in minced Baltic herring during cold storage. The analysis of PV and secondary volatile oxidation compounds showed that a relatively low concentration of press residue in the case of lingonberry-bilberry retarded lipid oxidation in minced Baltic herring stored at 1 °C. The addition of 1.5 g/100 g lingonberry-bilberry juice press residue or sea buckthorn residue after juice pressing and oil extraction prevented increases in fishy odor and fish oil odor, resulting in better preservation of the sensory quality during the 3-day storage of raw mince. Furthermore, the addition of sea buckthorn residue at a dosage of 1.5 g/100 g to fish mince kept the odor intensities low from several lipid oxidation-derived volatile compounds during storage. This study provides new insights into the potential use of berry processing side streams to improve the

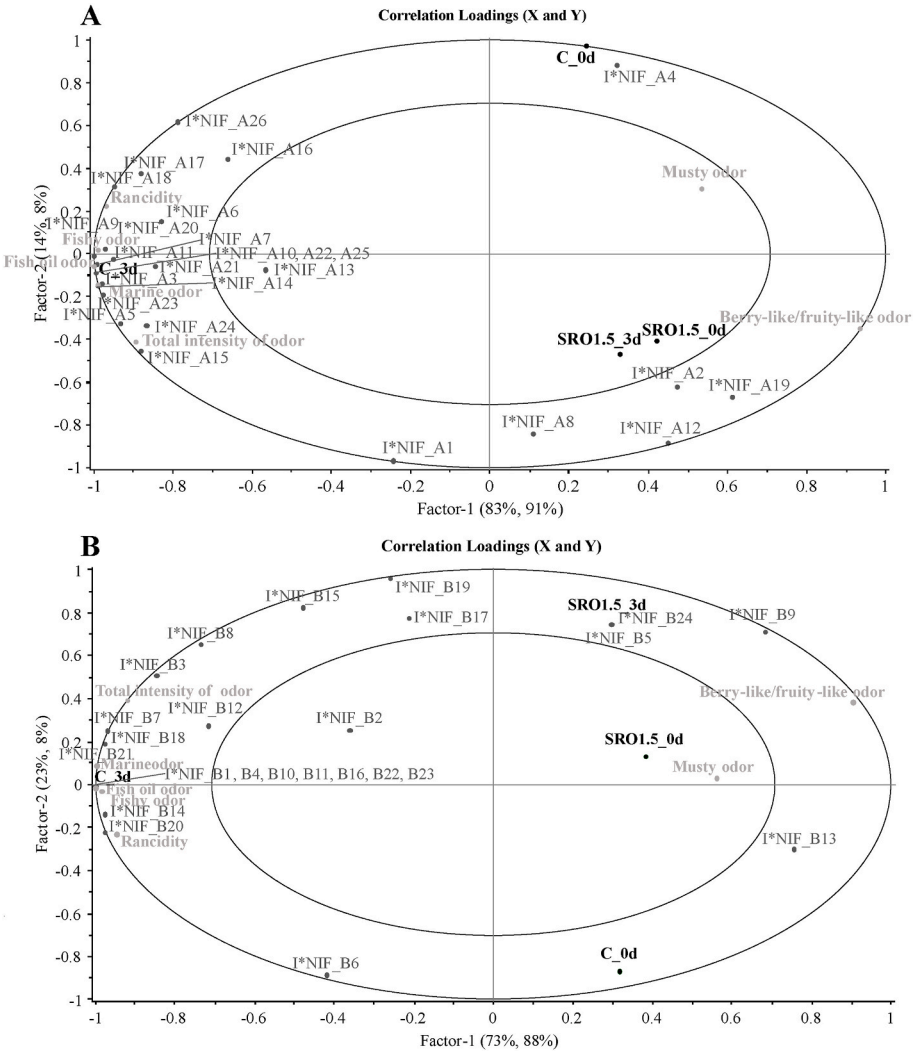

Fig. 4. Partial Least Squares (PLS) regression model with the odor-active compounds (Intensity*Nasal Impact Frequency) on the semipolar SPB-624 (A) or polar DB-WAX (B) column as X variables (n = 26 in case of column A, n = 24 in case of column B), intensities of GDA odor attributes as Y variables (n = 7), and samples as dummy variables. Sample abbreviations refer to raw minces without additions (C) or with 1.5 g/100 g sea buckthorn juice press and oil extraction residue (SRO1.5), after 0 (0d) or 3 days (3d) of storage at 1 °C, and codes for odor-active compounds are the same as in Tables 1 and 2.

stability of fish or fish products during cold storage.

CRediT authorship contribution statement

Tanja Kakko: Conceptualization, Methodology, Formal analysis, Investigation, Writing – original draft, Writing – review & editing, Visualization, Supervision, Funding acquisition. **Annelie Damerau:** Conceptualization, Writing – original draft, Writing – review & editing, Supervision. **Claudia Mejia Rios:** Methodology, Formal analysis, Investigation. **Oskar Laaksonen:** Conceptualization, Writing – original draft, Writing – review & editing, Supervision. **Baoru Yang:** Conceptualization, Methodology, Resources, Writing – review & editing, Supervision, Project administration, Funding acquisition.

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.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.lwt.2023.115044.

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