Shelf-life studies of flavour characteristics in model UHT liquid systems enriched with wholegrain oat

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Abstract

Development of malodourous compounds, hexanal and p-vinyl guaiacol (PVG), in UHT model liquid systems fortified with wholegrain oat were evaluated. Single and mixed systems using oat powder (3, 5, and 7% w/w), skim milk powder (SMP, 2.8% w/w) and sucrose (6.7% w/w) were subjected to UHT treatment and 29 days ambient storage. Both chromatographic analyses and panellists’ perceived aroma intensity show a positive relationship between the content of hexanal and PVG, storage time and oat concentrations trialled. Ratio of the odour activity values (OAV) plotted against time shows that although PVG aroma initially is dominant, hexanal aroma, with a ratio of about 0.5, has twice the intensity of the PVG aroma for the remaining 29 days. Oat samples (with skim milk) were unacceptable when hexanal concentration was 3—5 times its threshold whilst the PVG level was still below its threshold in the same samples.

Keyword: Food science

1. Introduction

Oats are an excellent source of macro- and micro-components that bring considerable benefits to human health including reduction in blood cholesterol and risk of
heart disease as well as protection against colorectal cancer. These advantages are associated with soluble dietary fibre, i.e. β-glucan (Kagimura et al., 2015) and phenolic compounds such as ferulic acid that can scavenge free radicals and serve as antioxidants (Carlotti et al., 2008).

However, increasing oat content in food products also increases off-flavour production during processing and subsequent storage. Of the numerous phenolic acids in oats, the primary one is ferulic acid (Hitayezu et al., 2015). Ferulic acid decarboxylation produces 2-methoxy-4-vinylphenol (PVG), which is an off-flavour compound in various foods, including beer (Cui et al., 2015), due to a clove or spice-like aroma. PVG’s threshold varies with food matrices: 0.3 mg/L in beer (Cui et al., 2015) and 0.1 mg/L in water (Belitz et al., 2009).

Besides the phenolic note, rancidity also adversely affects the sensory quality of oat-based products due to oat’s lipid content. Hexanal, the primary volatile from lipid-oxidation, is considered a rancidity marker in oat and other foods (Azarbad and Jeleni, 2015). It is associated with a grassy odour (Pino and Febles, 2013) with threshold from 3.9 μg/L in water (Ömür-Özbek and Dietrich, 2008) to 135 μg/L in orange juice (Plotto et al., 2004).

However, there is scant information regarding the off-odour development of PVG and hexanal in dairy-based beverages enriched with oat. Furthermore, as manufacturers rely on repeat sales for viability, it is essential to correlate analytical and sensory data in off-flavour studies (Ömür-Özbek and Dietrich, 2008). This study carries out analytical and sensorial tests in model UHT beverages containing wholegrain oat, skim milk and sucrose during storage at ambient temperature to identify the main flavour characteristics (PVG and hexanal) that largely determine the acceptability level for potential commercialisation of these materials.

2. Materials and methods

2.1. Materials

Sanitarium Health and Wellbeing Company (Cooranbong, NSW) supplied the oats, skim milk, and sucrose powders with the approximate composition (w/w) for oat powder being 60.2% total carbohydrate (29.3% dietary fibre), 1% total sugar, 16.8% protein, 9.9% fat, 5.2% ash and 6.9% moisture. Skim milk powder (w/w) had 54% total carbohydrate, 34% protein and 1.3% fat.

The PVG standard (2-methoxy-4-vinylphenol with 98% purity) was purchased from Merck KGaA (Darmstadt, Germany). Sigma Aldrich (Castle Hill, Australia) supplied the hexanal standard (98% purity), solvents (methanol, ethyl acetate and hexane; all with 99.9% purity), and enzymes (α-amylase from Bacillus licheniformis, alcalase protease from Bacillus licheniformis and amyloglucosidase from Aspergillus niger).
2.2. Methods

2.2.1. UHT processing and sample preparation before extraction

All samples (Table 1) were prepared at $22 \pm 1^\circ C$, homogenised at 3,000 psi before heating ($145 \pm 1^\circ C$ for 8 s) using an indirect tubular UHT unit (Hipex, Melbourne, Australia). Samples were then filled into sterile containers in a laminar flow cabinet and stored at $22 \pm 1^\circ C$ for periodic chemical and sensory analyses over 29 days. Oat powder was defatted by twice extracting lipids with hexane.

Samples were centrifuged at $986 \times g$ for 20 min for each sampling. The sediment, except about 3 g which was reserved for lipid-oxidation analysis, was vacuum dried overnight at $37^\circ C$ for phenolic aroma analysis.

2.2.2. Extraction of PVG

Dried precipitate was twice defatted with hexane before mixing with NaOH (2 M, 5 mL) in opaque bottles into which a N$_2$ stream is passed. A rotary shaker was then used for 6 h at $22 \pm 1^\circ C$ in a dark room for saponification. After adjusting the sample to pH 2.0, PVG was extracted with ethyl acetate (twice sample volume, three times), and centrifuged at $1753 \times g$ for 20 min before drying with a rotary evaporator at $40^\circ C$. The extracts were redissolved in methanol/water (50:50 v/v, 1 mL) and filtered (0.45 μm) before chromatographic analysis.

2.2.3. Separation of PVG and hexanal

PVG separation required a HPLC Shimadzu LC-6A liquid chromatograph (Tokyo, Japan) with a reverse phase C18 nonpolar column (Polaris C18-A5μ, PNA 2000 0.46 × 150 mm, 5 μm from Varian, Germany). The 10 min isocratic elution used a mobile phase of methanol:water:acetic acid (49.5%:49.5%:1%). The injection

Table 1. UHT model liquid systems (% w/w).

| Sample | Oat powder | Oat powder (defatted)* | Skim milk powder | Sucrose | Water |
|--------|------------|------------------------|------------------|---------|-------|
| W1     | 3          | –                      | –                | –       | 97    |
| W2     | 5          | –                      | –                | –       | 95    |
| W3     | 7          | –                      | –                | –       | 93    |
| W4     | –          | 3                      | –                | –       | 97    |
| W5     | –          | 5                      | –                | –       | 95    |
| W6     | –          | 7                      | –                | –       | 93    |
| M1     | 3          | –                      | 2.8              | 6.7     | 87.5  |
| M2     | 5          | –                      | 2.8              | 6.7     | 85.5  |
| M3     | 7          | –                      | 2.8              | 6.7     | 83.5  |

*Defatted oat powder is normal oat powder that had been defatted with hexane.
volume was 20 μL and solvent flow rate was 1 mL/min. A detector (Hitachi F1050 fluorescence spectrometer, Tokyo, Japan) with excitation and emission wavelength set as 290 and 335 nm, respectively, was used.

Hexanal was detected by solid phase micro extraction (SPME) coupled with gas chromatography-mass spectroscopy (GC-MS) technique. It operates in electron ionization mode, which can lead to fragmentation of a molecule whose spectral pattern is used for the identification of the sample compound. A 50/30 μm SPME fibre coated with divinylbenzene/carboxen/polydimethylsiloxane (Supelco, USA) was used for headspace analysis. Samples were heated (60 °C) with SPME fibre in the headspace for 20 min before injection into the GC-MS unit where they underwent desorption for 5 min.

For hexanal analysis, a HP 6890 series GC system with mass selective detector HP 5973 (Burwood, Australia) and a non-polar DB-5 column, 0.25 μm, 0.25 mm and 30 m for film thickness, internal diameter, and length, respectively, was used. The column was set at: 50 °C for 2 minutes, 50—110 °C at 10 °C/min, 110—200 °C at 5.7 °C/min, 200—250 °C at 40 °C/min, and 5 min. isothermal step at 250 °C. The carrier gas (helium) at 40 cm/s linear velocity and a splitless injection mode at 250 °C was employed. Compounds were identified with the GC-MS database (National Institute of Standards and Technology, 2011).

### 2.2.4. Quantification of PVG and hexanal

Quantification of aromas was based on calibration curves with PVG standards (10—6,250 μg/L) prepared in methanol/water (50:50) and hexanal standards, 0.05—1,000 μg/L, dissolved in water. Both chemicals were analysed as reported previously. The aroma concentrations, reported as μg/g, were calculated from regression equations (R² ≥ 0.990). Techniques used can detect hexanal and PVG levels to 0.05 μg/L and 7 μg/L, respectively.

### 2.2.5. Sensory evaluation: training of panellists

Shelf-life trials for oat-based samples using aroma intensity and hedonic studies were carried out. Participants (thirty) for the hedonic study were untrained, however, training (2 h × 2) for panellists (fourteen) undertaking the aroma intensity study was conducted over 2 weeks; all panellists were taken from the students and staff of RMIT University in Melbourne, Australia. For aroma familiarisation, concentrations (nine) of odourants in water were prepared from threshold; 100 and 4 μg/L for PVG and hexanal, respectively (Belitz et al., 2009), to maximum expected in this work, which is 8,000 μg/L (80 × threshold; labelled as P80) for PVG and 1,000 μg/L (250 × threshold; labelled as H250) for hexanal.
2.2.6. Aroma (PVG and hexanal) intensity study over 29 days

Aroma intensity in model UHT-treated oat samples (W1–W3 or W4–W6; Table 1) was evaluated regularly, i.e. at intervals of 1, 2, 4, 8, 15, 22 and 29 days (Morais et al., 2014). At each sensory session, panellists were supplied a freshly prepared sample compositionally identical to W1 (or W4 for the defatted trial) in Table 1, which had been heated to 95 °C to familiarize them with the oat aroma to be disregarded. Panellists then matched a sample’s PVG intensity with a PVG reference (0, P1 to P80) where 0 is water only. After palate cleansing, they evaluated the hexanal intensity of that same sample using hexanal’s references (0, H1 to H250). The averaged sensory result (±standard error) of each odorant was plotted against storage time and quantitatively determined PVG and hexanal concentrations for the same samples.

2.2.7. Hedonic study

Panellists determined the acceptability (Antúnez et al., 2016), relative to reference samples, of model UHT-treated liquid samples of oat powder, skim milk powder and sucrose (M1–M3 in Table 1) over 29 days. Reference samples compositionally identical to M1–M3, which were prepared freshly and heated to 95 °C for each sampling were deemed as ‘extremely liked’, thereby scoring 9 on a 9-point hedonic scale. Panellists smelled the reference sample, then the corresponding model sample and recorded the relative acceptability of the model sample. The averaged result (±standard error) was plotted against time, with samples scoring ≤7.5 (i.e. between liked and very much liked) on the 9-point scale deemed as unacceptable.

3. Results and discussion

3.1. Oat aroma profile

A chromatogram of stored UHT-treated single oat samples shows eleven volatile compounds, namely: pentanal, 1-pentanol, hexanal, 1-hexanol, 2-pentyl-furan, nonanal, 2-butanol, octane, heptanal, dodecane and tridecane (data not shown). Klensporf and Jeleń (2008) recorded the presence of the first six volatiles in oat flakes. Volatiles produce distinct odours, e.g. hexanal, the product of 13-hydroxyperoxide cleavage, from linoleic acid autoxidation (Azarbad and Jeleń, 2015), elicits a grassy rancid aroma (Zhu et al., 2016).

The UHT process appears to induce thermal decarboxylation of ferulic acid to PVG, an explanation supported by Arrieta-Baez et al. (2012). Consequently, PVG became the dominant phenolic acid in the UHT-treated oat samples (for pre- and post-UHT treatment; data not shown). This guaiacol compound produces an offensive spice-like medicinal aroma in many food products including juices (Huang et al., 2015).
3.2. Content of hexanal and PVG in single UHT-treated oat systems during storage

The overall hexanal content of samples W1–W3 ranged from 4.5 to 28.8 μg/L, whereas samples W4–W6 (Fig. 1a) only contained from 0 to 1.4 μg/L hexanal during the 29 days of experimentation, indicating that the defatting treatment has effectively removed free oat lipids, which are the predominant lipids present (Przybylski, 2006). Hexanal, in defatted samples, is attributed to the release during storage of bound oat lipids, which are hydrolysed into free fatty acids that are subsequently oxidized into hexanal.

A positive relationship exists between oat concentration and hexanal level in all samples (Fig. 1a). Thus, raising the oat concentration, e.g. from 3% w/w (W1) to 7% w/w (W3), increases the hexanal content from 4.5 to 11.4 μg/L, respectively, in samples kept for 2 days. Due to oxidative degradation of unsaturated free fatty acids (Azarbad and Jeleń, 2015), time, also, enhances hexanal development in all samples; e.g. for sample W3, hexanal increases from 10.7 μg/L on day 1 to 28.8 μg/L on day 29.

PVG development studies (Averbeck and Schieberle, 2011; Klimczak and Małecka, 2011) in citrus juice show that even for the same product, fruit or processing differences do alter PVG synthesis rate. Therefore, examining PVG changes during storage of food matrices containing oat is meaningful.

Traces of PVG, 14.5–47.4 μg/L (Fig. 1b), are detected on day 1 in all oat samples (W1–W6) indicating that UHT processing induces ferulic acid degradation into PVG relatively rapidly. To the best of our knowledge, this is the first time that PVG is associated with a UHT-processed oat product.

Like hexanal, PVG levels show a positive relationship with oat concentration and time (Fig. 1b). When oat concentration is raised from 3% w/w (W1) to 7% w/w (W3), PVG level for two-day samples was 22.4 and 50.0 μg/L, respectively. After 29 d, PVG level increased (4- to 6-fold), which supports Wang et al. (2011) who showed that PVG increased 2-fold from 200 to 420 μg/L over 28 days (at 35 °C), in orange juice. Defatting does not affect PVG levels which ranged from 14.5 to 241.6 μg/L in W1–W3 (not defatted oat), and 14.6–237.4 μg/L in W4–W6 (defatted oat) over 29 days.

Interestingly, PVG content and development (Fig. 1b) is greater than that of hexanal (Fig. 1a) in single oat samples during storage. For instance, there is an 8.5- and 3.8-fold increase in PVG and hexanal content, respectively for W1 samples over 29 days suggesting that ferulic acid decarboxylation occurs rapidly compared with oxidation of unsaturated fatty acids.
3.3. Perceived intensity of hexanal and PVG in single UHT-treated oat samples

The panellists’ perceived intensity of hexanal (Fig. 2a) in UHT-treated samples (W1—W6, the composition of these samples is given in Table 1) stored at 22 °C for 29 days. Analysis for one-day samples was performed 24 hours after UHT processing. Data are expressed as the mean of triplicate measurements ± standard error.

Fig. 1. Content of (a) hexanal and (b) PVG in single UHT-treated oat samples (W1—W6, the composition of these samples is given in Table 1) stored at 22 °C for 29 days. Analysis for one-day samples was performed 24 hours after UHT processing. Data are expressed as the mean of triplicate measurements ± standard error.

3.3. Perceived intensity of hexanal and PVG in single UHT-treated oat samples

The panellists’ perceived intensity of hexanal (Fig. 2a) in UHT-treated samples (with normal oat powder, W1—W3) increased as a function of time and oat concentration trialled. The average perceived intensity of hexanal in samples W1—W3 increases 4.5- to 5.0-fold over 29 days; starting with 6.0–8.0 µg/L on day 1. The trend for
the perceived intensity of PVG (Fig. 2b) follows that of hexanal. However, defatting oats depresses panellist’s assessment of PVG intensity. Thus for normal (not defatted) 7% w/w oat sample (W3), the PVG level was assessed as 500 μg/L but that was only 340 μg/L for the corresponding defatted oat sample (W6) on day 15 (Fig. 2b).

Overall in the presence of hexanal, PVG’s perceived intensity increased, possibly due to synergistic interactions (chemical or physical) between components of oat macromolecules and the matrix structure, the odorants (hexanal and PVG), and their precursors. Ferrer-Gallego et al. (2014) discussed odourant interactions with one
another and the substrate material to produce synergistic, antagonistic and suppression effects.

3.4. Correlation between sensory and analytical measurements in single oat samples

Plots (Fig. 3a and b) correlating panellists’ perceived intensity with analytically determined hexanal and PVG odorant levels, respectively, show an initial near-

![Graph](https://example.com/graph.png)

**Fig. 3.** Correlation between (a) hexanal and (b) PVG concentration and its perceived intensity (µg/L) in UHT-treated single oat samples made with normal oat powder (W1–W3, the composition of these samples is given in Table 1) stored at 22 °C for 1, 2, 4, 8, 15, 22 and 29 days, as indicated by the seven symbols in each curve.
vertical section. This suggests that panellists have difficulty assessing odourants near their threshold levels accurately. The hexanal concentration, e.g., for the 3% w/w oat sample (W1) was determined analytically to be 4.5–5.0 μg/L, whereas panellist rated these samples to have up to 3.6-fold more hexanal (6–18 μg/L). A similar trend was recorded for PVG (Fig. 3b). Thomas-Danguin et al. (2014) noted that ‘higher order mixtures’ i.e. beyond binary, are difficult for panellists. In this study, samples are multi-odourant; besides the two odourants in question, the UHT-treated oats have a distinct aroma of their own that panellists’ must disregard.

Beyond the vertical section (Fig. 3a and b); there is a straight-line relationship, with a positive gradient, between the analytical odorant data and the panellists’ perceived intensity level. However, for the same sample, the odourant concentration as determined analytically was less than the perceived odourant intensity. For example, the perceived hexanal intensity for W1–W3 (Fig. 2a) increases 4.5- to 5.0-fold (6 μg/L initially to 40 μg/L) but based on analytical data (Fig. 1a) for the same samples, the hexanal level increases only 2.7- to 3.8-fold (4.5–10.7 μg/L to 17.1–28.8 μg/L) over 29 days. As odourants can interact with each other and the background matrix to intensify their perception (Ferrer-Gallego et al., 2014), it is proposed that UHT-treated oat matrix ‘enhances’ the hexanal and PVG off-odor via synergistic interactions within the matrix. This is supported by the previous result of hexanal (in the normal oat samples) enhancing panellists’ perception of PVG level.

3.5. The relative odour activity values (OAV) of hexanal and PVG

Odour activity value (OAV) which enables comparison of the intensity of various odours (Miyazawa et al., 2012) is the perceived intensity/the threshold value of 4 μg/L for hexanal (or 100 μg/L for PVG). Significantly, the relationship between the OAV of hexanal and PVG (Fig. 4) is independent of the oat concentrations trialled. Thus, a manufacturer can alter product formulations within this oat range without concern for a shift in off-flavour development.

The ratio of OAV of PVG to OAV of hexanal plotted as a function of time (Fig. 5) shows that initially PVG aroma dominates. After that, hexanal, rather than PVG odour, was perceived as the more intense as an OAV ratio of about 0.5 indicates hexanal as having twice the aroma ‘strength’ of PVG. PVG aroma dominates initially because ferulic acid decarboxylates to PVG faster than oxidation of lipids and additionally, ferulic acid, being an antioxidant, reduces hexanal development by free fatty acid oxidation. However, utilisation of ferulic acid in other reactions decreases its availability enabling then hexanal to dominate.
Fig. 4. Correlation between the odour activity values (OAV) of hexanal and PVG in single UHT-treated oat samples prepared with normal oat powder (W1–W3, the composition of these samples is given in Table 1) stored at 22 °C for 29 days. Odour activity value (OAV) is the ratio of individual odour’s perceived intensity and its respective sensory threshold.

\[ y = 0.0197x^2 + 0.1946x + 1.7671 \]  
\[ R^2 = 0.9932 \]

\[ y = 0.0405x^2 - 0.0138x + 1.9078 \]  
\[ R^2 = 0.9771 \]

\[ y = 0.0263x^2 + 0.0726x + 1.341 \]  
\[ R^2 = 0.9926 \]

Fig. 5. Relative contribution of PVG’s OAV to hexanal’s OAV over 29 days of storage at 22 °C.
3.6. Hexanal and PVG content during storage in mixed UHT-treated samples

Hexanal and PVG development in mixed and single oat samples as a function of storage time and oat concentrations follow similar trends. In sample M1, hexanal and PVG levels increase about 3.8-fold over 29 days from 3.8 μg/L (Fig. 6a) and 9.4 μg/L (Fig. 6b), respectively. Raising the oat content from 3% w/w (M1) to 7%

Fig. 6. Content of (a) hexanal and (b) PVG in mixed UHT-treated samples (M1–M3, the composition of these samples is given in Table 1) stored at 22 °C for 29 days. Analysis for one-day samples was performed 24 hours after UHT processing. Data are expressed as the mean of triplicate measurements ± standard error.
w/w (M3) increases hexanal level from 4.1 to 11.1 μg/L (Fig. 6a) and PVG from 11.9 to 18.4 μg/L (Fig. 6b), respectively, after 2 days storage.

However, the addition of SMP and sucrose reduces hexanal development; e.g. oat only samples (W1–W3) have 4.5–28.8 μg/L (Fig. 1a) whereas M1–M3 samples that have SMP and sucrose produce 3.8–21.6 μg/L (Fig. 6a) of hexanal. Similarly, PVG level is 123.3 μg/L (Fig. 1b) in single oat samples (W1) after 29 days, yet, the equivalent mixed sample (M1) produced only 35.8 μg/L (Fig. 6b) of PVG. Wang and Arntfield (2015) demonstrated that heating promoted binding between hexanal and canola protein isolates. UHT treatment which induces milk protein unfolding exposing sites that now could be available for complexation with volatile compounds. Reiners et al. (2000) found that flavours including PVG bind with β-lactoglobulin and suggest that this binding occurs at the protein’s hydrophobic sites.

PVG development is more depressed by milk proteins and sucrose than hexanal. For example, there is 75.7% less PVG in mixed (M2) samples kept for 29 days compared with equivalent single oat (W2) samples (Figs. 6b and 1b, respectively), but in the same samples only 14.5% less hexanal (Figs. 6a and 1a). This outcome suggests that protein and PVG form a stronger interaction than the protein-hexanal counterparts. In addition, the PVG levels upon incorporation of SMP and sucrose could feasibly be affected by the interaction between oxidized ferulic acid, forming a semi-quinone, and SMP leading to a reduction in the availability of ferulic acid (precursor of PVG) for the thermal decarboxylation process. Recently, Kaur et al. (2018) stated that in milk protein-phenolic acid solutions, heat treatment oxidized phenolic acids leading to the formation of covalent bonds between sulphhydryl or amino groups of the protein and quinones.

3.7. Acceptability of mixed UHT-treated samples

Relative to fresh oat samples (rated 9, extremely liked), the mixed UHT-treated oat samples achieve an average score of 8 (very much liked) to 6.86 (<liked) over 29 days (Table 2). Mixed oat samples (3, 5 and 7% w/w) are deemed unacceptable after 27, 26, and 21 days storage, respectively when the hedonic score is ≤7.5 (i.e. <very much liked). A product’s acceptability threshold is set at 7.5; higher than ‘liked’, because commercial viability relies on repeat purchasing (Wettstein and Hanf, 2009).

When deemed unacceptable, mixed oat samples have a PVG content below threshold level (100 μg/L) at 32.7, 40.6 and 46.0 μg/L for 3, 5, and 7% w/w (M1–M3) samples, respectively (Fig. 6b). However, the same samples have hexanal levels, which are 3–4 times threshold level (4 μg/L), at 13.1, 17.0 and 18.0 μg/L for M1–M3, respectively (Fig. 6a). This data supports the OAV trend indicating that hexanal, rather than PVG, is the dominant off-flavour aroma.
Table 2. Acceptability\(^a\) based on a 9-point hedonic scale of fresh and stored UHT-treated samples.

| Storage time | Hedonic score\(^b\) for the following mixed UHT-treated samples |
|--------------|---------------------------------------------------------------|
| Days at room temperature | M1\(^b\) | M2\(^b\) | M3\(^b\) |
| 1 (fresh) | 7.91 ± 0.15 | 7.90 ± 0.14 | 8.03 ± 0.17 |
| 2 | 7.75 ± 0.17 | 7.54 ± 0.18 | 8.00 ± 0.18 |
| 4 | 7.63 ± 0.14 | 8.05 ± 0.14 | 8.00 ± 0.12 |
| 8 | 7.88 ± 0.14 | 7.90 ± 0.14 | 8.03 ± 0.15 |
| 15 | 7.85 ± 0.19 | 7.66 ± 0.21 | 7.50 ± 0.23 |
| 22 | 7.60 ± 0.19 | 7.40 ± 0.21 | 7.11 ± 0.23 |
| 29 | 7.11 ± 0.20 | 7.08 ± 0.21 | 6.86 ± 0.26 |

\(^a\)Results are expressed as mean of thirty measurements ± standard error.

\(^b\)Composition of samples M1–M3 is given in Table 1.

### 4. Conclusions

Off-flavour characteristics in oat-based products are usually associated with hexanal but, for the first time, this study shows the evolution of PVG that contributes phenolic off-flavour in UHT-treated oat-based products. Results show that aroma-protein complexes, possibly promoted by UHT, depress odourant development, with PVG levels being more affected than hexanal levels. So, hexanal aroma (feasibly, in conjunction with other aroma compounds also produced by linoleic acid oxidation) rather than PVG aroma, dominates during storage and consequentially affects acceptability of oat samples with SMP; this is despite PVG being enhanced by hexanal and developing faster than hexanal with time.

Multi-odourant systems, as in this study of UHT–treated oats, create challenges for panellists. This relates to the accurate intensity assessment near the aroma threshold when compared to analytically determined concentrations. This emphasises the need for further studies that correlate fundamental aroma analysis with data based on panellist intensity perception, to improve our understanding of this relationship.

### Declarations

**Author contribution statement**

Nashi K. Alqahtani, John Ashton, Lita Katopo, Elisabeth Gorczyca, Stefan Kasapis: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

References

Antúnez, L., Giménez, A., Ares, G., 2016. A consumer-based approach to salt reduction: case study with bread. Food Res. Int. 90, 66–72.

Arrieta-Baez, D., Dorantes-Álvarez, L., Martinez-Torres, R., Zepeda-Vallejo, G., Jaramillo-Flores, M.E., Ortiz-Moreno, A., Aparicio-Ozores, G., 2012. Effect of thermal sterilization on ferulic, coumaric and cinnamic acids: dimerization and antioxidant activity. J. Sci. Food Agric. 92, 2715–2720.

Averbeck, M., Schieberle, P., 2011. Influence of different storage conditions on changes in the key aroma compounds of orange juice reconstituted from concentrate. Eur. Food Res. Technol. 232, 129–142.

Azarbad, M.H., Jelen, H., 2015. Determination of hexanal—an indicator of lipid oxidation by static headspace gas chromatography (SHS-GC) in fat-rich food matrices. Food Analytical Methods 8, 1727–1733.

Belitz, H.-D., Grosch, W., Schieberle, P., 2009. Aroma compounds. In: Food Chemistry. Springer, Berlin, pp. 340–402.

Carlotti, M.E., Sapino, S., Ugazio, E., Peira, E., Vione, D., Minero, C., 2008. Photostability of ferulic acid and its antioxidant activity against linoleic acid peroxidation. J. Dispers. Sci. Technol. 29, 629–640.

Cui, Y., Wang, A., Zhang, Z., Speers, R.A., 2015. Enhancing the levels of 4-vinylguaiacol and 4-vinylphenol in pilot-scale top-fermented wheat beers by response surface methodology: enhancing the levels of 4VG and 4VG in top-fermented wheat beers by RSM. J. Inst. Brew. 121, 129–136.

Ferrer-Gallego, R., Hernández-Hierro, J.M., Rivas-Gonzalo, J.C., Escribano-Bailón, M.T., 2014. Sensory evaluation of bitterness and astringency sub-qualities of wine phenolic compounds: synergistic effect and modulation by aromas. Food Res. Int. 62, 1100–1107.
Hitayezu, R., Baakdah, M.M., Kinnin, J., Henderson, K., Tsopmo, A., 2015. Antioxidant activity, avenanthramide and phenolic acid contents of oat milling fractions. J. Cereal. Sci. 63, 35–40.

Huang, X.-C., Yuan, Y.-H., Guo, C.-F., Gekas, V., Yue, T.-L., 2015. Alicyclobacillus in the fruit juice industry: spoilage, detection, and prevention/control. Food Rev. Int. 31, 91–124.

Kagimura, F.Y., da Cunha, M.A.A., Barbosa, A.M., Dekker, R.F.H., Malfatti, C.R.M., 2015. Biological activities of derivatized d-glucans: a review. Int. J. Biol. Macromol. 72, 588–598.

Kaur, J., Katopo, L., Hung, A., Ashton, J., Kasapis, S., 2018. Combined spectroscopic, molecular docking and quantum mechanics study of β-casein and p-coumaric acid interactions following thermal treatment. Food Chem. 252, 163–170.

Klensporf, D., Jeleń, H.H., 2008. Effect of heat treatment on the flavor of oat flakes. J. Cereal. Sci. 48, 656–661.

Klimczak, I., Malecka, M., 2011. Evaluation of sensory profile and p-vinylguaiacol (PVG) content in orange juices during storage at different temperature: evaluation of sensory profile and PVG in orange juices. J. Food Qual. 34, 30–39.

Miyazawa, M., Hashidume, S., Takahashi, T., Kikuchi, T., 2012. Aroma evaluation of Gamazumi (Viburnum dilatatum) by aroma extract dilution analysis and odour activity value: aroma-active compounds in Viburnum dilatatum. Phytochem. Anal. 23, 208–213.

Morais, E.C., Cruz, A.G., Faria, J.A.F., Bolini, H.M.A., 2014. Prebiotic gluten-free bread: sensory profiling and drivers of liking. LWT Food Sci. Technol. 55, 248–254.

Ömür-Özbek, P., Dietrich, A.M., 2008. Developing hexanal as an odor reference standard for sensory analysis of drinking water. Water Res. 42, 2598–2604.

Pino, J.A., Febles, Y., 2013. Odour-active compounds in banana fruit cv. Giant Cavendish. Food Chem. 141, 795–801.

Plotto, A., Margaria, C.A., Goodner, K.L., Goodrich, R., Baldwin, E.A., 2004. Odour and flavour thresholds for key aroma components in an orange juice matrix: terpenes and aldehydes: odour/flavour thresholds for terpenes and aldehydes. Flavour Fragrance J. 19, 491–498.

Przybylski, R., 2006. Cereal grain oils. In: Shahidi, F. (Ed.), Nutraceutical and Specialty Lipids and Their Co-products. CRC/Taylor & Francis, Boca Raton.
Reiners, J., Nicklaus, S., Guichard, E., 2000. Interactions between β-lactoglobulin and flavour compounds of different chemical classes. Impact of the protein on the odour perception of vanillin and eugenol. Lait 80, 347–360.

Thomas-Danguin, T., Sinding, C., Romagny, S., El Mountassir, F., Atanasova, B., Le Berre, E., Le Bon, A., Coureaud, G., 2014. The perception of odor objects in everyday life: a review on the processing of odor mixtures. Front. Psychol. 5, 1–18.

Wang, K., Arntfield, S.D., 2015. Binding of selected volatile flavour mixture to salt-extracted canola and pea proteins and effect of heat treatment on flavour binding. Food Hydrocoll. 43, 410–417.

Wang, Q., Gao, X., Gong, H., Lin, X., Saint-Leger, D., Senee, J., 2011. Chemical stability and degradation mechanisms of ferulic acid (F.A) within various cosmetic formulations. J. Cosmet. Sci. 62, 483–503.

Wettstein, N., Hanf, J.H., 2009. What are “true” loyal customers in the food sector? Insights from an empirical study. In: European Association of Agricultural Economists. Presented at the 113th EAAE Seminar “A Resilient European Food Industry and Food Chain in a Challenging world.” Crete, Greece.

Zhu, J., Chen, F., Wang, L., Niu, Y., Chen, H., Wang, H., Xiao, Z., 2016. Characterization of the key aroma volatile compounds in cranberry (Vaccinium macrocarpon Ait.) using gas chromatography—olfactometry (GC-O) and odor activity value (OAV). J. Agric. Food Chem. 64, 4990–4999.