# Optimising water activity for storage of high lipid and high protein infant formula milk powder using multivariate analysis

Hong Cheng [a, c], Henriette Erichsen [c], John Soerensen [b], Mikael Agerlin Petersen [c], Leif H. Skibsted [c, *]

[a] College of Biology Science and Engineering, Hebei University of Economics and Business, No.47 Xuefu Road, Shijiazhuang 050061, China
[b] Arla Foods R&D, Arla Foods, Rørdrumvej 2, DK-8220 Brabrand, Denmark
[c] Department of Food Science, University of Copenhagen, Rolighedsvej 30, DK-1958 Frederiksberg C, Denmark

ABSTRACT

High lipid and high protein infant formula milk powders were stored at water activity of 0.11, 0.33 and 0.53 for up to fourteen weeks at 40 °C to investigate the effect of storage water activity on physico-chemical properties and formation of volatiles to thereby recommend optimal storage water activity conditions. Water activity of the powders was determined during storage together with surface colour, glass transition temperature combined with dynamic headspace sampling followed by gas chromatography/mass spectrometry. The principal component analysis (PCA) showed that the optimal water activity for storage of high lipid infant formula milk powder, for which lipid oxidation was found to be the critical quality parameter, is $a_w = 0.33$ with lowest lipid oxidation, while for high protein infant formula milk powder, for which protein degradation was found to be the critical quality parameter, $a_w = 0.11$ is optimal to limit formation of Maillard reaction products.

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## 1. Introduction

Storage stability of infant formula milk powders is dependent on many factors including powder composition, handling of powders, and storage conditions (Tham, Yeoh, & Zhou, 2017). Quality deterioration is caused by both physical changes and chemical reactions during storage and has been assigned mainly to lactose crystallisation, lipid oxidation, and Maillard reactions (nonenzymatic browning), all strongly affected by storage temperature and water activity (Novaes et al., 2019; Stapelfeldt, Nielsen, & Skibsted, 1997; Thomsen, Lauridsen, Skibsted, & Risbo, 2005).

Lactose crystallisation in milk powder during storage increases the amount of free fat and may also promote non-enzymatic browning due to the increasing water activity linked to a lower glass transition temperature (Nasirpour, Scher, Linder, & Desobry, 2006). The glassy state after spray drying will collapse at higher temperature and water activity, resulting in lactose crystallisation during longer storage (Tham et al., 2017; Thomsen et al., 2005).

Docosahexaenoic acid (DHA) and eicosapentaenoic acid (EPA) are often added to infant formula milk powder (Cesa, Casadei, Cerreto, & Paolicelli, 2015). These polyunsaturated fatty acids (PUFAs) are easily oxidised to secondary lipid oxidation products such as aldehydes, ketones and alcohols, which are potentially toxic and with unpleasant flavours.

Storage temperature and water activity have a strong effect on reaction rates for formation of various lipid oxidation products. Formation of hexanal and pentanal was found to be accelerated 5 times when storage temperature rose from 25 °C to 55 °C (Cheng et al., 2017). The effect of water activity on lipid oxidation is less clear (Stapelfeldt et al., 1997). Water seems to: (i) act as a solvent, (ii) mobilise prooxidative reactants and (iii) interact with more hydrophilic lipid oxidation products (Labuza & Dugan, 1971). These different effects may counteract each other leading to an increasing lipid oxidation for very low and for high water activity and with an optimal stability against lipid oxidation at intermediate water activities.

Proteins are differentially degraded by Maillard reactions depending on water activity and temperature in a manner different from that for lipid oxidation. The early stage of the browning reactions begins with condensation between reducing

* Corresponding author. Tel.: +45 3533 3221.
E-mail address: ls@food.ku.dk (L.H. Skibsted).

sugars and free amino groups in proteins followed by subsequent rearrangement leading to formation of Amadori compounds. Intermediate stages in the browning reactions lead to formation of compounds like hydroxymethylfurfural, and α-dicarbonyl and α-amino carbonyl compounds by reactions like cyclisation, sugar dehydration, fragmentation, enolisation and oxidation. At the final stage, reactions proceed further producing Strecker aldehydes, pyrazines, pyrroles, oxazoles and polymeric aggregates called advanced glycation end products (AGEs) (Cortés, Gagneten, Leiva, & Malec, 2017; Lee, Ho, Khoo, & Chow, 2012; Poulsen et al., 2013; Zhu et al., 2018).

The objective of the current study was to identify optimal water activity conditions for storage of two types of infant formula milk powder (high lipid and high protein). Therefore, gas chromatography-mass spectrometry (GC-MS) was used to identify the volatile compounds during storage. Principal component analysis was used to visualise analytical data in reduced-dimension plots (Delgado, González-Crespo, Cava, García-Parra, & Ramírez, 2010; Xu et al., 2014). Detailed knowledge of the effect of water activity on chemical transformations in different types of milk powders will be of increasing importance for optimising packaging and storage conditions.

## 2. Materials and methods

### 2.1. Chemicals

Lithium chloride, magnesium chloride, sodium bromide, and 4-methyl-1-pentanol were all purchased from Sigma–Aldrich (Steinheim, Germany).

### 2.2. Infant formula milk powder samples

Both high lipid infant formula milk powder (HLMP, for 0–6 month old infants) and high protein infant formula milk powder (HPMP, for 12–36 month old infants) based on Danish bovine milk were produced by Arla Foods amba, Viby J, Denmark as standard products. The main nutrient content of these two types of infant formula milk powders may be found in Table 1.

### 2.3. Sample preparation

Around 25 g of infant formula milk powder was transferred into 50 mL open beakers. The beakers were immediately placed in closed glass desiccators with saturated lithium chloride, magnesium chloride or sodium bromide aqueous solutions for control of water activity at 0.11, 0.33 and 0.53, respectively. The desiccators were stored at 40 °C in incubator chambers with sampling every two weeks for 14 weeks. There were 96 samples in total. The open beakers were closed with lids after sampling and placed in a refrigerator at 4 °C.

### 2.4. Water activity

The water activity of samples was determined after sampling according to the method described by Cheng et al. (2017). Around 1.5 g of milk powder samples were measured by an Aqua Lab CX-2 (Aqua Lab, Pullman WA. U.S.A.). All the measurements were done in duplicate.

### 2.5. Colour

The colour of the samples was determined after sampling according to the method described by Cheng et al. (2017). Hunter L, Hunter a and Hunter b were measured by A Byk-Gardner colour guide system (Byk-Gardner CB-6692, Geretsried, Germany). Only Hunter b was used to follow increasing yellow and brown colour. All the measurements were done in duplicate.

### 2.6. Glass transition

The glass transition temperature was determined according to the method described by Cheng et al. (2017). Around 15 mg milk powder samples placed in 40 μL aluminium vessels were subjected to conventional DSC using a DSC 820 (Mettler Toledo, Schwerzenbach, Swizerland) with a scanning rate of 10 °C min$^{-1}$ from 0 °C to 100 °C. Thermograms for determination of Tg were recorded induplicate for the first two samples for each of the experimental series.

### 2.7. Volatile compound and principal component analysis

Volatile compounds were determined after the end of sampling according to the method described by Cheng et al. (2017). Aroma compounds in the infant formula milk powder were extracted using dynamic headspace sampling (DHS), and a GC-MS system (7890A GC with a ZB-Wax capillary column, 30 m × 0.25 mm × 0.5 μm, interfaced with a 5975C VL MSD with Triple-Axis detector from Agilent Technologies, Palo Alto, CA, USA) was used to analyse the collected volatile compounds. Peak areas and mass spectra were extracted from the chromatograms using the PARAFAC2 based software PARADISe (University of Copenhagen, Copenhagen, Denmark) and mass spectra were identified using the NIST05 database. Peak areas were used as relative measures of intensity compared with the internal standards of 4-methyl-1-pentanol. Volatile compound identification was confirmed by comparison with retention indices (RI) of authentic reference compounds or retention indices reported in the literature.

Principal component analysis (PCA) was performed to investigate the relationships between the storage conditions, storage time and formation of volatile compounds using the software Latentix 2.12 (Latent5, Copenhagen, Denmark). All the measurements were done in duplicate.

**Table 1**
Composition of high lipid infant formula milk powder (HLMP) and high protein infant formula milk powder (HPMP).[a]

| Formula | Protein (g) | Casein (g) | Whey protein (g) | Fat (g) | Linoleic acid (g) | α-Linolenic acid (mg) | Docosahexaenoic acid (mg) | Arachidonic acid (mg) | Carbohydrate (g) | Lactose (g) | FOS (g) | GOS (g) |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| HLMP | 10.3 | 3.1 | 7.2 | 28 | 4.9 | 650 | 56 | 84 | 50.5 | 49 | 0.46 | 4.2 |
| HPMP | 16.5 | 11.9 | 4.6 | 17 | 1.8 | 200 | 10 | 20 | 56 | 49 | 0.11 | 0.99 |

[a] Values given are per 100 g powder. Abbreviations are: FOS, fructose oligosaccharides; GOS, galactose oligosaccharides.

## 3. Results and discussion

### 3.1. Water activity

Two types of infant formula milk powder, HLMP and HPMP, were stored at water activities of 0.11, 0.33 and 0.53 to study the effect on storage stability. The initial water activity of both types of powder was 0.2. The water activity increased to reach the storage water activity of 0.32 and 0.53, indicating that moisture sorption took place. For storage at water activity 0.11, the powders were dried out to reach the desired water activity, as may be seen in Fig. 1. Absorption and desorption of water occurs with slightly different rates depending on the water activity of storage.

### 3.2. Colour

The colour changes of infant formula milk powders were used as a marker of the progress of the Maillard reactions based on several studies showing that Maillard reactions can be followed by the colour changes of milk powders (Cortés et al., 2017; Norwood et al., 2016). In previous studies, we have shown that the relative importance of lipid oxidation and browning reactions for infant formula milk powders during the storage time strongly depends on temperature with browning reactions dominating at higher temperature (Cheng et al., 2017; Zhu et al., 2018). The current study focused on the effect of water activity on browning and lipid oxidation at the intermediate temperature of 40 °C for which both types of reactions are important. As shown in Fig. 2, colour of the two types of infant formula milk powders stored at water activity of 0.33 showed only a slight increase with storage time and even less for a water activity of 0.11. However, infant formula milk powders stored at water activity of 0.53 showed a rapid increase from start of storage until the eighth week, eventually reaching a more constant value. The water activity is clearly crucial to the browning of infant milk powder. At the higher storage water activity, lactose will absorb more water promoting the amorphous lactose to undergo crystallisation, further increasing non-enzymatic Maillard reactions leading to the brown colour (Kim, Saltmarch, & Labuza, 1981; Tham et al., 2017). Notably, for all three water activity
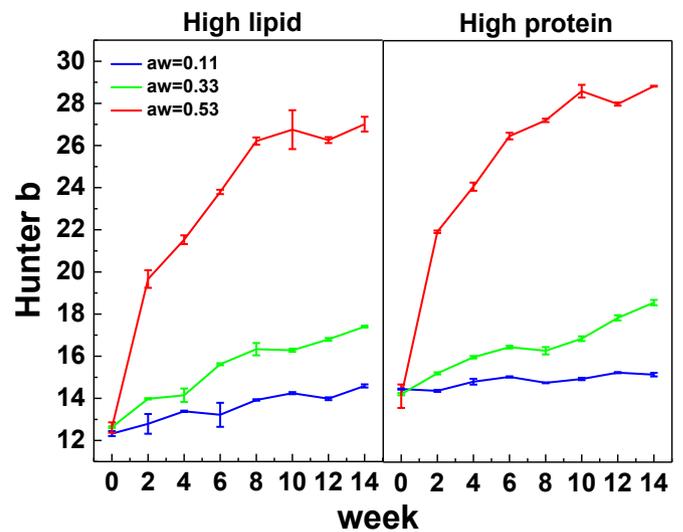


**Fig. 2.** Surface browning of HLMP (high lipid infant formula milk powder) and HPMP (high protein infant formula milk powder) stored at 40 °C in beakers placed in closed desiccators with saturated LiCl, MgCl₂ and NaBr solution with water activity of 0.11, 0.33 and 0.53, respectively.

conditions, HPMP turned browner than HLMP, see Fig. 2, in agreement with the higher content of protein amino groups as substrates for the Maillard reaction.

### 3.3. Glass transition

Infant formula milk powders produced by spray drying go through a rapid increase in viscosity hindering crystallisation, and finally leading to the formation of amorphous lactose (Haque & Roos, 2006; Tham et al., 2017). Amorphous lactose is hygroscopic and easily absorbs water and will undergo crystallisation. This second-order phase transition may be determined by a change in the glass transition temperature ($T_g$) measured by differential scanning calorimetry (DSC) (Schmitz, Gianfrancesco, Kulozik, & Foerst, 2011). As shown in Fig. 3, $T_g$ of infant formula milk powders stored at water activity of 0.11 showed a slight increase with the time in $T_g$ until the eighth week, reaching 72 °C for HLMP and 70 °C for HPMP. However, at storage water activity of 0.33, $T_g$ of the powders declined to 40 °C for HLMP, and to 35 °C to disappear at the tenth week for HPMP. In addition, Tg around 53 °C for both types of infant formula milk powders at storage water activity of 0.53 disappeared at the second week of storage, as explained by a more rapid absorption of water at the higher water activity. Water will, as the plasticiser increases the molecular mobility, promote the second-order phase transition (Cheng et al., 2017; Tham et al., 2017), leading to a lowering or disappearance of Tg at the higher water activity. HPMP contains more hydrophilic proteins such as β-lactoglobulin, which absorbs more water, in effect making its Tg slightly lower than for HLMP (Nasirpour et al., 2006).

### 3.4. Volatile compound and principal component analysis

A total of 51 volatile compounds were identified in the milk powders during storage (Table 2). To get an overview of the changes, a principal component analysis (PCA) was conducted and the resulting score and loadings plots are shown in Fig. 4 (PC1 versus PC2, explaining 70.3% of the variance). All samples start with a negative score in PC1 and a score close to zero in PC2. During storage, all samples exhibit an increase in PC1 score, while PC2
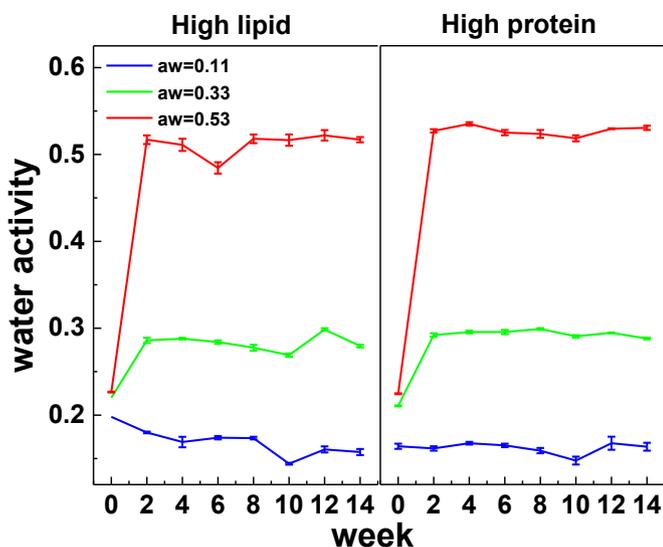


**Fig. 1.** Water activity of HLMP (high lipid infant formula milk powder) and HPMP (high protein infant formula milk powder) stored at 40 °C in beakers placed in closed desiccators with saturated LiCl, MgCl₂ and NaBr solution with water activity of 0.11, 0.33 and 0.53, respectively.
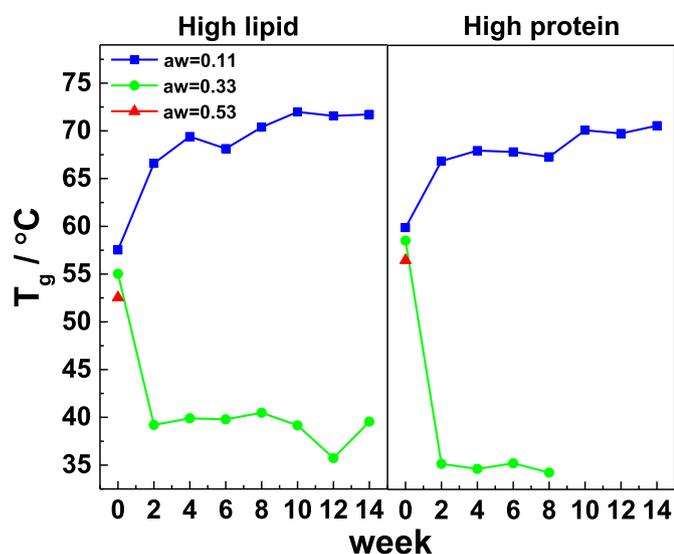
**Fig. 3.** Glass transition of HLMP (high lipid infant formula milk powder) and HPMP (high protein infant formula milk powder) stored at 40 °C in beakers placed in closed dessicators with saturated LiCl, $MgCl_2$ and NaBr solution with water activity of 0.11, 0.33 and 0.53, respectively.

scores develop differently. Since all volatile compounds have positive PC1 loadings, which means that almost all volatiles increase during storage but at different rates. The volatile patterns after storage are, therefore, different depending on the storage conditions. All linear aldehydes, which are typical lipid oxidation products (Fabiano, Roschel, & Castro, 2018; García-Moreno, Guadix, Guadix, & Jacobsen, 2016; Purriños, Bermúdez, Franco, Carballo, & Lorenzo, 2011), have positive PC1 loadings (placed to the right in the loadings plot). Among the HLMP samples, those stored at $a_w = 0.53$ move most to the right, which indicates the highest increase in the linear aldehydes. HLMP samples stored at $a_w = 0.11$ move less, and samples stored at $a_w = 0.33$ move the least, i.e., $a_w = 0.33$ leads to the lowest formation of lipid oxidation products. Among the HPMP, samples stored at $a_w = 0.53$ and 0.33 results in a movement towards negative PC2 scores. This indicates an increase in compounds with negative PC2 loadings, for example, 2- and 3-methylbutanal, dimethyldisulphide and dimethyltrisulphide, which are typical products of amino acid breakdown. Benzaldehyde and furfural are located in the same area of the loadings plot. HPMP samples stored at $a_w = 0.11$ have increasing scores in PC1 as well as PC2 (moving up-right), which indicates no or very limited formation of products from amino acid breakdown. To give a more detailed picture, the changes in the content of some selected compounds are shown individually in Fig. 5.

The formation of hexanal and (E,E)-2,4-nonadienal is much more pronounced in HLMP samples, and especially when stored at $a_w = 0.53$; see Fig. 5a,b. The lowest formation of hexanal and (E,E)-2,4-nonadienal is seen at $a_w = 0.33$. Some of the unsaturated linear aldehydes had the highest increase when stored at $a_w = 0.11$, but for all linear aldehydes storage at $a_w = 0.33$ resulted in lowest formation.

Hexanal is formed by the oxidation of linoleic acid, and heptanal is also generated in n-6 PUFA oxidation, while nonanal is from n-9 PUFA oxidation (Duflos, Coin, Cornu, Antinelli, & Malle, 2006; Prost, Hallier, Cardinal, Serot, & Courcoux, 2004; Xu et al., 2014). Hexanal with green, grassy and fatty odour and pentanal with woody, pungent, and fruity flavour have been identified as early signs of quality deterioration of infant milk powders (Cheng et al., 2017; Fenaille, Visani, Fumeaux, Milo, & Guy, 2003).

**Table 2**
Volatile compounds identified in two types of infant milk powders (high lipid infant formula milk powder and high protein infant formula milk powder) during storage.[a]

| Volatile compound | $RI_{exp}$ | $RI_{auth}$ | $RI_{litt}$ |
|---|---|---|---|
| **Aldehydes** | | | |
| Pentanal | 980 | 983 | |
| Hexanal | 1091 | 1087 | |
| Heptanal | 1192 | 1192 | |
| Octanal | 1306 | 1306 | |
| Nonanal | 1404 | 1402 | |
| (E)-2-Pentenal | 1136 | | 1129 |
| (E)-2-Decenal | 1660 | 1662 | |
| (E)-2-Octenal | 1442 | 1444 | |
| (E,E)-2,4-Nonadienal | 1720 | 1722 | |
| 1-Methyl-3-cyclohexene-1-carboxaldehyde | 1429 | | |
| (Z)-2-Heptenal | 1340 | 1339 | |
| (E,Z)-2,4-Heptadienal | 1478 | | 1463 |
| (E,E)-2,4-Heptadienal | 1507 | | 1492 |
| (E,Z)-2,4-Decadienal | 1783 | | 1756 |
| (E,E)-2,4-Decadienal | 1832 | | 1808 |
| Benzaldehyde | 1540 | 1539 | |
| Benzeneacetaldehyde | 1660 | 1662 | |
| 2-Methylbutanal | 913 | 912 | |
| 3-Methylbutanal | 916 | 916 | |
| **Ketones** | | | |
| Acetone | 817 | | 819 |
| 2-Butanone | 905 | 906 | |
| 2-Heptanone | 1190 | 1189 | |
| 4-Methyl-2-heptanone | 1215 | | 1211 |
| (E,E)-Octadien-2-one | 1586 | | 1566 |
| 6-Methyl-5-hepten-2-one | 1354 | 1353 | |
| 3-Octen-2-one | 1420 | | 1400 |
| Acetophenone | 1671 | | 1646 |
| 1-Penten-3-one | 1023 | | 1019 |
| **Alcohols** | | | |
| 1-Butanol | 1165 | 1166 | |
| 1-Pentanol | 1275 | 1274 | |
| 1-Hexanol | 1372 | 1372 | |
| 1-Octanol | 1572 | 1573 | |
| Isopropyl alcohol | 939 | | 926 |
| 2-Ethylhexanol | 1504 | 1499 | |
| 1-Penten-3-ol | 1179 | 1177 | |
| 1-Octen-3-ol | 1464 | 1464 | |
| (E)-2-Octen-1-ol | 1631 | | 1615 |
| **Furans** | | | |
| 2-Methylfuran | 872 | | 862 |
| 2-Ethylfuran | 952 | | 952 |
| 2-Pentylfuran | 1245 | | 1232 |
| Furfural | 1476 | 1476 | |
| 2-Furanmethanol | 1676 | 1679 | |
| **Sulfur compounds** | | | |
| Dimethyldisulphide | 1074 | | 1076 |
| Dimethyl trisulphide | 1391 | | 1376 |
| **Acids** | | | |
| Pentanoic acid | 1753 | 1751 | |
| Hexanoic acid | 1862 | 1860 | |
| **Esters** | | | |
| Ethyl acetate | 895 | | 887 |
| Ethyl propanonate | 958 | 962 | |
| **Others** | | | |
| 2-Methyl-2-undecanethiol | 1117 | | |
| Benzothiazole | 1988 | | 1961 |
| Phenol | 2025 | | 1997 |

[a] Abbreviations are: $RI_{exp}$, experimental RI; $RI_{auth}$, RI of authentic standard; $RI_{litt}$, mean of values reported in PubChem (2018).

Different 2,4-alkadienals have been reported as secondary oxidation products from n-3 fatty acids and have been used as more specific oxidative markers (Fabiano et al., 2018; García-Moreno et al., 2016; Shen, Augustin, Sanguansri, & Cheng, 2010).

Fig. 5c shows that the typical Strecker-aldehyde 3-methylbutanal increases most in the HPMP samples and when samples are stored at $a_w = 0.53$ and $a_w = 0.33$. At $a_w = 0.11$ formation is very low. The same pattern was seen for 2-methylbutanal.
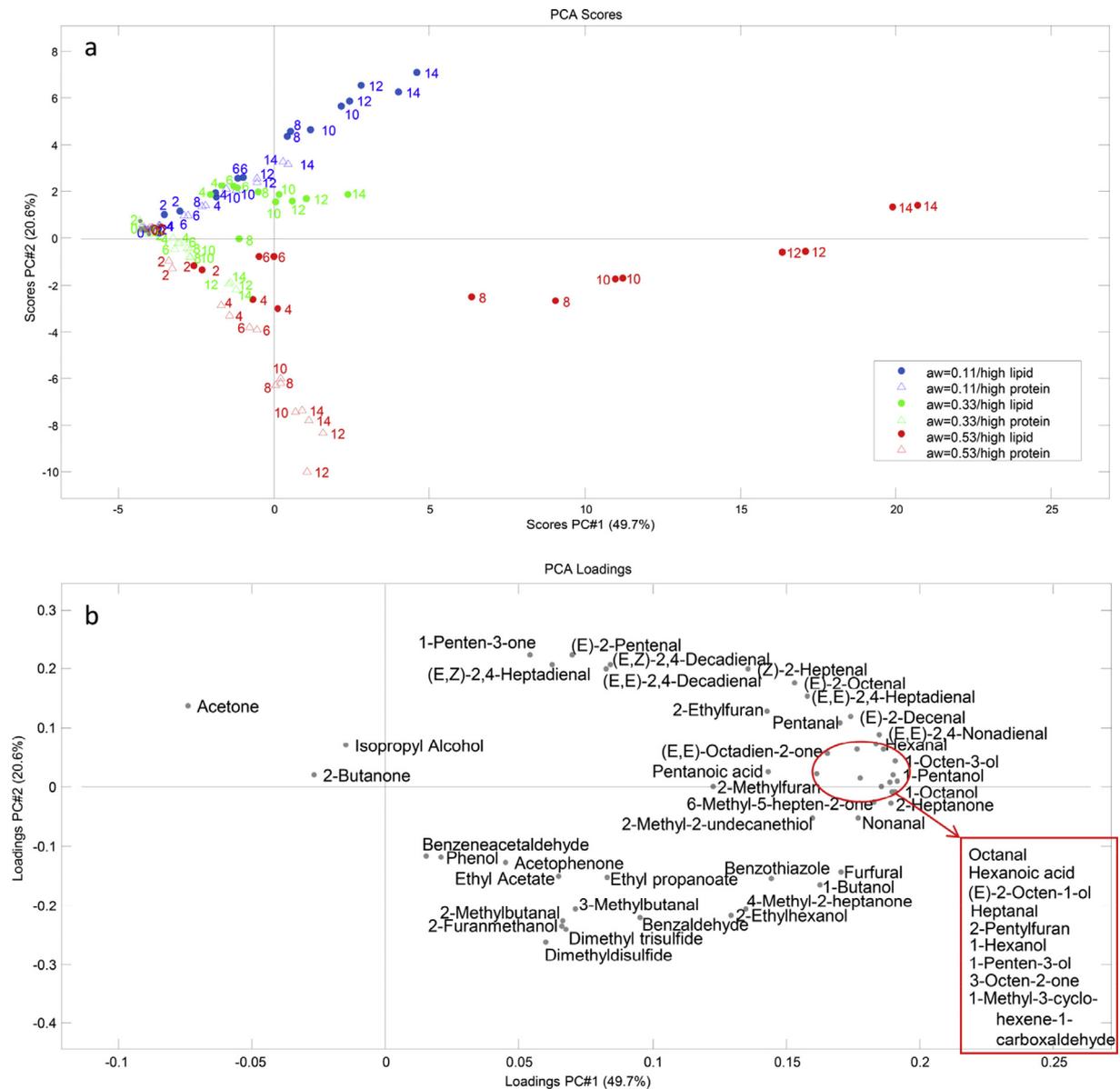
**Fig. 4.** Scores plot (a) and loadings plot (b) from principal component analysis (PCA) of volatile compounds formed during storage at 40 °C of two types of infant milk powders (high lipid infant formula milk powder and high protein infant formula milk powder) at different water activities ($a_w$).

3-Methylbutanal, with a spicy chocolate-like flavour (Juan, Barron, Ferragut, & Trujillo, 2007), and 2-methylbutanal, with a malty flavour (Griffith & Hammond, 1989), are produced by the deamination and decarboxylation of leucine and isoleucine, respectively, in the presence of α-dicarbonyl compounds (Estévez, Ventanas, & Heinonen, 2011; Mottram, 1998). Higher water activity storage conditions have earlier been found to promote Maillard reactions, in agreement with higher activation energies for decreasing moisture content (Cremer & Eichner, 2000; Hendel, Vernon, & Harrington, 1955).

Fig. 5d shows that dimethyltrisulphide reached the highest level at $a_w = 0.53$, especially in HPMP. At $a_w = 0.33$ and $a_w = 0.11$, almost no formation was seen. A similar pattern was seen for dimethyldisulphide. Dimethyltrisulphide and dimethyldisulphide with sulphury, onion-like flavour can be formed via the oxidation of methanethiol, which has been reported as a breakdown product of methional produced from the Strecker degradation of methionine (Dias & Weimer, 1999; Griffith & Hammond, 1989).

Fig. 5e shows that benzaldehyde is produced in the highest amount during storage at high $a_w$, and the production is higher in HPMP than in HLMP.

Benzaldehyde with almond or cherry-like flavour has been found to be produced by a combination of Strecker degradation and oxidation (Griffith & Hammond, 1989). The level of benzaldehyde was highest in HPMP stored at $a_w = 0.53$ confirming the link to Maillard degradation.

Fig. 5f shows that furfural is produced in the highest amount during storage at high aw. At $a_w = 0.53$, the production is higher in HLMP than in HPMP. The lactose level was similar in the two types of milk powders, and since furfural is formed by dehydration of sugars, no difference would be expected.

These examples confirm the general observation that water activity had a different effect on production of volatiles from lipid oxidation and Maillard reactions. Lipid oxidation was lowest at $a_w = 0.33$, which is in agreement with the most general conclusion for foods (Labuza & Dugan, 1971), while amino acid degradation
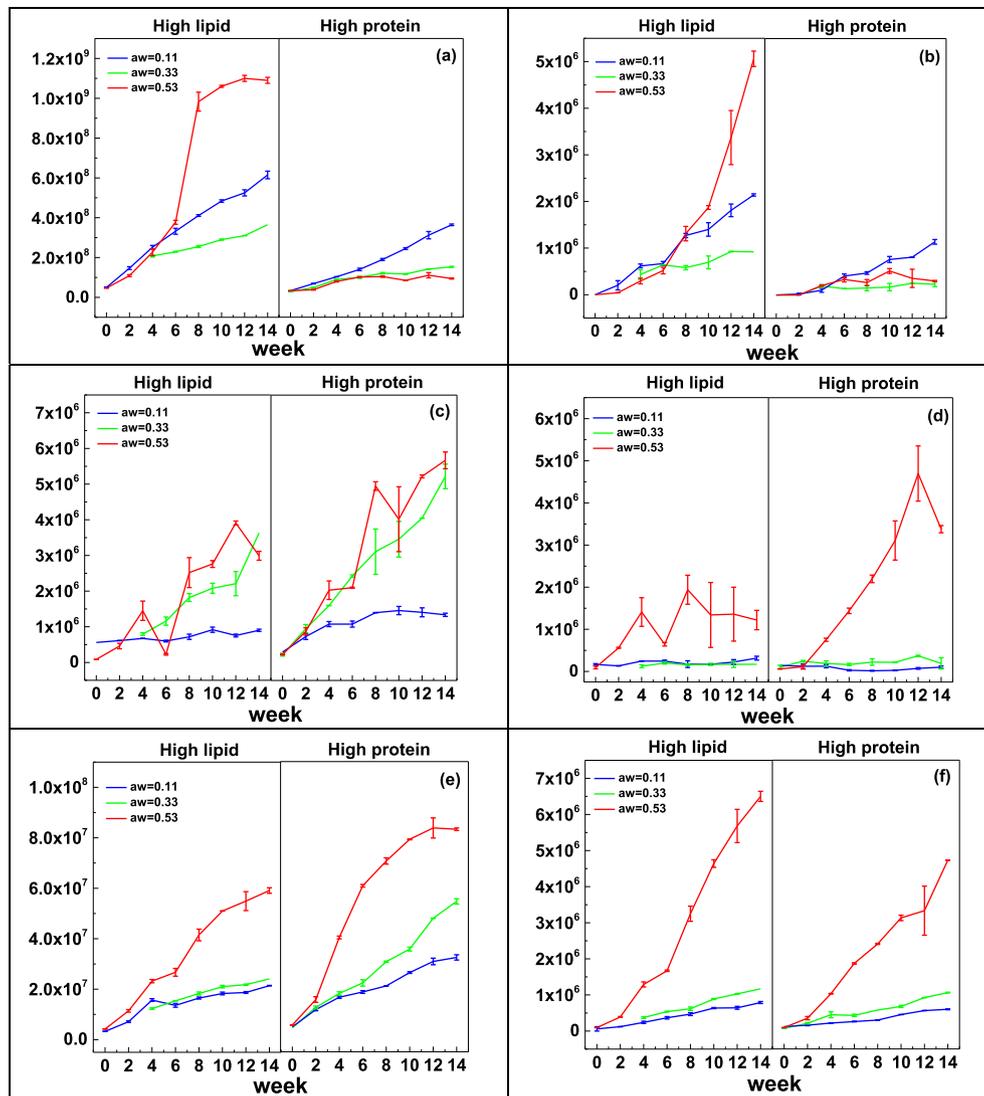
**Fig. 5.** Relative intensity of selected volatile compounds (a) hexanal, (b) (E,E)-2,4-nonadienal, (c) 3-methylbutanal, (d) dimethyltrisulphide, (e) benzaldehyde, (f) furfural, in two types of infant milk powders (high lipid infant formula milk powder and high protein infant formula milk powder) stored at 40 °C at different water activities (a_w).

was lowest at $a_w = 0.11$. The composition of the milk powder was also important since high lipid content (HLMP) resulted in higher lipid oxidation, and high protein content resulted in higher levels of amino acid breakdown products after storage.

## 4. Conclusion

High lipid and high protein infant formula milk powders were stored at water activities ($a_w$) of 0.11, 0.33, and 0.53 at 40 °C for up to 14 weeks. Both powders adjusted rapidly their initial water activity according to storage conditions, and powders turned brown rapidly especially at $a_w$ of 0.53, while browning was slower at 0.33, and hardly occurred at 0.11, as measured by the Hunter b parameter. A glass transition temperature around 53 °C disappeared for storage at $a_w = 0.53$, remained at a lower temperature around 38 °C for storage at 0.33 and increased to around 70 °C for $a_w = 0.11$ after a month of storage as determined by DSC. Protein degradation as detected by GC-MS as formation of Strecker aldehydes and benzaldehyde was most significant for the protein rich powder and increased with increasing water activity. Lipid oxidation as detected as several linear aldehydes by GC-MS had a clear minimum for

$a_w = 0.33$ for the lipid rich powder. For storage at ambient conditions, where lipid oxidation determines shelf-life, intermediate water activity is recommended for optimal storage stability for lipid rich powders. In contrast, storage at lower water activity is recommended for protein rich powders to minimise browning.

## References

Cesa, S., Casadei, M. A., Cerreto, F., & Paolicelli, P. (2015). Infant milk formulas: Effect of storage conditions on the stability of powdered products towards autoxidation. *Foods, 4*, 487–500.

Cheng, H., Zhu, R.-G., Erichsen, H., Soerensen, J., Petersen, M. A., & Skibsted, L. H. (2017). High temperature storage of infant formula milk powder for prediction of storage stability at ambient conditions. *International Dairy Journal, 73*, 166–174.

Cortés Yáňez, D. A., Gagneten, M., Leiva, G. E., & Malec, L. S. (2017). Antioxidant activity developed at the different stages of Maillard reaction with milk proteins. *LWT Food Science and Technology, 89*, 344–349.

Cremer, D. R., & Eichner, K. (2000). The reaction kinetics for the formation of Strecker aldehydes in low moisture model systems and in plant powders. *Food Chemistry, 71*, 37–43.

Delgado, F. J., González-Crespo, J., Cava, R., García-Parra, J., & Ramírez, R. (2010). Characterisation by SPME-GC-MS of the volatile profile of a Spanish soft cheese P.D.O. Torta del Casar during ripening. *Food Chemistry, 118*, 182–189.

Dias, B., & Weimer, B. (1999). Production of volatile sulfur compounds in Cheddar cheese slurries. *International Dairy Journal, 73*, 166–174.

Duflos, G., Coin, V. M., Cornu, M., Antinelli, J.-F., & Malle, P. (2006). Determination of volatile compounds to characterize fish spoilage using headspace/mass spectrometry and solid-phase microextraction/gas chromatography/mass spectrometry. *Journal of the Science of Food and Agriculture, 86*, 600–611.

Estévez, M., Ventanas, S., & Heinonen, M. (2011). Formation of Strecker aldehydes between protein carbonyls−α-aminoadipic and γ-glutamic semialdehydes−and leucine and isoleucine. *Food Chemistry, 128*, 1051–1057.

Fabiano, T. A. D., Roschel, G. G., & Castro, I. A. (2018). The use of factorial design to evaluate the oxidation of oils containing different types of omega-3 fatty acids. *Journal of the Science of Food and Agriculture, 98*, 2518–2529.

Fenaille, F., Visani, P., Fumeaux, R., Milo, C., & Guy, P. A. (2003). Comparison of mass spectrometry-based electronic nose and solid phase microextraction gas chromatographys-mass spectrometry technique to assess infant formula oxidation. *Journal of Agriculture and Food Chemistry, 51*, 2790–2796.

García-Moreno, P. J., Guadix, A., Guadix, E. M., & Jacobsen, C. (2016). Physical and oxidative stability of fish oil-in-water emulsions stabilized with fish protein hydrolysates. *Food Chemistry, 203*, 124–135.

Griffith, R., & Hammond, E. G. (1989). Generation of Swiss cheese flavor components by the reaction of amino acids with carbonyl compounds. *Journal of Dairy Science, 72*, 604–613.

Haque, M. K., & Roos, Y. H. (2006). Differences in the physical state and thermal behaviour of spray-dried and freeze-dried lactose and lactose/protein mixtures. *Innovative Food Science & Emerging Technologies, 7*, 62–73.

Hendel, C. E., Vernon, G. S., & Harrington, W. O. (1955). Rates of nonenzymic browning of white potato during dehydration. *Food Technology, 9*, 433–438.

Juan, B., Barron, L. J. R., Ferragut, V., & Trujillo, A. J. (2007). Effects of high pressure treatment on volatile profile during ripening of Ewe milk cheese. *Journal of Dairy Science, 90*, 124–135.

Kim, M. N., Saltmarch, M., & Labuza, T. P. (1981). Non-enzymatic browning of hygroscopic whey powders in open versus sealed pouches. *Journal of Food Processing and Preservation, 5*, 49–57.

Labuza, T. P., & Dugan, L. R., Jr. (1971). Kinetics of lipid oxidation in foods. *CRC Critical Reviews in Food Technology, 2*, 355–405.

Lee, T.-A., Ho, J.-H., Khoo, S. K., & Chow, C. F. (2012). Comprehensive stability evaluation of iron-fortified milk powder. *Food Science and Technology Research, 18*, 419–428.

Mottram, D. S. (1998). Flavour formation in meat and meat products: A review. *Food Chemistry, 62*, 415–424.

Nasirpour, A., Scher, J., Linder, M., & Desobry, S. (2006). Modeling of lactose crystallization and colour changes in model infant foods. *Journal of Dairy Science, 89*, 2365–2373.

Norwood, E.-A., Le Floch-Fouéré, C., Briard-Bion, V., Schuck, P., Croguennec, T., & Jeantet, R. (2016). Structural markers of the evolution of whey protein isolate powder during aging and effects on foaming properties. *Journal of Dairy Science, 99*, 1–8.

Novaes, S. S. C., Dantas, F. B. H., Alvim, I. D., de Oliveira Miguel, A. M. R., Dantas, S. T., & Alves, R. M. V. (2019). Stability of omega-3 enriched milk powder in different commercial packages stored under accelerated conditions of temperature and relative humidity. *International Dairy Journal, 88*, 1–9.

Poulsen, M. W., Hedegaard, R. V., Andersen, J. M., Courten, B. D., Bügel, S., Nielsen, J., et al. (2013). Advanced glycation endproducts in food and their effects on health. *Food and Chemical Toxicology, 60*, 10–37.

Prost, C., Hallier, A., Cardinal, M., Serot, T., & Courcoux, P. (2004). Effect of storage time on raw sardine (Sardina pilchardus) flavor and aroma quality. *Journal of Food Science, 69*, S198–S204.

PubChem. (2018). *Compound database [online]*. The National Center for Biotechnology Information. https://pubchem.ncbi.nlm.nih.gov/. (Accessed 8 November 2018).

Purriños, L., Bermúdez, R., Franco, D., Carballo, J., & Lorenzo, J. M. (2011). Development of volatile compounds during the manufacture of dry-cured "lacón" a Spanish traditional meat product. *Journal of Food Science, 76*, C89–C97.

Schmitz, I., Gianfrancesco, A., Kulozik, U., & Foerst, P. (2011). Influence of temperature and the physical state on available lysine in powdered infant formula. *Procedia Food Science, 1*, 1031–1038, 11th International Congress on Engineering and Food (ICEF11).

Shen, Z., Augustin, M. A., Sanguansri, L., & Cheng, L. J. (2010). Oxidative stability of microencapsulated fish oil powders stabilized by blends of chitosan, modified starch, and glucose. *Journal of Agriculture and Food Chemistry, 58*, 4487–4493.

Stapelfeldt, H., Nielsen, B. R., & Skibsted, L. H. (1997). Effect of heat treatment, water activity and storage temperature on the oxidative stability of whole milk powder. *International Dairy Journal, 7*, 331–339.

Tham, T. W. Y., Yeoh, A. T. H., & Zhou, W. B. (2017). Characterisation of aged infant formulas and physicochemical changes. *Food Chemistry, 219*, 117–125.

Thomsen, M. K., Lauridsen, L., Skibsted, L. H., & Risbo, J. (2005). Temperature effect on lactose crystallization, Maillard reactions, and lipid oxidation in whole milk powder. *Journal of Agriculture and Food Chemistry, 53*, 7082–7090.

Xu, Y. X., Liu, Y., Jiang, C. C., Zhang, C. M., Li, X. P., Zhu, D. S., et al. (2014). Determination of volatile compounds in turbot (Psetta maxima) during refrigerated storage by headspace solid-phasemicroextraction and gas chromatography−mass spectrometry. *Journal of the Science of Food and Agriculture, 94*, 2464–2471.

Zhu, R.-G., Cheng, H., Li, L., Erichsen, H., Soerensen, J., Petersen, M. A., et al. (2018). Temperature effect on formation of advanced glycation end products in infant formula milk powder. *International Dairy Journal, 77*, 1–9.