



An approach to adapt aroma in fat-free yoghurt systems: Modelling and transfer to pilot scale



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ABSTRACT

Aroma retention and flavour perception of dairy products largely depend on the fat content of their respective varieties. When the milk fat content of model milk solutions was increased from 2% to 12%, a linear increase in the matrix-air partition coefficient K_{MG} , as determined by the phase ratio variation (PRV) method, was observed for six aroma compounds in the log P range of +1.8 to +4.5, namely ethyl butanoate, ethyl-2-methylbutanoate, (*Z*)-3-hexenyl acetate, ethyl hexanoate and limonene. (*Z*)-3-hexenol, the least hydrophobic compound (log P = +1.6), was slightly less retarded at increased fat content. With the exception of limonene, it was observed that the higher the log P, i.e., the hydrophobicity of the aroma compound, the more pronounced was the linear increase of K_{MG} and hence retention. The successful scale-up from basic laboratory research into pilot plant for the aroma compound retention in fat-free yoghurt is presented.

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1. Introduction

Although fat content has been recognised as the decisive factor in aroma retention behaviour, surprisingly little attention has been drawn to the investigation and quantification of this effect in milk products. Brauss, Linforth, Cayeux, Harvey, and Taylor (1999) and Deleris, Zouid, Souchon, and Trelea (2009) investigated milk fat contents of 3.5% and 10%, and 2% and 4%, respectively. They investigated aroma compounds with a reported log P ranging from −1.3 to +4.4 (diacetyl, ethyl acetate, trans-2-hexenyl acetate, ethyl hexanoate, linalool, anethole and terpinolene). Other authors investigated milk fat contents of 2.7% (Martuscelli, Savary, Pittia, & Cayot, 2008), 4% (Deleris, Lauerjat, Trelea, & Souchon, 2007; Kopjar, Andriot, Saint-Eve, Souchon, & Guichard, 2010; Saint-Eve, Juteau, Atlan, Martin, & Souchon, 2006), 5% (Nongonierma, Springett, LeQuéré, Cayot, & Voilley, 2006) and 20% (Benjamin, Leus, & Everett, 2011) alone or in comparison to 0% fat. Aroma compounds analysed included diacetyl, ethyl acetate, ethyl butanoate, ethyl isobutanoate, ethyl-3-methylbutanoate, cis-3-hexenyl acetate, ethyl hexanoate, ethyl octanoate, benzyl acetate, hexanal,

cis-3-hexen-1-ol, linalool, limonene, decanoic acid, 1-propanol, heptanal, octanol and 2-decanone (reported log P-values from −1.3 to +4.6). Nevertheless, these studies applied different methods of retention analysis and a vast variety of analytical conditions as well as extensive primary data transformation and modelling, which impedes the quantitative comparison of their results. None of the above named studies has varied the milk fat content on more than two levels, and consequently, its effect on the retention of aroma compounds cannot be adequately described over the comparably wide range of fat contents that is characteristic for dairy products.

However, several studies focused on how changes in the composition, especially the fat content, and the processing of yoghurt made with bacterial starter cultures affect the retention and release of aroma compounds (Deleris et al., 2007, 2009; Saint-Eve, Juteau, Atlan, Martin, & Souchon, 2006a,b). Further studies exist on aroma compound behaviour in model yoghurts acidified with glucono- δ -lactone (GDL) (Heilig, Çetin, Erpenbach, Höhn, & Hinrichs, 2011; Merabtime, Lubbers, Andriot, Tromelin, & Guichard, 2010; Nongonierma et al., 2006). Thus, no studies exist on whether the aspects of aroma compound-dairy matrix interaction that have been observed in bacterially fermented yoghurt are valid in GDL-fermented yoghurt, and, more importantly, vice versa.

To close the gap of understanding aroma compound retention and flavour perception of dairy products this study proposes two

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objectives: (i) to extend the data base on the aroma retentive effect of milk fat in model milk matrices and, subsequently, (ii) to investigate to which degree aroma–matrix interactions observed in model milk matrices are valid under the conditions of yoghurt manufacture.

For this purpose, the matrix-air partition coefficient K_{MG} of seven aroma compounds with a log P range of -1.3 to $+4.5$ was determined by means of the PRV method. Due to its calibration-free approach to partition coefficient determination (Ettre, Welter, & Kolb, 1993), the PRV method has been increasingly applied in recent food-related research of aroma compound behaviour (Benjamin et al., 2011; Heilig et al., 2011; Morakul et al., 2011; Zhang, Kim, Park, & Chung, 2010). In contrast to other often applied techniques of single-volume static or dynamic headspace analysis (Stevenson, Chen, & Mills, 1996), which yield only relative retention data, the PRV method delivers a fundamental, though temperature-dependent and matrix-specific, physico-chemical constant. The K_{MG} indicates to which degree the nasal and retro-nasal aroma perception of foods may be altered by changes in product composition or processing (Leksrisompong, Barbano, Foegeding, Gerard, & Drake, 2010).

2. Materials and methods

2.1. Aroma composition

The aroma composition used for this study was based on a commercial strawberry aroma from Symrise AG (Holzminden, Germany). Limonene and diacetyl, obtained from Symrise AG, were added to extend the log P range of the aroma compounds under investigation. Table 1 lists the final composition of the propylene glycol based aroma used in this study.

2.2. Preparation of flavoured model milk matrices

Micellar casein concentrate was produced in-house by microfiltration-diafiltration following the method described by Kersten (2001). Micellar casein concentrate was spray-dried at an inlet air temperature of $190\text{ }^{\circ}\text{C}$ to obtain micellar casein powder. The outlet temperature was $80\text{--}90\text{ }^{\circ}\text{C}$. Cream was produced in-house by separation of raw milk at $55\text{ }^{\circ}\text{C}$ and two-stage homogenisation ($15/3\text{ MPa}$) at $65\text{ }^{\circ}\text{C}$, followed by a batch-pasteurisation process at $90\text{ }^{\circ}\text{C}$ with a holding time of less than 10 s.

The flavoured model milk matrices were prepared as follows. Ultrafiltration permeate was produced by reconstituting $5.2\text{ g } 100\text{ g}^{-1}$ ultrafiltration permeate powder (Bayolan PT, BMI, Landshut, Germany) in distilled water. Model milk matrices were prepared by dissolving micellar casein powder and whey protein isolate powder (DSE 5627, Fonterra Co-operative Group Ltd., Auckland, New Zealand) in the ultrafiltration permeate at room temperature to give milk matrices with a protein content of 4% and a casein protein to whey protein ratio CWR of 4. This fat-free milk protein solution was batch-pasteurised at $65\text{ }^{\circ}\text{C}$ with a holding time of 30 min. Overnight stored cream and fat-free milk protein solution ($4\text{ }^{\circ}\text{C}$) were then warmed up to $35\text{ }^{\circ}\text{C}$ and mixed for fat contents of 2, 4 and 12%. Fat content of matrices was determined by using LactoScope FTIR Advanced (Delta Instruments B.V., Drachten, The Netherlands). The resulting model milk matrices were then flavoured with 1.0% (w/w) of the aroma composition. The pH of the flavoured model milk matrices was 6.75 ± 0.05 .

2.3. Pilot-scale yoghurt production

Pilot-scale production of yoghurt systems was performed according to Krzeminski, Großhable, and Hinrichs (2011). Raw milk

was separated, pasteurised ($74\text{ }^{\circ}\text{C}$, 30 s), adjusted in protein content by using low heat skim milk powder and fat content by using cream resulting in two batches of standardised yoghurt milk (full-fat batch: 4% protein, 4% fat; fat-free batch: 4% protein, 0.1% fat). Both batches were homogenised ($65\text{ }^{\circ}\text{C}$, $15/3\text{ MPa}$), heated in a tubular heat exchanger ($95\text{ }^{\circ}\text{C}$, 5 min), cooled to $35\text{ }^{\circ}\text{C}$, fermented with FD-DVS Yo-Flex[®] 812 (Chr. Hansen GmbH, Nienburg, Germany) at $35\text{ }^{\circ}\text{C}$ for 14 h to pH 4.46–4.42. Set yoghurt gel was manually stirred while adding 1.0% (w/w) of a strawberry aroma-based composition of volatile compounds. Immediately after stirring, 2000 μL of the aromatised matrices were filled into five 22 mL glass vials each, sealed and stored overnight at $10\text{ }^{\circ}\text{C}$ before headspace analysis. The full-fat batch was aromatised with 1.0% (w/w) of the non-modified aroma composition (AC), previously specified by Heilig et al. (2011). One half of the fat-free batch was aromatised with the non-modified aroma composition, and the other half with the adapted aroma composition (AAC) as described in Table 1.

2.4. Determination of the matrix/gas partition coefficient K_{MG}

Vials containing flavoured model milk solutions were equilibrated for 15 min at $40\text{ }^{\circ}\text{C}$ in an automatic headspace sampler QHSS40 (QUAMA Elektronik & Analytik GmbH, Wuppertal, Germany). The vials were gently agitated using the QHSS40 integrated shaker. After equilibration, 1 mL of vial headspace was automatically withdrawn at a valve temperature of $90\text{ }^{\circ}\text{C}$ and a tube temperature of $150\text{ }^{\circ}\text{C}$.

Headspace analysis was performed on a CP-3800 gas chromatograph (Varian Deutschland GmbH, Darmstadt, Germany), equipped with a split/splitless injector CP-1177 ($240\text{ }^{\circ}\text{C}$) and a flame ionisation detector ($250\text{ }^{\circ}\text{C}$, H_2 28 mL min^{-1} , synthetic air 300 mL min^{-1} , N_2 30 mL min^{-1}). For samples with 2% fat, a split-ratio of 1:50 was used, and 1:20 for the higher fat contents. An HP-FFAP capillary column with an inner diameter of 0.32 mm, a film thickness of $0.25\text{ }\mu\text{m}$ and a length of 30 m (Agilent Technologies, Waldbronn, Germany), was used for chromatographic analysis. A deactivated silica-coated with an inner diameter of $0.53\text{ }\mu\text{m}$ and a length of 5 m served as pre-column. The carrier gas was H_2 (2 mL min^{-1}). The oven program started at $40\text{ }^{\circ}\text{C}$ for 5 min, followed by heating up with $5\text{ }^{\circ}\text{C min}^{-1}$ to $100\text{ }^{\circ}\text{C}$ and $40\text{ }^{\circ}\text{C min}^{-1}$ to $240\text{ }^{\circ}\text{C}$ with a holding time of 5 min.

Vials containing yoghurt samples were equilibrated at $40\text{ }^{\circ}\text{C}$ for 30 min, and split-ratios of 1:50 and 1:20 were used for 0% and 4% fat, respectively.

The phase ratio variation (PRV) method (Ettre et al., 1993) was used to determine the matrix/gas partition coefficient K_{MG} . Volumes of 50, 75, 100, 150, 200, 500, 1000 and 2000 μL flavoured model milk were filled into 22 mL headspace vials (QUAMA Elektronik & Analytik GmbH) and immediately sealed with PTFE septa in metallic caps (QUAMA Elektronik & Analytik GmbH).

As described by Ettre et al. (1993), the matrix/gas partition coefficient $K_{MG,i}$ was calculated from

$$1/A_{i,i} = \left(\frac{1}{f_i \cdot C_{M,i}} \cdot K_{MG,i} \right) + \left(\frac{1}{f_i \cdot C_{M,i}} \right) \cdot \beta \quad (1)$$

where $A_{i,i}$ is the chromatographic peak area, f_i is a proportional factor, $C_{M,i}$ is the initial aroma compound concentration in the matrix and β is the phase ratio of headspace volume V_G and matrix volume V_M . Inversion of the matrix/gas partition coefficient $K_{MG,i}$ yields the gas/matrix partition coefficient $K_{GM,i}$, which is reported in a number of studies using the PRV-method (Deleris et al., 2007, 2009; Kopjar et al., 2010; Martuscelli et al., 2008; Saint-Eve et al., 2006a).

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