Food Chemistry xxx (xxxx) xxx–xxx

Contents lists available at ScienceDirect

Food Chemistry

journal homepage: www.elsevier.com/locate/foodchem

Understanding fat, proteins and saliva impact on aroma release from flavoured ice creams

Charfedinne Ayed[a,d], Sara I.F.S. Martins[b], Ann-Marie Williamson[c], Elisabeth Guichard[a,*]

[a] Centre des Sciences du Goût et de l'Alimentation, AgroSupDijon, CNRS, INRA, Université Bourgogne Franche-Comté, F-21000 Dijon, France
[b] Unilever R&D Vlaardingen, Olivier van Noortlaan 120, 3133 AT Vlaardingen, The Netherlands
[c] Unilever R&D Colworth, Colworth Science Park, Bedford MK44 1LQ, United Kingdom
[d] Flavour group, Division of Food Sciences, University of Nottingham, Sutton Bonington Campus, Sutton Bonington, Loughborough, Leicestershire LE12 5RD, United Kingdom[1].

ARTICLE INFO

Keywords:
Aroma release
Ice cream
Fat
Protein
Solid phase microextraction
Gas chromatography
Saliva reactor

ABSTRACT

The release profile of fourteen aroma compounds was studied in ice cream samples varying in fat and protein, both in level and type. *In vitro* aroma release was monitored by solid phase micro-extraction gas chromatography using an innovative saliva reactor, which imitated human chewing under temperature control. The results showed that the effect of the fat type on aroma release was smaller than that of fat level. Ice creams with low fat level released more hydrophobic aroma compounds than ice creams with high fat level. At low fat level more aroma compounds were released from ice creams with lower protein content. At high fat level a small increase of aroma release was observed by the addition of saliva, which was explained by a salting out effect, due to the presence of proteins and salts in the saliva. These findings confirmed that the interactions between salivary proteins and aroma compounds occurring in aqueous solutions are not observed in emulsions.

1. Introduction

Ice cream is a complex aerated emulsion composed of protein and milk fat which play an important role in the stabilisation of its structure. In order to answer to nutritional recommendations, ice cream manufacturers have been reconsidering the formula of their products using less fat or different sources of fat and protein. It has been shown that modifications in the process conditions leading to different diameters of ice crystals and air bubbles induce differences in ice cream microstructure which thus impact sensory perception and more specifically mouthfeel (Inoue et al., 2012). Considering ice cream as an oil-in-water emulsion, its microstructure also impacts the physico-chemical properties and as an example, fat droplet size was found to have a significant impact on emulsion destabilisation, meltdown behaviour and creamy mouthfeel (Koxholt, Eisenmann, & Hinrichs, 2001). Moreover, the consumption of ice cream is highly determined by its overall sensory acceptability, mainly flavour perception, which justifies the need to better understand the impact of food reformulation on the release of aroma compounds in conditions as close as possible as in-mouth consumption.

The effect of fat type and fat level on either aroma release or sensory perception in ice creams has been the subject of different studies. Frost, Heymann, Bredie, Dijksterhuis, and Martens (2005) demonstrated that a modification of fat type and fat level induced differences in the perceived rate of melting of ice cream and also in flavour perception, because an increase in fat level delayed the perceived ice cream melting measured by time intensity and the time to reach maximum flavour intensity. The authors also noticed different effects according to the different aroma compounds used, which were partly explained by the boiling point and hydrophobicity. Prindiville, Marshall, and Heymann (1999) showed that chocolate flavour was perceived differently in ice creams manufactured with milk fat or with cocoa butter. The impact of the fat type on the headspace composition of chocolate ice cream was then studied by Welty, Marshall, Grün, and Ellersieck (2001) who demonstrated that the release in the vapour phase of two important aroma compounds in chocolate, 3,5-diethyl-2-methylpyrazine and 2-methyl-5-propyl pyrazines, was higher in ice creams containing milk fat than in ice creams containing cocoa butter. Another study on strawberry ice creams showed that the strawberry flavour was perceived faster and more intensely in ice creams realised with a higher level of unsaturated vegetable fat (Hyvonen, Linna, Tuorila, & Dijksterhuis, 2003). Similar results were observed by other authors on model food emulsions realised with animal or vegetal oils flavoured with a fruity aroma (Fabre, Guichard, Aubry, & Hugi, 2003). Emulsions realised with a vegetal oil

* Corresponding author.
E-mail address: elisabeth.guichard@inra.fr (E. Guichard).
[1] Present address.

https://doi.org/10.1016/j.foodchem.2017.10.127
Received 21 February 2017; Received in revised form 4 August 2017; Accepted 25 October 2017

presented the highest maximal perceived intensity and the longer duration, which was explained by the fact that the vegetable fats used in the experiment had a lower liquid proportion upon heating which increased the release of hydrophobic compounds such as ethyl hexanoate, (Relkin, Fabre, & Guichard, 2004). This could be explained by a lower solubility of this hydrophobic compound in solid fat as was observed in model systems composed of oils differing in their melting points (Roudnitzky, Irl, Roudaut, & Guichard, 2003). The opposite behaviour was observed for diacetyl, which was more released from emulsions containing anhydrous milk fat (Relkin et al., 2004). The authors explained the higher release by the higher triacylglycerol content of anhydrous milk fat in comparison with vegetal fat and also by the greater droplet size of the emulsions which favoured the release of this hydrophilic compound (Charles, Rosselin, Beck, Sauvageot, & Guichard, 2000). However the effect of emulsion droplet size on the release of hydrophilic aroma compounds seems to highly depend on the nature of the emulsion, because in another study no effect of droplet size was observed on the release of diacetyl from ice creams (Miettinen, Tuorila, Piironen, Vehkalahti, & Hyvönen, 2002). There seems to be a better consensus for the behaviour of hydrophobic compounds, because both studies showed that the smaller the droplet size, the more intense was the release of the most hydrophobic compounds. Besides fat, ice cream is also composed of proteins, mainly milk proteins which are known to interact with aroma compounds according to both the nature of aroma and the nature of the protein (Guichard, 2002; Tromelin, Andriot, & Guichard, 2006). The nature and amount of protein in the ice cream will change the structure of the emulsion by modifying the interfacial properties and the fat droplet agglomeration in the emulsion (Sourdet, Relkin, & Cesar, 2003). This will impact the rate of transfer of aroma compounds from oil to water and then from the emulsion to the gas phase (Druaux & Voilley, 1997).

During consumption, even if the role of chewing can be considered negligible in the case of emulsions, ice cream undergoes phase changes from semi-solid to liquid, due to the combined actions of temperature increase and dilution with saliva, before swallowing (Salles et al., 2011). In water and oil model systems, the addition of artificial saliva modifies the air/liquid partitioning of aroma compounds (van Ruth, Grossmann, Geary, & Delahunty, 2001), inducing either a retention or a salting out effect. This effect has not been explored yet in real food emulsions.

Even if some general trends of flavour release from ice cream during eating have already been reviewed (Chung, 2007) there is still a need for a better understanding of the relative impacts of fat level, fat type and protein content on aroma release from ice creams, taking into account thermal exchanges occurring in the mouth and the effect of human saliva. A salivary reactor has been previously developed within the research group to mimic the in-mouth breakdown of fat spreads (Poette et al., 2010), which highlighted the impact of human saliva on aroma release. This device reproduces as faithfully as possible the principal phenomena occurring in the mouth during eating (i.e. stirring, saliva flow, and temperature) by using data from real measurements on subjects consuming the same products.

Our aim was therefore to determine the combined effects of food composition and human saliva on the release of a wide range of aroma compounds from ice creams, using the saliva reactor in conditions reproducing as close as possible the in-mouth process. We will thus design our experimental protocol to reproduce the thermal exchanges occurring in the mouth during ice cream consumption and work with a pool of human saliva. The results will allow us to determine the respective impacts of saliva, fat and protein on aroma release. This work will provide innovative tools to guide food industries in the reformulation of low fat ice creams with a limited effect on aroma release.

2. Materials and methods

2.1. Samples composition

The study was done with different samples of ice creams realised with two fat types (A and B) varying in their solid fat content (SFC). Fat A had respectively 83.3% and 39.6% SFC and fat B, 0.9% and 0.1% SFC at the temperatures of 10°C and 20°C. Each fat type was added at two different fat levels (L for low = 3 %; H for high = 9 %). The ice creams contained two different levels of skimmed milk powder enriched with whey protein (level 1: standard – SMP: 6.4% Whey: 2.3%; level 2: low – SMP: 3.2% Whey: 1.15%). They were flavoured with a mixture of 14 aroma compounds (acetoin: 450 mg/kg ice cream; 2,5-dimethylpyrazine, 33.3 mg/kg; vanillin: 550 mg/kg; 2-methoxy phenol: 45 mg/kg; benzaldehyde: 18 mg/kg; phenyl ethyl alcohol: 18 mg/kg; 2-ethyl-3,5-dimethylpyrazine: 54 mg/kg; 2-methoxy-4-methylphenol: 18 mg/kg; hexanal: 54.9 mg/kg; *p*-anisaldehyde: 300 mg/kg; ethyl butyrate: 18 mg/kg; butyl propionate: 64.8 mg/kg; *cis*-3-Hexenyl acetate: 3.6 mg/kg; ethyl octanoate: 18 mg/kg). To study the impact of human saliva on aroma release the experiments were realised after diluting the samples in either ultra-pure water (MilliQ®, Bedford, MA) (W) or human saliva (S). Thus a total of 16 samples were analysed (Table 1).

2.2. Human saliva collection

Resting human saliva was collected from 20 volunteers as already described (Poette et al., 2014). Participants were not allowed to eat or drink one hour before sampling. Resting saliva was collected by instructing the subjects to spit out the saliva into a pre-weighed plastic bottle until it was full. To obtain the most representative salivary composition, the different saliva samples were pooled, mixed and centrifuged at 15000*g* for 15 min. The salivary pool was then sampled into aliquots of 10 mL stored at −80 °C until use. In a previous study, no effect of saliva storage was observed on the retention of 2-heptanone and ethyl heptanoate by human saliva in aqueous solution (Pagès-Hélary, Andriot, Guichard, & Canon, 2014).

2.3. Saliva reactor

A saliva reactor cell was used to reproduce ice cream breakdown as closely as possible to in mouth conditions (Fig. 1). This device was specifically designed to evaluate the particular role of saliva during liquid and semi-solid food consumption (Poette et al., 2010). It was composed of a water-jacketed glass flask (250 ml), which allowed a temperature control of the sample, equipped with four orifices, one for the temperature sensor, two others to introduce the sample and the SPME fibers and the last one was equipped with a 3-blade marine propeller with digital speed control.

The amount of water/saliva to be added in the reactor and the temperature changes of the ice cream was estimated from preliminary tests with a panel of 10 volunteers. As an average of 1.6 g of saliva was produced by consuming 8 g of ice cream and considering that 50 g was the minimum amount of ice cream in the reactor, 10 ml of water/saliva

Table 1
Samples compositions and codes after dilution in water or saliva.

code in water	code in saliva	fat type	fat level	Protein level
WAH1	SAH1	A	High	1
WAH2	SAH2	A	High	2
WBH1	SBH1	B	High	1
WBH2	SBH2	B	High	2
WAL1	SAL1	A	Low	1
WAL2	SAL2	A	Low	2
WBL1	SBL1	B	Low	1
WBL2	SBL2	B	Low	2