Food Chemistry 157 (2014) 252–256

Contents lists available at ScienceDirect

Food Chemistry

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

The effect of sugars on the retention of ethyl butyrate by gellan gels

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ARTICLE INFO

Article history:
Received 19 November 2013
Received in revised form 4 February 2014
Accepted 6 February 2014
Available online 19 February 2014

Keywords:
Gellan
Ethyl butyrate
Sugars
Aroma retention
Headspace
Partition coefficient

ABSTRACT

The effect of sucrose, glucose and fructose on the retention of ethyl butyrate by low acyl gellan gels was investigated by static headspace gas chromatography. The air/biopolymer partition coefficient (K) and percentage of retention ($R\%$) were determined. When 5 g of sample were left to equilibrate at 37 °C for 24 h, the obtained results were explained in terms of gel rigidity, as increased rigidity resulted in increased aroma retention. Glucose showed the greatest aroma release among the sugars and resulted in either the same or increased aroma release with increasing concentration. Increasing concentrations of fructose and sucrose did not alter aroma release significantly. For 15 g of sample mass, sucrose exhibited the lowest partition coefficient values among the sugars. The two higher sucrose concentrations resulted in decreased coefficient values. For fructose and glucose, aroma retention decreased with increasing concentration. The percentage of retention values were positive for all sugars, throughout their concentration range and for both experiments.

1. Introduction

Aroma is an important organoleptic parameter that contributes to the acceptability of a product. Aroma release from a food matrix depends on the type and the concentration of the food ingredients (Seuvre, Philippe, Rochard, & Voilley, 2006) along with the nature and the physicochemical properties of the aroma compound (e.g., volatility, hydrophobicity) (Guichard, 2002; Secouard, Malhiac, Grisel, & Decroix, 2003). The nature and the intensity of the interactions between the aroma compounds and the matrix are also of great importance (Landy, Druaux, & Voilley, 1995).

Biopolymers (i.e. proteins, carbohydrates) are widely used by the food industry as thickening, stabilizing and gelling agents. When they are present in a food matrix, two mechanisms are proposed in order to explain their effect on the release of aromatic compounds (Terta, Blekas, & Paraskevopoulou, 2006). These mechanisms involve the interactions between the biopolymer and the aromatic compound as well as the effect on the transport of the volatiles from the interior of the sample to the surface due to the observed increased viscosity (Baines & Morris, 1987).

The distribution of the aroma compound between the matrix and the vapour phase depends on both thermodynamic and kinetic factors (Voilley & Souchon, 2006). The interactions of the aroma compound with the matrix along with its partitioning in the different phases define the thermodynamic aspects of the release. The kinetic aspects are connected to the resistance to its mass transfer from the matrix to the vapour phase (Martuscelli, Savary, Pittia, & Cayot, 2008).

Several techniques are used to determine aroma compound release. The most common way of investigating the aroma retention is by determining the air/biopolymer partition coefficient (K), using static headspace analysis. The air/biopolymer partition coefficient describes the partition of the aroma compounds in the air phase and the matrix, by measuring the aroma concentration in both phases after equilibrium has been reached (Boland, Buhr, Giannouli, & Van Ruth, 2004).

Among carbohydrates, low acyl gellan is a water-soluble polysaccharide that finds many applications in the food area. Some of them include confectionery, jams and jellies, water-based gels and dairy products (Sanderson, 1990). It forms hard and brittle gels in the presence of cations with the concentration and valency of cations being of great importance (Grasdalen & Smisdrod, 1987; Moritaka, Fukuba, Kumeno, Nakahama, & Nishinari, 1991). However, the presence of co-solutes also affects the properties of gellan gels (Evageliou, Mazioti, Mandala, & Komaitis, 2010).

Sugars (mainly sucrose, glucose and fructose) are important in the human diet. They are either added to food products by the food industry in order to achieve the desired properties in sweetness and in texture or they are present *de novo* in foods (Anderson, 1997). Sucrose is the most commonly used sugar and studies on its mixtures with gellan had shown that sucrose acts complementarily with the calcium ions needed for gelation (Willoughby & Kasapis, 1994).

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http://dx.doi.org/10.1016/j.foodchem.2014.02.035
0308-8146/

Over the last years, the content of sucrose in food products has become a matter of great importance. The intake of sucrose, or sugars in general, is associated with obesity, cardiovascular disease, type 2 diabetes and decreased diet quality (Park, Onufrak, Sherry, & Blanck, 2013). Consumers are interested in healthier new products containing either a decreased amount of sucrose or other sweeteners with fewer calories. As aroma release for these new products should not be significantly altered, a study on aroma release of matrices containing sugars seems interesting.

The present work investigated the retention of ethyl butyrate from 0.5 wt% low acyl gellan gels in the presence of sucrose, glucose and fructose at concentrations up to 20 wt%. Potassium in the form of salt was the cation used to induce gelation. Moreover, the effect of sample mass in the vials on aroma release was also investigated.

2. Materials and methods

2.1. Materials

The deacylated gellan gum was provided by Sigma (Phytagel, P8169) and KCl was from Merck. Ethyl butyrate (19230) was from Fluka. Sucrose was normal food grade and purchased locally. Fructose and glucose were Reagent grade from Fluka. Glucose was in its monohydrated form, which was taken into account in our calculations. Distilled water was used throughout the experiments.

2.2. Preparation of samples

Samples were prepared by dissolving gellan in distilled water at 90 °C using gentle agitation. Following solvation, sugars and KCl were added. The salt concentration in all samples was 100 mM. The polysaccharide concentration was kept constant at 0.5 wt% whereas that of the sugars varied from 0 to 20 wt% (5 wt% increment). Then, the aroma compound at a concentration of 400 ppm was added. In order to ensure its complete dissolution, additional stirring for 2 min was applied. Subsequently, 5 or 15 g of each sample was transferred to a 40 mL screw-capped glass vial with a Mininert valve (Sigma–Aldrich, Athens, Greece) which was capped immediately and left to equilibrate at 37 °C for 24 h prior to analysis.

2.3. Static headspace analysis

For gas chromatography analysis 500 μL of vial headspace were sampled with a gas-tight syringe. The chromatograph (Fisons Instruments, GC 8000 series, Model 8060) was equipped with an FID detector and an Equity 5 capillary column (30 m × 0.25 mm, film thickness 0.25 μm; Supelco, Bellefonte, PA). Helium was used as carrier gas, at a flow rate of 1.0 mL/min. Oven temperature was increased from 40 to 250 °C at a rate of 4 °C/min and maintained at 250 °C for 5 min. The injector and the detector temperature were 230 and 270 °C, respectively. The analysis was carried out five times for each different sample.

Five aroma preparations differing in their concentration were analysed in triplicate, under the same experimental conditions, in order to construct the calibration curve. The determination of the concentration of the aroma compound in the air phase was achieved by converting the peak areas obtained from the chromatograph by using this calibration curve.

The air/biopolymer partition coefficient (K) was determined according to the following equation:

$$K_{a/biopol} = \frac{C^f_{air}}{C^f_{biopol}} \quad (1)$$

where C^f_{air} is the concentration of the aroma compound in the air phase and C^f_{biopol} the corresponding concentration in the biopolymer gel phase.

The concentration in the biopolymer phase (C^f_{biopol}) was calculated by the following equation:

$$C^f_{biopol} = C^i_{biopol} - C^f_{air}\left(\frac{V_{air}}{V_{biopol}}\right) \quad (2)$$

where C^i_{biopol} is the concentration of the aroma compound initially present in the biopolymer sample and V_{air}, V_{biopol} the volumes of the air and the biopolymer phase, respectively.

Moreover the percentage of retention (R%) can also be calculated:

$$R\% = \frac{K_{a/water} - K_{a/biopol}}{K_{a/water}} \times 100(\%). \quad (3)$$

$K_{a/water}$ is the partition coefficient for samples containing only water. A positive percentage value shows that the aroma compound is retained by the biopolymer matrix whereas a negative value shows that it is released by the matrix (Seuvre et al., 2006).

2.4. Statistical analysis

One way analysis of variance (ANOVA) and least significant difference tests (LSD) were carried out on the data in order to determine significant differences between the samples. The significant level was $p < 0.05$ throughout the study. Data analysis was carried out with Statistica (StatSoft, Inc., Tulsa, OK).

3. Results

Figs. 1 and 2 present the values of air/biopolymer partition coefficient of ethyl butyrate, as determined by Eq. (1), plotted against increasing sugar concentration for all sugars studied and for different sample weights. Fig. 1 shows the retention of ethyl butyrate from gellan gels in the presence of 100 mM KCl when 5 g of each sample were equilibrated at 37 °C for 24 h prior to analysis. A first observation is that glucose showed the greatest aroma release for all concentrations studied. Moreover, its addition in increasing concentrations resulted in either the same or increased aroma release compared to the sample with no sugar present. On the other

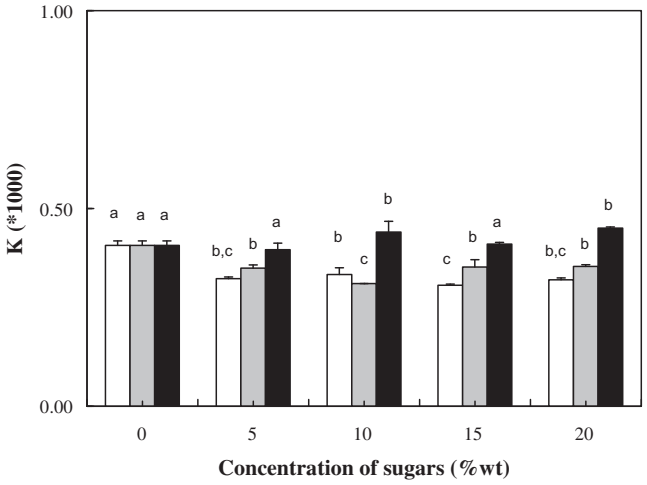

Fig. 1. Air/biopolymer partition coefficients ($K \times 1000$) of ethyl butyrate in gellan gels in the presence of sucrose (□), fructose (▨) and glucose (■). Samples were equilibrated at 37 °C for 24 h. Sample mass was 5 g. *Values with different superscripts (for the different concentrations of each sugar) are significantly different ($p < 0.05$).