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

Vasiliki Evageliou*, Ilianna Mavragani, Michael Komaitis

Department of Food Science and Technology, Agricultural University of Athens, 75 Iera Odos, 11855 Athens, Greece

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ABSTRACT

The effect of gellan gels with different texture on the retention of ethyl butyrate was investigated by Static Headspace Gas Chromatography. Calcium induced gels enriched with 400 ppm of the volatile showed not significantly different aroma release for calcium concentrations up to 40 mM, whereas higher concentrations exhibited greater partition coefficient values not significantly different from each other. Aroma release was not controlled by the mechanical properties when 1000 ppm of ethyl butyrate was added. When mixtures of calcium and potassium chloride, at a total molar concentration of 80 mM, were used to induce gelation, aroma release became greater with increasing calcium concentration in the mixtures. Moreover, elevated concentrations of the aroma compound (400–1000 ppm) added to gellan matrices, gelled by 10 mM calcium chloride, resulted in increased aroma release. For all samples, the percentage of retention was also calculated and both positive and negative values were determined.

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1. Introduction

The consumers' need for healthier diet has led the Food industry to change the composition of several food products. This alteration is done, mainly, by the addition of various biopolymers. The acceptance of a product is influenced by its organoleptic properties. In that aspect, aroma is an important parameter. Composition modifications can enhance or decrease aroma release from a food matrix. Therefore, the knowledge of the nature and the intensity of the interactions between the aroma compounds and the matrix is useful (Landy, Druaux, & Voilley, 1995).

When studying the retention of aroma compounds by a matrix, several parameters should be taken into account. These include the type and the concentration of the food ingredients along with the nature and the physicochemical properties (e.g. volatility, hydrophobicity) of the aromatic compound (Seuvre, Philippe, Rochard, & Voilley, 2006). Both thermodynamic and kinetic factors affect the distribution of the aroma compound between the matrix and the vapour phase (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 ones are connected to the resistance to its mass transfer from the matrix to the vapour phase (Martuscelli, Savary, Pittia, & Cayot, 2008).

In the case of biopolymers present in a food matrix, two mechanisms are proposed in order to explain their effect on the release of the 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).

Aroma compound release is usually investigated by Static Headspace Gas Chromatography. This technique determines the partition coefficient (K), which 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).

In a recent work of our research team the effect of the time of equilibration and the amount of sample in the headspace vials on the retention of ethyl butyrate by gellan gels in the presence of potassium ions was investigated (Evageliou, Galanaki, Gardeli, & Komaitis, 2011). The valency of the cations used to induce gellan's gelation is of great importance as divalent cations are more effective than the monovalent ones (Moritaka, Fukuba, Kumeno, Nakahama, & Nishinari, 1991). Potassium chloride was used in this former work. In order to explore further the effect of salts on the retention of ethyl butyrate by gellan gels, the present work investigated its retention in gels where calcium chloride and mixtures of calcium and potassium chloride was used to induce gelation. Furthermore, the effect of increasing aroma compound concentration was also investigated.

* Corresponding author. Tel./fax: +30 210 5294691.
E-mail address: evageliou@aua.gr (V. Evageliou).

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2. Materials and methods

2.1. Materials

The deacylated gellan gum was provided by Sigma (Phytagel, P8169). KCl was from Merck and $CaCl_2$ from Panreac Quimica SA. Ethyl butyrate (19230) was from Fluka. Table 1 shows its physicochemical properties (Seuvre, Philippe, Rochard, & Voilley, 2007; www.sigmaaldrich.com). Distilled water was used throughout the experiments.

2.2. Preparation of samples

In all experiments, the polysaccharide concentration was kept constant at 0.5 wt%. In the first set of experiments, calcium concentration varied from 10 to 100 mM, whereas ethyl butyrate was added at concentrations of 400 and 1000 ppm. The effect of mixtures of the two salts at a total concentration of 80 mM was also explored. Mixtures were prepared at KCl:$CaCl_2$ molar ratios of 20:60, 40:40 and 60:20 in the presence of 400 ppm of the aroma compound. In another set of experiments, gellan samples gelled by 10 mM calcium chloride were enriched with 400, 600, 800 and 1000 ppm of ethyl butyrate.

Samples were prepared by dissolving gellan in distilled water at 90 °C under gentle agitation. After solvation, appropriate amounts of the salts were added. Gellan preparations were cooled to ~50 °C and the aroma compound was added. In order to ensure its complete dissolution, additional stirring for 2 min was applied. Then, 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

Gas chromatography was used for the analysis of the headspace of each vial as 500 μL of it were sampled with a gas-tight syringe. The chromatograph (Fisons Instruments, GC 8000 series, Model 8060) was equipped with an FID detector and using an Equity 5 capillary column (30 m × 0.25 mm, film thickness 0.25 μm, Supelco). 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 °C and 270 °C, respectively. The analysis was carried out five times for each different sample.

Five aroma preparations 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 coefficient was calculated, in order to determine the retention of the aroma compound, by dividing the concentration of the aroma compound in the air phase (C^f_{air}) to the corresponding concentration in the biopolymer gel phase (C^f_{biopol}) according to the following equation:

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

Furthermore, the percentage of retention ($R\%$) was also calculated. A positive percentage value shows that the aroma compound is retained by the biopolymer matrix whereas a negative one, that it is released by the matrix (Seuvre et al., 2006). For its determination the following equation was used:

$$R\% = \frac{K_{a/water} - K_{a/biopol}}{K_{a/biopol}} \times 100(\%) \quad (2)$$

$K_{a/water}$ is the partition coefficient for samples containing only water.

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. Analysis of data was carried out with Statistica (Stat-Soft, Inc., Tulsa, OK, USA).

3. Results

Figs. 1–4 present the values of air/biopolymer partition coefficient, as determined by Equation (1), plotted against increasing salt (Figs. 1–3) and aroma compound (Fig. 4) concentration. Figs. 1 and 2 show the retention of ethyl butyrate from gellan gels in the presence of increasing calcium chloride concentration when 400 (Fig. 1) and 1000 ppm (Fig. 2) of ethyl butyrate was added, respectively. Samples were equilibrated at 37 °C for 24 h prior to analysis. The amount of gel present in the sample vial was 15 g. At the lower volatile concentration (400 ppm), and for salt concentrations up to 40 mM no significant changes at the partition coefficient values were seen. As calcium concentration increased, the coefficient values were increased but did not differ significantly among them. In the presence of 1000 ppm of ethyl butyrate (Fig. 2), the partition coefficient had its higher value for the calcium

Table 1
Physicochemical properties of ethyl butyrate.

Properties	
Formula	$C_6H_{12}O_2$
CAS number	105-54-4
Molecular weight (g/mol)	116.16
Boiling point (°C)	120
Refractive index	1.392
Hydrophobicity (log KOW)	1.73
Vapour pressure at 25 °C (mm Hg)	15.5
Solubility in water (25 °C)	5.3 g/L

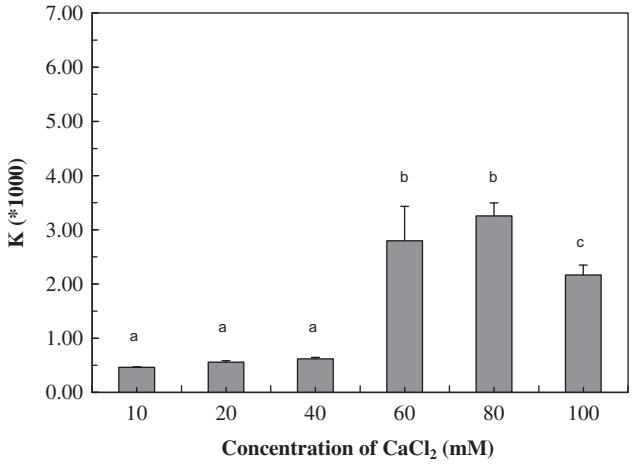

Fig. 1. Air/biopolymer partition coefficients ($K \times 1000$) of ethyl butyrate (400 ppm) in gellan gels in the presence of increasing concentrations of calcium chloride. *: Values with different superscripts are significantly different ($P < 0.05$).

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