Food Hydrocolloids 47 (2015) 94–98

Contents lists available at ScienceDirect

Food Hydrocolloids

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

Retention of *trans*-anethole by single and double layered films based on gelatine

Vasiliki Evageliou [a, *], Arhontoula Gerolymatou [a], Kyriaki Sotirakoglou [b], Chryssavgi Gardeli [a], Stavros Yanniotis [a]

[a] Department of Food Science and Human Nutrition, Agricultural University of Athens, 75 Iera Odos, 11855, Athens, Greece
[b] Department of Crop Science, Agricultural University of Athens, 75 Iera Odos, 11855, Athens, Greece

ARTICLE INFO

Article history:
Received 7 July 2014
Received in revised form
22 December 2014
Accepted 20 January 2015
Available online 30 January 2015

Keywords:
Gelatine
Gellan
trans-Anethole
Film
Aroma retention
Drying

ABSTRACT

The present work investigated the retention of *trans*-anethole by single and double layered films based on gelatine. In the case of double layered films, gelatine was the base component whereas gellan was the upper layer. The gelatine layer was always carrying the aroma compound. Films were formed under varying drying conditions. Aroma release was determined by hexane extraction. For some double layered films the aroma present in the headspace above the film was determined by static headspace analysis. Experimental conditions were of great importance. Longer extraction time, lower drying temperature and shorter drying time resulted in greater extraction yields. Longer extraction treatment did not compensate for the volatile losses during drying at high temperatures. Moreover, regardless the film composition or the drying conditions, no significant difference in the air/film partition coefficient was observed. In addition, when comparing single and double layered films under the same experimental conditions, the extraction yield was always higher for the double films. Thus, the double layered gelatine-gellan films are effective as aroma barriers.

1. Introduction

Aroma compounds are chemical compounds that can be found in food, perfumes and essential oils. They are responsible for flavour, an important parameter in determining food quality and acceptability. In order for flavour to be perceived, the aromatic compounds of food have to be released from the food (matrix) and carried to the olfactory and gustatory receptors (Juteau, Cayot, Chabanet, Doublier & Guichard, 2004).

Drying is a widely used by the food industry procedure in which a liquid, usually water, is removed from a wet solid. It should target to a rapid reduction of the moisture content of food products without affecting the quality of their active ingredients. Moisture content is an important factor as it affects the physical and chemical properties of food. Several drying processes can be used, including air, oven and freeze drying.

When drying a food product, apart from the drying parameters (e.g. duration, temperature), its surface properties are also very important as they may control the drying rate and the final product quality (e.g. retention of flavour) (Yamamoto, Morihiro, Ariyoshi, & Aktas, 2005). On that topic, literature shows that the presence of biopolymers can affect the surface properties. For example, it has been reported that the drying rate of sugar solutions gelled with a protein-based gel-forming agent, gelatine, was much lower than that of the agar-gelled sugar solutions, possibly due to the formation of gelatine skin (film) at the surface of the solution (Yamamoto et al., 2005). Moreover, it was found that an encapsulant consisting of 3 %w/w α-cyclodextrin and 27 %w/w maltodextrin resulted in the highest retention of shiitake flavours during spray-drying (Yoshii et al., 2005). In addition, it is known that below some water content, the surface of the drying drop reaches a moisture content no longer permeable to most volatile compounds, but quite permeable to the relatively smaller water molecules. Biopolymers (mainly proteins and carbohydrates) play an important role in the food industry mostly because of their interesting properties like hydration, water binding, viscosity and capacity of gelation (Glicksman, 1982). Moreover, they can be used to retain a variety of compounds like aroma compounds, antioxidants, antimicrobial agents, pigments, vitamins etc. This property is further exploited in the formation of edible films and coatings.

Regarding the retention of aroma/volatile compounds from a biopolymer matrix, it is affected by both thermodynamic and kinetic

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

http://dx.doi.org/10.1016/j.foodhyd.2015.01.016
0268-005X/

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 ones are connected to the resistance of the mass transfer from the matrix to the vapour phase. (Martuscelli, Savary, Pittia, & Cayot, 2008).

Furthermore, aroma retention depends not only on the ingredients present in the matrix but also on the physicochemical properties of the aroma compounds (e.g. hydrophobicity, pressure, solubility, structure) (Guichard, 2002; Secouard, Malhiac, Grisel, & Decroix, 2003; Seuvre, Philippe, Rochard, & Voilley, 2006).

In order to explain the effect of biopolymers present in the matrix on the volatility of the aromatic compounds two mechanisms are proposed (Terta, Blekas, & Paraskevopoulou, 2006). The first mechanism is connected to the increased viscosity in the presence of biopolymers. This alteration affects the transport of the volatiles from the interior of the sample to the surface. The second one is related to the interactions between the biopolymer and the aromatic compound. These interactions mainly involve binding, adsorption, complexation and encapsulation (Cayot, Taisant, & Voilley, 1998; Thijssen, 1974).

In a recent work of our research team (Gardeli, Evageliou, Poulos, Yanniotis, & Komaitis, 2010), fennel plants were freeze dried after being immersed to gelatin gels and starch solutions. The biopolymer systems were tested as surface barriers in order to minimise the losses of isoanethole and *trans*-anethole (the two main components of fennel's essential oil) during drying. According to our findings, both biopolymers led to reduced losses of the aroma compounds. In addition, it was suggested that they retained part of the volatiles during the drying procedure probably as a result of mechanical and/or chemical interactions between them and the aromatic compounds. Thus, they were acting as a second matrix.

The present work was based on these observations and aimed to make a first attempt to describe the behaviour of biopolymer matrices as aroma surface barriers. In order to simulate the behaviour of a biopolymer system covering the plant material, the study was organised in two steps. Initially, gelatine gels were enriched with *trans*-anethole and then dried. In a second step, and in order to simulate the behaviour of the system comprising both matrices (plant and biopolymer film), two biopolymer systems were used. These systems were formed by adding a second biopolymer gel system onto the aroma carrier initial biopolymer gel and then dried.

Several parameters (i.e. film thickness, drying time and temperature, extraction time) were taken into consideration in order to investigate the release of *trans*-anethole from single and double layered films based on gelatine and determine the optimum experimental conditions in terms of aroma retention.

2. Materials and methods

2.1. Materials

Gelatine was purchased from ICN Biomedicals (Catalogue. Number: 960317). Deacylated gellan gum (Phytogel, P8169), t*rans*-anethole (A8639) and hexane (208752) were provided by Sigma. The physicochemical properties of *trans*-anethole are shown in Table 1 (www.sigma.com; www.epa.gov/chemrtk/pubs/summaries/anethole/c14069tp.pdf). KCl (1.04936.1000), for inducing the gelation of gellan, and glycerol (4094.2500), which was used as a plasticiser, were from Merck. Distilled water was used throughout the experiments.

2.2. Preparation of samples

Gelatine solutions (5 wt%) were prepared by soaking the appropriate amount of granules at room temperature in a screw-capped bottle for about 1 h and then heating up to 60 °C under gentle agitation. Then, the samples were cooled to ~50 °C and the aroma compound, at a concentration of 1000 or 2000 ppm, was added. The bottle was then capped immediately and the sample was stirred for 2 min in order to ensure the complete dissolution of the aroma compound.

Gellan samples (0.5 wt%) were prepared by dissolving the appropriate amount of gellan in distilled water at 90 °C using gentle agitation. Following solvation, KCl and glycerol were added. The salt concentration in all samples was 80 mM, whereas that of glycerol 0.3 wt%.

Table 1
Physicochemical properties of *trans*-anethole.

	Properties
Formula	$C_{10}H_{12}O$
CAS number	4180-23-8
Molecular weight (g/mol)	148.20
Boiling point (°C)	234–237
Refractive index	1.561
Hydrophobicity (log KOW)	3.11
Vapour pressure at 25 °C (mm Hg)	0.05
Solubility in water (25 °C)	111 mg/L

2.3. Preparation of films

Gelatine was used for both single and double system formulations. For the preparation of single films, 10 or 15 g of the aroma enriched (1000 or 2000 ppm) gelatine solutions were poured onto glass Petri dishes (9 cm diameter), capped and refrigerated (4 °C) for 30 min in order to gel. The samples were then dried in a convection oven. Experiments differed in drying temperature (25 and 35 °C) and time (18, 24 and 48 h).

In the case of double layered films, gelatine was the base component whereas gellan the upper one. For their preparation, the gelatine layer was prepared as described in the previous paragraph. The gelatine layer was carrying the aroma compound at a standard concentration of 1000 ppm. For the upper layer, 22 g of gellan solution were poured onto glass Petri dishes (9 cm diameter) and then refrigerated (4 °C) for 30 min in order to gel. Then, the gellan gel was put on top of the gelatine gel and the double system was placed in the oven. Samples were dried either at 35 °C for 24 h or at 25 °C for 48 h.

2.4. Aroma compound retention in biopolymer matrix

In order to determine the retention of *trans*-anethole from both single and double layered films, 0.25 g of the dried film was dispersed in 2 mL of hexane (Monedero et al., 2010) for 24 or 48 h in order to achieve the best extraction scheme of the aroma compound. For the quantification of the aroma compound, 1 μL of the hexane phase was injected onto a gas chromatograph. The analysis was carried out five times for each sample. An external calibration curve was used to determine the concentration of the aroma compound in the hexane phase.

The extraction yield (in %) was calculated as the ratio of the determined amount of *trans*-anethole in the solvent to its amount initially present in the sample.

2.5. Aroma compound in gas phase

For some double layered films, and in order to measure the aroma compound release from the film into the headspace, 1 g of each film was transferred to a 20 mL screw-capped glass vial with a