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P3 - GC-MS ANALYSIS FOR EVALUATION OF QUALITY AND OXIDATIVE STABILITY IN CHEWING GUM BASES

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Introduction

Chewing gum is one of the very popular confectionery products. Generally, chewing gum is a combination of a water-insoluble phase, known as gum base, and some other ingredients, including sugar, various softeners, food colourings, preservatives such as synthetic antioxidants, flavourings, etc. (Wong et al, 2009). Natural ester-type gum bases, which are used worldwide as food additives, mainly consist of wax esters composed of long-chain fatty acids and long-chain fatty alcohols (Tada et al, 2014). The presence of volatile organic compounds (VOCs) generated from the breakdown of lipid hydroperoxides is an established indicator of lipid oxidation products in some food systems. Most of these compounds are generally aldehydes whose smell can be defined as pungent, "green (or erbaceous)", "fat", "food-fried" etc. and may easily be perceived by the consumers. Solid Phase Micro Exraction from Head Space (HS-SPME) or Dinamic Headspace Sampling (DHS) with Gas Chromatography-Mass Spectrometry (GC-MS) detection applied to VOCs allowed their characterization and quantification, as relative abundance, in food samples (Ieri et al, 2015). The objective of this study was, firstly, to develop a method for identifying the VOCs in gum base and then to determine the contribution of the antioxidant BHT (Butylated hydroxytoluene) as food additive in protecting lipids against oxidation in gum base during the storage. Part of the work has focused on the development and optimization of different sampling and analysis techniques for the volatile fraction of Gum Base samples.

Experimental

Specimens of gum base layers furnished by GUM BASE CO. SPA, Lainate (Milan) were analyzed (Figure 1). 1 g of each sample was cut in three slices and placed in a 20 ml screw cap airtight vial. In order to evaluate the stability of the sample in a short time, an accelerated aging protocol was followed as reported in the International Conference on Harmonisation (ICH) guidelines (Ieri et al, 2015). Vials of sample OUT

(without antioxidant) and sample BHT (with antioxidant BHT) were put in a static oven at 40°C±2. On the basis of the manufacturers' guidelines, the accelerated aging protocol assimilates the storage of one month at room temperature to 10 days of storage at 40°C in an oven. The VOCs profile of Gum Base was determined by DHS-GC-MS and by SPME-GC-MS. For HS-SPME, the VOCs profile was determined by absorption of VOCs at 40°C (for 10-min) on a DVB/Carboxen/PDMS 1 cm trivalent fiber, followed by desorption at 280°C and, then, analysis by GC-MS. An Agilent 7890 a GC equipped with a 5975C MSD was used. A column Agilent DB InnoWAX 50m, 0.20 μm id, 0.40 μm df, was used for analytes separation. Chromatographic conditions were: initial temperature was 40°C, then increasing 10°C min-1 up to 260°C. This temperature was maintained for 6.6 min. A tentative compounds identification was performed by comparing the mass spectra of each peak with those reported in NIST 08 mass spectral databases. The evolution of samples odour profile was also followed by performing an in-factory sensory evaluation during aging.

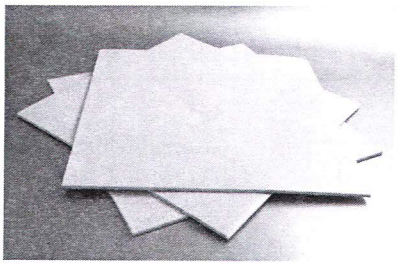

Figure 1. Gum base layers

Results

The use of different approaches in the pretreatment of the sample for the analysis of volatile compounds provides a wide range of opportunities to evaluate most of the components, even in such complexes matrices, such as gum base. SPME is a solvent-free, sensitive and reproducible technique easy to conduct and it was select for this work. A SPME-GC-MS profile obtained from BHT sample at time 0 is reported in Figure 2. Since oxidative stability is an important quality criterion for chewing gum, the identification of volatile compounds causing off-flavor is a key point for the quality improvement. In order to assess the presence of possible traceable markers of oxidation during aging, SPME-GC-MS analyses were carried out at the beginning of the experiment and periodically every 20 days up to 120 days and at 200 and 260 days, during accelerate aging.

Hexanal, a secondary product of lipid oxidation, was identified as an index of lipid oxidation in gum base samples.
Hexanal in sample OUT doubled its value after 40 days of accelerate aging, after 120 days the value was five times higher and after 260 days was 21 times higher and the samples were definitely perceived as oxidized at a sensory evaluation (Figure 3).

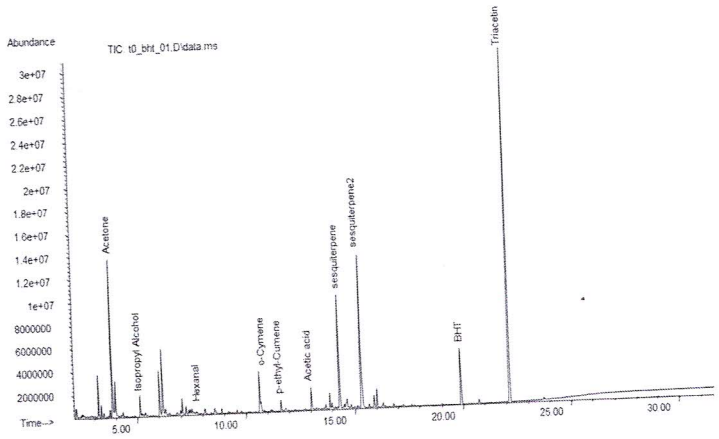

Figure 2. GC profile of BHT sample

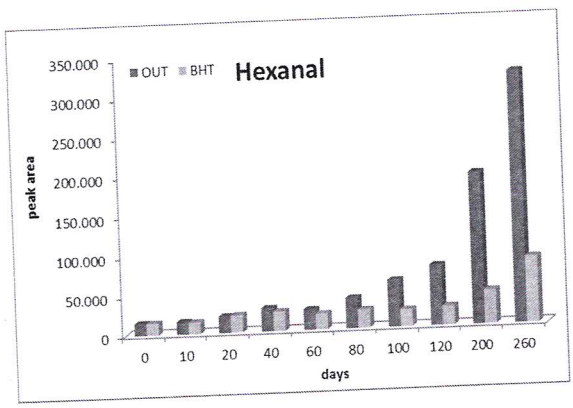

Figure 3. Hexanal trend during accelerate aging in OUT and BHT samples

On the contrary, BHT-containing samples had roughly the same Hexanal concentration from 20th to 120th days. After 200 and 260 days the presence of others VOCs such as hexanoic acid, ketones and aldehydes were evidenced in samples OUT stored in oven as well as at room

temperature. The onset of new VOCs such as hexanoic acid could be taken as marker to check the oxidation.

SPME-GC-MS, developed in this study for gum base samples, has proven to be a rapid method for monitoring lipid oxidation and chewing gum shelf-life by the quantification, as relative abundance, of hexanal, a reliable indicator for assessing the degree of oxidation of gum bases.

The results reported in this study come from a research agreement between GUM BASE CO. S.p.A. and the University of Florence.

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