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Aroma profile of Fuji apples treated with gelatin edible coating during their storage

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ABSTRACT

This study aimed to detect possible changes in the volatile organic compounds (VOCs) of Fuji apples induced by gelatin-based edible coating (EC), during 21 days of storage at room temperature. VOCs were analyzed by solid-phase micro extraction-gas chromatography-mass spectrometry. Data were analyzed by one-way ANOVA and principal component analysis. Control apples showed a greater presence of total aldehydes and acids at 7 and 14 days, respectively, while coated apples were characterized by higher proportions of alcohols (from 1.3- to 2-fold) at 7 day till the end of the storage. The higher ethanol proportions detected in coated apples (154-fold higher after 7 days) indicate a likely partial anaerobiosis, confirmed by the lower CO_2 emission (reaching -68% after 21 days). Esters responsible of the varietal aroma of Fuji were identified also in coated fruits, suggesting that gelatin did not modify the typical aroma extensively. Acetate esters, normally increasing with maturity, were less concentrated in coated apples (-78% 2-methylbutyl acetate and -73% hexyl acetate, after 1 and 7 days respectively), suggesting a likely slowdown of the ripening due to the EC.

Further investigation is needed to improve this storage technology considering that aroma is an important determinant of food quality.

1. Introduction

Fuji apple (*Malus* × *domestica* Borkh.) is one of the most consumed cultivar worldwide thanks to its typical sweetness, crunchiness and aroma. Apples are well known as a huge source of phytochemicals like flavonoids and phenolic acids (Francini & Sebastiani, 2013), which play an important role in the antioxidant defense of the human organism. Food packages are essential in increasing the shelf life of fresh foods, like fruits, preserving their safety and quality. Nowadays the most common package materials, like polyethylene terephthalate (PET), polyvinylchloride (PVC) and others, are produced from petrochemical plastics, which are not totally biodegradable and recyclable (Siracusa, Rocculi, Romani, & Rosa, 2008). Because of their high environmental impact, many recent studies have investigated new materials, more eco-friendly and user-friendly, to preserve food, such as edible coatings (ECs). ECs are prepared from edible materials, such as lipids, proteins or polysaccharides; they are applied and formed directly on the food product by spraying, dipping or brushing, constituting a thin layer around (Dhall, 2013; Falguera, Quintero, Jiménez, Muñoz, & Ibarz, 2011; Guilbert, Gontard, & Gorris, 1996). They can be consumed along with foods, but they can be easily removed as well, if the consumer does not like to eat them. Edible coatings can be obtained from natural biopolymers, also derived from wastes of agro-food industry, so they have low impact on the environment and may reduce the plastic packaging waste. The use of coatings for fruits is not a new concept: waxes have been used from a long time in China to prevent water transpiration loss and it has been reported that in 1930's hot-melt paraffin wax was used as EC for apples and pears (Dhall, 2013). In many studies, researchers demonstrated the effectiveness of ECs in decreasing weight loss, prolonging conservation and preventing deterioration of perishable fruits (Del-Valle,

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Hernàndez-Muñoz, Guarda, & Galotto, 2005; Dhall, 2013; Falguera et al., 2011; Zhang, Wang, Hu, & Liu, 2015).

Among the film-forming proteins, gelatin, obtained by hydrolysis of collagen, has effective barrier properties against oxygen and carbon dioxide (Jiang, Liu, Du, & Wang, 2010). Gelatin-starch coatings prolonged the postharvest shelf life of avocado (Aguilar-Méndez, San Martín-Martínez, Tomás, Cruz-Orea, & Jaime-Fonseca, 2008) and 10% gelatin coating, besides delaying ripening of mango fruit by suppressing the activity of softening enzymes, allowed the retention of higher ascorbic acid and phenolic content as compared with control (Gol & Ramana Rao, 2013). In the last few years, many scientific publications focused on physical and organoleptic changes in fruits covered with ECs, but at the best of our knowledge, only very few papers deal with aroma profile of coated apple fruit (Maya-Meraz et al., 2014; Olivas, Mattinson, & Barbosa-Cánovas, 2007; Sepulveda & Olivas, 2016), and no paper assessed possible changes in volatile organic compounds (VOCs) induced by gelatin coating.

VOCs contributing to apples aroma are over 300 and they belong to different chemical classes including carboxylic esters, alcohols, aldehydes, ketones, acids, terpenes and ethers (Ferreira, Perestrelo, Caldeira, & Câmara, 2009). However, only about 20 of them are character impact compounds (Dixon & Hewett, 2000) and each individual molecule has its own odor threshold and concentration. Fruit aroma is cultivar-specific (Dixon & Hewett, 2000) but it depends also from pre- and post-harvest conditions like seasonal variation (López, Lavilla, Riba, & Vendrell, 1998) or harvest date and storage technology (Echeverría, Fuentes, Graell, Lara, & López, 2004b). The major volatiles in apples are esters, which are synthesized by esterification of alcohols and acyl-coA from fatty acids or amino acids-pathways; another important group is represented by alcohols, derived from fatty acids and amino acids metabolism and lipoxygenase (LOX) activity, this latter also producing aldehyde volatiles (Dixon & Hewett, 2000).

From a quality point of view, it is extremely important that the aroma profile of produce treated with EC is preserved to ensure good palatability. Therefore, this work aimed to verify, by means of HS-SPME and GC/MS technique, whether a gelatin-based ECs induced changes in the VOCs profile of Fuji apples during three weeks of storage at room temperature to simulate home conditions.

2. Material and methods

2.1. Plant material and experimental design

Apple fruits (*Malus domestica* Borkh. L. cv. Fuji) were obtained from a commercial orchard (Marchetti Anna Paola, province of Pisa). After one month of cold storage, fruits of uniform size and free from any visual symptoms of diseases were washed, dried and randomly separated into two groups: the first group was coated with gelatin while the second one, uncoated, represented the control. Apples were stored at room temperature (20 ± 1 °C), to simulate home storage conditions, and samples were collected at four different times after coating treatment: 1, 7, 14 and 21 days. After this period, visual signs of ageing, as wrinkled skin, appeared on some fruits. For each storage time and coating treatment, 3 fruits were used for aroma analysis and 5 for measurement of fruit gas exchanges (64 fruits in total). Each fruit represented an independent biological replicate.

2.2. Edible coating

Edible coating was prepared using the gelatin sheets discarded during the production of soft gelatin capsules, obtained from the Chemical-Pharmaceutical Laboratory Tiaraju (Santo Ângelo, Rio Grande do Sul, Brasil). The EC solution was prepared at 1.5 g/L final gelatin concentration by melting gelatin sheets in water at 60 °C until complete dissolution. After cooling at 40 °C, Tween 20 (0.01 g/L) and potassium sorbate (0.01 g/L) were added. The edible coating was applied by dipping the apples into the solution and let them dry at room temperature.

2.3. Headspace solid-phase micro extraction (HS-SPME) procedure

The HS-SPME procedure was done according to Ferreira et al. (2009), with some modifications. Briefly, each apple was weighed and, after removing core but keeping pulp and peel, it was cut into small pieces within a beaker filled with saturated calcium chloride (1/1 apple weight/$CaCl_2$ volume) to inhibit enzyme activity and homogenized with a mixer. Two grams of the mixture were put into a glass vial and closed with aluminum cap provided with a PTFE-septum. The vial was placed in a water bath at 50 °C for 15 min. VOCs were collected by using a divinylbenzene/carboxen/polydimethysiloxane (DVB/Carboxen/PDMS) Stable Flex SPME fiber (50/30 μm; 2-cm long) (Supelco, Bellefonte, PA, USA). The SPME fiber was first preconditioned for 15 min in the GC injection port at 250 °C and then exposed to headspace for 30 min, after which the fiber was retracted prior removal from the sample and then inserted into the GC system. Before every sampling, the fiber was preconditioned and blank runs were done in-between to check the absence of volatile residues on it.

2.4. GC/MS analysis

The fiber was inserted into the injector of a single quadrupole GC/MS apparatus (TRACE GC/MS, Thermo-Finnigan, Waltham, MA, USA) set at 250 °C, 3 min in splitless mode, keeping the fiber into the injector for 15 min in order to obtain the complete desorption. The GC program conditions were the same as those described by Ferreira et al. (2009). The GC apparatus was coupled with a Varian CP-WAX-52 capillary column (60 m × 0.32 mm; coating thickness 0.5 μm). The transfer-line and the ion source were both set at 250 °C. The filament emission current was 70 eV. A mass range from 32 to 300 *m/z* was scanned at a rate of 1.6 amu/sec. The acquisition was carried out by electron impact, using the Full Scan (TIC) mode. Three replicates (n = 3) per sample were run. For the determination of LRI a C_8-C_{20} series was used (Sigma-Aldrich).

The VOCs were identified in three different ways: by comparison with the mass spectra of the Wiley library (version 2.0–11/2008); by injection of authentic standards previously analyzed and stored in the database; by calculation of LRI (Linear Retention Index) and comparing with those obtained in literature. For those compounds of which authentic standards were not used, the identification is to be considered tentative. Data of volatiles were expressed as peak area percentage of total chromatogram area (Budryn, Zaczyńska, & Oracz, 2016).

2.5. Gas exchange measurements

CO_2 gas exchange was determined using the LI-6400XT portable gas exchange system (LI-COR, Lincoln, NE, USA) equipped with a large chamber (6400-05, LI-COR, Lincoln, NE, USA). Measurements were performed on five individual fruits per treatment and time point, at CO_2 concentration of 400 μmol mol^{-1}, air temperature of 20 °C and relative humidity of 45–55%. Chamber was maintained under dark condition and fruits were allowed to adapt to the above conditions within the chamber for about 15–20 min for adjustment and stabilization of the gas exchange parameters.