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Talanta

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Novel approach for the determination of volatile compounds in processed onion by headspace gas chromatography–mass spectrometry (HS GC–MS)

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ARTICLE INFO

Article history:

Received 26 June 2012

Received in revised form

26 September 2012

Accepted 6 October 2012

Available online 11 October 2012

Keywords:

Headspace GC–MS

Scan mode

SIM mode

Onion

Volatile compounds

Sulfides

High-pressure processing

ABSTRACT

A novel headspace gas chromatography–mass spectrometry (HS GC–MS) method was developed for analysis of volatile compounds in onion (*Allium cepa* L. var. *cepa*, 'Recas'). MS was operated using full scan mode and selective ion monitoring (SIM) mode in order to quantify some specific compounds with increased sensitivity relative to full scan mode. The limits of detection and quantitation ranged from 0.01 to 0.10 µg/g and from 0.02 to 3.83 µg/g fresh weight, respectively, for studied compounds. The procedure allowed the identification of eighteen compounds and quantitation of nine compounds in the volatile fraction of onion, belonging mainly to di-, and trisulfides and aldehydes. These methods were applied to evaluate how high-pressure (HP) as a processing technology affects onion volatile compounds, responsible in part of the onion biological activity. Onion samples were treated at T1: 200 MPa/25 °C/5 min, T2: 400 MPa/25 °C/5 min and T3: 600 MPa/25 °C/5 min (treatments). In addition, the difference among diced, freeze-dried and pulverized onions (groups) was studied, in order to select the process more adequate for better preserving volatile compounds. The results obtained in full scan mode showed that both main factors (group and treatment) had a significant effect ($P < 0.001$). There were also significant differences between groups and treatments for all compounds, being the main effect of group more marked by HS GC–MS using selective ion monitoring (SIM) mode. For 2-methyl 2-pentenal, dimethyl trisulfide, and methyl propyl trisulfide it has been observed an increase in freeze-dried and pulverized onion samples compared with diced samples regardless the HP treatment. However, freeze-drying and pulverization processes affected the stability of propionaldehyde, 1-propanethiol, hexanal, dipropyl disulfide, and dipropyl trisulfide, diminishing their content regardless the HP treatment. HP at 200 and 400 MPa/25 °C/5 min were the least detrimental treatments to the total fraction of volatile compounds, not affecting or even increasing the levels of some volatile compounds.

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1. Introduction

Onion (*Allium cepa* L.) is an *Allium* vegetable widely used in cooking and as a food ingredient. Along with flavanols, the major bioactive constituents in onions are sulfur compounds, most of them are in the form of cysteine derivatives. Common to all *Allium* species is the enzyme alliinase that catalyzes the hydrolysis of S-alk(en)yl-L-cysteine sulfoxides (ACSOs) to produce pyruvate, ammonia and sulfenic acids. Upon tissue disruption produced by mastication, cooking, extraction, etc. the enzymatic hydrolysis of the ACSOs occurs. The major flavor compounds are generated

by the spontaneous reactions undergone by the sulfenic acids amongst themselves and other compounds. The result is a mixture of sulfur-containing compounds including thiosulfonates, thiosulfonates, mono-, di- and tri-sulfides as well as specific compounds such as the lachrymatory or tear factor, thiopropanal S-oxide [1,2]. Among the main sulfides in onions it could be mentioned dimethyl trisulfide, propenyl propyl disulfide, dipropyl disulfide, propenyl methyl disulfide, methyl propyl trisulfide and dipropyl trisulfides, responsible in part of its bioactivity [3]. These compounds are determined by GC–MS with the MS detector typically operating in the scan mode to aid identification of compounds. However, the scan mode does not achieve the optimal sensitivity, and the SIM mode with quadrupole systems is needed for accurate detection and quantification [4]. In addition, in this paper, headspace GC–MS will be use because is

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a valuable methodology with almost no sample treatment requirements [5].

The manner in which onion is processed and consumed when considering its potential biological activity and obtaining the maximum health effects has to be taken into account. The processing of foods is becoming more sophisticated and diverse in response to the growing demand for quality foods. Consumers today expect food products to provide fresh-like appearance, convenience, variety, appropriate shelf life and caloric content, reasonable cost, environmental soundness, and high nutritional and functional quality. Nonthermal processing of fruit and vegetable has been revealed as a useful tool to extend their shelf-life and quality as well as to preserve their nutritional and functional characteristics. In the last years, there has been an increasing interest in nonthermal technologies as high-pressure processing (HPP) [6,7]. HPP is a novel technology that has enormous potential in the food industry, controlling food spoilage, improving food safety, and extending product shelf life while retaining the characteristics of fresh, preservative-free, minimally processed food [8].

When processing onion it is important to consider not only the consumer's perception and onion safety and quality but also onion nutritional attributes. Onion HPP could offer safe new onion products with similar organoleptic properties of fresh onion that additionally could offer potential human health benefits. Dried onions are an attractive alternative to fresh onions for both domestic and industrial use. In addition, onions could be a source of a powdered functional food ingredient, very easy to use for consumers, with a great versatility and an extra added value due to its potential biological activity [9].

This paper evaluates the onion volatile compounds by headspace GC–MS using full scan mode and selective ion monitoring (SIM) mode in order to quantify some specific compounds with increased sensitivity relative to full scan mode. These methods were applied to evaluate how HPP affects onion volatile compounds, sulfides among them, as part of the compounds responsible of the onion biological activity. In addition, the difference among diced, freeze-dried and pulverized onions was studied, in order to select the process more adequate for better preserving volatile compounds.

2. Materials and methods

2.1. Reagents

n-Hexane (UV–IR–HPLC, 99.0%) was purchased from Panreac Química SAU (Barcelona, Spain). Propyl thioacetate, methyl propyl trisulfide, and dipropyl trisulfide were purchased from Endeavor Speciality Chemicals Ltd., Daventry, Northants, UK. Propionaldehyde, 1-propanethiol, allyl methyl sulfide, hexanal, 2-methyl 2-pentenal, dimethyl trisulfide, and dipropyl disulfide were purchased from Sigma-Aldrich Inc. (St. Louis, MO, USA).

Ultrapure water was produced from a Milli-Q[®] Integral system (Millipore Iberica SAU, Madrid, Spain).

2.2. Onion samples

Raw onions (*Allium cepa* L. var *cepa*, 'Recas') were supplied by Cebacat (Asociación Catalana de Productores-Comercializadores de Cebolla, Lleida, Spain). Onions were harvested in January 2011 in Spain. Their bulbs were free of external damages with 60–75 mm diameter, 61–78 mm height and 126–224 g weight.

2.3. High-pressure (HP) processing

Onions (*Allium cepa* L. var *cepa*, 'Recas') were hand peeled and cut into 10 mm pieces, packaged in a very low gas permeability bags type Doypack[®] (Polyskin XL, Flexibles Hispania, S.L.) and treated by high-pressure in a hydrostatic pressure unit with a 2925 mL pressure vessel capacity, a maximum pressure of 900 MPa, and a potential maximum temperature of 100 °C [Stansted ISO-LAB System (SW-IL-100-250-09-W)]. The rates of compression and decompression were both 3 MPa/s. During HP treatment, the vessel of the HP unit was thermostatically-controlled at 25 °C. Pressure, time and temperature were controlled by a computer program, being constantly monitored and recorded during the process. Due to compressive heating, an increase of the temperature of the pressurizing fluid, by up to a maximum of 0.5 °C, 1.7 °C or 5 °C at 200, 400 or 600 MPa, respectively, was measured.

The treatments were as follows: T1: 200 MPa/25 °C/5 min, T2: 400 MPa/25 °C/5 min and T3: 600 MPa/25 °C/5 min. Treatments were conducted in triplicate. After treatment, HP-treated (T1, T2 and T3) and untreated (T0) samples were immediately frozen with liquid nitrogen (diced samples). Subsequently, part of the diced samples were freeze-dried in a lyophilizer (model Lyoalfa, Telstar, S.A., Barcelona, Spain) (freeze-dried samples), and part of the freeze-dried samples were additionally pulverized with a pestle and mortar (pulverized samples). Samples were stored at –80 °C until analysis.

2.4. Headspace gas chromatography–mass spectrometry (HS GC–MS)

2.4.1. Gas chromatography

3.00 g of onion were transferred into 22 mL-headspace vials of headspace analyzer. The vials were sealed air-tight with a silicone/polytetrafluoroethylene (PTFE) septum. An aliquot of 10 µL of allyl methyl sulfide (340 µg/mL water) as internal standard was added to each vial before capping and heating. Samples were subjected to dynamic headspace for 40 min at 80 °C. Volatile compounds were concentrated in a Air Monitoring Trap (Perkin Elmer, Shelton, CT, USA), with 10 min dry purge, and desorbed during 3 min through a transfer line heated at 120 °C, directly into the injection port at 250 °C in splitless mode.

HS GC–MS analyses were performed using an Agilent 6890 Series gas chromatograph (Agilent Technologies, Heilbronn, Germany) coupled to an Agilent 5973 Series mass spectrometer (Agilent Technologies, Heilbronn, Germany) and equipped with a TurboMatrix 40 Trap Headspace sampler (PerkinElmer, Shelton, CT, USA).

Chromatographic separation was performed on capillary column (30 m × 0.25 mm I.D., 0.25 µm film thickness, HP-5MS 5% Phenyl Methyl Siloxane, Agilent 19091S-433, Agilent Technologies, Avondale, PA, USA). Helium (purity 99.999%) was used as a carrier gas at a constant pressure of 16 psi. The temperature program was as follows: 10 min at 35 °C, first ramp 10 °C/min to 190 °C, 10 min at 190 °C, second ramp at 20 °C/min to 250 °C, 3 min at 250 °C. Total analysis run was 41.50 min.

2.4.2. Mass spectrometry

The mass spectrometer was operated in Electron Impact (EI) ionization mode at 70 eV using full-scan mode from m/z 25 to 550. Source and quadrupole temperatures were 230 and 150 °C. Compound identification was carried out by injection of commercial standards, by spectra comparison using the Wiley Registry 7th Edition Mass Spectral Library (Wiley and Sons Inc., Germany) and the National Institute Standards and Technology (NIST) 2005 Mass Spectral Library and by calculation of linear retention indexes (LRI) relative to a series of alkanes (C6–C20). For a

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