Food Chemistry 197 (2016) 881–890

Contents lists available at ScienceDirect

Food Chemistry

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

Effect of drying method on volatile compounds, phenolic profile and antioxidant capacity of guava powders

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ARTICLE INFO

Article history:
Received 1 September 2015
Received in revised form 3 November 2015
Accepted 11 November 2015
Available online 12 November 2015

Chemical compounds studied in this article:
Furfural (PubChem CID: 7362)
Gallic acid (PubChem CID: 370)
Limonene (PubChem CID: 22311)
Myricetin (PubChem CID: 5281672)
Naringenin (PubChem CID: 932)
Quercetin (PubChem CID: 5280343)
Quercetin-3-*O*-rutinoside (PubChem CID: 5280805)
β-Caryophyllene (PubChem CID: 5281515)
(*Z*)-3-Hexenyl acetate (PubChem CID: 5363388)
2-Hexenal (PubChem CID: 10460)

Keywords:
Psidium guajava
Freeze drying
Oven drying
Soluble phenolics
Insoluble phenolics

ABSTRACT

We studied the chemical composition of oven and freeze dried guava powders for future use as antioxidant-rich flavour enhancers. Among thirty-one volatiles in guava powders, terpenes were predominant, even after both drying processes. In contrast, esters and aldehydes, volatiles characteristic of fresh guava fruit, appeared to have been decreased by drying. Insoluble phenolics were predominant and among the sixteen compounds identified, quercetin-3-*O*-rutinoside and naringenin corresponded to 56% of total phenolics. Drying processes decreased total phenolics contents by up to 44%. Oven drying promoted the release of insoluble flavonoids, generating mainly quercetin. Antioxidant capacity also decreased due to both drying processes, but guava powders still presented similar antioxidant capacity in comparison to other tropical fruit powders. Our results suggest that oven drying is a viable option for the production of a functional ingredient that would improve the phenolic content of cereal foods while adding desirable guava flavour.

1. Introduction

Consumption of fruits is known to lower the risk of development of several non-communicable diseases, such as cancer and stroke. Research on phenolic compounds and health has been a focus of great interest in the last decade. Phenolic compounds are known bioactives in foods and might help disease prevention.

Guava (*Psidium guajava* L.) is a tropical fruit native of the American continent rich in dietary fibre, lycopene, vitamin C and phenolic compounds, as well as pleasant and intense flavour, affording remarkable nutritional, functional and sensory properties (Flores, Wu, Negrin, & Kennelly, 2015). However, fresh guava fruit is perishable and rapidly deteriorates, leading to great economic losses. In addition, guava harvest season is brief and therefore the fruit is not available all year round, which limits its commercialisation and consumption.

In order to take advantage of the potential health benefits of guava and add value to the fruit, drying the fruit to produce a

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http://dx.doi.org/10.1016/j.foodchem.2015.11.050
0308-8146/

guava powder represents one of the possible conservation methods so as to extend shelf life, and potentially increase the use of the fruit (Zambrano-Zaragoza et al., 2013). Dehydrated foods can be easily stored for long periods, due to reduction in water content, and also offer the opportunity for the development of novel functional products when used as a replacement ingredient for nutrition bars, breakfast cereals, pasta and baking products. The use of fruit powders in food formulation is assuming a greater importance and has attracted the attention of the food industry and consumers. In fact, recent studies have reported the application of fruit powders as functional ingredients in foods such as breads, cakes and biscuits (Alves & Perrone, 2015; Ho, Aziz, & Azahari, 2013).

Drying processes occupy an important place in the food industry and different technologies can be viable options for manufacturing guava powders, including oven drying and freeze drying. The former is one of the most employed methods due to its low cost, but implies exposure to oxygen and high temperatures, which may affect fruit chemical composition, especially through oxidation and pyrolysis. On the other hand, freeze drying is generally considered as the best method for the production of high-quality dried fruit, but has a high cost. Nevertheless, although freeze drying preserves sensory attributes, some authors have reported that it might lead to loss of bioactive compounds (Aydin & Gocmen, 2015; Que, Mao, Fang, & Wu, 2008; Wojdyło, Figiel, Lech, Nowicka, & Oszmiański, 2014).

To the best of our knowledge, the effect of the drying method on volatile and phenolic compounds and antioxidant capacity (AC) of guava powders has been scarcely studied. Moreover, guava insoluble phenolic compounds have never been investigated. Therefore, the aim of the present study was to compare oven and freeze drying processing for the production of guava powders with desirable aroma and high concentrations of phenolic compounds with high AC. These powders could be used in the future as functional ingredients for the development of food products.

2. Material and methods

2.1. Materials, solvents, reagents and standards

Fluorescein, 2,4,6-tris(2-pyridyl)-*S*-triazine (TPTZ), (±)-6-hydroxy-2,5,7,8-tetramethylchromane-2-carboxylic acid (Trolox), 2,2′-azobis(2-methylpropionamidine) dihydrochloride (AAPH), alkanes standard (C_7–C_{30}), myricetin, naringenin, quercetin, quercetin-3-*O*-rutinoside and 2-hydroxybenzoic, 3-hydroxycinnamic, 4-hydroxy-cinnamic, 4-hydroxyphenylacetic, 2,4-dihydroxybenzoic, 3,4-dihydroxybenzoic, 3,4-dihydroxyphenylacetic, benzoic, ferulic, gallic, rosmarinic and syringic acids were purchased from Sigma–Aldrich Chemical Co. (St. Louis, MO). Iron (II) sulphate was purchased from Merck KGaA (Darmstadt, Germany). An SPME fibre assembly composed of divinylbenzene/Carboxen/polydimethylsiloxane (DVB/CAR/PDMS, 50/30 μm) was purchased from Supelco (Bellefonte, PA). All solvents were HPLC grade from Tedia (Fairfield, OH). HPLC-grade water (Milli-Q system; Millipore, Bedford, MA) was used throughout the experiments.

2.2. Guava samples and drying processes

Fresh red guava samples (*P. guajava* L. cv. Pedro Sato) from Rio de Janeiro state, Brazil, from the 2013 crop, were purchased at Rio de Janeiro's agricultural trading centre. Whole fruits were selected, washed, sanitised (immersion in sodium hypochlorite 100 ppm solution for 5 min) and manually fractioned in 1-cm cubes. Fresh guava fruit was dehydrated either in a freeze dryer (FreeZone®; Labconco, Kansas City, MO) at −50 °C and 0.025 mbar for 48 h or in a forced air circulation oven (330 drier, FANEM®, Sao Paulo, Brazil) at 55 °C for 22 h. Then, dried fruits were ground in a laboratory mill (MF 10 Basic; IKA® Werke, Staufen, Germany) and sieved to give a powder with a particle size of 20 mesh. Freeze dried guava powder (FDG) and oven dried guava powder (ODG) were vacuum-sealed and stored at −20 °C until analyses. The moisture contents of fresh guava fruit, FDG and ODG were 84.9%, 9.7% and 6.8%, respectively (AOAC, 2000). Both guava powders showed low water activity (up to 0.46), determined in a LabMASTER-aw analyser (Novasina, Pfäffikon, Switzerland).

2.3. Volatile compounds by HS-SPME-GC–MS

Volatiles from fresh guava fruit and guava powders were analysed by headspace solid-phase microextraction gas chromatography-mass spectrometry (HS-SPME–GC-MS), essentially as previously described (Carasek & Pawliszyn, 2006; Chin et al., 2008). For fresh guava fruit, 25 g of sample were softened with 50 mL Milli-Q water in a disperser (T-18 Basic, Ultra-Turrax; IKA®, Staufen, Germany) for 2 min at 10,000 rpm. Fruit homogenate (3.75 g) was transferred to a 20-mL vial together with a magnetic stirring bar and 1.25 g of NaCl. For guava powders, 0.8 g aliquots were placed in a 20-mL vial. All vials were sealed with an aluminium seal with a PTFE septum (Chromatography Research Supplies, Brazil). Samples were heated until equilibrium at 60 °C for 10 min.

For headspace extraction, the SPME fibre was exposed to the headspace of the sample for 25 min at 60 °C, and then was retracted and transferred immediately to the injection port of a Shimadzu GC-17A gas-chromatograph (Kyoto, Japan) for desorption for 3 min at 260 °C. The system was equipped with a Quadrex 007–5 capillary column (5% phenyl-methylpolysiloxane: 30 m, 0.32 mm i.d., 0.25 mm film; Quadrex, Woodbridge, CT). The temperatures of the injector and the interface were both 260 °C. Helium was used as carrier gas. The column oven was operated at 40 °C for 2 min, temperature programmed at 6 °C/min to 190 °C, held for 5 min, and then temperature programmed at 12 °C/min to 240 °C and held for 5 min. The GC was interfaced to a QP5050A mass spectrometer (MS) The MS was operated in electron impact mode at 70 eV ionisation energy and in scanned from m/z 40 to 500, at 1 scan/s. The fibre was conditioned in the injection port at 260 °C for 40 min before the first daily analysis and for 15 min between each chromatographic run. This procedure was enough to guarantee the absence of peaks in blank runs.

Chromatographic peaks were tentatively identified based on both mass spectra and retention index (RI). Mass spectra of volatile compounds were compared with those of the National Institute of Standards and Technology (NIST) mass spectral database. RI of each compound was calculated using *n*-alkanes (C_7–C_{30}) as external references and compared with literature data. Compounds were tentatively identified when mass spectra similarity index (SI) was higher than 90% and RI differed less than 5% from literature data. Semi-quantitative analysis of relative contents of volatile compounds was assessed by area normalisation. All analyses were performed in triplicate.

2.4. Soluble and insoluble phenolic compounds by HPLC-DAD

Extraction of soluble and insoluble phenolic compounds from fresh guava fruit and guava powders was performed in triplicate, according to the adapted methodology of Mattila and Kumpulainen (2002). For soluble phenolic compounds, 1 g of sample was extracted for 10 min with 20 mL of chilled ethanol:water solution (80:20, v/v) and centrifuged (2500*g*, 5 min, 10 °C, Sorvall ST 16R; Thermo Scientific™, Osterode, Germany). The supernatant was collected and the residue re-extracted. Supernatants were combined, the solvent was evaporated using a rotary evaporator