

Analysis of volatile compounds in 'Fuyu' persimmon: comparison of extraction techniques by GC-qMS

Maristella Martineli^{*1}; Andrea A. R. Alves¹; Thais M. Uekane¹; Agnelli H. Oliveira²; Rodrigo S. Campos²; Claudia M. Rezende¹; Marcos J. O. Fonseca²

¹Instituto de Química – Universidade Federal do Rio de Janeiro (UFRJ) - Rio de Janeiro, RJ, Brasil, Cep 21941-909

*email: maristella.martineli@yahoo.com.br; ²Empresa Brasileira de Pesquisa Agropecuária (Embrapa) – Rio de Janeiro, RJ, Brasil, Cep 23020-470

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Introduction

In Brazil, the culture of persimmon fruit has economic importance, being the South and Southeast regions the largest producers, due to their favorable climatic conditions.^[1]

The study of volatile constituents in foods by different sampling techniques is required, once factors such as temperature, rate of volatiles liberation from the matrix and their partition coefficients between the aqueous phase and the gas phase (ambient) cause intense changes in qualitative and quantitative profile of these extracts.^[6,8]

Few scientific studies report the volatile composition of persimmon. Thus, the objective of this study is to investigate the performance of different sampling techniques for the extraction of volatile compounds from persimmon cv. 'Fuyu' (*Diospyros kaki*, L.), using gas chromatography coupled with mass spectrometry (GC-qMS). For attempt the identification of the volatile profile obtained from different sampling techniques retention indices were calculated.

Experimental

Persimmon pulp six uniform fruits with total shell and mass approximately of 300 g were homogenized in a blender with distilled water, at a ratio of 1:1 (v/v) for 2 minutes.

• Liquid-Liquid Extraction with ultrasonic agitation (LLE):

50 mL of persimmon pulp was placed in an Erlenmeyer of 150mL, 25mL of dichloromethane and 2 g of sodium chloride (NaCl) were added and the mixture placed in an ultrasound bath for 10 min at ambient temperature. After, the sample was transferred to a separation funnel of 50 mL, and the organic phase (O.P) collected. This phase was dried with sodium sulfate (Na₂SO₄), and then concentrated in a fume hood to a volume of 2mL.

• Solid Phase Extraction (SPE):

The cartridges of SPE Florisil and TC18 were conditioned with 5 mL of acetone, according to Lanças (2004)^[5]. For the extraction, 2mL of

persimmon pulp was placed in the SPE cartridge and 5mL of dichloromethane was used for elution of the compounds, and concentrated in a fume hood to a volume of 1mL.

Both extractions were performed in triplicate and a volume of 2 µL of each were injected in GC-qMS.

• Solid Phase Microextraction (SPME):

100 mL of persimmon pulp was placed in an Erlenmeyer of 125mL, added 20g of sodium chloride (NaCl), heated under water bath at 50 °C and the system closed with a silicon septum. After 5 min of equilibrium the fiber was exposed into the headspace. The extraction period was 30 min, according to Barros^[2]. The volatile compounds were desorbed at injector port at 200 °C of the GC-qMS for 5 min. The extractions were performed in triplicate.

Results and discussion

Comparing the different techniques, it was observed that the extraction with SPE Florisil cartridges presented a larger number of volatile compounds (15), being identified as: limonene, γ-terpinene, nonanal, benzaldehyde, ethyl benzoate, 4-ethoxy-ethylbenzoate, 4-methyl-2-heptanone, acetophenone, 2,6-bis(1,1-dimethyl)-2,5-ciclohexadiene-1,4-dione, 4-hydroxy-4-methyl-2-pentanone, benzyl alcohol, 2-ethyl-hexanol, α-ethylbenzyl alcohol, linalyl propionate and bornyl acetate, when compared with LLE that presented four compounds only (limonene, tetradecanoic acid, dodecanoic acid and hexadecanoic acid). In SPME technique, the number of volatiles compounds observed also was lower (8 compounds with the triple fiber (PDMS/DVB/CAR): limonene, selina-1, 3,7(11)-trien-8-one, nonanal, decanal, 1,3-isobenzofurandione, octyl acetate, methyl decanoate and methyl nonanoate and 11 compounds with PDMS fiber: α-copaene, selina-1, 3,7(11)-trien-8-one, α-terpinolene, citronellyl acetate, β-elemene, selina-1,3,7(11)-trien-8-one oxide, decanal, methyl hexadecanoate, 1,3-isobenzofurandione, geranyl acetone and 2,6-bis(1,1-dimethyl)-2,5-1-ciclohexadiene-1,4-dione.

Previous studies in persimmon volatiles were performed by Taira et al. (1996)^[7], by steam

distillation, where a great amount of alcohols was observed, possibly due to an increased in matrix degradation because of prolonged time of heating. Horvat et al. (1991)^[4] detected bornyl acetate and (*E*)-2-hexenal as major compounds and nine other compounds as minor components in astringent and non-astringent persimmon varieties. In the present work, the extraction with SPE Florisil permitted to observe esters, ketones, alcohols and aldehydes, with aromatic notes of citrus, orange, fat, green, floral, fragrant, aromatic, sweet, fresh, oily-green-winey, alcoholic, mild pine, and herbaceous^[3]. Relative to SPME technique, distinguished profiles were observed with different fibers, once triple fiber captured more esters (fruit flavors, coconut and wine) and PDMS fiber a greater number of terpenes (monoterpenes and sesquiterpenes) with uncharacteristics aromas of persimmon such as wood, spice, rose, dust, herb, wax, fresh and gasoline^[3].

Conclusion

Volatile compounds in persimmon cv. 'Fuyu' were analysed by GC-qMS and by the best extraction method was with SPE Florisil using a nonpolar column (DB1). Different classes of organic compounds were observed and associated to the fruity aroma of persimmon.

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