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Valencia Late orange juice preserved by pulp reduction and high pressure homogenization: Sensory quality and gas chromatography–mass spectrometry analysis of volatiles

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A B S T R A C T

Low pulp Valencia Late orange juice was obtained by high-pressure homogenization (HPH) at 150 MPa as an alternative to thermal pasteurization processes. Colour and cloudiness were improved through a soft pre-homogenization at 20 MPa applied before centrifugation. Initially and until 0.5 months of refrigerated storage, it was not distinguishable from the fresh juice and was more acceptable than its counterpart pasteurized at 85 °C for 15 s. At 1.5 months onwards of storage (till 3.5 months), acceptability, colour and transmittance of both juices became similar. Moreover, the effects of processing and storage on the aroma profile of juices were evaluated by Gas Chromatography coupled to Mass Spectrometry (GC–MS) analysis with an automated data processing performed by the Automated Mass Spectral Deconvolution and Identification System (AMDIS). There were determined a total of 88 volatiles, following the evolution of 32 aroma descriptors. Compared with the pasteurized sample, the aroma profile of the double homogenized juice was initially closer to fresh juice and presented lower concentrations in off flavour compounds during storage.

1. Introduction

Commercial pasteurized chilled citrus juices are usually obtained by mechanical extraction of the whole fruit, removal of membrane debris and coarse pulp and thermal pasteurization at 90–92 °C for 30–60 s. Since pectinmethylesterase (PME) activity of citrus juices, responsible of their clarification, is by far more thermoresistant than the spoilage microorganisms, these strong pasteurization conditions are needed to reduce PME activity until a residual level compatible with refrigerated storage. As a consequence of thermal pasteurization, a decrease of "freshness" and sensory quality of juice is inevitable, appearing a perceptible "cooked" smell and taste (Berry & Veldhuis, 1997) promoted by the apparition of off flavour compounds.

A way to avoid this loss of freshness is the use of non-thermal processes such as pulsed electric fields (Sentandreu, Carbonell, Rodrigo, & Carbonell, 2006; Walkling-Ribeiro, Noci, Cronin, Lyng, & Morgan, 2009) or high hydrostatic (Katsaros, Tsevdou, Panagiotou, & Taoukis, 2010) and dynamic pressures (high-pressure homogenization, HPH, Donsi, Ferrari, & Maresca, 2010). All these processes minimally affect sensory quality of juices and seem to be effective for their microbial inactivation, but their effectiveness on the reduction of PME activity is reasonably low. Welti-Chanes, Ochoa-Velasco, and Guerrero-Beltrán (2009) achieved a decrease of only 50% in PME activity after a single pass through an HPH system working at 250 MPa meanwhile Lacroix, Fliss, and Makhlouf (2005) reported a decrease of only 20% after three passes through a homogenizer working at 170 MPa. Similarly, Katsaros et al. (2010) achieved a maximum inactivation of 85% in Valencia Late orange juice but under severe static pressure conditions (350 MPa at 35 °C for 2 min).

In a high-pressure homogenizer, suspended particles of juice suffer breakdowns caused by shear forces, impacts and sudden pressure drop. Part of the liberated energy in the homogenizer is consumed to break droplets and solids meanwhile the remaining energy is converted into friction heat, increasing the temperature of the sample. In this line, Belloch, Gurrea, Tárrega, Sampedro, and Carbonell (2012) described how in Valencia Late orange juice, the combined effect of pressure and temperature generated during the HPH treatment achieved an inactivation level higher than 5 log cycles in *Listeria innocua* (working at 110 MPa and 48 °C during 2 s) and in *Lactobacillus plantarum* (working at 150 MPa and 56 °C for 20 s).

Homogenization at 150 MPa increases up to 60–65 °C the temperature of a juice fed to the system at 22–28 °C. A temperature

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of 60 °C (held for 15 s) produced enough microbial inactivation in short life chilled juices (Torres, Bayarri, Sampedro, Martinez, & Carbonell, 2008) without thermal damage, since as pointed out by Sentandreu, Carbonell, Carbonell, and Izquierdo (2005) juices treated at temperatures below 70 °C did not differ in sensory acceptability from the original fresh juice. Nevertheless, heating at 60–65 °C does not induce efficient PME inactivation. Torres et al. (2008) reported that a treatment at 60 °C for 15 s reduced PME activity of a clementine juice around 50% of its initial value, far away the recommended 90% for short life chilled citrus juices (Irwe & Olson, 1994).

But there is an alternative way to reduce PME activity in citrus juices that consists in reducing the pulp content by centrifugation, since PME is cell-wall enzymes associated with pulp (Rouse, 1953). The pulp content reduction caused by centrifugation can lead to juices with less colour and cloudiness, a problem that can be easily avoided by converting part of the suspended pulp into coloidal pulp. This conversion decreases the particle size of pulp and can be achieved through a soft homogenization of the whole juice before centrifugation.

Evidently, technological processes aiming to stabilize orange juices can significantly modify their chemical composition, mainly for heat sensitive compounds and/or those directly linked to the pulp of juices such as volatile constituents. Compositional modifications affect flavour characteristics of the resultant products, having a key role in their acceptability by consumers. However, orange juice has a very complex aroma profile needing the use of high-throughput methodologies of analysis for its study, having Gas Chromatography coupled to Mass Spectrometry (GC–MS) as the traditional election of choice. But in addition to the intricate aroma composition of juices, the high sensitivity of GC–MS systems generates an immense quantity of experimental data that needs to be appropriately processed, with the subsequent consumption of time. To overcome this problem, bioinformatics can help analysts in their task of performing an accurate processing of such quantity of raw data. Ideally, analytical programs should be cheap, easy to use, versatile, fast and able to deconvolute overlapped chromatographic peaks, thus providing reliable qualitative and quantitative results. Currently, the National Institute of Standards and Technology-Automated Mass Spectral Deconvolution and Identification System (NIST-AMDIS) satisfies all these requirements.

Recently, Cerdán-Calero, Sendra, and Sentandreu (2012) demonstrated the utility of AMDIS to determine the aroma profile of freshly squeezed Valencia Late orange juice through Head Space Solid-Phase Microextraction (HS-SPME) and GC–MS analysis. Authors clearly remarked that the reliability of the automated analysis performed by AMDIS strongly depends on the optimization of different settings and conditions of analysis (belonging to AMDIS and the considered platform of analysis). Moreover, it was also concluded that although AMDIS was a very reliable system of analysis, the requested optimization as well as the generation of the customized in-house libraries needed to perform an automated and reliable data processing was tedious, only recommending AMDIS for bulky data analysis.

This research aims to study the residual PME activity, cloudiness, colour, acceptability and aroma composition of a chilled orange juice pre-homogenized at 20 MPa, partially depulped by centrifugation and homogenized at 150 MPa (with an outlet temperature of 65 °C for 15 s). Flavour and physicochemical properties of this juice were evaluated during its refrigerated storage taking as reference two partially depulped juices, one treated in a similar way but without pre-homogenization and the other one pasteurised in a heat exchanger at 85 °C for 15 s. Additionally, reliability of AMDIS to perform multiple data processing was tested, determining the aroma profiles of the assayed juices.

2. Material and methods

2.1. Sample preparation

Valencia Late orange (*Citrus sinesnsis* L. Osb.) fruits were harvested in June 2010 from an orchard located in Lliria (Valencia, Spain). Fruits (400 kg) were washed by immersion in tap water, drained, sized and squeezed in an industrial extractor with finger cups (Exzel, Luzzysa; El Puig, Valencia, Spain). Raw juice (180 L) was sieved in a paddle finisher (Ø 0.4 mm, Luzzysa) giving sample A as shown in Fig. 1 (scheme of operations to obtain the different juices studied). To prepare samples E and F, part of raw juice (100 L) was pre-homogenized with a Manton–Gaulin homogenizer (model 15M8TBA) working at 20 MPa, meanwhile the remaining juice (to prepare sample D) was not. Low pulp juices (LPJs, samples B and C) were obtained using a Westfalia centrifuge (model SAOH 205) fed at 60 L/h, finally blending 75% of centrifuged with 25% of non-centrifuged juice. Sample F was from the pasteurization of sample C using a plate heat-exchanger (model Junior, APV Ibérica S. A., Madrid) fed at 1 L/min in where the juice was heated at 85 °C for 15 s and finally cooled at 7 °C for 10 s. By their hand, samples D and E were from the homogenization at 150 MPa of samples B and C respectively using a GEA Niro-Soavi homogenizer, model NS3015 H with 420 mL of residence volume (GEA Niro Soavi S.p.A. Via da Erba Edoari 29, Parma, Italy). The homogenizer was fed at 100 L/h at 28 °C, reaching an outlet temperature of 65 °C for 15 s during the HPH treatment and finally cooling the juice at 7 °C in the cooling section of the system. Samples D, E and F were aseptically packed in 946 mL glass jars with twist-off cups that were previously sterilized with fluent steam. Delay between extraction and packaging of juices was about 40 min at room temperature (20 °C). Finally, samples were stored at 3 °C and analysed after 0, 0.5, 1.5, 2.5 and 3.5 months of storage.

2.2. Reagents and standards

HPLC gradient grade methanol was from Scharlab (Scharlab S. L., Barcelona, Spain). Analytical grade ethyl nonanoate standard was from Sigma (Sigma–Aldrich Co., St. Louis, MO, USA).

2.3. °Brix, acidity and pH

Total soluble solids were measured as °Brix with a digital refractometer (Pal-1; Atago Co., Ltd., Tokyo, Japan). Total acidity

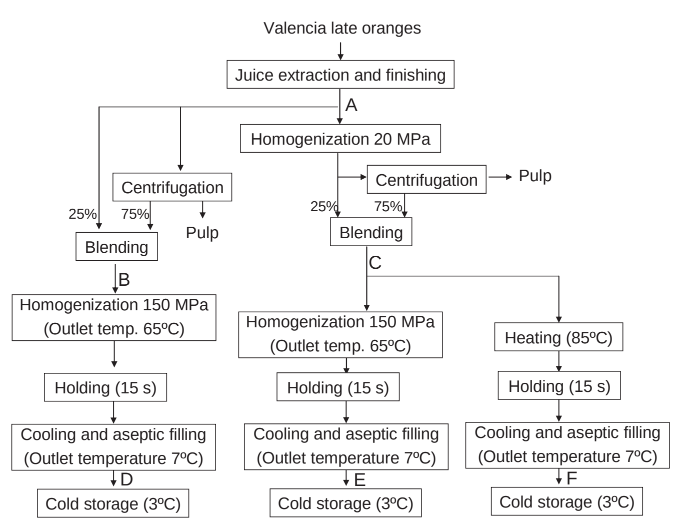

Fig. 1. Diagram of treatments applied to obtain the different Valencia Late orange juices (see Table 1 for nomenclature).