



Odor potency, aroma profile and volatiles composition of cold pressed oil from industrial passion fruit residues



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ABSTRACT

In Brazil, the industrial exploitation of passion fruit generates agro-industrial waste potentially dangerous to the environment. Thus, the objective of the present research was to explore the use of the waste produced during passion fruit processing (*Passiflora edulis f. flavicarpa*) for the manufacture of aromatic oil. The waste was dried, ground, cold pressed, and centrifuged. The oil was characterized for its moisture content, density, acidity, and for its refractive, iodine, and saponification indexes. The volatile compounds present in the headspace of the oil were isolated using the SPME technique and identified by GC-MS. Trained panelists evaluated the odoriferous strength of the oil as compared to fresh passion fruit pulp using the magnitude estimation scale, and quantitative descriptive analysis. Forty-nine volatile compounds were identified in the oil, the esters representing the major compounds with approximately 60% of the total area of the chromatogram, followed by the alcohols (15.5%), and terpenes (9.5%). Ethyl butanoate, ethyl hexanoate, and hexyl acetate were the major compounds found in the oil. The oil possessed high odoriferous strength, from two to three times superior to that of the pulp, an aroma profile similar to that of fresh passion fruit and good potential for use in the manufacture of aromatizing products.

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1. Introduction

In Brazil, the production of concentrated passion fruit juice almost tripled in a recent 5-year period, increasing from approximately 4.4 thousand tons in 2005 to approximately 11.2 thousand tons in 2010 (IBGE, 2013). This expansion is important and desirable, since this sector generates income and jobs and strengthens the economy. However, such growth also contributes to the generation of large amounts of agro-industrial residues, produced during the processing of the raw material.

The residues from passion fruit processing represent 76% of the weight of the processed fruits, of which 26% is made up of the seeds (Ferrari et al., 2004). Annually, these seeds amount to tons of solid residues whose disposal involves operational costs for the pulp and juice industries, and represents an environmental problem (Malacrida and Jorge, 2012).

After drying, passion fruit seeds show a lipid content of up to 30%, and are therefore a good source of oil (Malacrida and Jorge, 2012). Some researchers have studied different methods to extract this oil (Liu et al., 2009; Oliveira et al., 2013) whereas others have characterized the physical and chemical properties of the oil, notably the fatty acid profile, the presence of phenolic compounds, the antioxidant capacity of the material, the carotenoid content, and the contents of other bioactive compounds (Nyanzi et al., 2005; Oliveira et al., 2009; Ferreira et al., 2011). However, up to the present moment studies exploring the potential of passion fruit oil in the production of natural aromas are rare despite the increasing world demand for ingredients obtained from natural sources. Thus, the objective of the present study was to evaluate the odor potency, aroma profile, and volatile composition of the oil extracted from industrial passion fruit residues, so as to evaluate the potential of this material for the production of natural aromas.

2. Material and methods

2.1. Raw material

About 10 kg of industrial residues obtained after depulping passion fruit were dehydrated in a tray drier with the air flow

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temperature fixed at 45 °C, until they reached a moisture content of about 6%. The dehydrated residues were ground in a Willye TE 650 knife mill equipped with a 20 mesh sieve (0.85 mm), and subsequently analyzed chemically and submitted to oil extraction. The residues consisted mainly of the seeds.

2.2. Chemical characterization of the raw material

The moisture, lipid, ash, and crude fiber contents of the dehydrated, ground residues were determined according to the methods described in [AOCS \(2009\)](#). The protein content was determined according to the [AOAC \(2005\)](#) method and the carbohydrate content obtained by difference. All analyses were carried out in triplicate.

2.3. Extraction of the oil from the passion fruit processing residue

The dried, ground residues were submitted to cold pressing in order to extract the oil, using a hydraulic press (TECNAL model TE-098) with a force of 16 tons for a total period of 100 min. The material was then transferred to a 45 mL graduated Eppendorf tube with a screw top, and centrifuged at 4000 rpm for 20 min at a temperature of 25 °C in a model 5804R Eppendorf centrifuge, to break the water–oil emulsion and separate the impurities. The oil was extracted three times, and the yield calculated for each repetition by w/w difference and expressed as g oil/100 g dehydrated residue. The material obtained was stored at a temperature of –18 °C in amber glass jars with hermetic seals.

2.4. Characterization of the oil extracted from the passion fruit residues

The oil extracted from the passion fruit residues was characterized with respect to its moisture content, acid index (mg KOH/g), saponification index (mg KOH/g), iodine index (g I₂/100 g), relative density, and refractive index (40 °C) according to AOCS methodology (2009).

The volatile compound composition, odoriferous power and aroma profile of the oil were also determined using the methodologies specified below.

2.4.1. Isolation and identification of the volatile compounds

For the isolation of the volatiles, 2 g of the passion fruit residue oil were placed in a 10 mL amber flask and submitted to slow agitation for 15 min at a temperature of 60 ± 2 °C. After reaching equilibrium, a 75 µm SPME Carboxen/Polydimethylsiloxane fiber (CAR/PDMS, Supelco, Co, Bellefonte, PA, USA), previously tested by [Carasek and Pawliszyn \(2006\)](#) was exposed to the volatiles present in the headspace for adsorption during 30 min at 60 ± 2 °C, and subsequently inserted into the injector of the GC-MS for desorption of the volatiles.

The volatiles were separated in an Agilent model GC 7890A/5975C (Palo Alto, USA) gas chromatograph coupled to a mass spectrometer, operating under an ionization voltage of 70 eV in the scan mode. A fused silica capillary column DB-5 (30 m × 0.25 mm, 0.25 µm; JW Scientific, Folsom, CA, USA) was used to separate the volatiles. Initially, the column temperature was maintained at 40 °C for 2 min and then raised to 100 °C at 2 °C/min, then to 270 °C at 30 °C/min, and finally maintained at 270 °C for 10 minutes. Helium was the carrier gas at a flow rate of 1.2 mL/min. The temperature of the injector was maintained at 280 °C, and the fiber exposed for 5 min for heat desorption of the volatiles. The injection was made in the splitless mode and the interface temperature was maintained at 300 °C. The volatiles were also separated using a DB-Wax (JW Scientific) fused silica capillary column (30 m × 0.25 mm, 0.25 µm) under the same conditions described

above, but with the injector temperature maintained at 250 °C and the interface temperature maintained at 260 °C. The column temperature was maintained at 40 °C for 2 min and then raised to 100 °C at 2 °C/min, then to 250 °C at 30 °C/min, and finally maintained at 250 °C for 10 min.

To help in identification of the volatile compounds, a series of hydrocarbon standards (C7–C30, Polyscience 211C kit, Chicago, USA) was isolated using the same CAR/PDMS fiber and injected into the equipment (onto the two columns mentioned above), under the same conditions used for the sample analyses. The retention index of each column was calculated from the retention times of the standards (C7–C30) as described by [Van Den Dool and Kratz \(1963\)](#). Mass spectra and experimental retention indexes were also obtained from pure standards under the same conditions used for the sample analyses. The following techniques were used to identify the volatiles present in the oil isolated from the residues: the spectra available in the NIST (2008), the retention indexes reported in the scientific literature ([Jordán et al., 2002](#); [Carasek and Pawliszyn, 2006](#); [El-Sayed, 2012](#); [Janzantti et al., 2012](#)), and both the mass spectra and experimental retention indexes of the following pure standards were purchased from Sigma Aldrich (Steinheim, Germany): ethyl butanoate, ethyl hexanoate, benzaldehyde, hexanol, benzyl alcohol, β-myrcene, D-limonene and linalool.

As described by [Sampaio et al. \(2011\)](#), the compounds were considered positively identified if their mass spectra and retention indices were compatible with those of the pure standards. They were considered identified when their mass spectra matched those available in the computerized library, and their retention indices were comparable to those reported in the literature. Compounds were considered tentatively identified when their identification was based solely on their mass spectrometric data.

2.4.2. Sensory analysis

The passion fruit oil was characterized from a sensory point of view with respect to its aromatizing power and aroma profile. The analyses were carried out in comparison with the natural pulp of yellow passion fruit (*Passiflora edulis f. flavicarpa*) diluted to a concentration of 6.25 mL/100 mL in water. This is the dilution recommended by Brazilian frozen pulp processors for the elaboration of passion fruit juice. For all the sensory evaluations, the samples (30 mL) were served at room temperature in tulip-shaped glasses wrapped in aluminum foil, covered with a watch glass, and coded with random three digit numbers.

2.4.2.1. Training and selection of the sensory panel. Initially, 14 volunteers from the Department of Food Technology of the Federal University of Sergipe were trained to evaluate the aroma of yellow passion fruit pulp (*Passiflora edulis f. flavicarpa*) at different intensities using a 9-point structured scale and Spectrum® methodology, as described by [Meilgaard et al. \(2007\)](#). Each panelist then evaluated the intensity of the passion fruit aroma present in six samples prepared by diluting natural passion fruit pulp in water at different concentrations, using another 9-point structured scale. The individual results of each panelist were then evaluated by ANOVA using the methodology described by [Sampaio et al. \(2011\)](#) (sources of variation: sample and repetition), and the panelists who presented good discriminative power ($p_{\text{sample}} \leq 0.05$) and good repeatability in their scoring ($p_{\text{repetition}} > 0.05$), were selected to take part in the sensory panel that carried out the subsequent tests. This panel consisted of 12 judges.

2.4.2.2. Odoriferous power of the oil extracted from passion fruit residues. The selected judges were initially trained to use the magnitude scale proposed by [Stevens \(1956\)](#). They then analyzed the aroma of six samples of passion fruit pulp diluted in water at the following concentrations: 1.56 mL/100 mL,

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