

THERMAL AND STRUCTURAL STUDY OF GUAVA (*Psidium guajava* L) POWDERS OBTAINED BY TWO DEHYDRATION METHODS

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Two food products (powders) were obtained by hot-air drying or lyophilisation methods on the whole guava fruits. The powders were characterised by sensory and thermal analyses (TGA-DSC), infrared spectroscopy (IR), X-ray diffraction (XRD) and scanning electron microscopy (SEM). Thermal, morphological and structural characterisations showed a similar behaviour for the two solids. TGA-DSC and IR showed the presence of pectin as the main constituent of solids. A semi-crystalline profile was evidenced by XRD, and lamellar/spherical morphologies were observed by SEM. Sensory analyses revealed an aroma highly related to guava. These value-added food products are an alternative to process guava and avoid loss during postharvest handling.

Keywords: *Psidium guajava*; freeze-dried powder; hot air dried powder.

INTRODUCTION

Guava (*Psidium guajava* L) is a tropical fruit native of America, which has been spread to different countries, since Brazil until Mexico and Florida, due to its wide acceptance. This fruit exhibits excellent nutritious, sensory and biofunctional properties. Among them, it is remarkable the high vitamin C content (269 mg/100 g),¹ dietary fibre (48,55-49,42%),² antioxidant compounds,³ and its intense and pleasant aroma.^{4,5} Dietary fibre is largely composed of complex carbohydrates that are beneficial to digestion, because they contribute to maintain the colonic microflora and to remove waste and toxins.⁶ A major component of soluble fibre is pectin, which is an anionic polysaccharide mainly composed of partially esterified D-galacturonic acid monomers linked by α -(1-4) bonds.⁷ To this regard, red guava exhibits a high pectin content (1,04-1,74 g/100 g of fresh weight)⁸⁻¹⁰ in comparison with other fruits. Guava pectin has a high methoxy index that imparts viscous properties to guava purees or juices.¹¹

In Colombia, it has been selected as a productive promising species by the government. However, the production of red variety ("regional roja" variety) guava has some technological limitations as consequence of a deficient phytosanitary handling and its susceptibility to storage due to the accelerated metabolism during ripening, resulting in significant losses for producers. The relative humidity and temperature are the more critical variables to extend the shelf-life of the fruit and to preserve its sensory, physical and chemical properties. Thus, dehydration processes may be an efficient alternative for fruit storage, because the reduction of water activity is related to the decline of chemical and enzymatic reactions responsible for the deterioration of foods. Dehydration is based on the extraction of water contained in foods up to a minimal level which is enough to their conservation for long time. The hot-air drying is one of the most used methods for preservation of fruits since long time ago;¹² however the main problem is the possibility to lose or change some of their properties due to the thermal treatment.

Gentler drying techniques such as, osmodehydration or lyophilisation (freeze-drying) has also been used in fruits.^{13,14}

With the aim to find new alternatives for the commercialization of guava, and to reduce the difficulties for its handling and transport, hot-air drying and lyophilisation techniques were used to obtain added-value solids from guava. Thus, the objective of this study was to perform the physicochemical, structural, and morphological analyses of the above-mentioned dehydrated guava powders. A strict knowledge on the characteristics of those guava materials should be established in order to recognize the best technique for guava dehydration and for the future optimization of different parameters involved in this technological process at industrial scale.

EXPERIMENTAL

Material

Ripe red guava fruits (*Regional roja* variety) were purchased at Puente Nacional and Barbosa (Santander, Colombia) commercial crops. Fully ripe fruits were characterized by their pH (4,3) and solid soluble content (8,5 °Brix), in a Schott CG8220 pH-meter and an Abbe Atago 8682 (at 20 °C) refractometer, respectively.

Methods

Preparation of dehydrated guava powders

Two different guava powders were obtained by hot-air drying (G-HA) or lyophilisation (G-LF) processes, from 100 and 300 kg of guava, respectively. Before processing, the fruits were dipped into sodium hypochlorite solution (1 w/v %) to achieve an acceptable sanitary status and then processed without previous freezing. G-HA Powder was obtained by drying of guava slices of 0,5 cm in thickness at 70 ± 2 °C, during 8 h to reach a humidity value of ca 6%. After that, a portion of dried product was ground in a mill to obtain 10 kg of G-HA and storage them at 18 °C in darkness. For G-LF powder, guava slices (0,5 cm thickness) were frozen at -15 °C for 12 h before

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the freeze-drying in a Lyomac lyophiliser at 10^{-2} Pa (90 microbar) for 10 h, and then ground in a mill to get 10 kg of dried powder.

Physicochemical characterization

Powders moisture content was determined gravimetrically by drying in an oven at 105 °C until constant weight. The crude fibre content was determined following the procedure published by AOAC.¹⁵ Bulk density of powders was measured by weighing 260 g of sample and placing it into a 250 mL graduated cylinder. The bulk density was calculated by dividing the mass of powder by the volume filled in the cylinder.¹⁶ The fluidity was determined by weighing 150 g of each solid and placing it in a plastic funnel over a vibrator (VEB Koombinat, thyv2) and registering the time that powder spend to be deposited.

Thermal and morphological analyses

Thermogravimetric measurements were carried out with a thermogravimetric analyzer TGA 2050 (TA Instruments, accuracy $\pm 0,1\%$; resolution 0,2 μg) coupled with thermal analyzer Dupont 990. The powders were also analyzed by using a differential scanning calorimeter (DSC 2910, TA Instruments) and the TA Instruments software (Universal V2.5H). The apparatus was calibrated with high-purity indium. The experiments were performed under a nitrogen flow. Samples (2 mg) were heated from 0 to 400 °C in aluminium crucibles with a linear heating rate of 10 °C/min and using an empty aluminium crucible as a reference material.⁹

Morphology of the powders was evaluated using a scanning electron microscope FEI Quanta 200 (operating at 30 kV), coating them by gold sputtering before their examination.

X-ray diffraction (XRD) and infrared (IR) analyses

Infrared analyses were carried out in an IR Prestige-21 FTIR-spectrophotometer (Shimadzu) with transmission geometry. Before analysis each sample was maintained for 4 days into a vacuum desiccator with silica gel and CaCl_2 , and then 2 mg of sample was mixed (in agata mortar) with 200 mg of dried KBr to be compressed in a thin tablet. X-ray diffraction analyses were performed using an X-Pert Pro MPD PANalitical equipment with 2θ geometry and a Bragg-Brentano configuration, at room temperature and a step size of 0,01 and a step time of 2 s.

Volatiles analyses

The volatile compounds released from the headspace of dehydrated guava powders were analyzed by HS-SPME (headspace-solid phase microextraction). A portion of each sample (2 g) was placed during 45 min in a 20 mL sealed vial at 40 °C to reach equilibrium. The headspace was collected on a CAR/PDMS fibre (75 μm thickness, Supeltex) during 1 h, and then directly injected (desorption time was set at 5 min) into a GC17A Shimadzu gas chromatograph equipped with an FID and operated in splitless mode. A DB-FFAP fused silica column (30 m x 0,32 mm i.d., 0,25 μm film thickness) was used. The column oven was programmed from 50 (after 4 min) to 250 °C at 4 °C/min and the final temperature was held for 5 min; the injector temperature was maintained at 250 °C; carrier gas was 1,5 mL of He/min; and make up gas was nitrogen at 30 mL/min flow

rate. A DB-5 column was also used with the following conditions: the column oven was programmed from 50 (after 4 min) to 300 °C at 4 °C/min and the final temperature was held for 5 min; the injector temperature was maintained at 300 °C; carrier gas was 1,5 mL of He/min. GCMS analyses were performed in a gas chromatograph Shimadzu GC17A coupled to a selective mass detector QP5050, using the same conditions used in GC analyses (DB-FFAP column). Linear retention indices were calculated according to the Kovats method using a mixture of normal paraffin $\text{C}_6\text{-C}_{28}$ as external references. Mass spectral identification was completed by comparing spectra with commercial mass spectral databases Wiley and EPA/NIH and by comparison with authentic reference standards.¹⁷

An odour profile description of the volatile extracts was obtained using a sniffing port with an FID/sniffing 1:1 ratio (microvalve, OSS-2, SGE) connected to a Hewlett Packard 5890 series II and operated under the conditions above mentioned (DB-FFAP column). The effluent going to the sniffing port was heated (60 °C) through a glass-lined capillary and mixed with humidified air. Each sample was sniffed 3 times by trained panellists who described the odour of detectable compounds.

RESULTS AND DISCUSSION

Physicochemical analysis

The preliminary characterization of these powders showed that they had a residual content of vitamin C and also exhibited *in vitro* antioxidant activity, with the freeze-dried powder showing the highest values (G-HA 118,7 mg of ascorbic acid/100 g of solid and 1,44 mmol of Trolox/100 g of solid; G-LF 948,7 mg of ascorbic acid/100 g of solid and 32,6 mmol of Trolox/100 g of solid).¹⁸ These results were an important starting point for the characterization of these guava solids in order to a future development of value-added natural food products from them. In this sense, some physico-mechanical properties, such as bulk density and fluidity were evaluated (Table 1), which should be considered for pre-formulation studies. The values of fluidity and bulk density were higher in the G-HA solid. The residual moisture after drying processes was higher in the G-HA solid (5,37% for G-HA and 2,75% for G-LF). The composition of powders was around 50% of crude fibre with high pectin content.

Thermal analysis

Thermal analysis curves (Figure 1a and 1b) show some differences between G-HA and G-LF powders. Although the TGA curves show three general regions of weight loss (50-150; 150-350; 350-470 °C), a double stage of mass drop for the solid G-HA can be distinguished in the second mentioned region, related to a possible more complex volatilisation of matter. The first region (50-150 °C) reveals a weight loss around 6% for G-HA solid; while the solid G-LF show a loss less than 2% (Figure 1c). This drop of mass can be assumed as water loss during the temperature rise. The third region (350-470 °C) in the TGA analyses shows a more stable stage after volatilization of water and other compounds: between 150 and 350 °C dehydroxylation, demethoxylation and decarboxylation reactions

Table 1. Physicochemical analysis of guava dehydrated powders

Sample	Fluidity (g/min) ^a	Bulk density (g/cm ³) ^a	Moisture content (%) ^a	Crude fibre (%)
G-HA	20,27 \pm 0,91	0,43 \pm 0,02	5,37 \pm 1,63	47,0
G-LF	5,74 \pm 0,34	0,24 \pm 0,01	2,75 \pm 1,72	54,5

^a All data are the mean values of triplicate measurements \pm standard deviation.

should be occurring on pectin¹⁹ and similar polysaccharides (pectin is the main component of dehydrated guava). The total loss of weight for samples (63% for G-LF and 67% for G-HA) indicates that freeze drying is a technique slightly more suitable to give more thermally stable solids. So, it seems that lyophilisation is more effective than hot-air drying to yield dried powders of guava; however these thermogravimetric results are comparables.

On the other hand, DSC curves (Figures 1a, 1b and 1d) show a broad and complex endothermic peak for both samples (centred at 130 °C for the solid G-HA and at 150 °C for solid G-LF) followed of a shoulder (close to 200 °C) more pronounced for the G-LF material. This complex endothermic peak involves the volatilization

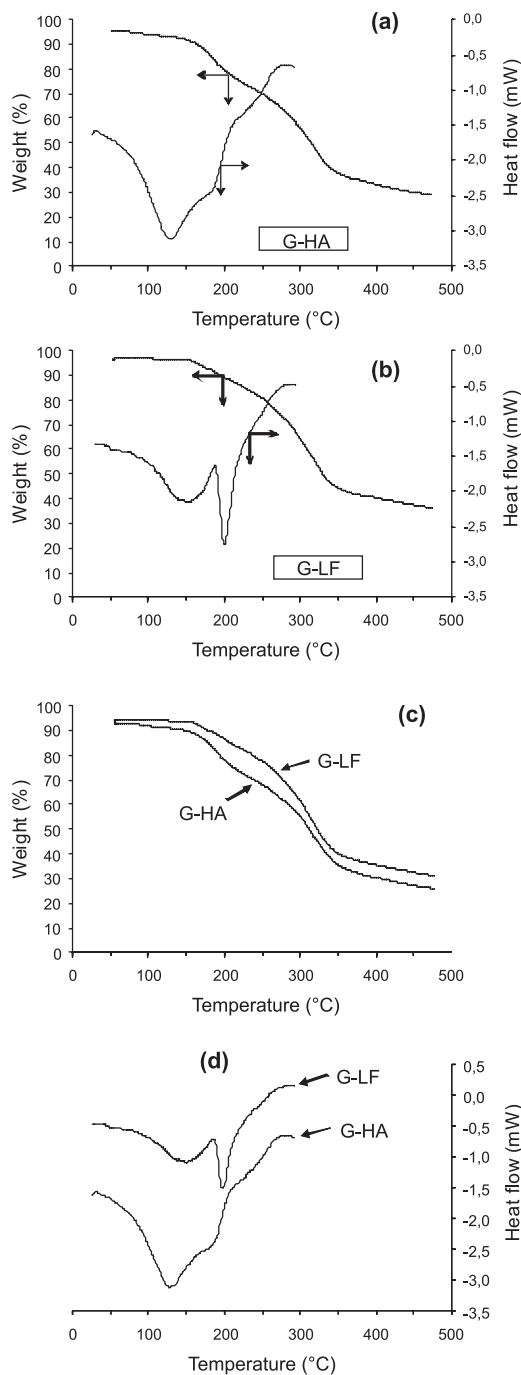


Figure 1. Thermal analysis curves (TGA-DSC) of powders obtained by dehydration of guava (a, b) and the comparison of TGA (c) and DSC (d) curves. G-HA: hot-air dried solid; G-LF: lyophilised solid

processes of water, melting of pectin contained in guava^{13,20} and a possible demethoxylation, dehydroxylation and decarboxylation of pectin and other polysaccharides.¹⁹ A slight shift of discussed peaks toward lower temperature values is observed for the G-HA sample (Figure 1d), probably due to a higher water content²⁰ and its lesser thermal stability. In addition, almost a double intensity of G-HA peak regarding G-LF peak is clearly observed, and so a larger flow of heat, revealing more significant changes on the G-HA sample and thereby a lower thermal stability of this material.

IR and XRD analyses

The IR spectra of dehydrated guava samples revealed the characteristic signals of polygalacturonic acid (pectin)²¹⁻²³ or similar components.²⁴ A broad and intense band at 3400 cm^{-1} is assigned to -OH stretching vibrations; an intense band at 2930 cm^{-1} corresponds to C-H stretching of $\text{-CH}_2\text{-}$ groups; bands that appear at 1743 and 1630 cm^{-1} can be assigned to C=O stretching vibration of methyl esterified carboxylic group and vibrations of the carboxylate (-COO^-) group, respectively. The 1630 cm^{-1} peak is accompanied of a signal at 1450 cm^{-1} , which is common in this group because they correspond to the asymmetrical and symmetrical stretching vibrations.^{21,24} The effect of shifting the carboxylate group vibrations to slightly higher wavenumbers regarding those described in IR spectroscopy literature could be attributable to several interactions of COO^- groups with other neighbour groups of galacturonic acid in the polymeric environment and a consequence of the samples in solid state.²¹ On the other hand, a vibration observed at 1250 cm^{-1} indicates the presence of C-O bond, which is common in several organic compounds, and the intense signal at 1064 cm^{-1} can be attributed to stretching vibrations of C-O-C bonds in ethers or related compounds.²⁴ This band is characteristic of carbohydrates, which show high absorbance between 1200 and 950 cm^{-1} wavenumbers values.²¹

Carboxylic groups of galacturonic acid monomers can be esterified with methanol, and the percentage of esterified groups is expressed by the degree of methoxylation (DM) or methoxy index. According to Manrique and Lajolo,²¹ the proportion between absorption bands at 1743 and 1630 cm^{-1} can be related with the esterification degree of pectin, taking into account the IR vibrations described above for these bands. In this way, a higher ratio of peak areas ($A_{(1743)}/A_{(1630)}$) was observed for G-LF than that of G-HA sample (1,9 for G-LF and 0,8 for G-HA), indicating a higher fraction of esterified groups in pectin contained into the lyophilised powder. This result reveals that hot-air drying technique is more aggressive on the structural components of guava, causing a de-esterification and perhaps a partial depolymerisation of pectin.

XRD analysis of dehydrated solids showed a similar semi-crystalline profile in both cases, with the characteristic peaks of pectin.^{7,22} The main interplanar spacings (separation of planes, d) for guava pectin were $d = 4,0 \text{ \AA}$, $d = 5,3 \text{ \AA}$ and $d = 8,8 \text{ \AA}$. Higher intensity of G-HA sample indicates a slightly higher level of crystallinity. This effect can be interpreted as a more organized structure of pectin molecules, probably due to the additional association of chains as a consequence of interactions produced by a higher quantity of carboxylic groups in the polygalacturonic structure.⁷ So, this observation is in agreement with the IR results that revealed a higher de-esterified (de-methoxylated) fraction of carboxylic groups in the solid G-HA.

Morphological analysis

In morphological terms, two phases were observed in both guava solids (Figure 2): a lamellar surface structure (with flake form) and a

spherical arrangement. These two morphologies indicate a possible separation of phases in the solids as a result of the dehydration processes. As discussed from IR and XRD results, a possible demethoxylation of galacturonic acid in pectin would generate more interactions and association of carboxylic groups with other molecules. So, these interactions between the structural components on the surface of these

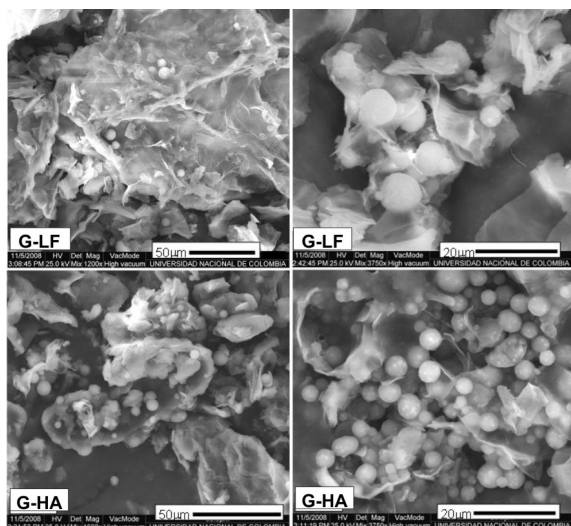


Figure 2. SEM images of powders obtained by dehydration of guava: (a) G-LF, (b) G-HA

materials can be understood as a surface tension increase, which lead to the separation and formation of spherical particles from the largely containing-pectin layered surface. However, a higher quantity of spherical particles (having a smaller size) was observed by SEM analysis for sample G-HA, according to a higher demethoxylation of pectin due to the hot-air drying process. Furthermore, an additional limitation to the formation of spherical particles in the G-LF solid can be expected, taking into account that the molecular motion of polysaccharides associated with bound-water is restricted by ice grown in the intermolecular spaces due to the freezing of free water by the lyophilisation process.²⁰

Sensory analysis

Sensory analyses of two solids were performed by a trained panel in comparison with guava puree, and evidenced that the aroma profile of the two solids were similar between them and different of the fresh fruit. The fruity, sweet, and woody odour notes were predominant in the solids, with the woody note being more intense in G-HA solid. The grassy, sulphury and fresh notes from the fruit were lost during the drying processes. In spite of this fact, a sensory assay performed by a non-trained panel (consumers) using a non-structured scale revealed that the flavour of the solids was pleasant and resembled guava jelly.

The volatiles composition of headspace of solids was analyzed by HS-SPME/GCMS showing a similar profile as was expected from sensory analyses (Table 2).

Table 2. Volatile compounds detected by HS-SPME/GC-MS in dehydrated guava powders and odour-note description

Compound	Retention Index		Amount		Odour description ^a	ID ^b
	FFAP	DB-5	G-HA	G-LF		
Limonene	1188	1032	+++	+++	Sweet, citric	A
β-Ocimene	1229	-	+	+	-	B
Hexyl acetate	1270	1016	+	-	-	A
3-Hydroxy-2-butanone	1284	-	+	+	Fresh	A
(Z)-3-Hexenyl acetate	1317	1010	+	+	-	A
6-Methyl-5-hepten-2-one	1339	989	+	++	-	B
Hexanol	1357	< 900	+	+	-	A
Allocimene	1369	-	+	-	-	B
(Z)-3-Hexenol	1389	< 900	+	+	-	A
Acetic acid	1465	-	+	+	acid	A
α-Copaene	1489	1380	+	++	guava peel-like, earthy	B
Linalool	1548	-	+	-	ripe guava-like	A
Butane-2,3-diol	1562	-	-	+	-	A
Farnesol	1578	-	+	+	-	B
Bergamotene	1596	-	++	++	-	B
trans-Caryophyllene	1602	1431	++++	++++	-	A
Junipene	1608	-	+	+	-	C
α-Humulene	1673	1460	++	++	sweet, creamy	B
β-Gurjunene	1681	-	+	+	sweet, guava jelly	C
β-Farnesene	1685	1442	+	+	-	B
Valencene (isomer I)	1715	-	+	+	fermented	C
Valencene (isomer II)	1723	-	++	++	-	C
β-Selinene	1729	1487	++	++	-	B
β-Bisabolene	1738	-	+	+	-	C
δ-Cadinene	1765	1509	+	+	-	C
1-Calamenene	1840	1525	+	+	creamy, sweet	C
Patchulane	1978	-	+	+	floral	C
β-Nerolidol	2054	-	+	+	-	B
Cinnamyl acetate	2142	-	+	+	-	A

^aOdour description as perceived at the sniffing port during GC-O; ^bIdentification: A, mass spectrum and Kovats index agreed with those of standards;¹⁶ B, mass spectrum and Kovats index agreed with literature data;²⁵ C, tentatively identified by only the mass spectrum. Relative abundance: + < 5%, ++ 5-15%, +++ 15-25%, ++++ > 25 %, - = not detected.

The major volatiles were limonene, bergamotene, *trans*-caryophyllene, α -humulene, and valencene. By comparison of these results with those of fresh fruit,⁴ it is clear that key aroma compounds of guava, such as (*Z*)-3-hexenal, hexanal, 3-sulfanyl-1-hexanol, 3-sulfanylhexyl acetate, 4-hydroxy-2,5-dimethyl-3(*2H*)-furanone, and ethyl butanoate were lost during drying processes. This is in agreement with the changes in the odour notes evidenced from the sensory panel of solids, because the grassy, sweet and sulphury notes due to C₆-aldehydes, furanic, and sulphur compounds, respectively, were diminished. However, the overall flavour of these solids was pleasant for consumers and somehow related to processed-guava characteristics.¹⁸ From GC-O (gas chromatography coupled to olfactometry) analyses, it was suggested that some sesquiterpenes are responsible for these guava characteristics and could be associated to the remarkable woody odour note detected through sensory analysis. These compounds were generated by drying processes because most of them were not detected as components in the aroma extracts of fresh fruit. It is important to point out that the flavour of guava remains in the powders after drying processes and although it is different to that exhibited for fresh fruit, no formation of off-flavours were detected, which is good an advantage for development of food products.

CONCLUSIONS

Two dehydration methods to obtain guava powders were compared, showing the potentiality of both techniques to integral use of guava since the bulk of whole fruit was processed. Physico-chemical analysis, thermal and structural study and morphology observations showed small differences between hot-air dried solid (G-HA) and lyophilised (G-LF) powder. TGA and DSC analyses revealed a higher thermal stability of G-LF sample and a thermal behaviour markedly determined by pectin containing in both samples. XRD analysis showed a semi-crystalline structure of the solids, indicating a slightly more crystalline structure in G-HA powder, related with a possible higher demethoxylation of this solid which was evidenced by IR spectra and reflected in the morphology results. On the other hand, sensory analysis showed similar characteristics between the two dehydrated powders and several differences between them and the fresh fruit, but a pleasant and resembled guava-jelly flavour was detected for both solids. So, the solids were satisfactorily accepted by consumers as food value-added products.

SUPPLEMENTARY MATERIAL

Available at <http://quimicanova.s bq.org.br>, in PDF file, with free access. The IR spectra and X-ray diffractograms, as well as the aroma profile analyses of solids (G-HA and G-LF) are showed.

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