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Chemical characteristics and colorimetric properties of non-centrifugal cane sugar ("panela") obtained via different processing technologies

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Abbreviated running header: Chemical characterization of non-centrifugal cane sugar under different processing

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Abstract

Non-centrifugal cane sugar (NCS) samples obtained by traditional moulding and granulation, and also via a novel spray-drying powdering process without additives, were assessed to characterise their sugar and phenolic profiles, flavonoid content, as well as colour parameters. As expected, sucrose was the predominant sugar (91.9 – 95.5%), followed by glucose (2.9-4.6%), and fructose (1.6-3.7%). Total phenolic content was between 0.4-0.6% and total flavonoid content into the range of 0.2-0.4%. Six phenolic acids were found in all NCS samples: protocatechuic acid (0.36-0.94 µg/100 g), vanillic acid (0.70-1.45 µg/100 g), chlorogenic acid (2.08-3.82 µg/100 g), syringic acid (1.08-2.80 µg/100 g), *p*-coumaric acid (0.69-1.35 µg/100 g), and ferulic acid (0.50-0.95 µg/100 g). The thermal treatment under high temperatures required in the production of granulated products was related with darker colours and changes in phenol and flavonoid contents. In contrast, spray drying generates clearer products, but with slightly less phenol and flavonoid contents.

Keywords: Panela; jaggery; spray drying; phenolic compounds; phenolic acids, colour parameters.

Chemical compounds studied in this article: Protocatechuic acid (PubChem CID: 528594); vanillic acid (PubChem CID: 8468); chlorogenic acid (PubChem CID: 1794427); syringic acid (PubChem CID: 10742); *p*-coumaric acid (PubChem CID: 637542); ferulic acid (PubChem CID: 445858); sucrose (PubChem CID: 5988); glucose (PubChem CID: 5793); fructose (PubChem CID: 2723872).

45

1. Introduction

Panela, jaggery, gur, muscovado, or piloncillo is a natural sweetener obtained by concentration of sugar cane juices (*Saccharum officinarum*), technically known as non-centrifugal cane sugar (NCS). According to the Food and Agriculture Organization of the United Nations (FAO), the NCS is more than a sweetener (Jaffé, 2015), and it can be considered as a whole food product of high nutritional value. Additionally, NCS is an advantageous substitute of white sugar, because its minimal chemical processing enables to retain a variety of minerals (Fe, Ca, P, K, Mg, Cu, Mn, Na, and Zn), vitamins (A, C, and D), carbohydrates (sucrose, glucose, and fructose), antioxidant substances, and other phytochemicals from sugar cane (Duarte-Almeida, Salatino, Genovese & Lajolo, 2011; Harish Nayaka, Sathisha, Manohar, Chandrashekar & Dharmesh, 2009; Feng, Luo, Zhang, Zhong, & Lu, 2014; Jaffé, 2015; Seguí, Calabuig-Jiménez, Betoret & Fito, 2015; Arif, Batool, Nazir, Khan, & Khalid 2019). The presence of these compounds gives NCS some functional properties, such as the ability to reduce the cells degenerative processes and to mitigate problems associated to cancer and cardiovascular diseases (Harish Nayaka, Sathisha, Manohar, Chandrashekar & Dharmesh, 2009). Also, NCS has been used to reduce the negative effects of some infections and diseases such as bronchitis, cough, anaemia, and jaundice (Seguí, Calabuig-Jiménez, Betoret & Fito, 2015).

Nowadays, world NCS production is around 12000 kt/yr. and the largest producers are India (60%), Colombia (14.9%), Pakistan (5%), China (3.9%), and Brazil (3.7%) (Minagricultura, 2018). Nevertheless, it is consumed worldwide as sweetener or as ingredient in different food products and beverages (Lamdande, Khabeer, Kulathooran, &

Dasappa, 2018; Mohan & Agrawal, 2020). Particularly in Colombia, the production of NCS is a traditional artisan activity with a strong economic and social significance. This productive sector generates around 1.2 million jobs, mainly in rural areas, and the country has the highest world per capita consumption (Minagricultura, 2018).

The traditional production of NCS is mostly an artisan activity, and it begins with the extraction of sugar-rich juices from sugar cane in a compression mill. Then the juices are heated up to carry out the clarification process by adding a natural mucilage as coagulant agent. Afterwards, the clarified solution is subjected to alkalisation with lime to adjust pH nearly to 6.0, in order to avoid sucrose hydrolysis. Subsequently, juices are concentrated by an open evaporation process, generating intermediate sugar solutions generally called sugar cane syrups (solutions from 50 to 80 °Brix). The evaporation process ends when the solution is concentrated to 90–92 °Brix, or even at higher concentrations when the product is intended for granulation. Finally, the concentrated liquid is stirred to induce crystals formation (Verma, Iyer, Shah, & Mahajani, 2021) and cooled down in moulds to obtain NCS blocks or bricks (Alarcón, Orjuela, Narváez & Camacho, 2020).

The solid bricks are the most common commercial forms of NCS because their regular shapes are convenient for piling, storage, packaging, and transportation, as well as to minimize water sorption. However, NCS bricks lack convenience for consumers because there is need for product grinding before use, and it exhibits a low dissolution rate at room temperature (18 °C). For these reasons, producers have developed granulated products with the aim to enhance the dissolution rate, and making them suitable as instantaneous

sweeteners (Verma, Shah, & Mahajani, 2019). Current granulation processes involve mechanical milling of the bricks and subsequent sieving. Alternatively, there is a technique that involves the addition of sodium bicarbonate to the concentrated syrup (above 92 °Brix). The bicarbonate thermally decomposes above 80 ºC releasing CO_2 and causing the product to rise. At the same time, the hot mass is manually shaken with a scrapper until it expands into a sponge-like form. After cooling the porous matrix bursts out generating the granulated solids (Prada Forero, García Bernal & Chaves Guerrero, 2015; Alarcón, Orjuela, Narváez, & Camacho, 2020). Any of these granulation methods increases the processing time and energy consumption, modifies the sensory properties of the final product, and reduces the production capacity in the NCS facilities (Hussain, Islam, Mohammad, Perveen, & Khan, 2011; Solís et al., 2019; Vera, García, Otalvaro, & Mendieta, 2019; Verma, Shah, & Mahajani, 2019). A pictorial image of commercial products is presented in Figure 1S in the supplementary material.

Recently, some alternative processes have been evaluated to produce powdered NCS with high dissolution rates via spray drying of juices and syrups (Cortes, Ciro, Rodríguez, & Largo, 2012; Khuenpet, Charoenjarasrerk, Jaijit, Arayapoonpong, & Jittanit, 2016; Palacios, Orjuela, Narváez, & Osorio, 2016). Spray drying is widely used in the food industry; however, its adaptation to produce powdered NCS is still a major challenge. The high sugar-content (i.e. sucrose, glucose, and fructose) in NCS's syrups can cause troubles during the drying process (e.g. filaments formation, caking/sticking of solids) due to the low glass transition temperatures of the dried materials as a result of their hygroscopic nature (Truong, Bhandari & Howes, 2005). Also, because this powdering method involves contact with air at high temperatures, chemical, and sensory properties might be affected by

potential oxidation, thermal degradation, or volatile losses. Nevertheless, if spray drying challenges can be overcome, this technique could boost NCS production and demand mainly in the food industry.

In this regard, the aim of this work was to develop a chemical characterisation and a comparative assessment of the NCS samples obtained by using different technologies, namely traditional moulding in blocks, granulation, and powdering via spray-drying. As part of their characterisation, the sugar profile, total phenolic and flavonoid content, as well as the profile of phenolic compounds of different NCS samples were determined. Thereafter, these properties were correlated with colour measurements. Furthermore, granulated and powdered NCS were analysed into the different particle sizes to verify composition homogeneity and to rule out the potential chemical stratification. The hypothesis behind this study was that the spray drying process would produce an equivalent NCS product (i.e. chemical compounds and colour) to those currently commercialized in the market (i.e. bricks and granules) but with improved preservation of nutritional compounds.

2. Materials and methods

2.1. Samples collection and/or preparation

Four types of NCS samples were studied, all of them obtained from producers in the state of Cundinamarca, Colombia. The traditional NCS bricks (NCS-B) were purchased at local store and corresponded to the brand Megapanela® (Pane-rapid Ltd.). The granulated

NCS (NCS-G) and syrups of 50 °Brix (S50) and 70 °Brix (S70) were acquired in a manufacturing facility located at SENA (Tobia, Cundinamarca, Colombia). Powdered NCS (NCS-P50 and NCS-P70) was obtained by spray drying of collected syrups (i.e. S50 and S70) without the use of additives. These syrups were collected from the same batch used for granulated NCS production at the SENA facilities, and they were stored in autoclaved and sterilized glass bottles (schott®), and maintained under refrigerated conditions prior use or characterization (max. 1 month). For that purpose, a LabPlant SD-06 (Huddersfield, UK) laboratory-scale (1110 mm x 825 mm x 600 mm main spray chamber) spray dryer was used. Approximately, 1 L of the feed-mixture (i.e. 50 or 70 °Brix) was kept at 40 ºC, and spray dried with a hot air flow of 3 kg/h, by using a 0.5 mm diameter nozzle. The feed flow was 0.597 kg/h, and the inlet and outlet air temperatures were 140 ± 2 ºC, and 65 ± 2 ºC, respectively (Palacios, Orjuela, Narváez & Osorio, 2016). The NCS-P was collected from the cyclone. The final solid (NCS-B, NCS-G, NCS-P50 and NCS-P70) and liquid (S50 and S70) samples were collected and stored in hermetic containers for further characterisation. A picture of the assessed samples is presented in figure 2S in the supplementary material.

In order to characterise the solid products (i.e. granules and powder), a series of sieves with different mesh sizes were used to separate the solid samples (NCS-G and NCS-P) into different fractions, as described in Table 1. The sieves were operated by mechanical vibration (Retsch AS200, Germany) at amplitude of 70 for 30 min. The weight of samples retained on each sieve was recorded and expressed as fraction (%) of the original sample.

2.2. Chemicals

Pure reference standards of sucrose, glucose, fructose, gallic acid, catechin, protocatechuic acid, vanillic acid, chlorogenic acid, syringic acid, *p*-coumaric acid, and ferulic acid were acquired from Sigma-Aldrich (Madrid, Spain). Acetonitrile, diethyl ether, methanol, and formic acid, all HPLC grade, as well as, sodium carbonate, sodium hydroxide, sodium nitrite, $AlCl_3$, and hydrochloric acid were purchased to Fisher Scientific (Loughborough, UK). Folin-Ciocalteu´s reagent was acquired from Scharlab (Sentmenat, Spain).

2.3. Physicochemical characterisation of NCS samples

2.3.1. Moisture content in solids

Moisture content was gravimetrically measured following a modification of the AOAC 925.45 standard method (AOAC, 2016a). A sample in-between 5 to 10 g was put into a Petri dish 5 cm in diameter and introduced into a forced convection air oven (LabTech Daihan, Korea) at 85 ± 0.1 °C for 24 h. In the case of the syrups, a 2 g sample was placed in the petri dish on top of 4 g of quartz sand (Merk, Darmstadt). Afterwards the samples were cooled down within a glass desiccator containing silica gel. Moisture was calculated in a wet basis by difference with respect to the initial weight. The moisture content corresponds to the average value of duplicate measurements of each sample.

2.3.2. Sugar analysis

Samples processing for sugar analysis was adapted from Asikin et al., 2014. Approximately 0.5 g of each sample was dissolved in a 10 ml flask with acetonitrile-water (15:85). One millilitre of each dissolution was filtered through a hydrophilic PVDF Millex-

189 HV 0.45 µm syringe filter (Millipore, Bedford, MA, USA) and injected into the HPLC
190 system. This unit corresponds to an Agilent 1100 chromatograph (Agilent Technologies,
191 Palo Alto, CA, USA) equipped with a Differential Refractive Index detector (RID). The
192 separation was performed on a Zorbax carbohydrate Analysis column (Agilent
193 Technologies, Palo Alto, CA, USA; 5 µm, 250 x 4.6 mm *i.d.*). The column was maintained
194 at 30 °C, and the mobile phase was acetonitrile-water (75:25, *v/v*) at a flow rate of 1.4
195 ml/min. The injection volume of the samples was 20 µl. Stock solutions of sucrose (50
196 mg/ml), glucose (6 mg/ml), and fructose (6 mg/ml) standards were used for the calibration.
197 The compounds were identified by comparison with the retention times of the
198 corresponding standards (sucrose 7.8 min, Glucose 9.5 min, Fructose 11.5 min). The
199 quantification was carried out by external calibration from the areas of the chromatographic
200 peaks obtained by RID, and the results were expressed as g of carbohydrate / 100 g of
201 sample in dry basis. The analyses were performed by quadruplicate.
202
203 *2.3.3. Total phenolic content*
204 The total phenolic content was determined using the Folin-Ciocalteu assay with a
205 minor modification (Kuś et al., 2014). For this purpose, 0.5 g of each sample was diluted in
206 10 ml of distilled water, then 100 µl of each dilution was separately added to 0.5 ml of
207 Folin-Ciocalteu´s reagent. After 5 min, 3 ml of 20 % Na_2CO_3 (w/v) was added, the mixture
208 was vigorously shaken, and then diluted with water to a final volume of 10 ml. After 2
209 hours of incubation at 18 °C, the absorbance was read at λ 725 nm in 10 mm quartz cuvette
210 with an Agilent Technologies Cary 8454 UV-vis (Santa Clara, CA, USA)
211 spectrophotometer, against a blank. The total phenolic content was calculated using a

calibration curve with gallic acid standard solutions (10 – 500 mg/l) and expressed as mg of gallic acid equivalent (GAE) /g of sample. The procedure was made by quadruplicate and the reported value corresponds to the mean ± standard deviation (SD).

2.3.4. Total flavonoid content

The flavonoid content was determined using the methodology described by Habib et al., (2014). The dilutions were prepared as described in section 2.3.3, as follows: 200 µl of each dilution was added to a 5 ml volumetric flask containing 2 ml of distilled water. Then, 0.15 ml of $NaNO_2$ (5 g/100 ml) was added to the flask. After 5 min, 0.15 ml of $AlCl_3$ (10 g/100 ml) was added. Then, after 6 min, 1 ml of NaOH 1M was added to the mixture under agitation, and diluted with H_2O to final volume of 5 ml. The absorbance of the mixture was measured at λ 510 nm versus prepared water blank in an Agilent Technologies Cary 8454 UV-vis (Santa Clara, CA, USA) spectrophotometer. Total flavonoids content was calculated using a calibration curve with prepared catechin standard solutions (20 – 200 mg/L) and expressed as mg of catechin equivalent (CE) per gram of sample. The procedure was made by quadruplicate and the reported value corresponds to the average of measurements.

2.3.5. Extraction of phenolic compounds

To quantitate the phenolic content, 2 g of each sample was separately diluted with deionized water in a 10 ml flask and acidified to pH 2 with HCl 37% (Harish Nayaka et al. 2009). After that, 500 µL of each dilution was mixed with 500 µL of HPLC-grade diethyl ether in an Eppendorf tube. The mixture was shaken in a vortex, sonicated for 3 min, and

centrifuged for 5 min at 1400 rpm (i.e. 175 x g). The supernatant was collected, and the remaining residue was subjected to five additional extractions. The supernatants were combined and dried under vacuum during 10 min at 30°C to obtain the extract. Each residue was dissolved in 100 µL of HPLC-grade methanol and filtered through a hydrophilic PVDF Millex-HV 0.45 µm syringe filter (Millipore, Bedford, MA, USA). The obtained extracts were used for the determination of individual phenolic compounds by UHPLC-DAD.

2.3.6. Analysis of phenolic compounds by UHPLC-PDA

Phenolic compound characterization was carried out according to a previously reported method (Jara-Palacios et al., 2014). Analysis were carried out in an Agilent 1260 chromatograph (Agilent Technologies, Palo Alto, CA, USA) equipped with a photodiode-array detector (PDA), which was set to scan from λ 200 to 770 nm. A C_{18} Eclipse Plus 120 (1.8µm, 50 x 2.5mm) column was used. The mobile phase was 0.1% formic acid in deionized water (solvent A), and acetonitrile (solvent B), with the following gradient: 0-5 min, 5% B linear; 5–20 min 50% B linear; 20–25 min, 100% A linear, washing and re-equilibration. The flowrate was 0.8 ml/min, and the temperature of the column was set at 25 °C.

The phenolic compounds were identified by their retention time, and UV-Vis spectroscopic characteristics by comparison with those of standards. The quantitation was carried out by external calibration from the areas of the chromatographic peaks obtained by PDA detection at λ 280 nm. The corresponding calibration curves were made up of the

following standards: protocatechuic acid, vanillic acid, chlorogenic acid, syringic acid, *p*-coumaric acid, and ferulic acid. Each sample extracted by quadruplicate was analysed with an injection volume of 0.2 µl to quantitate each compound, and the results were expressed as µg of phenolic compound/g sample in dry basis.

2.3.7. Colour measurement

Colour was evaluated by tristimulus colorimetry based on the reflectance spectra. Approximately, 0.5 g of sample was diluted with water in a 10 ml flask. The sample was homogenised, and the absorbance was measured into the visible region (λ = 380-770 nm), in an Agilent Technologies Cary 8454 UV-vis (Santa Clara, CA, USA) spectrophotometer. CIE standard illuminant D_{65} and 10° standard observer were used as references. The following CIELAB colour attributes were assessed using the CromaLab® software (Heredia et al., 2004): L^* (Lightness), a^* and b^* (colour coordinates), h_{ab} (hue angle) and C^*_{ab} (chroma).

2.4. Statistical analysis

One-way analysis of variance (ANOVA) was applied to determine whether significant differences ($p < 0.05$) exist among properties of the NCS obtained by the different processes. In addition, simple correlations between phenolic content and colorimetric parameters were studied. In all cases, statistically significant level was considered at $p < 0.05$. Stepwise Discriminant Analysis (SDA) was applied on experimental standardized data to identify the variables responsible for the differences between NCS samples. These statistical analyses were performed by using the Statsoft Statistica® V 8.0 software.

3. Results and discussion

To elucidate the origin of the potential differences among the assessed products, it is important to describe some details of the operation in the manufacture of each type of NCS product. The bricks (corresponding to NCS-B samples) are traditionally produced in open pan evaporation systems, in which the sugarcane juice is pool boiled in a series of stainless-steel pans that are heated by mean of combustion gases (La Madrid, Marcelo, Mendoza Orbegoso, & Saavedra, 2016; Vera, García, Otalvaro, & Mendieta, 2019). The liquid is manually moved and poured from one pan to the next at uneven time intervals that depends on the operator's experience. During the residence time, juices are concentrated to obtain syrups, and they are in direct contact with surfaces that can be at temperatures up to 500-600 °C. Under this condition, the liquid rapidly boils, and even under a vigorous stirring, a crust of caramel-like materials and carbonized products can be formed on the hot surface. These degradation products get re-dissolved or re-suspended as a result of the operator's manual agitation and surface scraping with a metal blade. To avoid the excessive foaming and overflow caused by boiling the concentrated liquid, a small amount of vegetable oil is added in the final stages as a de-foaming agent (García, Albarracin, Toscano, Santana, & Insuasty, 2007).

During this open evaporation process, the feed sugarcane juice with a solids content ranging from 16 to 20 °Brix concentrates up to ~ 92 °Brix. In the last pan, the bubble point reaches around 120 °C, and the syrup is "in point" for final stirring (that enhances crystallization, Verma, Iyer, Shah & Mahajani, 2021) and molding (García, Albarracin,

306 Toscano, Santana, & Insuasty, 2007; Velásquez, Espitia, Escobar & Mendieta, 2019). As
307 the concentration process is carried out in direct contact with air, oxidation products are
308 formed because of the long processing time and the high operating temperatures. Also, a
309 variety of volatile compounds present in the original sugarcane juice, or those formed
310 during reactions, can be steam distilled during the boiling operation. Finally, Maillard
311 products are formed by reaction of reducing sugars with primary amines present in the juice
312 or those coming from the mucilaginous material used for clarification. The final molded
313 product is wrapped and sealed with a thermoplastic film, and packed in cardboard boxes for
314 storage and distribution (Velásquez, Espitia, Mendieta, Escobar, & Rodríguez, 2019).
315
316 In comparison, when the product is granulated NCS (corresponding to NCS-G samples),
317 the syrups are boiled for longer times up to a higher solids content (i.e. > 95 °Brix), which
318 correspond to a higher bubble temperature (> 125 °C). The operation under these
319 conditions accelerates different chemical reactions. In general, a larger fraction of the
320 reducing sugars (i.e. glucose and fructose) can react to produce different side products
321 affecting the final sugars profile. Therefore, to reduce hydrolysis of sucrose to reducing
322 sugars, the operation is conducted under higher pH levels (> 6). In this case, a higher
323 amount of calcium hydroxide or calcium carbonate is added with respect to that used in
324 bricks manufacturing (Velásquez, Espitia, Mendieta, Escobar, & Rodríguez, 2019). After
325 the final hot syrup is removed from the last pan, it is subjected to aeration, cooling, manual
326 grinding and sieving. Large particles are subjected to mill grinding and sieving until
327 fulfilling the required particle size specifications. Typically, during the granulation steps
328 the particles can uptake some moisture from the environment, and a final drying could be
329 required. This last step is carried out in heated and agitated drums, or in convective tray

ovens. Finally, the product is packed in sacks or plastic bags of different sizes (García, Albarracin, Toscano, Santana, & Insuasty, 2007).

The other assessed product, spray dried powder (NCS-P), was obtained from intermediate syrups of 50 to 70 °Brix collected in the open evaporation process process intended for granulation (i.e. pH > 6). The corresponding bubble temperatures of these syrups are below 105 °C. During the spray drying, the liquid is fed at low temperature, and it is put in contact with air at 140 °C at the atomizer inlet nozzle. The residence time in this unit is very short (< 2 min), and the air rapidly cools down (~ 70-80 °C) because of water evaporation (Palacios, Orjuela, Narváez, & Osorio, 2016). As expected and described in Table 1, particle sizes of the spray dried powders are smaller than those obtained in the granulation process. While 70% of granulated products corresponds to particles larger than 0.5 mm, in the case of spray dried products nearly 58 to 64 % corresponded to particles smaller than 0.6 mm. In particular, it was observed that powders obtained from syrups with a lower solid content (P50) exhibited a larger fraction (> 40%) of particles with sizes below 0.3 mm. The higher content of fines was related with the more effective atomization in the nozzle when operating with a lower viscosity liquid that enabled the formation of smaller droplets. The proportion of larger particles (> 0.6 mm) after spray drying of assessed syrups was similar (i.e. 36 - 42 %), and it might be related with the aggregation of the particles during the drying process or in the collecting cyclone. Because of the light scattering effect, the spray dried particles have a clearer visual aspect than those in the granulated product, and both have a lighter apparent color to that of the syrups and bricks.

3.1. Physicochemical characterisation

354 *3.1.1. Moisture in samples*

355 According to the measurements, the moisture content (wet basis) of the bricks (NCS-
356 B) was 6.5 % (only one measurement), the granulated product (NCS-G) was 2.74 ± 0.11 %
357 wt., and the spray dried solids, i.e. NCS-P50 and NCS-P70, were 2.31 ± 0.03 % wt. and
358 1.69 ± 0.06 % wt., respectively. These values are consistent with required specifications for
359 brick and granulated NCS (ICONTEC, 2009).

360

361 *3.1.2. Sugars profile*

362 The sugars profile in the assessed NCS samples are reported in Table 2. As expected,
363 sucrose was the predominant sugar in all samples with a content greater than 91.9% (dry
364 basis). Glucose and fructose, exhibited a lower content corresponding to >2.9% and >1.6%,
365 respectively. This sugar profile is consistent with NCS standards (ICONTEC, 2009) and
366 agrees with those reported in previous studies on the characterisation of NCS samples
367 (Guerra & Mujica, 2010; Segui et al., 2015). The content of reducing sugars can help
368 differentiating NCS from brown sugar, which is a similar natural sweetener obtained by
369 crystallization of concentrated sugarcane molasses, and that has a much lower content of
370 fructose (0.75%), and glucose (0.46%) (Asikin et al., 2014).

371

372 Regarding the processing influence on the sugars profile on the different types of
373 NCS, there were slight differences between NCS-G and NCS-P products in comparison to
374 the brick (NCS-B). Particularly there is a lower content of sucrose in the NCS-B sample,
375 and a higher concentration of reducing sugars. This was expected as the higher pH used in
376 the processing of syrups for granulated products helps inhibiting hydrolysis. Interestingly,
377 there are no differences among syrups obtained at intermediate stages of the evaporation

process (i.e. S50 and S70). This agrees with a previous report (Alarcón, Orjuela, Narváez, & Camacho, 2020) and indicates that, during most part of the evaporation process, the chemical integrity of saccharides in the syrups is maintained, and that only the solids content changes due to water removal. The lower viscosity and the dissolving action of the boiling syrups below 70 °Brix prevent solids from sticking and caramelising on the pan's hot surface.

In average, when comparing the total reducing sugars content in NCS-G, NCS-P50 and NCS-P70 samples, there is a slightly higher content in the granulated product. This is related with a higher degree of hydrolysis as a results of long residence times (i.e. typically 10-20 min more compared to the syrup point for brick product) and higher temperatures during NCS-G production. Interestingly, the sugars profile is almost invariant when comparing the spray dried powders and their corresponding syrups of origin (S50 and S70), indicating that the drying conditions do not affect the sugars profile. As the reducing sugars content in the syrups and spray dried products is lower with respect to bricks, this can represent a commercial and operational advantage. A high content of glucose and fructose in NCS is related with a higher hygroscopicity of the product, which affects the texture, and reduces its stability and shelf life (Guerra & Mujica, 2010; Verma, Shah, & Mahajani, 2019). Nonetheless, while the reduced hygroscopicity of powdered and granulated material could affect their dissolving rates, the smaller particles sized can help overcoming such difficulties.

According to a generalized claim among some granulated NCS producers, the difference in particle sizes during granulation is driven by the uneven cluster crystallization

around local high concentrations of sucrose. Thus, they expect to have larger concentrations of sucrose in the larger particles during granulated NCS production. As observed in Table 2, the sugar profile is almost the same among the different fractions of NCS with different particle sizes. This was expected as the mother syrup before the granulation or the spray drying processes is homogeneous, and the solidification process is fast enough to avoid any internal segregation within the solid phase. The claimed behaviour is also disproved when verifying that the content of phenolic and flavonoid compounds among particles of different sizes is almost the same.

3.1.3. Phenolic and flavonoid compounds

Regarding the total phenolic content in the samples (Table 2), it was found that syrups and granulated NCS samples exhibited the highest phenolic contents (>5.6 mg GAE/ g for syrups, and >5.1 mg GAE/ g for NCS-G). Comparatively, the content in spray dried product was around 20% lower. This result agrees with those published by Gómez-Narváez et al. (2019), who compared NCS samples from Colombia, Peru, Italy, and Spain. Also, it is in concordance with the results obtained from different sugarcane sweeteners from Pakistan (Iqbal et al., 2017). In addition, total phenolic content (TPC) calculated in wet base extracts varies over the range of 2.4 and 5.2 mg GAE/g of product. These values are slightly higher than those reported for NCS samples from Spain (1.0 – 2.6 mg GAE/g product, Segui et al., 2015) and India (3.8 mg GAE/g of product, Harish Nayaka et al. 2009). The observed differences are attributed to the cane variety and the agro-climatic conditions during the sugar cane crop. The reduction of the phenolic content of the samples NCS-P50 and NCS-P70 with respect to the original syrups is related with losses during the drying process (Rothwell et al., 2015), which are most probably caused by air dragging and

426 oxidation at the drying chamber conditions. This indicates that the spray drying process
427 might have an impact on the nutritional value of the powdered NCS.
428
429 The total flavonoid content of NCS samples and syrups was also determined (Table 2).
430 The NCS-B sample presented the lowest value, but still being in the same order of
431 magnitude of the other samples. The observed differences are most probably related to the
432 variety of sugarcane as it was different from the other NCS samples. The higher content of
433 phenolic and flavonoids in NCS-G samples can be explained by the long exposure time at
434 high temperatures during this process. Several phenolic compounds have been reported in
435 NCS samples and syrups, such as, chlorogenic acid, ferulic acid, gallic acid, gentisic acid,
436 *p*-coumaric acid, *p*-hydroxy benzoic acid, protocatehuic acid, syringic acid, vanillic acid,
437 and coniferyl alcohol, among others. Additionally, flavonoids such as, apigenin, luteolin
438 and tricin, have also been found in NCS samples (Duarte-Almeida et al., 2011; Harish
439 Nayaka et al., 2009; Jaffé, 2015; Singh et al., 2015).
440
441 It is known that phenolic compounds and flavonoids are highly unstable during NCS
442 processing (Jaffé, 2015). Thermal decomposition or thermal reactions of those compounds
443 can produce new chemical species (Segui et al., 2015) or generate an increase in the
444 phenolic content (Payet et al., 2006). Thus, in a further stage, six phenolic acids were
445 quantified (Table 3) in all NCS samples and syrups, being the most abundant chlorogenic
446 acid and syringic acid, followed by *p*-coumaric acid, vanillic acid, ferulic acid, and
447 protocatechuic acids. The effect of thermal treatment during processing is more significant
448 for protocatechuic acid, vanillic acid, chlorogenic acid, and ferulic acid, after the
449 comparison of their amount in the S-50, S-70, NCS-G, NCS-P50 and NCS-P70 samples.

Interestingly, syringic acid amount is higher in NCS-G and in NCS-B samples than in the S-50 and S-70, likely because heating promotes the oxidative breaking of cinnamic acids present in sugar cane syrups (chlorogenic acid, ferulic acid, and *p*-coumaric acid) (Duarte-Almeida et al., 2011). This can also explain the slight differences between S50 and S70, considering that the latter is a syrup that has been subjected to higher temperature for longer times.

The NCS-G, and NCS-P50 and NCS-P70 samples had a slight reduction in the phenolic acids content in comparison with those of the syrups, with the exception of syringic acid that can be produced from cinnamic acids structurally related such as, ferulic acid or sinapic acid. The granulated material also exhibited a slightly higher content of phenolic acids than the spray dried product. Considering this and taking into account that the spray dried powder is obtained directly from the intermediate syrups that had a higher content of phenolic compounds, it is verified that the drying process can affect the content of phenolic acids. Despite the low residence time, this can be related to oxidation due to the high air temperatures (140 °C), or volatilization of some of the components during the spray drying process promoted by a process occurring in the small drops and not in a liquid bulk.

The processing influence on the nature and content of phenolic compounds can vary because some of them (e.g. chlorogenic acid) are substrates in enzymatic browning reactions (Payet et al., 2006), while other are more stable. These processes can occur not only during the open evaporation of the juices, but also in the granulation operations (either

in the milling or in the manual shaking method), and even in the spray drying (depending on the temperature conditions and residence time).

3.2. Colour measurement

The average of CIELAB colour space parameters of aqueous solutions of NCS samples obtained in the different processes are presented in Figure 1. NCS-G solutions have the lowest values of lightness among samples. Relating to *b** parameter, all values were positive. These values proportionally increased from the lowest to highest thermal NCS treatment (from syrups and powdered to block and granulated NCS samples). Furthermore, the data obtained for NCS-G and NCS-B samples are located in the first *a**, *b** quadrant, while NCS-S and NCS-P samples were spread out between both, first and second *a**, *b** quadrant, because of the variation of *a** value.

NCS colour is a quality feature that influences consumers in their purchase decision. They usually prefer light colours, but this property depends on many variables, such as, phenolic content, sugar cane variety and maturity, agroecological conditions, and processing conditions (heating type, temperature, and use of bleach agents, García et al., 2017). While the commercial NCS has colours between gold yellow and dark brown (Shrivastav et al., 2016), it is expected that the granulated and powdered products might have clearer colours due to light scattering. Nevertheless, after preparing hot beverages of the same solids concentration (~ 20 % wt. in boiling water) in a similar way to a previous work (García et al., 2017), the perceived colour was similar among all the different solutions. This is a valuable finding because it indicates that the novel spray dried product

497 can be used as ingredient in food and beverages applications, and it most certainly would
498 exhibit similar organoleptic behaviour (i.e. colour) than that of the traditional bricks and
499 granules. This was expected considering that generally, spray drying has minor impact on
500 colour-imparting compounds. It has been reported that low molecular weight phenolic
501 compounds extracted from the canes during the milling process are responsible for ~ 30%
502 of colour; while some chemicals produced during the open evaporation process through
503 degradation reactions (i.e. enzymatic and non-enzymatic) contribute to 70% of final colour
504 (Schlumbach, Pautov & Flöter, 2017). The oxidation of phenolic compounds to dark
505 polymers are enzymatic reactions, and caramelisation and Maillard reactions are non-
506 enzymatic reactions (Guerra & Mujica, 2010); both of them generate a complex matrix of
507 coloured compounds.
508
509 Statistical relationships between the CIELAB colour coordinates and the phenolic
510 content were made. The complete definition of the colour of any object by means of the
511 CIELAB coordinates requires a joint consideration among the scalar (L^*, a^*, b^*) and the
512 angular (L^*, h_{ab} and C^*_{ab}) values. Hence, a multiple regression study by means of general
513 linear model (GLM) was carried out to get a more meaningful evaluation of the correlation
514 existing between the colour of NCS samples and their phenolic content. For this purpose,
515 the content of each phenolic compounds was considered as dependent variable, and the sets
516 of L^*, a^*, b^* and L^*, h_{ab}, C^*_{ab} coordinates as predictor or independent variables. The
517 corresponding R^2 values are listed in Table 4. When the set L^*, a^*, b^* was considered, the
518 highest R^2 values were obtained for the contents of vanillic, syringic, protocatechuic, and
519 chlorogenic acids, as well as for total phenolic content. Similarly, when the set L^*, h_{ab}, C^*_{ab}
520 was analyzed, the same phenolic compounds presented the highest R^2 values, being the

protocatechuic acid the one that showed the highest correlation. These results mean that benzoic acids have more influence in the colour of NCS samples that hydroxy cinnamic acids (ferulic and *p*-coumaric acids).

Additionally, to determine which variables were the most appropriate for discriminating among samples, Stepwise Discriminant Analysis (SDA) were performed (Figure 2a). L^*, glucose and syringic acid content were found statistically significant ($p < 0.05$). Two classification functions were obtained, which yielded a good separation (100% correct classification) among samples. According to data presented in Figure 2b, the discriminant function 1 (root 1) was mainly related to lightness (L^*) (with positive sign) and glucose content (negative sign), whereas discriminant function 2 (root 2) was mainly linked to syringic acid content (negative sign). The sets of the points show a distribution pattern in which three well-defined groups can be distinguished.

Regarding the initial hypothesis, this study enabled to determine that most phenolic compounds and the colour are preserved during spray drying process. However, the use of syrups as raw material for powdered NCS has slight negative impacts in the content of flavonoids. This might represent a challenge in terms of the sensory profile for the spray dried NCS powder as it is intended to be used as substitute for traditional brick or granulated products. Further studies might involve the use of more concentrated syrups or even reconstituted solutions as feed for the spray drying. Also, the slight reduction of phenolic acids in the NCS-P with respect to the original syrups indicates that spray drying conditions must be optimized (e.g. using encapsulating agents, reducing temperatures).

4. Conclusion

Non-centrifugated cane sugar (NCS) samples obtained by different manufacturing process were characterized. Samples corresponded to the traditional bricks, granules, and novel spray dried powders without additives. According to the results, there were differences in the evaluated properties attributed to the different processing techniques. Most changes of sugars profile, flavonoid content, phenolic compounds nature and content, and colour parameters, were related to thermal and enzymatic degradation processes. It was confirmed that the difference in particle sizes is the granulated NCS is not related with differences in composition, which was a common believing among producers. Regarding phenolic compounds, it was found that when the amount of protocatehuic, vanillic and chlorogenic acids increases, a positive effect was found in L^*, and a^* parameters of the CIELAB space. Additionally, L^* a^*, and syringic acid were the variables that showed more influence into the differences observed among NCS samples. There are differences on colorimetric properties, sugar content, and phenolic compounds related to the NCS manufacturing treatment. As expected the thermal treatment under high temperatures required in the production of granulated products is related with darker colors and higher phenolic compounds and flavonoids content. The early removal of syrups for spray drying might has slight negative impacts in the content of flavonoids. This might represent a challenge in terms of the sensory profile for the spray dried powder as it is intended to be used as substitute for traditional block or granulated NCS. Nevertheless, all evaluated NCS samples have more health-promoting phytochemicals than traditional refined and brown sugars.

CRediT authorship contribution statement

Angela Liliana Alarcón was responsible for the laboratory investigation, writing of original draft preparation. **Laura M. Palacios** contributed with the laboratory investigation for collection of samples and obtaining powdered material. **Coralia Osorio** contributed with formal analysis, resources, original draft preparation, writing, review, and editing. **Paulo César Narváez** contributed in the conceptualization stage and in the definition of the experimental methodologies used in the collection and characterization of obtained materials, as well as the supervision of the research activities and writing the results. **Francisco J. Heredia** contributed in the conceptualization stage, in the definition of the experimental methodologies used during the colour analysis, supervision of physicochemical characterisation. **Alvaro Orjuela** contributed with formal analysis, resources, original draft preparation, writing, review, editing, project administration, and funding acquisition. **Dolores Hernanz** supervised the laboratory investigation on colour analysis and phenol compound characterisation, as well as writing, review and editing. All authors contributed to the final version of the manuscript and have read and agreed to the published version of the manuscript.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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747

748 **Figure captions**

749

750 **Fig. 1.** Projection of the colour points corresponding to each sample on the (*a* b**) diagram
751 of colour parameters. NCS-G (○), NCS-P (□), NCS-S (●), NCS-B (Δ).

752 **Fig. 2.** Stepwise Discriminant Analysis (SDA). Loading plot for colour parameters,
753 phenolic, and sugar content measured in NCS samples (NCS-G (○), NCS-P (□), NCS-S
754 (●), and discriminant power (*F*-value) of the variables selected and cooeficients of
755 discriminant functions

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767 **Figure 1.**

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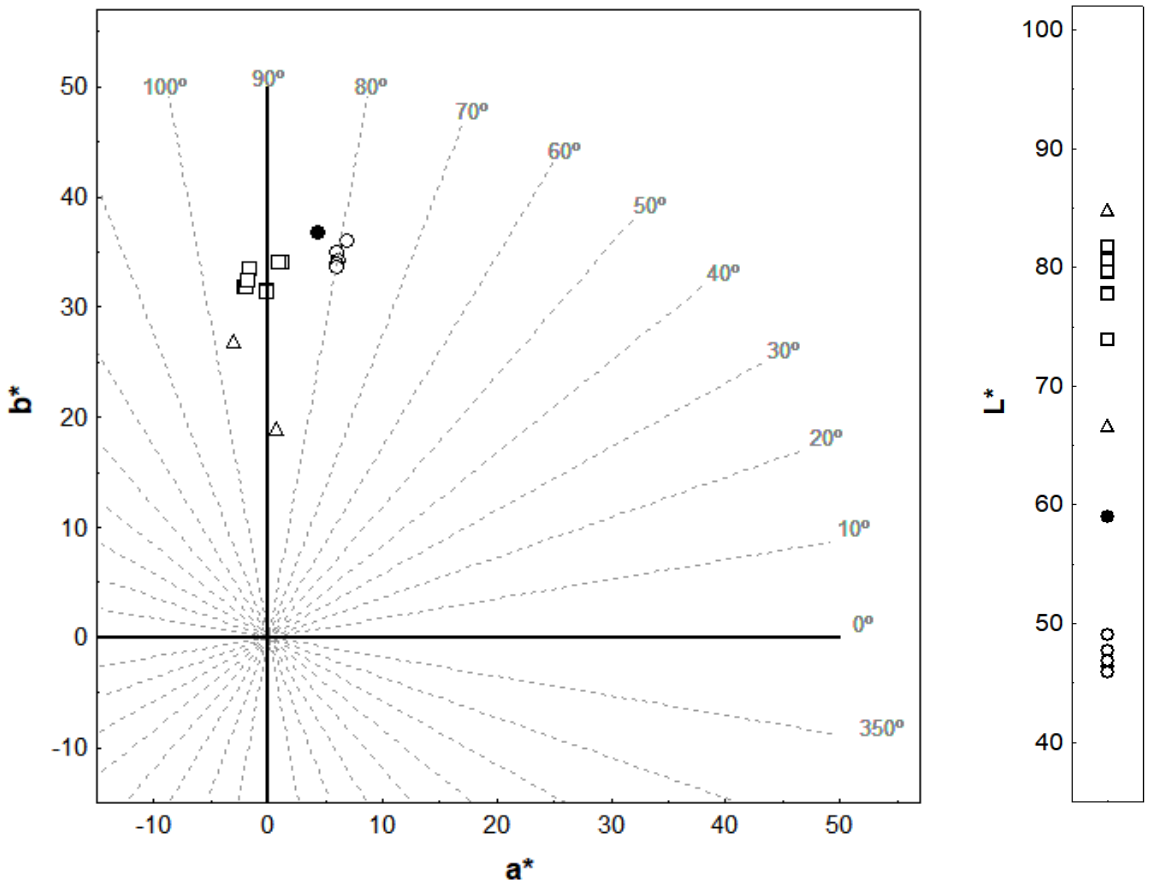
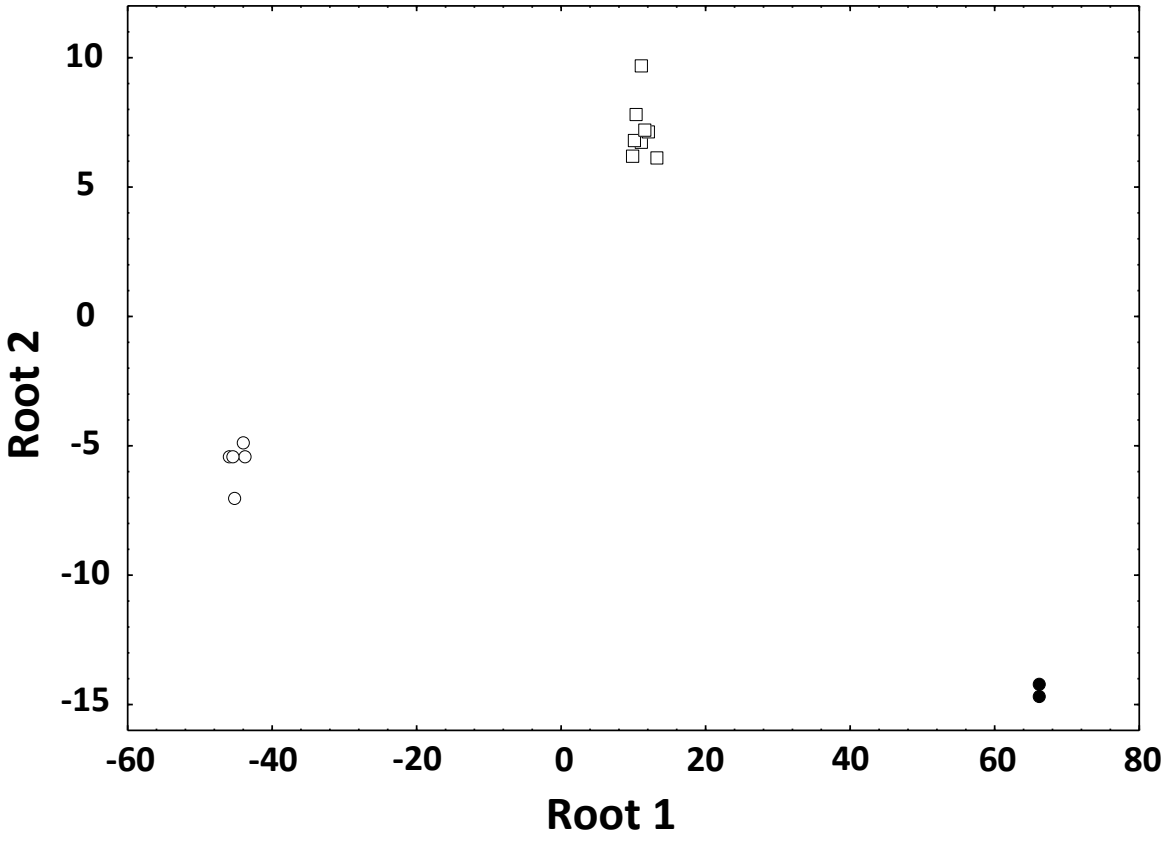


Figure 2.



Variable	F-value	Standardized coefficients	
		Root 1	Root 2
*L**	86.447	7.801	0.650
Glucose	18.431	-6.409	1.696
Syringic acid	12.035	2.312	-2.663
Eigenval		1650.912	82.637
Cum.Prop		0.952	1.000

Table 1

Description of NCS samples obtained by different processes

Sample	Nomenclature	Mesh size (mm)	Fraction %
NCS-G (granulated)	G1	> 1	23.64
	G2	0.8	4.95
	G3	0.6	5.92
	G4	0.5	35.60
	G5	<0.5	29.89
NCS - P50 (Powdered from 50 °Brix syrups)	P50-1	> 0.6	36.14
	P50-2	0.4	9.46
	P50-3	0.3	12.20
	P50-4	< 0.3	42.20
NCS - P70 (Powdered from 70°Brix syrups)	P70-1	> 0.6	41.99
	P70-2	0.4	26.89
	P70-3	0.3	23.43
	P70-4	< 0.3	7.69
NCS - B (Block)	B	NA	NA
NCS - S50 (50 °Brix syrup, AOAC 932.12)	S50	NA	NA
NCS - S70 (70 °Brix syrup, AOAC 932.12)	S70	NA	NA

NA. Not applicable

Table 2

Sugar, total phenol and flavonoid-contents (dry basis) in NCS samples obtained by different processing

Sample	Sucrose (g / 100g product)	Glucose (g / 100g product)	Fructose (g / 100g product)	Total phenolic content (mg GAE/g product)	Total flavonoid content (mg CE/g product)
NCS-G1	92.9 ± 2.9^{a}	4.6 ± 0.2^{a}	3.7 ± 0.2^{a}	5.8 ± 0.1^{a}	2.8 ± 0.1^{a}
NCS-G2	93.0 ± 2.9^{a}	4.3 ± 0.2^{a}	2.7 ± 0.1^{b}	5.9 ± 0.2^{a}	3.0 ± 0.3^{a}
NCS-G3	93.1 ± 2.2^{a}	4.2 ± 0.0^{a}	2.7 ± 0.2^{b}	5.9 ± 0.2^{a}	3.0 ± 0.1^{a}
NCS-G4	94.1 ± 1.5^{a}	3.6 ± 0.2^{b}	2.4 ± 0.0^{c}	5.4 ± 0.2^{b}	2.9 ± 0.1^{a}
NCS-G5	94.2 ± 2.9^{a}	3.6 ± 0.2^{b}	2.2 ± 0.1^{d}	$5.1 \pm 0.2^{b,e,g}$	3.8 ± 0.1^{b}
NCS-P50-1	94.5 ± 2.2^{a}	$3.4 \pm 0.3^{b,c}$	$2.1 \pm 0.2^{d,e}$	4.0 ± 0.2^{c}	$2.5 \pm 0.2^{a,c}$
NCS-P50-2	94.6 ± 5.0^{a}	3.5 ± 0.1^{b}	1.9 ± 0.1^{e}	4.4 ± 0.1^{d}	$2.7 \pm 0.2^{a,c}$
NCS-P50-3	$94.6 \pm 1.0^{a,b}$	$3.4 \pm 0.1^{b,c}$	2.0 ± 0.1^{d}	4.6 ± 0.1^{d}	2.9 ± 0.1^{a}
NCS-P50-4	$95.5 \pm 1.4^{a,c}$	2.9 ± 0.4^{c}	1.6 ± 0.1^{f}	4.5 ± 0.2^{d}	3.2 ± 0.2^{d}
NCS-P70-1	94.6 ± 4.3^{a}	$3.5 \pm 0.4^{b,c}$	$1.9 \pm 0.2^{d,e,f}$	4.9 ± 0.1^{e}	3.3 ± 0.1^{d}
NCS-P70-2	94.9 ± 2.9^{a}	$3.4 \pm 0.6^{b,c}$	$1.8 \pm 0.3^{d,e,f}$	4.7 ± 0.1^{f}	2.6 ± 0.2^{a}
NCS-P70-3	94.6 ± 3.2^{a}	$3.3 \pm 0.7^{b,c}$	$2.1 \pm 0.2^{d,e}$	4.6 ± 0.0^{f}	$2.3 \pm 0.2^{c,e}$
NCS-P70-4	95.0 ± 5.0^{a}	$2.9 \pm 0.9^{b,c}$	$2.1 \pm 0.1^{d,e}$	4.7 ± 0.2^{g}	2.4 ± 0.1^{c}
NCS-B	$91.9 \pm 1.5^{a,b,c}$	4.4 ± 0.2^{a}	3.7 ± 0.1^{a}	4.3 ± 0.1^{c}	2.0 ± 0.2^{e}
NCS-S50	94.4 ± 4.8^{a}	$3.5 \pm 0.5^{b,c}$	$2.1 \pm 0.2^{d,e}$	$5.7 \pm 0.1^{a,b}$	3.2 ± 0.1^{d}
NCS-S70	94.9 ± 1.8^{a}	$3.2 \pm 0.2^{b,c}$	$1.9 \pm 0.2^{d,e}$	$5.6 \pm 0.3^{a,b}$	$2.7 \pm 0.1^{a,c}$

NCS-G: granulated NCS; NCS-P: powdered NCS at different mesh from 50 °Brix (P50) and 70 °Brix (P70); NCS-B: NCS block sample; NCS-S50: syrup 50 °Brix, and NCS-S70: 70 °Brix. GAE = gallic acid equivalent; CE = catechin equivalent. $^{a–g}$ Means within a column, values with different superscript letters differ significantly ($p < 0.05$).

Table 3

Quantitation of phenolic compounds in NCS samples obtained by different processing methods

Sample	Protocatechuic acid	Vanillic acid	Chlorogenic acid	Syringic acid	*p*-Coumaric acid	Ferulic acid
	µg phenolic compound/g of product (dry basis)					
NCS-G1	$0.54 \pm 0.05^{a,b}$	$0.97 \pm 0.09^{a,b,e}$	2.68 ± 0.12^{a}	2.56 ± 0.04^{a}	1.31 ± 0.03^{a}	0.81 ± 0.01^{a}
NCS-G2	0.54 ± 0.03^{a}	$0.97 \pm 0.08^{a,b,e}$	2.62 ± 0.12^{a}	2.61 ± 0.05^{a}	1.30 ± 0.03^{a}	0.95 ± 0.10^{b}
NCS-G3	$0.49 \pm 0.03^{a,b,c}$	$0.93 \pm 0.02^{a,b,e}$	2.80 ± 0.06^{a}	2.57 ± 0.04^{a}	1.35 ± 0.01^{a}	0.76 ± 0.10^{b}
NCS-G4	$0.45 \pm 0.05^{a,b}$	0.80 ± 0.01^{b}	2.48 ± 0.07^{b}	2.27 ± 0.10^{b}	0.71 ± 0.04^{b}	0.50 ± 0.04^{c}
NCS-G5	0.49 ± 0.02^{b}	0.86 ± 0.03^{a}	$2.48 \pm 0.10^{a,b}$	2.35 ± 0.10^{b}	0.69 ± 0.05^{b}	0.66 ± 0.07^{d}
NCS-P50-1	$0.39 \pm 0.03^{b,c}$	0.85 ± 0.04^{a}	3.09 ± 0.12^{c}	1.76 ± 0.03^{c}	0.99 ± 0.05^{c}	0.94 ± 0.08^{b}
NCS-P50-2	0.36 ± 0.01^{c}	0.83 ± 0.02^{b}	3.09 ± 0.14^{c}	1.73 ± 0.07^{c}	0.94 ± 0.05^{c}	0.86 ± 0.04^{b}
NCS-P50-3	$0.41 \pm 0.06^{b,c}$	0.82 ± 0.04^{b}	2.94 ± 0.07^{c}	1.77 ± 0.04^{c}	0.90 ± 0.06^{c}	0.80 ± 0.06^{b}
NCS-P50-4	$0.38 \pm 0.10^{a,b,c}$	0.83 ± 0.03^{b}	2.85 ± 0.08^{c}	1.77 ± 0.07^{c}	1.07 ± 0.03^{d}	0.91 ± 0.10^{b}
NCS-P70-1	$0.46 \pm 0.08^{a,b,c}$	0.77 ± 0.03^{b}	2.57 ± 0.07^{d}	1.58 ± 0.05^{d}	1.01 ± 0.04^{c}	0.79 ± 0.09^{b}
NCS-P70-2	$0.43 \pm 0.15^{a,b,c}$	0.80 ± 0.09^{b}	2.52 ± 0.04^{d}	1.58 ± 0.02^{d}	0.99 ± 0.04^{c}	0.73 ± 0.08^{d}
NCS-P70-3	$0.42 \pm 0.12^{a,b,c}$	0.81 ± 0.03^{b}	2.66 ± 0.27^{a}	1.60 ± 0.05^{d}	1.01 ± 0.07^{c}	0.77 ± 0.09^{d}
NCS-P70-4	0.43 ± 0.01^{b}	0.71 ± 0.04^{c}	2.62 ± 0.08^{d}	1.09 ± 0.05^{e}	0.95 ± 0.02^{c}	0.89 ± 0.07^{c}
NCS-B	0.49 ± 0.02^{b}	0.89 ± 0.03^{b}	2.08 ± 0.11^{e}	2.81 ± 0.04^{f}	0.93 ± 0.05^{c}	0.60 ± 0.00^{d}
NCS-S50	0.95 ± 0.19^{d}	1.45 ± 0.07^{d}	3.82 ± 0.05^{f}	1.43 ± 0.10^{g}	1.03 ± 0.04^{c}	0.93 ± 0.00^{c}
NCS-S70	0.79 ± 0.18^{d}	1.09 ± 0.06^{e}	2.83 ± 0.03^{g}	1.79 ± 0.03^{c}	1.01 ± 0.04^{c}	1.51 ± 0.07^{e}

NCS-G: granulated NCS; NCS-P: powdered NCS at different mesh from 50 °Brix (P50) and 70 °Brix (P70); NCS-B: NCS block sample; NCS-S50: syrup 50 °Brix, and NCS-S70: 70 °Brix. $^{a–g}$ Means within a column, values with different superscript letters differ significantly ($p < 0.05$).

Table 4

Coefficient of determination from the multiple regressions between the content of phenolic compounds and the corresponding L_{ab} parameters (scalar and angular coordinates*) [a]

	L^*, a^*, b^*	L^*, h_{ab}, C_{ab}^*
Total phenolic content	0.6768[a]	0.7813[a]
Total flavonoid content	0.2430	0.2371
Protocatechuic acid	0.7685[a]	0.8058[a]
Vanillic acid	0.8190[a]	0.7896[a]
Chlorogenic acid	0.7423[a]	0.7886[a]
Syringic acid	0.7916[a]	0.7649[a]
p-Coumaric acid	0.0423	0.0544
Ferulic acid	0.3044	0.3335

[a] Significant effect ($p < 0.05$).

Figure(s)

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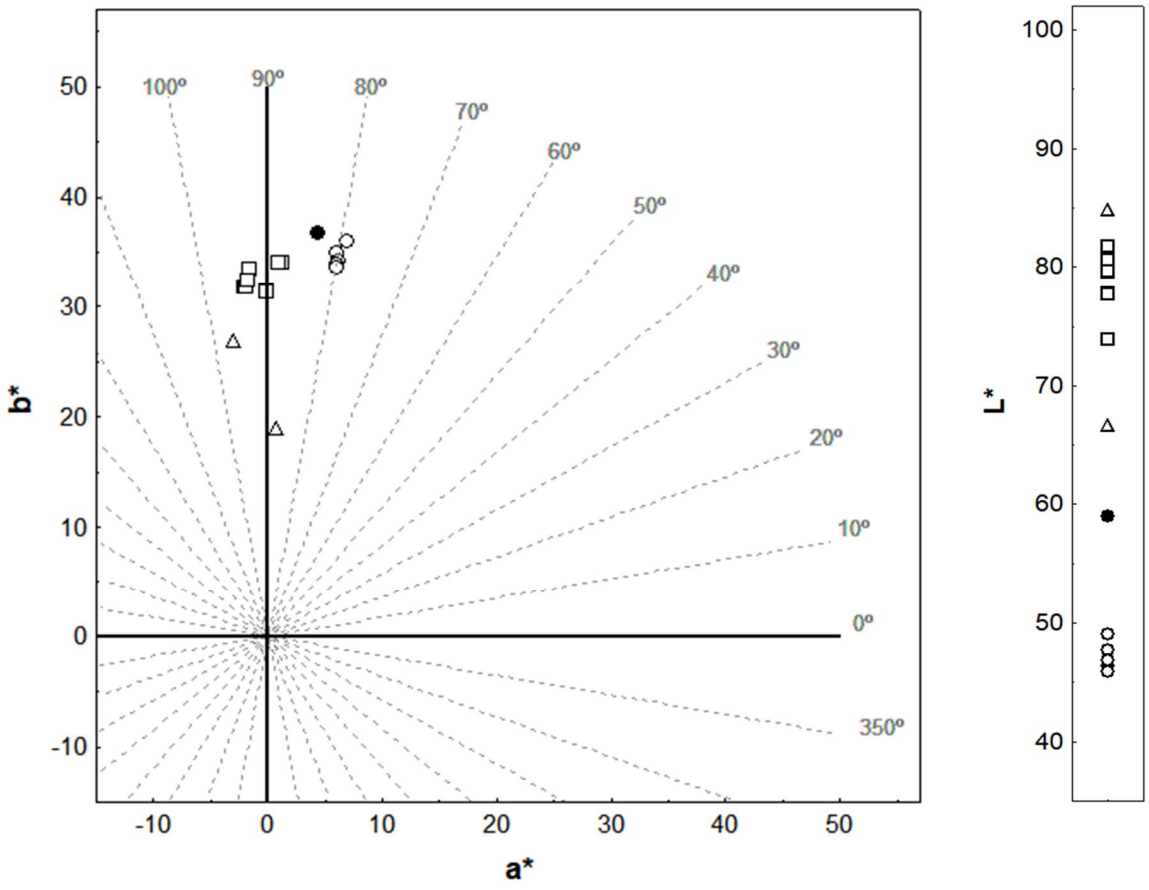

Figure(s)
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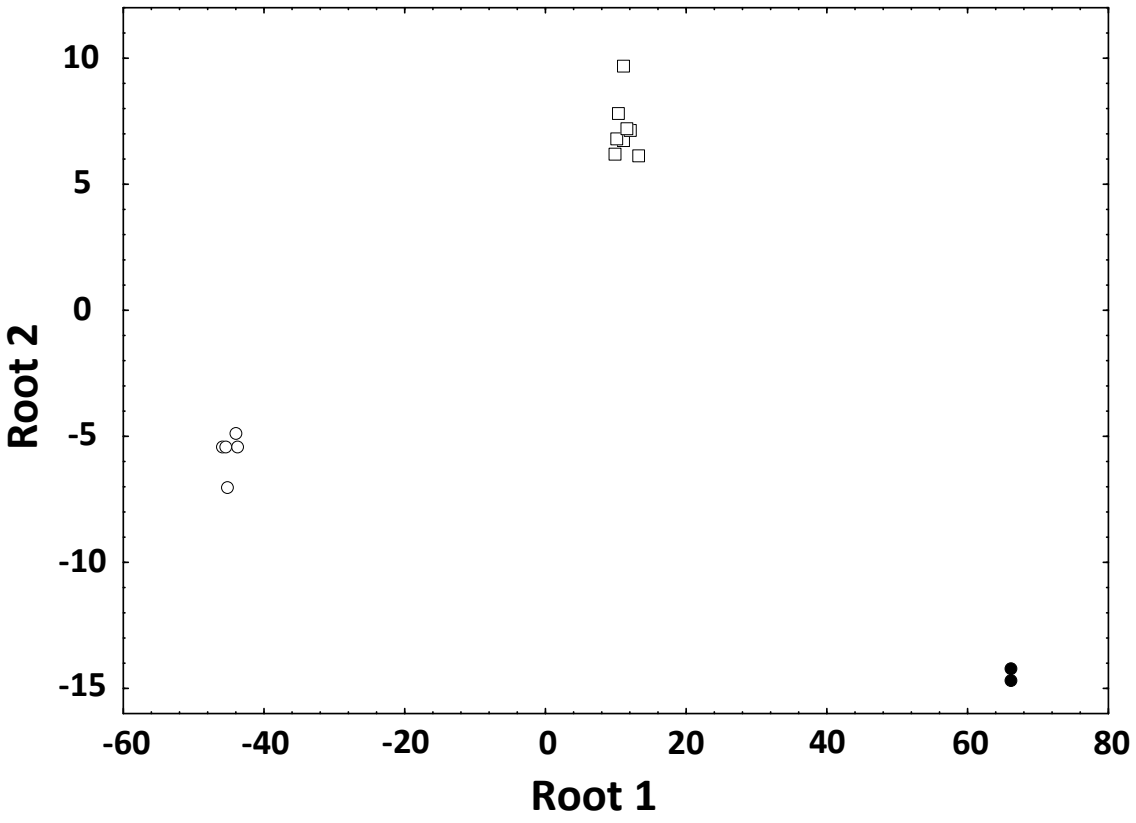

Variable	F-value	Standardized coefficients	
		Root 1	Root 2
*L**	86.447	7.801	0.650
Glucose	18.431	-6.409	1.696
Syringic acid	12.035	2.312	-2.663
Eigenval		1650.912	82.637
Cum.Prop		0.952	1.000

CRediT authorship contribution statement

Angela Liliana Alarcón was responsible for the laboratory investigation, writing of original draft preparation. **Laura M. Palacios** contributed with the laboratory investigation for collection of samples and obtaining powdered material. **Coralia Osorio** contributed with formal analysis, resources, original draft preparation, writing, review, and editing. **Paulo César Narváez** contributed in the conceptualization stage and in the definition of the experimental methodologies used in the collection and characterization of obtained materials, as well as the supervision of the research activities and writing the results. **Francisco J. Heredia** contributed in the conceptualization stage, in the definition of the experimental methodologies used during the colour analysis, supervision of physicochemical characterisation. **Alvaro Orjuela** contributed with formal analysis, resources, original draft preparation, writing, review, editing, project administration, and funding acquisition. **Dolores Hernanz** supervised the laboratory investigation on colour analysis and phenol compound characterisation, as well as writing, review and editing. All authors contributed to the final version of the manuscript and have read and agreed to the published version of the manuscript.

Supplementary Material

Chemical characteristics and colorimetric properties of non-centrifugal cane sugar ("panela") obtained via different processing technologies

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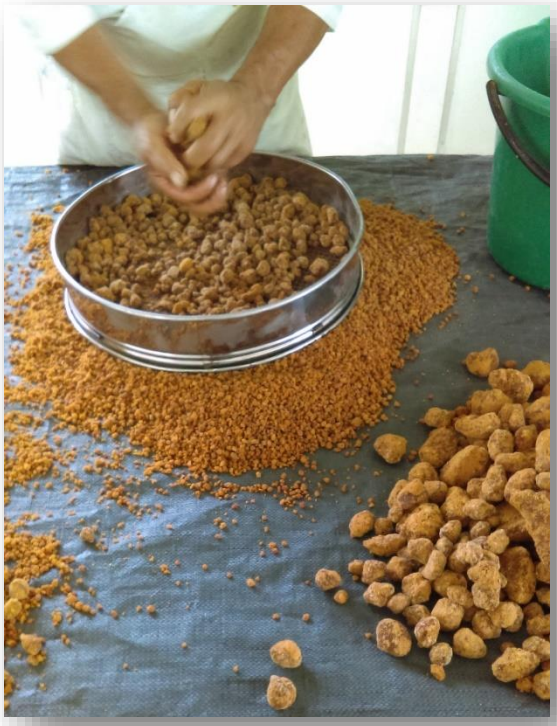
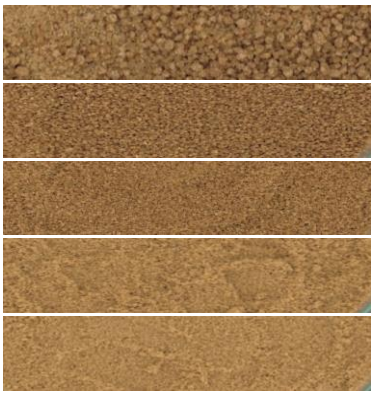


Figure 1S. Images of commercial NCS bricks and granulates manufacturing

Syrups (S)

Brick (B)



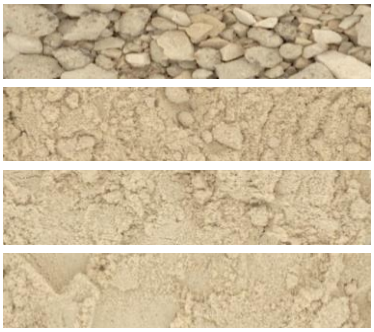


Figure 2S. Images of samples used during experiments. Syrup (S), NCS brick (B), granulated NCS of different particle sizes (G1, G2, G3, G4, G5), and spray dried products of different particle sizes (P1, P2, P3, P4).