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Utilization of Yacon Damaged Roots as a Source of FOS-Enriched Sweet-Tasting Syrup

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Featured Application: This work can be used for the development of sweet-tasting syrups rich in fructooligosaccharides by taking advantage of yacon agri-food by-products.

Abstract: Damaged yacon roots, often discarded, are a rich source of sweet carbohydrates. In this context, yacon roots from the Hualqui and Crespo varieties were characterized and processed into low-calorie and low-glycemic syrups for sugar reduction in foods. Syrups were obtained using, as technological adjuvants, lemon juice and its most relevant components: citric acid and ascorbic acid. The Hualqui variety was found to be mostly composed of fructose (210 g/kg), while the Crespo variety was rich in inulin (352 g/kg). The use of lemon juice during syrup production promoted the hydrolysis of inulin to fructooligosaccharides and fructose, yielding syrups with competitive relative sweetness (0.52–0.91), glycemic index (0.21–0.40), and caloric values (186–263 kcal/100 g) to commercial syrups. The increase in citric acid concentrations promoted inulin hydrolysis, yielding, at the highest concentration, syrups with higher fructose (333–445 g/kg) and kesto-type fructooligosaccharides (11–85 g/kg) content and lower surface stickiness and stringiness. The addition of ascorbic acid, as an antioxidant agent, decreased by 10% the free sugar content, negatively impacting the sweetness level. These results evidence that fructooligosaccharides-rich syrup can be obtained from yacon-damaged roots with tailored sweetness and low glycemic and caloric properties.

Keywords: inulin; fructooligosaccharide; syrup; clean label; sweetener; caloric value; lemon juice; citric acid; ascorbic acid

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1. Introduction

Yacon (*Smallanthus sonchifolius*) is a perineal plant from the family Asteraceae characteristic of the Andean region. This crop has been promoting the development of many Andean communities with low incomes, where it represents a relevant food source. The adaptability of yacon to moderate edaphoclimatic conditions has also allowed the expansion of yacon cultivation to many other geographical areas, such as in the USA, New Zealand, the Czech Republic, Italy [1], and Portugal. In Europe, the most common varieties are Morado, Blancom Llanera, among others, that have been cultivated according to their adaptability to the local edaphoclimatic conditions [2]. Depending on the variety, yacon plants form underground tuberous roots of yellowish, pink, purple, or brownish color, with an average of 15–20 cm long, a thickness of 10 cm, and a weight that might reach 2 kg [3]. Yacon cultivation presents a major issue related to its breakage during harvesting, packaging, and transportation. Such a process causes the darkening of the damaged tissues by polyphenol oxidation reactions, which are perceived as spoiled by the consumer [3]. In this context, the damaged roots account for an agro-industrial by-product that, when not channeled to low-commercial-value applications, is treated as an agro-industrial disposable due to its high perishability.

Many studies have focused on yacon valuation for flour applications [4], dried chips [5], or beverages [6]. However, the current association between type 2 diabetes, obesity, tooth decay, and high daily intakes of sugar, highlighted by the World Health Organization [7], has promoted the use of yacon for sweetening purposes in the form of syrups [8]. This results from the fact that, unlike many other plants that accumulate starch, yacon accumulates inulin, a polysaccharide composed of a sucrose unit extended by 1)→Fru*f*-(β2→ chain [9,10]. This structure differs from that of branched inulins, such as those found in agave, that present long 1)→Fru*f*-(β2→ and short 6)→Fru*f*-(β2→ chains at C1 and C6 of fructose of the sucrose unit, respectively. In agave inulins, the glucose unit of sucrose might also be branched at C6 with Fru [11]. Although the high temperatures of extraction can promote hydrolysis and debranching, these structural features are still present in the extracted fractions, allowing us to clearly distinguish linear and branched fructans. The decrease in the degree of polymerization of fructans due to the high temperatures of extraction also results in an increase in the sweetness of the products [12]. For linear inulin and derived fructooligosaccharides (FOS), the estimated sweetness sensation is 10% and 40% compared to sucrose [13]. Notably, they are not degraded by digestive enzymes. Consequently, both inulin and FOS have a role as dietary fiber and are reported to stimulate the growth of beneficial bacteria in the gut, acting as prebiotics [4,14]. In addition, antioxidants such as chlorogenic and caffeic acid derivatives and minerals such as potassium, calcium, and phosphorus, which also comprise relevant yacon components [15], together with the low content of fructose, glucose, and sucrose when compared to fruits, render yacon a low-caloric and high-nutrition food [16]. As a natural product, yacon has the potential to be a "clean label" sugar reduction strategy, currently demanded by informed consumers [8]. Accounting for this, yacon-damaged roots could be mitigated as agricultural by-products by their use in the development of syrups for sugar reduction in foods.

This work studies the potential use of the damaged roots of Hualqui and Crespo yacon varieties harvested in Portugal, with a specific emphasis on the carbohydrates present to produce syrups. This aims to comprehensively understand the fate of carbohydrates, especially when using "clean label" technological adjuvants, such as lemon juice. The work will consider various dimensions of syrup properties, taking into consideration different pH levels and the presence of ascorbic acid as an antioxidant in the FOS content of the syrups. The approach extends to relating the effects to syrup caloric value, glycemic index, sweetening power, and texture properties. In addition, the study will cover the impact of the water evaporation technique on the overall carbohydrate composition. This comprehensive perspective ensures an innovative and thorough examination of the factors impacting the FOS content in yacon syrup and its diverse applications.

2. Materials and Methods

2.1. Sample Preparation

The yacon samples, from the varieties Hualqui and Crespo (Figure 1a,b), comprised a heterogeneous mixture of small and larger roots that originated from pieces broken off during the harvesting process by YaconPortugal. For the Hualqui variety, the initial water content was 89%, while for the Crespo variety, it was 86%. To avoid enzymatic browning and inulin and FOS degradation as well as tuberous root spoilage upon storage, the samples were cut into small pieces, frozen, and freeze-dried. The freeze-dried step is necessary because, under vacuum conditions, the availability of oxygen is residual. This absence of oxygen prevents oxidation reactions from taking place during freeze-drying, avoiding the darkening of the juice. The dehydrated yacon by-products were then peeled. The isolated skins and pulps were then milled separately into fine powders that were stored at room temperature until further use. The skins and pulp were used for the characterization of the carbohydrate composition of the yacon varieties following ethanol extractions.

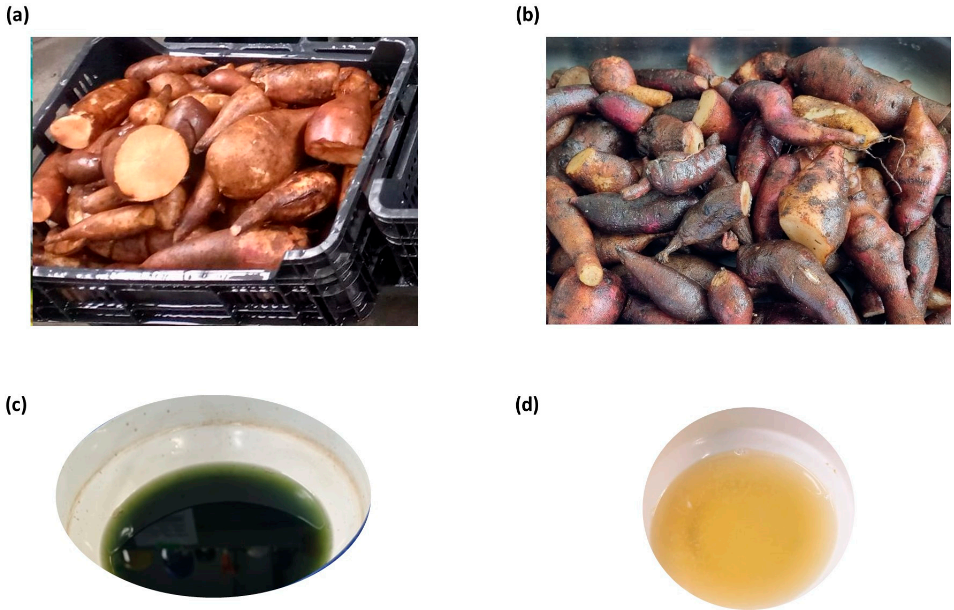

Figure 1. Yacon-damaged roots comprehending the pieces broken off during the harvest process of (**a**) Hualqui and (**b**) Crespo varieties and coloration of the extraction juice when obtained (**c**) with and (**d**) without the skins.

2.2. Hydroethanolic Extractions

To pursue the characterization of carbohydrates composition of the skin and pulp of the different yacon varieties, an aqueous ethanol extraction was performed, allowing the recovery of low-molecular-weight carbohydrates (EtSn extract) [17,18]. For this, 10 g of yacon pulp or skin were suspended in 100 mL of distilled water, followed by the addition of 400 mL of ethanol up to a final concentration of 80% (v/v). Samples were then boiled for 10 min under reflux, left to cool down for 3 h at 4 °C, and then centrifuged at 20,000 $\times$ g for 20 min at 4 °C. The obtained residue was then subjected to a new alcohol extraction following the same procedure. The obtained EtSn was then filtrated and combined with the previous extract. The combined EtSn was evaporated under reduced pressure at 40 °C to remove ethanol and then frozen and freeze-dried. The insoluble residue obtained upon filtration was washed with ethanol and acetone and left to dry at room temperature, yielding EtRe.

2.3. Syrup Production

For syrup production, 40 g of freeze-dried Hualqui pulp powder was suspended in 364 mL of water to reach a concentration of 11% solids, accounting for the water proportion of fresh roots (89%). Afterwards, commercially available lemon juice (SonaeMC, Porto, Portugal) was added at a concentration of 5% (v/v), taking into account the concentration of citric acid reported in lemon juices (48 g/L) [19], as a clean label solution to: (1) Promote inulin hydrolysis by taking advantage of the low pH of lemon juice provided by citric acid; and (2) avoid enzymatic browning reactions by taking advantage of ascorbic acid, an abundant antioxidant found in lemon juice. The obtained suspension was then filtered using a nylon cloth, and the residue was washed with an aqueous solution following the same process. The obtained filtered juices were then combined and concentrated by boiling at atmospheric pressure up to 70–75 °Brix. The boiling temperature was 100 °C in the diluted juices and reached higher values with the sugar concentration. This process resulted in one yacon syrup prepared with 5% lemon juice.

To unveil implications for the variation in citric acid (CitA) content of lemon and other citrus juices, tests were also performed with pure CitA: 0.1, 0.25, and 0.5% (w/v). The concentration range selected for citric acid was based on the concentrations reported by Penniston, Nakada, Holmes, and Assimos [19] for lemon juice (48 g/L). Assuming the addition of this juice at a 5% (v/v) level, the resulting concentration of citric acid is 0.25%. A concentration of 0.5% citric acid was considered the upper limit, as beyond this point, the perceived sweetness of the syrup becomes overshadowed by the sourness imparted by citric acid. The concentration of 0.1% was used as it accounted for the minimal amount of citric acid that allowed it to inhibit the enzymatic browning of the juice. A total of six syrups were obtained, three for each variety.

As the minimal concentration of ascorbic acid reported to efficiently inhibit the enzymatic browning by polyphenol oxidase is 0.08% w/v [20], avoiding the enzymatic oxidation of yacon polyphenols [21], an experiment was also performed by combining ascorbic acid at 0.08% with CitA at 0.25%. The juice mixture was concentrated at 100 °C at atmospheric pressure, yielding two syrups, one for each variety. The concentration at 60 °C using a rotary evaporator, until reaching a syrup of ≈70 °Brix, yielded one syrup for the Crespo variety.

2.4. Chemical Composition

2.4.1. Moisture and Total Solids Content

The moisture content of yacon by-products was determined for a subset of yacon samples, considering consistent values between the different roots within each variety. The determination accounted for the difference between the weight of the damaged roots before and after freeze-drying (Equation (1)).

$$\% \text{ Moisture} = (w_{\text{fresh by-product}} - w_{\text{freeze-dried by-product}})/(w_{\text{fresh by-product}}) \times 100. \quad (1)$$

The syrup's total solids content was measured following a 10-fold dilution with water and using a refractometer (HANNA HI96813, Hanna Instruments Portugal-Póvoa de Varzim, Amorim, Portugal), with a scale ranging from 0 to 50 °Brix.

2.4.2. Carbohydrate Composition

The neutral sugars composition of EtSn and EtRe was determined following the Saeman acid hydrolysis, reduction, and acetylation steps for the conversion of the monosaccharides to alditol acetates. Gas chromatography with a flame ionization detector (GC-FID, Perkin Elmer–Claus 400, PerkinElmer Inc., Shelton, CT, USA) equipped with a DB-225 (J&W Scientific-Agilent Technologies, Santa Clara, CA, USA) column was used for quantification purposes. 2-deoxyglucose was used as an internal standard [22]. The *m*-Phenylphenol method was used to determine colorimetrically the uronic acids (UA) [23].

The free glucose (Glc) and fructose (Fru), sucrose (Suc), fructooligosaccharides (FOS), and inulin content in the syrup samples were determined by high-performance anion-exchange chromatography/pulsed amperometric detection (HPAEC-PAD) using a Dionex ICS-600 system equipped with a DC oven and SP, controlled by Chromeleon 7.3 software (Thermoscientific Dionex – Waltham, MA, USA). Carbohydrates were detected using an electrochemical detector in integrated amperometry mode with an AgCl reference electrode and a conventional electrode. The carbohydrate separation was performed using a Dionex CarboPac PA100 pre-column (50 mm × 4 mm) and a Dionex CarboPac PA100 analytical column (250 mm × 4 mm). The eluents used were eluent A—MilliQ water (resistance of ≈18 MΩ.cm), eluent B—500 mM NaOH (prepared from a 50% sodium hydroxide solution, MERCK), and eluent C—1 M sodium acetate (sodium acetate, Thermo Scientific™ Dionex™–Waltham, MA, USA) with 100 mM NaOH. The eluents were filtered through a 0.2 nylon filter µm and degassed under vacuum with sonication for 30 min. Afterwards, the eluents were transferred to 2 L Thermo Fisher Dionex plastic bottles and placed under a nitrogen atmosphere. The column and detector temperatures were 30 °C. The initial equilibrium was performed with 12.0% solvent B and 0.5% solvent C. After injecting 25 µL of sample into the column, the compounds were eluted at a flow rate of 1 $mL.min^{-1}$ by the following method: An initial step of A:B:C 87.5:12.0:0.5 ($v/v/v$) for 5 min; a gradient from 87.5:12.0:0.5 to 61:24:15 from 5 to 50 min; followed by a 0:25:75 gradient from 50 to 105 min. After a gradient of 0:0:100 for 5 min, kept for 10 min to remove impurities, the eluent proportion was returned to the initial conditions and held for an equilibration time of 20 min. Glc, Fru, Suc, kestose, kestotetraose, inulotriose, and kestopentaose were used as standards for quantification. FOS solely composed of Fru units (inulo-type FOS) were quantified as inulotriose equivalents. FOS containing the sucrose unit (kesto-type FOS), presenting a degree of polymerization (DP) between 5–9, and inulins (DP $\geq$ 10) were quantified as kestopentaose equivalents. The FOS and inulin average degree of polymerization (DP) was determined by accounting for the sum of the product between the relative proportion of each FOS/inulin and the respective DP.

To determine other polysaccharides composing the polymeric material, yacon syrups were dialyzed with a cut-off membrane of 12 kDa (Medicell). Before dialysis, syrup samples were previously diluted in water and centrifuged at 20,000$\times$ g (Thermo Scientific Multifuge X1R) for 10 min for the removal of particulate material. The polymeric material of the syrups was freeze-dried and subjected to neutral sugar and uronic acid analysis.

2.4.3. Protein Analysis

The protein content of the polymeric material in syrups was determined following elemental analysis. Briefly, this analysis comprehended a four-stage process: (1) The purge where all the gases are removed from the sample; (2) the combustion by burning the sample at 1100 °C in the presence of a controlled amount of oxygen; (3) oxidation in a second oven at 850 °C; and (4) analysis step where the gases from combustion pass through detectors. Nitrogen is measured by thermal conductivity in a Truspec 630-200-200 elemental analyzer (LECO–Vouersweg, The Netherlands) with a thermal conductivity detector (TDC). Protein content was quantified, accounting for the total nitrogen determined and using a conversion factor of 6.25.

2.5. Texture Analysis

Syrup texture was analyzed using a Texture Analyzer TA.XT Plus C (Stable Micro Systems, Cardiff, UK) with a 5 kg load cell. About 20 g of syrups, on a fresh weight basis, was placed on acrylic cylindrical containers (50 mm internal diameter and 75 mm height) and submitted to an adhesive test using a compression cylindrical probe of 35 mm diameter. A force of 6 g at a velocity of 1 mm/s was exerted on the surface of the sample and held for 2 s. After the compression test, the probe returned from the sample at a velocity of 8 mm/s, stopping at 170 mm.

From the force–time curve, the empirical attributes of surface stickiness and stringiness were obtained using the Exponent Connect version 8,0,3,0 equipment software. Surface stickiness accounted for the maximum force needed to separate the probe from the syrup sample. This attribute describes the stickiness experienced when the syrup comes into contact with another surface, such as the mouth. Stringiness was recorded as the maximum distance traveled by the probe before the force dropped to 2.5 g. Stringiness is a textural attribute that refers to the capacity to form string-like structures when poured or manipulated. Commercial yacon (YaconPortugal, Tábua, Portugal) and agave syrups (Go BIO, Mexico) were used for comparison purposes.

2.6. Syrups Sweetness and Nutritional Features

The theoretical relative sweetness (TRS), theoretical glycemic index (TGI), and caloric values of syrups were determined by accounting for the relative proportion of free sugars (Glc and Fru), Suc, FOS, and inulin, and their estimated relative sweetness, glycemia, and calories reported in the literature. For the estimation of syrup TRS (Equation (2)), a parameter that only accounts for the contribution of sugars to the sweetness, excluding potential synergisms and matrix effects, a relative sweetness level of 1.7 was used for Fru, 0.8 for Glc, and 0.4 for FOS [24–26]. Yacon inulin, being linear, was assigned a value of 0.1 for calculating the sweetening power of the syrups [13]. These values are normalized to sucrose, used as a reference sugar with a sweetening power of 1.0 at room temperature [24–26].

$$TRS = Fru\ (\%_{w/w}) \times 1.7 + Glc\ (\%_{w/w}) \times 0.8 + Suc\ (\%_{w/w}) \times 1.0 + FOS\ (\%_{w/w}) \times 0.4 + inulin\ (\%_{w/w}) \times 0.1. \quad (2)$$

For the estimation of the TGI (Equation (3)), a parameter that only accounts for the contribution of sugars to the glycemic index, excluding potential synergisms, matrix effects, and variability among the health status of consumers, a relative glycemic level of 0.3 was used for Fru (average of 7 studies) and 1.5 for Glc (average of 11 studies) [27]. A relative glycemic index of 0.015 was used for FOS and inulin [25,28]. These values were obtained by accounting for the relative glycemic indexes of each sugar and their normalization to sucrose, the reference sugar, with a relative glycemic index of 1.0 (average of 12 studies [27]).

$$TGI = Fru\ (\%_{w/w}) \times 0.3 + Glc\ (\%_{w/w}) \times 1.5 + Suc\ (\%_{w/w}) \times 1 + FOS\ (\%_{w/w}) \times 0.015 + inulin\ (\%_{w/w}) \times 0.015. \quad (3)$$

For the estimation of syrups caloric value (Equation (4)), a caloric value of 4 kcal was used for Fru, Glc, and Suc and 2 kcal for FOS and inulin [29,30].

$$Caloric\ value = Fru\ (\%_{w/w}) \times 4 + Glc\ (\%_{w/w}) \times 4 + Suc\ (\%_{w/w}) \times 4 + FOS\ (\%_{w/w}) \times 2 + inulin\ (\%_{w/w}) \times 2. \quad (4)$$

2.7. Statistical Analysis

All chemical analyses were performed in triplicate unless otherwise stated. The reproducibility of the results is expressed as the average ± standard deviation.

3. Results and Discussion

3.1. Yacon Varieties Composition

The damaged roots of the Hualqui and Crespo yacon varieties were found to have a pH of 6.0, with dry matter contents of 11% and 14%, respectively. The total soluble solids accounted for 6.7 °Brix for the Hualqui variety and 10.1 °Brix for the Crespo variety. These features were comparable to the varieties cropped in the Peru region [9,31], which are reported to have pH levels ranging from 6.0 to 6.5, dry matter contents of 8 to 13%, and total soluble solids of 8 to 11 °Brix. Cultivars grown in the southwest region of Germany exhibit dry matter contents of 9 to 17% [2,32]. The proportion of pulp in Hualqui and Crespo-damaged roots was 80%, consistent with the percentages remaining in yacon juice extraction [21].

Accounting for the chromatogram (Figure 2), the overall composition and yields of EtSn and EtRe (Tables 1 and S1), as well as the proportion of skin and pulp, the whole carbohydrate composition of the yacon Hualqui and Crespo varieties were determined. As shown in Figure 3a (values in Table S1), the Hualqui variety exhibited higher concentrations of Glc (73 g/kg dry weight) and Fru (210 g/kg) compared to the Crespo variety (41 g/kg of Glc and 90 g/kg of Fru). When comparing these findings to varieties grown in Southwest Germany [2], the Crespo and Hualqui varieties had nearly 4 to 30 times more Fru. While other factors likely contribute to these differences, variations in the edaphoclimatic conditions and genetic factors [2,33] play a significant role. The Hualqui variety contained a higher proportion of lower molecular weight FOS (Figure 3b), with an average DP of 5, in contrast to the Crespo variety, which had FOS with an average DP of 6. The Crespo variety was found to have a total fructan (FOS + inulin) content of 509 g/kg. These levels are consistent with the values reported for yacon flour (360–670 g/kg) from varieties cultivated in tropical climates of Brazil [20] and the Andean region of Peru (540–620 g/kg) [31]. The fructan content of Crespo-damaged roots is also comparable with highly regarded fructan sources such as artichokes (290 g/kg) [34], Jerusalem artichoke (400–690 g/kg) [35], chicory (450–587 g/kg) [36,37], asparagus (579 g/kg) [37], and agave (809 g/kg) [37,38]. In contrast, the damaged roots of the Hualqui varieties herein studied displayed a lower fructan content (150 g/kg). The Hualqui variety also contained lower levels of Ara (3 g/kg) and Gal (2 g/kg) (Table 2), sugars characteristic of the neutral side chains of pectic polysaccharides [9,22], in contrast to the Crespo variety, which featured 12 and 6 g/kg, respectively. The occurrence of Glc in the Hualqui variety also suggests the occurrence of about 26 g/kg glucans, including cellulose [9]. Due to the presence of inulin and FOS in the EtRe fractions of the Crespo variety, the determination of glucans was not performed because the methodology used does not distinguish Fru from Man and Glc [39].

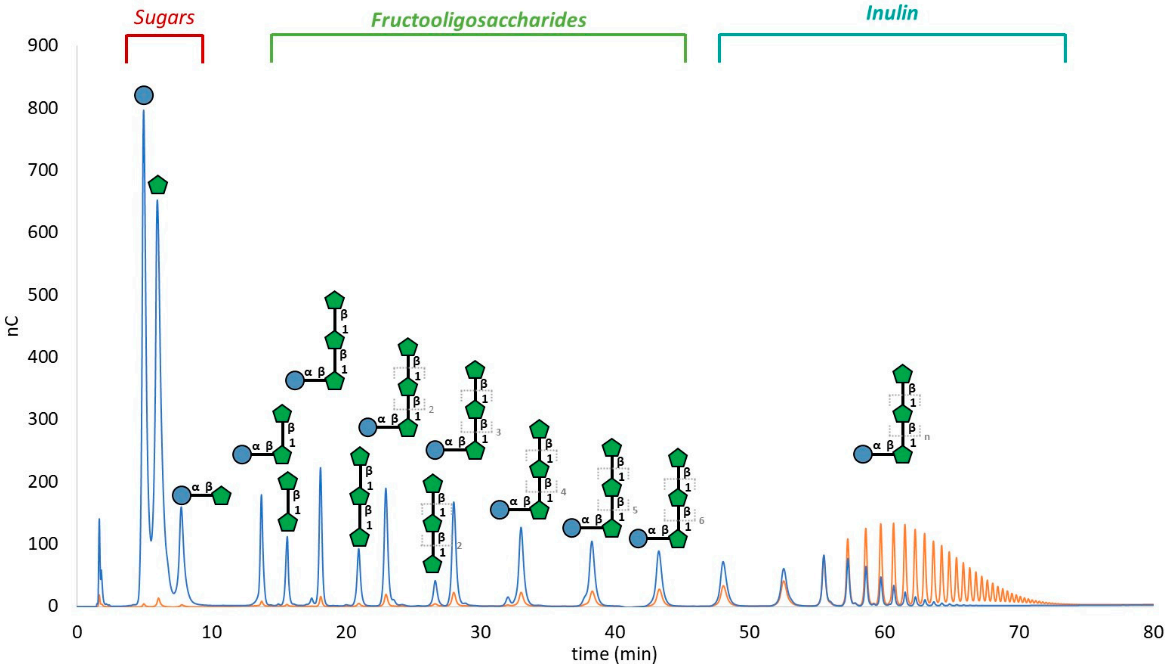

Figure 2. HPAEC-PAD chromatogram of the ethanol soluble material (EtSn, blue) and ethanol residue (EtRe, orange) of the pulp from the damaged roots of yacon Crespo variety. Blue circle—glucose; green pentagon—fructose.

Table 1. Relative proportion (η_{root} (%)) of skin and pulp, yields of the ethanol soluble (EtSn), and ethanol residue (EtRe), and respective carbohydrate composition of the skin and pulp of the yacon Hualqui and Crespo varieties as determined by HPAEC-PAD without any hydrolysis step. Glc—glucose; Fru—fructose, Suc—sucrose; FOS—fructooligosaccharides; DP—degree of polymerization.

	Hualqui				**Crespo**			
	Skin		**Pulp**		**Skin**		**Pulp**	
η_{root} (%)	20		80		20		80	
Fraction	**EtRe**	**EtSn**	**EtRe**	**EtSn**	**EtRe**	**EtSn**	**EtRe**	**EtSn**
η_{EtPp} (%)	31	49	9	86	42	56	64	34
Sugar								
Glc	-	100 ± 1	tr	92 ± 7	tr	78 ± 3	tr	117 ± 13
Fru	-	243 ± 5	tr	272 ± 7	tr	212 ± 21	tr	240 ± 9
Suc	-	34 ± 0	tr	47 ± 2	tr	57 ± 0	tr	51 ± 1
FOS (kesto type)								
DP3	-	23 ± 1	tr	43 ± 1	tr	24 ± 0	tr	22 ± 0
DP4	-	29 ± 0	tr	40 ± 2	tr	31 ± 3	3 ± 0	38 ± 1
DP5	-	29 ± 1	tr	37 ± 2	tr	42 ± 2	8 ± 0	55 ± 1
DP6	-	16 ± 0	tr	21 ± 1	tr	33 ± 2	10 ± 0	53 ± 2
DP7	-	13 ± 1	tr	15 ± 1	tr	32 ± 4	12 ± 0	50 ± 2
DP8	-	12 ± 0	tr	11 ± 1	2 ± 0	32 ± 2	15 ± 1	45 ± 6
DP9	-	10 ± 0	1 ± 0	9 ± 0	3 ± 0	33 ± 2	20 ± 2	40 ± 2
FOS (Inulo type)								
DP2	-	3 ± 0	tr	4 ± 0	tr	12 ± 1	tr	15 ± 0
DP3	-	-	tr	1 ± 0	tr	11 ± 1	1 ± 0	16 ± 0
DP4	-	-	tr	-	tr	4 ± 0	tr	8 ± 0
DP5	-	-	tr	-	tr	tr	-	-
DP6	-	-	tr	-	tr	tr	-	-

Table 1. *Cont.*

	Hualqui				Crespo			
	Skin		**Pulp**		**Skin**		**Pulp**	
Inulin								
DP10	-	7 ± 0	1 ± 0	6 ± 0	5 ± 1	31 ± 3	25 ± 1	36 ± 1
DP11	-	5 ± 0	2 ± 0	4 ± 0	8 ± 1	27 ± 1	28 ± 0	29 ± 0
DP12	-	4 ± 0	1 ± 0	2 ± 0	12 ± 0	23 ± 1	31 ± 2	21 ± 0
DP13	-	3 ± 0	1 ± 0	1 ± 0	15 ± 1	18 ± 1	34 ± 2	17 ± 0
DP14	-	2 ± 0	1 ± 0	1 ± 0	17 ± 2	13 ± 1	37 ± 1	15 ± 0
DP15	-	1 ± 0	1 ± 0	tr	19 ± 2	9 ± 0	40 ± 1	9 ± 0
DP16	-	1 ± 0	tr	-	20 ± 2	6 ± 1	34 ± 6	6 ± 0
DP17	-	1 ± 0	tr	-	20 ± 2	3 ± 0	36 ± 1	4 ± 0
DP18	-	tr	-	-	19 ± 1	2 ± 0	31 ± 4	3 ± 0
DP19	-	Tr	-	-	18 ± 2	1 ± 0	30 ± 1	2 ± 0
DP20	-	-	-	-	16 ± 1	1 ± 0	28 ± 0	1 ± 0
DP21	-	-	-	-	15 ± 1	tr	24 ± 1	1 ± 0
DP22	-	-	-	-	14 ± 1	tr	22 ± 1	tr
DP23	-	-	-	-	12 ± 1	-	19 ± 0	-
DP24	-	-	-	-	11 ± 1	-	17 ± 0	-
DP25	-	-	-	-	10 ± 1	-	15 ± 0	-
DP26	-	-	-	-	9 ± 0	-	13 ± 0	-
DP27	-	-	-	-	8 ± 0	-	11 ± 0	-
DP28	-	-	-	-	7 ± 0	-	10 ± 0	-
DP29	-	-	-	-	6 ± 0	-	8 ± 0	-
DP30	-	-	-	-	6 ± 0	-	7 ± 0	-
DP31	-	-	-	-	5 ± 0	-	6 ± 0	-
DP32	-	-	-	-	4 ± 0	-	5 ± 0	-
DP33	-	-	-	-	4 ± 0	-	4 ± 0	-
DP34	-	-	-	-	3 ± 0	-	4 ± 0	-
DP35	-	-	-	-	3 ± 0	-	3 ± 0	-
DP36	-	-	-	-	3 ± 0	-	3 ± 0	-
DP37	-	-	-	-	2 ± 0	-	2 ± 0	-
DP38	-	-	-	-	2 ± 0	-	2 ± 0	-
DP39	-	-	-	-	2 ± 0	-	2 ± 0	-
DP40	-	-	-	-	2 ± 0	-	1 ± 0	-
DP41	-	-	-	-	1 ± 0	-	1 ± 0	-
DP42	-	-	-	-	1 ± 0	-	1 ± 0	-
DP43	-	-	-	-	1 ± 0	-	tr	-
DP44	-	-	-	-	1 ± 0	-	tr	-
DP45	-	-	-	-	1 ± 0	-	tr	-
DP46	-	-	-	-	1 ± 0	-	tr	-
DP47	-	-	-	-	1 ± 0	-	tr	-
DP48	-	-	-	-	1 ± 0	-	tr	-
DP49	-	-	-	-	tr	-	tr	-
DP50	-	-	-	-	tr	-	tr	-
Total	-	532 ± 10	9 ± 0	606 ± 8	311 ± 21	734 ± 41	608 ± 23	888 ± 17

Considering the overall carbohydrate composition of yacon-damaged roots, it was possible to conclude that the Hualqui variety presented a higher theoretical sweetness and glycemic index than the Crespo variety (Table 3). The estimated caloric index for these varieties ranged from 157 kcal/100 g to 162 kcal/100 g on a dry weight basis.

Accounting for the composition of skins and pulps, both tissues could be used for syrup production. However, when using the skin during processing, the juice obtained acquired a dark coloration due to pigment diffusion from the skins (Figure 1c,d). For this reason, only the pulp of yacon was further used to produce the syrups.

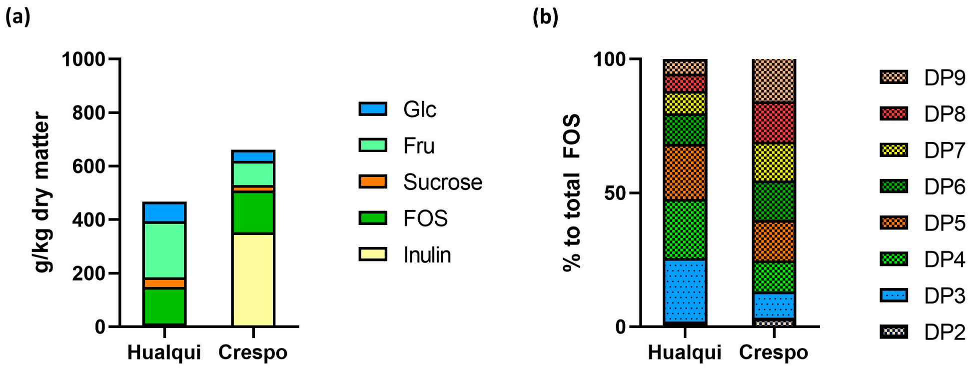

Figure 3. Representation of the (**a**) carbohydrate composition (g/kg dry matter) of the damaged roots of yacon Crespo and Hualqui varieties and (**b**) relative proportion of the different degrees of polymerization of fructooligosaccharides as determined by HPAEC-PAD. Glc—glucose; Fru—fructose; FOS—fructooligosaccharides; DP—degree of polymerization.

Table 2. Neutral sugar and uronic acid composition of the ethanol-soluble material (EtSn) and residue (EtRe) of the Hualqui and Crespo yacon varieties. Rha—rhamnose; Fuc—fucose; Ara—arabinose; Xyl—xylose; Man—mannose; Gal—galactose; Glc—glucose; UA—uronic acids.

Variety	Fraction	Yield (%)	Carbohydrate Composition (molar %)								Total (g/kg)
			Rha	Fuc	Ara	Xyl	Man	Gal	Glc	UA	
Hualqui											
Skin	*EtSn*	49	-	-	1 ± 0	-	22 ± 1	-	70 ± 2	7 ± 1	331 ± 37
	EtRe	31	tr	tr	3 ± 0	1 ± 0	tr	1 ± 0	9 ± 1	86 ± 1	220 ± 9
Pulp	*EtSn*	86	-	-	-	-	23 ± 1	-	71 ± 1	6 ± 2	402 ± 15
	EtRe	9	tr	-	2 ± 0	1 ± 0	tr	1 ± 0	10 ± 2	84 ± 2	335 ± 11
Crespo											
Skin	*EtSn*	64	-	-	-	-	24 ± 1	-	71 ± 1	5 ± 0	538 ± 38
	EtRe	34	1 ± 0	1 ± 0	13 ± 1	1 ± 0	22 ± 3	5 ± 0	48 ± 6	9 ± 1	338 ± 47
Pulp	*EtSn*	56	-	-	-	-	28 ± 2	-	67 ± 1	5 ± 1	547 ± 26
	EtRe	42	tr	tr	4 ± 0	1 ± 0	28 ± 1	2 ± 0	49 ± 1	15 ± 1	500 ± 16

Table 3. Yield, total soluble solids (°Brix), theoretical relative sweetening index (TRS), theoretical relative glycemic index (TRG), and caloric value (kcal/100 g dry weight) of the initial yacon juice and the obtained yacon syrups from Hualqui and Crespo varieties damaged roots. CitA—citric acid; AA—ascorbic acid.

Yacon Sample	Evaporation Pressure	Yield (%) *	°Brix	pH	TRS	TRG	Caloric Value (kcal/100 g)
Hualqui							
Root	-	-	6.7	6.03	0.51 ± 0.00	0.21 ± 0.01	157 ± 1
Syrups							
5% lemon	Atmospheric	-	72	4.15	0.88 ± 0.02	0.40 ± 0.01	256 ± 6
0.1% CitA	Atmospheric	72	68	4.87	0.71 ± 0.01	0.30 ± 0.02	214 ± 4
0.25% CitA	Atmospheric	70	70	4.20	0.89 ± 0.03	0.35 ± 0.01	263 ± 8
0.25% CitA + 0.08% AA	Atmospheric	57	75	4.23	0.62 ± 0.00	0.30 ± 0.00	186 ± 2
0.5% CitA	Atmospheric	79	68	3.90	0.91 ± 0.02	0.38 ± 0.00	253 ± 4
Crespo							
Root	-	-	10.1	6.00	0.30 ± 0.01	0.12 ± 0.01	162 ± 5
Syrups							
0.1% CitA	Atmospheric	79	73	5.12	0.55 ± 0.00	0.21 ± 0.01	223 ± 1
0.25% CitA	Atmospheric	68	72	4.47	0.60 ± 0.01	0.23 ± 0.01	224 ± 1
0.25% CitA + 0.08% AA	Atmospheric	68	70	4.34	0.59 ± 0.02	0.23 ± 0.00	212 ± 5
0.25% CitA + 0.08% AA	Reduced	79	70	4.36	0.52 ± 0.05	0.21 ± 0.02	216 ± 14
0.5% CitA	Atmospheric	78	76	3.79	0.73 ± 0.03	0.28 ± 0.01	214 ± 8

* Dry matter of syrup obtained from 100 g of pulp.

3.2. Syrup Production

Yacon syrup production follows a juice extraction and filtration process to remove solid particles in suspension. To prevent enzymatic browning, an antioxidant is commonly added to the clarified juice. The juice is then concentrated to at least 70 °Brix [21]. In this study, lemon juice was added as an antioxidant to yacon water-soluble material, inhibiting the polyphenol oxidation reaction by the decrease in pH, promoted by citric acid, and by the reducing power of ascorbic acid [40,41]. The addition of lemon juice resulted in a decrease in the juice pH from 6.0 to 4.2 (Table 1). Its concentration by boiling at atmospheric pressure yielded a syrup at 72 °Brix, representing an estimated water loss of about 91%. The dry matter accounted for 155 g/kg of Glc, 394 g/kg of Fru, 46 g/kg of Suc, and 90 g/kg of FOS (Figure 4a). Of the FOS, 26% were of the inulo-type (Figure 4b), representing a 10-fold increase compared to the initial raw material (Table S2). This was attributed to inulin hydrolysis, favored by the acidic pH of the juice combined with the boiling temperatures achieved during concentration [42]. This was also supported by the traceable amounts of inulin detected in this syrup. On a dry weight basis, the theoretical relative sweetness, theoretical glycemic index, and caloric value of the syrup were higher than the raw material by 72%, 90%, and 63%, respectively (Table 2). This was attributed to the higher proportion of sweet-tasting carbohydrates (Glc, Fru, Suc, and FOS) in the syrup [8,24].

The commercial yacon syrup (Yacon Portugal) presented a theoretical relative sweetness of 1.05 ± 0.07, a theoretical relative glycemic index of 0.28 ± 0.03, and a caloric value of 292 ± 19 kcal/100 g. The theoretical relative sweetness and caloric value values were higher than those obtained for 5% lemon juice syrup. The commercial agave syrup (Go Bio), produced from an inulin source [12], also presented higher theoretical relative sweetness (1.34 ± 0.10), theoretical relative glycemic index (0.53 ± 0.03), and caloric value (361 ± 25 kcal/100 g). These differences result from the higher prevalence of Glc and Fru in the commercial syrups than in the 5% lemon juice syrup (Figure 4a and Table S2), due to the extensive saccharification of inulin and FOS for commercial sweetening purposes.

Citric acid and ascorbic acid proportions in citrous juices may vary among species, varieties, seasons, and geographic locations [43]. To understand how these variations might affect the FOS proportion in syrups, citric acid and ascorbic acid were tested as pure components at different concentrations. These tests were conducted using the damaged roots of the yacon Hualqui variety, chosen as a representative source with high free sugar content, and yacon Crespo damaged roots, selected as a representative source of high inulin content.

3.2.1. Effect of Citric Acid Concentration on Syrup Properties

The addition of 0.1% CitA to yacon juice decreased the pH from 6.0 to the range of 4.9–5.1 (Table 3). Upon concentration, the water loss was 87–90%, resulting in syrups of a dark/brown color (Figure S1) presenting 68–73 °Brix and containing 72–79% (*w*/*w*) of yacon dry matter. The carbohydrates accounted for 460 g/kg of syrup obtained from the Hualqui variety and 598 g/kg of syrup for the Crespo variety (Figure 4c,e and Table S3). In the 0.1% CitA Hualqui syrup, free sugars accounted for the most abundant carbohydrates (263 g/kg of syrup), while in the 0.1% CitA Crespo syrup, these accounted for FOS (212 g/kg of syrup) and inulin (165 g/kg of syrup). This composition led to a lower theoretical relative sweetness and theoretical relative glycemic index compared to the Hualqui variety syrup (Table 3). The presence of GalA (5 mol%), Ara (1 mol%), and Gal (1 mol%) in the polymeric material of the syrup also suggested that a portion of yacon pectic polysaccharides becomes solubilized during juice extraction (Table 4). It was also determined that the protein content of yacon syrup is 2.7 g/kg (0.3%), making a negligible contribution to the syrup's caloric value. This protein content was lower than the 10 to 16 g/kg reported for other yacon syrups [21,44,45] and the 15 g/kg reported for maple syrups [46]. However, it falls within the range typically found in honey [47]. The back extrusion test showed that the resistance provided by the liquid to probe penetration (surface stickiness) was lower for the 0.1% CitA Hualqui syrup than for the Crespo syrup (Table 5). The distance traveled by the probe until the liquid no longer offered resistance due to physical separation (stringiness) was also lower for the Hualqui syrup than for the Crespo syrup. This difference can be attributed to the viscosity conferred by inulin [12].

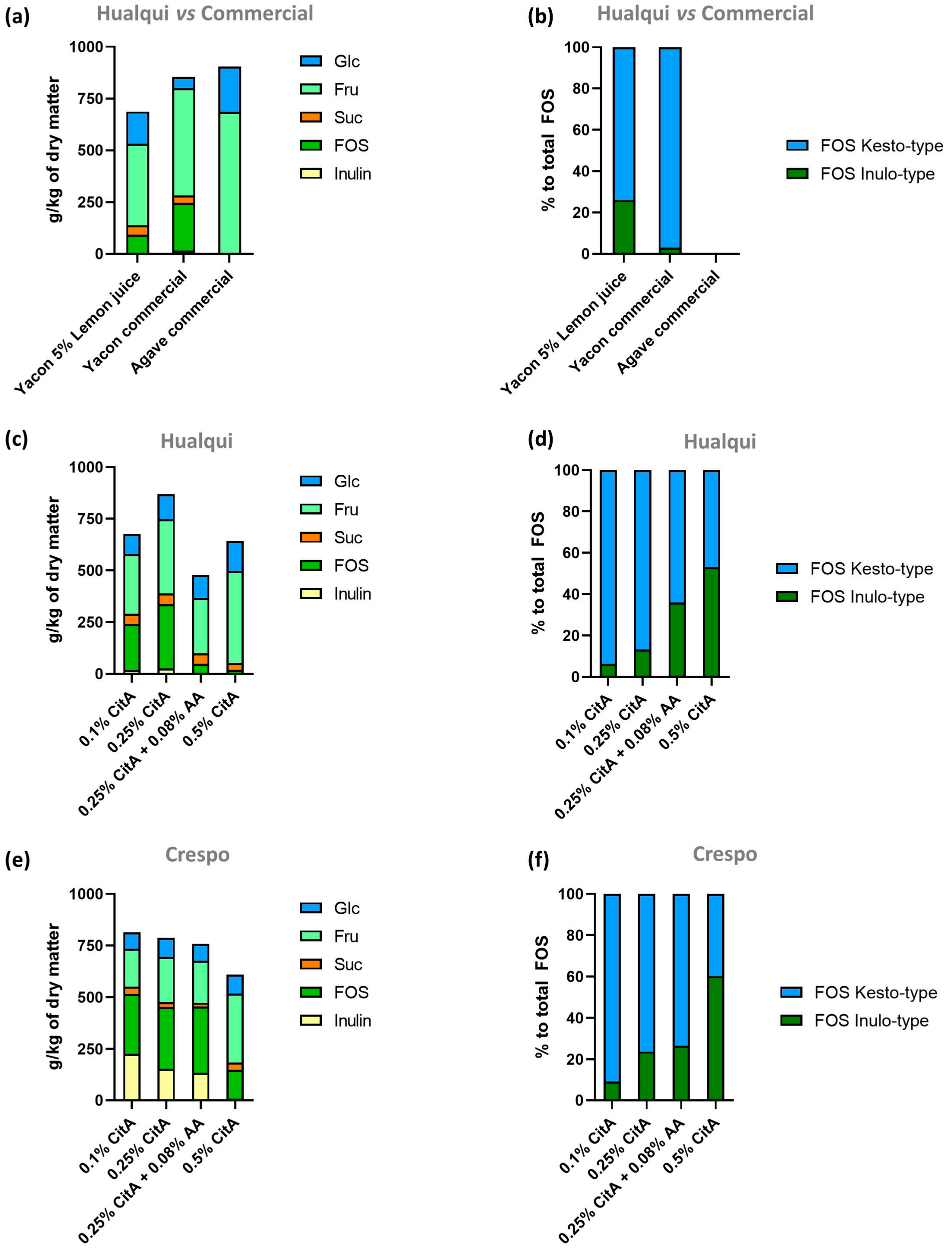

Figure 4. Composition of (**a**) Hualqui 5% lemon juice, commercial yacon syrup, and commercial agave syrup and of the (**b**) Hualqui syrups and (**c**) Crespo syrups produced with citric acid (0.1%, 0.25%, 0.25%, and 0.08% ascorbic acid and 0.5%). In (**d**–**f**) is represented the proportion of FOS of kesto-type and inulo-type for the corresponding syrup. CitA—citric acid; AA—ascorbic acid; Glc—glucose; Fru—fructose, Suc—sucrose; FOS—fructooligosaccharides.

When 0.25% CitA was introduced into yacon juice, the juice pH decreased to a range of about 4 (Table 3). The resulting syrups presented a brown color (Figure S1) and carbohydrate content of 572 g/kg of syrup and 566 g/kg of syrup for the Hualqui and Crespo syrups, respectively (Figure 4c,e and Table S3). Comparing these syrups to the 0.1% CitA syrups, it was observed that the use of 0.25% CitA led to increased Glc content by 14–23%, Fru content by 20–24%, and FOS content by 4–40%. Additionally, it resulted in an increase in the proportion of inulo-type FOS to 13–24% for both varieties (Figure 4d,f). This suggested a more extensive degradation rate of FOS and inulin than was observed with 0.1% of CitA. In fact, for the Hualqui syrup, the average DP of inulin was 14, lower than that found in the syrup at 0.1% CitA. For the Crespo syrup, the inulin content was reduced by 33%, supported by the lower recovery of polymeric material (100 g/kg, Table 4). The protein content of the yacon syrup decreased to 1.8 g/kg (Table 4), which was 33% lower than in the 0.1% syrup. Given that these syrups only differed on the amount of citric acid added during juice extraction, these differences in protein content can be attributed to protein precipitation in a highly concentrated acid medium, rendering the proteins close to their isoelectric point, at which proteins are less soluble [48]. Compared to 0.1% CitA syrup, the 0.25% CitA syrups had an increase of 10–25% in the theoretical relative sweetness, 7–17% in the relative glycemic index, and 1–24% in the caloric value (Table 3). Regarding the texture properties of 0.25% CitA Hualqui and Crespo syrups (Table 5), surface stickiness and stringiness decreased by 7% and 92%, respectively. For stringiness, this decrease was between 7% and 82%. These changes were due to the increase in the free sugars/inulin ratio.

Table 4. Yield (%), neutral sugar, and uronic acid composition of the polymeric material obtained from the dialysis of the Crespo syrups with 0.1% CitA, 0.25% CitA, and 0.5% CitA using dialysis membranes with a molecular weight cut-off of 12 kDa. CitA—citric acid; AA—ascorbic acid.

Syrup	Yield (%)	Carbohydrate Composition (Molar %)								Total (g/kg)	Protein (g/kg)
		Rha	Fuc	Ara	Xyl	Man	Gal	Glc	UA		
0.1% CitA	21	-	tr	1 ± 0	1 ± 0	32 ± 0	1 ± 0	60 ± 1	5 ± 0	510 ± 43	12 ± 2
0.25% CitA	10	-	tr	1 ± 0	2 ± 0	32 ± 2	1 ± 0	57 ± 1	7 ± 0	493 ± 28	14 ± 2
0.5% CitA	3	-	1 ± 0	2 ± 0	2 ± 0	30 ± 1	3 ± 1	53 ± 0	10 ± 2	502 ± 91	23 ± 0

Table 5. Surface stickiness (g) and stringiness (mm) of yacon syrups produced from Hualqui and Crespo-damaged roots and of commercially available yacon and agave syrups. CitA—citric acid.

Syrup	Additive	Surface Stickiness (g)	Stringiness (mm)
Yacon (Hualqui)	0.1% CitA	7.57 ± 0.25	9.54 ± 0.30
	0.25% CitA	7.01 ± 0.14	8.89 ± 0.18
	0.5% CitA	9.06 ± 0.39	10.36 ± 0.26
Yacon (Crespo)	0.1% CitA	69.21 ± 5.72	43.18 ± 3.42
	0.25% CitA	5.81 ± 0.33	7.36 ± 0.11
	0.5% CitA	4.70 ± 0.08	6.91 ± 0.07
Yacon (commercial)		8.59 ± 0.27	10.06 ± 0.26
Agave (commercial)		7.44 ± 0.08	9.14 ± 0.08

The introduction of 0.5% CitA to the yacon juice decreased the juice pH to about 3.8 (Table 3). When compared to the 0.25% CitA syrups, the 0.5% CitA Hualqui and Crespo syrups presented an orange color (Figure S1) and an increase in Glc content from 2–21%, as well as in Fru content to 24–51% (Figure 4c,e and Table S3). Model systems, which attest to the hydrolysis of FOS and inulin [49], support the conclusion that the increase in free Fru is associated with the depolymerization of short-chain FOS. This FOS decreased by 91% and 52% in the Hualqui and Crespo syrups, respectively, compared to the 0.1% CitA syrups. Furthermore, inulin almost entirely disappeared, indicating its depolymerization into FOS, which is corroborated by the lower recovery of polymeric material from the Crespo syrup (30 g/kg, Table 4). Consequently, the proportion of inulo-type FOS increased to 55–60% (Figure 4d,f). These results align with the trend observed for the syrups made with 0.25% CitA and 0.1% CitA. The lower proportion of Fru molecules in the 0.5% CitA syrups (Figure 5) also suggests the occurrence of side reactions that might influence syrup properties. These reactions may include caramelization processes that yield, among other products, difructosedianhydrides known for their reported sweet taste and prebiotic properties [50]. Additionally, the occurrence of protein, accounting for 0.75 g/kg in the syrup, can contribute to Maillard reactions [51]. One of the most relevant products generated during syrup production is hydroxymethylfurfural, a compound known to influence flavor [38,52], and likely affect perceived sweetness. In comparison to the 0.25% CitA, the 0.5% CitA syrups exhibited a higher theoretical relative sweetness and theoretical relative glycemic index while demonstrating lower surface stickiness and stringiness for the Crespo syrup.

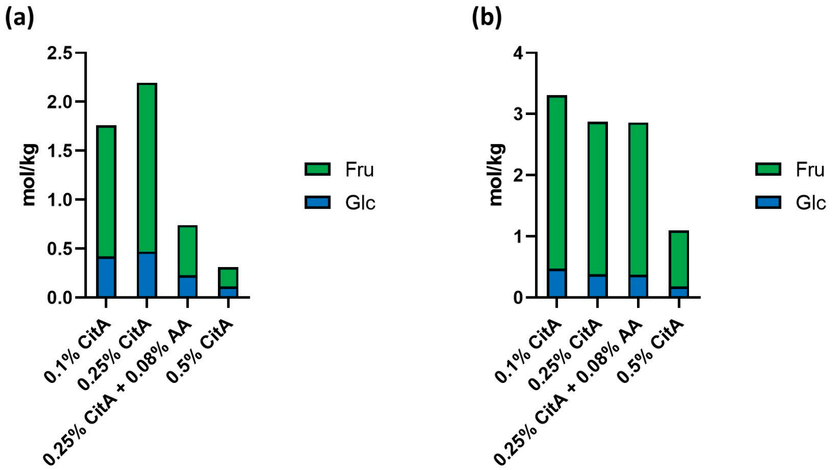

Figure 5. Proportion of total Fru and Glc units (mol/kg) present as free sugar and as part of FOS and inulin found in the syrups made from (**a**) Hualqui and (**b**) Crespo varieties. CitA—citric acid; AA—ascorbic acid; Glc—glucose; Fru—fructose.

The three syrups of the Crespo variety, produced with increasing citric acid concentrations, presented a lower theoretical glycemic index and caloric values and 52–70% of the theoretical sweetness than the commercially available yacon syrup (Yacon Portugal). This is likely due to the higher efficiency of saccharification to free sugars rather than FOS, catalyzed by endo- and exo-inulinases prior to syrup production, than by only performing thermal acid hydrolysis of FOS and inulin. These findings imply that, in an industrial setting, the production of sweet-tasting syrups with elevated FOS content should involve different processing approaches. For roots with low inulin content, processing up to 0.25% citric acid appears adequate, while roots with high inulin content may require processing up to 0.5% citric acid.

3.2.2. Effect of Ascorbic Concentration on Syrup Properties

The incorporation of 0.08% ascorbic acid alongside 0.25% CitA decreased the yacon juice pH to around 4.3, with values falling within the range achieved by using only 0.25% CitA (Table 3). The obtained syrups presented 70–75 °Brix and contained 57–78% of yacon material. The syrups produced using the Hualqui and Crespo damaged roots presented a carbohydrate content of 377 g/kg of syrup and 531 g/kg of syrup, respectively (Figure 4b,c and Table S3). In comparison to the 0.25% CitA syrup, the addition of 0.08% ascorbic acid decreased Glc content by 3–9% and Fru content by 8–21%. This effect could be attributed to the thermal degradation, occurring between 70 and 90 °C, of ascorbic acid through a non-enzymatic browning reaction with the available carbonyl groups of sugars [53]. Furthermore, the capacity of Fru to rearrange as a furanose makes it more reactive than pyranoses as Glc, in particular when in the presence of citric acid [54]. In the Hualqui syrups, a significant decrease in FOS content of 83% was observed, suggesting that oligosaccharides rich in Fru might also react with ascorbic acid. Such an effect was not as pronounced in the

Crespo syrups, possibly due to a balance between the reaction rate of FOS with ascorbic acid and their formation by inulin depolymerization. As observed in the CitA experiments, the newly formed FOS were predominantly of the inulo-type, increasing to a proportion of 27–36% compared to the initial yacon raw material (Figure 4e,f). However, when following syrup production using reduced pressure via rotary evaporation, which lowers the boiling point of water, the amount of inulo-type FOS formed only increased to 9% of a total of 263 g/kg (Table S3). Furthermore, the Glc (79 g/kg) and Fru (172 g/kg) content was lower due to a reduced rate of hydrolysis of inulin, resulting in a theoretical relative sweetness lower than the syrup made at atmospheric pressure (Table 3). However, the decrease in processing temperatures from 90 °C to 70 °C has been reported to limit ascorbic acid degradation [54], as well as the formation of bitter compounds [55] that likely mask the sweetness estimated for the syrup produced at atmospheric pressure. The syrup produced under reduced pressure exhibited a caloric value coherent with the 196–222 kcal/100 g reported for yacon syrups produced under vacuum pressures and was reported to be composed of 140 g of Glc/kg, 229 g of Fru/kg, and 307 g of FOS/kg of syrup [44,45]. Therefore, in an industrial context, the use of 0.08% ascorbic acid negatively impacts the syrup's sweetness as it reacts with sugars. In this context, ascorbic acid addition as an antioxidant should be limited to assure the production of FOS-rich syrup with high sweetening power.

4. Conclusions

The potential of yacon-damaged roots as a resource for the development of sweet-tasting syrups enriched in FOS has been demonstrated. Yacon roots with a higher content of free sugars are a resource that yields syrup with higher sweetening power. However, achieving this sweetness comes at the cost of a higher glycemic index and caloric value when compared to varieties with a higher proportion of FOS and inulin.

The use of citrous juices has demonstrated their effectiveness as a clean-label technological adjuvant for enhancing the sweetness of yacon during syrup production. As the concentration of citric acid increases, so does the extent of inulin hydrolysis to form primarily inulo-type FOS and Fru. This hydrolysis results in sweeter and more fluid syrups due to the higher sweetness and lower water retention of Fru and FOS when compared with inulin. However, the involvement of citric acid in caramelization reactions at the high temperatures used during syrup production may raise concerns regarding the final color of the syrups. Color may also be an issue of concern with ascorbic acid, which can form brown coloring compounds with unpleasant tastes as it degrades. A possible solution to mitigate these issues involves adopting emerging technologies that require lower temperatures for syrup concentration. Nevertheless, one should keep in mind that the successful utilization of yacon-damaged roots as agricultural by-products depends on the sensory and texture attributes that the derived syrups bring to low-sugar foods. The cost-to-health benefit ratio is also a significant consideration in making yacon syrups a competitive sweetener and expanding the availability of low-sugar foods. If reaching such a competitive level, environmental benefits are also expected to rise using yacon-damaged roots, usually discarded due to low market acceptability.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/app14020894/s1, Table S1. Carbohydrate composition (g/kg) of the skin, pulp and whole yacon from the Hualqui and Crespo varieties as determined by HPAEC-PAD without any hydrolysis step. Glc—glucose; Fru—fructose, Suc—sucrose; FOS—fructooligosaccharides. Table S2. Carbohydrate composition (g/kg) of Hualqui 5% lemon juice, commercial yacon syrup and commercial agave syrup as determined by HPAEC-PAD without any hydrolysis step. Table S3. Carbohydrate composition (g/kg) of Hualqui 5% lemon juice and of the Hualqui syrups and Crespo syrups produced with citric acid (0.1%, 0.25%, 0.25%, and 0.08% ascorbic acid and 0.5%) as determined by HPAEC-PAD without any hydrolysis step. Figure S1. Color of the Crespo syrups made que 0.1% citric acid, 0.25% citric acid, and 0.5% citric acid.

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Conflicts of Interest: Authors Vitor D. Alves and Adriana Silva were employed by the company Frulact. The remaining authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

References

1. Caetano, B.F.R.; De Moura, N.A.; Almeida, A.P.S.; Dias, M.C.; Sivieri, K.; Barbisan, L.F. Yacon (*Smallanthus sonchifolius*) as a Food Supplement: Health-Promoting Benefits of Fructooligosaccharides. *Nutrients* **2016**, *8*, 436. [CrossRef] [PubMed]
2. Kamp, L.; Hartung, J.; Mast, B.; Graeff-Hönninger, S. Tuber Yield Formation and Sugar Composition of Yacon Genotypes Grown in Central Europe. *Agronomy* **2019**, *9*, 301. [CrossRef]
3. Reis, F.R.; Marques, C.; Moraes, A.C.S.d.; Masson, M.L. Effect of processing methods on yacon roots health-promoting compounds and related properties. *Trends Food Sci. Technol.* **2021**, *113*, 346–354. [CrossRef]
4. Martino, H.S.D.; Kolba, N.; Tako, E. Yacon (*Smallanthus sonchifolius*) flour soluble extract improve intestinal bacterial populations, brush border membrane functionality and morphology in vivo (*Gallus gallus*). *Food Res. Int.* **2020**, *137*, 109705. [CrossRef] [PubMed]
5. Castro, A.; Caballero, M.; Herbas, A.; Carballo, S. Antioxidants in yacon products and effect of long term storage. *Food Sci. Technol.* **2012**, *32*, 432–435. [CrossRef]
6. Marques, C.; Bortolan Toazza, C.E.; Sari, R.; Mitterer-Daltoé, M.L.; Amaral, W.D.; Masson, M.L. Long-term storage of yacon (*Smallanthus sonchifolius*) juice: Phytochemical profile, in vitro prebiotic potential and discriminant bioactive properties. *Food Biosci.* **2021**, *41*, 100970. [CrossRef]
7. World Health Organization. *Taxes on Sugary Drinks: Why Do It?* World Health Organization: Geneva, Switzerland, 2017.
8. Gomes, A.; Bourbon, A.I.; Peixoto, A.R.; Silva, A.S.; Tasso, A.; Almeida, C.; Nobre, C.; Nunes, C.; Sánchez, C.; Gonçalves, D.A.; et al. Strategies for the reduction of sugar in food products. In *Food Structure Engineering and Design for Improved Nutrition, Health and Well-Being*; Cerqueira, M.Â.P.R., Pastrana Castro, L.M., Eds.; Academic Press: Massachusetts, MA, USA, 2023; pp. 219–241. [CrossRef]
9. Castro, A.; Céspedes, G.; Carballo, S.; Bergenståhl, B.; Tornberg, E. Dietary fiber, fructooligosaccharides, and physicochemical properties of homogenized aqueous suspensions of yacon (*Smallanthus sonchifolius*). *Food Res. Int.* **2013**, *50*, 392–400. [CrossRef]
10. Paredes, L.L.R.; Smiderle, F.R.; Santana-Filho, A.P.; Kimura, A.; Iacomini, M.; Sassaki, G.L. Yacon fructans (*Smallanthus sonchifolius*) extraction, characterization and activation of macrophages to phagocyte yeast cells. *Int. J. Biol. Macromol.* **2018**, *108*, 1074–1081. [CrossRef]
11. Lopez, M.G.; Mancilla-Margalli, N.A.; Mendoza-Diaz, G. Molecular Structures of Fructans from Agave tequilana Weber var. azul. *J. Agric. Food Chem.* **2003**, *51*, 7835–7840. [CrossRef]

12. Shoaib, M.; Shehzad, A.; Omar, M.; Rakha, A.; Raza, H.; Sharif, H.R.; Shakeel, A.; Ansari, A.; Niazi, S. Inulin: Properties, health benefits and food applications. *Carbohydr. Polym.* **2016**, *147*, 444–454. [CrossRef]
13. Franck, A. Technological functionality of inulin and oligofructose. *Br. J. Nutr.* **2002**, *87*, S287–S291. [CrossRef]
14. Sales, S.d.S.; Dionísio, A.P.; Adriano, L.S.; Melo, B.R.C.d.; Abreu, F.A.P.d.; Sampaio, H.A.d.C.; Silva, I.D.C.G.d.; Carioca, A.A.F. Previous gut microbiota has an effect on postprandial insulin response after intervention with yacon syrup as a source of fructooligosaccharides: A randomized, crossover, double-blind clinical trial. *Nutrition* **2023**, *109*, 111948. [CrossRef] [PubMed]
15. Choquechambi, L.A.; Callisaya, I.R.; Ramos, A.; Bosque, H.; Mújica, A.; Jacobsen, S.-E.; Sørensen, M.; Leidi, E.O. Assessing the Nutritional Value of Root and Tuber Crops from Bolivia and Peru. *Foods* **2019**, *8*, 526. [CrossRef] [PubMed]
16. Delzenne, N.M.; Roberfroid, M.R. Physiological Effects of Non-Digestible Oligosaccharides. *LWT-Food Sci. Technol.* **1994**, *27*, 1–6. [CrossRef]
17. Ferreira, S.S.; Passos, C.P.; Cardoso, S.M.; Wessel, D.F.; Coimbra, M.A. Microwave assisted dehydration of broccoli by-products and simultaneous extraction of bioactive compounds. *Food Chem.* **2018**, *246*, 386–393. [CrossRef] [PubMed]
18. Knee, M. Polysaccharides and glycoproteins of apple fruit cell walls. *Phytochemistry* **1973**, *12*, 637–653. [CrossRef]
19. Penniston, K.L.; Nakada, S.Y.; Holmes, R.P.; Assimos, D.G. Quantitative Assessment of Citric Acid in Lemon Juice, Lime Juice, and Commercially-Available Fruit Juice Products. *J. Endourol.* **2008**, *22*, 567–570. [CrossRef]
20. Rodrigues, O.R.L.; Asquieri, E.R.; Orsi, D.C. Prevention of enzymatic browning of yacon flour by the combined use of anti-browning agents and the study of its chemical composition. *Food Sci. Technol.* **2014**, *34*, 275–280. [CrossRef]
21. Manrique, I.; Parraga, A.; Hermann, M. *Yacon Syrup: Principles and Processing*; Series: Conservación y uso de la Biodiversidad de Raíces y Tubérculos Andinos: Una década de Investigación para el Desarrollo. (1993–2003); No. 8B; International Potato Center, Universidad Nacional Daniel Alcides Carrión, Erbacher Foundation, Swiss Agency for Development and Cooperation: Lima, Peru, 2005; pp. 1–31.
22. Fernandes, P.A.R.; Silva, A.M.S.; Evtuguin, D.V.; Nunes, F.M.; Wessel, D.F.; Cardoso, S.M.; Coimbra, M.A. The hydrophobic polysaccharides of apple pomace. *Carbohydr. Polym.* **2019**, *223*, 115132. [CrossRef]
23. Blumenkrantz, N.; Asboe-Hansen, G. New method for quantitative determination of uronic acids. *Anal. Biochem.* **1973**, *54*, 484–489. [CrossRef]
24. Edwards, C.H.; Rossi, M.; Corpe, C.P.; Butterworth, P.J.; Ellis, P.R. The role of sugars and sweeteners in food, diet and health: Alternatives for the future. *Trends Food Sci. Technol.* **2016**, *56*, 158–166. [CrossRef]
25. Tiefenbacher, K.F. Chapter Three—Technology of Main Ingredients—Sweeteners and Lipids. In *Wafer and Waffle*; Tiefenbacher, K.F., Ed.; Academic Press: Massachusetts, MA, USA, 2017; pp. 123–225. [CrossRef]
26. Moskowitz, H.R. Ratio scales of sugar sweetness. *Percept. Psychophys.* **1970**, *7*, 315–320. [CrossRef]
27. Atkinson, F.S.; Foster-Powell, K.; Brand-Miller, J.C. International Tables of Glycemic Index and Glycemic Load Values: 2008. *Diabetes Care* **2008**, *31*, 2281–2283. [CrossRef]
28. Goldman, F. Sugar Substitute and Bulking Agent and Chocolate. U.S. Patent US20060088637A1, 27 April 2006.
29. Godshall, M.A. The expanding world of nutritive and non-nutritive sweeteners. *Sugar J.* **2007**, *69*, 12–20.
30. European Parliament. *Regulation (EU) No. 1169/2011*; The Council of the European Union, Ed.; Official Journal European Union: Brussels, Belgium, 2011; Volume 304, pp. 18–63.
31. Graefe, S.; Hermann, M.; Manrique, I.; Golombek, S.; Buerkert, A. Effects of post-harvest treatments on the carbohydrate composition of yacon roots in the Peruvian Andes. *Field Crops Res.* **2004**, *86*, 157–165. [CrossRef]
32. Khajehei, F.; Merkt, N.; Claupein, W.; Graeff-Hoenninger, S. Yacon (*Smallanthus sonchifolius* Poepp. & Endl.) as a Novel Source of Health Promoting Compounds: Antioxidant Activity, Phytochemicals and Sugar Content in Flesh, Peel, and Whole Tubers of Seven Cultivars. *Molecules* **2018**, *23*, 278. [CrossRef]
33. Kamp, L.; Hartung, J.; Mast, B.; Graeff-Hönninger, S. Impact of Nitrogen Fertilization on Tuber Yield, Sugar Composition and Nitrogen Uptake of Two Yacon (*Smallanthus sonchifolius* Poepp. & Endl.) Genotypes. *Agronomy* **2019**, *9*, 151. [CrossRef]
34. Leroy, G.; Grongnet, J.F.; Mabeau, S.; Corre, D.L.; Baty-Julien, C. Changes in inulin and soluble sugar concentration in artichokes (*Cynara scolymus* L.) during storage. *J. Sci. Food Agric.* **2010**, *90*, 1203–1209. [CrossRef]
35. Bekers, M.; Grube, M.; Upite, D.; Kaminska, E.; Linde, R.; Scherbaka, R.; Danilevich, A. Carbohydrates in Jerusalem artichoke powder suspension. *Nutr. Food Sci.* **2007**, *37*, 42–49. [CrossRef]
36. Cabezas, M.J.; Rabert, C.; Bravo, S.; Shene, C. Inulin and Sugar Contents in *Helianthus tuberosus* and *Cichorium intybus* Tubers: Effect of Postharvest Storage Temperature. *J. Food Sci.* **2002**, *67*, 2860–2865. [CrossRef]
37. Hamdi, A.; Viera-Alcaide, I.; Guillén-Bejarano, R.; Rodríguez-Arcos, R.; Muñoz, M.J.; Monje Moreno, J.M.; Jiménez-Araujo, A. Asparagus Fructans as Emerging Prebiotics. *Foods* **2023**, *12*, 81. [CrossRef] [PubMed]
38. Waleckx, E.; Gschaedler, A.; Colonna-Ceccaldi, B.; Monsan, P. Hydrolysis of fructans from *Agave tequilana* Weber var. azul during the cooking step in a traditional tequila elaboration process. *Food Chem.* **2008**, *108*, 40–48. [CrossRef]
39. Fernandes, P.A.R.; Le Bourvellec, C.; Renard, C.M.G.C.; Nunes, F.M.; Bastos, R.; Coelho, E.; Wessel, D.F.; Coimbra, M.A.; Cardoso, S.M. Revisiting the chemistry of apple pomace polyphenols. *Food Chem.* **2019**, *294*, 9–18. [CrossRef] [PubMed]
40. Martinez, M.V.; Whitaker, J.R. The biochemistry and control of enzymatic browning. *Trends Food Sci. Technol.* **1995**, *6*, 195–200. [CrossRef]
41. Yoruk, R.; Marshall, M.R. Physicochemical properties and function of plant polyphenol oxidase: A review. *J. Food Biochem.* **2003**, *27*, 361–422. [CrossRef]

42. Ávila-Fernández, Á.; Galicia-Lagunas, N.; Rodríguez-Alegría, M.E.; Olvera, C.; López-Munguía, A. Production of functional oligosaccharides through limited acid hydrolysis of agave fructans. *Food Chem.* **2011**, *129*, 380–386. [CrossRef] [PubMed]
43. Lu, X.; Zhao, C.; Shi, H.; Liao, Y.; Xu, F.; Du, H.; Xiao, H.; Zheng, J. Nutrients and bioactives in citrus fruits: Different citrus varieties, fruit parts, and growth stages. *Crit. Rev. Food Sci. Nutr.* **2023**, *63*, 2018–2041. [CrossRef]
44. Silva, M.F.G.d.; Dionísio, A.P.; Abreu, F.A.P.; Brito, E.S.d.; Wurlitzer, N.J.; Silva, L.M.A.E.; Ribeiro, P.R.V.; Rodrigues, S.; Taniguchi, C.A.K.; Pontes, D.F. Evaluation of nutritional and chemical composition of yacon syrup using 1H NMR and UPLC-ESI-Q-TOF-MSE. *Food Chem.* **2018**, *245*, 1239–1247. [CrossRef]
45. Adriano, L.S.; Dionísio, A.P.; Pinto de Abreu, F.A.; Wurlitzer, N.J.; Cordeiro de Melo, B.R.; Ferreira Carioca, A.A.; de Carvalho Sampaio, H.A. Acute postprandial effect of yacon syrup ingestion on appetite: A double blind randomized crossover clinical trial. *Food Res. Int.* **2020**, *137*, 109648. [CrossRef]
46. Mohammed, F.; Sibley, P.; Guillaume, D.; Abdulwali, N. Chemical composition and mineralogical residence of maple syrup: A comprehensive review. *Food Chem.* **2022**, *374*, 131817. [CrossRef]
47. Seraglio, S.K.T.; Schulz, M.; Brugnerotto, P.; Silva, B.; Gonzaga, L.V.; Fett, R.; Costa, A.C.O. Quality, composition and health-protective properties of citrus honey: A review. *Food Res. Int.* **2021**, *143*, 110268. [CrossRef] [PubMed]
48. Grossmann, L.; McClements, D.J. Current insights into protein solubility: A review of its importance for alternative proteins. *Food Hydrocoll.* **2023**, *137*, 108416. [CrossRef]
49. Blecker, C.; Fougnies, C.; Van Herck, J.-C.; Chevalier, J.-P.; Paquot, M. Kinetic study of the acid hydrolysis of various oligofructose samples. *J. Agric. Food Chem.* **2002**, *50*, 1602–1607. [CrossRef] [PubMed]
50. Christian, T.J.; Manley-Harris, M.; Field, R.J.; Parker, B.A. Kinetics of Formation of Di-D-fructose Dianhydrides during Thermal Treatment of Inulin. *J. Agric. Food Chem.* **2000**, *48*, 1823–1837. [CrossRef] [PubMed]
51. Moreira, A.S.P.; Nunes, F.M.; Simões, C.; Maciel, E.; Domingues, P.; Domingues, M.R.M.; Coimbra, M.A. Transglycosylation reactions, a main mechanism of phenolics incorporation in coffee melanoidins: Inhibition by Maillard reaction. *Food Chem.* **2017**, *227*, 422–431. [CrossRef] [PubMed]
52. Mancilla-Margalli, N.A.; López, M.G. Generation of Maillard compounds from inulin during the thermal processing of *Agave tequilana* Weber var. azul. *J. Agric. Food Chem.* **2002**, *50*, 806–812. [CrossRef] [PubMed]
53. Rojas, A.M.; Gerschenson, L.N. Ascorbic Acid Destruction in Sweet Aqueous Model Systems. *LWT-Food Sci. Technol.* **1997**, *30*, 567–572. [CrossRef]
54. Rojas, A.M.; Gerschenson, L.N. Ascorbic acid destruction in aqueous model systems: An additional discussion. *J. Sci. Food Agric.* **2001**, *81*, 1433–1439. [CrossRef]
55. Dennison, D.B.; Brawley, T.G.; Hunter, G.L.K. Rapid high-performance liquid chromatographic determination of ascorbic acid and combined ascorbic acid-dehydroascorbic acid in beverages. *J. Agric. Food Chem.* **1981**, *29*, 927–929. [CrossRef]