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# Extraction of valuable compounds from granadilla (*Passiflora ligularis* Juss) peel using pressurized fluids technologies

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# ABSTRACT

Valuable products, including carotenoids, phenolic compounds, and pectin were obtained from granadilla peel using green technologies based on pressurized fluids. The processes of pressurized liquid extraction (PLE) and  $CO_2$ -expanded ethanol (GXE) were evaluated to recover the carotenoids and phenolic compounds, while pressurized hot water extraction (PHWE) was evaluated to extract pectin from the granadilla peel. The results demonstrated that although PLE and GXE had no significant difference in the carotenoids yield and antioxidant activity of the extracts obtained, PLE was faster than GXE to achieve the highest extraction yield. The pectin yield obtained by PHWE was affected by the process temperature with the highest value obtained at 160 °C. Further studies are needed to characterize the chemical composition of the pectin obtained, but the present results enable establishing a biorefinery for the complete use of granadilla wastes (seeds and peel).

# 1. Introduction

The natural extracts obtained from tropical fruit wastes contain several valuable bioactive compounds potentially beneficial to health. which can be used as food additives, such as antioxidants, antimicrobials, dyes, flavorings, etc. In this way, the use of granadilla wastes as source of new products represents an alternative to minimize the environmental impacts of waste disposal simultaneously creating an opportunity to increase incomes (de Souza et al., 2018). Granadilla is a fruit that belongs to the *Passiflora* genus, native to the Andes mountains and mainly produced in Peru (Lim, 2012). The edible parts of granadilla are mostly used for juice processing but seeds and peel are discarded as waste (Nascimento et al., 2012). Waste reduction is of especial importance for the food industry and it has been addressed in the Sustainable Development Goals (SDGs) of the United Nations as a specific target (SDG 12.3) to "by 2030, halve per capita global food waste at the retail and consumer levels and reduce food losses along the production and supply chains, including post-harvest losses" (United Nations, 2015).

It has enhanced the efforts of researchers to develop new strategies to reduce food waste by using it as a source of valuable compounds and energy following the biorefinery approach. Recently, granadilla seeds have already been explored as a source of polyun-

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*Abbreviations*: CER, Constant extraction rate period; DC, Diffusion-controlled period; FER, Falling extraction rate period; GXE, CO<sub>2</sub>-expanded ethanol; GXL, Gasexpanded liquids; GRAS, Generally recognized as safe; PHWE, Pressurized hot water extraction; PLE, Pressurized liquid extraction; SWE, Subcritical water extraction; SFE, Supercritical fluid extraction; SDG, Sustainable development goal; TPC, Total phenolic compounds.

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saturated fatty acids and phenolic compounds, which were successfully obtained by supercritical fluid extraction (SFE) and pressurized liquid extraction (PLE) (Vardanega et al., 2023). However, the use of granadilla peel remains a challenge for the establishment of the granadilla biorefinery, since no reports have been found about the recovery of bioactive compounds from granadilla peel, although it could be a source of carotenoids and pectin, as it has been reported for the use of passion fruit wastes (Viganó et al., 2016).

Apart from obtaining these valuable components using conventional techniques, recent works have demonstrated the potential of green technologies to obtain carotenoids (Tiwari et al., 2022) and pectin (Adetunji et al., 2017) from food wastes. Among them, extraction with pressurized fluids using ethanol, water, and CO<sub>2</sub> as green solvents is being successfully used for this purpose (Rodríguez-Pérez et al., 2016). PLE uses solvents at elevated pressure to maintain them in the liquid state, thus enhancing the solubility and mass transfer properties, in addition to reducing the extraction time and amount of solvent required (Herrero et al., 2015). When the solvent used is pure water, this technique is so-called subcritical water extraction (SWE) or pressurized hot water extraction (PHWE). In recent years, gas-expanded liquids (GXL) have been proposed as potential green solvents for isolating valuable components, in addition to be considered a prominent media for performing chemical reactions (Al-Hamimi et al., 2016). In extraction processes, GXL uses a liquid solvent acting as the primary solvent, and a compressible gas (e.g., CO<sub>2</sub>) to assist in the extraction, resulting in a solvent with greater extraction power, even at lower temperatures (Herrero et al., 2017). The main solvent used in GXL is ethanol (GXE) due to its GRAS (Generally Recognized As Safe) label (Poletto et al., 2021). By adding compressed CO<sub>2</sub> to ethanol, the bulk liquid expands and the systematic viscosity declines, leading to an enhancement of diffusion mainly controlled by steric effects (Jessop and Subramaniam, 2007). Thus, the present study aimed to develop a green extraction platform utilizing pressurized fluids in order to provide a mechanism for fully valorizing granadilla peel in a biorefinery approach.

# 2. Material and methods

# 2.1. Preparation and characterization of granadilla peel

The granadilla fruits were purchased in the local market of Antofagasta, Chile, and prepared according to described by Vardanega et al. (2023). After, peels were blanched using saturated steam at 100 °C for 4 min (Jiménez et al., 2021). Afterward, the peels were dried at  $30 \pm 2$  °C in an oven with forced air ventilation for 24 h. The dried peels were ground using an analytical mill (A-11 basic Analytical, IKA, Germany) and the particle size was determined according to ASAE methodology. The proximate composition of granadilla peel was determined regarding the moisture (AOAC method n° 920.151), ash (AOAC method n° 923.03), lipids (AOAC method n° 945.16), and protein (AOAC method n° 970.22 with the conversion factor of 6.25) contents. All analyses were performed in triplicate and the carbohydrates content was calculated by difference.

# 2.2. Conventional extractions

Initially, different solvents were evaluated for the extraction of granadilla peel using the convention method of maceration at 30 °C for 20 h under agitation at 200 rpm. The solvents evaluated were ethanol, acetone, ethyl acetate, and hexane. The extracts obtained were further analyzed regarding the carotenoid content, total phenolic compounds, and antioxidant activity.

The conventional extraction of pectin was performed according to Pinheiro et al., (2008). Briefly, 3.0 g of biomass were mixed with 150 mL of water acidified with 5% citric acid and extracted under reflux for 1 h. After the extraction, the mixture was filtered, and the supernatant was centrifuged at 3000 rpm for 30 min. Finally, the supernatant was precipitated with ethanol at a proportion of 1:2 (v/v) for 1h at 4 °C, filtered, and dried at 45 °C for 24 h. The experiment was performed in triplicate.

# 2.3. Pressurized liquid extraction (PLE) and CO<sub>2</sub>-expanded ethanol (GXE)

PLE using ethanol as solvent and GXE using ethanol and  $CO_2$  at a proportion of 50% (m/m) were evaluated to recover bioactive compounds from granadilla peel. All the experiments were performed in the Spe-ed SFE Helix equipment (Applied Separations, Allentown, PA, USA). The pressure was maintained at 10 MPa for all experiments and the effect of temperature (40 and 60 °C) was evaluated for both PLE and GXE. The extraction time was fixed at 30 min and the experiments were performed in duplicate. The amount of biomass and specific flow rates used for each process are presented in Table 1. After the extractions, 4 mL aliquots of extract were dried in an oven at 90 °C overnight to determine the extraction yield and the extract was stored at -18 °C until analysis.

# 2.4. Pressurized hot water extraction (PHWE)

Pectin extraction by PHWE was performed using the same Spe-ed SFE Helix equipment (Applied Separations, Allentown, PA, USA). The effect of temperature (120, 140 and 160 °C) was evaluated at a fixed pressure of 10 MPa. For each assay, 3.0 of biomass were packed in the 24-mL extraction vessel and the empty space was filled with glass beads of 3.0 mm. The water was pre-heated at

Parameter	PLE	GXE
Biomass (g)	1.0	2.5
CO2 proportion (%)	0	50
Ethanol flow rate (g/min)	1.58	3.94
CO2 flow rate (g/min)	0	3.62
S/F	47.40	90.72

# Experimental parameters used for the PLE and GXE processes.

Table 1

80 °C before entering the extraction vessel at a flow rate of 1 mL/min for 60 min. At the exit, the temperature of the mixture containing the extract + water was decreased in a condenser cold by fluid recirculation at -5 °C. The experiments were performed in duplicate. After the extraction, the pectin was precipitated with ethanol at a proportion of 1:2 (v/v) for 1h at 4 °C. The precipitated pectin was filtered and dried at 45 °C for 24 h. The pectin extraction yield was determined as the ratio between the mass of dried pectin and the mass of biomass used for the PHWE (Dias et al., 2020).

# 2.5. Extracts characterization

# 2.5.1. Carotenoids content

The carotenoids content of the extracts obtained by conventional extraction, GXE and PLE was determined by HPLC (Merck Hitachi LaChrom 7100, Tokyo, Japan) according to the method described by Jimenéz et al., (2021) using a RP-18 column (150 mm × 4.6 mm, 5  $\mu$ m). The mobile phase was water:acetone (1:1, v/v) at isocratic flowrate of 1 mL/min. The wavelength was 450 nm and the injection volume was 20  $\mu$ L. Prior to the analysis, 4 mL of extracts were dried at 40 °C with N<sub>2</sub>, resuspended in 2 mL of methanol, and filtered using PTFE filters 13 mm, 0.22  $\mu$ m. Calibration curves of β-carotene and lutein were used to identify and quantify the compounds in the extracts.

# 2.5.2. Total phenolics compounds (TPC)

The TPC of the extracts was quantified by the Folin-Ciocalteau method using 96-multiwell plates (Jiménez et al., 2021). Aliquots of 20  $\mu$ L of the extracts and 100  $\mu$ L of 10% Folin-Ciocalteau reagent were added to the microplate. After 5 min, 75  $\mu$ L of 700 mM sodium carbonate were added and the reaction was incubated in darkness for 60 min. After the incubation, the absorbance was measured at 765 nm in microplates reader (Biotek Synergy HTX multi-mode reader, software Gen 5 2.0, Winooski, VT, USA). The calibration curve to quantify TPC was obtained with gallic acid at concentrations in the range of 50 – 500  $\mu$ g/mL. The TPC results were expressed as mg acid gallic equivalent per g of biomass (mg GAE/g biomass).

# 2.5.3. In vitro ABTS antioxidant activity

The ABTS scavenging activity assay was performed according to the method described by Re et al., (1999), with some modifications. The ABTS<sup>•+</sup> radical cation was produced by the reaction of 7 mM ABTS and 2.45 mM of potassium persulfate in darkness at room temperature for 16 h. The aqueous solution of ABTS was diluted with 5 mM sodium phosphate buffer at pH 7.4 and measured in a microplate reader (Biotek Sinergy, HTX multimode reader, software Gen 5 2.0, Winooski, VT, USA) at 734 nm to an absorbance of  $0.7 \pm 0.2$ . Subsequently, aliquots of 20 µL of the extracts and 180 µL of ABTS<sup>•+</sup> solution were added to a 96-multiwell plates and measured in the microplate reader at 734 nm. Trolox in concentrations of 5–60 µg/mL was used as a reference antioxidant and the results were expressed as mg Trolox equivalent per g of biomass (mg Trolox/g biomass).

# 2.6. Statistical analysis

Analyses of variance (ANOVA) and Tukey's tests at a significant level of 5% (p-value < 0.05) were performed using Minitab 20® (Minitab Inc., State College, PA, USA) to evaluate the effect of process parameters on the extraction yield, chemical composition, and antioxidant activity of the extracts.

# 3. Results and discussion

# 3.1. Proximate composition and conventional extraction

The proximate composition of granadilla peel presented in Table 2 can be considered a novelty since it was not found in the current literature. Nonetheless, the proximate composition of peels from other fruits that belong to the genus *Passiflora* is already known, such as the passion fruit (*Passiflora edulis* sp.) (Klinchongkon et al., 2017; Viganó et al., 2016). It can be seen in Table 2 that the proximate composition of passion fruit peel was comparable to the granadilla peel, with a high content of carbohydrates, commonly observed in fruit peels from  $32.16 \pm 1.22$  to  $63.80 \pm 0.16\%$  (Romelle et al., 2016). Most of the carbohydrates of fruit peels are constituted of dietary fibers which are rich in pectin with potential industrial application due to its hydration and gelling properties (Schalow et al., 2018). In Table 2 it can also be seen that the protein content in granadilla peel is more than double that reported for passion fruit peel (Klinchongkon et al., 2017).

# Table 2

Proximate composition of granadilla (Passiflora ligularis Juss) peel and its comparison with passion fruit (Passiflora edulis) peel.

Macronutrient	Content (%)		
	Granadilla (Passiflora ligularis Juss)	Passion fruit (Passiflora edulis) <sup>a</sup>	
Moisture	$7.91 \pm 0.04$	9.43 ± 0.05	
Lipids	$0.54 \pm 0.05$	$0.36 \pm 0.04$	
Protein	8.59 ± 0.04	$3.9 \pm 0.20$	
Ash	$5.5 \pm 0.10$	$6.91 \pm 0.07$	
Carbohydrates	77.5	$79.4 \pm 0.2$	

<sup>a</sup> Klinchongkon et al., (2017).

In order to characterize the bioactive compounds that can be obtained from granadilla peel, conventional extractions were conducted using different extracting solvents. The results of carotenoids content, total phenolic compounds, and antioxidant activity are presented in Table 3. Although the solvent acetone resulted in the extract with the highest carotenoids content, the ethanolic extract presented the highest TPC content. Taking these results into account in addition to the fact that ethanol is recognized as a GRAS (Generally Recognized As Safe) solvent, this was selected for further studies.

# 3.2. CO<sub>2</sub>-expanded ethanol (GXE) and pressurized liquid extraction (PLE)

#### 3.2.1. Extraction yield

The extraction yield of granadilla peel obtained by GXE and PLE at different temperatures is presented in Table 4. The ANOVA results are presented in the Supplementary material (Table S1). The temperature increases significantly (*p*-value = 0.007) favored the extraction yield for both methods, since the increment in temperature helps the diffusion and solubility of the compounds, thus increasing the extraction rate. On the other hand, the presence of 50% of  $CO_2$  in the GXE decreased the extraction yield. Similar results were observed for the GXE of *Moringa oleifera* leaves using different  $CO_2$  concentrations, where it was observed that by increasing the  $CO_2$  concentration the extraction yield was decreased due to the reduction of the polarity of the solvent mixture (Rodríguez-Pérez et al., 2016). Inversely, for the extraction of garlic husk, the GXE process resulted in a higher extraction yield in comparison with pressurized ethanol, which was attributed to the reduction of surface tension and viscosity of ethanol caused by the addition of  $CO_2$ , thus increasing the diffusivity of the solvent into vegetable material (Chhouk et al., 2017).

# 3.2.2. Extraction of carotenoids

The main carotenoids obtained from the granadilla peel were  $\beta$ -carotene and lutein, which were also the main carotenoids obtained from the acerola by-products using GXE (Poletto et al., 2021). The  $\beta$ -carotene was in the range of  $0.42 \pm 0.04 - 0.49 \pm 0.03$  mg/g granadilla peel and lutein in the range of  $0.50 \pm 0.001 - 0.067 \pm 0.001$  mg/g granadilla peel (Table 5). The process parameters had no significant effect (*p*-value > 0.05) on the total carotenoids yield, and the highest value ( $0.54 \pm 0.03$  mg/g granadilla peel) was obtained by PLE at 60 °C. Assuming the total carotenoids obtained by conventional extraction using acetone as solvent as the total amount of extractable carotenoids of granadilla peels (Table 3), this value obtained by PLE represents a carotenoids recovery of 102.3%. By comparing PLE and conventional extraction using ethanol as solvent, the carotenoids yield was 35% higher for PLE confirming the potential of the green PLE process to recover carotenoids in a shorter time (30 min) than the conventional process that required 20 h of extraction. The positive effect of PLE can be attributed to the high pressure used that favors the penetration of the solvent into the matrix thus increasing the recovery of target compounds.

The extraction of carotenoids from passion fruit peel by ultrasound-assisted extraction using olive oil as solvent yielded 0.01 mg carotenoids/g of passion fruit peel (Chutia and Mahanta, 2021), similarly to the carotenoids yield obtained from the passion fruit bagasse by supercritical fluid extraction (0.01 mg/g bagasse) (Viganó et al., 2016). On the other hand, the extraction of carotenoids

#### Table 3

Carotenoids, total phenolic compounds (TPC) and antioxidant activity of the extracts obtained from granadilla peel by conventional extraction.

Solvent	Polarity Snyder (Snyder, 1974)	Carotenoids (mg/g biomass)	TPC (mg GAE/g biomass)	ABTS (mg Trolox/g biomass)
<i>n</i> -hexane	0.0	$0.11 \pm 0.01$	$0.008 \pm 0.001$	a
Ethyl acetate	4.3	$0.25 \pm 0.04$	$0.03 \pm 0.01$	a
Ethanol	5.2	$0.40 \pm 0.04$	$0.13 \pm 0.02$	$2.8 \pm 0.2$
Acetone	5.4	$0.53 \pm 0.04$	a	a

<sup>a</sup> Samples that presented a precipitated layer in the microplate resulting unreliable reading.

#### Table 4

Extraction yield of the extracts obtained from granadilla peel by GXE and PLE.

	Extraction yield (%)	
	40 °C	60 °C
GXE PLE	$0.83 \pm 0.10$ $1.14 \pm 0.09$	$1.55 \pm 0.06$ $1.99 \pm 0.11$

# Table 5

Process	Temperature (°C)	β-carotene	Lutein	Total carotenoids
GXE	40	0.48 ± 0.05	$0.046 \pm 0.001$	$0.52 \pm 0.03$
	60	$0.42 \pm 0.04$	$0.067 \pm 0.001$	$0.49 \pm 0.03$
PLE	40	$0.48 \pm 0.09$	$0.051 \pm 0.004$	$0.5 \pm 0.1$
	60	$0.49 \pm 0.03$	$0.050 \pm 0.001$	$0.54 \pm 0.03$

<sup>a</sup> The process parameters had no significant effect (p-value >0.05) on the total carotenoids yield evaluated by Tukey's test.

from acerola by-products obtained by GXE resulted up to 3.17 mg/g of biomass (Poletto et al., 2021). These results demonstrate that the granadilla peel can be considered a source of carotenoids among the other by-products that have been explored for this purpose.

## 3.2.3. Total phenolic compounds and antioxidant activity

The TPC and antioxidant activity of the extracts obtained from granadilla peel are presented in Table 6. The TPC was significantly affected by the extraction process (p-value = 0.002) and temperature (p-value = 0.045). The TPC results obtained by GXE were higher than those obtained by PLE, but the antioxidant activity presented the inverse behavior. It indicates that the antioxidant activity of the extracts from granadilla peel is not correlated to the presence of TPC, as observed for the extracts obtained from passion fruit peel by PLE that presented a high correlation between TPC and antioxidant activity (Viganó et al., 2016). The antioxidant activity of the extracts obtained from granadilla peel can be attributed to the presence of carotenoids (Poletto et al., 2021).

The TPC content of the extracts from granadilla peel obtained by the pressurized methods was higher than those obtained by conventional extraction. As conventional extractions were conducted at 30 °C, the higher values observed for PLE and GXE can be attributed to the effects of both pressure and temperature, since these processes were performed at higher temperatures (40 and 60 °C) as well. Viganó et al., (2016) observed the same behavior for the extraction of passion fruit peel and the TPC values reported are comparable with those observed in the present study for granadilla peel extracts. The main phenolic compounds found in passion fruit peel extracts were isoorientin, vicenin, orientin, isovitexin, and vitexin (Viganó et al., 2016), which may can be also expected to be found in granadilla peel extracts, since the both belong to same genus *Passiflora*. Nonetheless, the TPC values observed for the extracts obtained from the granadilla seeds (96  $\pm$  8 mg GAE/g biomass) (Vardanega et al., 2023) were more than 30 times higher than those observed for the granadilla peel.

# 3.2.4. GXE and PLE kinetics

Table 6

Considering that there was no significant difference on the carotenoids yield, the overall extraction curves were performed for both GXE and PLE at the lowest temperature of 40 °C and 10 MPa and the curves were adjusted to a spline model (Fig. 1). A typical extraction curve has two or three time periods according to how quickly the solute migrates into the solvent. The first thing that happens is a constant extraction rate period (CER), in which the easily accessible solute is extracted from the particles' surfaces or near the surface, in which the mass transfer is controlled by convection mechanisms. After this, the falling extraction rate period (FER) follows, with some of the easily accessible solutes already being extracted and mass transfer now controlled by diffusion. Finally, there is the diffusion-controlled (DC) rate period, when the easily extractable solutes have been exhausted, and the mass transfer is controlled by the diffusion of solvent inside the particles, leading to the diffusion of solute + solvent to the surface.

At the end of the extraction period, the extraction yield was similar for the processes, however, the trajectories described by the GXE and PLE processes differed substantially (Fig. 1). PLE presented a faster extraction rate, which can be associated with the higher solvating power of ethanol in comparison with the mixture containing 50% of  $CO_2$  used in GXE. Rodríguez-Perez et al., (2016) observed the same tendency for the downstream processing of *Moringa oleifera* leaves using SFE, GXE, and PHWE, where the highest extraction rate was observed for PWHE, followed by GXE, and SFE, respectively.

Process	Temperature (°C)	TPC (mg GAE/g biomass)	ABTS (mg Trolox/g biomass)	
GXE	40	$2.65 \pm 0.01$	$1.9 \pm 0.2$	
	60	$3.0 \pm 0.3$	$2.9 \pm 0.2$	
PLE	40	$1.5 \pm 0.1$	$3.3 \pm 0.5$	
	60	$2.01 \pm 0.07$	$3.6~\pm~0.1$	

Total phenolic compounds (TPC) and antioxidant activity (ABTS) of the extracts obtained from granadilla peel.



Fig. 1. Overall extraction curves (OECs) of granadilla peels obtained by PLE and GXE at 10 MPa and 40 °C and the predicted data by spline linear model.

According to the PLE overall extraction curve (Figs. 1), 96.2% of the total extractable material was recovered within 13.2 min corresponding to the CER period (Table 7). As a result, the extraction rate ( $M_{ext}$ ) of the CER period was 1.2 mg/min, and the solubility of the extract in ethanol ( $Y_{EtCH \ 100\%}$ ) was 7.8 × 10<sup>-4</sup> mg/g ethanol. The fast removal of the extractable material during the CER period, lead to the absence of the FER period for the PLE process, passing directly to the DC stage, abruptly lowering the  $M_{ext}$  of the DC period to 0.013 mg/min.

For the GXE extraction curve, the FER period was observed (Fig. 1), but in this case, the extraction can be considered less efficient. The first period (CER) extracted only 33.82% of the total extractable material at a rate of 3.3 mg/min in 4.12 min (Table 7). Then, the second period (FER) extracted 24.91% of the total extractable material at a lower extraction rate (0.68 mg/min). Finally, in the diffusion DC period, the last 41.27% of the extractable material was recovered at an extraction rate of 0.4 mg/min.

# 3.3. Pectin extraction by PHWE

Considering the high content of carbohydrates observed for the granadilla peel (Table 2), the extraction of pectin could also be an alternative to improve the usage of this biomass following the biorefinery concept. Thus, a preliminary study of PHWE at different temperatures was performed to recover pectin from granadilla peel. Information about pectin from granadilla peel is limited in the literature and further work is necessary to evaluate its chemical composition and properties, however, the pectin yield calculated after ethanolic precipitation according to described by Dias et al., (2020) demonstrated that granadilla peel is a potential source of pectin and PHWE is a sustainable alternative to extract it. The temperature increases had a significantly positive effect (*p*-value = 0.004) on the yield, increasing from 5.3  $\pm$  0.1% at 120 °C to 8.3  $\pm$  0.4% at 160 °C (Table 8). These values are approximately 50% of pectin yield reported for grapefruit (*Citrus grandis* L. Osbeck) peels under PHWE conditions of 120°C and 3 MPa (Liew et al., 2018). Furthermore, it has been determined that the optimal PHWE conditions for achieving maximum pectin yield (5.70%) in gabiroba fruits (*Campomanesia xanthocarpa* Berg) were 15 MPa, 120 °C, and flow rate of 1.5 mL/min (Dias et al., 2020).

The positive effect of increasing temperature on the pectin yield was also observed by Ueno et al. (2008), for the PHWE of Yuzu lemon (*Citrus junos*) until the temperature of 160 °C, which was attributed to the decreased dielectric constant of water that was responsible for increasing the solubility of pectin in water. In another study, the pectin yield from sugar beet pulp obtained by PLE was increased from 80 to 120 °C, and then decreased for temperatures above 120 °C (Chen et al., 2015). This gradual reduction of pectin yield at high temperatures can be attributed to thermal degradation of pectin that may begin after the time needed for its complete dissolution in the solvent has been exceeded (Adetunji et al., 2017). It means that the extraction time can also affect the pectin yield significantly as well. Chen et al., (2015) observed that pectin yield was increased up to 30 min of extraction, while decreasing sharply for higher extraction times, demonstrating that pectin dissolution required a certain time, but thermal degradation may occur at long extraction times. The combination of high temperatures and long extraction times used in PWHE can explain the lower pectin yields observed in comparison with that obtained by conventional extraction (17  $\pm$  1%).

In addition to the pectin yield, the extraction conditions also affect the pectin properties. It has been reported that pectin obtained from different sources using PHWE resulted in pectin with degree of esterification lower than 50% (Liew et al., 2018; Ueno et al., 2008), characterized as low methoxyl pectin, which enables the formation of stable gels in absence of sugar and is less sensitive to pH alterations than high methoxyl pectin, forming gels in the range of pH 2.6 – 6.0 (Munhoz et al., 2010). Thus, low methoxyl pectin is often used to produce low sugar or sugar free jellies (Liew et al., 2018) in addition to be used as soluble dietetic fiber and stabilizer in food emulsions (Munhoz et al., 2010).

#### Table 7

Adjusted parameters of the spline linear model from granadilla peels for PLE and GXE at 10 MPa and 40 °C.

Parameters	Stages of the General Extraction Curve				
	PLE		GXE		
	CER	DC	CER	FER	DC
Time (min.)	13.2	120.0	4.12	20.0	60.0
Accumulated extract (%)	1.61	1.67	0.54	0.93	1.59
Recovery (%)	96.2	3.8	33.82	24.91	41.27
M <sub>ext</sub> (g/min)	$1.2  imes 10^{-3}$	$1.3 \times 10^{-5}$	$3.3  imes 10^{-3}$	$6.8 \times 10^{-4}$	$4.0 \times 10^{-4}$
Y (g extract/g EtOH 100%)	$7.8 \times 10^{-4}$	$6.9 \times 10^{-4}$	-	-	-
Y (g extract/g EtOH <sub>50%</sub> )	-	-	$7.5 \times 10^{-4}$	$5.5 \times 10^{-4}$	$8.8  imes 10^{-5}$
R <sup>2</sup>	0.8416	1.0000	0.9285	1.0000	1.0000

#### Table 8

Pectin yield obtained from granadilla peel by PHWE at a fixed pressure of 10 MPa.

Temperature (°C)	Pectin yield (%)
120	$5.3 \pm 0.1 {}^{\rm b}$
140	$5.7 \pm 0.3$ b
160	$8.3 \pm 0.4$ <sup>a</sup>

Different letters in the same column indicate statistically significant difference at a confidence level of 95%.

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Integrating the results obtained in the present study with those previously reported for the granadilla seeds (Vardanega et al., 2023), a biorefining processing for the granadilla waste can be proposed. The biorefinery concept presumes the sustainable processing of raw materials resulting in different marketable products. The application of this concept by obtaining valuable compounds from food wastes using green technologies, such as those used in the present work is totally aligned with the achievement of SDGs (Vardanega et al., 2022).

# 4. Conclusions

Different extraction methods were carried out to obtain valuable compounds from granadilla peel aiming at stablishing a possible route for the biorefining of the granadilla by-products. The results demonstrated that although PLE and GXE had no significant difference in the carotenoids yield and antioxidant activity of the extracts obtained, PLE required a shorter extraction time to achieve the highest extraction yield. In addition, it was observed that the pectin yield obtained by PHWE was affected by the process temperature, however, further chemical characterization is required to evaluate the effect of the process conditions on the physicochemical properties of the pectin.

# **CRediT** author statement

Renata Vardanega: Conceptualization, Methodology, Formal analysis, Investigation, Writing – Original draft, Review & Editing, Visualization. Francisca Salinas Fuentes: Investigation. Jenifer Palma: Investigation. Waldo Bugueño-Miñoz: Investigation. Pedro Cerezal-Mezquita: Conceptualization, Resources, Supervision, Project administration, Funding acquisition. Mari Carmen Ruiz-Dominguéz: Conceptualization, Resources, Supervision, Project administration, Funding acquisition.

# 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.

# Data availability

Data will be made available on request.

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# Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.scp.2023.101135.

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