

# Centrifugal Cryoconcentration of Prickly Pear Juice: Effect on the Polyphenolic Content and their Antioxidant Activity

Carlos Alberto Márquez-Montes <sup>1</sup>, José Alberto Gallegos-Infante <sup>1,\*</sup>, Guillermo Rodrigo Petzold-Maldonado <sup>2</sup>, Patricio Antonio Orellana-Palma <sup>3</sup>, Rubén Francisco González-Laredo <sup>1</sup>, Nuria Elizabeth Rocha-Guzmán <sup>1</sup>, Luz Araceli Ochoa-Martínez <sup>1</sup>

<sup>1</sup> TecNM/Durango Institute of Technology, Department of Chemical and Biochemical Engineering, Blvd. Felipe Pescador 1830 Ote., Col Nueva Vizcaya, 34080, Durango, Dgo., México; [agallegos@itdurango.edu.mx](mailto:agallegos@itdurango.edu.mx) (J.A.G.-I.); [camm@itdurango.edu.mx](mailto:camm@itdurango.edu.mx) (C.A.M.-M.); [rfgl@itdurango.edu.mx](mailto:rfgl@itdurango.edu.mx) (R.F.G.-L.); [nerg@itdurango.edu.mx](mailto:nerg@itdurango.edu.mx) (N.E.R.-G.);

<sup>2</sup> University of Bío-Bío, Faculty of Health and Food Sciences, Department of Food Engineering, Campus Fernando May, Av. Andrés Bello 720, Chillán, Region del Ñuble, Chile; [gpetzold@ubiobio.cl](mailto:gpetzold@ubiobio.cl) (G.R.P.-M.);

<sup>3</sup> University of La Serena, Faculty of Engineering, Food Engineering Department, Av. Raúl Bitrán 1305, La Serena, Chile; [patricio.orellanap@userena.cl](mailto:patricio.orellanap@userena.cl) (P.A.O.-P.);

\* Correspondence: [agallegos@itdurango.edu.mx](mailto:agallegos@itdurango.edu.mx) (J.A.G.-I.);

Scopus Author ID 6602935659

Received: 29.01.2022; Accepted: 4.03.2022; Published: 23.03.2022

**Abstract:** Centrifugal cryoconcentration has been shown to be effective in the desalination of seawater, concentration of proteins, and liquid foods, mainly fruit juices; in the latter, it is particularly interesting to be a non-destructive technology on various bioactive compounds, such as polyphenols, which have been found in several fruit juices such as prickly pear. An important issue is related to centrifugation's effect on the concentration efficiency of cryoconcentration. The present work's main objective was to study the Block cryoconcentration assisted by centrifugation (BCCC), their effects on the polyphenols, and antioxidant activity applied to prickly pear juice. Prickly pear juice was obtained, Bx, pH, titratable acidity, color, total reducing sugars, total phenolic content (TPC), flavonoids, FRAP, DPPH, and process parameters (efficiency ( $\eta$ ), concentration index (CI), and solute yield (Y)) were evaluated. Data obtained for physicochemical parameters showed high values of °Bx in cryoconcentrated samples, pH values decreased in treated samples, and total titratable acidity increased, total reducing sugars were higher in samples treated at the lower time of centrifugation, all experimental samples showed noticeable color changes. Higher total phenolic and flavonoid content were observed in samples treated at 15 min of centrifugation; DPPH and FRAP tests were highly correlated with TPC and flavonoid content ( $R^2 > 0.96$ ). High efficiency and concentration index were observed; however, a low value of solute yield was found in cryoconcentrated juice. The use of cryoconcentration for prickly pear juices shows good results for physicochemical parameters, preservation of bioactive, and process parameters with the exception of solute yield.

**Keywords:** centrifugation; cryoconcentration; polyphenols; prickly pear juice.

© 2022 by the authors. This article is an open-access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

## 1. Introduction

Prickly pear (*Opuntia ficus-indica* L.) has been reported as a rich source of nutrients [1]. In addition, several studies have indicated its exclusive characteristics since it grows in particular environmental conditions such as low precipitation with high or low temperatures [2].

Precisely, prickly pears belong to the Cactaceae family, commonly known as *tuna*. The fruits are cultivated in many countries such as Mexico, Italy, South Africa, Argentina, the United States, and Chile [3;4].

The fruits possess a wide genetic variability, reflected in the color diversities since it is possible to find red, violet, green and yellow prickly pear fruits [5]. Furthermore, studies have reported that they contain a high isorhamnetin and quercetin glycosides content, with significant betalains presence in purple prickly pears [6;7].

In addition, *Opuntia* spp. fruits have been added as an ingredient in diverse food products (juices, jams, concentrates, etc.). Traditionally, thermal treatment has been used to elaborate on different prickly pear beverages. However, the high bioactive compounds degradation by heat processing has been documented. Thus, the application of innovative non-thermal processing technologies has emerged as a solution to the protection and retention of various bioactive components [8].

Several investigations have described emerging non-thermal technologies to process prickly pear juice without affecting its quality characteristics. Thus, microfiltration [9;10], high-pressure processing (HPP) [5, 11], and pulsed electric fields (PEF) [12] were applied with excellent results in bioactive components, antioxidant activity, and physicochemical properties terms.

An alternative to concentrating juices rich in thermosensitive bioactive components is cryoconcentration (CC). Concretely, CC is an emerging technology used to concentrate liquids; in this phenomenon, the water is frozen (below its freezing point), and the ice crystals fraction is separated from the aqueous fraction (also called cryoconcentrate fraction) [13;14]. According to the authors, block CC (BCC) has advantages over suspension CC systems in the simplicity of equipment terms [15]. In BCC, the whole liquid food is frozen, and later, it is thawed, and the concentrated fraction is separated from the ice phase. Different external forces have been applied in BCC to improve the separation process [16], such as microwave [17], centrifugation [18], and vacuum suction [19], with promising results in the total solid soluble content concentration and thermosensitive bioactive compounds protection.

Specifically, diverse fruit juices have been concentrated by BCC. [20] produced a concentrate with high total solid soluble content from pomegranate juice. [21] reported high polyphenols retention in BCC assisted by centrifugation (BCCC) applied to blueberry juices. [22] concentrated strawberry juice by BCCC, obtaining high anthocyanins retention. [23] informed high polyphenol, anthocyanin, and flavonoid retention in pineapple juice.

Nevertheless, there are no reports on the BCCC use in prickly pear juices to our knowledge. Therefore, the objective of the present work was to study the BCCC effects on the polyphenols and antioxidant activity applied to prickly pear juice.

## 2. Materials and Methods

### 2.1. Materials.

Prickly pear fruits (without apparent damage) were acquired in a local market in Santiago (Región Metropolitana, Chile), and immediately, the fruits were transported in a refrigerated truck to Chillán (Región del Ñuble, Chile) and then were stored under refrigeration condition until use.

## 2.2. Juice preparation.

Prickly pears were washed and peeled manually, and the fruits were processed in a mechanical pulper (Tecmint, Pouso Alegre, Brazil) to obtain the pulp. It was filtered to separate any solids from the juice.

## 2.3. BCC procedure.

Prickly pear juice (45 mL) into centrifuge tubes (internal diameter  $\approx$ 22 cm was insulated with polystyrene (8 mm thickness, thermal conductivity  $K=0.035$  W/m K) in order to obtain an unidirectional frozen propagation, and later, the tubes were frozen in a static freezer at  $-20^{\circ}\text{C}$  for 12 h [24]. After the freezing process was complete, the tubes were separated under different experimental (centrifugal, RCF and time, min) conditions.

## 2.4. Physicochemical parameters.

Total soluble solids (TSS) were obtained using a digital refractometer PAL-3 (range: 0-93  $^{\circ}\text{Brix}$ , precision:  $\pm 0.1$   $^{\circ}\text{Brix}$ , Atago Inc., Tokyo, Japan). The pH of samples was analyzed using a pH-meter HI 2221 (Hanna Instruments, Woonsocket, RI, USA). Total titratable acidity (TTA) was measured by using 4 mL of sample mixed with 40 mL of degassed deionized water. The pH was adjusted to 8.2 with 0.1 M NaOH solution. The TTA was expressed as grams of citric acid per 100 mL of sample (g CA/100 mL). The maturity index was calculated by dividing the TSS by TTA. Total reducing sugars (TRS) were evaluated using the dinitrosalicylic acid (DNS) method reported by [26]. Color parameters were investigated using the CIE  $L^*a^*b^*$  scale ( $L^*$ : black-white,  $a^*$ : red-green and  $b^*$ : yellow – blue) in a Konica Minolta CR-400 (Ramsey, NJ, USA), with D65 illuminant and the  $10^{\circ}$  standard observer. The total color difference ( $\Delta E^*$ ) between cryo-concentrated samples and fresh juice was calculated according to [27].

## 2.5. Total bioactive compounds.

The total polyphenol content (TPC) of samples was estimated colorimetrically by the Folin-Ciocalteu method [28], with some modifications. Briefly, 25  $\mu\text{L}$  of the sample was mixed with 500  $\mu\text{L}$  of 10-fold diluted Folin-Ciocalteu reagent and 80  $\mu\text{L}$  of  $\text{Na}_2\text{CO}_3$  (7.5%). The mixture was kept in the dark at room temperature for 30 min (incubation), and the absorbance was measured at 750 nm. The TPC was calculated using gallic acid (GA) as a standard curve, and the results were expressed as mg GA equivalents (GAE) per liter of the sample (mg GAE/L).

Total flavonoid content (TFC) was determined by the aluminum chloride colorimetric method following the procedure reported by [29], with minor modifications. 250  $\mu\text{L}$  of the sample was mixed with 1000  $\mu\text{L}$  of distilled water and 6  $\mu\text{L}$  of  $\text{NaNO}_2$  (5%) solution. After 6 min, 12.5  $\mu\text{L}$  of  $\text{AlCl}_3$  (10%) solution, 40  $\mu\text{L}$  of NaOH (1M), and 122  $\mu\text{L}$  of distilled water were added to the solution. The solution was kept in the dark at room temperature for 5 min (incubation), and later, the absorbance was measured at 515 nm. TFC was calculated using catechin (C) as a standard curve, and the results were expressed as mg C equivalent per liter of the sample (mg CE/L).

TPC and TFC were quantified on a UV/vis spectrophotometer (T70, Oasis Scientific Inc., Greenville, USA).

### 2.6. Bioactive compound retention (BCR).

BCR is the TPC (or TFC) percentage in the  $C_s$  with respect to the fresh juice. The BCR was calculated using the following equation (Eq. (1)) [16].

$$BCR (\%) = \left(\frac{C_0}{C_c}\right) * \left(\frac{TPC_c}{TPC_0}\right) \quad (1)$$

where  $C_0$  is the TSS (°Brix) in the initial solution,  $C_c$  is the TSS (°Brix) in cryo-concentrated fraction,  $TPC_c$  is the total polyphenol (or flavonoid) content in cryo-concentrated fraction, and  $TPC_0$  is the total polyphenol (or flavonoid) content in the fresh juice.

### 2.7. Antioxidant capacity.

DPPH (2,2-diphenyl-1-picrylhydrazyl) assay was assessed based on [30] method, with minor modifications. 10  $\mu$ L of the sample was mixed with 2990  $\mu$ L of DPPH methanolic solution. The mixture was kept in the dark at room temperature for 30 min (incubation). Later, the absorbance was measured at 515 nm.

Ferric reducing antioxidant power (FRAP) assay was performed according to [31] procedure, with some modifications. Briefly, FRAP reagent was prepared with 50 mL of sodium acetate buffer (300 mM, pH 3.6), 5.0 mL of TPTZ (10 mM in hydrochloric acid (40 mM)), and 5.0 mL of  $FeCl_3 \cdot 6H_2O$  (20 mM) (10:1:1 ratio), respectively, and then, the mixed solution was incubated at 37 °C for 30 min. 150  $\mu$ L of the sample was mixed with 3000  $\mu$ L of FRAP reagent. The solution was kept in the dark at room temperature for 10 min (incubation), and the absorbance was measured at 595 nm.

For all assays, Trolox (T) was used as a standard curve, and the results were expressed as  $\mu$ M Trolox equivalents (TE) per liter of the sample ( $\mu$ M TE/L).

DPPH and FRAP were quantified on a UV/vis spectrophotometer (T70, Oasis Scientific Inc., Greenville, USA).

### 2.8. Process parameters.

Efficiency ( $\eta$ ), solute yield (Y), and concentration index (CI) were evaluated for each experimental condition, according to Equations 2, 3, and 4, respectively [32].

$$\eta (\%) = \left[ \frac{(C_c - C_i)}{C_c} \right] * 100 \quad (2)$$

where  $\eta$  is the efficiency (%),  $C_c$  and  $C_i$  are the solutes (°Brix) in cryo-concentrated and ice fractions, respectively.

$$Y (kg/kg) = \frac{m_s}{m_0} \quad (3)$$

where Y is the solute yield (kg solute per 1 kg initial),  $m_s$  is the solute mass in concentrated solution, and  $m_0$  is the initial solute mass.

$$CI = \frac{C_c}{C_0} \quad (4)$$

where CI is the concentration index, and  $C_0$  is the solute (°Brix) in the initial solution.

## 2.9. Experimental design.

A factorial design of  $2^2$  (centrifugal force (RCF) at two levels and time (min) at two levels) was used to identify the desirable separation conditions. Specifically, two independent variables and three dependent variables were selected for optimization. The independent variables were centrifugal force and time. The dependent variables were the process parameters, physicochemical parameters, bioactive compound, and antioxidant capacity—Table 1 shows in detail the factors and levels of the experimental design.

**Table 1.** Experimental design used in the experimental work.

Name	Sample	Centrifugal force (RCF)	Time (min)
FJ	Fresh juice	--	--
a	Cryoconcentrated	1860	15
b	Cryoconcentrated	2910	15
c	Cryoconcentrated	1860	20
d	Cryoconcentrated	2910	20
a*	Ice	1860	15
b*	Ice	2910	15
c*	Ice	1860	20
d*	Ice	2910	20

## 2.10. Statistical Analysis.

Data were reported as mean value  $\pm$  standard deviation. All examinations were analyzed by analyses of variance (ANOVA) combined with the Tukey test ( $\alpha = 0.5$ ) to determine the statistical differences. All data were statistically analyzed with Statistica 12.5 (Stat-Soft, Tulsa, OK, USA).

## 3. Results and Discussion

### 3.1. Physicochemical parameters.

Physicochemical parameters are presented in Table 2. Firstly, the fresh prickly pear juice has a TSS value of approximately 13 °Brix. This TSS value has been related to fruits with high water content [18], a condition that was mentioned in prickly pear fruit by [33].

In cryoconcentrate samples, all TSS values were higher than the fresh juice (13.8 °Brix), with 30.3 °Brix, 33.1 °Brix, 25.4 °Brix, and 25.3 °Brix, for the combination 1860 RCF at 15 min, 2910 RCF at 15 min, 1860 RCF at 20 min and 2910 RCF at 20 min, respectively. These values were higher than other centrifugal CC studies with fruit juices such as pineapple juice [18], blueberry juice [16], and orange juice [21], in which the first cycle presented values between 18-20 °Brix. These results could be related to fresh juice since prickly pear juice was filtered, and it avoids any interference in the CC process. Thus, when the temperature decreased, the ice/cryoconcentrate separation was performed better than the other juices since these had seeds or some solids that complicated the ice growth purity.

The TSS values were lower than the initial sample in ice fraction terms, with values between 8.5-12 °Brix. The results indicate that the CC process applied to natural fruit juice without added components and/or any previous treatment allows a significant separation in the centrifugal step since few cryoconcentrate solution remains between the ice crystals [34].

**Table 2.** Physicochemical parameters in Prickly pear juice.

	Fresh Juice	Cryo-concentrate fraction name				Ice fraction name			
		a	b	c	d	a*	b*	c*	d*
<b>TSS (°Brix)</b>	13.8 ± 0a	30.3 ± 1b	33.1 ± 1c	25.4 ± 1.8d	25.3 ± 1.6d	12 ± 0.8e	11.9 ± 0.2e	8.3 ± 0.8f	8.5 ± 0.5f
<b>pH</b>	6.2 ± 0a	5.9 ± 0.0b	6 ± 0.1ab	6.1 ± 0ab	6.2 ± 0a	6.5 ± 0c	6.5 ± 0c	6.5 ± 0c	6.5 ± 0c
<b>TTA (g CA/100 mL)</b>	0.013± 0a	0.019 ± 0b	0.019± 0b	0.016± 0c	0.017±0c	0.006 ± 0d	0.006 ± 0d	0.006± 0d	0.006± 0d
<b>Reducing sugars (g/L)</b>	49.4±0.1a	140.4±2.1b	150.1±2.6c	92±2d	90.8±2.1d	47.2±0e	47.1± 0.1e	28.9± 0.6f	30.3± 0.9f
<b>ΔE*</b>	-	4.7 ±0.1a	5.3 ± 0.4a	4.8 ± 0.1a	4.2 ± 0.3b	-	-	-	-

Data showed mean ± standard deviation. Different literals in the same row indicate statistical differences (p<0.5)

The pH of the cryo-concentrates decreased in relation to the fresh juice, and the acidity has a contrary effect due to it increasing with respect to the initial value.

Both pH and TTA values presented significant statistical differences with the fresh juice. This inverse performance has been accredited to the TSS values since as the TSS concentration increases, the organic acid content in the sample increases [20].

TRS, in all experimental conditions, indicated a high increase in the cryoconcentrate samples, which equivalent an increase up to 2.8 (1860 RCF at 15 min), 3.0 (2910 RCF at 15 min), 1.9 (1860 RCF at 20 min), and 1.8 (2910 RCF at 20 min) times compared to the initial value (≈49.4 g/L). The presence of sugars in water or ice is important because the presence of sugar molecules in the water cluster model [35], the increasing distance among water cluster model, means a structure-breaker a produce more fragile ice [36], opposite, an increase of sugars content as obtained at 15 min of centrifugation time, increase solute – solute interaction reaches a more stable cluster that induces more strong ice, this condition was named as water structure-maker by [36], resulting in higher efficiency of the cryo-concentration process.

Color difference (Table 2) shows the difference (p<0.05) at 15 min and 2910 RCF of speed. The total color difference (ΔE\*) indicates the magnitude of the color difference. Depending on the value of ΔE, the color difference between samples can be described as not noticeable (0–0.5), slightly noticeable (0.5–1.5), noticeable (1.5–3.0), well visible (3.0–6.0) [37], in all experimental conditions, well visible changes were observed, however, obtained ΔE values was lower than reported for other fruits juices as pineapple [23]. Several authors reported that color change could be attributed to the increase in solutes (°Bx), which in turn facilitates the increase of different components such as bioactive and volatile compounds [22].

### 3.2. Bioactive compounds and antioxidant activity.

Total Phenolic Content (TPC) data are shown in Table 3. Higher total polyphenol content (TPC) was observed at 15 min of centrifugation, independent of the RCF (centrifugation speed). Freeze block concentrate at any experimental condition showed a higher value of (TPC) than prickly pear fresh juice. Similar behavior has been reported for different fruit juices and herbal infusions [38]. The increase of TPC was related to the concentration effect; TPC at 15 min of centrifugation showed an increase of 1.9 – 2 times the fresh prickly pear juice values. On the other side, the concentration range of TPC for samples treated at 20 min showed a concentration increase of 1.38 – 1.47 times. However, in the present work, no differences of TPC in ice fraction were observed (p>0.05). [39] indicate that the process efficiency in cryoconcentration decreased when the retention of phenolic

compounds increased in ice fraction. These results are highly related to the phenolic content thus. They could also be related to the presence of soluble polysaccharide molecules or insoluble dietary fiber (IDF), then in prickly pear fruits, has been mainly reported as pectin [5]. Therefore, a weak gel could be formed at the moment of increasing concentration, trapping polyphenols present into the prickly pear juice and reaching higher retention values. However, this behavior could be related to the solid soluble content, as can be seen by the high determination coefficient ( $r^2 = 0.92$ ) observed by multiple correlations of the linear model (Total phenolic content vs. °Bx).

Results obtained for total flavonoid content (TFC) (Table 3) showed similar behavior to TPC, i.e., only centrifugation time on the concentrate fraction showed a significant effect ( $p < 0.05$ ). This result is interesting because [5] did not find flavonoids content in prickly pear juices from four different varieties, although several authors have already reported the presence of flavonoids in prickly pear juices [7].

The importance of bioactive has been related to their antioxidant capacity, as obtained results shown in Table 3. A higher linear correlation was found between the TPC and DPPH results ( $r^2 = 0.96$ ), similar behavior was founded for TFC and DPPH data ( $r^2 = 0.96$ ). These results indicate that the antioxidant capacity shown by prickly pear juice (fresh, cryo-concentrated, or present into the ice fraction) is related mainly to the presence of flavonoids. Also, similar behavior was observed for FRAP results with a slightly higher value of  $R^2$  (0.97 for the relationship of FRAP vs. TPC and FRAP vs. TFC). Also, it is interesting to note the high levels of total polyphenolic content present in the ice fraction and high antioxidant capacity. Therefore, more studies need to be addressed to use these residues.

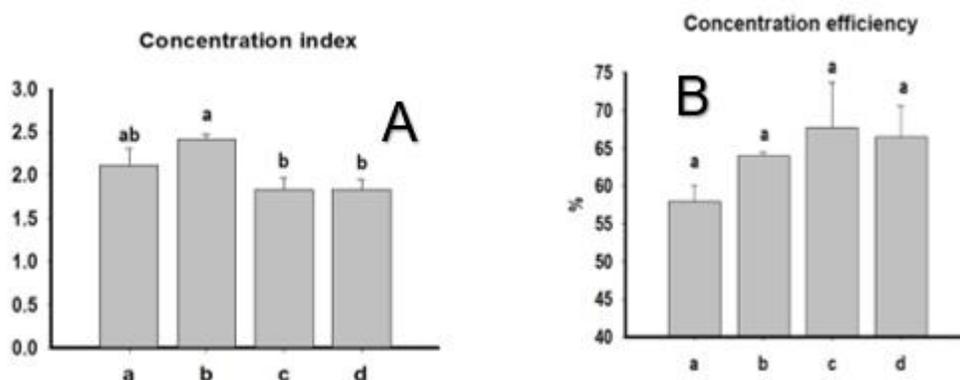
### 3.3. Process parameters.

Results of CI are shown in Figure 1A. About obtained results, [18] reported a CI of 2.5 for pineapple juice, similar to the value found in the present work. The low relative concentration of solutes found in prickly pear juice could be affected solute yield (Y), a) diffusion rate of solutes lower at high initial solute values, b) increase of viscosity limit separation of liquid at thawing stage, c) dendritic growth of crystal ice occluding higher amount of solutes in ice, and d) high concentration of solutes, a lower amount of water can be frozen and separated. solute yield (Y) was low in comparison to other cryo-concentrated fruit juices as orange juice (kg solute/kg initial) (0.4 – 0.8) [24], Blueberry juice (0.78 kg solute/kg initial), [16] and apple juice (0.79 – 0.83 kg solute/kg initial) [23].

In the block freeze concentration process, separating solutes from the concentrated phase of prickly pear juices is a consequence of using an external driving force (i.e., centrifugation). Under these conditions, the ice block acts as a porous solid through which the concentrated solution percolates by drainage channels among ice crystals in a similar fashion as reported for centrifugal freeze concentration of sucrose solutions [13]. Several authors have demonstrated that assuming a local surface porosity in a porous media (as an ice matrix in block freeze concentration) does not depend on the pore pressure itself; it depends on its difference from the hydrostatic pressure. Thus, solutes in the frozen sample were discharged with melting and with a solute diffusion from the cryo-concentrated phase. Reference [40] suggested that ice-melted concentration in a freeze-thawing process is governed by the inter-phase equilibrium and the kinetics of mass transfer during the phase

transition, increasing (Y) value on the ice fraction and diminishing the (Y) value into the concentrated phase.

On the opposite, a good value of cryo-concentration efficiency ( $\eta$ ) was observed (Figure 1B) (60 – 74%) for all conditions; similar values were reported by [18] for blueberry and pineapple juice. No influence of time and speed centrifugation were observed in the experimental range used, which could be related to a high level of solutes found in the prickly pear juice. The presence of the high efficiency and low yield of solutes has been observed for cryoconcentration of orange juice at the first cycle of the process, [24], they claimed that as the solute content is high, a large mass accumulates at the solid-liquid interface, and then extracted by centrifugation, however, if solutes are not transported to the interface, thus, solute yield is low, as can see in the present work.



**Figure 1.** (A), Concentration index; (B) concentration efficiency of cryo-concentrated prickly pear juice. Different literals indicate statistical differences ( $p < 0.05$ , Tukey test).

**Table 3.** Bioactive compounds and antioxidant activity in Prickly pear juice.

	Fresh Juice	Cryo-concentrate fraction name				Ice fraction name			
		a	b	c	d	a*	b*	c*	d*
<i>Bioactive compounds</i>									
<b>TPC (mg GAE/L)</b>	307±12c	584±53a	645±22a	451±22b	424±3b	38.4±0.2d	43±4d	42±1d	41±1d
<b>TFC (mg CE/L)</b>	89.5±1d	156±6a	163±1a	122±6b	109.±0.2c	12±1e	10±1e	9.4±0.4e	11±0.2e
<i>Antioxidant capacity</i>									
<b>DPPH (µM TE/L)</b>	252±5c	597±12b	699±19a	664±2a	554±40b	84±2d	72±0d	77±7d	91±7d
<b>FRAP (µM TE/L)</b>	277±7d	492±14b	537±11a	516±1ab	458±14c	69±4e	52±1e	60±4e	73±1e

Data showed mean ± standard deviation. Different literals in the same row indicate statistical differences ( $p < 0.5$ ).

#### 4. Conclusions

The use of cryoconcentration assisted by centrifugation for prickly pear juice show good results for physicochemical parameters ( $^{\circ}\text{Bx}$ , total titratable acidity, total reducing sugars, pH, color), preservation of bioactive (total phenolic content and flavonoids), and antioxidant capacity (DPPH and FRAP) and process parameters (Concentrate index, efficiency), however, a low solute yield was observed.

#### Funding

This research received no external funding.

#### Acknowledgments

C.A.M.M. acknowledges a scholarship grant for his M.Sc. studies and a Travel Grant to CONACyT.

## Conflicts of Interest

The authors declare no conflict of interest.

## References

1. Valero-Galván, J.; González-Fernández, R.; Sigala-Hernández, A.; Núñez-Gastélum, J. A.; Ruiz-May, E.; Rodrigo-García, J.; del Rocío Martínez-Ruiz, N. Sensory attributes, physicochemical and antioxidant characteristics, and protein profile of wild prickly pear fruits (*O. macrocentra* Engelm., *O. phaeacantha* Engelm., and *O. engelmannii* Salm-Dyck ex Engelm.) and commercial prickly pear fruits (*O. ficus-indica* (L.) Mill.). *Food Res Intl* **2021**, *140*, 109909, <https://doi.org/10.1016/j.foodres.2020.109909>.
2. El-Guezzane, C.; El-Moudden, H.; Harhar, H.; Chahboun, N.; Tabyaoui, M.; Zarrouk, A. A comparative study of the antioxidant activity of two Moroccan prickly pear cultivars collected in different regions. *Chemical Data Collections* **2021**, *31*, 100637, <https://doi.org/10.1016/j.cdc.2020.100637>.
3. Mejia, J. A. A.; Yáñez-Fernandez, J. Clarification Processes of Orange Prickly Pear Juice (*Opuntia* spp.) by Microfiltration. *Membranes* **2021**, *11*, 354 – 360, <https://doi.org/10.3390/membranes11050354>.
4. Álvarez, S. A.; Rocha-Guzmán, N. E.; Moreno-Jiménez, M. R.; Gallegos-Infante, J. A.; Pérez-Martínez, J. D.; Rosas-Flores, W. Functional fermented beverage made with apple, tibicos, and pectic polysaccharides from prickly pear (*Opuntia ficus-indica* L. Mill) peels. *J Food Process Preserv* **2021**, *45*, e15745, <https://doi.org/10.1111/jfpp.15745>.
5. Alexandre, E.; Coelho, M. C.; Ozcan, K.; Pinto, C. A.; Teixeira, J. A.; Saraiva, J. A.; Pintado, M. Emergent technologies for the extraction of antioxidants from prickly pear peel and their antimicrobial activity. *Foods* **2021**, *10*, 570, <https://doi.org/10.3390/foods10030570>.
6. Tesoriere, L.; Fazzari, M.; Allegra, M.; Livrea, M.A. Biothiols, taurine, and lipid-soluble antioxidants in the edible pulp of Sicilian cactus pear (*Opuntia ficus-indica*) fruits and changes of bioactive juice components upon industrial processing. *J Agric and Food Chem* **2005**, *53*, 7851-7855, <https://doi.org/10.1021/jf050636f>.
7. García-Cayuela, T.; Gómez-Maqueo, A.; Guajardo-Flores, D.; Welti-Chanes, J.; Cano, M.P. Characterization and quantification of individual betalain and phenolic compounds in Mexican and Spanish prickly pear (*Opuntia ficus-indica* L. Mill) tissues: A comparative study. *J Food Comp and Analysis* **2019**, *76*, 1-13, <https://doi.org/10.1016/j.jfca.2018.11.002>.
8. Casas-Forero, N.; Moreno-Osorio, L.; Orellana-Palma, P.; Petzold, G. Effects of cryoconcentrate blueberry juice incorporation on gelatin gel: A rheological, textural and bioactive properties study. *LWT- Food Sci Tech* **2021**, *138*, 110674, <https://doi.org/10.1016/j.lwt.2020.110674>.
9. Moßhammer, M.R.; Stintzing, F.C.; Carle, R. Development of a process for the production of a betalain-based colouring foodstuff from cactus pear. *Inn Food Sci Emerging Tech* **2005**, *6*, 221-231, <https://doi.org/10.1016/j.ifset.2005.02.001>.
10. Moßhammer, M.R.; Stintzing, F.C.; Carle, R. Evaluation of different methods for the production of juice concentrates and fruit powders from cactus pear. *Inn Food Sci Emerging Tech* **2006**, *7*, 275-287, <https://doi.org/10.1016/j.ifset.2006.04.003>.
11. Moussa-Ayoub, T.E.; Jäger, H.; Knorr, D.; El-Samahy, S.K.; Kroh, L.W.; Rohn, S. Impact of pulsed electric fields, high hydrostatic pressure, and thermal pasteurization on selected characteristics of *Opuntia dillenii* cactus juice. *LWT - Food Sci and Tech* **2017**, *79*, 534-542, <https://doi.org/10.1016/j.lwt.2016.10.061>.
12. Moussa-Ayoub, T.E.; Jaeger, H.; Youssef, K.; Knorr, D.; El-Samahy, S.; Kroh, L. W.; Rohn, S. Technological characteristics and selected bioactive compounds of *Opuntia dillenii* cactus fruit juice following the impact of pulsed electric field pre-treatment. *Food Chem* **2016**, *210*, 249-261, <https://doi.org/10.1016/j.foodchem.2016.04.115>.
13. Petzold, G.; Aguilera, J. M.. Centrifugal freeze concentration. *Inn Food Sci Emerging Tech* **2013**, *20*, 253-258, <https://doi.org/10.1016/j.ifset.2013.05.010>.
14. Martín, V. S.; Bastías-Montes, J. M.; Villagra-Jorquera, C.; Salinas-Huenchulao, G.; Flores-Ríos, A.; Gonzáles-Díaz, N.; Quevedo-León, R. Effect of Cryoconcentration Assisted by Centrifugation-Filtration on Bioactive Compounds and Microbiological Quality of Aqueous Maqui (*Aristotelia chilensis* (Mol.) Stuntz) and Calafate (*Berberis microphylla* G. Forst) Extracts Pretreated with High-Pressure Homogenization. *Processes* **2021**, *9*, 692, <https://doi.org/10.3390/pr9040692>.

15. Sánchez, J.; Ruiz, Y.; Auleda, J. M.; Hernández, E.; Raventós, M. Freeze concentration in the fruit juices industry. *Food Sci Tech Intl* **2009**, *15*, 303-315, <https://doi.org/10.1177/1082013209344267>.
16. da Silva Haas, I. C.; de Espindola, J. S.; de Liz, G. R.; Luna, A. S.; Bordignon-Luiz, M. T.; Prudêncio, E. S.; Fedrigo, I. M. T. Gravitational assisted three-stage block freeze concentration process for producing enriched concentrated orange juice (*Citrus sinensis* L.): Multi-elemental profiling and polyphenolic bioactives. *J Food Eng* **2022**, *315*, 110802, <https://doi.org/10.1016/j.jfoodeng.2021.110802>.
17. Aider, M.; de Halleux, D.J. Passive and microwave-assisted thawing in maple sap cryoconcentration technology. *J Food Eng* **2008**, *85*, 65-72, <https://doi.org/10.1016/j.jfoodeng.2007.07.025>.
18. Petzold, G.; Moreno, J.; Lastra, P.; Rojas, K.; Orellana, P. Block freeze concentration assisted by centrifugation applied to blueberry and pineapple juices. *Inn Food Sci Emerging Tech* **2015**, *30*, 192-197, <https://doi.org/10.1016/j.ifset.2015.03.007>.
19. Orellana-Palma, P.; Petzold, G.; Torres, N.; Aguilera, M. Elaboration of orange juice concentrate by vacuum-assisted block freeze concentration. *J Food Process Preserv* **2018**, *42*, e13438, <https://doi.org/10.1111/jfpp.13438>.
20. Orellana-Palma, P.; Guerra-Valle, M.; Gianelli, M. P.; Petzold, G. Evaluation of freeze crystallization on pomegranate juice quality in comparison with conventional thermal processing. *Food Bioscience* **2021**, *41*, 101106, <https://doi.org/10.1016/j.fbio.2021.101106>.
21. Casas-Forero, N., Orellana-Palma, P., & Petzold, G. (2021). Recovery of Solutes from Ice and Concentrated Fractions in Centrifugal Block Cryoconcentration Applied to Blueberry Juice. *Food and Bioprocess Technology*, *14*(6), 1155-1168. <https://doi.org/10.1007/s11947-021-02626-x>
22. Adorno, W. T.; Rezzadori, K.; Arend, G.D.; Chaves, V.C.; Reginatto, F. H.; Di Luccio, M.; Petrus, J.C. Enhancement of phenolic compounds content and antioxidant activity of strawberry (*Fragaria× ananassa*) juice by block freeze concentration technology. *International J Food Sci Tech* **2017**, *52*, 781-787, <https://doi.org/10.1111/ijfs.13335>.
23. Orellana-Palma, P.; Zúñiga, R.N.; Takhar, P.S.; Gianelli, M.P.; Petzold, G. Effects of centrifugal block freeze crystallization on quality properties in pineapple juice. *Chem Eng Tech* **2020**, *43*, 355-364, <https://doi.org/10.1002/ceat.201900387>.
24. Orellana-Palma, P.; González, Y.; Petzold, G. Improvement of centrifugal cryoconcentration by ice recovery applied to orange juice. *Chem Eng Tech* **2019**, *42*, 925-931, <https://doi.org/10.1002/ceat.201800639>.
25. Cruz-Cansino del Socorro, N.; Ramírez-Moreno, E.; León-Rivera, J.E.; Delgado-Olivares, L.; Alanís-García, E.; Ariza-Ortega, J.A.; Manríquez-Torres, J.J.; Jaramillo-Bustos, D.P. Shelf life, physicochemical, microbiological and antioxidant properties of purple cactus pear (*Opuntia ficus indica*) juice after thermoultrasound treatment. *Ultrasonics sonochemistry* **2015**, *27*, 277-286, <https://doi.org/10.1016/j.ultsonch.2015.05.040>.
26. Miller, G. L. Use of dinitrosalicylic acid reagent for determination of reducing sugar. *Analytical chem* **1959**, *31*, 426-428, <https://doi.org/10.1021/ac60147a030>.
27. Cserhalmi, Z.; Sass-Kiss, A.; Tóth-Markus, M.; Lechner, N. Study of pulsed electric field treated citrus juices. *Inn Food Sci Emerging Tech* **2006**, *7*, 49-54, <https://doi.org/10.1016/j.ifset.2005.07.001>.
28. Velioglu, Y.S.; Mazza, G.; Gao, L.; Oomah, B.D. Antioxidant activity and total phenolics in selected fruits, vegetables, and grain products. *J Agric Food Chem* **1998**, *46*, 4113-4117, <https://doi.org/10.1021/jf9801973>.
29. Rocha-Guzmán, N.E.; Medina-Medrano, J.R.; Gallegos-Infante, J.A.; Gonzalez-Laredo, R.F.; Ramos-Gómez, M.; Reynoso-Camacho, R.; González-Herrera, S.M. Chemical evaluation, antioxidant capacity, and consumer acceptance of several oak infusions. *J Food Sci* **2012**, *77*, C162-C166, <https://doi.org/10.1111/j.1750-3841.2011.02524.x>.
30. Brand-Williams, W.; Cuvelier, M.E.; Berset, C.L.W.T. Use of a free radical method to evaluate antioxidant activity. *LWT-Food Sci Tech* **1995**, *28*, 25-30, [https://doi.org/10.1016/S0023-6438\(95\)80008-5](https://doi.org/10.1016/S0023-6438(95)80008-5).
31. Thaipong, K.; Boonprakob, U.; Crosby, K.; Cisneros-Zevallos, L.; Byrne, D.H. Comparison of ABTS, DPPH, FRAP, and ORAC assays for estimating antioxidant activity from guava fruit extracts. *J Food Comp and Analysis* **2006**, *19*, 669-675, <https://doi.org/10.1016/j.jfca.2006.01.003>.
32. Qin, F. G.; Ding, Z.; Peng, K.; Yuan, J.; Huang, S.; Jiang, R.; Shao, Y. Freeze concentration of apple juice followed by centrifugation of ice packed bed. *J Food Eng* **2021**, *291*, 110270, <https://doi.org/10.1016/j.jfoodeng.2020.110270>.
33. Andreu-Coll, L.; García-Pastor, M. E.; Valero, D.; Amorós, A.; Almansa, M. S.; Legua, P.; Hernández, F. Influence of Storage on Physiological Properties, Chemical Composition, and Bioactive Compounds on Cactus Pear Fruit (*Opuntia ficus-indica* (L.) Mill.). *Agriculture* **2021**, *11*, 62, <https://doi.org/10.3390/agriculture11010062>.

34. Bastías-Montes, J. M.; Vidal-San-Martín, C.; Tamarit-Pino, Y.; Muñoz-Fariña, O.; García-Figueroa, O.; Quevedo-León, R.; Cespedes-Acuña, C. L. Cryoconcentration by Centrifugation–Filtration: A Simultaneous, Efficient and Innovative Method to Increase Thermosensitive Bioactive Compounds of Aqueous Maqui (*Aristotelia chilensis* (Mol.) Stuntz) Extract. *Processes* **2022**, *10*, 25, <https://doi.org/10.3390/pr10010025>.
35. Moussaoui, C.; Blanco, M.; Muñoz, I. D. B.; Raventós, M.; Hernández, E. An approach to the optimization of the progressive freeze concentration of sucrose solutions in an agitated vessel. *Separation Sci Tech* **2021**, *56*, 746-756, <https://doi.org/10.1080/01496395.2018.1508231>.
36. Guerra-Valle, M.; Lillo-Perez, S.; Petzold, G.; Orellana-Palma, P. Effect of Freeze Crystallization on Quality Properties of Two Endemic Patagonian Berries Juices: Murta (*Ugni molinae*) and Arrayan (*Luma apiculata*). *Foods* **2021**, *10*, 466, <https://doi.org/10.3390/foods10020466>.
37. Vazquez-Cabral, D.; Valdez-Fragoso, A.; Rocha-Guzman, N. E.; Moreno-Jimenez, M. R.; Gonzalez-Laredo, R. F.; Morales-Martinez, P. S.; Gallegos-Infante, J. A. Effect of pulsed electric field (PEF)-treated kombucha analogues from *Quercus obtusata* infusions on bioactives and microorganisms. *Inn Food Sci Emerging Tech* **2016**, *34*, 171-179, <https://doi.org/10.1016/j.ifset.2016.01.018>.
38. Meneses, D. L.; Ruiz, Y.; Hernandez, E.; Moreno, F. L. Multi-stage block freeze-concentration of green tea (*Camellia sinensis*) extract. *J Food Eng* **2021**, *293*, 110381, <https://doi.org/10.1016/j.jfoodeng.2020.110381>.
39. Tobar-Bolaños, G.; Casas-Forero, N.; Orellana-Palma, P.; Petzold, G. Blueberry juice: Bioactive compounds, health impact, and concentration technologies—A review. *J Food Sci* **2021**, *134*, 135 – 160, <https://doi.org/10.1111/1750-3841.15944>.
40. Burdo, O.; Bezbakh, I.; Zykov, A.; Fatieieva, Y.; Pour, D. R.; Osadchuk, P.; Mazurenko, I.; Zhengzheng, S.; Phylipova, L. Development of the Design and Determination of Mode Characteristics of Block Cryoconcentrators for Pomegranate Juice. *Eastern-European J of Enterprise Tech* **2021**, *2*, 110, <https://doi.org/10.15587/1729-4061.2021.230182>.