processes

MDPI

Article

Centrifugal Filter-Assisted Block Freeze Crystallization Applied to Blueberry Juice

Patricio Orellana-Palma [1,*], María Guerra-Valle [2,3] and Rommy N. Zúñiga [1,4]

1 Departamento of Biotecnología, Universidad Tecnológica Metropolitana, Las Palmeras 3360, P.O. Box, Ñuñoa, 7800003 Santiago, Chile; rommy.zuniga@utem.cl
2 Laboratory of Cryoconcentration, Department of Food Engineering, Universidad del Bío-Bío, Av. Andrés Bello 720, 3800708 Chillán, Chile; maria.guerra1601@egresados.ubiobio.cl
3 Doctorado en Ingeniería de Alimentos, Universidad del Bío-Bío, Av. Andrés Bello 720, 3800708 Chillán, Chile
4 Programa Institucional de Fomento a la I+D+i, Universidad Tecnológica Metropolitana, Ignacio Valdivieso 2409, San Joaquín, 8940577 Santiago, Chile
* Correspondence: p.orellanap@utem.cl; Tel.: +56-2-27877032

Abstract: The impact of centrifugal-filter assisted block freeze crystallization (CFBFC) on the physicochemical parameters, total phenolic content (TPC), total anthocyanin content (TAC), and total flavonoid content (TFC), antioxidant activity (AA) and process parameters applied to blueberry juice was studied. Additionally, CFBFC was contrasted with gravitational BFC (GBFC) and centrifugal BFC (CBFC) techniques. For CFBFC process, the solutes values were ≈35.9 °Brix (fresh juice ≈13.8 °Brix), with a very dark red/purple color. Moreover, the bioactive components values presented a significant increase of 2.1, 2.0, 1.8, and 3.1 times compared to the initial TPC, TAC, TFC, and AA values, respectively, and these values were higher than GBFC and CBFC techniques. For efficiency, percentage of concentrate, and solute yield, CFBFC showed values close to 86%, 81%, and 0.9 (kg/kg), respectively, which were higher values than GBFC (48%, 38%, and 0.5 (kg/kg)) and CBFC (79%, 68%, and 0.7 (kg/kg)). Therefore, this research offers new benefits with the addition of the filter in the centrifugal BFC, and thus, CFBFC offers an advantage due to the better separation than GBFC and CBFC, since the filter can be designated as a second separation stage, and only one cycle is necessary to obtain high quality properties in the final solution.

Keywords: freeze crystallization; centrifugation; filter; blueberry juice; preservation; physicochemical properties; polyphenolic compounds; antioxidant capacity; process parameters

Citation: Orellana-Palma, P.; Guerra-Valle, M.; Zúñiga, R.N. Centrifugal Filter-Assisted Block Freeze Crystallization Applied to Blueberry Juice. *Processes* **2021**, *9*, 421. https://doi.org/10.3390/pr9030421

Received: 25 January 2021
Accepted: 24 February 2021
Published: 26 February 2021

1. Introduction

Consumers are becoming more aware of the health benefits of fruits and vegetables and their protective effects against various diseases. Thus, consumers have been interested in consumption of fresh blueberries or derived products such as yogurt, jams, jellies, purees, and fruit juices due to their minerals, vitamins, high amount of potential bioactive compounds, high antioxidant activity, and acceptable sensory quality compared to other berries [1].

The food industry has evolved in the application of traditional thermal concentration processing to improve physicochemical properties, phenolic compounds retention, while keeping nutritional and organoleptic characteristics in the final product due to improvements in the operation conditions (pressure, temperature, time, among others) [2]. However, the use of high temperatures presents an important number of disadvantages, since the nutrients (minerals, bioactive compounds (vitamins, polyphenols, anthocyanins, flavonoids, flavonols, among others), and antioxidant activity, among others) have a high degree of deterioration, depending on the temperature and time under treatment [3]. Hence, emerging treatments such as ultrasound [4], microwave [5], radio-frequency [6], pulsed electric field [7], ohmic heating [8], plasma treatment [9], high hydrostatic pressure [10] and

freeze crystallization [11] have been studied with the intention to preserve and stabilize nutritional and organoleptic characteristics of each food product.

In particular, freeze crystallization (FC) is an emerging and environmentally friendly technology that uses low temperatures to concentrate liquid solutions containing heat-sensitive compounds, based on the partial or total freezing of the solution, and then, the unfrozen (cryoconcentrated) solution is separated from the ice crystals. Among FC techniques, block FC (BFC) process has received attention due to the concentration method and simplicity of separation step.

Specifically, in BFC, the entire solution is subjected to freezing process, thawing of the frozen solution, and separating of the cryoconcentrated solution from the frozen matrix [12]. Hence, BFC protects and retains various valuable bioactive components from fresh juices such as polyphenols [13], anthocyanins [14], ascorbic acid [15], flavonoids [16], and flavonols [17].

In BFC, the separation step has been carried out by gravitational thawing [18] or by addition of external forces to improve the BFC process [19]. Hence, studies have reported the use of microwave heating as an assisted thermal heating to accelerate the thawing step [20], and the use of external mechanical forces to help in the extraction step of the cryoconcentrated solution such as centrifugation [21], and vacuum [22], and thus, the external forces-assisted BFC allow the increase of process parameters and quality of the cryoconcentrated fraction [23]. However, despite the results with each external force have shown good performance in comparison to the gravitational BFC, it is possible to increase even more the concentration of solutes in the liquid phase (cryoconcentrated fraction) and the process parameters in the BFC process.

Specifically, Orellana-Palma et al. [24] added a filter to the centrifugal BFC process applied to sucrose solution, allowing to increase the solutes, efficiency, percentage of concentrate and solute yield. Nevertheless, to the best of our knowledge, there is still a lack of studies on centrifugal-filter as a potential new assisted technique in BFC process, and particularly, the use of centrifugal-filter assisted BFC (CFBFC) on fruit juices and their effects on process parameters and retention of bioactive compounds. With this knowledge, this new assisted technique would allow a significant role for the accelerated improvement of the FC technology. Furthermore, the application of CFBFC could be focused on the scale-up in the amount of fresh juice to concentrate.

Therefore, the objective of this study was to evaluate the addition of centrifugation with a filter as assisted technique in the BFC process to improve process parameters, and quality properties of fresh blueberry juice, since these berries have been recognized by their high levels in phenolic compounds and antioxidant activity. Additionally, the centrifugal-filter assisted BFC results were compared with gravitational BFC and centrifugal BFC.

2. Materials and Methods

2.1. Chemicals

Folin-Ciocalteu reagent, sodium carbonate (Na_2CO_3), potassium chloride (KCl), sodium acetate (CH_3COONa), sodium nitrite ($NaNO_2$), aluminum chloride ($AlCl_3$), sodium hydroxide (NaOH), 1,1-diphenyl-2-picryl-hydrazyl (DPPH), 2,4,6-tris(2-pyridyl)-S-triazine (TPTZ), ferric chloride ($FeCl_3$), ABTS (2,2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid)), fluorescein solution, 2,2′-azobis(2-methylpropionamidine)-dihydrochloride (AAPH), (±)-6-hydroxyl-2,5,7,8-tetramethyl-2-carboxylic acid (Trolox), acid gallic, cyanidin-3-glucoside and (+)-catechin, were purchased from Sigma-Aldrich Co. (St. Louis, MO, USA). All reagents used were of analytical grade. Distilled water was used throughout all experiments.

2.2. Raw Material and Juice Preparation

Blueberries (*Vaccinium corymbosum* L., cv. *Brigitta*) were purchased in Chillán (Región del Ñuble, Chile) and the blueberries were washed with tap water to remove any dirt adhered on the surface. Then, the blueberry juice was obtained using a domestic juicer (JE2001, 850 Watts, Nex, Barcelona, Spain), and later, the liquid solution was filtered (nylon

cloth, 0.8 mm mesh) to remove large fragments such as pulp, skin and seeds. Finally, the juice was placed in dark bottles at 4 °C until processing.

2.3. BFC Procedures

The centrifugal-filter BFC (CFBFC) procedure was made according to the method reported by Orellana-Palma et al. [24]. Specifically, the blueberry juice (12 mL) was placed in plastic centrifugal filter tubes (Amicon Ultra 15 mL, Merck Millipore Ltd., Dublin, Ireland) isolated with foamed polystyrene (8 mm thickness, thermal conductivity K = 0.035 W/mK) to produce an axial freezing. The samples were frozen in a static freezer overnight at −20 °C, and later, the cryoconcentrated solution was separated from the ice fraction by centrifugation (Eppendorf 5430R, Hamburg, Germany) at 20 °C for 15 min at 3850 rpm.

Results of CFBFC procedure were compared with gravitational BFC (GBFC) and centrifugal BFC (CBFC) procedures under the same CFBFC conditions (axial freezing at −20 °C and centrifugation parameters). For both GBFC and CBFC procedures, the filter was blocked to prevent that the cryoconcentrated or water remains inside the filter.

For GBFC, the blueberry juice (12 mL) was placed in plastic centrifugal filter (previously blocked) tubes, and then, the tubes were isolated with foamed polystyrene (similar to CFBFC), and later, the freezing conditions were applied (overnight at −20 °C). Once the freezing step was finished, the samples were removed from the static freezer; the tubes were put in inverted position (for gravitational thawing) through all-function clamp holder in a metal support. Hence, the samples were thawed at 20 °C for 15 min, and the cryoconcentrated fraction was received in a glass beaker.

For CBFC, the blueberry juice (12 mL) was placed in plastic centrifugal filter (previously blocked) tubes, and then, the tubes were isolated with foamed polystyrene (similar to CFBFC), and later, the freezing conditions were applied (overnight at −20 °C). Once the freezing step was finished, the samples were transferred to centrifuge equipment with a 50 mL-tube rotor (Eppendorf 5430R), and thus, the separation step was applied at 20 °C for 15 min at 3850 rpm to obtain a cryoconcentrated solution and ice fraction.

Thus, in all the cryoconcentrated samples, process parameters and different quality properties such as physicochemical properties, polyphenolic compounds and antioxidant activity were determined.

2.4. Determination of Physicochemical Properties

Total soluble solids (TSS, °Brix) were determined using a hand-held digital refractometer (PAL-3, ≈1 mL, range: 0–93 °Brix, precision: ±0.1 °Brix, Atago Inc., Tokyo, Japan). The pH was measured with a digital pH meter (HI 2210, Hanna Instruments, Woonsocket, RI, USA) calibrated with commercial buffer solutions at pH 7.0 and 4.0. The titratable acidity (TTA) was determined by acid-base titration method [14]. The samples (5 mL) were diluted with deionized water (50 mL) and it was titrated to the phenolphthalein endpoint (pH = 8.1 ± 0.1) with sodium hydroxide solution (0.1 M NaOH). The TTA values were expressed as grams of malic acid (MA) per 100 mL of sample (g MA/100 mL).

The CIELab color space parameters were measured directly using a spectrophotometer (CM-5, Konica Minolta, Osaka, Japan), and it was standardized each time with a standard white tile using illuminant D65 and an observation angle of 10°. The sample was filled in a glass cuvette, and thus, the CIELab values of L^* (lightness; 0 = black, 100 = white), a^* ($-a^*$ = greenness, $+a^*$ = redness) and b^* ($-b^*$ = blueness, $+b^*$ = yellowness) were measured. Moreover, the total color difference (ΔE^*) was calculated by Equation (1):

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

where ΔE^* is the change variation between fresh juice and concentrated samples, $\Delta L^* = (L^* - L_0^*)$, $\Delta a^* = (a^* - a_0^*)$ and $\Delta b^* = (b^* - b_0^*)$. The subscript 0 corresponds to the initial CIELab values in the fresh blueberry juice, and L^*, a^* and b^* are the color properties in each cryoconcentrated sample.

2.5. Determination of Total Phenolic Content (TPC), Total Anthocyanin Content (TAC), and Total Flavonoid Content (TFC)

TPC, TAC, and TFC assays were measured with a spectrophotometer UV/Vis (T70, Oasis Scientific Inc., Greenville, SC, USA) according to the method described by Moyer et al. [25], Khanal et al. [26], and Hwang et al. [27], where gallic acid (GA), cyanidin-3-glucoside (C3G), and catechin (C) were used for the standard curve, and the results were expressed in milligrams of GA equivalents (GAE) per grams (g) of dry matter (mg GAE/g d.m.), milligrams of C3G equivalents per grams (g) of dry matter (mg C3G/g d.m.), and milligrams of C equivalents (CEQ) per grams (g) of dry matter (mg QE/g d.m.), respectively.

Additionally, the polyphenolic retention (PR) represents the polyphenolic compounds retained from the fresh juice in the cryoconcentrated fraction [18], and it was calculated by Equation (2):

$$\text{PR } (\%) = \left(\frac{C_o}{C_c}\right) \times \left(\frac{TBC_c}{TBC_o}\right) \times 100\% \quad (2)$$

where C_0 is the initial TSS value, C_c is the TSS in the cryoconcentrated sample, TBC_c is the polyphenolic compounds value in the cryoconcentrated sample, and TBC_0 is the initial polyphenolic compounds value.

2.6. Determination of Antioxidant Activity (AA)

DPPH, FRAP (Ferric reducing antioxidant power), and ABTS assays were measured with a spectrophotometer UV/Vis (T70, Oasis Scientific Inc.) according to the method described by Brand-Williams et al. [28], Koca and Karadeniz [29], and Re et al. [30], respectively.

ORAC assay was evaluated based on the method described by Huang et al. [31] with a multimode plate reader (Victor X3, Perkin Elmer, Hamburg, Germany). The absorbance was measured at 485 nm ($\lambda_{excitation}$) and at 535 nm ($\lambda_{emission}$) every 3 min for 300 min.

Trolox (T) was used for the standard curve, and the AA assays were expressed as μM Trolox equivalents (TE) per gram (g) of dry matter (μM TE/g d.m.).

2.7. Determination of the Process Parameters

2.7.1. Efficiency (η)

η denotes the amount of solutes in the cryoconcentrated fraction (C_c) relative to the solutes remaining in the ice fraction (C_I), and it was calculated by Equation (3):

$$\eta\ (\%) = \frac{C_c - C_I}{C_c} \times 100\% \quad (3)$$

where C_c is the TSS in the cryoconcentrated fraction, and C_I is the TSS in the ice fraction.

2.7.2. Solute Yield (Y)

Y (kg solutes/kg initial solutes, kg/kg) represents the recovered mass in the cryoconcentrated fraction from the initial mass, and it was calculated by Equation (4):

$$Y\ (\frac{kg}{kg}) = \frac{m_c}{m_o} \quad (4)$$

where m_c is the mass of the cryoconcentrated fraction and m_0 is the initial mass.

2.7.3. Percentage of Concentrate (PC)

PC is defined as the removed solute from the initial frozen fraction, and it was calculated by Equation (5):

$$\text{PC } (\%) = \frac{W_0 - W_I}{W_0} \times 100\% \quad (5)$$

where W_0 is the initial weight of the frozen fraction and W_I is the final weight of the frozen fraction.

2.8. Statistical Analysis

The treatments were conducted in triplicate at ambient temperature (≈22 °C), and the results were presented as mean ± standard deviation. One-way analysis of variance (ANOVA) was used to the evaluation of statistical analysis and the treatment means were compared via least significant difference (LSD) test at a confidence level of 0.95 ($p \leq 0.05$). The data were analyzed through Statgraphics Centurion XVI software (v. 16.2.04, StatPoint Technologies Inc., Warrenton, VA, USA).

3. Results and Discussion

3.1. Determination of Physicochemical Properties

TSS, pH, TTA, and color (L*, a*, b* and ΔE*) values of the samples are summarized in Table 1. Firstly, the physicochemical properties values presented significant statistical differences ($p \leq 0.05$) when related each cryoconcentrated sample with the fresh juice, and in turn, the same tendency was observed when compared the results between each BFC technique.

Table 1. Physicochemical characteristics of fresh juice, cryoconcentrated and ice fractions.

	Fresh Juice	GBFC	CBFC	CFBFC
TSS (°Brix) (cryoconcentrated)	13.8 ± 0.6 ^{a}	23.4 ± 1.2 ^{b}	29.5 ± 1.3 ^{c}	35.9 ± 2.1 ^{d}
TSS (°Brix) (ice fraction)	13.8 ± 0.6 ^{a}	12.1 ± 0.8 ^{b}	6.3 ± 0.2 ^{c}	5.2 ± 0.4 ^{d}
pH	3.1 ± 0.1 ^{a}	2.8 ± 0.1 ^{b}	2.5 ± 0.0 ^{c}	2.3 ± 0.0 ^{d}
TTA (g MA/100 mL)	0.7 ± 0.0 ^{a}	1.0 ± 0.1 ^{b}	1.4 ± 0.0 ^{c}	1.9 ± 0.1 ^{d}
L*	32.5 ± 1.4 ^{a}	15.2 ± 1.5 ^{b}	12.0 ± 1.1 ^{c}	7.2 ± 2.5 ^{d}
a*	7.3 ± 1.2 ^{a}	4.0 ± 0.9 ^{b}	2.9 ± 0.4 ^{c}	1.2 ± 0.3 ^{d}
b*	4.0 ± 0.0 ^{a}	1.1 ± 0.0 ^{b}	0.8 ± 0.2 ^{c}	0.1 ± 0.1 ^{d}
ΔE*	-	17.9 ± 1.3 ^{a}	21.6 ± 1.7 ^{b}	26.3 ± 1.0 ^{c}

Different letters in a row are significantly different ($p \leq 0.05$) according to LSD test. GBFC, CBFC, CFBFC are gravitational BFC, centrifugal BFC, and centrifugal-filter BFC, respectively.

Hence, the physicochemical properties values in the fresh blueberry juice were higher than those reported by Cheng et al. [32] and Madrera et al. [33], who study diverse blueberry cultivars in the world. The differences among the physicochemical properties can be due mainly to aspects such as type of harvesting, time of harvesting, ripening process, climatic conditions, and/or the interaction genotype-environment [34].

In particular, the lowest TSS value was obtained with GBFC (23.4 °Brix), while the CBFC process achieved a lower TSS value (29.5 °Brix) than CFBFC (35.9 °Brix), indicating an increase of 1.7, 2.1 and 2.6 times with respect to the initial TSS value, for GBFC, CBFC, and CFBFC, respectively. Specifically, the TSS values obtained by CFBFC in only one cycle of centrifugation were higher than any results in our previous works with CBFC as concentration method, since the TSS values in the first cycle were close to 22 °Brix, 23 °Brix, 26 °Brix, 31 °Brix, and 32 °Brix for blueberry juice [35], calafate juice [12], pineapple juice [36], orange juice [15], and apple juice [17], respectively. Also, the TSS values obtained by CFBFC were superior to those described by Ding et al. [37] and Meneses et al. [38], who used suspension FC and multi-stage BFC in apple juice and green tea, respectively. Therefore, these high values through CFBFC method implies that the addition of a filter improved the separation process, since the filter is a second separation step that retains components (even water) post-centrifugation process, and thus, the TSS values in CFBFC were higher than a separation without filter, corroborating the high TSS values in the final cryoconcentrated fraction reached by Orellana-Palma et al. [24], who concentrate sucrose solution through BFC method, and then, the separation was produced by centrifugal-filter as assisted technique.

In the ice fractions, a contrary trend was observed in the TSS values, since the lowest TSS value was obtained with CFBFC (5.2 °Brix), while the CBFC process achieved a lower TSS value (6.3 °Brix) than GBFC (12.1 °Brix). Thus, the CBFC methods without filter had higher TSS concentration than the CFBFC method. A similar behavior was specified by Orellana-Palma et al. [24], reinforcing that the filter is a second separation step, since it allows retaining a considerable amount of water from the feed solution.

In all the BFC methods, the pH and TTA values presented an opposite behavior, since the pH increased and TTA decreased with respect to the initial pH and TTA values. Thus, CFBFC presented the lowest pH and the highest TTA values in relation to the other samples, with values of 2.3 and 1.9, respectively. These effects had good agreements with previous reports on other cryoconcentrated fruit juices [12,13,21,35], i.e., the samples had lower pH and higher TTA than the original sample (fresh juice). The pH and TTA changes can be attributed to the increase in TSS concentration, since it provokes an increase in the organic acids, causing the decrease in TTA values, i.e., the pH and TTA values have an inverse behavior [36]. In this context, pH and TTA values were correlated, and a negative linear correlation ($r = -0.98$) was found between the parameters, corroborating that the increase in TSS concentration produces an inversely proportional behavior between pH and TTA, i.e., as the organic acids increased, the TTA values decreased.

In terms of colorimetric CIELab parameters, a decrease in L*, a* and b* values was recorded in the cryoconcentrated samples in comparison to the fresh juice, where CFBFC showed the lowest values in each parameter. Specifically, in CFBFC, the L*, a* and b* values were close to 7.2, 1.2, and 0.1 units, which is equivalent to a reduction of 78%, 84% and 99% in relation to the initial CIELab values, respectively. Hence, as shown in Figure 1, the fresh blueberry juice had a characteristic red tone [39] (Figure 1a), while post BFC process, all the samples presented a very dark red tone, and in particular, the CFBFC obtained the darkest color compared to the other cryoconcentrated samples. These results are in concordance with studies on BFC applied to blueberry juice, since BFC generated very dark cryoconcentrate juices with a red/purple tone [13,40,41]. Moreover, according to the total color difference (ΔE*), Szczepańska et al. [42] indicated that a $\Delta E^* \geq 3.0–5.0$ reflects noticeable color difference through human eye between the original sample and treated samples, and thus, there is a visual difference between the fresh juice and samples produced for each BFC technique, since the ΔE* values were higher than 17, 21, and 25 CIELab units, for GBFC, CBFC, and CFBFC, respectively. Thereby, all the data and visual color had similar trends to the results reported in various studies on FC applied to fresh juices [13,35,40], and it can be connected to the high TSS values post BFC, since the low temperature of concentration permits a best separation of cryoconcentrated samples from the unfrozen fraction, allowing a significant increase in the initial solutes from the fresh juice, and in turn, producing a gradual and significant darkening of fresh juices [41].

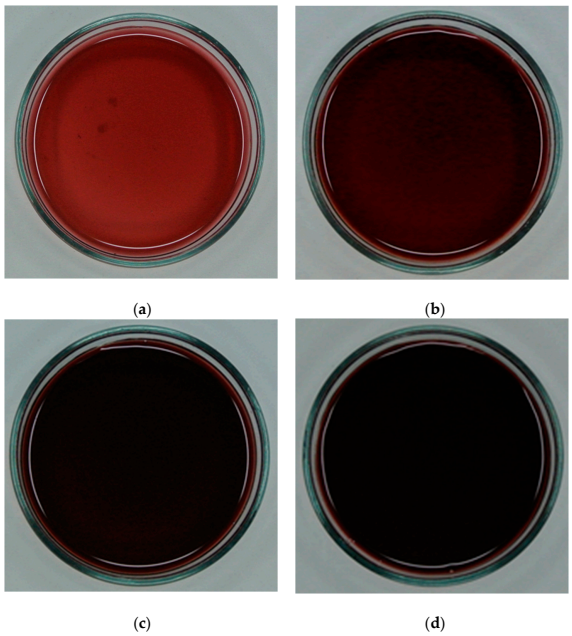
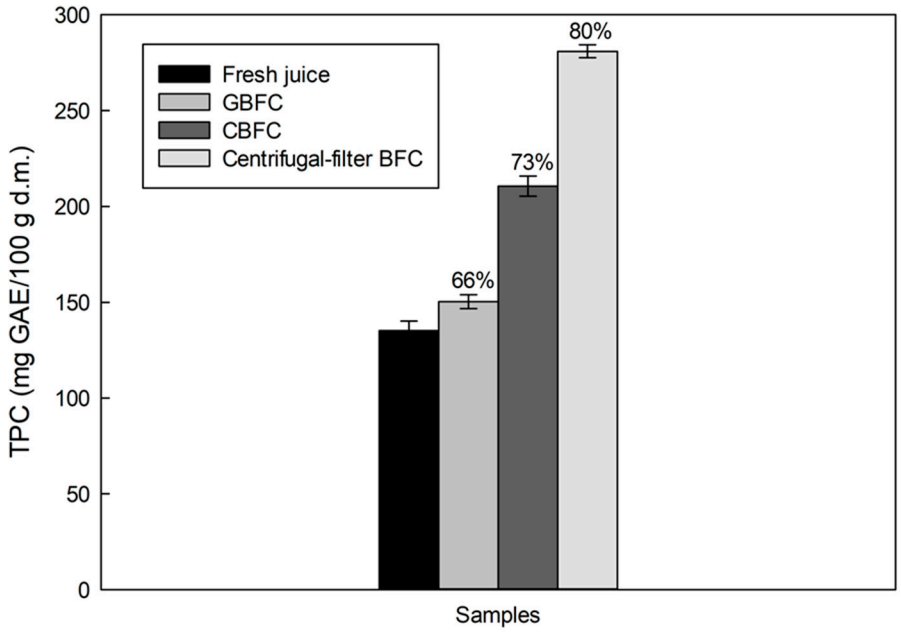


Figure 1. Effect on visual color of different BFC applied to fresh blueberry juice: (**a**) Fresh juice; (**b**) GBFC; (**c**) CBFC; (**d**) CFBFC.

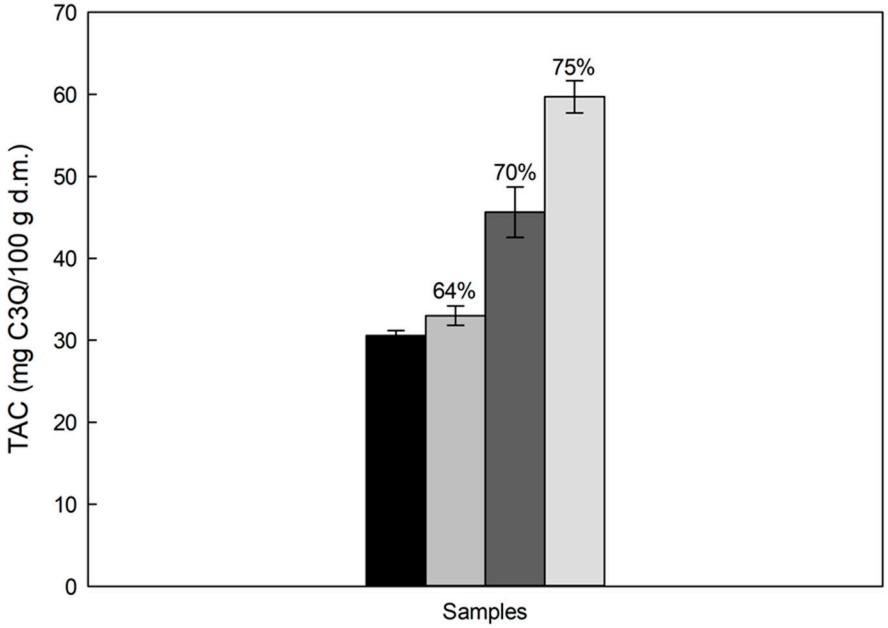
3.2. Determination of Total Phenolic Content (TPC), Total Anthocyanin Content (TAC), and Total Flavonoid Content (TFC)

The polyphenolic compounds values of the samples (fresh blueberry juice, GBFC, CBFC and CFBFC) are shown in Figure 2. Firstly, fresh blueberry juice had TPC, TAC, and TFC values close to 135 mg GAE/100 g d.m., 33 mg C3G/100 g d.m., and 41 mg CEQ/100 g d.m., respectively. These results were lower than the reported by Casas-Forero et al. [43] in our laboratory, who studied the incorporation of cryoconcentrated blueberry juice (cv. *Brigitta*) inside aerated gelatin gels, where the fresh blueberry juice had polyphenolic compounds values of 120 mg GAE/100 g d.m, 10 mg C3G/100 g d.m., and 25 mg CEQ/100 g d.m., for TPC, TAC, and TFC, respectively. However, our polyphenolic compounds values were lower than the values stated by González-Villagra et al. [44], who studied diverse blueberries cultivars (cv. *Legacy*, cv. *Brigitta*, and cv. *Bluegold*) from Southern Chile. Therefore, the variation in polyphenolic compounds values can be associated to the fruit culture, final maturity, growth, agroclimatic and storage conditions, and different extraction and processing methods [45].

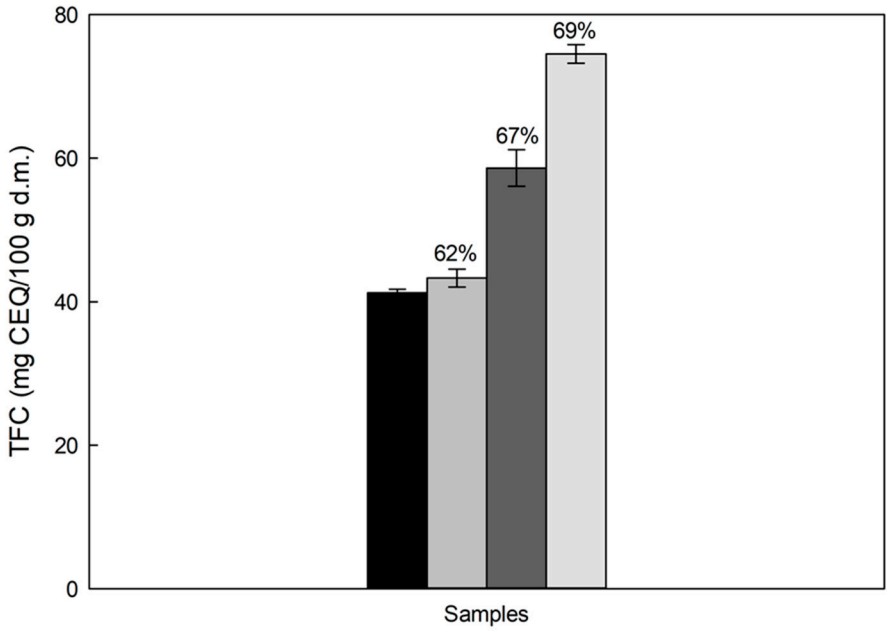


(**a**)

Figure 2. *Cont.*



(**b**)



(**c**)

Figure 2. Polyphenolic compounds in samples: (**a**) TPC; (**b**) TAC; (**c**) TFC.

All the cryoconcentrated samples had a significant increase in the polyphenolic compound values after the BFC process. The minimum TPC, TAC and TFC values were observed in GBFC, with values close to 150 mg GAE/100 g d.m., 33 mg C3G/100 g d.m., 43 mg CEQ/100 g d.m., respectively. While, the CFBFC results revealed that polyphenolic compounds values were higher than GBFC and CBFC (similar to TSS values), with ≈281 mg GAE/100 g d.m., ≈60 mg C3G/100 g d.m., ≈75 mg CEQ/100 g d.m., for TPC, TAC, and TFC, respectively. Hence, the cryoconcentrated solution achieved by CFBFC had an increased up to 2.1 (TPC), 2.0 (TAC) and 1.8 (TFC) times compared to the fresh

blueberry juice. These polyphenolic compound values increases (in any BFC process) with respect to the original sample have been detected in liquid samples such as *Centella Asiatica* extract [46], maqui juice [47], and broccoli extract [48], corroborating that the concentration at low temperature is a good option to retain potentially polyphenolic compounds, and in turn, the addition of the filter allow a better separation than other typical BFC process, i.e., an extra system in the BFC procedure manages to separate even more cryoconcentrated fraction from the ice fraction [49].

In the same way, the PR retention values (Figure 2, percent values) indicated that the highest PR values were detected in the CFBFC process, with values close to 80%, 75%, and 69%, for TPC, TAC, and TFC, respectively. The results are equivalent to those informed by Orellana-Palma et al. [13], Casas-Forero et al. [35] and Correa et al. [50], who indicated PR retention values from 53% to 95% after multiple cycles for different food liquids. The high retention in TPC, TAC and TFC displays the advantage of the filter in the centrifugal BFC process, since the results indicate that only one centrifugal cycle is necessary to obtain a high retention in comparison to other BFC studies, which used three concentration cycles [24].

3.3. Determination of the Antioxidant Activity (AA)

Table 2 presents the AA in fresh blueberry juice and after each BFC technique. The AA of the fresh juice obtained from blueberry fruits was found to be ≈264, ≈312, ≈575, and ≈815 μM TE/100 g d.m., for DPPH, FRAP, ABTS, and ORAC, respectively. These AA values were higher than those reported by Casas-Forero et al. [35,39], who study the bioactive components and antioxidant activiy in fresh blueberry juice from the same cultivar (cv. *Brigitta*). Nevertheless, our AA values were lower than those reported by Ribera [51], who evaluated bioactive components and antioxidant activity from different genotype and maturity stages in blueberry fruits (cv. *Brigitta*, cv. *Bluegold* and cv. *Legacy*) grown in Southern Chile. Hence, the variability of the antioxidant capacity in the fruits depends on multiple factors such as fruit culture, growth sector, maturity stage, storage conditions, and extraction methods, and in turn, all these factors affect the concentration of bioactive components, and thus, this concentration has a direct relationship on the antioxidant capacity in each fruit [52].

Table 2. Antioxidant activity of fresh juice and cryoconcentrated samples.

	Fresh Juice	GBFC	CBFC	CFBFC
DPPH	263.6 ± 10.3 ^{a}	315.3 ± 22.9 ^{b}	560.7 ± 60.3 ^{c}	850.5 ± 32.7 ^{d}
FRAP	312.2 ± 17.9 ^{a}	498.3 ± 37.1 ^{b}	855.8 ± 29.1 ^{c}	1301.3 ± 55.9 ^{d}
ABTS	574.6 ± 33.0 ^{a}	739.6 ± 19.2 ^{b}	1344.1 ± 27.9 ^{c}	1768.2 ± 70.3 ^{d}
ORAC	816.0 ± 22.7 ^{a}	1091.4 ± 55.7 ^{b}	1588.1 ± 43.7 ^{c}	2741.1 ± 101.3 ^{d}

Different letters in a row are significantly different ($p \leq 0.05$) according to LSD test. GBFC, CBFC, CFBFC are gravitational BFC, centrifugal BFC, and centrifugal-filter BFC, respectively.

After the BFC method, a increase in AA values was denoted in comparison to the fresh juice. Accordingly, CFBFC had the highest AA values, with ≈850, ≈1301, ≈1768 and ≈2741 μM TE/100 g d.m., equivalenting to an increase close to 3.2, 4.2, 3.1 and 3.4 times in relation to the fresh blueberry juice, whereas GBFC had the lowest AA values, with ≈315, ≈498, ≈740 and ≈1091 μM TE/100 g d.m., for DPPH, FRAP, ABTS, and ORAC, respectively, confirming that the addition of a filter improves the separation and concentration process, since the final sample presented higher AA values than other samples obtained by BFC without filter. Therefore, the CFBFC can be viewed as emerging alternative to the progressive increase in solutes, bioactive components, and antioxidant capacity. All the results (high AA values) are consistent with those of previous studies in apple juice [15,16], yogurt enriched with cryoconcentrated strawberry [53], and sapucaia nut [54], demostrating the positive effects of the BFC on polyphenolic compounds (principally anthocyanins), which finally leads to concentrate and preserve the antioxidant components in the fresh juice [19].

3.4. Determination of Process Parameters

The process parameters of cryoconcentrated samples are shown in Table 3. In general, significant statistical differences were found between the cryoconcentrated samples, where CFBFC had higher η, PC, and Y values than GBFC and CBFC techniques.

Table 3. Process parameters in the BFC techniques.

Process Parameters	GBFC	CBFC	CFBFC
Efficiency, %	48.3 ± 5.4^{a}	78.6 ± 2.9^{b}	85.5 ± 1.4^{c}
Percentage of concentrate, %	38.3 ± 3.1^{a}	68.1 ± 4.0^{b}	81.3 ± 2.0^{c}
Solute yield, kg/kg	0.5 ± 0.1^{a}	0.7 ± 0.0^{b}	0.9 ± 0.0^{c}

Different letters in a row are significantly different ($p \leq 0.05$) according to LSD test. GBFC, CBFC, CFBFC are gravitational BFC, centrifugal BFC, and centrifugal-filter BFC, respectively.

Thus, for η, PC, and Y, the values were close to 48%, 78%, and 86%, 38%, 68%, and 81%, and 0.5, 0.7, and 0.9 (kg/kg), for GBFC, CBFC, and CFBFC, respectively. Specifically, the process parameter values in CFBFC were higher than other BFC studies applied to fruit juices such as blueberry juice [13], orange juice [15,21], apple juice [17], pineapple juice [36,40], where the effects of BFC were studied and only centrifugation was used as an external force to remove the cryoconcentrate solution from the ice matrix. Our results allow corroborating that a filter facilitates the separation process, and in turn, it could be defined as a second stage of separation, since the filter retains the multiple components extracted in the centrifugation process. Therefore, as an assisted technique a centrifugal filter allows recovering and collecting a high mass amount from the original mass [55].

An important point, the CFBFC procedure should be standardized and scaled up to the pilot plant level, since it allows corroborating the process parameters values obtained at the laboratory scale. In our case, the differences in the process parameters values can be explained from the components of the juice, since the components (sugars, proteins, pectin, among others) interact differently in the counter diffusion process in the freezing step, and it can be associated with their molecular structures, and a high number of hydrogen bonds can be formed between the molecules with the water, and thus, these structures can be remained in the water molecules (between the ice crystals), which can reduce or increase the amount of final concentrate extracted from the frozen matrix, and it can finally influence the final mass and concentrated obtained post-cycle. In addition, a future study could be focused on the fraction in the filter, since there are three fractions in the CFBFC procerdure, ice fraction, cryoconcentrated solution and the mixture fraction in the filter. In this way, a mass balance could be focused to determine the quantities in the filter post-cycle [56]. Additionally, a previous study indicated that flavanols have a lower affinity to remain between ice crystals and tend to be easily extracted from the ice fraction, affecting the process parameters [57].

4. Conclusions

CFBFC method presented higher TSS, TPC, TAC, and TFC, and AA values than GBFC and CBFC methods, leading to high polyphenolic retention (69–80% retention). Additionally, CFBFC improves the separation/extraction of cryoconcentrated fraction from the ice fraction due to the high values in η (86%), PC (81%), and Y (0.9 kg/kg), compared to the other BFC techniques. Thus, the present results can provide important information on the improvement of the freeze crystallization process through the addition of a filter, since the filter acts as a second separation step, retaining the water extracted from the feed solution in the separation process by the centrifugal force, increasing the concentration of solutes in the liquid phase. Therefore, CFBFC technology at one cycle offers an interesting procedure to concentrate different food solutions, and thus, from the results obtained, to scale (pilot plant and industrial) this food processing in order to have a better concentration technology for the food industry.

Author Contributions: Conceptualization, P.O.-P., M.G.-V. and R.N.Z.; methodology, P.O.-P., M.G.-V. and R.N.Z.; software, P.O.-P. and R.N.Z.; validation, P.O.-P. and R.N.Z.; formal analysis, P.O.-P. and M.G.-V.; investigation, P.O.-P. and M.G.-V.; resources, P.O.-P. and R.N.Z.; data curation, P.O.-P. and M.G.-V.; writing—original draft preparation, P.O.-P. and M.G.-V.; writing—review and editing, P.O.-P. and R.N.Z.; visualization, P.O.-P.; supervision, R.N.Z.; project administration, P.O.-P.; funding acquisition, P.O.-P. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by ANID-Chile (Agencia Nacional de Investigación y Desarrollo de Chile) through the FONDECYT Postdoctoral Grant 2019 (Folio 3190420).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Acknowledgments: Patricio Orellana-Palma acknowledges the financial support of ANID-Chile (Agencia Nacional de Investigación y Desarrollo de Chile) through the FONDECYT Postdoctoral Grant 2019 (Folio 3190420).

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Silva, S.; Costa, E.M.; Veiga, M.; Morais, R.M.; Calhau, C.; Pintado, M. Health promoting properties of blueberries: A review. *Crit. Rev. Food Sci. Nutr.* **2020**, *60*, 181–200. [CrossRef]
2. de Corato, U. Improving the shelf-life and quality of fresh and minimally-processed fruits and vegetables for a modern food industry: A comprehensive critical review from the traditional technologies into the most promising advancements. *Crit. Rev. Food Sci. Nutr.* **2020**, *60*, 940–975. [CrossRef] [PubMed]
3. Yildiz, G.; Aadil, R.M. Comparison of high temperature-short time and sonication on selected parameters of strawberry juice during room temperature storage. *J. Food Sci. Technol.* **2020**, *57*, 1462–1468. [CrossRef]
4. Chen, Y.H.; Yang, C.Y. Ultrasound-assisted extraction of bioactive compounds and antioxidant capacity for the valorization of *Elaeocarpus serratus* L. leaves. *Processes* **2020**, *8*, 1218. [CrossRef]
5. Garofulić, I.E.; Kruk, V.; Martić, A.; Martić, I.; Zorić, Z.; Pedisić, S.; Dragović, S.; Dragović-Uzelac, V. Evaluation of polyphenolic profile and antioxidant activity of *Pistacia lentiscus* L. leaves and fruit extract obtained by optimized microwave-assisted extraction. *Foods* **2020**, *9*, 1556. [CrossRef] [PubMed]
6. Ling, B.; Cheng, T.; Wang, S. Recent developments in applications of radio frequency heating for improving safety and quality of food grains and their products: A review. *Crit. Rev. Food Sci. Nutr.* **2020**, *60*, 2622–2642. [CrossRef] [PubMed]
7. Niu, D.; Zeng, X.A.; Ren, E.F.; Xu, F.Y.; Li, J.; Wang, M.S.; Wang, R. Review of the application of pulsed electric fields (PEF) technology for food processing in China. *Food Res. Int.* **2020**, *137*, 109715. [CrossRef] [PubMed]
8. Salari, S.; Jafari, S.M. The influence of ohmic heating on degradation of food bioactive ingredients. *Food Eng. Rev.* **2020**, *12*, 191–208. [CrossRef]
9. Munekata, P.E.; Domínguez, R.; Pateiro, M.; Lorenzo, J.M. Influence of plasma treatment on the polyphenols of food products—A review. *Foods* **2020**, *9*, 929. [CrossRef] [PubMed]
10. Moreira, S.A.; Pintado, M.; Saraiva, J.A. High hydrostatic pressure-assisted extraction: A review on its effects on bioactive profile and biological activities of extracts. In *Present and Future of High Pressure Processing*, 1st ed.; Barba, F., Tonello-Samson, C., Puértolas, E., Lavilla, M., Eds.; CRC Press: Boca Raton, FL, USA, 2020; pp. 317–328.
11. Miyawaki, O.; Inakuma, T. Development of progressive freeze concentration and its application: A review. *Food Bioproc Technol.* **2021**, *14*, 39–51. [CrossRef]
12. Orellana-Palma, P.; Tobar-Bolaños, G.; Casas-Forero, N.; Zúñiga, R.N.; Petzold, G. Quality attributes of cryoconcentrated calafate (*Berberis microphylla*) juice during refrigerated storage. *Foods* **2020**, *9*, 1314. [CrossRef]
13. Orellana-Palma, P.; Petzold, G.; Guerra-Valle, M.; Astudillo-Lagos, M. Impact of block cryoconcentration on polyphenol retention in blueberry juice. *Food Biosci.* **2017**, *20*, 149–158. [CrossRef]
14. 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. *Int. J. Food Sci.* **2017**, *52*, 781–787. [CrossRef]
15. Orellana-Palma, P.; González, Y.; Petzold, G. Improvement of centrifugal cryoconcentration by ice recovery applied to orange juice. *Chem. Eng. Technol.* **2019**, *42*, 925–931. [CrossRef]
16. Zielinski, A.A.; Zardo, D.M.; Alberti, A.; Bortolini, D.G.; Benvenutti, L.; Demiate, I.M.; Nogueira, A. Effect of cryoconcentration process on phenolic compounds and antioxidant activity in apple juice. *J. Sci. Food Agric.* **2019**, *99*, 2786–2792. [CrossRef]
17. Orellana-Palma, P.; Lazo-Mercado, V.; Gianelli, M.P.; Hernández, E.; Zúñiga, R.N.; Petzold, G. Influence of cryoconcentration on quality attributes of apple juice (*Malus Domestica* cv. *Red Fuji*). *Appl. Sci.* **2020**, *10*, 959. [CrossRef]
18. Aider, M.; de Halleux, D.; Melnikova, I. Skim milk whey cryoconcentration and impact on the composition of the concentrated and ice fractions. *Food Bioproc. Technol.* **2009**, *2*, 80–88. [CrossRef]

19. Gunathilake, K.D.P.P. Emerging technologies available for the enhancement of bioactives concentration in functional beverages. In *Biotechnological Progress and Beverage Consumption*, 1st ed.; Grumezescu, A., Holban, A.M., Eds.; Academic Press: New York, NY, USA, 2020; pp. 39–69.
20. Aider, M.; de Halleux, D. Passive and microwave-assisted thawing in maple sap cryoconcentration technology. *J. Food Eng.* **2008**, *85*, 65–72. [CrossRef]
21. Orellana-Palma, P.; Petzold, G.; Andana, I.; Torres, N.; Cuevas, C. Retention of ascorbic acid and solid concentration via centrifugal freeze concentration of orange juice. *J. Food Qual.* **2017**, *2017*, 5214909. [CrossRef]
22. Canella, M.H.M.; Dantas, A.; Blanco, M.; Raventós, M.; Hernandez, E.; Prudencio, E.S. Optimization of goat milk vacuum-assisted block freeze concentration using response surface methodology and NaCl addition influence. *LWT-Food Sci. Technol.* **2020**, *124*, 109133. [CrossRef]
23. Orellana-Palma, P.A. External Forces Assisted Cryoconcentration to Improve the Concentration Process and the Quality of Fruit Juices. Ph.D. Thesis, Universidad del Bío-Bío, Chillán, Chile, May 2018.
24. Orellana-Palma, P.; Takhar, P.; Petzold, G. Increasing the separation of block cryoconcentration through a novel centrifugal filter-based method. *Sep. Sci. Technol.* **2019**, *54*, 786–794. [CrossRef]
25. Moyer, R.A.; Hummer, K.E.; Finn, C.E.; Frei, B.; Wrolstad, R.E. Anthocyanins, phenolics, and antioxidant capacity in diverse small fruits: *Vaccinium*, *Rubus*, and *Ribes*. *J. Agric. Food Chem.* **2002**, *50*, 519–525. [CrossRef] [PubMed]
26. Khanal, R.C.; Howard, L.R.; Brownmiller, C.R.; Prior, R.L. Influence of extrusion processing on procyanidin composition and total anthocyanin contents of blueberry pomace. *J. Food Sci.* **2009**, *74*, H52–H58. [CrossRef]
27. Hwang, H.; Kim, Y.J.; Shin, Y. Assessment of physicochemical quality, antioxidant content and activity, and inhibition of cholinesterase between unripe and ripe blueberry fruit. *Foods* **2020**, *9*, 690. [CrossRef]
28. Brand-Williams, W.; Cuvelier, M.E.; Berset, C.L.W.T. Use of a free radical method to evaluate antioxidant activity. *LWT-Food Sci. Technol.* **1995**, *28*, 25–30. [CrossRef]
29. Koca, I.; Karadeniz, B. Antioxidant properties of blackberry and blueberry fruits grown in the Black Sea Region of Turkey. *Sci. Hortic.* **2009**, *121*, 447–450. [CrossRef]
30. Re, R.; Pellegrini, N.; Proteggente, A.; Pannala, A.; Yang, M.; Rice-Evans, C. Antioxidant activity applying an improved ABTS radical cation decolorization assay. *Free Radic. Biol. Med.* **1999**, *26*, 1231–1237. [CrossRef]
31. Huang, D.; Ou, B.; Hampsch-Woodill, M.; Flanagan, J.A.; Prior, R.L. High-throughput assay of oxygen radical absorbance capacity (ORAC) using a multichannel liquid handling system coupled with a microplate fluorescence reader in 96-well format. *J. Agric. Food Chem.* **2002**, *50*, 4437–4444. [CrossRef]
32. Cheng, K.; Peng, B.; Yuan, F. Volatile composition of eight blueberry cultivars and their relationship with sensory attributes. *Flavour Fragr. J.* **2020**, *35*, 443–453. [CrossRef]
33. Madrera, R.R.; Valles, B.S.; Negrillo, A.C.; Fernández, J.F. Physicochemical characterization of blueberry (*Vaccinium* spp.) juices from 55 cultivars grown in Northern Spain. *Acta Aliment.* **2019**, *48*, 260–268. [CrossRef]
34. Spinardi, A.; Cola, G.; Gardana, C.S.; Mignani, I. Variation of anthocyanins content and profile throughout fruit development and ripening of highbush blueberry cultivars grown at two different altitudes. *Front. Plant Sci.* **2019**, *10*, 1045. [CrossRef]
35. Casas-Forero, N.; Orellana-Palma, P.; Petzold, G. Influence of block freeze concentration and evaporation on physicochemical properties, bioactive compounds and antioxidant activity in blueberry juice. *Food Sci. Technol.* **2020**, *40*, 387–394. [CrossRef]
36. Orellana-Palma, P.; Zuñiga, R.N.; Takhar, P.S.; Gianelli, M.P.; Petzold, G. Effects of centrifugal block freeze crystallization on quality properties in pineapple juice. *Chem. Eng. Technol.* **2020**, *43*, 355–364. [CrossRef]
37. Ding, Z.; Qin, F.G.; Yuan, J.; Huang, S.; Jiang, R.; Shao, Y. Concentration of apple juice with an intelligent freeze concentrator. *J. Food Eng.* **2019**, *256*, 61–72. [CrossRef]
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. [CrossRef]
39. 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. Technol.* **2021**, *138*, 110674. [CrossRef]
40. Petzold, G.; Moreno, J.; Lastra, P.; Rojas, K.; Orellana, P. Block freeze concentration assisted by centrifugation applied to blueberry and pineapple juices. *Innov. Food Sci. Emerg.* **2015**, *30*, 192–197. [CrossRef]
41. Orellana-Palma, P.; Petzold, G.; Pierre, L.; Pensaben, J.M. Protection of polyphenols in blueberry juice by vacuum-assisted block freeze concentration. *Food Chem. Toxicol.* **2017**, *109*, 1093–1102. [CrossRef]
42. Szczepańska, J.; Barba, F.J.; Skąpska, S.; Marszałek, K. High pressure processing of carrot juice: Effect of static and multi-pulsed pressure on the polyphenolic profile, oxidoreductases activity and colour. *Food Chem.* **2020**, *307*, 125549. [CrossRef]
43. Casas-Forero, N.; Orellana-Palma, P.; Petzold, G. Comparative study of the structural properties, color, bioactive compounds content and antioxidant capacity of aerated gelatin gels enriched with cryoconcentrated blueberry juice during storage. *Polymers* **2020**, *12*, 2769. [CrossRef]
44. González-Villagra, J.; Reyes-Díaz, M.; Alberdi, M.; Mora, M.L.; Ulloa-Inostroza, E.M.; Ribera-Fonseca, A.E. Impact of cold-storage and UV-C irradiation postharvest treatments on quality and antioxidant properties of fruits from blueberry cultivars grown in Southern Chile. *J. Soil Sci. Plant Nutr.* **2020**, *20*, 1751–1758. [CrossRef]
45. Zhang, J.; Nie, J.Y.; Jing, L.I.; Zhang, H.; Ye, L.I.; Farooq, S.; Bacha, S.A.S.; Wang, J. Evaluation of sugar and organic acid composition and their levels in highbush blueberries from two regions of China. *J. Integr. Agric.* **2020**, *19*, 2352–2361. [CrossRef]

46. Amran, N.A.; Rafee, N.H.M.; Samsuri, S. Retention of bioactive compound in the concentration of *Centella asiatica* extract through progressive freezing. *IOP Conf. Ser. Mater. Sci. Eng.* **2020**, *778*, 012175. [CrossRef]
47. Bastías-Montes, J.M.; Martín, V.S.; Muñoz-Fariña, O.; Petzold-Maldonado, G.; Quevedo-León, R.; Wang, H.; Yang, Y.; Céspedes-Acuña, C.L. Cryoconcentration procedure for aqueous extracts of maqui fruits prepared by centrifugation and filtration from fruits harvested in different years from the same localities. *J. Berry Res.* **2019**, *9*, 377–394. [CrossRef]
48. Azhar, A.N.; Panirselvam, M.; Amran, N.A.; Ruslan, M.S.; Samsuri, S. Retention of total phenolic content and antioxidant activity in the concentration of broccoli extract by progressive freeze concentration. *Int. J. Food Eng.* **2020**, *16*, 20190237. [CrossRef]
49. Ahmed, F.E.; Hashaikeh, R.; Hilal, N. Hybrid technologies: The future of energy efficient desalination—A review. *Desalination* **2020**, *495*, 114659. [CrossRef]
50. Correa, L.J.; Ruiz, R.Y.; Moreno, F.L. Effect of falling-film freeze concentration on bioactive compounds in aqueous coffee extract. *J. Food Process. Eng.* **2018**, *41*, e12606. [CrossRef]
51. Ribera, A.E.; Reyes-Diaz, M.; Alberdi, M.; Zuñiga, G.E.; Mora, M.L. Antioxidant compounds in skin and pulp of fruits change among genotypes and maturity stages in highbush blueberry (*Vaccinium corymbosum* L.) grown in Southern Chile. *J. Soil Sci. Plant Nutr.* **2010**, *10*, 509–536. [CrossRef]
52. Hashemi, S.M.B.; Khaneghah, A.M.; Barba, F.J.; Nemati, Z.; Shokofti, S.S.; Alizadeh, F. Fermented sweet lemon juice (*Citrus limetta*) using *Lactobacillus plantarum* LS5: Chemical composition, antioxidant and antibacterial activities. *J. Funct. Foods* **2017**, *38*, 409–414. [CrossRef]
53. Jaster, H.; Arend, G.D.; Rezzadori, K.; Chaves, V.C.; Reginatto, F.H.; Petrus, J.C.C. Enhancement of antioxidant activity and physicochemical properties of yogurt enriched with concentrated strawberry pulp obtained by block freeze concentration. *Food Res. Int.* **2018**, *104*, 119–125. [CrossRef] [PubMed]
54. Demoliner, F.; de Carvalho, L.T.; de Liz, G.R.; Prudêncio, E.S.; Ramos, J.C.; Bascuñan, V.L.A.F.; Vitali, L.; Block, J.M. Improving the nutritional and phytochemical compounds of a plant-based milk of sapucaia nut cake using block freeze concentration. *Int. J. Food Sci. Technol.* **2020**, *55*, 3031–3042. [CrossRef]
55. Petzold, G.; Aguilera, J.M. Centrifugal freeze concentration. *Innov. Food Sci. Emerg. Technol.* **2013**, *20*, 253–258. [CrossRef]
56. Vuist, J.E.; Boom, R.M.; Schutyser, M.A. Solute inclusion and freezing rate during progressive freeze concentration of sucrose and maltodextrin solutions. *Dry. Technol.*. accepted. [CrossRef]
57. Casas-Forero, N.; Orellana-Palma, P.; Petzold, G. Recovery of solutes from ice fractions in centrifugal block cryoconcentration applied to blueberry juice. *Food Bioprocess. Technol.* under review.