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Research Article

Mathematical Modeling for Cactus Pear Juice Concentration Kinetics and a Study of the Physicochemical Changes During the Concentration Process

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Cactus pear juice was concentrated by applying a rotary vacuum evaporator at different temperatures (50°C, 60°C, 70°C, and 80°C). The kinetics of the juice concentration based on the change in total soluble solid content was investigated by applying five mathematical models. The modeling of this process was essential to accurately describe the evolution of total soluble solids and to identify the most suitable model for predicting concentration behavior. The constants of the mathematical models were estimated with nonlinear regression and according to the determination of three statistical parameters: the coefficient of determination (R^2), the adjusted R^2 (adj R^2), and the root mean square error (RMSE); it was revealed that the model known as Weibull distribution is the best descriptive one ($R^2 > 0.980$, adj $R^2 > 0.971$, and RMSE < 2.12). The activation energy for the concentration process was estimated at 254.09 kJ/mol. To study the effect of the vacuum concentration on physicochemical characteristics and on bioactive compounds, several parameters were determined, namely, total soluble solid content, moisture, pH, acidity, viscosity, ascorbic acid, phenolic compounds, betalains, antioxidant activity, and reducing power. The results showed a significant increase in acidity (from 0.037 to 0.129 g citric acid eq/100 mL) and viscosity (from 0.048 to 0.275 Pa·s), after juice concentration. Bioactive compounds were concentrated along with the increase in °Bx value (from 15 to 60°Bx), with betalains reaching up to 59.66 mg/100 g, total phenolic compounds up to 246.74 mg GAE/100 g, and ascorbic acid up to 39.36 mg/100 g in the juice concentrates. However, statistical evaluation of the results made for the reconstituted juices from juice concentrates revealed that the bioactive compounds and the antioxidant activity of the cactus pear juice were better preserved when applying the lowest temperatures (50°C and 60°C).

Keywords: antioxidant activity; betalains; juice concentrate; modeling; *Opuntia ficus-indica*; phenolic compounds

1. Introduction

Opuntia ficus-indica (also known as cactus pear (CP) and prickly pear) is a plant associated with the semiarid and arid zones of the world but also well adapted to the climate of the Mediterranean zone [1, 2]. This plant possesses the particularity of being able to be cultivated in zones that offer very little growth possibility of other fruits [3, 4]. *O. ficus-indica* is most often used for fruit production, because of its nutritional properties [5]. CP fruits are also widely used for seed oil production [6, 7]. However, the production of seed oil alone leads to the discarding of the rest of the fruit. The CP fruit is one of the rare natural sources of betalains, a class of yellow and orange pigments, thus the fruits being an alternative to synthetic pigments. In addition, the consumption of betalains is beneficial to human health due to their antioxidant properties [8–10].

The nutritional properties of CP fruit are also related to relevant ascorbic acid (AA) amount, free amino acids, and phenolic compounds and its high amounts of calcium and magnesium [1, 11–13]. Moreover, its sugar content is higher than prune, peach, and apricot [4]. Furthermore, the high glucose amount in CP fruit can be a good energy source for nerve cells and the brain, while fructose enhances the fruit's sweetening power [1, 14].

Regarding technological properties of the CP fruit, it is considered as a useful component for production of juice concentrates (from here on referred to as "concentrates") and dehydrated products because of its important sugar concentration [5, 15]. In addition, CP concentrate might be incorporated in several products, such as fruit syrup, jellies, and ice creams [16].

Fruit juice concentration is one of the processes where food stabilization is ensured by reducing water activity (increasing the soluble solids content) and minimizing microorganisms' growth [5, 17]. Juice concentration techniques play a crucial role in improving shelf-life and reducing storage and transportation costs. Various methods are commonly employed, including reverse osmosis, osmotic membrane distillation, and vacuum evaporation. Reverse osmosis, a membrane-based method, allows for juice concentration without thermal degradation, but it requires high operational pressures [16]. Osmotic membrane distillation is a method that utilizes a selective membrane to separate water vapor from juice, reducing thermal degradation during concentration [5, 16]. Vacuum evaporation, the method used in this study, is widely applied in the food industry as it facilitates concentration at lower temperatures, thereby preserving heat-sensitive bioactive compounds such as phenolics and betalains [17, 18].

In the CP concentrate production, the required final soluble solid content is achieved using vacuum evaporators [18]. Therefore, the choice of the process temperature is an important parameter to preserve bioactive compounds of the processed fruit.

Recently, many studies have described the advantages of mathematical modeling and kinetics in order to control and understand the drying and concentration processes [19]. In fact, empirical or physical models are widely used to describe the evaporation process. The theoretical modeling, also known as physical modeling, is based on physical principles and flow of the fluid [19, 20].

Several researchers have studied the modeling of food drying/concentration process by different models such as Page, Henderson and Pabis, Weibull distribution, parabolic, and Wang and Singh [21–25]. Nevertheless, there are no studies in the literature concerning the kinetic modeling of CP juice concentration.

At lab scale, the rotary vacuum evaporator is widely used in the concentration process due to its ability to operate at lower temperatures, thus preserving heat-sensitive compounds and improving the overall quality of the final product. The study of concentration kinetics in juice processing is essential for optimizing product quality and efficiency in the food industry. Understanding the behavior of CP juice under different processing conditions can help in enhancing its nutritional attributes while minimizing energy consumption. By investigating the concentration kinetics and related changes in physicochemical properties, this study intended to provide valuable insights for the development of more effective and sustainable juice concentration techniques.

Although the effect of the temperature on bioactive compounds and vitamins has been widely studied, its specific impact during the vacuum concentration of CP juice is limited. This study integrates statistical analysis to assess bioactive compound retention and antioxidant properties in "concentrates" as well as in reconstituted juices. On the other hand, despite extensive studies on modeling food drying and concentration processes, no research has specifically addressed the kinetic modeling of CP juice concentration. The lack of literature on this topic highlights the need for a deeper understanding of its concentration dynamics.

This study had two main objectives. Firstly, the concentration kinetics of CP juice was investigated using a rotary vacuum evaporator by evaluating the change in TSS content. To achieve this, five different mathematical models were tested to describe the change in TSS concentration over time and find the most appropriate model.

The second objective focused on evaluating the effect of the concentration process on the physicochemical properties of both the CP concentrates and the reconstituted juices derived from them. Particular attention was paid to examine alterations related to the levels of bioactive compounds and antioxidant activity during the concentration process. The corresponding results were assessed using both univariate and multivariate statistical approaches.

2. Materials and Methods

2.1. Preparation of Samples

2.1.1. Concentration of the CP Juice. Sixty kilograms of fruits (*O. ficus-indica*) were harvested at full maturity from the Bouzoulem agricultural field in El Kseur, Bejaia, located in the northeast of Algeria. The harvest was conducted through random samplings from 20 plants. The CP juice was then prepared and stored according to the procedures outlined in [5]. The obtained CP juice with initial total soluble solid

TABLE 1: Mathematical models applied to cactus pear juice concentration.

Model name	Model equation	Eq.	Reference
Page	$B - B_0 = \exp(-k \cdot t^n)$	(1)	[21] Page
Henderson and Pabis	$B - B_0 = a \cdot \exp(-k \cdot t)$	(2)	[22] Henderson and Pabis
Weibull distribution	$B - B_0 = a - b \cdot \exp[-(k \cdot t^n)]$	(3)	[23] Babalis et al.
Parabolic	$B - B_0 = a + bt + ct^2$	(4)	[24] Sharma and Prasad
Wang and Singh	$B - B_0 = 1 + at + bt^2$	(5)	[25] Wang and Singh

Note: t: concentration time (minute); B: total soluble solid (TSS) content at time t (°Brix); B_0: initial total soluble solid (TSS) content (°Brix); k, a, n, b, c: constants.

(TSS) content approximately of 15°Brix was concentrated until a TSS content of about 60°Brix, using a laboratory rotary vacuum evaporator (Rotavapor R-210, Büchi Advanced, Switzerland). The experiments were carried out in triplicate at four different temperatures (50°C, 60°C, 70°C, and 80°C) and under a constant pressure of 72 mbar. The concentrate was packed in HDPE plastic bottles and stored at +4°C until the analysis.

2.1.2. Reconstitution of Juice From Concentrates. Reconstituted juices with the same initial TSS content of 15°Brix were prepared from CP concentrates through a dilution with deionized water. TSS content was measured regularly during concentration, and samples were taken from juice at four or five stages of the concentration and analyzed for AA, betalains, and moisture content.

CP fresh, concentrates, and reconstituted juices were all analyzed. For each chemical parameter, the analyses were performed in triplicate, and for each sample, tree aliquots were analyzed.

2.1.3. Kinetic Models of Concentration. CP juice concentration curves were fitted to five mathematical models, namely, Page, Henderson and Pabis, Weibull distribution, parabolic, and Wang and Singh. The mathematical expressions (1–5) for these models are shown in Table 1.

The Page model is a semiempirical model commonly used in drying and concentration kinetics, incorporating an empirical exponent to improve accuracy. The Henderson and Pabis model is a simple exponential model describing the moisture ratio decrease over time, often applied in drying studies. The Weibull distribution model is a probabilistic approach frequently used for nonlinear kinetics in food processing, allowing flexibility for various process conditions. The parabolic model represents concentration kinetics using a second-order polynomial equation, and finally, the Wang and Singh model is a diffusion-based approach that describes the moisture content variation over time using a second-degree equation.

2.2. Analytical Methods

2.2.1. TSS Content. The TSS content in the samples was measured according to AOAC [26], using an Abbe-refractometer (AR-12, Schmidt & Haensch) and expressed as °Bx. Each determination was made in triplicate.

2.2.2. Moisture Content. The moisture content determination in the analyzed samples was made according to AOAC [26]. The procedure consists of placing 2 g of the sample in a ventilated oven at 105 ± 2°C at atmospheric pressure, until constant weight is obtained. Each determination was made in triplicate.

2.2.3. Determination of pH. The determination of the pH value of each sample was performed according to AOAC [26]. The measurements were made in triplicate at a room temperature (~25°C) using a pH meter (PHM 210, Hanna Instruments, France), and the electrode was placed directly into each sample. The pH meter was first standardized using proper buffer solutions.

2.2.4. Titratable Acidity. The titratable acidity was determined as described by Friedrich [27]. The sample was centrifuged for 5 min at 700 *g* at 5°C. Then, 10 mL of the clear supernatant was introduced into conical flask, 100 mL of deionized water was added, and the solution was shaken on a magnetic stirrer plate. Subsequently, juice sample was titrated against a 0.1 N NaOH solution until 8.2 pH, using a pH meter. The titratable acidity was calculated using Equation (1):

$$\mathrm{TA(g/100\,mL)} = \frac{(V)(N)(\mathrm{meq.\,wt.})(100)}{1000(v)} \quad (1)$$

where V is the volume of the used NaOH solution (in milliliter), N is the normality of the NaOH solution; meq. wt. is the milliequivalent weight of the standard (citric acid), and v is the sample volume (in milliliter).

2.2.5. Viscosity Measurement. The determination of the CP juice viscosity before and after the concentration process was performed using a SNB-1 digital viscometer (Princeton Instruments) at room temperature and expressed as pascal-second. The measurements were made in triplicate for each sample.

2.2.6. Determination of the Concentration of the Main Betalain Classes. The content of the main betalain classes was realized according to the method described by Khatabi et al. [28]. A mixture of sample and methanol (1 : 5 *v*/*v*) was prepared and homogenized through magnetic stirring. Then, the solution was filtered using a syringe filter (0.45 μm). Finally, the absorbance values of the extracts were measured at two wavelengths, 482 nm for betaxanthin and 532 nm for indicaxanthin, using a UV-visible spectrophotometer (SpectroScan 50, Biotech Engineering Management

Company Limited, United Kingdom). The quantification of the betalain concentrations was made according to Lambert–Beer's law (2):

$$A = \varepsilon \Lambda c \quad (2)$$

where A is the absorbance, ε is the molar extinction coefficient ($L\,mol^{-1}\,cm^{-1}$), Λ is the optical path length, and c is the molar concentration ($L\,mol^{-1}$).

The molar extinction coefficients were 48,000 $L\,mol^{-1}\,cm^{-1}$ for indicaxanthin and 62,000 $L\,mol^{-1}\,cm^{-1}$ for betaxanthin. The results were expressed in $mg_{betalain}/100\,g$ of the juice sample.

2.2.7. AA Determination. The AA concentration was determined according to the method described by Mau et al. [29]. AA was extracted from 1 g of sample with 30 mL of oxalic acid (0.4% *w*/*v* in water). An aliquot of 300 μL of sample extract was mixed with 2.7 mL of 2,6-dichloroindophenol (0.006% *w*/*v* in water). The absorbance was measured at 515 nm using a UV-visible spectrophotometer. The content of AA was calculated based on the calibration curve of AA, and the results were expressed as $mg_{AA}/100\,mL$ of the sample.

2.2.8. Total Phenolic Content (TPC). The TPC level was assessed via the Folin–Ciocâlteu method [30]. Each sample (20 g) was homogenized with a solution of acetone–water, 7 : 3 *v*/*v* for 30 min. Then, the solutions obtained were filtered using a syringe filter (0.45 μm). Thereafter, a volume of 2.5 mL of diluted Folin–Ciocâlteu reagent (1/10) was mixed with 200 μL of each extract and 300 μL of deionized water. The incubation of the mixture was made at room temperature for 2 min, and then, 2 mL of the sodium carbonate solution (75 g/L) was added. After that, the incubation was made in the darkness at 50°C for 15 min, and finally, the sample was cooled down in a water ice bath up to room temperature. The absorbance values were immediately read at 760 nm using a UV-visible spectrophotometer. The TPC was expressed as milligram gallic acid equivalents (GAE)/100 g juice f.w. according to a calibration curve realized with standard solutions of pure gallic acid.

2.3. Antioxidant Activity. The antioxidant activity was assessed using the free radical DPPH (1,1-diphenyl-2-picrylhydrazyl) [31]. An aliquot of 0.1 mL of each juice extract (prepared with a solution of acetone–water, 7 : 3 *v*/*v*) was added to 3 mL of a methanol solution of 6×10^{-5} M DPPH, and the mixture was incubated for 15 min at room temperature in the darkness. The measurements of the absorbance values of the extracts were obtained using a UV-Vis spectrophotometer.

The absorbances were transformed into inhibition percentage of the DPPH concentrations so that the results are expressed as $mg_{GAE}/100\,g$ juice f.w. according to a calibration curve realized with standard solutions of pure gallic acid.

2.4. Reducing Power Assay. The reducing power of the CP fresh juices and the concentrates was evaluated using the method described by Rohman et al. [32]. The same extracts analyzed for the TPC (2.3.8) and for antioxidant activity (2.3.9) were used to determine the reducing power of the samples. Each extract (1 mL) was added to 2.5 mL of a phosphate buffer solution (pH 6.6, 200 mM) and 2.5 mL of a potassium ferricyanide solution (1%) and incubated for 20 min at a temperature of 50°C. The obtained mixture was added with 2.5 mL of a TCA solution (10%) and then centrifuged for 10 min at 1000 *g*. A volume of 2.5 mL of the obtained supernatant was added with 2.5 mL of deionized water and 0.5 mL of a solution of $FeCl_3$ (0.1%). Finally, absorbances were read at 700 nm using a UV-visible spectrophotometer. The reducing power was expressed as $mg_{GAE}/100\,g$ juice f.w. according to a calibration curve realized with standard solutions of pure gallic acid.

2.5. Statistical Analysis. In the present work, the constants of the mathematical models were estimated through nonlinear regressions using the statistic program Curve Expert Professional (version 2.6.3, United States). The evaluation of the effectiveness of each fit was made based on three statistical parameters: the coefficient of determination (R^2), the adjusted R^2 (adj R^2), and the root mean square error (RMSE) (3).

$$\mathrm{RMSE} = \sqrt{\frac{1}{n-p}\sum_{i=1}^{n}\left(Y_{\mathrm{pred}} - Y_{\mathrm{Exp}}\right)^2} \quad (3)$$

where n is the number of data points, p is the number of parameters, Y_{pred} is the model predicted value, and Y_{Exp} is the experimental value.

The experimental results of the physicochemical analyses were expressed as mean values ± standard deviation, for triplicate measurements. Differences among samples were determined using the one-way ANOVA, followed by Tukey's honestly significant difference (HSD) at $p < 0.05$ using JMP program (version 7.0, United States).

Furthermore, the results of chemical analyses performed on fresh and reconstituted juices were elaborated using principal component analysis (PCA) [33]. This step allowed to gain a global evaluation of the variation in bioactive compounds and antioxidant activity between fresh and reconstituted juices. Before PCA, the dataset was preprocessed using autoscaling and the model was calculated using the PLS Toolbox software (v8.8.1, Eigenvector Inc.) working in MATLAB environment (The MathWorks).

3. Results and Discussions

3.1. Moisture and TSS. Figure 1 illustrates the variation in TSS content (Figure 1a) and moisture content (Figure 1b) in CP juice samples as they undergo vacuum concentration over time at four different temperatures. The concentration rate exhibits an upward trend, aligning with the increase in temperature. This behavior can be attributed to the increased molecular velocity (acceleration) associated with the rise in temperature [19, 34].

The final concentration, approximately 60°Brix, corresponding to a moisture content of around 40%, was achieved

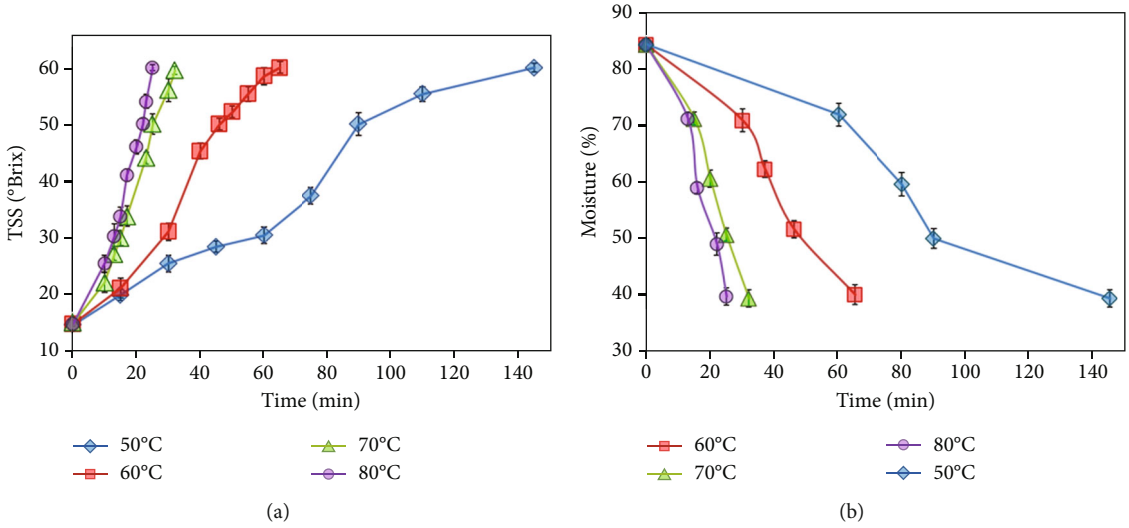

FIGURE 1: Total soluble solid (TSS) content (a) and moisture content (b) of cactus pear juice versus time during concentration applying four temperatures (50°C, 60°C, 70°C, and 80°C).

within different processing times: 145, 65, 32, and 25 min at temperatures of 50°C, 60°C, 70°C, and 80°C, respectively. By contrast, Moßhammer et al. [17], employing a rotary vacuum evaporator at 25°C for CP concentrate production, reported a longer processing time of 6 h to reach the desired final concentration (65°Brix). This difference is clearly due to the variations in the applied temperatures between their study and the current work and, secondarily, possibly to differences in operational pressure.

3.2. Modeling of the Juice Concentration

3.2.1. Selection of Mathematical Models. In the literature, various mathematical models are available for kinetic modeling. In this work, five models commonly used were applied to estimate the CP juice concentration, based on TSS change (Table 1). The choice of these mathematical models was based on their ability to describe different types of drying and concentration kinetics. The Weibull distribution model is particularly useful for describing nonlinear behavior and has been applied successfully in food processing studies. The Page model is known for its empirical adaptability, while the Henderson and Pabis model provides a simple exponential approach. The parabolic and Wang–Singh models allow for a more polynomial-based representation of concentration kinetics [21–25].

Several authors have reported that the Weibull distribution model is a suitable model for different foods drying kinetic [20, 23, 35–37]. Conversely, Lahsasni et al. [38] have concluded that the two-term drying model described adequately the CP fruit drying kinetic. However, all the research studies cited previously have referred to moisture change during the drying processes, and limited works have been achieved for the modeling of the juice concentration process based on TSS content change. Assawarachan and Noomhorm [39] have reported that "the modified Page" was the best descriptive model for pineapple juice concentration using a vacuum-microwave, Goula et al. [19] reported that the evaporation rate was better described by the logarithmic model for concentration kinetics of pomegranate juice using a rotary vacuum evaporator, and finally, Maskan [40] used the three parameter exponential equation to describe the TSS concentration change during production of pomegranate juice concentrate.

3.2.2. Results of the Statistical Analysis. Table 2 shows the results of the statistical analyses. The R^2, the adj R^2, and the RMSE values were determined to evaluate the goodness of each fit. According to Table 2, R^2, adj R^2, and RMSE values varied from 0.867 to 0.996, 0.845–0.995, and 0.992–5.879, respectively.

Figure 2 depicts the mathematical model curves at the four different temperatures. The effectiveness of the simulation provided by the models improved with the increase of the process temperature. Similar observation was noticed by Babalis et al. [23] for the Page and Henderson and Pabis models. The Page, Henderson and Pabis, and Wang and Singh models all gave a good fit at higher temperatures, while at low temperatures these models tended to overestimate in the early stages and underestimate in the later stages of the concentration. The parabolic model was a good fit for 60°C, 70°C, and 80°C. In fact, at 50°C, the parabolic model underestimated in some stages and overestimated in other stages of the concentration (Figure 2).

The Weibull distribution was the best descriptive model as its curves tended to fit well, and the RMSEs of this model were the smallest values at all temperatures tested (Table 2).

Figure 3 shows that the data from the Weibull distribution model exhibited the highest R^2 values, confirming the results through the comparison of experimental data plotted against predicted data. Consequently, the Weibull distribution model adequately described the kinetic of the CP juice concentration.

3.2.3. Activation Energy Determination. Knowledge of the activation energy is necessary to predict the extent of

TABLE 2: Found values of the constants of the applied models for cactus pear juice concentration and results of the statistical analysis.

Temperature (°C)	Constants				Statistical parameters		
					R^2	Adj R^2	RMSE
			Page				
	k (min^{-1})	n (–)					
50	−0.866	0.302			0.935	0.925	4.101
60	−0.972	0.332			0.943	0.934	3.572
70	−0.891	0.421			0.961	0.955	3.035
80	−0.806	0.484			0.988	0.985	1.462
			Henderson and Pabis				
	a (–)	k (min^{-1})					
50	9.487	−0.0115			0.867	0.845	5.879
60	9.337	−0.0259			0.887	0.869	5.024
70	6.394	−0.0628			0.933	0.921	4.021
80	5.387	−0.0866			0.979	0.974	1.939
			Weibull distribution				
	a (–)	b (–)	k (min^{-1})	n (–)			
50	45.60	37.71	$2.18 * 10^{-7}$	3.432	0.982	0.979	2.119
60	45.841	41.922	$1.89 * 10^{-5}$	2.920	0.995	0.994	0.992
70	46.692	43.309	$1.32 * 10^{-4}$	2.872	0.996	0.995	0.965
80	145.39	143.90	$8.19 * 10^{-4}$	1.893	0.993	0.992	1.228
			Parabolic				
	a (–)	b (min^{-1})	c (min^{-2})				
50	−2.038	0.398	$-3.98 * 10^{-4}$		0.951	0.943	3.568
60	−12.992	1.280	$-5.60 * 10^{-3}$		0.982	0.979	1.987
70	−17.216	2.490	$-1.70 * 10^{-2}$		0.990	0.988	1.510
80	−2.678	0.923	$3.77 * 10^{-2}$		0.993	0.992	1.236
			Wang and Singh				
	a (min^{-1})	b (min^{-2})					
50	0.318	$5.95 * 10^{-5}$			0.947	0.938	3.710
60	0.576	$2.36 * 10^{-3}$			0.954	0.946	3.193
70	0.632	$2.49 * 10^{-2}$			0.969	0.964	2.699
80	0.469	$5.29 * 10^{-2}$			0.990	0.988	1.291

Note: R^2: coefficient of determination; adj R^2: adjusted R^2; k, a, n, b, c: constants.
Abbreviation: RMSE, root mean square error.

chemical reactions and consequently, to optimize it [41]. In this context and to better evaluate the temperature effect on the juice concentration kinetics, the temperature dependence of the Weibull distribution rate constant can be represented by an Arrhenius relationship (Equation (4)).

$$k = k_0 \cdot e^{-E_a/RT} \quad (4)$$

where k is the rate content, given by the Weibull distribution model, k_0 is the frequency factor (per minute), E_a is the activation energy (joule/mole), R is the universal gas constant (8.314 $J \cdot K^{-1} \cdot mol^{-1}$), and T is the temperature (in Kelvin).

The constant k used in this study corresponds to the rate parameter of the Weibull distribution model, which was identified as the most suitable model for describing the concentration kinetics of CP juice. This constant represents the juice concentration rate and was determined by fitting the experimental curves of TSS variation over time. The estimation of k was performed using nonlinear regression through the Curve Expert Professional software.

The logarithm of the rate constant versus the reciprocal of the temperature is shown in Figure 4. The results show a linear relationship due to the Arrhenius type dependence ($R^2 = 0.9565$). From the slope of this line, an activation energy value of 254.09 kJ mol^{-1} was determined. Similar observation was reported by Corzo et al. [20], who reported an activation energy of 214.93 kJ mol^{-1}, when applying the Weibull distribution model.

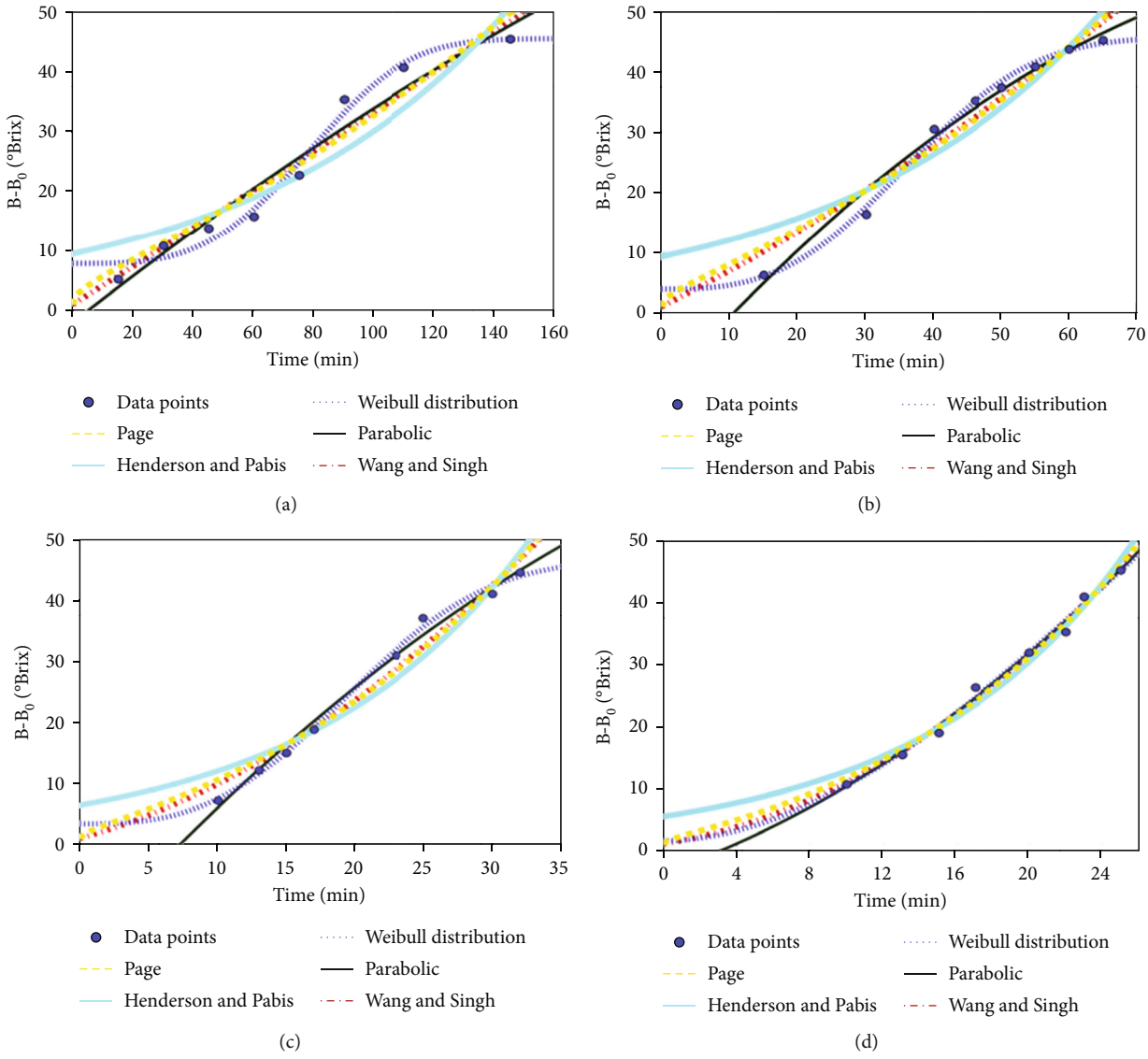

FIGURE 2: Experimental data and mathematical model curves of TSS content change at 50°C (a), 60°C (b), 70°C (c), and 80°C (d) during cactus pear juice concentration.

3.2.4. pH, Titratable Acidity, and Viscosity. Table 3 shows pH values and titratable acidity of CP fresh juice and concentrate. The pH value observed in the fresh juice was 6.52 ± 0.05, similar to the results obtained by Gurrieri et al. [42].

The titratable acidity of CP juice was 0.037 ± 0.004 $mg_{citric\ acid\ eq}$/100 mL. This value is similar to that reported by Chougui et al. [43] and Hernández-Pérez et al. [44]. It is well known that the CP juice acidity is rather low compared to other fruits such as grapes, pineapple, grapefruit, and orange, which are commonly used to be transformed into juices [45].

As expected, the pH of the juice was significantly higher than the pH values observed in the CP concentrates, while the juice acidity increased significantly after concentration. These results are the natural effect of the concentration of organic acids in the juice after water evaporation. Similar observation was reported by Hojjatpanah et al. [46] referring to black mulberry juice concentration. However, the low initial acidity, combined with the lower concentration temperatures, makes it possible to limit the formation of furanic compounds and acrylamide at the same time [47, 48].

As shown in Table 3, the measured viscosity of the CP juice was 0.048 Pa·s. This value is close to that noticed by Sepúlveda and Sáenz [49] for CP var. *Orange* pulp (0.045 Pa·s). Sáenz et al. [1] have reported that pectin (0.17–0.21 g/100 g) is partially responsible for viscosity of the CP pulp and is a useful ingredient to produce jams and jellies. More generally, the viscosity of the pulp is influenced by the presence of mucilaginous substances, which have the ability to retain and bind water [50].

In the present work, the viscosity of the concentrates reached values above 0.27 Pa·s, that is, 5.4 times higher than the viscosity of fresh juice. It is documented in the literature that the viscosity of the juice increases exponentially with the rise in TSS content [51, 52].

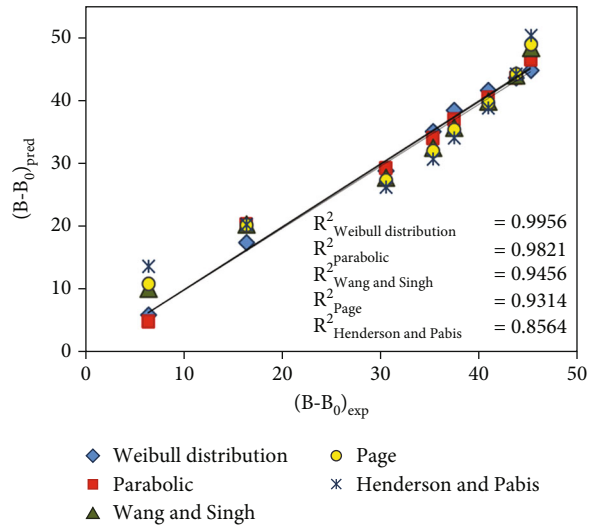

FIGURE 3: Experimental and predicted values of TSS content change provided by different models at 60°C.

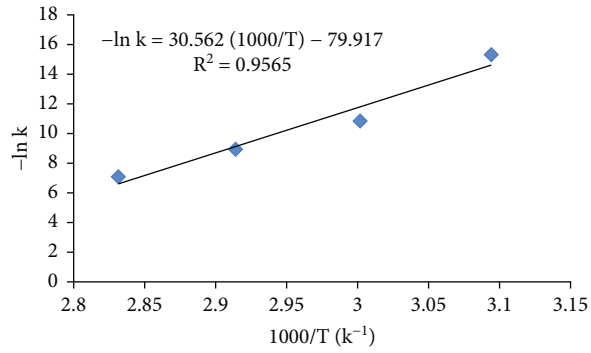

FIGURE 4: Correlation between the natural logarithm of rate constants (given by the Weibull distribution model) and the reciprocal of the absolute temperature.

3.3. Bioactive Compounds

3.3.1. Betalains. Betalains are the main pigments in cactus fruit juice, and their preservation during different thermal treatments could be used as a good indicator regarding product quality aspects. However, the preservation of betalains during the concentration process of CP juice is a remarkable challenge due to the sensitivity of these pigments to heat. On the other hand, the determination of the betalain content in CP concentrate is crucial due to its exploitation as a natural food colorant.

The determined betaxanthin and indicaxanthin contents in different samples (fresh, concentrates, and reconstituted juices) at different temperatures are presented in Figure 5a,b. According to the obtained results, the mean content of betaxanthin in the fresh juice was 13.88 ± 0.94 mg/100 g, while the indicaxanthin content was 0.59 ± 9.97 mg/100 g. Several authors have already reported the presence of similar level of betalains in CP juice. Indeed, values reported in the literature range from 3.78 to 25 mg/100 g for betaxanthin and from 0.13 to 1.04 mg/100 g for indicaxanthin [53–56].

The betaxanthin content in concentrates produced at different temperatures varied from 49.19 ± 1.23 to 59.66 ± 1.71 mg/100 g, while indicaxanthin content varied from 1.81 ± 0.08 to 2.26 ± 0.07 mg/100 g. That reveals the heat treatments' effect on the CP juice pigments. Moreover, the reconstituted juices from concentrates produced at 70°C and 80°C had significantly lower betalains content than the initial fresh juice. However, the preservation of both pigments was noticed when applying the temperature of 50°C. Similarly, it was reported in the literature that betalain stability can be impacted by thermal concentration processes, thus resulting in color loss and a decrease in antioxidant capacity [17, 57, 58].

The instability of the betalain pigments has been studied by several authors. Joubert [59] has noticed that the indicaxanthin content in CP juice decreased steadily with increasing temperature and heating time. Fernández-López and Almela [54] have studied the effect of temperature on the betaxanthin content and noticed that the lowest recovery was found at the highest temperatures (80°C–100°C). Ruiz-Gutierrez et al. [60] and Cruz-Bravo et al. [61] have reported that the temperature is the main factor affecting color stability of betalain pigments, and finally, according to Dehbi et al. [62], the loss in betalains and, in turn, yellow intensity during heat treatment can be attributed to a degradation due to isomerization, cleavage, or decarboxylation.

On the other hand, in the present work, betalain content significantly increased during the concentration process (Figure 6a,b), thus indicating that these pigments were concentrated during water evaporation. Nevertheless, content variations of both pigments depended on the applied temperatures, so the temperature as low as 50°C yielded higher betalain contents at all concentration stages when compared to higher temperatures. Moreover, the application of vacuum during the concentration process is crucial for preserving the quality of CP juice. By reducing the boiling point of water, it allows for concentration at lower temperatures, minimizing the thermal degradation of sensitive compounds like betalains. This technique helps maintain higher levels of these pigments, preserving the natural color and antioxidant properties of the juice.

3.3.2. AA Determination Results. High temperatures cause significant degradation of AA, thereby reducing the nutritional value of CP juice. Figure 5c illustrates the AA content in CP fresh, concentrates, and reconstituted juices at different temperatures. According to the obtained results, the AA content in the fresh juice was 7.5 ± 0.2 mg/100 g. As a matter of fact, CP contains a higher level of vitamin C compared to other fruits, such as pear, apple, banana, and grapes [63]. The determination of the AA content in food during treatments is an index of the nutritional quality because of its instability during thermal processing [35].

Figure 5c shows that the CP concentrates presented higher AA level than not processed fresh juice and varied from 20.21 ± 1.06 mg/100 g (in the CP concentrate at 80°C) to 39.36 ± 1.06 mg/100 g (in the concentrate at 50°C). Moreover, to assess in more detail the impact of applied

TABLE 3: pH, acidity, and viscosity in cactus pear juice and concentrates.

	Fresh juice	Concentrates			
		50°C	60°C	70°C	80°C
pH	6.52 ± 0.05a	5.96 ± 0.06b	5.92 ± 0.07b	5.99 ± 0.06b	6.02 ± 0.05b
Titratable acidity $g_{citric\ acid\ eq}$/100 mL	0.0373 ± 0.004c	0.123 ± 0.005ab	0.129 ± 0.003a	0.117 ± 0.006ab	0.113 ± 0.004b
Viscosity (Pas·s)	0.048 ± 0.001b	0.274 ± 0.005a	0.275 ± 0.004a	0.276 ± 0.005a	0.273 ± 0.003a

Note: Concentrates: juice concentrated until a total soluble solid (TSS) content of 60°Brix. Same letters refer to means not statistically different according to ANOVA and Tukey's test (a > b > c), at $p < 0.05$.

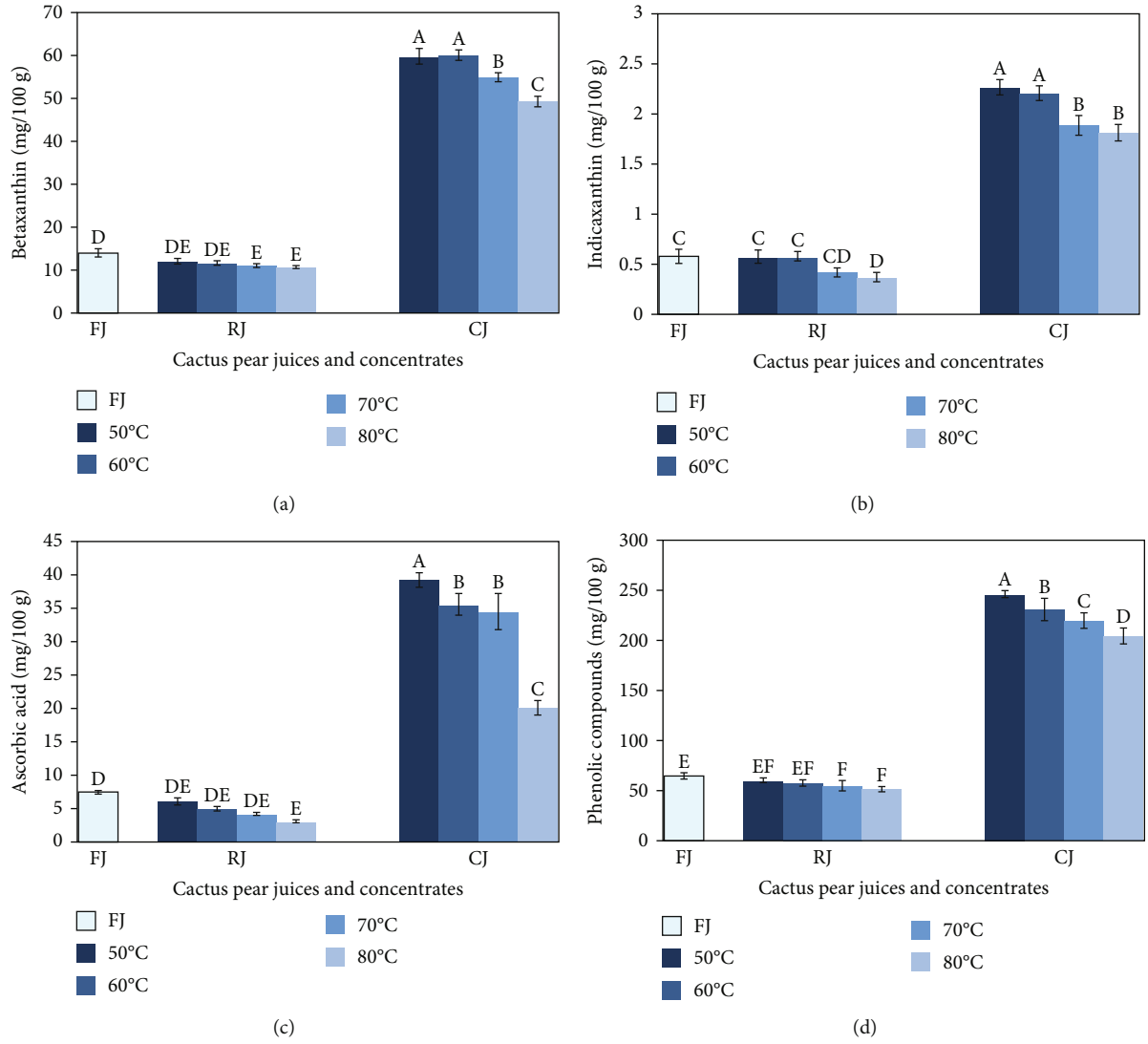

FIGURE 5: Bioactive compound levels: betaxanthin (a), indicaxanthin (b), ascorbic acid (c), and phenolic compounds (d) in different cactus pear juice samples: FJ (fresh juice), RJ (reconstituted juice), and CJ (concentrate until 60°Brix). Same letters refer to means not statistically different according to ANOVA and Tukey's test (A > B > C > D > E > F), at $p < 0.05$.

temperatures on the juice quality, the AA content was determined in the juices reconstituted from the CP concentrates (Figure 5c).

It was noticed that the prepared reconstituted juices from the concentrate carried out at 80°C showed the lowest AA level among all analyzed samples. In a similar context, da Silva et al. [64] have reported that an increase of the air-drying temperature increased the loss of AA. However, even studies using a vacuum concentration system, such as the one used by Moßhammer et al. [17], have shown a loss of as much as 90% in vitamin C content. This very high loss can be attributed to a long exposure to heat: the concentration phase in the rotary evaporator took about 6 h, which is a much longer process time than the process

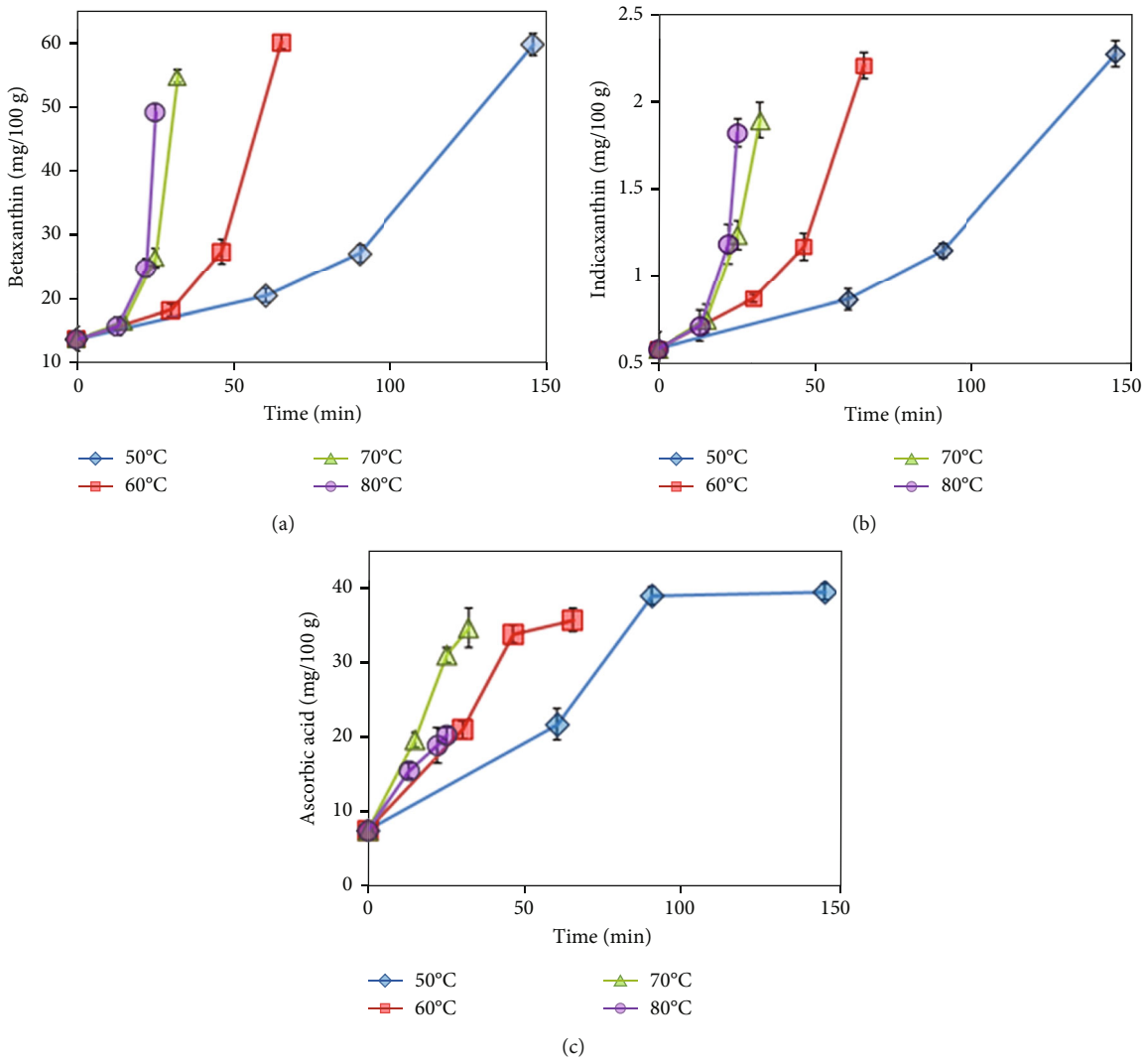

FIGURE 6: Bioactive compound level changes: betaxanthin (a), indicaxanthin (b), and ascorbic acid (c), during cactus pear juice concentration.

time described in the present work (25 to 145 min). AA is preserved in foods that undergo minimal heat treatment [65, 66].

According to Figure 6c, the AA level increased during juice concentration at all temperatures. Moreover, it was revealed that the higher the process temperatures, the lower the AA level in the juice. The lowest temperature (50°C) ensured the best recovering of AA compared to the higher temperatures. In fact, the preservation of AA is probably due to the ascorbate-sparing effect of the phenolic compounds [31].

The inverse relationship between juice concentration temperature and AA levels suggests significant implications for industrial applications, leading to the selection of lower processing temperatures and reduced pressure processing.

3.3.3. TPC. Preserving phenolic compounds in CP fruit juices is crucial due to their antioxidant properties and potential health benefits. These compounds not only enhance the nutritional value and sensory appeal of juices but also contribute to consumer perceptions of freshness and naturalness. Therefore, maintaining optimal processing conditions such as temperature and pressure to retain phenolic content is essential for ensuring the quality and health-promoting attributes of CP concentrate. Figure 5d shows the TPC values in the CP fresh, concentrates, and reconstituted juices at different temperatures. It was found a TPC amount of $64.81 \pm 3.21\ mg_{GAE}/100\ g$ of fresh CP juice. The TPC content increased significantly after the juice concentration, and it was noticed a significant difference among concentrates produced at different temperatures, ranging from 205.26 ± 7.71 to $246.74 \pm 3.52\ mg_{GAE}/100\ g$ of fresh CP juice. However, the reconstituted juices from concentrates produced at higher temperatures (70°C and 80°C) had significantly lower TPC when compared with the fresh juice. Indeed, it was reported in the literature that phenolic compounds are lost during food treatments due to several causes including heat degradation [46]. For this reason, a temperature of 50°C (at a pressure of 72 mbar) allowed for the most effective preservation of phenolic compounds in comparison to higher temperatures.

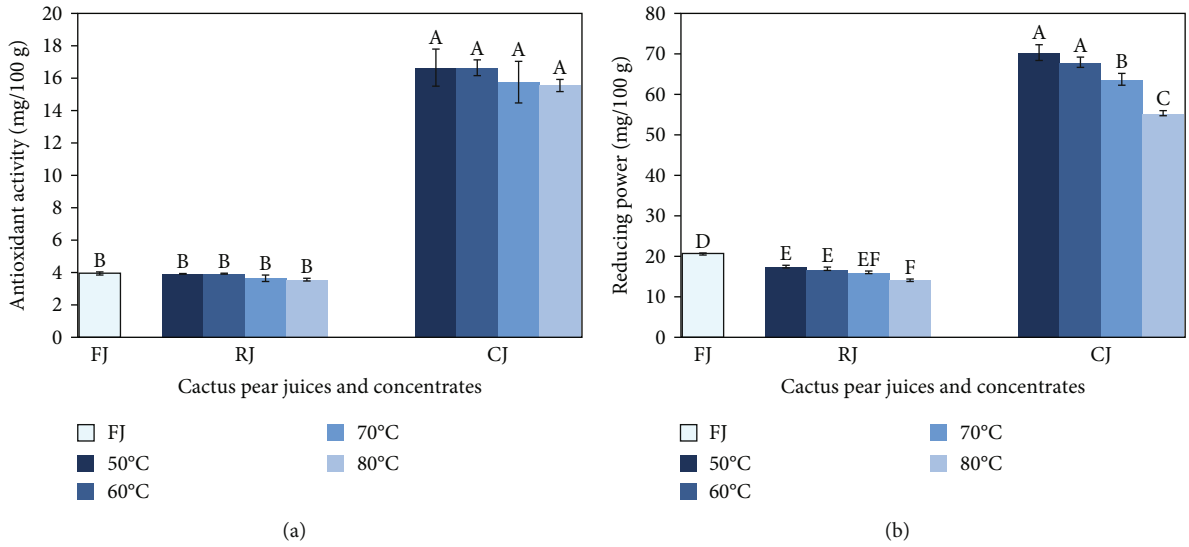

FIGURE 7: Antioxidant activity (a) and reducing power (b) of different cactus pear juice samples: FJ (fresh juice), RJ (reconstituted juice), and CJ (concentrate until 60°Brix). Same letters refer to means not statistically different according to ANOVA and Tukey's test (A > B > C > D > E > F), at $p < 0.05$.

3.3.4. Results of the Antioxidant Activity (DPPH). The measurement of antioxidant activity is crucial for assessing the potential health benefits of food samples [32]. Antioxidant activity reflects the ability of substances in the sample to neutralize harmful free radicals, thereby contributing to overall health and wellness. In the context of CP juice, understanding and enhancing its antioxidant capacity through concentration processing can lead to products with improved nutritional value and consumer appeal.

Figure 7a shows that fresh juice has an antioxidant activity of 3.94 ± 0.04 $mg_{GAE}/100$ g of fresh juice. The capacity of the CP to effectively reduce DPPH radical had already been confirmed in the literature [5, 31, 67].

As shown in Figure 7a, the antioxidant activity increased significantly after concentration, and it was noticed that there is no significant difference among concentrates produced at different temperatures. In addition, the reconstituted juices had similar antioxidant capacities in comparison with the fresh juice. On the other hand, the correlation between antioxidant activity and bioactive compound levels in samples showed highly significant correlation coefficients ($p < 0.001$) of 0.998, 0.952, 0.996, and 0.991 for TPC, AA, betaxanthin, and indicaxanthin, respectively. These results confirm that the antioxidant capacity of the CP juice and its concentrate was mainly linked to phenolic compounds and betalains. Piga et al. [31] have also noticed a high correlation of the antioxidant activity with AA and with phenolic compounds in the study of minimally processed CP fruits.

In summary, leveraging processing techniques to enhance antioxidant capacity in CP juice underscores its potential as a functional food. This approach not only preserves nutritional integrity but also meets consumer preferences for products with enhanced health benefits and antioxidant properties.

3.3.5. Reducing Power Results. The reducing power of the samples was determined by direct electron transfer in the reduction of ferricyanide to ferrocyanide [32]. Figure 7b shows that the fresh juice presented a reducing power of 20.62 ± 0.17 $mg_{GAE}/100$ g of fresh juice. The reducing power significantly increased after concentration, and it was noticed that the concentrates produced at the different temperatures displayed different reducing capacities, ranging from 70.42 ± 1.88 to 55.83 ± 0.68 $mg_{GAE}/100$ g of fresh juice.

The reconstituted juices showed lower reducing powers in comparison with fresh juice. As in the description of the previous parameters, it was noticed that the temperature of 50°C allowed the characteristics of the initial product to be better preserved. However, in agreement with the results based on the DPPH method, the reducing power assay also showed very high linear correlation coefficients with TPC ($r = 0.998$), betaxanthin ($r = 0.998$), and indicaxanthin ($r = 0.996$). The linear correlation between reducing power and AA was also very high ($r = 0.981$). Butera et al. [53] observed that the reducing power was mainly related to the betalain content. However, in the present study, the TPC and AA content are also of paramount importance.

3.3.6. Results of PCA Applied to Fresh and Reconstituted Juices. The PCA applied to the dataset of bioactive compounds (total phenolic compounds, AA, betaxanthin, indicaxanthin), antioxidant activity, and reducing power determined on fresh juices and concentrates was calculated considering two PCs, representing 94.25% of the explained variance (EV). The corresponding biplot is reported in Figure 8, where the loadings are reported in gray color while the scores represent the triplicate analyses performed on fresh juice (pink color in Figure 8) and on reconstituted juice obtained from the concentrate ones at 50°C (red color), 60°C (green color), 70°C (blue color), and 80°C (cyan color).

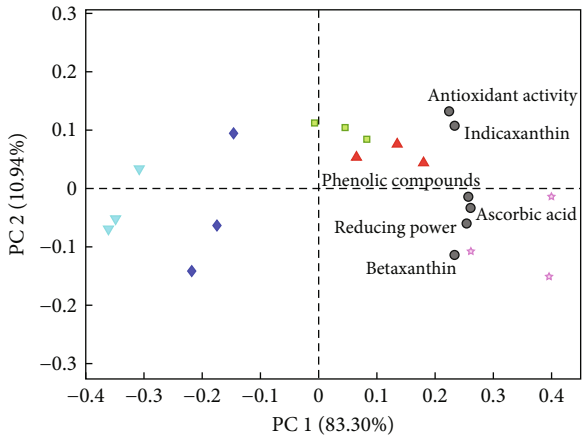

FIGURE 8: PC1 and PC2 biplot of the PCA model calculated on the dataset of the chemical analyses of fresh juice and reconstituted juices from concentrate obtained at 50°C, 60°C, 70°C, and 80°C.

PC1 (83.30% EV) describes a general decrease in the concentration of the variables measured from fresh to reconstituted juice, as already observed in the previous discussion. Concerning concentration temperature, the juices reconstructed from those concentrate carried out at 50°C and 60°C have a chemical composition closer to fresh juice, suggesting that the thermal treatment at temperatures equal or higher than 70°C determines a general degradation of phenolic and bioactive compounds, which in turn is reflected in a lower antioxidant activity and reducing power. Furthermore, antioxidant activity and indicaxanthin are less influenced to concentration at 50°C and 60°C since their value in the corresponding reconstituted juices is closer to fresh juice and much higher than the value observed for reconstituted samples from concentrate produced at 70°C and 80°C.

PC2 (10.94%) mainly describes the differences between triplicate analysis performed on the same sample. It is interesting to observe that triplicate analyses performed on juices reconstituted from concentrate produced at 50°C and 60°C have a lower variability from triplicate analyses performed juices reconstituted from concentrate obtained at 70°C and 80°C.

Therefore, based on the PCA results, it is possible to conclude that of course there are relevant differences between fresh and reconstituted juices; however, juices reconstituted from concentrate produced at 50°C and 60°C are better preserved and have a chemical composition closer to fresh juice. In addition, the reconstituted juices from concentrate obtained at 50°C and 60°C have an overall comparable chemical composition in terms of bioactive compounds. Considering that at 50°C the concentration time is almost double the concentration time at 60°C (145 min at 50°C vs. 65 min at 60°C), it is possible to perform the concentration of CP juice at 60°C without having a relevant decrease in bioactive compound but saving time and energy.

4. Conclusion

In conclusion, this study investigated the dynamics of CP juice concentration, employing a comprehensive mathematical modeling approach and examining the associated physicochemical changes. CP stands out for its ability to thrive in arid regions, offering a resilient source of nutrition with unique bioactive compounds, such as betalains, AA, and phenolic compounds.

The mathematical models, including Page, Henderson and Pabis, Weibull distribution, parabolic, and Wang and Singh, were systematically applied to elucidate the concentration kinetics of CP juice using a rotary vacuum evaporator. The Weibull distribution model emerged as a robust and fitting descriptor for the concentration process, highlighting its potential as a valuable tool in food processing kinetics. Furthermore, the elucidation of activation energy (254.09 kJ mol^{-1}) provided insights into the temperature dependence of the Weibull distribution rate constant, thus contributing to a deeper understanding of the chemical reactions underlying the concentration kinetics.

The results confirm that mathematical modeling is crucial for describing the concentration process, thus enabling a quantitative assessment of the impact of different temperatures. This approach provides a predictive tool to optimize processing conditions and improve product quality.

Throughout the concentration process, varying temperatures significantly influenced physicochemical parameters, revealing temperature-dependent alterations in the content of bioactive compounds and reducing power. The study confirmed the pivotal role of temperature in shaping the concentration process, where lower temperatures, particularly at 50°C and 60°C, demonstrated superior preservation of essential bioactive compounds, including betalains, AA, and phenolic compounds.

This research study not only enhances our knowledge of the CP juice concentration process but also underscores the significance of selecting optimal processing conditions to preserve the nutritional and bioactive quality of the final product. The findings offer valuable insights for the food industry, opening the way for the development of enhanced processing strategies that balance efficiency with the retention of essential bioactive compounds in CP-derived products.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Ethics Statement

The authors have nothing to report.

Consent

The authors have nothing to report.

Disclosure

The work described has not been published before; it is not under consideration for publication elsewhere; its publication has been approved by all coauthors.

Conflicts of Interest

The authors declare no conflicts of interest.

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The authors have nothing to report.

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