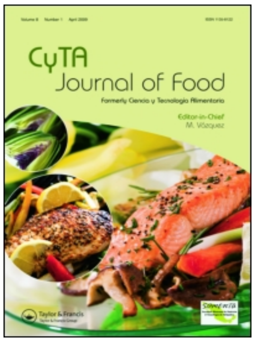

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Identifying non-destructive growth and maturity indexes of Prickly pear (*Opuntia albicarpa* S. Var. Burrona) and evaluation of freeze-drying conditions

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ABSTRACT

Around the world, prickly pear fruits are valued as a source of dietary functional compounds and ingredients for innovative foods. Growth and physicochemical changes of *Opuntia albicarpa* S. fruits were recorded from 0 to 132 days-after-flowering (DAF) to identify non-destructive maturity-indices. Optimum-ripened fruits were freeze-dried to study physicochemical and functional characteristics of dried and rehydrated pulp. Principal component analysis confirmed growth turned into fruit ripening in DAF 99, and it lasted until DAF 132. Changes in color parameters of the peel correlated with fruit texture and pulp sugar content and taste index ($P < 0.01$). During freeze-drying, plate temperature had more significant effects than the thickness ($P < 0.05$). At 30°C, color ΔE between dried and fresh slices augmented, but, texture Δ´s (medium force) between rehydrated and fresh pulp was lower. Color tests could be used to harvest commercially-ripened fruits. Freeze-drying at 30°C improves the rehydrated slices texture regarding thickness maintaining rehydration coefficients.

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KEYWORDS
Non-destructive indexes; fruit growth; maturity; prickly pear; *Opuntia albicarpa* S.; freeze-drying; rehydration; principal component analysis; discriminant analysis

Identificación de índices no destructivos de crecimiento y maduración de tunas (*Opuntia albicarpa* S. Var. Burrona) y evaluación de las condiciones de liofilización

RESUMEN

Las tunas son apreciadas en todo el mundo como fuente de compuestos funcionales dietarios e ingredientes para alimentos innovadores. Para identificar índices de maduración no destructivos, se registraron cambios en el crecimiento y en los parámetros fisicoquímicos de frutos de *Opuntia albicarpa* S. entre los días después de la floración (DAF) 0 al 132. Frutos con maduración óptima fueron liofilizados para evaluar características fisicoquímicas y funcionales de pulpa seca y rehidratada. El análisis de componentes principales confirmo que el crecimiento dio lugar a la maduración en el DAF 99 y ésta prosiguió hasta el DAF 132. Los cambios en parámetros de color en cáscara correlacionaron con la textura del fruto, y en pulpa, con el contenido de azúcares y el índice de sabor ($P< 0.01$). Durante la liofilización, la temperatura de placa tiene más efectos significativos que el espesor ($P\leq 0.05$). A 30°C, el ΔE de color entre la pulpa seca y fresca aumentó, pero, el Δ de textura (fuerza media) fue menor entre la rehidratada y la fresca. Se pueden emplear evaluaciones de color para cosechar frutos en su madurez comercial, y liofilizar estos frutos a 30°C mejora la textura sin importar el espesor, manteniendo los coeficientes de rehidratación.

PALABRAS CLAVE
Índices no destructivos; crecimiento de frutos; maduración; tuna; *Opuntia albicarpa* S.; liofilización; rehidratación; análisis de componentes principales; análisis discriminante

1. Introduction

The origin of the cactus species (*Opuntia* sp.) is tropical or subtropical America and initially were cultivated in semiarid lands of Mesoamerica (Pimienta, 1990). Currently, they are distributed in all continents, where wild or cultivated plants are mainly used as food (Sáenz et al., 2013). Prickly pears are the most consumed parts of the plant; it presents peel and a fleshy pulp in variable proportions with numerous seeds in the edible portion (López-Palacios et al., 2015). They are a source of functional compounds such as fiber, hydrocolloids and antioxidant vitamins that are valued for their contribution to a healthy diet and as an ingredient for innovative foods (Sáenz, Sepúlveda, & Matsuhiro, 2004).

During fruit ripening, various physiological and biochemical alterations promote the development of aroma and flavor, as well as changes in external appearance, for example, the peel color (McAtee, Karim, Schaffer, & David, 2013). The color, firmness, total acidity (TA) and total soluble solids

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(TSS), are variables frequently used to evaluate fruit quality. Additionally, they are useful markers of specific stages of development and maturation, as well as essential tools in the prediction of consumer acceptance and preference of use (Cliff & Toivonen, 2017). So, the studies on maturation are fundamental to define the collection time (Dantas, Silva, de Lima, Dantas, & Mendonça, 2013), or the shelf life after harvest (Maldonado-Astudillo et al., 2014). However, the optimum harvest time ultimately depends on the final use (Cocci, Sacchetti, Rocculi, & Dalla Rosa, 2014).

Prickly pears as *Opuntia albicarpa* S. with green pulp present the best texture, and also are the main species cultivated in several countries (Sáenz et al., 2013); but characteristics such as the high pH values and TSS are a challenge for processing (Sepulveda & Saenz, 1990). Conventional thermal processing affects the color and aroma due to the development of brown compounds and hay or grass aromas, being more critical in fruits with green pulp (Sáenz et al., 2013). The drying extends the availability of the fruits, and the selection of the optimal method minimizes the losses of quality and make it possible to obtain convenient products for the consumer (Salazar, Alvarez, & Orrego, 2017). Through freeze-drying, the removal of water is carried out by sublimation at low temperatures. As a result, the products in their dry state are very porous, brittle, hygroscopic, and with excellent rehydration capacity. Once rehydrated, it retains most of the characteristics of the original food (Ceballos, Giraldo, & Orrego, 2012). However, they tend to be fragile, and after rehydration, the texture of the native material is not wholly recovered (Marques, Silveira, & Freire, 2006). These changes are due to factors related, among others, to the dehydrated product microstructure, the treatments, freezing conditions before lyophilization, as well as, to the dehydration and rehydration conditions (Vergeldt et al., 2014).

The objectives of this study were to characterize the ripening kinetics of prickly pear (*Opuntia albicarpa* S.) variant Burrona fruits, to identify non-destructive parameters associated to physicochemical changes, and in the optimum ripening stage, to determinate the best freeze-drying conditions according to the physicochemical and functional characteristics of dehydrated pulp slices.

2. Material and methods

2.1. Chemicals

All chemicals used were analytical reagent grade unless otherwise stated. Sodium hydroxide, 3,5-dinitrosalicylic acid, phenol, sodium sulfite, potassium sodium tartrate, ethanol, sulfuric acid, and acetone where supplied by Sigma-Aldrich (St. Louis, Missouri).

2.2. Plant materials

Fruits were obtained from a three-year-old orchard located in central Mexico; where 130 prickly pear (*Opuntia albicarpa* S.) variant Burrona plants were selected. When bloom started, two to three peripheral fully open flowers per plant were hand-pollinated (98% of fruits were set). Morphological magnitudes were measured to evaluate the growing pattern. For this purpose, the samplings were done every three weeks from 0 to 99 days after flowering (DAF), and weekly from 99 to 132 DAF in a total of eleven collections. From the DAF 99 were included physicochemical determinations in the whole fruit, peel, and pulp to study their changes during the ripening phase.

In each sampling point, three batches of five prickly pear fruits were randomly collected at each stage of development. Data of morphological and physicochemical parameters were measured for each fruit into the batch. Then, the peel and pulp were hand-separated and measured. Immediately, the fractions obtained from each sample were homogenized in a domestic blender, and the seeds were recovered. Homogenates were storage in vessels protected from light under refrigeration (4°C) for further analysis.

2.3. Fruit morphological and physicochemical determinations

2.3.1. Morphological parameters

The polar fruit diameter (POD), equatorial fruit (EQD) and pulp diameter (PUD), depth of the flower cavity (DFC), and peel thickness (PTH) were measured using a caliper (cm). The fruit (FRW), peel (PEW), and seeds dry (SDW) weights and humidity (%) were tested gravimetrically according to 925.09 method (Association of Official Analytical Chemists, 1990).

2.3.2. Physicochemical parameters

Pulp density ($g{\cdot}cm^{-3}$) was calculated in 0.5 cm side cubes. The weight was determined in an analytical balance (Ohaus), and the volume was taken as the volume increase after submerging the pieces in a graduated cylinder of 10 mL capacity, partially filled with mineral oil.

External color of the whole fruit was determined as the mean of three measures taken around the equatorial diameter. It was used a spectrophotocolorimeter (Ultra Scan XE, Hunter Lab, Virginia, USA) whit an illuminant D65, 10° viewing angle, and an observation diameter of 2.45 cm. The pulp color was determined in 40 mL of liquid pulp using a $2 \times 5 \times 5$ cm cuvette. CIE color (L^*, a^*, b^*) parameters were recorded. Where L^* represents color lightness (0 = black and 100 = white). The a^* parameter indicates the axis from green (-) to red (+) while b^* ranged from blue (-) to yellow (+). These data were converted into hue and chroma values to refer to color tones and chromaticity, respectively.

Texture evaluation was done on the intersection of the polar and equatorial diameters; practicing one test in each fruit with a universal texturometer (TA-TX2 Stable Micro Systems, Texture Technologies Corps. New York). The penetration test probe TA-54 was used according to a program with the following parameters: 50% of penetration distance, a speed of 2 $mm{\cdot}s^{-1}$, and a penetration force of 0.07 N. Results were expressed as maximum (MAF) and medium (MEF) penetration forces (N).

Data of total soluble solids (TSS) were obtained in pulp samples using a bench refractometer (Abbe Mark 11, Reichert-Jung NY, USA) according to AOAC method 932.12 (Association of Official Analytical Chemists, 1990) and expressed as °Brix. For the sugars assays, ethanolic extracts were obtained from the samples according to the procedure previously described by Southgate (1976). Reducing sugar (RS) content (%) was quantified by the DNS method (Wood & Bhat, 1988). The reading was done with a spectrophotometer at 540 nm (Jenway 6305, UK). Total sugars (TS) content (%) was measured by the phenol-sulfuric method, taking the absorbance value at 490 nm (Wood & Bhat, 1988).

Direct measurements of pH were done in homogenates of peel and pulp with a potentiometer (Thermo scientific A211, USA, MA). Total acidity (TA) data was obtained according to 945.26 method (Association of Official Analytical Chemists, 1990), and the results were expressed as citric acid content (mg·g^{-1} fresh weight (f.w.)). The taste index (TI) of pulp samples was calculated as the TSS and TA quotient (Reig et al., 2017).

Chlorophyll (CHL) contents (μg·g^{-1} f.w.) of peel and pulp acetone extracts were calculated from the spectrophotometric measurements at 663 and 645 nm according to the procedure previously described by Castelfranco (1977). Water activity (aw) was measured in the homogenized pulp at 25°C with a water activity meter (AquaLab 4TE Meter Group, WA, USA).

2.4. Freeze-drying of prickly pear slices

2.4.1. Freeze-drying assays

For this step, 80 fruits with a maturation level corresponding to DAF 111 were selected from freshly harvested fruits. The selection was performed instrumentally from the measurement of color parameter *a** according to ANOVA and correlation results from ripening study. The pulp of selected fruits was sliced into 1 and 1.5 cm thickness pieces. In twenty units of each thickness were studied the characteristics of fresh (F) slices. The rest were placed in aluminum trays (14 cm) that were kept at −40°C (Scientemp, MI, USA) for at least 24 hours until freeze-drying experiments. The weights of the slices and the empty and filled trays were recorded before freezing and after freeze-drying them.

The experiments were conducted in a pilot-scale freeze-dryer (Usifroid SMH-15, Maurepas, France) assaying the two slice thickness (1 and 1.5 cm) and two plate temperatures (15 and 30°C). An initial plate temperature of −50°C was maintained for 60 min. Subsequently, the total pressure inside de vacuum chamber was kept at 100 Pa.Immediately, the plate temperature was raised to 15 or 30°C (1°C·min^{-1}). The freeze-drying process of the slices was carried out for approximately 24 hours, allow samples to obtain a moisture content of 4% or less. The process was repeated to get at least 40 units of the four combinations of thickness and temperatures to study the characteristics of freeze-dried (FD) and rehydrated (RH) prickly pear slices. The dried samples were immediately packed into nylon-polyethylene coextruded bags and stored in dark conditions at 18°C.

2.4.2. Characteristics of fresh, freeze-dried, and rehydrated slices

To evaluate color in F, FD, and RH pulp slices, color parameters *L**, *a**, *b**, hue, and chroma were measured in the center of 10 units under the same conditions described for the external color of the fruit. Also, to evaluate the color change of FD and RH slices compared to F ones were calculated the Δ*L**, Δ*a**, Δ*b** values and the global color change parameter (ΔE) with Eq. 1 (Duan, Liu, Ren, & Yang, 2017):

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

Texture parameters, MAF and MEF of F and RH material were obtained from the media of three tests performed in each slice. The penetration test probe TA-10 was used according to a program with the following parameters: 25% of penetration distance, a speed of 0.1 mm·s^{-1}, and a penetration force of 0.07 N. The ΔMAF and ΔMEF resulted from the quotient between the parameter measured in RH and F materials (Δ > 1 rise, Δ < 1 lost in texture).

Final humidity and dry matter content were quantified in the F, FD, and RH material as described in section 2.3.

To measure the water absorption capacity (WAC), the rehydration process was conducted at 25°C by submerging the tested samples into 300 mL of distilled water. The weight of each FD slice was registered along time until obtaining constant weight (m_{RH}). The WAC of the FD prickly pear pulp was defined as the quotient of the recovered water mass and the eliminated water mass during freeze-drying. WAC index was calculated according to the Eq. 2 (Marques, Prado, & Freire, 2009):

$$WAC = \frac{m_{RH}(100 - s_{RH}) - m_{FD}(100 - s_{FD})}{m_F(100 - s_F) - m_{FD}(100 - s_{FD})} \quad (2)$$

Where m is the mass of the slice (g), and s is the dry matter content (g·kg^{-1}) of the fresh, freeze-dry and rehydrated material identified with subscripts. The result was multiplied by 100 to express values as a percentage (%).

2.5. Statistical analysis

The statistical analyses were performed with the software Statistica 13.3 (TIBCO Software, Inc). Data obtained for fresh fruits evaluation throughout ripening were divided into two different data sets, one for the fruits morphological parameters and other for the fruits physicochemical parameters. Same statistical analyses were applied for both data sets. Analysis of variance (ANOVA) and Fisher's least significant differences (LSD) tests were applied to identify parameters showing significant differences throughout fruit ripening. Subsequently, Simple Linear Correlation (Pearson r) analysis was used (P < 0.05). Then, Principal Component Analysis (PCA) was performed to identify the most important parameters changing throughout the study. Finally, General Discriminant Analysis (GDA) was carried out to select the parameters discriminating fruits according to DAF. A forward stepwise method (P inclusion 0.05, P exclusion 0.05) was applied to both data sets. The selected variables were those with a significant (P < 0.05) F value.

For freeze-drying tests, ANOVA and LSD test were applied to measure the effect of evaluated factors and their interactions over different quality characteristics. This study allowed to select those treatments that show the best features.

3. Results and discussions

3.1. Fruit growth

3.1.1. Morphological changes during the ripening process

All parameters evaluated showed highly significant differences (P < 0.001) (Table 1). Along with the growth of fruits, FRW, POD, EQD, and PUD increased dramatically. FRW increased from DAF 38, while the others from DAF 0 to 59. The pulp weight and PEW augmented in alternate stages. The pulp remained unchanged from DAF 0 until day 80 to continue with a pattern of changes identical to FRW, while the PEW changed from the beginning of the study until the DAF 59 and then the increases ceased. During the period studied were observed decreases in the PTH and the DFC. In PTH occurred a drop between the DAF 80 and 99, in DFC

Table 1. Fruit's morphological magnitudes from 0 d to 132 days after flowering.

Tabla 1. Magnitudes morfológicas de frutos de 0 a 132 días después de la floración.

		Days after flowering										
Magnitude	*P*-value	0	22	38	59	80	99	104	111	118	125	132
FRW (g)	0.000***	22.310^{d}	41.639^{b}	56.654^{b}	87.631^{c}	93.050^{c}	151.364^{a}	157.522^{a}	166.671^{a}	191.101^{e}	162.750^{a}	154.022^{a}
POD (cm)	0.000***	4.868^{d}	5.625^{e}	6.377^{f}	7.462^{b}	7.507^{bc}	8.238^{a}	8.587^{a}	8.794^{a}	8.695^{a}	8.304^{a}	8.173^{ac}
EQD (cm)	0.000***	2.902^{c}	3.476^{d}	4.203^{e}	4.970^{c}	4.959^{c}	5.976^{a}	6.046^{a}	6.171^{a}	6.548^{f}	6.198^{a}	6.090^{a}
PEW (g)	0.000***	19.857^{b}	31.127^{c}	45.053^{d}	71.793^{a}	67.032^{a}	64.285^{a}	72.923^{a}	71.288^{a}	70.717^{a}	65.266^{a}	69.733^{a}
PUD (cm)	0.000***	0.950^{d}	1.658^{e}	2.222^{f}	2.919^{b}	3.117^{b}	4.845^{c}	5.058^{ac}	5.192^{a}	5.617^{g}	5.246^{a}	5.270^{a}
PTH (cm)	0.000***	1.942^{de}	1.881^{df}	1.969^{de}	2.051^{e}	1.774^{f}	0.812^{d}	0.773^{d}	0.694^{bc}	0.564^{ab}	0.526^{a}	0.581^{ab}
DFC (mm)	0.000***	0.956^{c}	0.975^{c}	0.333^{b}	0.374^{b}	0.329^{b}	0.112^{a}	0.075^{a}	0.043^{a}	0.037^{a}	0.058^{a}	0.052^{a}
SDW (g)	0.000***	0.030^{c}	0.262^{c}	1.538^{e}	4.266^{f}	5.632^{bd}	6.808^{a}	6.371^{ad}	6.476^{a}	6.670^{a}	5.178^{b}	5.347^{b}

FRW: Fruit weight; POD: Polar diameter; EQD: Equatorial diameter; PEW: Peel weight; PUD: Pulp diameter; PTH: Peel thickness; DFC: Depth of flower cavity; SDW: Seed dry weight.
Results are presented as means (n = 3). Significant difference at * $P < 0.05$, ** $P < 0.01$ and *** $P < 0.001$. Values with different letters within the same row are significantly different ($P < 0.05$).

FRW: Peso del fruto; POD: Diámetro polar; EQD: Diámetro ecuatorial; PEW: Peso cáscara; PUD: Diámetro de pulpa; PTH: Grosor de cáscara; DFC: Profundidad de la cicatriz receptacular; SDW: Peso seco de semillas.
Los resultados se presentan como medias (n = 3). Diferencias significativas a * $P < 0.05$, ** $P < 0.01$ y *** $P < 0.001$. Valores con distintas letras en la misma fila son significativamente diferentes ($P < 0.05$).

between DAF 22 and 38, and between DAF 80 and 99. For its part, the SDW increased steadily from DAF 22 until 99.

During growth, most of the morphological characteristics showed two stages of change ($P < 0.001$) (Table 1). The first stage started on DAF 38 except for DFC (DAF 22) and PTH (DAF 59). The second stage lasted from DAF 80 to DAF 99.

3.1.2. Correlations

All the parameters correlated in a highly significant way ($P < 0.001$). The highest correlation coefficient was between FRW and PUD (R = 0.99), and the lowest one (−0.56) between PEW and PTH. According to the sequence of changes observed, the growth curve of Burrona fruits showed a double sigmoid pattern. This behavior agreed with that described for prickly pear Guialla, Rossa, and Bianca cultivated in Italy (Barbera, Carimi, Inglese, & Panno, 1992) and *Opuntia inermis* (Kuti, 1992).

3.1.3. Principal component analysis (PCA)

After analysis, the first two PC obtained explained 96.53% of the variability (89.97% and 6.56% for PC1 and PC2, respectively). The factorial map (Figure 1(a)) showed an evolution in two phases, the first one (UR) comprised between dates 0 and 80, and the second one (RP) among DDF 99 to 132. The UR phase was determined by a combination between PC1 and PC2, the eigenvectors (Figure 1(b)) showed a substantial effect of all the parameters in PC1, while in PC2 were PEW and PTH. The evolution pattern shows the fruit stopped the growth and began ripening between DAF 80 and 99. Previous reports mentioned that fruit development period changes with climatic conditions and its length vary from 70 to 150 days (Inglese, Barbera, & La Mantia, 1995; Kuti, 1992; Nerd & Mizrahi, 1997; Pimienta, 1990).

3.1.4. Discriminant analysis

The forward stepwise general discriminant analysis led to the selection of FRW, DFC, EQD, PEW, SDW, and PTH, with a correct classification of 96.97% of the samples. Misclassification was observed in samples of 132 DDF (66.68% correctly classified) this could be due to senescence process. Figure 2(a) shows the Cooman´s graph according to magnitudes that follows the evolution of the fruit. The results confirm the observed in ANOVA and PCA.

3.2. Characterization of fruit ripening process

3.2.1. Fruit and pulp physicochemical changes through the ripening process

From DAF 99 throughout the end of the study (Table 2), the MAF and MEF decreased significantly ($P < 0.001$), between DAF 111 and 118, and DAF 104 and 118, respectively. Values of MAF were 23.3 to 18.5 N, and MEF ranged from 9.0 to 5.0 N. Fruit firmness is considered an important maturity index. In previous works has been referred penetration force values of 11.5 N in green *Opuntia ficus-indica* fresh fruits (Díaz-Lima & Vélez-Ruiz, 2016), and 8.2 N in fresh fruits of Burrona (González-González, Morales, Olivares-Sáenz, Aranda, & Gallegos-Vázquez, 2001). Those values are inferior to the results of MAF but were in accordance with MEF values.

Pulp color parameter L^* diminished ($P = 0.002$) and a^* increased ($P = 0.015$). L^* values from DAF 99 to 111 were darker than those from DAF 118 to 132. a^* increased moderately thought maturation remaining on the green scale. On the other hand, changes in b^*, hue, and chroma were not significant. But CHL content drops dramatically ($P = 0.002$) after DAF 99. Similar changes of *Opuntia ficus-indica* pulp color parameters in response to maturity stages and harvest season have been reported before (Allegra et al., 2015).

As maturation progressed, TA decreased, and pH had the opposite tendency, with significant changes between DAF 99 and 104 ($P = 0.000$). TA and pH values ranged from 0.061 to 0.035 mg of citric acid·g^{-1} f.w., and 5.862 to 6.045, respectively. The low TA values were in accordance with previous studies (Allegra et al., 2015; Inglese et al., 1995). Obtained pH values were in the range of no acidic foods and agreed with those reported in Mexican (Pimienta-Barrios, 1994) and Argentine (Felker et al., 2005) *Opuntia* fruits.

In the case of sugars, TSS ($P = 0.009$) and RS ($P = 0.000$) increased progressively until DAF 125 (13.150 and 12.244%, respectively), then decreased due to senescence, while TS remained steady. Taste index also augmented all over the maturity with a sharp climb between DAF 104 and 111 ($P = 0.000$). Cactus pears are usually harvested when they

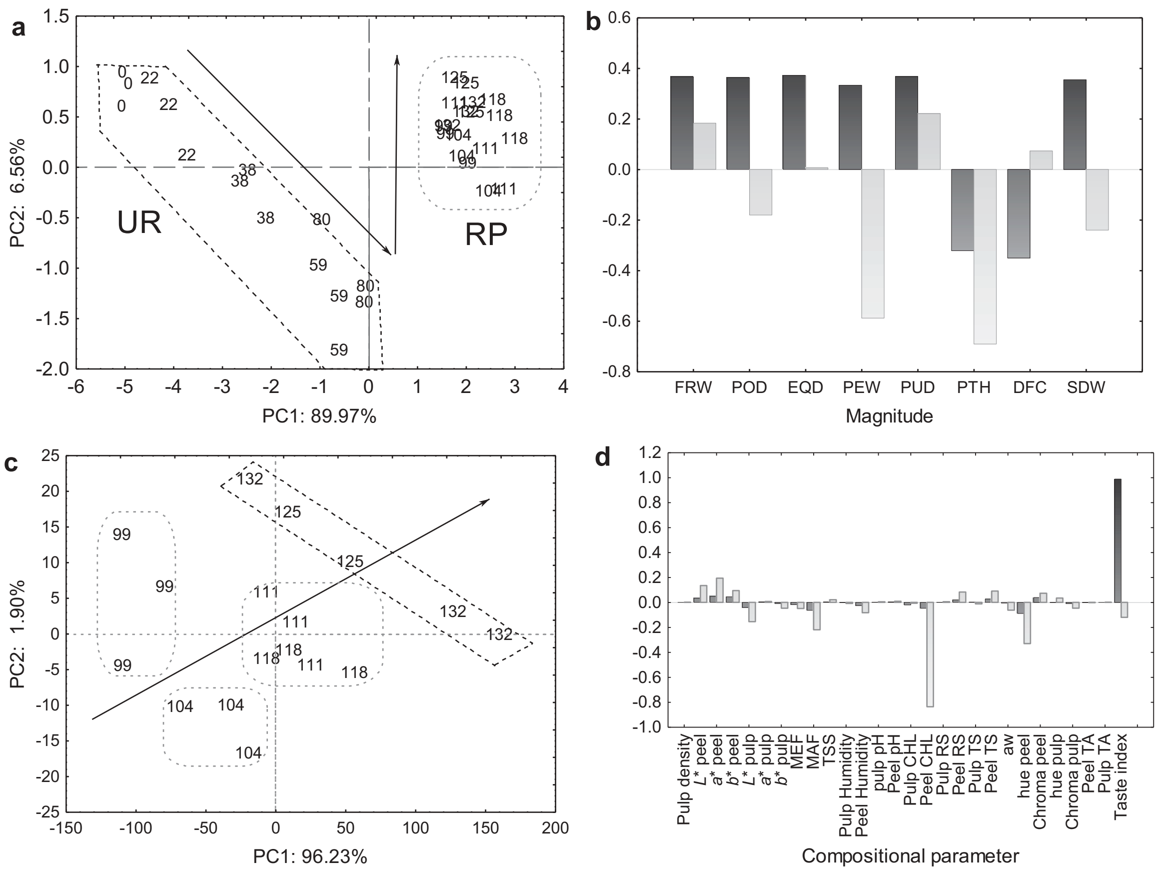

Figure 1. Principal component analysis (PCA) plots for first two principal components. Fruit magnitudes: a) Factorial map; and b) Eigenvectors (■ PC1, ■ PC2). Compositional parameters: c) Factorial map; and d) Eigenvectors (■ PC1, ■ PC2). FRW: Fruit weight; POD: Polar diameter; EQD: Equatorial diameter; PEW, Peel weight; PUD: Pulp diameter; PTH: Peel thickness; DFC: Deep of flower cavity; SDW: Seeds dry weight; MEF: Mean force; MAF: Maximum force; TSS: Total soluble solids; CHL: chlorophyll; RS: Reducing sugars; TS: Total sugars; TA: Total acidity.

Figura 1. Gráficas del análisis de componentes principales (ACP) para dos primeros componentes principales. Magnitudes: a) Mapa factorial y b) Vectores propios (■ PC1, ■ PC2). Parámetros composicionales: c) Mapa factorial y d) Vectores propios (■ PC1, ■ PC2). FRW: Peso del fruto; POD: Diámetro polar; EQD: Diámetro ecuatorial; PEW: Peso cáscara; PUD: Diámetro pulpa; PTH: Espesor cáscara; DFC: Profundidad cavidad floral; SDW: Peso seco de semillas; MEF: Fuerza media; MAF: Fuerza máxima; TSS: Sólidos solubles totales; CHL: Clorofila; RS: Azúcares reductores; TS: Azúcares totales; TA: Acidez total.

reach a minimum of 13% of TSS (Gurrieri et al., 2000), although other authors had reported lower values of 12.1 and 12.7% (Allegra et al., 2015). The density, humidity content, and aw remained without significant changes during the study.

3.2.2. Peel physicochemical changes through the ripening process

External color parameters *L**, *a**, *b** and chroma raised significantly according to DAF ($P < 0.010$) (Table 2). *L** and *a** values were lighter and greener, respectively, from DAF 99 to 111. During the following DAF, peel turned darker and red areas emerged. Nevertheless, values of *b** and chroma increased continuously. Hue drops steadily ($P = 0.000$) changing from green to red tones throughout maturation. CHL content drops dramatically ($P = 0.004$) after DAF 118. Results are similar to that reported at the beginning of the study in *Opuntia albiacarpa* (Burrona variant) fruits (Ochoa-Velasco & Guerrero-Beltrán, 2016). Other authors had pointed out that the increase in yellow appearance is a result of CHL loss (González-González et al., 2001).

The TA ranged from 0.580 to 0.227 mg citric acid·g^{-1} f.w., with a significant reduction between DAF 111 to 118 ($P = 0.000$). As TA decreased, pH raised (4.522 to 5.207) in a significant way until DAF 118 ($P = 0.000$), then remained steady. TA and pH values were higher than in the pulp, although changes were parallel. The same trend was referred before (Gurrieri et al., 2000).

The increase in contents of RS and TS was constant and, in both cases, highly significant between all sampling dates ($P = 0.000$). The concentrations were lower than those quantified in the pulp. Humidity content declined throughout the study ($P = 0.000$).

3.2.3. Correlations

Highly significant correlations were observed between physicochemical parameters of pulp and peel ($P < 0.01$). Outstanding those between nondestructive peel color parameters, and variables whose determination are destructive such as pulp TI, as well as fruit MAF and MEF ($P < 0.05$). Correlations between *a**, *b**, chroma, and hue, with TI, showed high coefficients (0.77, 0.63, 0.61, and −0.79 respectively), as well as with MAF (0.76, 0.82, −0.79 and 0.82 respectively); and MEF (0.78, 0.86, −0.84 and 0.86 respectively). More correlations of peel color with other pulp parameters were: *L**, *a**, *b**, hue and chroma with TSS (r = 0.64, 0.61, 0.60, −0.63 and 0.58, respectively) and TA (r = −0.62, −0.66, 0.65, 0.71 and 0.64, respectively); *L** with RS (r = 0.57); hue with CHL (r = 0.49); and chroma with RS (r = 0.53). On the other hand, additional high correlations ($P < 0.05$) of the meaningful destructive variable pulp TI were observed with peel humidity, pH, RS, TS and TA (r = −0.78, 0.738, 0.76, 0.79,

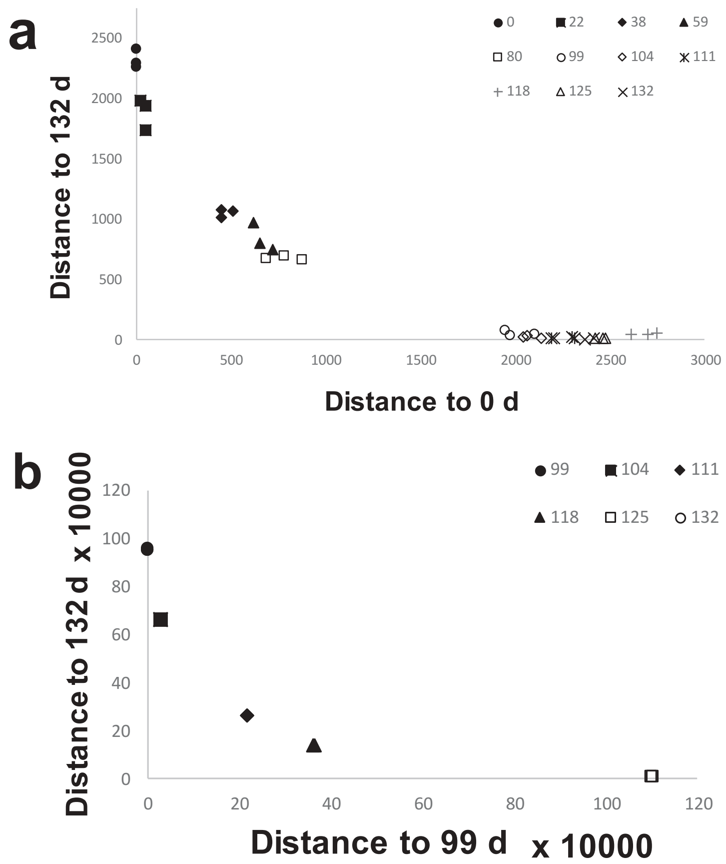

Figure 2. Cooman´s graphs of Mahalanobis distances for fruit classification according to a) Magnitudes and b) Compositional parameters.

Figura 2. Gráficas de Cooman de las distancias de Mahalanobis para la clasificación de los frutos según a) Magnitudes y b) Parámetros composicionales.

–0.77, respectively) and with pulp *L**, *a**, TSS, pH, CHL and TA (r = –0.67, 0.65, 0.56, 0.56, –0.61 and –0.96, respectively). The texture parameters of the fruit, MAF and MEF, correlated in a significant way ($P < 0.05$) with pulp *L** (r = 0.68 and 0.78), aw (r = 0.49 and 0.55), TA (r = 0.70 and 0.80), and TI (r = – 0.75 and –0.77). In the peel, all the correlations with MAF and MEF were highly significant ($P < 0.001$), as the the case of humidity (r = 0.89 and 0.95, respectively), and TA (r = 0.88 and 0.92, respectively).

The most important correlations were those between the color parameters in the peel with TI in the pulp and with MAF and MED of the fruit. As maturity index, the *a** parameter better differentiated between DAF (Table 2) and can be obtained in a direct, easy, quick and no destructive way. It is known that size, percent flesh, color, TSS, and seed content are the main parameters characterizing fruit quality (Inglese et al., 1995). In relation to prickly pear, Kuti (1992) stated the importance of establishing the maturity indexes through the external color as long as it contributes to predicting the sensory quality of the fruit during growth and development. To our knowledge, there are no publications showing correlations between peel and pulp characteristics in prickly pear. However, in fruits such as peach and nectarine was referred that changes in CHL were highly and consistently correlated with fruit firmness (Reig et al., 2017; Zhang, Peng, Zhang, Song, & Ma, 2017). In tomato also were reported high correlations between color parameters such as *a**, hue, and chroma, with lycopene and flesh or skin firmness (Radzevičius et al., 2016).

3.2.4. Principal component analysis

After analysis, the first two PC obtained explained 98.13% of the variability. The factorial map (Figure 1(c)) showed a continuous evolution forming four groups according to DAFs. The first group includes DAF 99, the second comprised DAF 104, the third grouped DAF 111 and 118, the last one comprises 125 and 132 DAF. The prickly pears from DDF 125 and 132 were misclassified; it may be attributed to the start of the senescence stage immediately after maturation. The eigenvector 1 (Figure 1(d)) showed an important effect of the taste index, while in PC2 predominated the effect of CHL followed by hue, both measured in the peel.

3.2.5. Discriminant analysis

The forward stepwise GDA led to the selection of density, RS, pH, and hue of the pulp, and MEF with a correct classification of 100% of the samples. Figure 2(b) shows the Cooman´s graph according to physicochemical parameters that follow the evolution of the fruit. The results confirm the observed in ANOVA and PCA.

3.3. Freeze-drying of prickly pear slices

3.3.1. Influence of temperature and thickness on freeze-dried slices characteristics

Color parameters *L** and *a** did not change significantly ($P > 0.05$) in response to the thickness and temperatures assayed (Table 3). On the other hand, *b** diminished significantly showing a reduction in the yellow color at 30°C. The hue

Table 2. Fruit's physicochemical parameters from 99 d to 132 days after flowering.

Tabla 2. Parámetros fisicoquímicos de los frutos de 0 a 132 días después de la floración.

		Days after flowering					
Physicochemical Parameter	*P*-value	99	104	111	118	125	132
Pulp density (g·cm^{-3})	0.905	1.051^{a}	1.074^{a}	1.058^{a}	1.054^{a}	1.062^{a}	1.079^{a}
*L** peel	0.000***	55.626^{a}	56.644^{a}	58.260^{a}	63.724^{b}	65.283^{b}	62.861^{b}
*a** peel	0.000***	−8.980^{a}	−8.414^{a}	−7.613^{a}	−5.430^{b}	−1.219^{b}	2.624^{b}
*b** peel	0.010**	28.542^{a}	31.014^{a}	32.781^{ac}	39.011^{b}	39.780^{b}	37.154^{b}
hue peel	0.000***	107.469^{b}	105.366^{ab}	103.387^{a}	97.919^{e}	91.236^{d}	85.882^{c}
Chroma peel	0.007**	29.922^{a}	32.154^{ab}	33.683^{ab}	39.391^{c}	40.065^{c}	37.273^{bc}
*L** pulp	0.002**	48.750^{b}	47.137^{b}	42.227^{a}	41.983^{a}	38.760^{a}	38.257^{a}
*a** pulp	0.015*	−6.533^{a}	−6.410^{a}	−6.227^{a}	−6.113^{a}	−6.005^{ab}	−5.573^{b}
*b** pulp	0.309	16.997^{ab}	17.403^{b}	15.847^{ab}	16.607^{ab}	15.890^{ab}	14.163^{a}
hue pulp	0.684	111.242^{a}	110.378^{a}	111.457^{a}	110.209^{a}	110.292^{a}	111.502^{a}
Chroma pulp	0.213	18.216^{a}	18.556^{a}	17.026^{ab}	17.696^{ab}	16.863^{ab}	15.222^{b}
MEF (N)	0.000***	9.025^{b}	8.050^{b}	6.827^{c}	5.244^{a}	4.902^{a}	5.063^{a}
MAF (N)	0.001***	29.338^{b}	32.127^{b}	26.361^{b}	19.661^{a}	18.787^{a}	18.481^{a}
TSS (%)	0.009**	12.100^{a}	12.133^{a}	12.567^{ab}	12.733^{bc}	13.150^{c}	12.767^{bc}
Pulp humidity (%)	0.101	87.226^{b}	87.095^{b}	87.064^{b}	86.951^{ab}	86.443^{b}	86.997^{b}
Peel humidity (%)	0.000***	90.858^{c}	90.120^{c}	88.860^{d}	86.114^{b}	85.101^{ab}	85.121^{a}
pH pulp	0.000***	5.862^{b}	5.725^{d}	6.160^{d}	6.063^{ac}	5.970^{ab}	6.042^{a}
pH peel	0.000***	4.522^{b}	4.642^{bc}	4.738^{c}	5.158^{a}	5.260^{a}	5.207^{a}
CHL pulp (μg·g^{-1} fresh wt)	0.002**	10.878^{c}	6.654^{a}	5.415^{ab}	7.575^{a}	7.552^{a}	4.092^{b}
CHL peel (μg·g^{-1} fresh wt)	0.004**	14.742^{ab}	28.652^{c}	10.312^{a}	22.052^{bc}	5.233^{a}	5.858^{a}
RS pulp (%)	0.000***	10.819^{a}	11.284^{b}	11.153^{b}	11.484^{c}	12.244^{d}	10.872^{a}
RS peel (%)	0.000***	0.865^{a}	1.594^{b}	3.209^{c}	3.694^{d}	5.926^{f}	5.218^{e}
TS pulp (%)	0.095	10.496^{b}	11.482^{a}	10.842^{ab}	11.253^{ab}	11.779^{a}	10.824^{ab}
TS peel (%)	0.000***	1.182^{a}	2.117^{b}	3.688^{c}	5.643^{d}	6.544^{e}	7.441^{f}
aw	0.364	0.981^{a}	0.972^{a}	0.98^{a}	0.967^{a}	0.952^{a}	0.962^{a}
TA peel (mg citric acid·g^{-1} fresh wt)	0.000***	0.580^{b}	0.511^{b}	0.483^{b}	0.335^{a}	0.291^{a}	0.277^{a}
TA pulp (mg citric acid·g^{-1} fresh wt)	0.000***	0.061^{c}	0.047^{b}	0.041^{ab}	0.041^{ab}	0.041^{ab}	0.035^{a}
Taste index	0.000***	198.42^{a}	259.03^{a}	308.39^{bc}	315.99^{bc}	325.33^{bc}	384.11^{bc}

MEF: Mean penetration force; MAF: Maximum penetration force; TSS: Total soluble solids; CHL: Chlorophyll; RS: Reducer Sugars; TS: Total sugars; aw: Water activity; TA: Total acidity.

Results are presented as means (n = 3). Significant difference at * $P < 0.05$, ** $P < 0.01$ and *** $P < 0.001$. Values with different letters within the same row are significantly different ($P < 0.05$).

MEF: Fuerza media de penetración; MAF: Fuerza máxima de penetración; TSS: Sólidos solubles totales; CHL: Clorofila; RS: Azúcares reductores; TS: Azúcares totales; aw: Actividad de agua; TA: Acidez total.

Los resultados se presentan como medias (n = 3). Diferencias significativas a * $P < 0.05$, ** $P < 0.01$ y *** $P < 0.001$. Valores con distintas letras en la misma fila son significativamente diferentes ($P < 0.05$).

angle corresponded to a green tone that did not change significantly at any condition tested ($P > 0.05$). For chroma, it was observed that the slices processed at 30°C were less intense than those at 15°C. When compared with F slices, the dried ones showed significant Δ of color parameters *L**, *a**, and *b** ($P = 0.000$) indicating that thinner slices were brighter (Δ*L** = 1.273), greener (Δ*a** = −0.064), and yellower (Δ*b** = 0.393) than the fresh ones, while temperature did not show a significant effect. The same effect in *L** color parameter was referred in soursop FD powder as a result of the freezing step.

For all the parameters analyzed, there were no significant interactions (Data not shown).

The effect was related to a scattering of light in the small pores formed during freezing (Ceballos et al., 2012). In Hawthorn freeze-dried were referred values of hue angle corresponding to a brighter color (red) as a result of the water remotion by sublimation during freeze-drying (Duan et al., 2017).

The values of total color difference (ΔE) obtained from all the treatments (Table 3) corresponded to very high color variation with respect to F material; although the differences were not significant for any of the factors studied ($P > 0.05$). According to the analytical criteria proposed before (Adekunte, Tiwari, Cullen, Scannell, & O'Donnell, 2010); differences in perceivable color are classified as very distinct (ΔE > 3), distinct (1.5 < ΔE < 3) and small different (ΔE < 1.5). Francis and Clydesdate (1975) reported good correlations between the ΔE values calculated from color parameters *L**, *a**, and *b** in green vegetables and the minimum differences detectable by trained sensory judges.

3.3.2. Influence of temperature and thickness on rehydrated slices characteristics

In the evaluation of all color parameters on rehydrated slices characteristics (Table 3) were found significant variations according to temperatures assayed ($P < 0.05$). At 30°C color parameter *L** and *b** were brighter and yellower, and *a** greener, hue approached more to 90° (green), and from the higher value of chroma can be inferred that are perceived as more saturated. Nevertheless, the influence of thickness was significant only in color parameter *L** where the thinnest slices had the highest values. When compared with fresh slices, the RH of 1 cm turned significantly darker, while those treated at 30°C were significantly darker, less green and yellower. The ΔE for RH slices was considerably higher (7 to 11 times) than the identified in FD slices; it means that rehydration caused more visible differences in color. ΔE was significant higher just in RH slices obtained at 15°C.

The texture of RH material expressed as MEF did not show significant differences according to thickness and temperatures but varied significantly when expressed as MAF being higher in samples lyophilized applying plate temperatures of 30°C. When compared RH with F slices according to Δ values, the results of MAF and MEF were

Table 3. Characteristics of freeze-dried and rehydrated prickly pear pulp slices.

Tabla 3. Características de rebanadas de tuna liofilizadas y rehidratadas.

		Thickness			Plate temperature	
Parameter	*P*-value	1 cm	1.5 cm	*P*-value	15	30
*L** FD	0.057	80.37^a	78.75^a	0.114	78.82^a	80.22^a
*a** FD	0.248	-4.053^a	-3.751^a	0.518	-3.807^a	-3.984^a
*b** FD	0.187	24.83^a	25.78^a	0.010**	26.31^b	24.36^a
Hue FD	0.068	99.27^a	98.31^a	0.066	98.27^a	99.26^a
Chroma FD	0.215	25.16^a	26.06^a	0.012*	26.60^b	24.69^a
Δ*L** FD	0.050*	1.273^b	1.246^a	0.114	1.248^a	1.270^a
Δ*a** FD	0.000***	-0.064^a	2.044^b	0.552	1.008^a	1.025^a
Δ*b** FD	0.000***	0.393^b	0.023^a	0.456	0.202^a	0.205^a
ΔE FD	0.199	18.94^a	16.91^a	0.572	17.42^a	18.36^a
*L** RH	0.022*	56.83^b	54.12^a	0.000***	52.43^a	58.30^b
*a** RH	0.867	0.549^a	0.536^a	0.002**	1.134^b	-0.020^a
*b** RH	0.886	26.68^a	26.79^a	0.047*	25.57^a	27.85^b
Hue RH	0.924	92.27^a	92.26^a	0.034*	92.98^b	91.58^a
Chroma RH	0.889	26.72^a	26.83^a	0.050*	25.63^a	27.87^b
Δ*L** RH	0.020*	0.900^b	0.856^a	0.000***	0.830^a	0.923^b
Δ*a** RH	0.523	-0.401^a	-0.292^a	0.001***	-0.748^a	0.038^b
Δ*b** RH	0.092	1.395^a	1.297^a	0.048*	1.285^a	1.401^b
ΔE RH	0.544	149.53^a	171.14^a	0.050*	195.62^b	127.35^a
MAF RH	0.625	95.53^a	89.81^a	0.020*	79.61^a	104.93^b
MEF RH	0.326	34.79^a	46.42^a	0.108	30.70^a	50.31^a
ΔMAF RH	0.014*	0.109^b	0.080^a	0.023*	0.081^a	0.107^b
ΔMEF RH	0.962	0.070^a	0.068^a	0.090	0.053^a	0.084^a
WAC RH	0.003**	31.33^a	33.07^b	0.008*	33.02^b	31.47^a

FD: Freeze-dried; RH: Rehydrated; ΔE: Total color difference; MAF: Maximum penetration force; MEF: Mean penetration force; WAC: Water absorption capacity.
Results are presented as means (n = 3). Significant difference at * $P < 0.05$, ** $P < 0.01$ and *** $P < 0.001$. Values with different letters within the same row are significantly different ($P < 0.05$).

FD: Liofilizado; RH: Rehidratado; ΔE: Diferencia total de color; MAF: Fuerza máxima de penetración; MEF: Fuerza media de penetración; WAC: Capacidad de absorción de agua.
Los resultados se presentan como medias (n = 3). Diferencias significativas a * $P < 0.05$, ** $P < 0.01$ y *** $P < 0.001$. Valores con distintas letras en la misma fila son significativamente diferentes ($P < 0.05$).

considerably lower in RH than those measured in F slices (Δ < 1). In the case of ΔMAF were significantly lower in thickest slices and those treated at 15°C.

For all the parameters analyzed there were no significant interactions (data not shown) except Δ*a** RH ($P = 0.032$).

In RH raspberries also was reported a loss of firmness after air and freeze-drying (Sette, Salvatori, & Schebor, 2016). In apple, banana, potato, and carrots dried and hydrated by various methods were found that freeze-drying produces materials with fragile structure, due to loss of elasticity of cellular tissue and higher values of porosity (Krokida, Kiranoudis, & Maroulis, 1999).

The FD slices showed water absorption capacity (WAC) around 30% (Table 3) with significant variations according to thickness and temperature, with the highest values in samples with 1.5 cm and those treated at 15°C. Those values were lower than that reported for FD raspberries (42%) (Sette et al., 2016), mushrooms (45.3%), and cauliflower (51%), but higher than the obtained in Brussels sprouts (21.4%) (Jambrak, Mason, Paniwnyk, & Lelas, 2007).

4. Conclusion

According to the fruit magnitudes, maturation started at DAF 99. External nondestructive characteristics such as peel color parameters reflect changes in the texture of the fruit, taste index, sugar contents, and TA in the pulp. So, color tests could be used in the field to harvest commercially ripened fruits for fresh consumption or to select optimal raw material for freeze-drying processing. Freeze-drying at 30°C improves the texture of rehydrated prickly pear slices regarding thickness and maintain water absorption capacity around 30%.

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