

## Research Article

# Effect of Calcium and Osmotic Pretreatments on Mass Transfer and Texture Parameters during Processing of Chilacayote (*Cucurbita ficifolia* Bouché)

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The aim of this work was to analyze the effect of calcium and osmotic pretreatments on mass transfer and texture parameters of chilacayote (*Cucurbita ficifolia* Bouché). Samples were immersed in  $\text{Ca}(\text{OH})_2$  solutions at concentrations of 0.5, 1, and 1.5 g/100 mL water; solution temperature of 20, 35, and 50 °C; and immersion times of 1.5, 3, and 4.5 h. Subsequently, pretreated samples with a Ca solution were osmotically dehydrated for 2 h with sucrose solutions at 30, 45, and 60 °Brix and temperatures of 30, 50, and 70 °C. Chilacayote tissue showed a positive response to interaction with  $\text{Ca}^{2+}$  ions, increasing the calcium content in the samples. The osmotic effect increased the water loss and the solute gain in the calcium impregnated samples. However, the calcium gained was leached, so the calcium retained decreased considerably. The hardness and adhesiveness increased significantly during calcium pretreatment, and it was maintained during osmosis by impregnated sucrose on the surface. Also, calcium increased the cohesiveness, but its values decreased after osmosis due to brittle behavior acquired. Samples acquired a more plastic than elastic behavior because the springiness decreased during both pretreatments. The gumminess and chewiness were affected by the decrease in cohesiveness and springiness, so less work is required to digest the osmosed samples. Calcium pretreatment (1 g/100 mL water, 35 °C, and 3 h) and osmosis (45 °Brix, 50 °C, and 2 h) were selected as appropriate process conditions for adequate calcium content, mass transfer, and texture parameters.

## 1. Practical Applications

Pretreatments with calcium and osmotic dehydration modify the mass transfer and the physical texture parameters of minimally processed fruits and vegetables. Chilacayote (*Cucurbita ficifolia* Bouché) minimally processed is a product that can be packed with syrups or with different osmotic solutions, and they can also be used as an ingredient (raw material) in the production of foods such as yogurt, jams, cereals, confectionery, and bakery products. In addition, pretreated chilacayote is a food with a higher calcium content and intermediate moisture available to test a drying process

(convective, freeze drying, and microwave vacuum) to obtain another final product.

## 2. Introduction

Chilacayote (*Cucurbita ficifolia* Bouché) is a vegetable consumed in México, Central and South America. The acceptability for consumption is due to its edible portion of 80–90%, carbohydrates, lipids, protein, fiber, calcium, iron, ascorbic acid, and thiamine content [1], in addition, its hypoglycemic effect that acts as an insulin secretagogue [2]. Chilacayote is consumed as dishes prepared with seeds and

pulp only, but chilacayote mesocarp is an edible portion with great potential for the development of new minimally processed products by applying pretreatments with calcium solution [3] and osmotic dehydration [4, 5].

Calcium pretreatment has been analyzed in several works because it improves the nutritional and mechanical properties of food [6–9]. In other words, it increases the calcium content and changes the response of plant tissues to moisture loss and the resulting stresses of the moisture concentration gradient during dehydration [10]. Calcium (Ca) interacts with the middle lamella and the cell wall, improving structural integrity and promoting greater tissue firmness [11] and fortification of plant matrices [12]. Indeed, Ca strengthens the cellular walls cements, avoiding cellular collapse [13]. Also, due to Ca pretreatment, the tensile force and cutting force of cooked noodle increase significantly [14]. In an alginate-guar gum system, the gel strength increases considerably by adding Ca; also, the hardness of Ca lactate-alginate gels is higher than calcium citrate-alginate gels at the same calcium level [15].

Osmotic dehydration (OD) is a pretreatment that consists of immersing a food in a concentrated solution (hypertonic), simultaneously causing a flow of water from the food and a flow of solutes into the plant tissue. The mass transfer during this process is based on two main phenomena taking place: (i) moisture loss and (ii) solid gain [16]. With osmotic pretreatment, an intermediate moisture food is obtained [10], a gain solute efficiently [17], and improved texture parameters [18]. In some cases, a pretreatment with just OD does not show significant differences in the hardness [17], and the work to fracture decrease until equilibrium is reached during OD [19, 20]. However, the combination of calcium-osmotic pretreatments preserves tissue structure, showing a firming effect on stress at rupture values [11]. In the same sense, calcium influences the efficiency of the osmotic process [3]. Also, Barragán-Iglesias et al. [10] mention that the combination of calcium-osmotic pretreatment improves the physical texture parameters during the application of convective drying, and this is due to tissue restructuring caused by  $\text{Ca}^{2+}$  and solutes impregnation during OD.

Pretreatments with calcium and osmotic dehydration influence on the microstructure, calcium content, water loss, solute gain, and texture parameters. Also, these pretreatments can provide an edible food with a nutritional quality and acceptable texture parameters. However, in some foods such as chilacayote, the physical texture parameters are not yet available; therefore, these can be determined objectively measured through a force-time curve [21] and considering the same extensive properties [22]. The objective of this work was to analyze the effect of processing conditions during calcium and osmotic dehydration pretreatments on mass transfer and texture parameters of chilacayote (*Cucurbita ficifolia* Bouché).

### 3. Materials and Methods

**3.1. Plant Material.** Chilacayote fruits (*Cucurbita ficifolia* Bouché) were cultivated on a single orchard with coordinates  $17^{\circ} 56' 27''$  N and  $97^{\circ} 58' 9''$  W (Oaxaca, México) at



FIGURE 1: External appearance and mesocarp of chilacayote (*Cucurbita ficifolia* Bouché).

2, 080 meters above sea level. The plant material was uniformly selected according to the following characteristics: the fruits were harvested in the ripeness stage of consumption (opaque green color of the fruit peel), approximately 210 days after the anthesis, a weight of 3 to 4 Kg, an elongated shape, and without physical damage such as broken skin (Figure 1). The chilacayote fruits were washed and subsequently sanitized using a 0.4% v/v solution of water and commercial Microdyn® sanitizer (Tavistock Holding AG, Switzerland) for 10 minutes. Fresh mesocarp was sectioned into cylinders (10 mm/height and 20 mm/diameter); then, approximately 300 g of cylindrical samples was used for each experiment. Before each experiment, the physicochemical characteristics were determined in duplicate considering three randomly selected cylindrical samples. Table 1 shows the physicochemical characteristics determined in cylindrical samples of fresh chilacayote mesocarp.

**3.2. Calcium Hydroxide Pretreatment.** Calcium solutions were formulated with distilled water and calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) (Fermont PQ36251, GRAS  $\geq 95\%$  of purity).  $\text{Ca}(\text{OH})_2$  solutions were prepared in a beaker with a capacity of 4 L; then, the chilacayote cylinders were immersed in the different formulated solutions. During calcium pretreatments, the fruit/solution mass ratio was 1 : 10. The temperature of the solution was controlled with a digital hot plate (Thermolyne®, Mirak™) adapted with a thermocouple with an accuracy of  $\pm 2$  °C of the set point. The system was kept under magnetic stirring at  $105 \pm 5$  rpm to keep the solution homogeneous, the temperature uniform throughout the system, and to avoid sedimentation of  $\text{Ca}(\text{OH})_2$ . At the end of the pretreatment, the samples were washed three times with distilled water and the excess water was removed from the surface with absorbent paper without pressing the cylinders.

**3.3. Osmotic Dehydration Pretreatment.** Chilacayote samples pretreated with calcium solution under conditions of 1 g/100 mL of water, 35 °C, and 3 h were considered for the application of osmotic dehydration. Conditions of calcium pretreatment prior to osmotic dehydration were selected considering an increase of 200% in calcium content, energy

TABLE 1: Physicochemical characteristics of cylindrical samples obtained in the fresh mesocarp of chilacayote (*Cucurbita ficifolia* Bouché).

Chemical analysis	
Moisture content, $X_{wb}$ (%)	$91.4 \pm 0.80$
Total soluble solids, TSS (°Brix)	$7.10 \pm 0.10$
Calcium content (mg Ca/100 g fresh sample)	$15.0 \pm 1.50$
Texture parameters	
Hardness ( $N$ )	$121 \pm 25.8$
Cohesiveness (adim)	$0.07 \pm 0.01$
Adhesiveness (N.mm)	$0.26 \pm 0.33$
Springiness (adim)	$0.43 \pm 0.08$
Gumminess ( $N$ )	$8.34 \pm 2.45$
Chewiness ( $N$ )	$3.51 \pm 0.93$

savings, and shorter immersion time. Osmotic pretreatment was applied during 2 h of dehydration because at this time equilibrium was reached. Osmotic solution was formulated with distilled water and commercial sucrose (refined, 99.9% sucrose). The osmotic solutions were prepared in a beaker with a capacity of 4 L; then, the chilacayote cylinders were immersed in the different solutions formulated. The solution temperature was controlled with a digital hotplate (Thermolyne®, Mirak™) adapted with a thermocouple with an accuracy of  $\pm 2$  °C of set point. The system was maintained under magnetic stirring at  $105 \pm 5$  rpm to keep the solution homogeneous and the temperature uniform throughout the system. At the end of each osmotic treatment, excess of osmotic solution on surface of the samples was removed with absorbent paper without pressing the cylinders.

**3.4. Physicochemical Analysis.** Moisture content ( $X_{wb}$ ) was determined gravimetrically according to Method 20.013 [23]. Moisture content values were expressed as % (wet basis).

Total soluble solids (TSS) were measured according to Method 932.12 [23]. A semisolid sample of cylindrical samples was obtained by grinding, and the TSS content was measured using a manual refractometer (ICSA-OPTIC, REF113ATC). Data were expressed as °Brix.

The calcium content in fresh chilacayote, pretreated with calcium hydroxide solutions and osmotically dehydrated, was determined with an adapted technique from Method 944.03 [23] based on potassium permanganate titration ( $\text{KMnO}_4$ ). Chilacayote samples (2.5 g) were calcined at 550 °C for 6 hours. The white ashes obtained were dissolved with 40 mL of chloride acid (HCl) 3%  $v/v$  and 5 drops of nitric acid ( $\text{HNO}_3$ ) 70%  $v/v$ , and the solution was boiled and then allowed to cool. Then, the solution was gauged in a 250 mL volumetric flask. An aliquot of 100 mL was measured and mixed with two drops of methyl red; then, ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) 1.5%  $v/v$  was dissolved dropwise until the solution turned orange, and this solution was immediately titrated with HCl 3% until it turned pink. This solution

was diluted with 50 mL of water, boiled, and added with stirring 10 mL of ammonium oxalate [ $(\text{NH}_4)_2\text{C}_2\text{O}_4$ ] 4.2%  $v/v$  solution. The pH was adjusted with HCl 3% to return to pink. The solution was allowed to stand for 6 hours; then, the solution was filtered, and the precipitate was washed with the  $\text{NH}_4\text{OH}$  1.5%  $v/v$  solution. The filter paper with the precipitate was placed in a beaker, and a mixture of 125 mL of water/5 mL of sulfuric acid ( $\text{H}_2\text{SO}_4$ ) 98%  $v/v$  was added; then, the solution was heated to 70 °C and titrated with the  $\text{KMnO}_4$  solution (0.05 N). The results were expressed as mg Ca/100 g of fresh sample.

After calcium pretreatment, the calcium gain in the samples was calculated according to Equation (1), and at the end of osmotic dehydration, the calcium retained in the samples was calculated according to Equation (2).

$$CaG = Ca^f - Ca^i, \quad (1)$$

$$CaR = Ca^{OD} - Ca^i, \quad (2)$$

where  $CaG$  is the calcium gain in mg Ca/100 g of fresh sample,  $Ca^f$  is the calcium content after calcium treatment,  $Ca^i$  is the calcium content of fresh samples,  $CaR$  is the calcium retained after osmotic dehydration in mg Ca/100 g of fresh sample, and  $Ca^{OD}$  is the calcium content after osmotic dehydration.

**3.5. Texture Parameters.** The texture parameter analysis was performed on fresh samples, pretreated with calcium hydroxide, and osmotically dehydrated. Physical parameters of texture, such as hardness, cohesiveness, adhesiveness, springiness, gumminess, and chewiness, were determined from double compression test [24, 25]. Double compression was made with a TA1 texture analyzer (Lloyd Instruments AMETEK™). This equipment was equipped with a load cell of 500 N and a probe cylindrical of 50 mm diameter. The probe compressed the cylinders through 70% of its height and the compression speed set before, during, and after the test was 1 mm/s.

**3.6. Experimental Design and Data Analysis.** Calcium pretreatment consisted of a  $3^3$  completely randomized factorial design; factors and levels considered were calcium concentration (0.5, 1, and 1.5 g/100 mL of water), solution temperature (20, 35, and 50 °C), and immersion times (1.5, 3, and 4.5 h), where 27 experiments were applied with one replicate for each. Osmotic pretreatment consisted of a  $3^2$  completely randomized factorial design; sucrose concentrations (30, 45, and 60 °Brix) and solution temperature (30, 50, and 70 °C) were considered as factors and levels; that is, nine experiments were applied with one replicate for each. Response variables as calcium content, total solids soluble, moisture content, and texture parameters were determined in triplicate for each of the experiments (total experiments: 54 with calcium and 18 with osmosis), and the values were expressed as mean  $\pm$  standard deviation.

The effect of factors and their statistical interactions were analyzed by ANOVA with a significant level  $\alpha = 0.05$ .

TABLE 2: Changes in moisture content ( $X_{wb}$ ), total soluble solids (TSS), and calcium gain of chilacayote cylinders pretreated with  $\text{Ca}(\text{OH})_2$  solutions.

$\text{Ca}(\text{OH})_2$ concentration (g/100 mL w)	Temperature ( $^{\circ}\text{C}$ )	Time (h)	$X_{wb}$ (%)	TSS ( $^{\circ}\text{Brix}$ )	Calcium gain (mg/100 g f. s.)
0.5	20	1.5	$93.6 \pm 0.69$	$2.75 \pm 0.07$	$11.8 \pm 1.48$
		3.0	$93.7 \pm 1.53$	$2.80 \pm 0.03$	$29.2 \pm 1.53$
		4.5	$94.6 \pm 0.40$	$1.70 \pm 0.14$	$32.4 \pm 0.15$
	35	1.5	$91.3 \pm 0.30$	$4.65 \pm 0.21$	$18.0 \pm 1.70$
		3.0	$92.3 \pm 0.50$	$2.35 \pm 0.07$	$30.0 \pm 1.70$
		4.5	$93.0 \pm 0.60$	$1.90 \pm 0.14$	$46.8 \pm 1.70$
	50	1.5	$91.2 \pm 0.34$	$3.10 \pm 0.14$	$30.0 \pm 1.70$
		3.0	$95.5 \pm 3.34$	$2.50 \pm 0.14$	$35.4 \pm 4.24$
		4.5	$96.0 \pm 0.10$	$3.05 \pm 0.07$	$46.8 \pm 1.70$
1	20	1.5	$93.5 \pm 2.36$	$3.10 \pm 0.14$	$26.4 \pm 0.28$
		3.0	$94.0 \pm 0.12$	$2.75 \pm 0.07$	$32.4 \pm 1.70$
		4.5	$94.5 \pm 1.10$	$1.95 \pm 0.07$	$39.6 \pm 1.70$
	35	1.5	$92.8 \pm 0.02$	$3.30 \pm 0.28$	$25.2 \pm 1.70$
		3.0	$92.3 \pm 0.10$	$2.85 \pm 0.07$	$34.8 \pm 1.70$
		4.5	$93.2 \pm 0.03$	$1.85 \pm 0.07$	$44.4 \pm 1.70$
	50	1.5	$93.6 \pm 0.72$	$3.40 \pm 0.14$	$32.4 \pm 1.70$
		3.0	$94.0 \pm 0.51$	$2.55 \pm 0.07$	$39.6 \pm 1.70$
		4.5	$95.6 \pm 0.40$	$1.90 \pm 0.14$	$46.8 \pm 1.70$
1.5	20	1.5	$90.9 \pm 0.02$	$6.45 \pm 0.07$	$25.2 \pm 1.70$
		3.0	$90.8 \pm 0.22$	$6.10 \pm 0.14$	$32.4 \pm 1.70$
		4.5	$90.4 \pm 0.20$	$5.00 \pm 0.14$	$44.4 \pm 1.70$
	35	1.5	$92.1 \pm 0.79$	$4.40 \pm 0.14$	$26.4 \pm 3.39$
		3.0	$92.6 \pm 0.20$	$3.00 \pm 0.14$	$34.8 \pm 1.70$
		4.5	$93.0 \pm 0.20$	$1.95 \pm 0.07$	$42.0 \pm 1.70$
	50	1.5	$93.4 \pm 0.11$	$3.10 \pm 0.14$	$26.4 \pm 3.39$
		3.0	$94.3 \pm 0.61$	$2.95 \pm 0.07$	$33.0 \pm 3.39$
		4.5	$96.0 \pm 0.30$	$1.95 \pm 0.07$	$44.4 \pm 1.70$

Statistical analyses were performed with Minitab® Statistical Software package (2022 Minitab, LLC, Pennsylvania, USA).

## 4. Results and Discussion

**4.1. Calcium Pretreatment Effect.** Table 2 shows changes in moisture content ( $X_{wb}$ ), total soluble solids (TSS), and calcium gain under different processing conditions. After calcium pretreatment, the higher moisture content values were found at the higher temperature ( $50^{\circ}\text{C}$ ) and the prolonged immersion time (4.5 h) regardless of the calcium concentration used. The TSS content was degraded to  $1.70 \pm 0.14^{\circ}\text{Brix}$ , and the calculated calcium gain was from  $11.8 \pm 1.48$  to  $46.8 \pm 1.70$  mg/100 g of fresh sample, when higher conditions were used.

The effects of the processing conditions on the moisture content ( $X_{wb}$ ), total soluble solids (TSS), and calcium gain

are shown in Table 3. Significant differences ( $\alpha = 0.05$ ) were found between the factors and their interaction, except for the  $\text{Ca}(\text{OH})_2$  conc.-time interaction in the moisture content ( $P = 0.82$ ).  $X_{wb}$  showed significant differences, and higher values were obtained at  $50^{\circ}\text{C}$  due to tissue relaxation that allowed a greater water diffusion [26].  $X_{wb}$  increased during 4.5 h of immersion due to exposure of tissue to hypotonic solution; similarly, Oey et al. [27] observed a hypotonic effect in apple tissue. In contrast, a concentration of 1.5 g/100 mL  $\text{H}_2\text{O}$  conditioned the water flow towards the tissue, that is, the saturation effect of  $\text{Ca}^{2+}$  on surface samples was observed. The TSS values were significantly different, the highest degradation of TSS was found under conditions of  $50^{\circ}\text{C}$ , 4.5 h, and 0.5 g/100 mL  $\text{H}_2\text{O}$ , where a higher water content was obtained, which means that the degradation of carbohydrates by the alkaline effect depends on the  $\text{OH}^-$  ions [28], and the diffusion of  $\text{OH}^-$  was synergistic with the

TABLE 3: Effect of the processing conditions and their interactions on moisture content ( $X_{wb}$ ), total soluble solids (TSS), and calcium gain during calcium pretreatment of chilacayote cylinders.

Processing conditions	Main effects			
		$X_{wb}$ (%)	TSS (°Brix)	Calcium gain (mg/100 g sample)
Ca(OH) <sub>2</sub> concentration (g/100 mL of water)	0.5 *	94.0 ± 1.57 *	2.6 ± 0.84 *	31.1 ± 11.0
	1.0	93.8 ± 1.09	2.6 ± 0.58	35.7 ± 7.22
	1.5	92.7 ± 1.79	3.9 ± 1.61	34.3 ± 7.47
Temperature (°C)	20 *	92.9 ± 1.83 *	3.6 ± 1.68 *	30.4 ± 8.83
	35	92.6 ± 0.65	2.9 ± 1.00	33.6 ± 9.26
	50	94.9 ± 1.00	2.6 ± 0.54	37.2 ± 7.30
Time (h)	1.5 *	93.0 ± 1.44 *	3.8 ± 1.13 *	24.6 ± 6.04
	3.0	93.4 ± 1.40	3.1 ± 1.09	33.5 ± 3.19
	4.5	94.1 ± 1.80	2.3 ± 0.99	43.1 ± 4.54
Combined effects				
Ca(OH) <sub>2</sub> conc.-temperature		*	*	*
Ca(OH) <sub>2</sub> conc.-time		$P = 0.82$	*	*
Temperature-time		*	*	*
Ca(OH) <sub>2</sub> conc.-temperature-time		*	*	*

\*Significant differences with  $\alpha = 0.05$ .

diffusion of water to the tissue. A significant increase in calcium content was observed mainly at a higher solution temperature and a longer immersion time; Barrera et al. [29] reported a similar increase in apple tissue. A higher concentration gradient favored the calcium content; therefore, with calcium pretreatment, two ways of calcium are identified: (1) bound to carboxyl groups and (2) free or remaining unbound in the plant tissue [30].

Table 4 shows the values of texture parameters determined after calcium pretreatment. All the experiments with calcium solution increased the hardness of the samples with respect to their initial value, where it is probable that the cross-links formed between Ca<sup>2+</sup>-pectins and the turgor pressure exerted by the increase in moisture content have reinforced the cell walls. The lowest hardness value (139 ± 8.85 N) was obtained under lower processing conditions, while the highest value was obtained at 1 g/100 mL w, 20 °C, and 4.5 hours. This could indicate pretreatments with an unsaturated calcium solution, low temperature, and longer immersion time; the force necessary to deform the pretreated samples will be greater. Peng et al. [30] indicate that the maximum shear force increases with the amount of Ca<sup>2+</sup>-pectin bonds generated due to the addition of calcium and endogenous pectin methylesterase activated at 50–70 °C and 1% calcium concentration. The cohesiveness increased with a longer immersion time, which can be related to the increase in the formation of calcium structures from surface to inner of the samples; however, at higher temperatures, the cohesiveness is affected by the softening of the tissue. The adhesiveness increased in comparison fresh samples, and the work to overcome the attractive forces between the chilacayote cylinders and the probe was significantly increased with processing conditions. Possibly

due to moisture on the surface samples where the adhesion force is relevant. According to Nishinari et al. [31], conditions such as moisture content of samples and surface of probes are key during adhesiveness test. In general, the springiness decreased under processing conditions applied compared to initial value; in some experiments, the capacity of the cylinders to return to their undeformed state after compression remained with changes compared to control samples. Barrera et al. [32] mention that the formation of bonds in middle lamellae and cell walls, due calcium effect, increases the elastic behavior, rigidity, and fragility of cell network. Finally, the gumminess and chewiness increased in relation to fresh samples, mainly due to the increase in hardness values. The work required to chew a solid and semisolid food to a ready-to-swallow state increased significantly at a longer immersion time due to greater formation of cross-links from the surface to the inner of the samples; on the contrary, both parameters decreased at higher temperature because the cohesion force also decreases.

Figure 2 shows the effect of factors and their interactions on the texture parameters of chilacayote cylinders pretreated with calcium solutions. Calcium pretreatment conditions had significant effects ( $\alpha = 0.05$ ) on hardness, cohesiveness, adhesiveness, gumminess, and chewiness at different levels of importance and magnitude. Regarding springiness, the combination of conditions such as calcium concentration-time and calcium concentration-temperature-time did not show significant effects.

**4.2. Osmotic Pretreatment Effect.** The changes in moisture content ( $X_{wb}$ ), total soluble solids (TSS), and calcium retained of chilacayote cylinders pretreated osmotically during 2 h are presented in Table 5. The highest loss of water

TABLE 4: Changes in texture characteristics of chilacayote cylinders pretreated with calcium hydroxide solutions.

Ca(OH) <sub>2</sub> concentration (g/100 mL w)	Temperature (°C)	Time (h)	Hardness (N)	Cohesiveness (adim)	Adhesiveness (N.mm)	Springiness (adim)	Gumminess (N)	Chewiness (N)
0.5	20	1.5	139 ± 8.85	0.11 ± 0.03	0.28 ± 0.05	0.35 ± 0.02	15.3 ± 4.48	5.34 ± 1.49
		3.0	149 ± 9.97	0.12 ± 0.02	0.45 ± 0.14	0.37 ± 0.01	17.4 ± 4.41	6.43 ± 1.64
		4.5	228 ± 15.9	0.16 ± 0.02	1.92 ± 0.22	0.43 ± 0.03	46.8 ± 4.09	19.9 ± 1.97
	35	1.5	156 ± 25.6	0.13 ± 0.01	0.14 ± 0.03	0.42 ± 0.02	20.2 ± 2.99	8.52 ± 1.45
		3.0	169 ± 20.7	0.16 ± 0.01	0.24 ± 0.10	0.39 ± 0.03	26.9 ± 3.07	10.5 ± 1.84
		4.5	201 ± 9.17	0.17 ± 0.01	0.61 ± 0.12	0.36 ± 0.02	33.7 ± 2.09	12.3 ± 1.03
	50	1.5	210 ± 27.5	0.14 ± 0.02	1.31 ± 0.05	0.40 ± 0.04	29.8 ± 2.88	12.0 ± 1.56
		3.0	216 ± 13.9	0.08 ± 0.01	1.99 ± 0.14	0.35 ± 0.01	17.0 ± 1.03	5.93 ± 0.36
		4.5	194 ± 16.3	0.09 ± 0.01	2.15 ± 0.31	0.33 ± 0.01	16.6 ± 2.92	5.42 ± 1.02
1	20	1.5	221 ± 7.76	0.10 ± 0.02	0.34 ± 0.13	0.35 ± 0.01	21.1 ± 4.07	7.45 ± 1.34
		3.0	241 ± 16.3	0.16 ± 0.01	2.00 ± 0.15	0.37 ± 0.01	38.8 ± 3.36	14.3 ± 0.98
		4.5	250 ± 23.7	0.14 ± 0.01	2.47 ± 0.53	0.39 ± 0.01	35.9 ± 4.33	14.0 ± 1.75
	35	1.5	168 ± 24.1	0.13 ± 0.01	0.25 ± 0.04	0.42 ± 0.05	22.5 ± 2.95	9.51 ± 1.51
		3.0	183 ± 15.8	0.16 ± 0.02	0.46 ± 0.06	0.40 ± 0.02	28.6 ± 4.49	11.6 ± 1.90
		4.5	214 ± 22.9	0.15 ± 0.01	0.76 ± 0.07	0.38 ± 0.03	31.9 ± 3.57	12.1 ± 1.85
	50	1.5	221 ± 11.0	0.13 ± 0.02	1.67 ± 0.16	0.39 ± 0.04	27.7 ± 4.43	11.0 ± 2.81
		3.0	232 ± 5.85	0.07 ± 0.01	2.23 ± 0.14	0.35 ± 0.01	17.3 ± 0.97	6.06 ± 0.29
		4.5	185 ± 28.7	0.08 ± 0.01	2.85 ± 0.59	0.32 ± 0.01	15.2 ± 2.08	4.92 ± 0.66
1.5	20	1.5	228 ± 17.2	0.12 ± 0.01	0.77 ± 0.17	0.40 ± 0.03	27.7 ± 4.08	11.0 ± 1.32
		3.0	233 ± 12.4	0.11 ± 0.01	1.92 ± 0.16	0.42 ± 0.02	25.6 ± 1.51	10.7 ± 0.91
		4.5	227 ± 18.1	0.11 ± 0.01	2.68 ± 0.09	0.43 ± 0.02	25.4 ± 2.94	10.8 ± 1.36
	35	1.5	175 ± 19.8	0.12 ± 0.02	0.62 ± 0.06	0.43 ± 0.04	21.0 ± 2.98	9.14 ± 1.86
		3.0	201 ± 13.9	0.15 ± 0.01	0.87 ± 0.05	0.40 ± 0.03	29.4 ± 1.01	11.7 ± 1.11
		4.5	223 ± 12.5	0.16 ± 0.01	1.79 ± 0.29	0.37 ± 0.01	35.6 ± 2.35	13.3 ± 0.60
	50	1.5	218 ± 13.0	0.15 ± 0.01	1.20 ± 0.41	0.39 ± 0.04	33.5 ± 3.52	13.1 ± 2.14
		3.0	201 ± 11.0	0.09 ± 0.01	2.07 ± 0.46	0.36 ± 0.01	18.4 ± 0.86	6.56 ± 0.24
		4.5	171 ± 16.7	0.08 ± 0.02	2.66 ± 0.31	0.33 ± 0.01	13.7 ± 1.22	4.50 ± 0.35

was determined at high concentration and temperature, the moisture content decreased to  $53.0 \pm 0.57\%$  compared to  $81.3 \pm 0.26\%$  determined at low concentration and temperature. Similarly, the solid gain increased to  $50.0 \pm 0.21$  °Brix, and it was attributed to an increase in the concentration of sucrose in the osmotic solution. Barragán-Iglesias [10] found that mass transfer during osmotic dehydration is more effective at high concentration and high temperature of sucrose solution until equilibrium is reached. In contrast, the calcium gain obtained during the calcium pretreatment changed negatively by the effect of the osmotic solution; therefore, the calcium retained in the tissue decreased to  $12.6 \pm 0.21$  mg Ca/100 g sample. Barrera et al. [29, 32] reported a reduction in calcium content after osmosis due to the concentration gradient. Also, Martínez-Sánchez et al. [33] observed a leaching effect in banana tissue at higher concentration and temperature of the osmotic solution.

The effects of the processing conditions on the moisture content ( $X_{wb}$ ), total soluble solids (TSS), and retained calcium are shown in Table 6. Moisture content was significantly affected ( $\alpha = 0.05$ ) during all applied processing conditions and their interactions. In general, the reduction of the moisture content was directly proportional to the processing conditions of the osmotic solution, that is, at higher temperature and concentration, the water loss of the plant tissue was also greater. Therefore, it was considered that the calcium pretreatment did not limit the outflow of water from plant tissue, but rather facilitated it due to microstructure modification. TSS content increased significantly with higher osmotic concentration and temperature, demonstrating that the  $\text{Ca}^{2+}$  contained within the tissue did not affect sucrose transfer. Barragán-Iglesias et al. [10] indicates that with calcium pretreatment and osmosis, the main effect obtained is the moisture loss and solute gain, independently

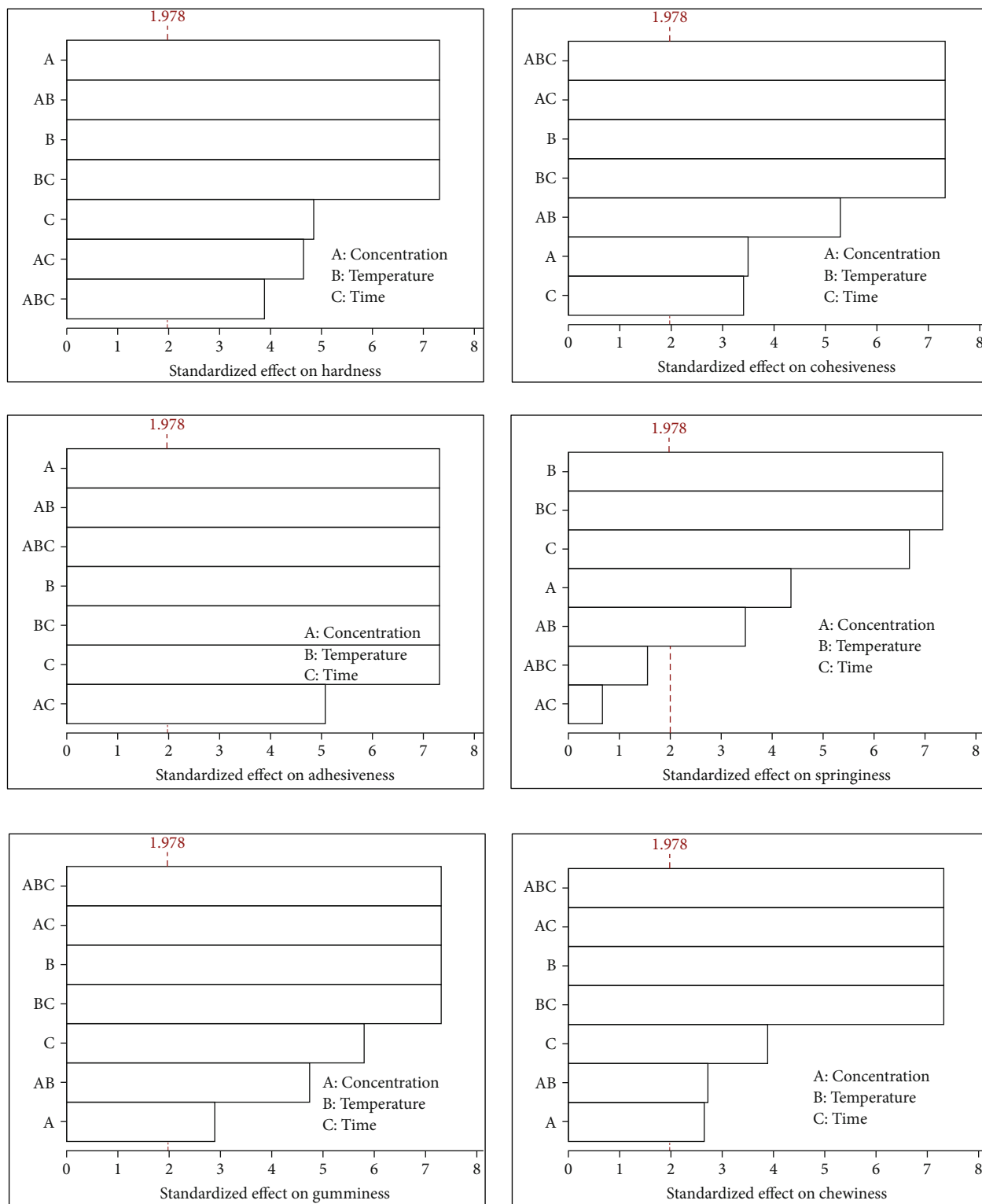


FIGURE 2: Effect of factors and their interactions on texture parameters of chilacayote cylinders pretreated with calcium hydroxide solution. Factors and their interactions that cross the reference line (1.978) are statistically significant with  $\alpha = 0.05$ .

of calcium gain. The calcium retained was affected negatively by the factors and their interactions because the osmotic solution favors the lixiviation of calcium; also, the relaxation of the tissue due to thermal effect favored the calcium loss, free or not bound to pectins, within the tissue.

The changes in texture parameters of chilacayote cylinders pretreated osmotically during 2h are presented in Table 7. The hardness value (Table 4) obtained after the selected calcium pretreatment (1 g Ca/100 ml H<sub>2</sub>O, 35 °C, and 3 h) increased during osmosis at temperatures of 30

TABLE 5: Changes in moisture content ( $X_{wb}$ ), total soluble solids (TSS), and calcium content of chilacayote cylinders pretreated osmotically during 2 h.

Osmotic concentration (°Brix)	Temperature (°C)	$X_{wb}$ (%)	TSS (°Brix)	Calcium retained (mg/100 g fresh sample)
30	30	81.3 ± 0.26	21.1 ± 0.21	18.0 ± 1.70
	50	80.4 ± 1.45	22.0 ± 0.14	14.4 ± 0.21
	70	74.0 ± 0.88	24.2 ± 0.42	14.3 ± 0.22
45	30	71.6 ± 0.02	28.4 ± 0.14	15.2 ± 0.14
	50	72.7 ± 0.90	30.0 ± 0.21	13.2 ± 0.14
	70	66.7 ± 1.30	35.7 ± 0.14	13.7 ± 0.14
60	30	65.0 ± 0.09	32.0 ± 0.16	13.0 ± 0.17
	50	61.1 ± 1.14	49.0 ± 0.21	12.6 ± 0.21
	70	53.0 ± 0.57	50.0 ± 0.21	13.0 ± 0.12

TABLE 6: Processing conditions effect and their interaction on the moisture content ( $X_{wb}$ ), total soluble solids (TSS), and calcium retained after osmotic dehydration of chilacayote cylinders.

Processing conditions	Main effects			
	$X_{wb}$ (%)	TSS (°Brix)	Calcium retained (mg/100 g sample)	
Osmotic concentration (°Brix)	30 *	78.6 ± 3.42 *	22.4 ± 1.39 *	15.6 ± 1.87
	45	70.3 ± 2.75	31.4 ± 3.21	14.3 ± 1.09
	60	59.7 ± 5.11	43.6 ± 8.50	12.9 ± 0.25
Temperature (°C)	30 *	72.6 ± 6.97 *	27.1 ± 4.68 *	15.4 ± 2.18
	50	71.4 ± 8.19	33.6 ± 11.7	13.4 ± 0.78
	70	64.6 ± 8.96	36.6 ± 10.4	13.7 ± 0.58
Combined effects				
Osmotic conc.-temperature	*	*	*	

\*Significant differences with  $\alpha = 0.05$ .

TABLE 7: Changes in texture characteristics of chilacayote cylinders pretreated osmotically during 2 h.

Osmotic conc. (°Brix)	Temperature (°C)	Hardness (N)	Cohesiveness (adim)	Adhesiveness (N.mm)	Springiness (adim)	Gumminess (N)	Chewiness (N)
30	30	270 ± 17.2	0.07 ± 0.01	1.84 ± 0.53	0.34 ± 0.01	18.4 ± 3.66	6.33 ± 1.24
	50	231 ± 15.4	0.06 ± 0.01	2.31 ± 0.10	0.33 ± 0.01	14.2 ± 2.34	4.62 ± 0.75
	70	147 ± 11.2	0.05 ± 0.01	2.59 ± 0.42	0.32 ± 0.01	7.29 ± 1.09	2.32 ± 0.33
45	30	224 ± 8.60	0.05 ± 0.01	2.17 ± 0.26	0.33 ± 0.01	11.4 ± 1.71	3.81 ± 0.60
	50	223 ± 11.8	0.05 ± 0.01	2.51 ± 0.17	0.32 ± 0.01	11.7 ± 2.27	3.80 ± 0.75
	70	144 ± 4.70	0.04 ± 0.01	2.69 ± 0.31	0.31 ± 0.01	6.20 ± 0.56	1.94 ± 0.18
60	30	216 ± 6.30	0.05 ± 0.01	2.32 ± 0.20	0.32 ± 0.01	11.2 ± 1.87	3.60 ± 0.53
	50	220 ± 8.70	0.05 ± 0.01	2.52 ± 0.08	0.31 ± 0.01	11.0 ± 1.55	3.24 ± 0.57
	70	142 ± 7.06	0.04 ± 0.01	2.85 ± 0.13	0.30 ± 0.02	6.02 ± 0.79	1.80 ± 0.24

and 50 °C regardless of the concentration of the osmotic solution; however, the loss of hardness was evident at 70 °C. Osmotic treatment at high temperatures has been reported to cause softening of plant tissue [34]. The cohesion

force decreased to  $0.04 \pm 0.01$  compared to  $0.16 \pm 0.02$  obtained after calcium treatment, possibly loss of water, leaching of  $\text{Ca}^{2+}$ , and degradation of other substances decreased the hardness and the cohesiveness. Truong et al.



[35] mention that the compressive strength decreases during cooking due to the magnitude of starch degradation and substances in the cell wall.

Adhesiveness shows a positive trend at higher osmotic concentration and temperature, it was accompanied by a gain of solutes (sucrose); in other words, the ability of the samples to adhere depends on the sucrose impregnated on the surface of the samples. With loss of water and incorporation of solutes, springiness showed a negative trend; therefore, the recovery capacity of the samples was affected by the processing conditions used during the osmotic pretreatment. Lastly, the force to chew and disintegrate the samples to a ready-to-swallow state decreased at higher concentration and temperature, this means that osmotic dehydration softened the tissue making samples easier to taste. Generally, osmotic pretreatment affects the samples due to the temperature and concentration of the osmotic solution, which exerts a high osmotic pressure on the tissue, causing a loss of water and therefore a loss of turgidity and cohesion force.

Figure 3 shows the effect of factors and their interactions on the texture parameters of osmotically dehydrated chilacayote cylinders. Processing conditions used during osmotic dehydration had significant effects ( $\alpha = 0.05$ ) on the texture parameters. The temperature levels used caused the most important effect on hardness, adhesiveness, springiness, gumminess, and chewiness; in this sense, cohesiveness is mostly affected by the concentration of the osmotic solution. The combined concentration-temperature effect can significantly modify the hardness values; on the contrary, this combination did not cause significant changes in the other texture parameters.

**4.3. General Analysis of Texture Parameters.** Figure 4 shows the changes in physical texture parameter during calcium and osmotic pretreatments. The mean values of the texture parameters obtained during the pretreatments indicate that the hardness increased significantly with the calcium pretreatment and that such a parameter was maintained during osmotic dehydration despite the osmotic pressure exerted on the plant tissue. The cohesion force increased significantly for any condition of calcium pretreatment, but this acquired force is lost during the osmotic dehydration, mainly due to the osmotic pressure exerted on tissue, and therefore water loss. Calcium-pretreated samples showed greater detachment work from the probe because the adhesiveness increased significantly ( $\alpha = 0.05$ ) compared to fresh samples, and after osmotic pretreatment, the highest adhesiveness values were observed due to the influence of the impregnated osmotic agent (sucrose) on the surface. The recovery capacity of samples was significantly affected with the osmotic dehydration, possibly because at high temperature and concentration, and the structure of plant tissue was softened causing a loss in the elasticity (tendency to plastic behavior) compared with samples pretreated with calcium and control. Guminess and chewiness had a similar behavior when applying pretreatments, both parameters showed a significant increase due to interaction of  $\text{Ca}^{2+}$  ions with plant tissue, and the force required to disintegrate the sam-

ples was higher; however, after osmotic dehydration, this force decreased significantly ( $\alpha = 0.05$ ).

Texture parameters were modified with the application of calcium pretreatment, osmotic dehydration, or both pretreatments combined. The hardness is adequately represented by instrumentally recorded force in food materials with different sizes and shapes [36]. Barragán-Iglesias et al. [37] reported that the hardness of plant tissue increases with the application of calcium. It is because pectin compounds are the key substances responsible for the mechanical strength of the primary cell wall [38], that is, the acidic part ( $\text{COO}^-$ ) of pectin interacts with  $\text{Ca}^{2+}$  during pretreatment to increase tissue hardness. Yang et al. [39] showed that at high pH of the calcium solution, crosslinking formation ( $\text{COO}^- - \text{Ca}^{2+}$ ) was favored by  $\beta$ -elimination reactions. On the contrary, calcium solutions with neutral pH did not show significant differences in the hardness of potato and apple tissue [40]. During osmosis, the increase in hardness is related to the gain of solute [17]. This means that the impregnation of sucrose mainly on surface samples is responsible for the resistance to double compression.

Cohesiveness is related to the change of the curve during the second compression with respect to first compression [41]. A smaller decrease in the second-curve area with respect to the first curve caused an increase in the cohesion force. Calcium pretreatment increases the cohesiveness due to a major cross-link formation inner tissue of plant; this is, calcium pretreatment allows small displacements of the cell walls with each other when a force is applied [37]. Nishinari et al. [31] mention that cohesiveness can be interpreted as a rate of recoverability to an applied force on the sample. However, OD affects the cohesiveness due to moisture loss and consequently the loss of turgor pressure of the plant tissue. Ochoa-Martínez et al. [42] found a decrease in cohesiveness values during osmotic pretreatment at high temperature in apple samples. Additionally, osmotic pretreatment after a calcium pretreatment decreases the cohesion force according to Udomkun et al. [43].

Adhesiveness is adequately represented when considering a practically superficial compression (not a puncture test) and surface properties of the plunger [31, 36]. In this work, the variations in the adhesiveness values during the calcium pretreatment are affected by the processing conditions, such as the temperature according to Brenner & Nishinari [44]. The increase in the adhesiveness values of chilacayote is correlated with the results for papaya cubes pretreated with a  $\text{Ca}(\text{OH})_2$  solution reported by Barragán-Iglesias et al. [37]. The solutes impregnated during osmosis increased the adhesiveness values due to the stickiness that sucrose impregnation provides on the surface of the samples. According to Muñoz-Becerá et al. [45], the sucrose concentration is higher on the surface of the plant tissue during osmosis, and the stickiness due to the osmotic agent provides greater uniformity in the adhesiveness values [18]. Furthermore, with different osmotic agents such as sucrose and glucose, the adhesiveness values increase during osmosis [18].

The springiness of processed fruits provides an idea of the elastic or plastic nature of plant tissue. An elastic nature

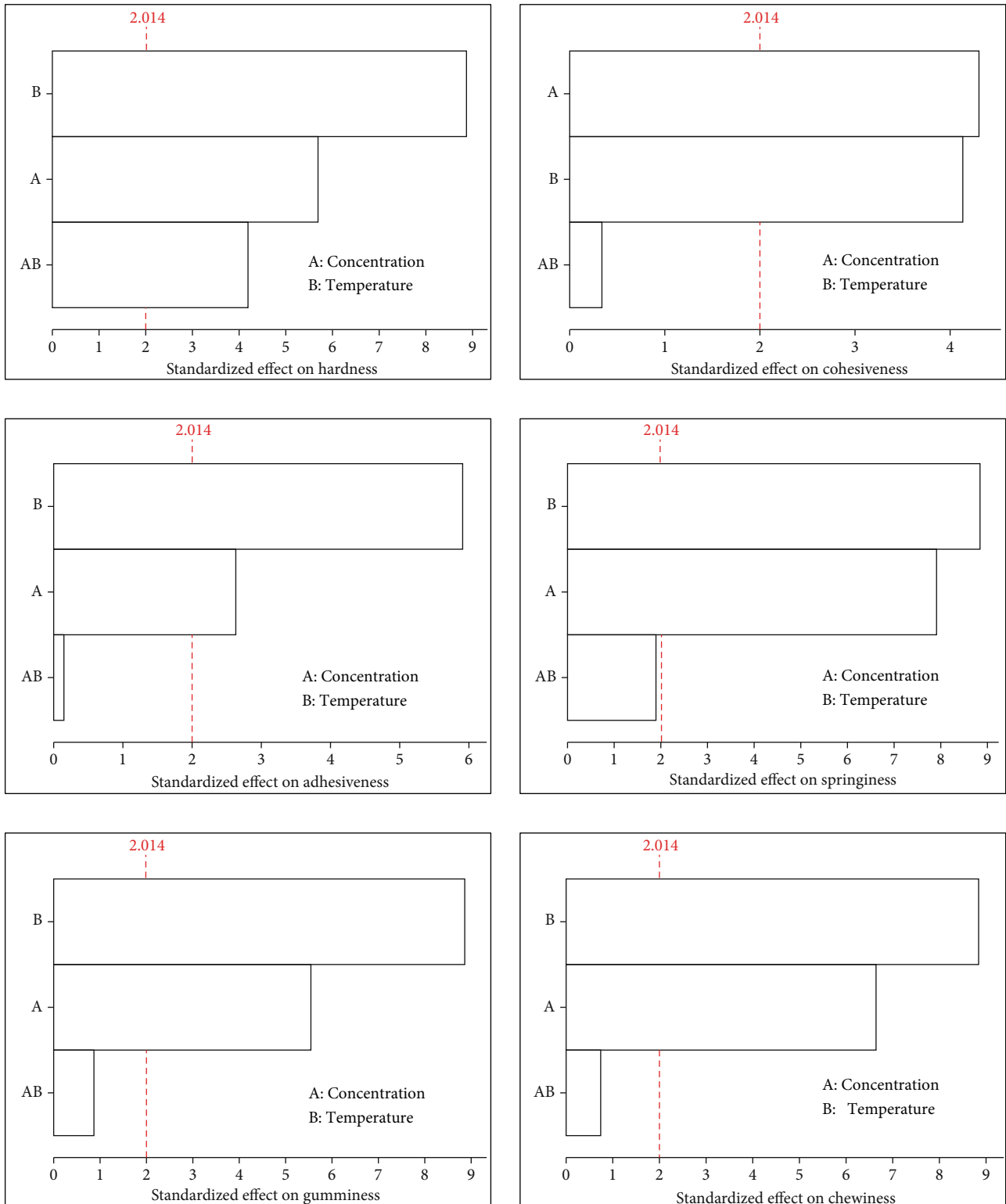


FIGURE 3: Effect of factors and their interactions on texture parameters of osmotically dehydrated chilacayote cylinders. Factors and their interactions that cross the reference line (2.014) are statistically significant with  $\alpha = 0.05$ .

was observed in fresh plant tissues, that is, fruits and vegetables are formed by extensible and somewhat elastic cell walls [46]. When calcium pretreatment is applied, the tissue was firmer and more rigid, so the tissue loses a certain degree

of springiness (elastic nature). Fruit tissues pretreated with calcium and osmotically acquire a “crystallized” appearance, so springiness is even lower compared to fresh samples or only pretreated with calcium. Cao et al. [17] showed that

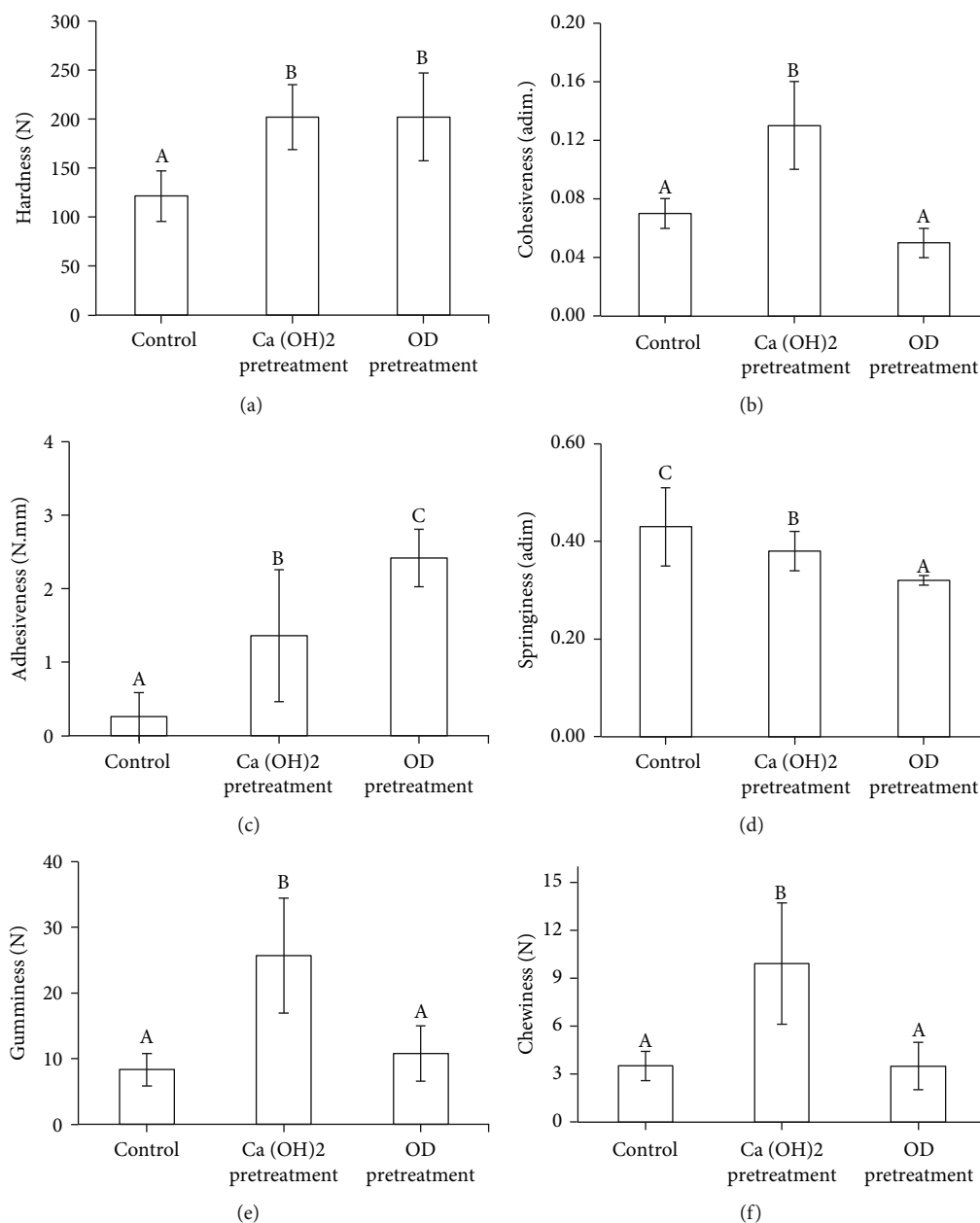


FIGURE 4: Changes in physical texture parameters of chilacayote cylinders during calcium and osmotic pretreatments with respect to fresh samples. Different letters above of  $M \pm SD$  indicate significant differences within factors with Tukey test ( $\alpha = 0.05$ ).

springiness decreases during OD with sucrose solutions. This means that with moisture loss and solutes gain, the osmodehydrated fruits become more brittle [18].

The gumminess of fresh tissue is low because they do not have a gummy characteristic like confectionery products. In fresh plant matrix, the gumminess is low due to the high moisture content and the crumbling behavior presented during compression force [10]. Gumminess was influenced by the higher hardness and the higher cohesion force acquired during alkaline calcium pretreatment. In cape gooseberry fruits pretreated under alkaline conditions, gumminess also increases [47]. In contrast, the gummy characteristic is affected by the decrease in the recovery capacity of tissue after osmosis. This modification can be attributed to cell tur-

gor and cell wall integrity [18]. This means that the force required to disintegrate the pretreated chilacayote samples to a ready state for swallowing was as fresh samples.

The chewiness of food is considered the force required to chew a solid food until it is ready to swallow and is a key parameter during oral processing [48]. Chewing movements are highly correlated with oral processing time, and both parameters increase as the hardness of the cellular food structure increases [49, 50]. In this work, the chewiness increased during calcium pretreatment mainly due to an increase in hardness values. In support of these data, similar results in papaya tissue have been reported by Barragán-Iglesias et al. [37]. After OD, chilacayote tissue maintains its hardness and loses its recoverability and springiness;

consequently, chewiness decreases. In dehydrated products, low chewiness is key because it facilitates the process of mastication and swallowing of solid food [50].

## 5. Conclusions

Processing conditions used in calcium and osmotic pretreatments significantly modified the mass transfer and the texture in mesocarp samples of chilacayote (*Cucurbita ficifolia* Bouché). Processing conditions used during calcium pretreatment significantly increased calcium gain and moisture content. In contrast, the total soluble solids decreased due to alkaline effect of the solution. Furthermore, calcium pretreatment allowed the transfer of ions ( $\text{Ca}^{2+}$  and  $\text{OH}^-$ ) and water to chilacayote tissue. The effect of osmotic dehydration on mesocarp samples was significant, and water loss and solid gain increased significantly under the highest conditions, showing that previous calcium pretreatment did not affect mass transfer between the osmotic solution and the tissue. However, the calcium gained was leached, so the calcium retained decreased considerably.

Regarding texture, the main factors and their combined effect modified the texture values after calcium pretreatment, increasing hardness, cohesiveness, adhesiveness, gumminess, and chewiness. However, with osmotic dehydration, only the hardness was maintained, and the adhesiveness increased because sucrose was impregnated on the surface samples.

The conditions of calcium pretreatment (1 g/100 mL water, 35 °C, and 3 h) and osmosis (45 °Brix, 50 °C, and 2 h) provide a minimally processed product with intermediate moisture, calcium gain of up to 100%, and higher solid gain.

## Data Availability

Data are available on request to corresponding author.

## Ethical Approval

This research did not involve any human or animal ethics issues to be considered.

## Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

## Authors' Contributions

Juan Rodríguez-Ramírez did the data curation, investigation, and writing—review and editing. Josué Barragán-Iglesias did the conceptualization, investigation, methodology, supervision, visualization, and writing—review and editing. Atenea J. Ramírez-Palma did the methodology, data collection, and analysis tools. Lilia L. Méndez-Lagunas did the financial, supervision, visualization, and writing—review and editing.

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## Supplementary Materials

The complementary material section shows the values of texture parameters obtained at different process conditions used during the calcium and osmotic pretreatments, as well as the corresponding statistical analysis. The effect of factors and their interactions during calcium pretreatment on texture parameters is shown in Supplementary Table 1. Most of the factors, as well as their interactions, had a significant effect ( $\alpha = 0.05$ ) on texture parameters. However, the interactions such as the  $\text{Ca}(\text{OH})_2$  concentration-time and  $\text{Ca}(\text{OH})_2$  concentration-temperature-time did not show a significant effect ( $P > 0.05$ ) on the springiness. Supplementary Table 2 shows the effect of factors and their interactions during osmotic dehydration on texture parameters. The interaction osmotic concentration-temperature had no significant effect ( $P > 0.05$ ) on cohesiveness, adhesiveness, springiness, gumminess, and chewiness. In all conditions used an important effect showed changes on texture parameters, which allows select process conditions considering saving energy and less intake sucrose. (*Supplementary Materials*)

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