# Effect of calcium salts on the texture, structure and sensory acceptance of osmotically dehydrated guavas



Leila M Pereira,<sup>1</sup> Sandra M Carmello-Guerreiro,<sup>2</sup> Helena MA Bolini,<sup>3</sup> Rosiane L Cunha<sup>1</sup> and Miriam D Hubinger<sup>1</sup>\*

<sup>1</sup>Department of Food Engineering, Faculty of Food Engineering, State University of Campinas, P.O. Box 6121, CEP 13083-862, Campinas, SP, Brazil

<sup>2</sup>Department of Botany, Biology Institute, State University of Campinas, Brazil

<sup>3</sup>Department of Food Planning and Nutrition, Faculty of Food Engineering, State University of Campinas, Brazil

Abstract: The effect of additives, calcium chloride and calcium lactate  $(5-25 \text{ g kg}^{-1})$ , on the osmotic dehydration of guavas with sucrose solutions was studied, aiming at the structural preservation of processed fruits. The osmotic process was evaluated from the reduction in weight of the guavas, water loss and solids gain, and the samples were analyzed with respect to calcium content, texture (stress and strain at failure, relaxation time and residual stress), structure by light microscopy and sensory acceptance. Calcium salts had a strong influence on the texture and structure of the processed guavas, resulting in the maintenance of tissue structure when calcium lactate was used at concentrations up to  $15 \text{ g kg}^{-1}$ , and calcium chloride was used at  $5 \text{ g kg}^{-1}$ . The sensory acceptability of guava was related to the structural and texture results. Calcium treatments did not improve guava's sensory acceptance. Guavas treated with calcium lactate showed good sensory acceptance, presenting slight inferior scores only at concentrations above  $20 \text{ g kg}^{-1}$ , while CaCl<sub>2</sub> treated guavas showed average scores statistically equal to the sucrose and calcium lactate treated fruits only at  $5 \text{ g kg}^{-1}$ .

© 2007 Society of Chemical Industry

Keywords: guava; osmotic dehydration; structure; sensory analysis; calcium chloride; calcium lactate

## INTRODUCTION

Consumer demand for high-quality products with fresh-like characteristics has promoted an important change in food preservation techniques during the last few years. Mild treatments, which could preserve these characteristics, have been preferred to severe treatments such as drying, freezing and pasteurization. The osmotic dehydration process has appeared as an important technology for the development of fruit products. The slight reduction in water activity and the possible incorporation of additives, such as antimicrobials, antioxidants and firming agents, promoted by the osmotic process, associated with mild heat treatments, modified atmosphere packaging and refrigeration or other mild preservation techniques, can provide stable products with good nutritional and sensorial quality and with characteristics similar to those of the fresh products. $^{1-3}$ 

However, structural changes noticed in the product texture, are frequently observed as a result of the osmotic process.<sup>4–6</sup> Loss of cell turgidity, deformation and/or cell wall rupture, splitting and degradation of the middle lamella, lysis of membranes (plasmalemma and tonoplast), cellular collapse, plasmolysis and tissue shrinkage are indicated as the main effects of osmotic dehydration on the cellular structure of plant tissues.<sup>2,6-9</sup>

The application of calcium salts to fruit, before or after harvest, is usually used to delay ripening and senescence and to prevent physiological disorders in several fruits.<sup>10</sup> These salts have also been widely used in recent years for the structural preservation of processed tissues. The addition of salts to the osmotic solution, calcium infiltration as a pre-treatment to the osmotic dehydration process or calcium salt dips in combination with mild heat treatments have resulted in improvements in product texture and have shown a protective effect on tissue structure.<sup>4,6,8,11-13</sup> The action of calcium on the cellular structure has been explained by its effect on the pectin matrix present in the cell wall of plant tissues. The interaction of  $Ca^{2+}$ and pectin provide rigidity to the cell wall, favoring the maintenance of product texture.<sup>14</sup>

Treatment with calcium salts has also shown an effect on the mass transfer kinetics of osmotically dehydrated products. As observed by Lewicki *et al.*,<sup>15</sup>

\* Correspondence to: Miriam D Hubinger, Department of Food Engineering, Faculty of Food Engineering, State University of Campinas, P.O. Box 6121, CEP 13083-862, Campinas, SP, Brazil

E-mail: mhub@fea.unicamp.br

- Contract/grant sponsor: FAPESF
- Contract/grant sponsor: CNPq
- Contract/grant sponsor: CAPES/GRICES

(Received 25 October 2005; revised version received 28 June 2006; accepted 5 July 2006) Published online 12 March 2007; DOI: 10.1002/jsfa.2836

© 2007 Society of Chemical Industry. J Sci Food Agric 0022–5142/2007/\$30.00



an increase in water and sucrose transport was verified when calcium salts were used as a pre-treatment in the osmotic process of tomato quarters. Penetration of calcium ions and formation of bridges between pectin molecules stiffen the tissue, increasing the resistance to deformation. As a result of this, an open structure is formed favoring the mass transport.<sup>15</sup>

Calcium chloride is a calcium salt commonly used in the preservation of fruits and vegetables but in some products like apples<sup>11</sup> and cantaloupes,<sup>16</sup> it may impart bitterness or changes in flavor. The use of calcium lactate has been proposed as an alternative source of calcium<sup>4,6,17</sup> but the effects of both calcium salts on osmotically dehydrated guavas have not yet been reported.

Thus, the objective of this work was to compare the effect of the application of two different calcium salts, calcium chloride or calcium lactate, on the texture (stress and strain at failure, relaxation time and residual stress), structure (light microscopy) and sensory characteristics (acceptance tests) of osmotically dehydrated guavas, aiming at the structural preservation of processed fruits. The action of the calcium salt on the mass transfer of the osmotic process and the incorporation of calcium into the fruit were also evaluated and related to its structural preserving effect.

#### MATERIALS AND METHODS Materials

Red guavas (*Psidium guajava* L) of the Paluma cultivar (Table 1) supplied by Val Fruits Industry (Vista Alegre do Alto, SP, Brazil) were used in the trials approximately 4–5 days after harvest, when the fruits reached a suitable ripening grade. A lot of around 200 guavas (four boxes of 7 kg each) was bought and fruit sampling was based on ripening grade  $(7-8^{\circ}\text{Brix} \text{ and } 80\% \text{ of skin yellowness})$ , shape and size  $(7.8 \pm 0.4 \text{ cm length}, 6.7 \pm 0.2 \text{ cm diameter}, 1.0 \pm 0.1 \text{ cm pericarp thickness}, and weight of <math>152.0 \pm 7.8 \text{ g}$ ), using six guavas (12 halves) for each treatment, totaling 66 guavas for the whole experiment. After the processing, the guava halves were stored at 5 °C until their evaluation, which was carried out no longer than 72 h after treatment.

Table 1. Composition of guava (Paluma cultivar)

Mean value <sup>a</sup> (g kg <sup>-1</sup> )	Method
$872.6 \pm 4.8$	Ranganna <sup>18</sup>
$5.4 \pm 0.3$	Ranganna <sup>18</sup>
$14.2 \pm 0.6$	Ranganna <sup>18</sup>
$3.1 \pm 0.5$	Bligh and Dyer <sup>19</sup>
$28.0 \pm 3.1$	Ranganna <sup>18</sup>
69.1	By difference
$7.6\pm0.1$	Ranganna <sup>18</sup>
	$\begin{array}{c} \text{Mean value}^{a} \\ (g \ \text{kg}^{-1}) \\ \hline 872.6 \pm 4.8 \\ 5.4 \pm 0.3 \\ 14.2 \pm 0.6 \\ 3.1 \pm 0.5 \\ 28.0 \pm 3.1 \\ 69.1 \\ 7.6 \pm 0.1 \\ \end{array}$

 $^{\rm a}$  All data were obtained by triplicate analyses and expressed as mean  $\pm$  standard deviation.

## **Osmotic dehydration**

The guavas were washed with tap water (pH 7.9 and electrical conductivity (EC)  $105 \mu S \text{ cm}^{-1}$ ) and dipped in a 5.5 g kg<sup>-1</sup> solution of chlorinated sanitizer (165 ppm active chlorine and pH 6.2) for 10 min (Diversey Lever, São Paulo, SP, Brazil). The sanitized fruits were peeled using NaOH solution at 20 g kg<sup>-1</sup>, washed with tap water and dipped in a new solution of the chlorinated sanitizer at 5.5 g kg<sup>-1</sup> for 10 min. The guavas were then cut into halves and the seeds removed.

Guava halves (one half guava per flask) were soaked in a 60°Brix sucrose solution (pH 6.7) with or without the addition of 5, 10, 15, 20 and 25 g kg<sup>-1</sup> of calcium chloride, with pH 5.9, 5.8, 5.7, 5.7 and 5.6 respectively, or calcium lactate, with pH 8.5, 8.9, 9.2, 9.3 and 9.5, respectively.<sup>16</sup> The mass ratio of product to solution of 1:10 was used in the process. The solutions with the samples were placed in a thermostatic shaker (TE 420, Tecnal, Piracicaba, SP, Brazil) at 120 rpm and 40 °C for 2 h, process conditions optimized in a previous study performed by Argandoña.<sup>20</sup> After this process, the samples were rinsed with the chlorinated sanitizer solution at 2 g kg<sup>-1</sup> (60 ppm active chlorine and pH 6.2) and placed on absorbent paper to remove excess solution.<sup>3</sup>

The samples were named FR (without treatment), SUC (osmotically dehydrated in a 60°Brix sucrose solution), CaLAC 5 to CaLAC 25 (osmotically dehydrated in a 60°Brix sucrose solution with the addition of  $5-25 \text{ g kg}^{-1}$  of calcium lactate) and CaCl<sub>2</sub> 5 to CaCl<sub>2</sub> 25 (osmotically dehydrated in a 60°Brix sucrose solution with the addition of  $5-25 \text{ g kg}^{-1}$  of calcium chloride). FR and SUC samples were used as controls in order to evaluate the effect of adding low to high concentrations of calcium salts. An additional comparison between FR and SUC samples was done to evaluate the effect of osmotic dehydration on guava properties.

Weight reduction (WR), water loss (WL) and solids gain (SG), expressed in g  $100 \text{ g}^{-1}$  of the initial fresh fruit, were analyzed using Equations 1, 2 and 3. The changes in weight were obtained by weighing the samples before and after the osmotic treatment and the weight of water and solids calculated from the sample moisture content, determined according to AOAC methods.<sup>21</sup> Three guava halves from each treatment, taken from different fruits, were used for these measurements and the mean values were reported.

$$WR = \frac{w_i - w_f}{w_i} \times 100$$
(1)

$$WL = \frac{w_{wi} - w_{wf}}{w_i} \times 100$$
 (2)

$$SG = \frac{w_{sf} - w_{si}}{w_{i}} \times 100$$
(3)

where  $w_i$  is the initial weight of the sample (g),  $w_f$  is the final (after osmotic dehydration) weight of the sample (g),  $w_{wi}$  is the initial weight of water in the sample (g),

 $w_{\rm wf}$  is the final weight of water in the sample (g),  $w_{\rm si}$  is the initial weight of solids in the sample (g), and  $w_{\rm sf}$  is the final weight of solids in the sample (g).

#### **Determination of calcium content**

The calcium concentrations of the fresh and osmotically dehydrated samples were determined using an inductively coupled plasma atomic emission spectrometer (ICP 2000, Baird, MA, USA).<sup>21</sup> The calcium content was determined in three different guava halves from each treatment and the mean of the values obtained was reported.

#### **Evaluation of texture**

The guava texture was analyzed by uniaxial compression tests using a Universal Testing Machine (TA.XT2i Texture Analyzer, Stable Micro Systems, Godalming, Surrey, UK). The stress and strain at failure were determined using a 30 mm diameter lubricated acrylic plate at a crosshead speed of 1 mm s<sup>-1</sup> until 70% sample deformation. A 10mm diameter cylindrical sample removed from the center of the guava halves was used for these assays. The force and height data obtained from this test were converted to Hencky stress  $\sigma_{\rm H}$  and strain  $\varepsilon_{\rm H}$ . The stress and strain at failure were determined from the peak of the stress-strain curve.<sup>22</sup> The relaxation time and residual stress were determined by using the same acrylic plate at an initial speed of  $7 \text{ mm s}^{-1}$ , keeping the samples under 5% strain for 600s. The resulting stress-relaxation curve was linearized (Equation 4) according to Peleg<sup>23</sup> and the relaxation time,  $\tau$ , and residual stress,  $S_r$ , calculated using the constants  $k_1$ (viscous element) and  $k_2$  (elastic element), according to Equations 5 and 6:24

$$\frac{\sigma_0 t}{\sigma_0 - \sigma_t} = k_1 + k_2 t \tag{4}$$

$$\tau = \frac{k_1}{4 - k_2} \tag{5}$$

$$S_r = 1 - \frac{1}{k_2} \tag{6}$$

where  $\sigma_0$  and  $\sigma_t$  are the initial stress and the stress at time *t*, respectively. Five guava halves from each treatment were taken for texture measurements and the mean of values obtained was reported.

## Light microscopy

Samples ( $\sim 5 \text{ mm} \times 3 \text{ mm} \times 3 \text{ mm}$ ) from the flesh tissue of fresh and osmotically dehydrated guavas were fixed in 40 g kg<sup>-1</sup> glutaraldehyde in phosphate buffer (pH 7.0) with 40 g kg<sup>-1</sup> added sucrose, and dehydrated in a graded ethanol series. The dehydrated samples were embedded in hydroxyethyl methacrylate historesin (Leica Microsystems, Jung, Heidelberg, Germany) and sectioned using a rotary microtome (820 Spencer Microtome, American Optical Corporation, New York, USA). Sample sections measuring 8 µm were stained with toluidine blue O in acetate buffer (pH 4.7)<sup>25</sup> and examined using an Olympus BX 51 light microscope (Olympus Optical Co., Tokyo, Japan). Two samples of different fruits, from each treatment, were used for microscopic evaluation.

## Sensory analysis

Sensory acceptance tests were carried out in a standardized test room, 1 day after processing the guavas. The samples were presented in completely randomized blocks, in monadic form, with a time interval of 30 min between each sample, using white saucers labeled with three-digit random number codes. Color, aroma, flavor, texture and overall impression of the samples were evaluated by 30 panelists who were guava consumers, and representative of the target public, using a 9 cm unstructured hedonic scale anchored with 'I dislike very much' on the left side and 'I like very much' on the right side. An average score of 4.5 was considered the limit for acceptability.<sup>26</sup>

#### Statistical analysis

The experimental design used was completely randomized. Data were subjected to one-way analysis of variance using STATISTICA 5.0 (StatSoft, Inc., Tulsa, OK, USA). Mean separation was done using Tukey's test at P < 0.05.

#### **RESULTS AND DISCUSSION**

## Calcium content, weight reduction, water loss and solids gain

The calcium content of the fresh guavas was  $0.815 \pm 0.007 \, g \, kg^{-1}$  dry matter (mean value  $\pm$  standard deviation) and the osmotic dehydration process using sucrose solution retained around 70% of the calcium present in the raw material (Fig. 1). The use of calcium salts in the osmotic treatment resulted in a meaningful increase in calcium content of the osmotically dehydrated guavas, reaching values of  $7.800 \pm 0.052 \, g \, kg^{-1}$  dry matter.

According to Lewicki *et al.*<sup>15</sup> the osmotic process for tomatoes pre-treated in 20 g kg<sup>-1</sup> of CaCl<sub>2</sub> for 24 h retained more than 50% of the calcium content of the fruits and when the osmotic treatment was done in the presence of calcium (20 g kg<sup>-1</sup> CaCl<sub>2</sub>), a substantial increase in tomato calcium content was also verified, showing values around seven-fold higher than the raw tomato.

The calcium content of osmo-dehydrated guavas increased with increasing concentration of the calcium salt in the osmotic solution, and calcium chloride provided a calcium uptake of up to twice that obtained using calcium lactate, at the same concentrations (Fig. 1). The calcium content in the fruit varied in the range from  $1.672 \pm 0.008$  to  $4.341 \pm 0.051$  g kg<sup>-1</sup> dry matter for guavas treated with calcium lactate and from  $2.367 \pm 0.047$  to  $7.800 \pm 0.052$  g kg<sup>-1</sup> dry matter for guavas treated with CaCl<sub>2</sub>.

The osmotic dehydration process using sucrose solutions caused a water loss (WL) of 31% and solids



**Figure 1.** Calcium content of guavas subjected to different treatments. FR: without osmotic treatment; SUC: osmotically dehydrated in a 60°Brix sucrose solution; CaLAC 5 to CaLAC 25: osmotically dehydrated in a 60°Brix sucrose solution with addition of 5–25 g kg<sup>-1</sup> calcium lactate; and CaCl<sub>2</sub> 5 to CaCl<sub>2</sub> 25: osmotically dehydrated in a 60°Brix sucrose solution with addition of 5–25 g kg<sup>-1</sup> calcium lactate; and CaCl<sub>2</sub> 5 to CaCl<sub>2</sub> 25: osmotically dehydrated in a 60°Brix sucrose solution with addition of 5–25 g kg<sup>-1</sup> calcium chloride. Mean separation by the Tukey test (*n* = 3). Different letters indicate statistically significant differences at *P* < 0.05.

gain (SG) of around 5% for guavas (Fig. 2). Weight reduction (WR) of the guavas was a combination of the effects of water loss and solids gain (SG), and in this study a WR of around 26% was observed.

A slight statistically significant increase in water loss was verified when calcium salts were added to the osmotic solution, showing WL values of around 34%.



**Figure 2.** Weight reduction (WR), water loss (WL) and solids gain (SG) of guavas subjected to different treatments. (a) 60°Brix osmotic dehydration with or without the addition of calcium lactate; (b) 60°Brix osmotic dehydration with or without the addition of calcium chloride. Mean separation by the Tukey test (n = 3). Different letters indicate statistically significant differences at P < 0.05.

As calcium concentration increased from 5 to 25 g kg<sup>-1</sup> (for both salts), the water content of the samples remained practically constant, as can be seen in Fig. 2. An increasing trend in the content of fruit solids was also observed, due to calcium lactate addition (Fig. 2a), but statistically significant differences are remarkable only for the highest calcium concentration used (25 g kg<sup>-1</sup>), with a solid uptake around 7%. However, this increase in solids did not cause a significant decline in the product weight reduction. The addition of calcium chloride resulted in increased calcium uptake but did not alter the increase in fruit solids, resulting in a weight reduction behavior similar to that observed for water loss (Fig. 2b).

Lewicki *et al.*<sup>15</sup> also verified an improvement in water and sugar diffusion in tomatoes pre-treated with calcium and subjected to a de-watering and drying process. The formation of an open structure due to the bridges formed between pectin molecules by calcium ion linkages, favored mass transport, resulting in greater water loss and solids incorporation in the product.

According to del Valle *et al*.<sup>12</sup> calcium cross-linking of cell walls should result in increased mass transfer resistance to the outflow of water and sugar intake of osmotically dehydrated apples pre-treated with calcium chloride, but this behavior was not observed. The higher water loss and sugar gain verified in the experiments were attributed to membrane damage as a result of osmotic shock.

#### Texture

Osmotic treatment with sucrose solution resulted in an increase in failure stress or hardness of the guavas, which was enhanced by the calcium addition (Fig. 3a). As the calcium concentration in the osmotic solution increased, higher stress at failure values were observed for the guavas treated with calcium lactate. A more intense effect of calcium on fruits hardness was verified for guavas treated with calcium chloride. The stress at failure value of the sample treated with 5 g kg<sup>-1</sup> of CaCl<sub>2</sub> (211.97  $\pm$  32.48 kPa) was similar to the value for the sample treated with  $20 \text{ g kg}^{-1}$  of calcium lactate  $(191.57 \pm 54.32 \text{ kPa})$ , showing no statistically significant difference. Moreover, rupture did not occur for samples treated with CaCl<sub>2</sub> at concentrations higher than 5 g kg<sup>-1</sup> (10, 15, 20 and 25 g kg<sup>-1</sup> of CaCl<sub>2</sub>), making not possible to obtain the mechanical properties at failure.

The effect of osmotic dehydration, with or without calcium addition, on guava strain at failure or elasticity was also noticed, but in a less pronounced way (Fig. 3b). The guavas osmotically dehydrated in sucrose solutions showed greater strain at failure values than the fresh ones, but the effect of calcium lactate addition was only observed at concentrations above  $10 \text{ g kg}^{-1}$ . Samples treated with 5 g kg<sup>-1</sup> of CaCl<sub>2</sub> and  $20 \text{ g kg}^{-1}$  of calcium lactate also showed similar strain at failure values,  $0.591 \pm 0.068$  and  $0.550 \pm 0.058$  respectively, in a similar way to hardness. The effect of



**Figure 3.** Stress (a) and strain (b) at failure of guavas subjected to different treatments. FR: without osmotic treatment; SUC: osmotically dehydrated in a 60°Brix sucrose solution; CaLAC 5 to CaLAC 25: osmotically dehydrated in a 60°Brix sucrose solution with addition of  $5-25 \text{ g kg}^{-1}$  of calcium lactate. Mean separation by the Tukey test (n = 5). Different letters indicate statistically significant differences at P < 0.05.

 $CaCl_2$  concentration on strain at failure values could not be evaluated, once samples treated with this salt did not break at concentrations higher than 5 g kg<sup>-1</sup>.

The firming effect provided by calcium salts was also observed by several researchers<sup>4,6,11,13</sup> and was explained by the linkage of calcium ions with cell wall and middle lamella pectin. The different behavior shown by the calcium salts studied here, at the same concentration, can be attributed to the higher availability of calcium ions able to cross-link with guava pectin in calcium chloride solutions, which can be proved by the greater calcium content of fruits treated with this salt, as shown previously (Fig. 1). CaCl<sub>2</sub> and calcium lactate are salts from strong and weak acids, respectively, with a strong base. In CaCl<sub>2</sub> solutions all the calcium ions are dissociated, but in calcium lactate solutions only some of these ions are available, resulting in a higher amount of free  $Ca^{2+}$ available for pectin linkage using CaCl<sub>2</sub> solutions.

In order to obtain an equivalent amount of calcium in the treatments, resulting in a similar effect of the calcium salts on fruit firmness, Agar *et al.*<sup>27</sup> suggested the use of calcium lactate at double the concentration of CaCl<sub>2</sub>. In this study, despite the fact that the calcium content of the guavas treated with CaCl<sub>2</sub> was, in general, twice the value found in the calcium lactate treated guavas at the same concentration, the effect of the calcium salts on fruit hardness did not directly correspond to this behavior (Fig. 3). The stress at failure value for guavas treated with 5 g kg<sup>-1</sup> of CaCl<sub>2</sub> was considerably greater than the value for guavas treated with 10 g kg<sup>-1</sup> of calcium lactate, corresponding to 211.97  $\pm$  32.48 kPa and 104.75  $\pm$  11.83 kPa, respectively (approximately two-fold).

The relaxation time and residual stress are rheological properties associated with the visco-elastic characteristics of the material. These rheological properties are indirectly related with texture and, consequently, reflect the macroscopic and microscopic features of foods. The definition of relaxation time is not simple but could be thought of as the time it takes a macromolecule to be stretched out when deformed or the ratio between viscous and elastic characteristics.<sup>28</sup> Thus, greater values of relaxation time can be associated with a greater difficulty to deform the material structure. Residual stress is the measure of the elastic part of a visco-elastic material, which means that higher values of this parameter are associated with a more solid or more rigid material.

Osmotic dehydration with sucrose solution caused a decrease in guava relaxation time and addition of calcium lactate to this solution did not change this behavior (Fig. 4). On the other hand, the use of calcium chloride avoided the decrease in the relaxation time of the osmotically dehydrated guavas, showing values statistically equal to those of the fresh fruit. The osmotic process with sucrose solution also caused a decrease in guava residual stress and the addition of calcium salts did not present a meaningful effect on this texture characteristic. These results showed that the addition of both calcium salts to the osmotic solution did not alter the elastic character of the osmodehydrated guavas, while the viscous character of the treated guavas was only influenced by addition of calcium chloride. Besides, for both salts used, an increase in calcium concentration in the sugar solution did not change the texture features of the osmotically dehydrated guavas under small deformations.

Mastrangelo *et al.*<sup>6</sup> also verified a similar effect of osmotic dehydration on the relaxation time and residual stress of melons, but the characteristics of raw melon were more altered than those of guavas. The fruits subjected to atmospheric or vacuum osmotic treatment, with or without calcium lactate, showed residual relaxation force more than eight times lower than the fresh melon and the relaxation time for raw melon was remarkably higher than that for osmotically dehydrated melon, showing high internal fracture suffered by the fruits during the osmotic process.

#### Structural changes

The cellular structure of guava was strongly influenced by the osmotic process and addition of calcium (Fig. 5). Fresh guava tissue showed turgid cells with a consistent cell wall structure. Tonoplast and



**Figure 4.** Relaxation time of guavas subjected to different treatments. FR: without osmotic treatment; SUC: osmotically dehydrated in a 60° Brix sucrose solution; CaLAC 5 to CaLAC 25: osmotically dehydrated in a 60°Brix sucrose solution with addition of 5–25 g kg<sup>-1</sup> of calcium lactate; and CaCl<sub>2</sub> 5 to CaCl<sub>2</sub> 25: osmotically dehydrated in a 60° Brix sucrose solution with addition of 5–25 g kg<sup>-1</sup> of calcium lactate; and CaCl<sub>2</sub> 5 to CaCl<sub>2</sub> 25: osmotically dehydrated in a 60° Brix sucrose solution with addition of 5–25 g kg<sup>-1</sup> of calcium lactate; and CaCl<sub>2</sub> 5 to CaCl<sub>2</sub> 25: osmotically dehydrated in a 60° Brix sucrose solution with addition of 5–25 g kg<sup>-1</sup> of calcium chloride. Mean separation by the Tukey test (*n* = 5). Different letters indicate statistically significant differences at *P* < 0.05.

plasmalemma appeared associated with the cell wall and, in some areas, a well-defined middle lamella between the cells was also observed (dashed arrows) (Fig. 5(a)). Osmotic dehydration in sucrose solutions caused extensive cellular plasmolysis (solid arrows). Cells appeared deformed and collapsed (open arrows) (Fig. 5(b)). The addition of calcium lactate to the osmotic solution promoted the structural preservation of the guavas. Calcium-treated samples showed turgid cells with a thick cell wall and well-defined cellular contour as observed for fresh guava (Fig. 5(c)and (e)). However, calcium lactate concentrations above  $15 \text{ g kg}^{-1}$  seemed to cause some cellular damage, showing slight cell plasmolysis (solid arrows), although the plasmalemma was apparently intact (Fig. 5(g)).

The negative impact of the osmotic process on cellular tissue integrity and the advantageous effect of adding calcium lactate during the osmotic treatment have already been reported in the literature. Structural preservation due to the addition of calcium was verified in different types of fruit, such as melon,<sup>6</sup> kiwifruit<sup>4</sup> and strawberry.<sup>17</sup>

On the other hand, the addition of CaCl<sub>2</sub> only had the same preserving effect on guava structure at 5 g kg<sup>-1</sup> salt concentration (Fig. 5d). Although guavas treated with CaCl<sub>2</sub> showed a strengthening of the cell walls and a more structured cellular arrangement than osmo-dehydrated guavas without a calcium salt, for calcium chloride concentrations above  $5 \text{ g kg}^{-1}$ , severe cellular plasmolysis (solid arrows) and some cell collapse was verified (Fig. 5f and h). This unexpected behavior of CaCl<sub>2</sub> on the cellular structure of guavas could be attributed to the great calcium uptake provided by this salt, presenting a calcium content three-fold to ten-fold higher than fresh guava and almost double the value showed by guavas treated with calcium lactate at the same concentration, resulting in guava tissue damage. Moreover, these structural changes can be associated with the strong effect of this salt on the texture features under high deformations, which led to no rupture of samples at calcium concentrations greater than  $5 \text{ g kg}^{-1}$ .

Despite the severe cellular plasmolysis, caused by cytoplasmic water loss as verified in the presence of high amounts of calcium, especially when  $CaCl_2$  was used, the water content of guavas remained practically constant with increases in salt concentration, as shown in Fig. 2. The rigid and structured cell wall provided by the  $Ca^{2+}$  and pectin linkages seemed to hinder the outflow of water from the cells, resulting in shrinkage of the cytoplasm but with no alteration in the total water content of the fruit.

#### Sensory acceptability

Guavas osmo-dehydrated in sucrose solutions, with or without the addition of calcium lactate, showed good sensory acceptance for all the sensory attributes evaluated (Table 2). Slightly lower scores were only observed at calcium lactate concentrations above 20 g  $kg^{-1}$ , but with average scores above the limit of acceptability (4.5). However, treatment with CaCl<sub>2</sub> only showed good sensory acceptance at  $5 \text{ g kg}^{-1}$ salt concentration, showing scores statistically equal to the sucrose and calcium lactate treated fruits. The fruits treated with calcium chloride presented average scores below or equal to the acceptability limit for all concentrations above  $5 \text{ g kg}^{-1}$  in relation to texture and, for flavor, this was also verified at  $25 \text{ g kg}^{-1}$ salt concentration. For these treatment conditions the scores were also statistically lower.

The poor sensory acceptance of guavas treated with  $CaCl_2$  is probably associated with the strong effect of this salt on guava stress and strain at failure and was also demonstrated in the guava structure, showing cell damage when used in concentrations greater than 5 g kg<sup>-1</sup>. In addition, the good acceptance of

**Table 2.** Sensory acceptance scores of guavas subjected to different treatments, in relation to color, aroma, flavor, texture and overall impression

	Sensory acceptance scores*				
Treatment	Color	Aroma	Flavor	Texture	Overall impression
Sucrose	6.6 <sup>ab</sup>	6.4 <sup>a</sup>	6.4 <sup>a</sup>	6.4 <sup>a</sup>	6.4 <sup>a</sup>
CaLAC 5 g kg <sup>-1</sup>	6.9 <sup>a</sup>	5.6 <sup>a</sup>	5.8 <sup>ab</sup>	5.7 <sup>ab</sup>	5.9 <sup>ab</sup>
CaLAC 10 g kg <sup>-1</sup>	6.5 <sup>ab</sup>	5.7 <sup>a</sup>	5.9 <sup>ab</sup>	6.1 <sup>a</sup>	6.0 <sup>ab</sup>
CaLAC 15 g kg <sup>-1</sup>	6.5 <sup>a</sup>	5.4 <sup>a</sup>	5.3 <sup>bc</sup>	5.8 <sup>ab</sup>	5.8 <sup>ab</sup>
CaLAC 20 g kg <sup>-1</sup>	6.3 <sup>ab</sup>	5.5 <sup>a</sup>	5.2 <sup>bc</sup>	6.0 <sup>a</sup>	5.4 <sup>bc</sup>
CaLAC 25 g kg <sup>-1</sup>	5.5 <sup>b</sup>	5.5 <sup>a</sup>	4.6 <sup>c</sup>	4.9 <sup>bc</sup>	5.1 <sup>bc</sup>
CaCl <sub>2</sub> 5 g kg <sup>-1</sup>	6.3 <sup>ab</sup>	6.0 <sup>a</sup>	5.7 <sup>ab</sup>	5.7 <sup>ab</sup>	5.7 <sup>ab</sup>
CaCl <sub>2</sub> 10 g kg <sup>-1</sup>	6.1 <sup>ab</sup>	5.4 <sup>a</sup>	5.1 <sup>bc</sup>	4.5 <sup>c</sup>	4.9 <sup>bc</sup>
CaCl <sub>2</sub> 15 g kg <sup>-1</sup>	6.5 <sup>ab</sup>	6.2 <sup>a</sup>	5.3 <sup>bc</sup>	4.3 <sup>c</sup>	5.3 <sup>bc</sup>
$CaCl_2 20 \text{ g kg}^{-1}$	6.4 <sup>ab</sup>	5.7 <sup>a</sup>	5.4 <sup>bc</sup>	4.5 <sup>c</sup>	5.3 <sup>bc</sup>
CaCl <sub>2</sub> 25 g kg <sup>-1</sup>	6.4 <sup>ab</sup>	5.6 <sup>a</sup>	4.3 <sup>c</sup>	4.1 <sup>c</sup>	4.7 <sup>c</sup>

\*The values represent the mean of the acceptance scores by 30 panelists, using a 9 cm unstructured hedonic scale.

Mean separation by the Tukey test. Means with the same letter in a column did not differ significantly at P < 0.05.



**Figure 5.** Micrographs of guavas subjected to different treatments. (a) Fresh, (b) osmotically dehydrated in a 60°Brix sucrose solution, (c), (e) and (g) osmotically dehydrated in a 60°Brix sucrose solution with 5, 15 and 25 g kg<sup>-1</sup> of calcium lactate; and (d), (f) and (h) osmotically dehydrated in a 60°Brix sucrose solution with 5, 15 and 25 g kg<sup>-1</sup> of calcium lactate; and (d), (f) and (h) osmotically dehydrated in a 60°Brix sucrose solution with 5, 15 and 25 g kg<sup>-1</sup> of calcium lactate; and (d), (f) and (h) osmotically dehydrated in a 60°Brix sucrose solution with 5, 15 and 25 g kg<sup>-1</sup> of calcium chloride. Scale bar: 70  $\mu$ m.

osmo-dehydrated guavas with the addition of calcium lactate up to  $20 \text{ g kg}^{-1}$  and calcium chloride at  $5 \text{ g kg}^{-1}$  is in agreement with the results for texture. It is interesting to note that both these limits of sensory

texture acceptability corresponded to the same results of instrumental texture. However, the evaluation of guava structure showed that at concentrations of calcium lactate higher than  $15 \text{ g kg}^{-1}$  there was already

some cell damage, suggesting that such analysis was capable of detecting some structural changes at lower salt concentrations than the instrumental texture measurements and the sensory evaluation.

## CONCLUSIONS

The addition of calcium salts to the sucrose solutions used for the osmotic dehydration of guavas had a strong influence on the texture and structure of processed fruits, resulting in maintenance of the tissue structure when calcium lactate was employed at concentrations of up to  $15 \text{ g kg}^{-1}$  and calcium chloride at  $5 \text{ g kg}^{-1}$ .

The use of calcium salts also influenced the mass transfer in the osmotic process without remarkable differences between the two salts. However, calcium chloride provided a higher calcium uptake, resulting in a fruit calcium content up to twice that of calcium lactate when used at the same concentration, but this behavior was apparently not advantageous to product quality, according to the sensory analysis.

Guavas treated with calcium lactate presented good acceptance regarding all the sensory attributes up to  $20 \text{ g kg}^{-1}$  salt concentration but CaCl<sub>2</sub> treated guavas were acceptable only at 5 g kg<sup>-1</sup> of the salt.

The use of calcium salts did not improve sensory testing scores of osmotically dehydrated guavas under any of the circumstances studied, although adding the salt to the osmotic solution might be important in enhancing storage stability and product characteristics after storage times. Then, shelf-life studies should be done in future research.

## ACKNOWLEDGEMENT

The authors thank FAPESP, CNPq and CAPES/ GRICES for their financial support. LMP was a recipient of a FAPESP fellowship.

## REFERENCES

- Alzamora SM, Tapia MS, Argaíz A and Welti J, Application of combined methods technology in minimally processed fruits. *Food Res Int* 26:125–130 (1993).
- 2 Alzamora SM, Gerschenson LN, Vidales SL and Nieto A, Structural changes in the minimal processing of fruits: some effects of blanching and sugar impregnation, in *Food Engineering 2000*, ed. by Fito P, Ortega-Rodriguez E and Barbosa-Canovas G. Chapman and Hall, New York, pp. 117–139 (1997).
- 3 Pereira LM, Rodrigues ACC, Sarantópoulos CIGL, Junqueira VCA, Cunha RL and Hubinger MD, Influence of modified atmosphere packaging and osmotic dehydration on the quality maintenance of minimally processed guavas. *J Food Sci* 69:172–177 (2004).
- 4 Muntada V, Gerschenson LN, Alzamora SM and Castro MA, Solute infusion effects on texture of minimally processed kiwifruit. *J Food Sci* 63:616–620 (1998).
- 5 Sormani A, Maffi D, Bertolo G and Torreggiani D, Textural and structural changes of dehydrofreeze-thawed strawberry slices: effects of different dehydration pretreatments. *Food Sci Technol Int* 5:479–485 (1999).

- 6 Mastrángelo MM, Rojas AM, Castro MA, Gerschenson LN and Alzamora SM, Texture and structure of glucose-infused melon. *J Sci Food Agric* 80:769–776 (2000).
- 7 Nieto AB, Salvatori DM, Castro MA and Alzamora SM, Structural changes in apple tissue during glucose and sucrose osmotic dehydration: shrinkage, porosity, density and microscopic features. *J Food Eng* 61:269–278 (2004).
- 8 Quiles A, Hernando I, Pérez-Munuera I, Llorca E, Larrea V and Lluch MA, The effect of calcium and cellular permeabilization on the structure of the parenchyma of osmotic dehydrated 'Granny Smith' apple. J Sci Food Agric 84:1765–1770 (2004).
- 9 Lewicki PP and Porzecka-Pawlak R, Effect of osmotic dewatering on apple tissue structure. J Food Eng 66:43-50 (2005).
- 10 Lara I and Vendrell M, ACC oxidase activation by cold storage on 'Passe Crassane' pears: effect of calcium treatment. J Sci Food Agric 76:421–426 (1998).
- 11 Monsalve-González A, Barbosa-Cánovas GV and Cavalieri RP, Mass transfer and textural changes during processing of apples by combined methods. *J Food Sci* 58:1118–1124 (1993).
- 12 del Valle JM, Aranguiz V and Leon H, Effects of blanching and calcium infiltration on PPO activity, texture, microstructure and kinetics of osmotic dehydration of apple tissue. *Food Res Int* 31:557–569 (1998).
- 13 Luna-Guzmán I, Cantwell M and Barret DM, Fresh-cut cantaloupe: effects of CaCl<sub>2</sub> dips and heat treatments on firmness and metabolic activity. *Postharvest Biol Technol* **17**:201–213 (1999).
- 14 Jackman RL and Stanley DW, Perspectives in the textural evaluation of plant foods. *Trends Food Sci Technol* **6**:187–194 (1995).
- 15 Lewicki PP, Vu Le H and Pomaranska-Lazuka W, Effect of pre-treatment on convective drying of tomatoes. J Food Eng 54:141-146 (2002).
- 16 Luna-Guzmán I and Barret DM, Comparison of calcium chloride and calcium lactate effectiveness in maintaining shelf stability and quality of fresh-cut cantaloupes. *Postharvest Biol Technol* 19:61–72 (2000).
- 17 Vidales SL, Castro MA and Alzamora SM, The structure-texture relationship of blanched and glucoseimpregnated strawberries. *Food Sci Technol Int* **4**:169–178 (1998).
- 18 Ranganna S, Manual of Analysis of Fruit and Vegetables Products. Central Food Technological Research Institute, Mysore, India (1977).
- 19 Bligh EG and Dyer WJ, A rapid method of total lipid extraction and purification. Can J Biochem Physiol 37:911-917 (1959).
- 20 Argandoña EJS. Guavas dehydration by immersion and drying. M.Sc dissertation. Faculty of Food Engineering, State University of Campinas, Campinas, SP (1999). (in Portuguese).
- 21 Association of Official Analytical Chemists, *Official Methods* of *Analysis*, 17th edition. AOAC, Gaithersburg, Maryland (2002).
- 22 Costell E, Fiszman SM and Durán L, Physical properties I – Rheology of solids and texture, in *Themes in Food Technology*, ed. by Aguilera JM. National Polytechnic Institute, Mexico, pp. 215–260 (1997). (in Spanish).
- 23 Peleg M, Linearization of relaxation and creep curves of solid biological materials. J Rheol 24:451–463 (1980).
- 24 Lewicki PP and Lukaszuk A, Effect of osmotic dewatering on rheological properties of apple subjected to convective drying. *J Food Eng* 45:119–126 (2000).
- 25 O'Brien TP, Feder N and McCully ME, Polychromatic staining of plant cell walls by toluidine blue. *Protoplasma* 59:368–373 (1964).
- 26 Meilgaard M, Civille GV and Carr BT, Sensory Evaluation Techniques, 2nd edition. CRC Press Inc., Florida (1988).
- 27 Agar IT, Massantini R, Hess-Pierce B and Kader AA, Postharvest CO<sub>2</sub> and ethylene production and quality maintenance of fresh-cut kiwifruit slices. *J Food Sci* 64:433–440 (1999).
- 28 Steffe JF, *Rheological Methods in Food Process Engineering*, 2nd edition. Freeman Press, Michigan (1996).