The impact of the use of vacuum and calcium lactate on the osmotic dehydration of papaya with isomaltulose solution

O impacto do uso de vácuo e do lactato de cálcio na desidratação osmótica de mamão com solução de isomaltulose

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ABSTRACT
Papayas are one of the most consumed fruits in the world. Enriching papayas with isomaltulose and calcium through osmotic dehydration (OD) is a noteworthy strategy for providing these substances to consumers. The present study evaluated the influence of the application of vacuum and the addition of calcium lactate in an osmotic solution of isomaltulose (35%) on the OD of papaya. Papaya cubes with an edge length of 10 mm were immersed in osmotic solutions, with or without calcium lactate, for a total time of 300 min, with or without application of vacuum during the first 20 min, totaling four treatments. The osmotic processes reduced the moisture content (MC) and water activity and caused a change in the color of the papaya. The

application of vacuum caused greater shrinkage and color change. Calcium lactate impregnation increased the water loss (WL), weight reduction (WR), hardness, and chroma of the papaya and reduced the solid gain (SG), MC and shrinkage. Therefore, OD proved to be an appealing alternative for the production of papaya predehydrated and enriched with isomaltulose and calcium.

Keywords: pulsed vacuum osmotic dehydration, palatinose, mass exchange, shrinkage, hardness.

RESUMO
O mamão é uma das frutas mais consumidas no mundo. Enriquecer o mamão com isomaltulose e cálcio por meio da desidratação osmótica (OD) é uma estratégia de destaque para o fornecimento dessas substâncias aos consumidores. O presente estudo avaliou a influência da aplicação de vácuo e da adição de lactato de cálcio em solução osmótica de isomaltulose (35%) na OD de mamão. Cubos de mamão com borda de 10 mm foram imersos em soluções osmóticas, com ou sem lactato de cálcio, por um tempo total de 300 min, com ou sem aplicação de vácuo durante os primeiros 20 min, totalizando quatro tratamentos. Os processos osmóticos reduziram o teor de umidade (MC) e a atividade de água e causaram alteração na cor do mamão. A aplicação de vácuo causou maior encolhimento e mudança de cor. A impregnação com lactato de cálcio aumentou a perda de água (WL), redução de peso (WR), dureza e croma do mamão e reduziu o ganho de sólidos (SG), MC e encolhimento. Portanto, OD mostrou-se uma alternativa atraente para a produção de mamão pré-desidratado e enriquecido com isomaltulose e cálcio.

Palavras-chave: desidratação osmótica por vácuo pulsado, palatinose, troca de massa, encolhimento, dureza.

1 INTRODUCTION

The development of different foods from healthy ingredients is necessary due to greater consumer interest in health and well-being. Thus, interest in isomaltulose, also known as palatinose, a carbohydrate with a low glycemic and cariogenic index because the bond between its two monosaccharides (glucose and fructose) is at α-1,6, unlike sucrose, which is linked at α-1,2, is increasing. The α-1,6 bond is more difficult to hydrolyze by gastrointestinal enzymes and cannot be broken by most bacteria in the mouth. The low glycemic and insulin index promotes a prolonged feeling of satiety and release of energy. These characteristics are desirable for good physical and mental performance and for diabetic patients. The low cariogenic index of isomaltulose, in turn, preserves the health of the teeth by not promoting cavities [1–3].

Isomaltulose occurs naturally in honey and sugarcane juice but can also be produced through an enzymatic process from sucrose [2]. The addition of this carbohydrate to fruits is an interesting strategy for enriching them [4, 5], combining the beneficial properties of isomaltulose with the natural properties of the fruit. Fruits are an important natural source of nutrients in the human diet [6]. Papaya (*Carica papaya* L.) is one of the most consumed fruits and stands out because of its composition of vitamins, minerals, fibers and antioxidants, low caloric value, and excellent sensory acceptance [7, 8].

Osmotic dehydration (OD) is a unitary operation that promotes the impregnation of carbohydrates in fruits. This process is carried out by immersing fruit in a hypertonic solution, resulting in the loss of water from the food to the solution and the gain of solids in the food from the solution [6, 9–12]. Some procedures can be used to improve the OD process. The application of vacuum in the first minutes of the osmotic process is called pulsed vacuum osmotic dehydration (PVOD), and this method has been used to maximize mass exchange flows. The reduction of the pressure followed by the resumption of atmospheric pressure results in the replacement of the occluded gas with the osmotic solution, increasing the contact area with the osmotic solution [13–15].

Some physical changes in food tissue may occur in fruits subjected to OD, such as the softening of the surface layers, detachment of one cell from another, and breakdown of cells, which can reduce the shelf life. Calcium compounds can be added to the osmotic solution because calcium can increase cell stiffness, decreasing the fragility of the material [9, 16]. In addition, calcium is an important mineral for growth, maintenance, and reproduction in the human body. However, currently, the calcium intake of many populations is below the recommended value. According to Palacios et al. (2021), nutritional rickets in children and osteomalacia in adults and an increased risk of osteoporosis are the result of very low calcium intake. The fortification of foods and beverages represents a good alternative for improving calcium intake, as long as simple, safe, and low-cost technologies are used [17, 18].

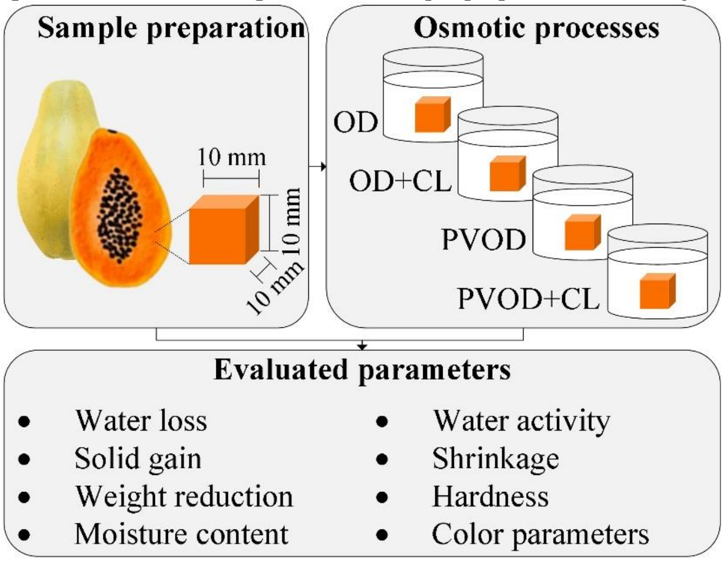
This study aimed to evaluate the influence of vacuum pulsing and the addition of calcium lactate on mass changes during papaya OD using isomaltulose osmotic solution and moisture content (MC), water activity, shrinkage, hardness and color parameters.

2 MATERIALS AND METHODS

2.1 SAMPLE PREPARATION

Fresh papaya fruits (*Carica papaya* L.) were selected, washed in running water, sanitized with chlorinated water (200 ppm for 10 min), rinsed, and drained on absorbent paper. The fruit peel was removed, and the pulp was cut into cubes (10 × 10 × 10 mm), as shown in Figure 1.

Figure 1 - Illustrative diagram of the sample preparation and analyses.



2.2 OSMOTIC DEHYDRATION (OD) AND PULSED VACUUM OSMOTIC DEHYDRATION (PVOD)

Isomaltulose solutions (Beneo-Palatinit, Mannheim, Germany) were prepared at a concentration of 35% (ww^{-1}), dissolving the solute in deionized water [4]. Calcium lactate pentahydrate was added to the osmotic solution at 0% and 2% (ww^{-1}) [16].

Papaya cubes were immersed in osmotic solutions at a ratio of 1:20 (ww^{-1}) for a total of 300 min and 25 °C in a controlled temperature oven (Solab SL104/40, Piracicaba, Brazil) [4]. Vacuum (160 mbar) was applied in the first 20 min in the PVOD process. Then, the atmospheric pressure was restored. OD was performed at atmospheric pressure throughout the process [19].

After the OD and PVOD processes, the samples were removed from the solution and immersed in a cold distilled water bath for 10 s to interrupt the mass flow and to remove the soluble solids and the solution from the food surface [19]. The sample surfaces were drained on absorbent paper to remove excess water from the sample surface [20].

2.3 PARAMETERS EVALUATED

The samples were evaluated in terms of water loss (WL), solid gain (SG), weight reduction (WR), MC, water activity, shrinkage, hardness, and color parameters.

2.3.1 Mass exchange parameters

The WL, SG, and WR were calculated according to Equations 1, 2, and 3, respectively.

$$WL(\%) = \frac{W_0 M_0 - W_f M_f}{W_0} \times 100 \quad (1)$$

$$SG(\%) = \frac{W_f(1-M_f) - W_0(1-M_0)}{W_0} \times 100 \quad (2)$$

$$WR(\%) = \frac{W_0 - W_f}{W_0} \times 100 \quad (3)$$

where W is the sample weight (kg); M is the moisture content of the sample (kg water kg sample^{-1}); and the subindices "0" and "f" indicate the initial and final samples, respectively.

2.3.2 Moisture content (MC)

The MC was determined by the gravimetric method, in which the samples were placed in an oven at 70 °C under vacuum according to the AOAC 934.06 method [21].

2.3.3 Water activity (a_w)

The a_w of the samples was determined using an electronic hygrometer (Aqualab, series 3TE, Washington, USA) at 25 °C.

2.3.4 Shrinkage

The edges of each axis (x, y, and z) of the fresh and osmotically dehydrated samples were measured. The determination was performed using a Vernier digital caliper (± 0.01 mm) (Western, DC-60 model, Zheiang, China). The volume of the samples was calculated by the product of the measurement of each axis (x, y, and z). The shrinkage of the samples was calculated according to Equation 4.

$$Shrinkage = \left(1 - \frac{V}{V_0}\right) \times 100 \quad (4)$$

where V and V_0 are the volumes (m^3) after and before the osmotic process, respectively.

The coefficient of variation was calculated by the ratio between the standard deviation and the mean of the three measurements of each sample to determine how isotropic the shrinkage of the samples of each treatment was. The result was expressed in %.

2.3.5 Hardness

The hardness of the samples was determined with the aid of a texture analyzer (Stable Micro Systems, TA-X2T, Surrey, England). The equipment included a 50-kg load cell, a 6-mm diameter probe, a test speed of 2 mm s^{-1}, and a penetration distance of 3 mm. The response was expressed in newtons (N) [4].

2.3.6 Color

A digital colorimeter (Konica Minolta, model CR-10, Osaka, Japan) was used to obtain the colorimetric parameters (L^*, C^* and °h) of the samples with the D65 illuminant using the CIELab color scale. The L^* indicates lightness, ranging from 0 (black) to 100 (white). The C^* indicates the chroma. The °h indicates the hue of the sample, where the angles of 0° or 360°, 90°, 180° and 270° represent red, yellow, green and blue, respectively. The total color difference (ΔE) indicates the difference between the color of each osmo-dehydrated sample and that of the fresh sample, where the parameters of the fresh sample are represented by the subindex "0". The ΔE was calculated according to Equation 5 [22].

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2} \quad (5)$$

2.4 STATISTICAL ANALYSIS

The experiment was conducted with a completely randomized design in a factorial scheme (2 × 2) (Table 1) with five replicates. The data were subjected to analysis of variance, followed by Tukey's test. The Dunnett test was used to compare the control sample (fresh papaya) with each treatment individually.

Table 1 - Experimental conditions

Treatment	Osmotic process	Calcium lactate
Fresh	No	No
OD	OD	No
PVOD	PVOD	No
OD+CL	OD	Yes
PVOD+CL	PVOD	Yes

A multivariate analysis was conducted using principal component analysis (PCA) on the WL, SG, WR, MC, water activity, shrinkage, hardness, and color parameter responses to analyze the influence of the application of vacuum and the addition of calcium lactate on their distinctive interrelationships.

Statistical analyses were performed at the 5% error probability level using the software Statistica (StatSoft Inc., Tulsa, OK, USA).

3 RESULTS AND DISCUSSION

3.1 MASS EXCHANGE

The responses of WL, SG, and WR after OD and PVOD are shown in Table 2.

Table 2 - Water loss (WL), solid gain (SG), and weight reduction (WR) of the osmotically dehydrated papayas

Response	Osmotic process	Calcium lactate	
		No	Yes
WL (%)	OD	12.91±0.48 Ab	23.11±0.92 Ba
	PVOD	14.42±0.82 Ab	28.06±0.98 Aa
SG (%)	OD	10.51±0.19 Aa	8.32±0.82 Ab
	PVOD	11.10±0.48 Aa	8.12±0.43 Ab
WR (%)	OD	2.40±0.63 Ab	14.79±1.44 Ba
	PVOD	3.32±0.79 Ab	19.95±1.25 Aa

Means followed by the same uppercase letter vertically and the same lowercase letter horizontally are not significantly different according to Tukey's test ($p<0.05$).

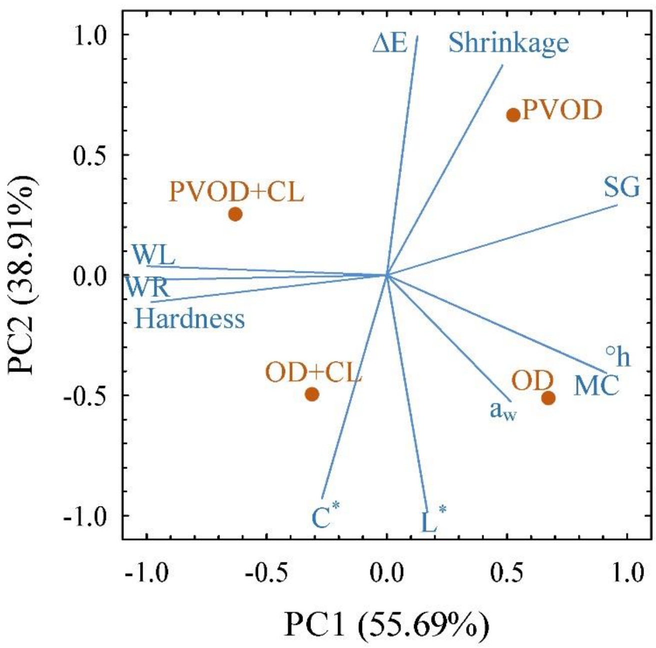
The WL is the mass flow that characterizes OD and PVOD as proper dehydration processes. This phenomenon occurs through several mechanisms but mainly in a passive manner by diffusion [16]. The osmotic processes caused WL from 12.91% to 28.06% in papaya (Table 2). Similar values were observed in other studies [19, 23–27]. The low water solubility of isomaltulose precludes the development of more concentrated osmotic solutions, where 40% and 60% is the most commonly used concentration range [6, 9]. Thus, the osmotic pressure gradient between the solution and the material is not very large, which tends to result in low mass exchange values.

According to Table 2, the addition of calcium lactate to the osmotic solution increased the WL in both OD and PVOD. The impregnation of solutes with physiological functions, such as calcium, can affect mass exchange due to its influence on the cellular structure of the fruit [16]. The degree of influence on and how calcium influences the material depend on several factors, such as the fruit type, size, and shape, amount of calcium added to the osmotic solution, concentration and type of solute, and total time of the osmotic process.

The application of vacuum did not significantly influence ($p > 0.05$) the WL when using osmotic solution without calcium lactate. On the other hand, in the presence of calcium lactate, the application of vacuum increased the WL. The vacuum applied during the first minutes removes the occluded gas in the material. With the resumption of atmospheric pressure, the osmotic solution fills this space. This increases the contact between the food matrix and the osmotic solution, tending to increase the mass exchange between both systems [6].

Figure 2 shows the biplot of the two principal components. The first (PC1) and the second (PC2) axes were responsible for 55.69% and 38.91%, respectively, totaling 94.60% of the explained variance. According to Figure 2, high WL values were associated with the treatment in which vacuum and calcium lactate were used.

Figure 2 - Principal component analysis biplot of the water loss (WL), solid gain (SG), weight reduction (WR), moisture content (MC), water activity (a_w), shrinkage, hardness, and color parameters of osmo-dehydrated papayas.



OD: osmotic dehydration; PVOD: pulsed vacuum osmotic dehydration; OD+CL: osmotic dehydration with calcium lactate; PVOD+CL: pulsed vacuum osmotic dehydration with calcium lactate.

The SG values ranged from 8.12% to 11.10% (Table 2). Similar values were observed in other studies [27]. The application of vacuum did not significantly influence the SG. On the other hand, the addition of calcium lactate to the osmotic solution reduced the SG. The same was reported in other studies [28, 29]. According to Mavroudis et al. (2012), the reduction in solute impregnation due to the addition of calcium may be related to the reduction in cell wall porosity. Thus, SG was more associated with processes in which calcium lactate was absent (Figure 2).

Based on the SG results obtained, OD and PVOD were noteworthy processes in terms of promoting the enrichment of papaya with isomaltulose, conferring the beneficial properties of this carbohydrate to the fruit. This makes the product nutritionally richer and, consequently, increases its added value. In parallel, calcium impregnation makes papaya another source of this important mineral for consumer health. In addition to the health effects, calcium decreases the metabolic activity of the tissue, contributing to the reduction in the respiration rate due to increased membrane rigidity, blocking the exchange of gases and, consequently, prolonging the shelf life of the product [31].

WR occurs because the WL is greater than the SG because water molecules are smaller than the solute molecules, passing through the membrane more easily [28]. The WR values obtained ranged from 2.40% to 19.95%. As with the WL, the application of vacuum did not influence the WR of samples subjected to osmotic processes without calcium lactate but only those in which calcium lactate was added to the osmotic solution (Table 2). In addition, the use of calcium lactate increased the WR for both osmotic processes, as occurred for WL. Similar behavior between WR and WL can be observed in Figure 2.

3.2 MOISTURE CONTENT (MC)

Fresh papaya has a high MC value, as shown in Table 3 and reported in other studies [7, 26, 32], indicating that water is a major compound in the composition of papaya, as well as for most fruits.

Table 3 – Moisture content (MC), water activity (a_w), shrinkage, and hardness of fresh and osmotically dehydrated papayas

Response	Osmotic process	Calcium lactate		Fresh
		No	Yes	
MC (%)	OD	74.12±0.10 Aa⁺	72.93±0.66 Ab⁺	85.25±0.72
	PVOD	73.26±0.49 Aa⁺	71.43±0.36 Bb⁺	
a_w	OD	0.968±0.002 Aa⁺	0.963±0.003 Aa⁺	0.976±0.004
	PVOD	0.963±0.004 Aa⁺	0.964±0.002 Aa⁺	
Shrinkage (%)	OD	14.66±4.85 Ba	11.68±1.80 Bb	-
	PVOD	19.93±3.57 Aa	15.28±4.77 Ab	
Hardness (N)	OD	0.419±0.030 Ab	0.789±0.070 Ba⁺	0.486±0.084
	PVOD	0.352±0.028 Ab	0.992±0.105 Aa⁺	

Means followed by the same uppercase letter vertically and the same lowercase letter horizontally are not significantly different according to Tukey's test (p<0.05). A plus sign indicates a significant difference between the treatment and the control sample (fresh) by the Dunnett test (p<0.05).

Both OD and the PVOD resulted in a significant reduction in the MC of the papaya (Table 3). These reductions were due to the WL and SG that occurred during the osmotic processes due to the osmotic pressure gradient between the fruit and the hypertonic solution [6].

The application of vacuum resulted in a significant reduction (p <0.05) in the MC only when calcium lactate was added (Table 3). In addition, the use of calcium lactate reduced the MC in both osmotic processes. The reduction in MC was associated with the mass flows that occurred during OD and PVOD, especially the water flow, as it was the most significant. Thus, there was a tendency for the samples that had lower MC to have higher WL. This association can be observed in Figure 2.

OD and PVOD partially reduced the MC and are commonly used as pretreatments for drying to decrease the volume of water to be removed in the next process. This may result in a shorter drying time and lower electricity consumption. The main economic problem related to OD is that in many cases, the osmotic solution is discarded after the process. However, the reuse of this solution is an alternative that can solve this problem, for example, by using it again for a new OD process or for the elaboration of other products since the osmotic solution is rich in sugars and in leached compounds of fruit [9].

3.3 WATER ACTIVITY (A_W)

Fresh papaya has a high a_w value (Table 3), which makes the fruit highly perishable since microorganisms and enzymes need water to be active [33]. OD and PVOD significantly reduced (p <0.05) the a_w of papaya. However, there were no significant differences (p > 0.05) regarding the use of vacuum and calcium lactate (Table 3).

The a_w is one of the main parameters in food preservation because it is related to the growth rates of microorganisms and chemical and enzymatic reactions that promote food spoilage. According to Jay et al. (2005), an a_w less than 0.6 is recommended for safe storage because, in this condition, the activities of food-spoiling and intoxicating microorganisms are inhibited. Although it is not able to sufficiently reduce the a_w, OD/PVOD reduces the respiration rate of fruits, contributing to the preservation of the product.

Although OD and PVOD reduce the a_w of fruits, this reduction is not sufficient to inhibit microbial growth and minimize enzymatic reactions. Thus, osmo-dehydrated foods require a complementary preservation method, such as drying, refrigeration or pasteurization, to increase product stability. Of these methods, drying with heated air is the most commonly used to complete the reduction in a_w of the material to a safe level [4, 9].

3.4 SHRINKAGE

Both osmotic processes, using calcium lactate or not, caused shrinkage of the samples (Table 3). Volumetric shrinkage is one of the main physical changes that occur in food during OD/PVOD. This phenomenon occurs due to the pressure imbalance created between the inside and the outside of a food when the water leaves, causing a series of changes in the cellular structure of the food matrix [20, 34].

The application of vacuum increased papaya shrinkage, as shown in Table 3 and Figure 2. The same was observed by de Jesus Junqueira et al. (2018) when subjecting beet and eggplant to the OD and PVOD processes. On the other hand, the addition of calcium lactate decreased papaya shrinkage. Calcium preserves the cell wall structure [28], restricting the volumetric changes of the sample. According to Barragán-Iglesias et al. (2019), the osmotic process combined with the addition of calcium forms a firm external structure and a soft internal structure, reducing shrinkage and deformation.

The shrinkage of the samples occurred in an isotropic manner, i.e., the reduction in the volume of the material occurred similarly in the three axes (x, y, and z) of the cube. This is confirmed by the low coefficient of variation values between the three measurements of each sample, where the values for the samples subjected to OD, PVOD, OD+CL, and PVOD+CL were 2.98 ± 0.85%, 2.39 ± 0.31%, 2.59 ± 0.40% and 2.46 ± 0.49%, respectively.

3.5 HARDNESS

The hardness of the fresh and osmo-dehydrated samples is shown in Table 3. The fresh papaya had a low hardness value, confirming its physical fragility, which may limit the shelf life of the fruit [32]. Therefore, hardness is a very important quality parameter in fresh and processed fruits [16].

OD and PVOD, without calcium lactate, did not significantly affect hardness, preserving it in relation to that of the fresh fruit (Table 3). On the other hand, the use of the osmotic solution with isomaltulose and calcium lactate resulted in higher hardness in osmo-dehydrated papayas (Table 3). Figure 2 shows the association of hardness with the processes in which calcium lactate was used. This beneficial effect of calcium has also been reported in other studies [26].

3.6 COLOR

Color is the first quality attribute evaluated by consumers. The color of the fruit is related to the degree of ripeness and freshness. By visual evaluation of the color, the consumer can define their choices and preferences, deciding whether to buy and consume the fruit [4, 7, 35]. In papaya, the orange color is attributed to carotenoids synthesized throughout fruit ripening [7].

The colorimetric parameters of fresh and osmotically dehydrated papaya are shown in Table 4.

Table 4 - Colorimetric parameters of fresh and osmotically dehydrated papayas

Response	Osmotic process	Calcium lactate		Fresh
		No	Yes	
L^*	OD	37.17±0.32 Aa⁺	35.75±0.40 Ab	35.64±0.41
	PVOD	28.50±0.24 Bb⁺	30.22±0.49 Ba⁺	
C^*	OD	41.16±0.72 Ab⁺	46.11±0.65 Aa⁺	43.78±0.31
	PVOD	32.67±0.06 Bb⁺	37.08±0.28 Ba⁺	
h°	OD	49.06±0.06 Aa	47.26±0.38 Aa	47.32±1.23
	PVOD	47.76±1.89 Aa	45.98±0.84 Aa	
ΔE	OD	3.31±0.71 Ba	2.38±0.63 Ba	-
	PVOD	13.25±0.07 Aa	8.68±0.45 Ab	

Means followed by the same uppercase letter vertically and the same lowercase letter horizontally are not significantly different according to Tukey's test (p<0.05). A plus sign indicates a significant difference between the treatment and the control sample (fresh), by Dunnett test (p<0.05).

With the exception of treatment OD+CL, the evaluated treatments influenced L^* and reduced C^* compared to those of fresh papaya. The application of vacuum resulted in a reduction in the L^* and C^* values. Calcium impregnation in the sample increased C^*, which is a beneficial effect because higher C^* values indicate a purer and more intense color [35].

The °h parameter indicates the hue of the material. According to Table 4, the values of °h were in the range corresponding to orange, characteristic of papaya pulp. Significant differences between fresh and osmo-dehydrated fruits were not observed, and there was no significant influence from the application of vacuum and the addition of calcium lactate regarding °h.

ΔE is a good tool for expressing how much color is influenced by each treatment in relation to the control sample (fresh fruit) [4, 35]. The mean values of ΔE obtained were between 2.38 and 13.25. According to Pathare et al. (2013), a noticeable difference in color can be classified as a small difference (ΔE <1.5), distinct (1.5 <ΔE <3), or very distinct (ΔE> 3). Thus, the color of the osmo-dehydrated fruits was distinct or very distinct from the color of the fresh papaya. This is a reflection of the water outflow, impregnation of solids, and leaching of compounds that occurred during the osmotic processes and the physical changes caused, such as shrinkage.

The application of vacuum was the main factor that influenced ΔE, resulting in an increased response. According to Figure 2, it was possible to observe similarity between the responses of ΔE and shrinkage associated with the treatments in which vacuum was used. This suggests that shrinkage exerts a strong influence on color due to changes in the structure of the material [36].

Given all the responses evaluated, Figure 2 shows that the PCA provided a clear distinction between the four treatments, in which each one occupied a quadrant. On the left side of the axis of principal component 1 (PC1), the treatments in which calcium lactate was used were found, while on the right were those that did not use calcium lactate. In addition, the OD processes were found at the bottom of the axis of principal component 2 (PC2), while the PVOD processes were located at the top.

4 CONCLUSION

The impregnation of calcium and isomaltulose can be performed through osmotic dehydration, enriching papaya with an important mineral and a carbohydrate with a low glycemic and cariogenic index. The osmotic processes partially dehydrated the papaya, reducing the moisture content of the fruit due to water loss and solid gain.

The application of vacuum decreased the lightness and chroma and increased the shrinkage and the total color difference between the osmo-dehydrated samples and the fresh papaya. Calcium impregnation increased the water loss, weight reduction, hardness and chroma of the samples and reduced the solid gain, moisture content and shrinkage. Water activity was reduced with osmotic dehydration but was not influenced by the use of vacuum and calcium lactate. The hue of the samples was not altered by the osmotic processes.

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