

## DETERMINATION OF PHYSICO-CHEMICAL PROPERTIES OF OSMO-DEHYDROFROZEN PINEAPPLES

Lee-Fong Siow & Kar-Hing Lee

Monash University Sunway Campus,  
Jalan Lagoon Selatan, 46150 Bandar Sunway, Selangor, Malaysia

**ABSTRACT.** *The effect of osmotic pretreatment using 50 % (w/v) sucrose, trehalose or maltitol syrups on the physicochemical properties of frozen pineapple pieces (Josapine variety) of ~6.7 cm in external diameter (without core) and ~0.9 cm in thickness was investigated. Maltitol-treated pineapple pieces had a higher water loss than those treated with sucrose and trehalose ( $p \leq 0.05$ ). All three syrups resulted in similar solid gain. Prior to freezing, pineapple pieces were subjected to osmotic pretreatment for 1 hour. This was followed by cooling the pineapple pieces at either slow (0.25°C/min) or fast (6.13°C/min) cooling rates to -20°C with an untreated sample used as a control. Physicochemical properties including drip loss, firmness, pH, titratable acidity, total soluble solids and colour of samples stored at -20°C for 0, 3, 10, 14, 17 and 24 days were determined. Untreated pineapple pieces exhibited a smaller change in firmness at fast cooling rate compared to slow cooling rate. At fast cooling rate, a higher drip loss was observed for thawed pineapple pieces compared to slow cooling rate. In the presence of maltitol and trehalose, a small change in firmness and higher drip loss was found at slow cooling compared to fast cooling. In the presence of sucrose, there was no significant change in drip loss at both slow and fast cooling rates. Fast cooling in the presence of sucrose, maltitol or trehalose had no effect on firmness of osmo-dehydrofrozen pineapple pieces. Frozen storage of 24 days did not affect the physicochemical properties of osmo-dehydrofrozen pineapples.*

**KEYWORDS.** *Ananas comosus*, cooling rate, maltitol, sucrose, trehalose

### INTRODUCTION

Pineapple is one of the tropical fruits commonly available in Malaysia. It is sold as a whole fruit or freshly cut. Pineapple is juicy and yellow, with an outer skin resembling pine cones. Pineapple is a good source of the proteolytic enzyme, bromelain, which breaks down protein. Surplus of the fruits is canned or juiced, both of which affect the nutritional properties, flavour and texture of the fruit. Freshly cut pineapple maintains its freshness for about 5 days at 5°C (Torri *et al.*, 2010). Freezing technology has been increasingly popular to preserve fruits. Cooling rate is an important parameter in determining the quality of fruits as slow cooling promotes cell dehydration and shrinkage through the solute concentration effect of the unfrozen matrix. Fast cooling rate is normally related to intra-cellular freezing that eventually leads to cell damage as a result of intra-cellular ice re-crystallization. A recent study shows freezing has a significant effect in texture, colour and soluble solids of two varieties of apples (Chassagne-Berces *et al.*, 2010). Fast cooling rate has been reported to generate small ice crystals that lead to small mean pore diameter in the dried external layer of freeze-dried sour op fruit pulp, thus resulting in difficulty for ice sublimation and consequently high final moisture content in the sample (Ceballos *et al.*, 2012). In another study, freezing of carrot at rapid rates of -4.5 °C/min and -2.4 °C/min showed less softening than slow rates of -0.19 °C/min and -0.05 °C/min (Roy *et al.*, 2001). Freezing alone is not effective in preserving the quality of freshly-cut pineapple due to its juicy nature that causes

ice formation, leading to cellular damage. During thawing, fruits may lose physicochemical and organoleptic properties. Therefore it is important to reduce the moisture content of the fruit prior to freezing.

Osmotic dehydration is used as a pre-treatment to reduce the moisture content of fruits prior to freezing. This process involves soaking the fruit in a concentrated sugar or salt solution to partially remove water from fruits through osmotic pressure (Ramallo & Mascheroni, 2005; Tortoe, 2010). The combination of osmotic dehydration and freezing is known as osmo-dehydrofreezing (Lowithun & Charoenrein, 2009). Reduction in moisture content also decreases the refrigeration load during freezing and costs for subsequent packaging, distribution and storage (Dermensonlouoglou *et al.*, 2007a; Lowithun & Charoenrein, 2009; Ramallo & Mascheroni, 2010). Drip loss or exudation during thawing of osmo-dehydrofrozen fruits is also reported to be minimized (Torreggiani & Bertolo, 2001; Dermensonlouoglou *et al.*, 2007b; Ramallo & Mascheroni, 2010). Various types of sugars can be used for osmotic dehydration such as sucrose, glucose, maltose, maltitol, sorbitol and trehalose (Lowithun & Charoenrein, 2009).

Several researchers have reported the changes of fruits subjected to osmo-dehydrofreezing (Lowithun & Charoenrein, 2009; Agnelli *et al.*, 2005). In this study, sucrose, maltitol and trehalose were used as the osmotic agents for osmotic dehydration of pineapples. Sucrose is a disaccharide which consists of glucose and fructose. Maltitol is a sugar alcohol and has the same degree of sweetness as sucrose. It is hydrolyzed and absorbed at a slower rate than sucrose, and thus, it is used as an alternative sweetener in food products for diabetics (Lowithun & Charoenrein, 2009). Trehalose is an isomer of sucrose, and is a non-reducing disaccharide with approximately 45% of the sweetness of sucrose (Lowithun & Charoenrein, 2009).

In the current study, Josapine variety, which is a hybrid between the 'Johor' variety ('Singapore Spanish' x 'Smooth Cayenne') and the 'Sarawak' variety ('Smooth Cayenne') developed by the Malaysian Agricultural Research and Development Institute (MARDI) was used due to its popularity among Malaysians and its wide availability in Malaysia. Freezing of freshly cut pineapples is important to extend their shelf life. The objective of this study is to determine the effects of different osmotic agents, cooling rate and frozen storage period on the physicochemical properties of osmo-dehydrofrozen pineapple.

## MATERIALS AND METHODS

### *Materials*

Dry ice was purchased from Cryo Gases, Selangor, Malaysia. Filter paper (no. 1, 90 mm) was purchased from Advantec, Japan. Maltitol syrup (75%, food grade) was obtained from Deluxe Melody, Selangor, Malaysia. Pineapples of Josapine variety at commercial maturity were purchased from local hypermarkets in Bandar Sunway, Selangor, Malaysia. Sodium hydroxide pellets were obtained from BDH Laboratory Supplies, Poole, UK. Sucrose, (analytical grade) was purchased from R & M Chemicals, Essex, UK and trehalose (food grade) was purchased from Fukui Confectionery, Selangor, Malaysia.

### *Sample Preparation*

Pineapples of the Josapine variety at commercial maturity were purchased from local hypermarkets around Bandar Sunway, Malaysia. The pineapples were washed with tap water and manually peeled. The top and bottom parts of the pineapple were removed. The total soluble solids were determined using a refractometer (Master-T, Atago, Tokyo, Japan). The ripeness of the pineapples used for this study was controlled by selection of fruits between 10

and 13.5 °Brix (Bx). The remaining portion of the pineapple was cut into rings of 6.7 cm in external diameter and thickness of  $0.9 \pm 0.1$  cm using a doughnut shaper, which helped to remove the core of the fruit. Each individual ring was then cut into quarters. Each pineapple quarter ring was in the range of  $5 \pm 1$ g.

### ***Determination of Water Loss and Solid Gain of Pineapples***

Osmotic dehydration of pineapple pieces was carried out according to Lowithun and Charoenrein (2009) with slight modifications. Individual quarter rings were immersed in 50% (w/v) sucrose, 50% (w/v) trehalose and 50% (v/v) maltitol at a fruit to syrup ratio of 1:5 (w/v). The beakers were sealed with parafilm and placed in an incubator shaker (ZHWHY 200B, Labwit, Shanghai, China) preset at a speed of 150 rpm and temperature of 30°C. The osmotic dehydration was conducted for 1, 2, 3 and 4 hours for each sugar solution. A control fresh fruit without osmotic treatment was also prepared. After the osmotic treatment, samples were blotted dry on baking paper to remove excess solution. The blotting technique was controlled by flipping each piece of fruit from one side to the other at 2 minute intervals for the first 4 minutes, 3 minute intervals for the next 12 minutes, and 2 minute intervals for the last 4 minutes for a total blotting time of 20 minutes. The control fresh fruit and osmotic treated fruits were then weighed and subjected to drying in an oven (UFE400, Memmert, Schwabach, Germany) at 105°C for 20 hours to determine the dry mass of the fruits after all the water had been removed. A preliminary study indicated that the drying time required to achieve a constant dried fruit mass was 18 hours. The mass transfer parameters which were water loss (WL) and solid gain (SG) were calculated using the following equations (Panagiotou *et al.*,1999):

$$\text{WL (g H}_2\text{O/g initial dry matter)} = \frac{(M_0 - m_0) - (M - m)}{m_0} \dots (\text{Eq. 1})$$

$$\text{SG (g solid/g initial dry matter)} = \frac{m - m_0}{m_0} \dots (\text{Eq. 2})$$

where  $M_0$  is the initial mass of the fresh fruit before osmotic treatment (g),  $M$  is the mass of the fruit after osmotic treatment (g),  $m_0$  is the dry mass of fresh fruit (g) and  $m$  is the dry mass of fruit after osmotic treatment.

### ***Osmo-dehydrofreezing***

Pineapple quarter rings were immersed in 50% (w/v) sucrose, trehalose or maltitol solutions for 1 hour in an incubator shaker (ZHWHY 200B, Labwit, Shanghai, China) preset at 150 rpm and 30°C. After osmotic treatment, samples were blotted as for osmotic dehydration. For slow freezing, samples were arranged on a tray and placed into a chest freezer (S500, Ardo, UK) preset to -20°C. A four channel data logging thermo-couple (94461-30, Cole Parmer, Illinois, US) was used to monitor the time required for the samples to be frozen to -20°C by placing the thermo-couple probes in the centre of selected samples. For rapid freezing, samples were arranged on a tray and placed into an ice box containing dry ice. The dry ice temperature was measured to be about -78°C. Samples were fully covered by dry ice (Cryo Gases, Malaysia). The average cooling rate for the chest freezer is 0.25°C /min (hereafter referred to as slow cooling rate) while that of dry ice is 6.13°C/min (hereafter referred to as fast cooling rate). Samples were then placed into freezer bags in the chest freezer and kept at -20°C for arbitrary sampling for 3, 10, 14, 17 and 24 days. A frozen sample without osmotic pre-treatment was prepared as control.

### ***Determination of Physicochemical Properties pH, Titratable Acidity, Total Soluble Solids and Colour***

Frozen samples stored for 3, 10, 17 and 24 days were thawed in beakers sealed with parafilm at 30°C for 1 hour. Thawed samples were then mashed in their respective beakers using the flat end of a spatula prior to measurement of the physical and chemical properties. Values of L\*, a\*, and b\* of the samples were measured using a Colourflex spectrophotometer (45/0 CX2367, HunterLab, Virginia, US). The pH and total soluble solids of samples were measured using a pH meter (SG2, Mettler Toledo, Schwerzenbach, Switzerland) and refractometer (Master-T, Atago, Tokyo, Japan) respectively. Titratable acidity of the samples was determined according to the AOAC official method 942.15. Five gram of the samples was homogenized in 25 mL of Milli-Q water and titrated with 0.1 M NaOH until the pH was 8.1. The sample mixture was stirred with a magnetic stirrer on a hot plate (HMS100, Chemolab Supplies, Malaysia) throughout titration to ensure it was well mixed. The titratable acidity was calculated according to Equation (3).

$$\text{Titratable acidity (g of citric acid/kg of pineapple)} = \frac{V \times 0.1 \times 1000 \times 0.064}{m} \dots (\text{Eq. 3})$$

where 0.1 is the normality of NaOH (N), 0.064 is the conversion factor for citric acid, V is the volume of NaOH required (mL) and m is the mass of pineapple sample used (g).

### ***Drip Loss***

Frozen samples were removed from the freezer after 14 days and placed onto pre-weighed filter papers (Advantec, Japan) in Petri dishes. Samples were thawed in an oven (UFE400, Memmert, Schwabach, Germany) preset at 30°C for 1 hour. The mass of the wet filter paper was measured and the drip loss (DL) was calculated according to Equation (4) (Ramallo & Mascheroni, 2010).

$$\text{DL (g liquid loss/g of fruit)} = \frac{W_t - W_0}{M} \dots (\text{Eq. 4})$$

where  $W_t$  is the mass of the wet filter paper (g),  $W_0$  is the mass of the dry filter paper (g), and M is the mass of the fruit before freezing (g).

### ***Firmness***

Firmness of the osmotic treated and control samples before subjecting to freezing was measured using a texture analyzer (TA-XT *plus*, Stable Micro Systems, Surrey, UK) with the following parameters:

Test mode: compression, pre-test speed: 1.00 mm/s, test speed: 2.00 mm/s, post-test speed: 10.00 mm/s, target mode: distance, distance: 3.00 mm, trigger type: auto (force), trigger force: 5.0 g. After thawing and DL measurement, the samples were subjected to the same firmness measurement. Each individual fruit sample was measured at 3 locations and the average firmness of the fruit was determined. Measurements were performed in triplicate and data were reported as the mean of the triplicates.

### ***Statistical Analysis***

All experiments were conducted in three independent runs ( $n = 3$ ) and data were reported as mean  $\pm$  standard deviation. For determination of water loss and solid gain, drip loss and firmness, each run was conducted using one quarter ring. For determination of pH, titratable acidity, total soluble solids, and colour, each run (osmo-dehydrofreezing and subsequent storage) was conducted with two homogenized quarter rings. The results were tested for normality using Statistical Package for the Social Sciences (SPSS) version 16 software. One-way variance analysis (ANOVA) followed by post-hoc Tukey's test was performed to

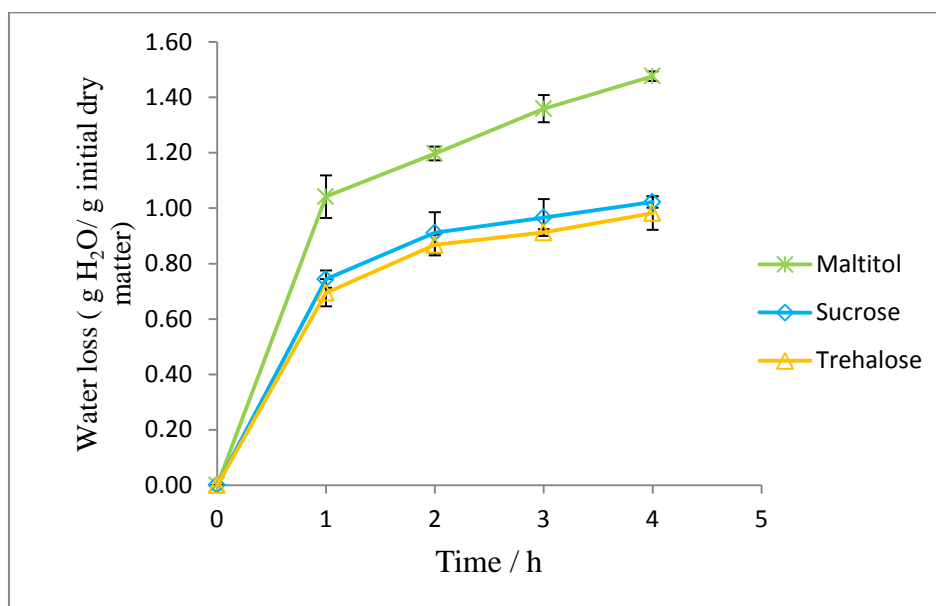
determine significant differences ( $p \leq 0.05$ ). Data which failed the normality test were transformed and retested for normality. Data which failed normality after transformation were analyzed using Kruskal-Wallis test in MedCalc software to determine significant differences ( $p \leq 0.05$ ).

## RESULTS AND DISCUSSION

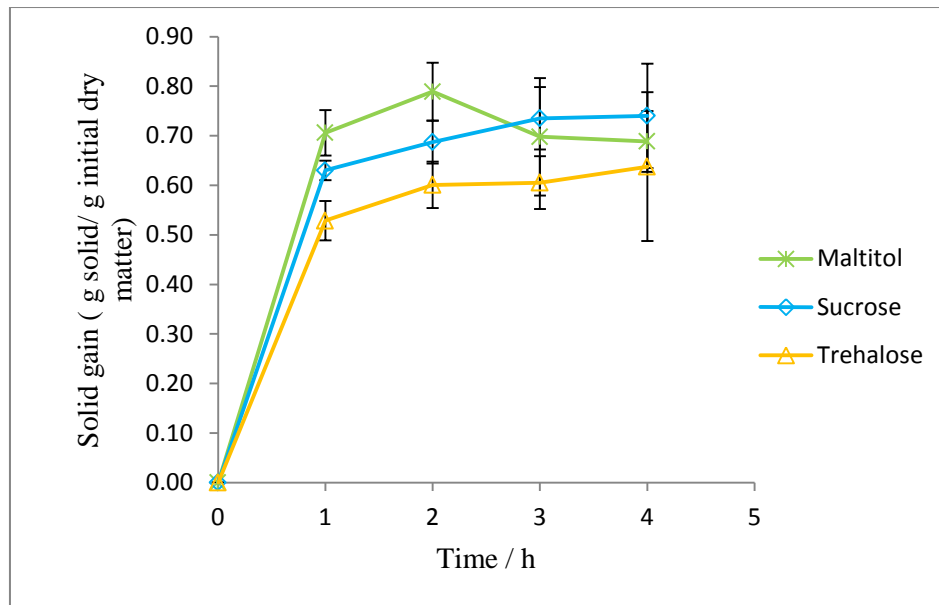
### *Mass Transfer During Osmotic Dehydration*

Water loss and solid gain of pineapple pieces was found to be the highest during the first hour (Figure 1a and b). Subsequently, both water loss and solid gain rate were slower than during the first hour. A similar trend was observed in a previous study on osmotic dehydrated rambutans (Lowithun & Charoenrein, 2009). The difference in rate of water loss observed was due to the large difference in osmotic pressure at the initial stage of osmotic treatment which gradually decreased as more water was removed from the fruit with time (Giannakourou & Taoukis, 2003; Rincon & Kerr, 2010).

Water loss of pineapples treated with maltitol after 4 hours of osmotic treatment was found to be significantly ( $p \leq 0.05$ ) higher than that treated in sucrose or trehalose (Figure 1a). This is in contrast with Lowithun & Charoenrein (2009) who reported no difference in the water loss of rambutans subjected to osmotic dehydration in the same concentration of sucrose, maltitol and trehalose. In this study, the higher water loss in maltitol treated pineapples was probably because maltitol is a sugar alcohol that may have a stronger interaction between its hydroxyl groups with water molecules (Siniti *et al.*, 1999) than sucrose and trehalose. This suggestion, however, needs to be confirmed by further studies. There was no significant ( $p > 0.05$ ) difference in solid gain from the different sugars used for osmotic dehydration (Figure 1b). This could be due to the similar molecular weight of the sugars.



(a)



(b)

**Figure 1. Water loss (a) and solid gain (b) in pineapple slices against osmotic treatment time. Mean  $\pm$  S.D, n = 3**

### *Physicochemical Properties of Osmo-dehydrofrozen Pineapples pH & Titratable Acidity*

In this study, pH of all thawed pineapples was in the range of  $3.44 \pm 0.05$  to  $3.97 \pm 0.03$  (Table 1). Titratable acidity of all thawed samples was found to be in the range of  $6.68 \pm 0.24$  to  $10.1 \pm 0.1$  g citric acid/kg of pineapple (Table 2).

**Table 1. pH of thawed pineapples frozen at slow and fast cooling rates and stored at  $-20^{\circ}\text{C}$  for 0, 3, 10, 17 and 24 days. Mean  $\pm$  S.D, n = 3**

Cooling rate	Storage time (days)	Control	Sucrose	Maltitol	Trehalose
<b>Slow</b>	0	$3.66 \pm 0.15^{\text{aB}}$	$3.76 \pm 0.02^{\text{aB}}$	$3.62 \pm 0.06^{\text{aA}}$	$3.73 \pm 0.03^{\text{aB}}$
	3	$3.73 \pm 0.19^{\text{aB}}$	$3.55 \pm 0.09^{\text{aA}}$	$3.56 \pm 0.07^{\text{aA}}$	$3.61 \pm 0.02^{\text{aA}}$
	10	$3.82 \pm 0.10^{\text{aB}}$	$3.86 \pm 0.04^{\text{aBC}}$	$3.90 \pm 0.06^{\text{aB}}$	$3.91 \pm 0.04^{\text{aC}}$
	17	$3.55 \pm 0.02^{\text{aB}}$	$3.63 \pm 0.03^{\text{aA}}$	$3.59 \pm 0.03^{\text{aA}}$	$3.60 \pm 0.05^{\text{aA}}$
	24	$3.90 \pm 0.16^{\text{aB}}$	$3.97 \pm 0.03^{\text{aC}}$	$3.94 \pm 0.05^{\text{aB}}$	$3.92 \pm 0.05^{\text{aC}}$
<b>Fast</b>	0	$3.66 \pm 0.09^{\text{aB}}$	$3.86 \pm 0.06^{\text{aBC}}$	$3.80 \pm 0.13^{\text{aA}}$	$3.73 \pm 0.06^{\text{aB}}$
	3	$3.44 \pm 0.05^{\text{aA}}$	$3.71 \pm 0.13^{\text{bBC}}$	$3.85 \pm 0.08^{\text{bA}}$	$3.71 \pm 0.09^{\text{bB}}$
	10	$3.65 \pm 0.08^{\text{aB}}$	$3.85 \pm 0.06^{\text{bBC}}$	$3.80 \pm 0.04^{\text{bA}}$	$3.82 \pm 0.03^{\text{bB}}$
	17	$3.50 \pm 0.07^{\text{aA}}$	$3.64 \pm 0.12^{\text{abB}}$	$3.82 \pm 0.04^{\text{bA}}$	$3.71 \pm 0.03^{\text{bB}}$
	24	$3.70 \pm 0.03^{\text{aB}}$	$3.93 \pm 0.09^{\text{cC}}$	$3.85 \pm 0.08^{\text{bcA}}$	$3.78 \pm 0.04^{\text{abB}}$

<sup>a,b,c</sup> Means within the same row with different letters are significantly different ( $p \leq 0.05$ )

<sup>A,B,C</sup> Means within the same column with different letters are significantly different ( $p \leq 0.05$ )

**Table 2. Titratable acidity of thawed pineapples frozen at slow and fast cooling rates and stored at -20°C for 0, 3, 10, 17 and 24 days. Mean ± S.D, n = 3**

Cooling rate	Storage time (days)	Control	Sucrose	Maltitol	Trehalose
Slow	0	7.38 ± 0.73 <sup>aA</sup>	9.86 ± 0.68 <sup>bA</sup>	10.1 ± 0.8 <sup>bA</sup>	9.90 ± 0.64 <sup>bA</sup>
	3	7.98 ± 0.90 <sup>aA</sup>	9.43 ± 0.58 <sup>aA</sup>	9.94 ± 0.27 <sup>aA</sup>	8.83 ± 1.22 <sup>aA</sup>
	10	8.19 ± 0.77 <sup>aA</sup>	9.77 ± 0.70 <sup>aA</sup>	9.51 ± 1.23 <sup>aA</sup>	9.07 ± 0.13 <sup>aA</sup>
	17	8.19 ± 0.77 <sup>aA</sup>	7.98 ± 0.74 <sup>aA</sup>	9.32 ± 0.29 <sup>aA</sup>	9.08 ± 0.34 <sup>aA</sup>
	24	7.38 ± 0.94 <sup>aA</sup>	8.36 ± 1.33 <sup>aA</sup>	8.62 ± 0.93 <sup>aA</sup>	9.26 ± 0.83 <sup>aA</sup>
Fast	0	8.83 ± 0.77 <sup>aA</sup>	7.81 ± 0.59 <sup>aA</sup>	8.02 ± 0.78 <sup>aA</sup>	7.51 ± 0.15 <sup>aA</sup>
	3	8.66 ± 1.37 <sup>aA</sup>	7.42 ± 0.68 <sup>aA</sup>	7.30 ± 0.44 <sup>aA</sup>	8.06 ± 0.26 <sup>aA</sup>
	10	9.04 ± 1.41 <sup>aA</sup>	7.94 ± 0.89 <sup>aA</sup>	8.04 ± 0.33 <sup>aA</sup>	7.94 ± 0.59 <sup>aA</sup>
	17	8.96 ± 0.68 <sup>bA</sup>	7.68 ± 1.28 <sup>abA</sup>	6.68 ± 0.24 <sup>aA</sup>	8.36 ± 0.16 <sup>abA</sup>
	24	8.19 ± 1.44 <sup>aA</sup>	7.38 ± 0.63 <sup>aA</sup>	8.15 ± 0.98 <sup>aA</sup>	9.13 ± 0.73 <sup>aA</sup>

<sup>a,b</sup> Means within the same row with different letters are significantly different ( $p \leq 0.05$ )

<sup>A,B</sup> Means within the same column with different letters are significantly different ( $p \leq 0.05$ )

### Total Soluble Solids

Total soluble solids of all thawed samples with osmotic treatment were significantly ( $p \leq 0.05$ ) higher than the untreated frozen samples (Table 3). This could be due to the uptake of sugar molecules by the fruit cells during osmotic dehydration. The same trend had previously been reported for other osmotic treated fruits such as rambutans, strawberries, mangoes and kiwi (Lowithun & Charoenrein, 2009; Moraga *et al.*, 2006; Rincon & Kerr, 2010; Talens *et al.*, 2002).

There was no difference ( $p > 0.05$ ) in total soluble solids of thawed pineapples treated with sucrose or maltitol upon fast cooling after storage. At slow cooling, maltitol showed higher soluble solids than trehalose after 17 days of storage. At fast cooling, higher soluble solids were observed for pineapples treated with maltitol than trehalose after 3 days of storage. Duration of frozen storage did not alter the total soluble solids of thawed pineapples ( $p > 0.05$ ).

**Table 3. Total soluble solids of thawed pineapples cooled at slow and fast cooling rates and stored at -20°C for 0, 3, 10, 17 and 24 days. Mean ± S.D, n = 3**

Cooling rate	Storage time (days)	Control	Sucrose	Maltitol	Trehalose
Slow	0	16.3 ± 0.5 <sup>aA</sup>	23.3 ± 1.5 <sup>bA</sup>	23.9 ± 2.9 <sup>bA</sup>	22.0 ± 0.8 <sup>bA</sup>
	3	15.1 ± 0.6 <sup>aA</sup>	21.5 ± 1.2 <sup>bA</sup>	22.4 ± 1.4 <sup>bA</sup>	20.9 ± 0.6 <sup>bA</sup>
	10	15.5 ± 0.4 <sup>aA</sup>	21.9 ± 1.5 <sup>bA</sup>	23.1 ± 2.4 <sup>bA</sup>	22.0 ± 1.0 <sup>bA</sup>
	17	15.9 ± 1.7 <sup>aA</sup>	21.1 ± 2.5 <sup>bcA</sup>	21.1 ± 0.1 <sup>cA</sup>	20.0 ± 0.9 <sup>bA</sup>
	24	15.1 ± 0.4 <sup>aA</sup>	22.6 ± 1.6 <sup>bcA</sup>	24.3 ± 1.1 <sup>cA</sup>	20.5 ± 1.6 <sup>bA</sup>
Fast	0	15.4 ± 0.5 <sup>aA</sup>	25.0 ± 0.1 <sup>cA</sup>	23.7 ± 1.4 <sup>bA</sup>	23.3 ± 0.6 <sup>bA</sup>
	3	14.7 ± 0.6 <sup>aA</sup>	23.8 ± 0.0 <sup>bcA</sup>	25.0 ± 1.0 <sup>cA</sup>	22.7 ± 1.5 <sup>bA</sup>
	10	14.7 ± 1.5 <sup>aA</sup>	24.1 ± 0.8 <sup>cA</sup>	22.9 ± 0.8 <sup>bcA</sup>	22.6 ± 0.7 <sup>bA</sup>
	17	15.9 ± 0.9 <sup>aA</sup>	24.1 ± 0.8 <sup>cA</sup>	25.3 ± 1.2 <sup>cA</sup>	22.4 ± 0.7 <sup>bA</sup>
	24	16.2 ± 1.1 <sup>aA</sup>	24.3 ± 1.2 <sup>bcA</sup>	25.4 ± 0.6 <sup>cA</sup>	21.9 ± 1.4 <sup>bA</sup>

<sup>a,b,c</sup> Means within the same row with different letters are significantly different ( $p \leq 0.05$ )

<sup>A,B</sup> Means within the same column with different letters are significantly different ( $p \leq 0.05$ )

**Colour**

Both untreated and osmotic-treated pineapples subjected to a slow cooling rate were found to be lighter in colour (higher L\* values), lower intensity of red (less positive a\* values) and lower intensity of yellow (lower b\* values) compared to those subjected to a fast cooling rate (Table 4(a-c)). The effect of freezing rate on colour change of pineapple slices of ‘Smooth Cayenne’ and ‘Red Spanish’ cultivars was previously reported by Bartolome *et al.* (1996). It was reported that there was no significant difference in the colour of the two pineapple cultivars at freezing rates achieved using an air blast freezer (-50°C) and cold room (-18°C) (Bartolome *et al.*, 1996).

Colour of pineapples was not influenced by the osmotic treatment as there was no significant ( $p > 0.05$ ) difference in colour between the osmotic treated and untreated pineapples. No significant ( $p > 0.05$ ) changes in colour parameters were observed throughout the 24 days of frozen storage.

**Table 4 (a-c). Colour parameters of thawed pineapples cooled at slow and fast cooling rates and stored at -20°C for 0, 3, 10, 17 and 24 days. Mean ± S.D, n = 3**

a)

Colour parameter	Cooling rate	Storage				
		time (day)	Control	Sucrose	Maltitol	Trehalose
L*	slow	0	64.31 ± 3.50 <sup>ab</sup>	62.39 ± 1.06 <sup>ab</sup>	64.13 ± 1.24 <sup>ab</sup>	63.77 ± 0.69 <sup>ab</sup>
		3	60.12 ± 1.17 <sup>ab</sup>	63.09 ± 1.04 <sup>ab</sup>	62.01 ± 0.56 <sup>ab</sup>	62.40 ± 0.26 <sup>ab</sup>
		10	61.86 ± 0.73 <sup>ab</sup>	63.19 ± 0.95 <sup>ab</sup>	63.78 ± 3.31 <sup>ab</sup>	63.06 ± 1.38 <sup>ab</sup>
		17	63.57 ± 1.30 <sup>ab</sup>	62.12 ± 1.14 <sup>ab</sup>	65.69 ± 0.16 <sup>ab</sup>	63.85 ± 2.52 <sup>ab</sup>
		24	61.95 ± 1.82 <sup>ab</sup>	62.52 ± 1.29 <sup>ab</sup>	61.16 ± 0.67 <sup>ab</sup>	63.69 ± 1.22 <sup>ab</sup>
	fast	0	59.24 ± 0.65 <sup>ab</sup>	57.42 ± 0.34 <sup>ab</sup>	58.94 ± 2.28 <sup>ab</sup>	58.17 ± 0.49 <sup>ab</sup>
		3	62.63 ± 1.41 <sup>ab</sup>	58.36 ± 0.79 <sup>ab</sup>	57.11 ± 1.37 <sup>ab</sup>	58.64 ± 2.43 <sup>ab</sup>
		10	60.15 ± 2.12 <sup>ab</sup>	56.47 ± 1.27 <sup>ab</sup>	59.83 ± 1.06 <sup>ab</sup>	56.62 ± 1.36 <sup>ab</sup>
		17	58.68 ± 2.37 <sup>ab</sup>	57.18 ± 0.32 <sup>ab</sup>	57.08 ± 1.93 <sup>ab</sup>	58.44 ± 1.08 <sup>ab</sup>
		24	58.02 ± 0.57 <sup>ab</sup>	56.43 ± 0.54 <sup>ab</sup>	57.44 ± 2.15 <sup>ab</sup>	59.65 ± 2.25 <sup>ab</sup>

<sup>a,b</sup> Means within the same row with different letters are significantly different ( $p \leq 0.05$ )

<sup>A,B</sup> Means within the same column with different letters are significantly different ( $p \leq 0.05$ )



b)

Colour parameter	Cooling rate	Storage				
		time (day)	Control	Sucrose	Maltitol	Trehalose
a*	slow	0	1.23 ± 0.95 <sup>aA</sup>	0.74 ± 0.69 <sup>aA</sup>	0.22 ± 1.11 <sup>aA</sup>	-0.12 ± 0.52 <sup>aA</sup>
		3	-0.46 ± 1.43 <sup>aA</sup>	-0.54 ± 0.93 <sup>aA</sup>	-1.40 ± 0.64 <sup>aA</sup>	0.01 ± 0.31 <sup>aA</sup>
		10	-0.97 ± 1.16 <sup>aA</sup>	-0.21 ± 1.89 <sup>aA</sup>	-0.96 ± 0.96 <sup>aA</sup>	0.17 ± 0.11 <sup>aA</sup>
		17	-0.94 ± 0.60 <sup>aA</sup>	0.73 ± 0.30 <sup>aA</sup>	-0.93 ± 0.53 <sup>aA</sup>	-0.34 ± 1.64 <sup>aA</sup>
		24	-0.21 ± 1.13 <sup>aA</sup>	0.72 ± 0.60 <sup>aA</sup>	0.36 ± 0.55 <sup>aA</sup>	-0.14 ± 0.70 <sup>aA</sup>
	fast	0	0.30 ± 0.63 <sup>aA</sup>	1.67 ± 0.16 <sup>aA</sup>	0.23 ± 1.72 <sup>aA</sup>	1.01 ± 0.78 <sup>aA</sup>
		3	0.10 ± 0.51 <sup>aA</sup>	2.78 ± 0.86 <sup>aA</sup>	3.07 ± 1.48 <sup>ab</sup>	0.99 ± 2.54 <sup>aA</sup>
		10	0.02 ± 0.95 <sup>aA</sup>	1.16 ± 1.36 <sup>aA</sup>	-0.29 ± 1.37 <sup>aA</sup>	1.99 ± 1.04 <sup>aA</sup>
		17	2.11 ± 0.40 <sup>AB</sup>	2.81 ± 1.32 <sup>aA</sup>	2.86 ± 1.08 <sup>AB</sup>	2.42 ± 0.43 <sup>aA</sup>
		24	2.15 ± 0.50 <sup>ab</sup>	2.76 ± 1.04 <sup>aA</sup>	1.98 ± 1.29 <sup>ab</sup>	-0.30 ± 1.16 <sup>aA</sup>

<sup>a,b</sup> Means within the same row with different letters are significantly different ( $p \leq 0.05$ )

<sup>A,B</sup> Means within the same column with different letters are significantly different ( $p \leq 0.05$ )

c)

Colour parameter	Cooling rate	Storage				
		time (day)	Control	Sucrose	Maltitol	Trehalose
b*	slow	0	45.86 ± 6.62 <sup>aA</sup>	50.24 ± 2.90 <sup>aA</sup>	48.88 ± 3.17 <sup>aA</sup>	48.68 ± 2.01 <sup>aA</sup>
		3	45.33 ± 4.06 <sup>aA</sup>	46.02 ± 3.66 <sup>aA</sup>	42.62 ± 3.61 <sup>aA</sup>	48.00 ± 1.92 <sup>aA</sup>
		10	45.71 ± 2.35 <sup>aA</sup>	47.54 ± 6.60 <sup>aA</sup>	44.89 ± 4.92 <sup>aA</sup>	50.87 ± 0.39 <sup>aA</sup>
		17	45.92 ± 3.08 <sup>aA</sup>	51.81 ± 1.71 <sup>aA</sup>	44.91 ± 1.74 <sup>aA</sup>	46.84 ± 6.03 <sup>aA</sup>
		24	47.48 ± 3.66 <sup>aA</sup>	50.96 ± 1.95 <sup>aA</sup>	49.02 ± 2.73 <sup>aA</sup>	49.27 ± 2.63 <sup>aA</sup>
	fast	0	44.87 ± 0.91 <sup>aA</sup>	49.53 ± 0.46 <sup>aA</sup>	44.41 ± 8.48 <sup>aA</sup>	46.65 ± 2.75 <sup>aA</sup>
		3	46.71 ± 1.10 <sup>aAB</sup>	54.15 ± 2.68 <sup>ba</sup>	54.45 ± 2.91 <sup>ba</sup>	48.95 ± 6.42 <sup>abA</sup>
		10	45.59 ± 2.42 <sup>aA</sup>	50.65 ± 4.04 <sup>aA</sup>	45.17 ± 4.63 <sup>aA</sup>	52.09 ± 2.13 <sup>aA</sup>
		17	50.48 ± 0.38 <sup>aB</sup>	54.79 ± 3.46 <sup>aA</sup>	54.05 ± 2.61 <sup>aA</sup>	52.29 ± 0.79 <sup>aA</sup>
		24	50.06 ± 1.37 <sup>ab</sup>	53.81 ± 1.61 <sup>aA</sup>	52.20 ± 2.64 <sup>aA</sup>	45.94 ± 5.22 <sup>aA</sup>

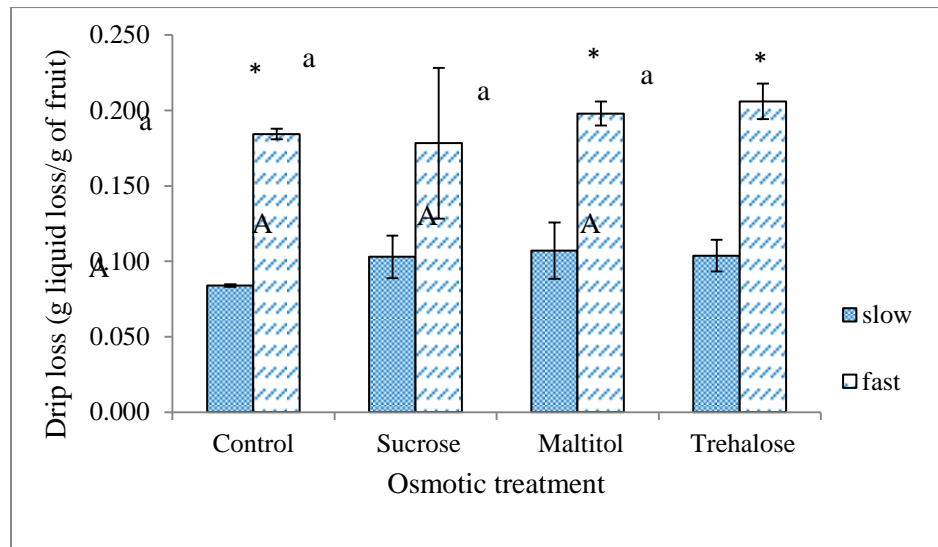
<sup>a,b</sup> Means within the same row with different letters are significantly different ( $p \leq 0.05$ )

<sup>A,B</sup> Means within the same column with different letters are significantly different ( $p \leq 0.05$ ).

### **Drip loss**

Drip loss of thawed pineapples frozen at fast cooling rate was found to be significantly ( $p \leq 0.05$ ) higher than that at slow cooling rate for control and pineapple pieces treated with maltitol or trehalose (Figure 2). In general, slow cooling results in a higher drip loss in thawed samples compared to fast cooling (Goswami, 2010). Slow cooling causes extracellular growth of large ice crystals (Aguilera & Stanley, 1990) that disrupts membrane and makes a higher proportion of solution to drain from cells. However, if damage is limited,

water could diffuse back to the cells (Grout *et al.*, 1991). This postulation, however, needs to be further confirmed. There was no significant difference in the drip loss between osmotic treated and untreated samples subjected to both slow and fast cooling rate ( $p > 0.05$ ) (Figure 2).



**Figure 2. Comparison of drip loss of thawed pineapples at slow and fast cooling rates.**

Mean  $\pm$  S.D, n = 3

<sup>A, B</sup> Means at slow cooling rate, different letters indicate significant difference ( $p \leq 0.05$ )

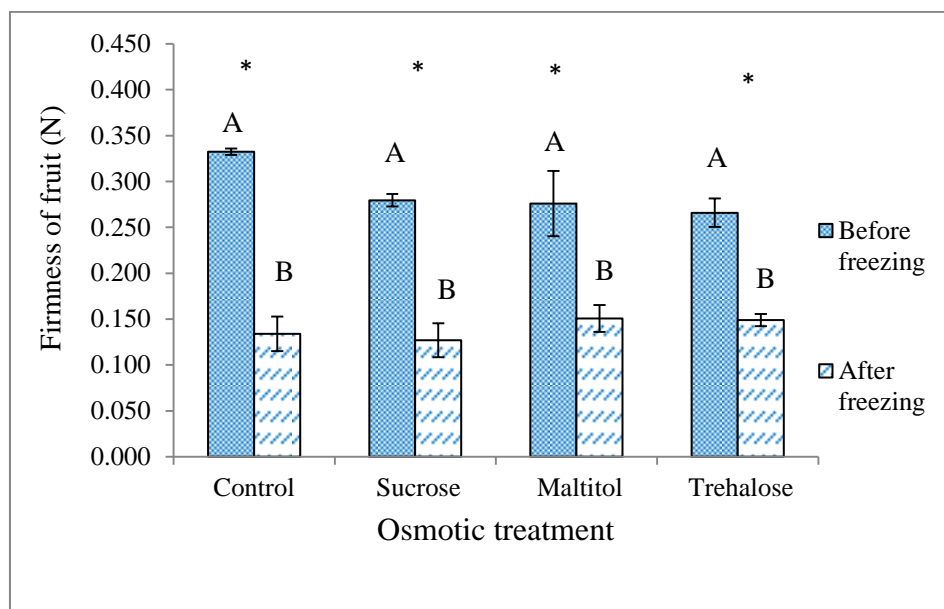
<sup>a, b</sup> Means at fast cooling rate, different letters indicate significant difference ( $p \leq 0.05$ )

\* Means within osmotic treatment, there is a significant difference between slow and fast cooling rate ( $p \leq 0.05$ ).

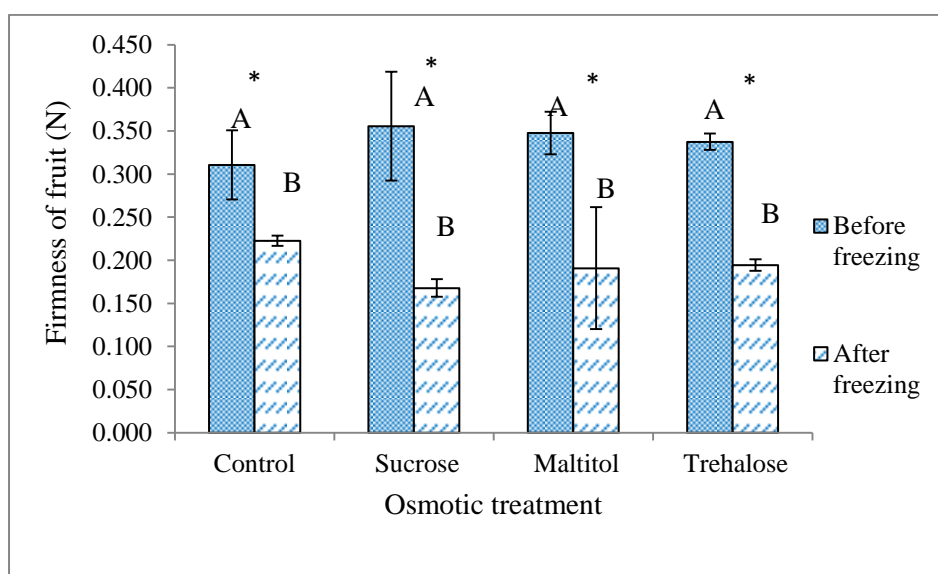
### ***Firmness***

All pineapples decreased ( $p \leq 0.05$ ) in firmness after freezing and thawing (Figure 3a and b). The decrease in firmness could be attributed to the ice crystal damage and solute concentration effects during freezing, which cause cellular damage to the fruit cells. Another potential mechanism for the decreased firmness after 14 days storage is the recrystallization of ice into larger ice crystals during thawing, which further damaged the cellular structure of pineapples (Kerr, 2004).

The change in firmness was significantly lower in osmotic treated samples compared to untreated samples at slow cooling rate ( $p \leq 0.05$ ) (Figure 4). This was probably due to the water loss from the fruit during osmotic treatment, which reduced the amount of available water to form ice. Therefore, less ice crystals were available to cause mechanical damage of fruit cellular structures (Moraga *et al.*, 2006). The presence of sugar in osmotic treated samples also depressed the freezing point of water. Therefore, less ice was formed and less textural damage occurred in the fruits. There was no difference in the change of firmness between osmotic treated and untreated samples at fast cooling rate ( $p > 0.05$ ). Fast cooling resulted in a smaller change of the firmness of pineapple pieces compared to slow cooling. This could be due to the subtle effect of fast cooling on cellular damage, thus leading to minimal firmness change of the samples upon freezing and thawing (Roy *et al.*, 2001).



(a)



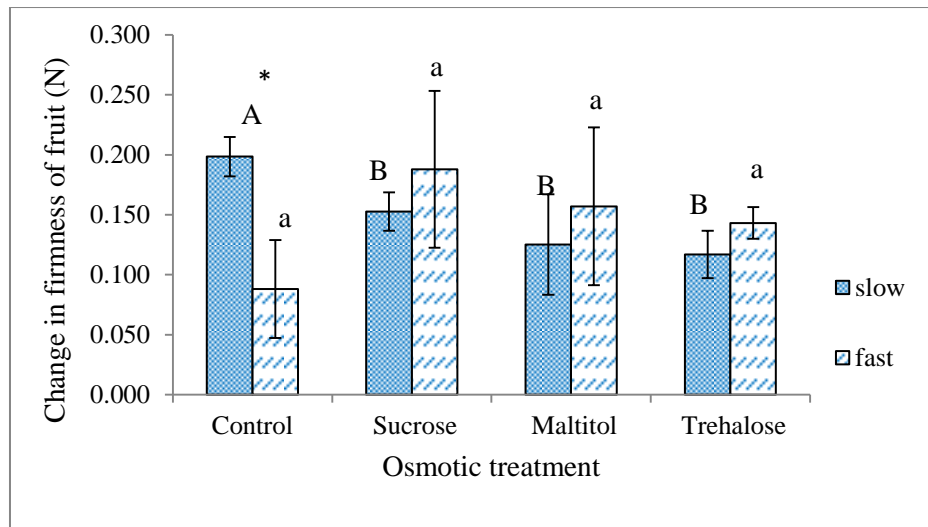
(b)

**Figure 3. Firmness of pineapple slices before and after freezing at (a) slow and (b) fast cooling rates.**

Mean  $\pm$  S.D, n = 3

<sup>A, B</sup> Means within osmotic treatment, different letters indicate significant difference ( $p \leq 0.05$ )

\* Means within osmotic treatment, there is a significant difference between before and after freezing treatment ( $p \leq 0.05$ )



**Figure 4. Comparison of change in firmness of thawed pineapples subjected to slow and fast freezing.**

Mean  $\pm$  S.D, n = 3

<sup>A, B</sup> Means at slow cooling rate, different letters indicate significant difference ( $p \leq 0.05$ )

<sup>a, b</sup> Means at fast cooling rate, different letters indicate significant difference ( $p \leq 0.05$ )

\* Means within osmotic treatment, there is a significant difference between slow and fast cooling rate ( $p \leq 0.05$ ).

## CONCLUSION

pH and titratable acidity of thawed osmo-dehydrofrozen pineapple pieces were in the range of  $3.44 \pm 0.05$  to  $3.97 \pm 0.03$  and  $6.68 \pm 0.24$  to  $10.1 \pm 0.1$  g citric acid/kg of pineapple, respectively. In the absence of sugar solution, slow cooling resulted in a higher change in firmness but lower drip loss than that of fast cooling. The current result shows that firmness is not correlated with drip loss and this needs to be further confirmed in future studies. In the presence of maltitol and trehalose, a small change in firmness and higher drip loss was found at slow cooling compared to fast cooling. In the presence of sucrose, there was no significant change in drip loss at both slow and fast cooling rates. Fast cooling in the presence of sucrose, maltitol or trehalose had no effect on the firmness of samples. Frozen storage for 24 days did not affect the physiochemical properties of osmo-dehydrofrozen pineapples.

## ACKNOWLEDGEMENT

The authors would like to thank School of Science, Monash University Sunway Campus for the financial support for this study.

## REFERENCES

- Agnelli, M. E., Marani, C. M., & Mascheroni, R. H. 2005. Modelling of Heat and Mass Transfer during (Osmo) Dehydrofreezing of Fruits. *Journal of Food Engineering*, **69**: 415-424.

- Aguilera J. M., & Stanley D. W. 1990. *Microstructural Principles of Food Processing and Engineering*. London: Elsevier Applied Science.
- Association of Official Analytical Chemists (AOAC). 2000. *Official Methods of Analysis of AOAC International*. 17th edn, Gaithersburg, Maryland.
- Bartolome, A. P., Ruperez, P., & Fuster, C. 1996. Freezing Rate and Frozen Storage Effects on Color and Sensory Characteristics of Pineapple Fruit Slices. *Journal of Food Science*, **61**: 154-156.
- Ceballos, A. M., Giraldo, G. I., & Orrego, C. E. 2012. Effect of Freezing Rate on Quality Parameters of Freeze Dried Soursop Fruit Pulp. *Journal of Food Engineering*, **111**: 360–365.
- Chassagne-Berces, S., Fonseca, F., Citeau, M., & Marin, M. 2010. Freezing Protocol Effect on Quality Properties of Fruit Tissue according to the Fruit, the Variety and the Stage of Maturity. *LWT: Food Science and Technology*, **43**: 1441-1449.
- Dermensonlouoglou, E. K., Giannakourou, M. C., & Taoukis, P. S. 2007a. Kinetic Modeling of the Degradation of Quality of Osmo-Dehydrofrozen Tomatoes during Storage. *Food Chemistry*, **103**: 985-993.
- Dermensonlouoglou, E. K., Giannakourou, M. C., & Taoukis, P. S. 2007b. Stability of Dehydrofrozen Tomatoes Pretreated with Alternative Osmotic Solutes. *Journal of Food Engineering*, **78**: 272- 280.
- Giannakourou, M. C., & Taoukis, P. S. 2003. Stability of Dehydrofrozen Green Peas Pretreated with Nonconventional Osmotic Agents. *Journal of Food Science*, **68**: 2002-2010.
- Goswami, T. K. 2010. Role of Cryogenics in Food Processing and Preservation. *International Journal of Food Engineering*, **6**: 1-29.
- Grout, B. W, Morris, G. J., & McLellau, M. R. 1991. *Freezing of Fruit and Vegetables*. In *Food Freezing Today and Tomorrow*. (Bald W.B., ed.), London: Springer-Verlag: 113–121.
- Kerr, W. L. 2004. *Texture in frozen foods*. In Handbook of Frozen Foods. (Hui, Y. H., Cornillon, P., Legaretta, I. G., Lim, M. H., Murrell, K. D., and Nip, W. K. eds.), New York: Marcel Dekker: 149-168.
- Lowithun, N., & Charoenrein, S. 2009. Influence of Osmodehydrofreezing with Different Sugars on the Quality of Frozen Rambutan. *International Journal of Food Science and Technology*, **44**: 2183-2188.
- Moraga, G., Martinez-Navarette, N., & Chiralt, A. 2006. Compositional Changes of Strawberry Due to Dehydration, Cold Storage and Freeze-thawing Process. *Journal of Food Processing and Preservation*, **30**: 458-474.
- Panagiotou, N. M., Karathanos, V. T., & Maroulis, Z. B. 1999. Effect of Osmotic Agent on Osmotic Dehydration of Fruits. *Drying Technology*, **17**: 175-189.
- Ramallo, L. A., & Mascheroni, R. H. 2005. Rate of Water Loss and Sugar Uptake During the Osmotic Dehydration of Pineapple. *Brazilian Archives of Biology and Technology*, **48**: 761-770.
- Ramallo, L. A., & Mascheroni, R. H. 2010. Dehydrofreezing of Pineapple. *Journal of Food Engineering*, **99**: 269-275.
- Rincon, A., & Kerr, W. L. 2010. Influence of Osmotic Dehydration, Ripeness, and Frozen Storage on Physicochemical Properties of Mango. *Journal of Food Processing and Preservation*, **34**: 887 903.
- Roy, S. S., Taylor, T. A., & Kramer, H. L. 2001. Textural and Ultrastructural Changes in Carrot Tissue as Affected by Blanching and Freezing. *Journal of Food Science*, **66**: 176-180.

- Siniti, M., Jabrane, S., & Letoffe, J. M. 1999. Study of the Respective Binary Phase Diagrams of Sorbitol with Mannitol, Maltitol and Water. *Thermochimica Acta*, **325**: 171-180.
- Talens, P., Martinez-Navarrete, N., Fito, P., & Chiralt, A. 2002. Changes in Optical and Mechanical Properties during Osmodehydrofreezing of Kiwi Fruit. *Innovative Food Science and Emerging Technologies*, **3**: 191-199.
- Torreggiani, D., & Bertolo, G. 2001. Osmotic Pretreatments in Fruit Processing: Chemical, Physical and Structural Effects. *Journal of Food Engineering*, **49**: 247-253.
- Torri, L., Sinelli, N., & Limbo, S. 2010. Shelf Life Evaluation of Fresh-Cut Pineapple by using an Electronic Nose. *Postharvest Biology and Technology*, **56**: 239-245.
- Tortoe, C. 2010. A Review of Osmodehydration for Food Industry. *African Journal of Food Science*, **4**: 303-324.