

สมบัติทางเคมีกายภาพของมะเกี๋ยงแช่อิ่มอบแห้ง  
ที่มีผลมาจากสารละลายออสโมติกที่แตกต่างกัน

Physicochemical Properties of Osmo-Dried Ma-Kiang

(*Cleistocalyx nervosum* var. *paniala*) as Affected by Different Osmotic Solutions

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บทคัดย่อ

การตรวจสอบสมบัติทางเคมีกายภาพของมะเกี๋ยงแช่อิ่มอบแห้งที่ผ่านการเตรียมด้วยสารละลายออสโมติกชนิดที่แตกต่างกัน สารละลายออสโมติกที่ศึกษาได้แก่ สารละลายน้ำตาลซูโครส (ความเข้มข้น 60 และ 70 องศาบริกซ์) ร่วมกับกรดซิตริก และแคลเซียมคลอไรด์ ร้อยละ 1 จากนั้นทำแห้งมะเกี๋ยงแช่อิ่มด้วยตู้อบลมร้อนที่อุณหภูมิ 60 องศาเซลเซียส เป็นเวลา 8 ชั่วโมง ผลการทดลองแสดงให้เห็นว่ามะเกี๋ยงแช่อิ่มอบแห้งที่ผ่านการเตรียมขึ้นต้นด้วยสารละลายออสโมติกชนิดต่างๆ ไม่มีความแตกต่างกันอย่างมีนัยสำคัญ ในด้านของค่าสี ( $L^*$ ,  $a^*$  และ  $b^*$ ), ความชื้น, ปริมาณน้ำอิสระ, ความเป็นกรด-ด่าง, ปริมาณของแข็งที่ละลายน้ำได้ทั้งหมด ปริมาณกรดทั้งหมด และปริมาณกรดแอสคอร์บิก เมื่อพิจารณาคุณภาพทางประสาทสัมผัสพบว่าตัวอย่างมะเกี๋ยงแช่อิ่มอบแห้งที่ผ่านการเตรียมขึ้นต้นด้วยสารละลายออสโมติก ชนิดที่ใช้สารละลายน้ำตาลซูโครส ความเข้มข้น 70 องศาบริกซ์ ร่วมกับกรดซิตริก ร้อยละ 1 ( $70^\circ\text{Bx-CA}$ ) มีค่าคะแนนความชอบโดยรวมสูงที่สุด การแช่อิ่มสามารถใช้เป็นวิธีการเตรียมขึ้นต้นก่อนการอบแห้งด้วยลมร้อนได้ ซึ่งพบว่าสารละลายออสโมติก ชนิดที่ใช้สารละลายน้ำตาลซูโครส ความเข้มข้น 70 องศาบริกซ์ ร่วมกับกรดซิตริก ร้อยละ 1 สามารถช่วยปรับปรุงคุณภาพทางประสาทสัมผัส ของมะเกี๋ยงอบแห้งได้

คำสำคัญ : มะเกี๋ยง, การแช่อิ่ม, สารละลายออสโมติก, การอบแห้งด้วยลมร้อน, สมบัติทางเคมีกายภาพ

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## Abstract

Physicochemical properties of osmo-dried Ma-kiang (*Cleistocalyx nervosum* var. *paniala*) prepared by various osmotic solutions were investigated. Sucrose solution (60 and 70 °Bx) mixed with 1% citric acid (CA) and calcium chloride ( $\text{CaCl}_2$ ) were used. Osmo-dried Ma-kiang fruits were hot air dried at 60°C for 8 hr. Results showed that there were no significant differences in all samples with respect to color ( $L^*$ ,  $a^*$ , and  $b^*$  values), moisture content, water activity, pH, total soluble solid, titratable acidity and ascorbic acid content ( $p > 0.05$ ). However, hardness and firmness were significantly different ( $p \leq 0.05$ ). When considering sensory evaluation, the highest hedonic score of overall liking was found in the 70°Bx sucrose solution containing 1% citric acid (70°Bx-CA) sample. Osmotic dehydration was could be used as the promising pre-treatment before hot-air drying, in which 70°Bx-CA was an appropriate osmotic solution that enabled improvement of the sensory quality of dried Ma-kiang fruits.

**Keywords :** *Cleistocalyx nervosum* var. *paniala*, osmotic dehydration, osmotic solution, hot-air drying, physicochemical properties

## Introduction

*Cleistocalyx nervosum* var. *paniala* is a tropical fruit belonging to the Myrtaceae family. They are widely grown throughout India, Burma, Bangladesh, Vietnam and particularly the Northern provinces of Thailand such as Chiang Mai, Chiang Rai, Lamphun, Lampang and Mae Hong Son (Subhadrabandhu, 2001). This fruit is commonly known as Ma-kiang and is also categorized as an indigenous fruit of the Plant Genetic Conservation Project under The Royal Initiation of Her Royal Highness Princess Maha Chakri Sirindhorn (RSPG) in Thailand. The edible fruits are a purplish-red fleshy berry with a sour and astringent taste. Moreover, they are a rich source of vitamins, minerals and bioactive compounds which have a great impact on human health and disease (Manosroi *et al.*, 2015; Poontawee *et al.*, 2016). This fruit is only in season in July and August (Subhadrabandhu, 2001). Besides the limitation of harvest time, it can also deteriorate easily due to its high water content, therefore preserving techniques have been employed to enhance its shelf life.

Osmotic dehydration is a traditional water removal process by which water is partially eliminated from the cellular materials when they are immersed in a concentrated solution of soluble solute (such as sugar or salt). As a result of this process,  $a_w$  in foods are reduced (da Costa Ribeiro *et al.*, 2016; Singh *et al.*, 2007). This method is often used as a pre-treatment and is subjected to convective drying methods. This is because it can retain the quality in terms of morphological, organoleptic, and nutritional characteristics of dried fruits (Sablani and Shafiqur Rahman, 2003). Osmotic dehydration through further air-drying was extensively proposed for fruits by several research studies (da Costa Ribeiro *et al.*, 2016; Fernandes *et al.*, 2006; González-Martínez *et al.*, 2006; Kaushal and Sharma, 2016; Park *et al.*, 2003; Sette *et al.*, 2016). It should be noted that the osmotically dried fruit quality is

dependent on many factors such as osmotic dehydration manner (Phisut *et al.*, 2013), immersion time and temperature (Barat *et al.*, 2001; Mandala *et al.*, 2005), sample characteristics (Sablani and Shafiur Rahman, 2003), sample and osmotic solute ratio (Khoji and Hesari, 2007; Singh *et al.*, 2007), osmotic agent type (Chauhan *et al.*, 2011; Lerici *et al.*, 1985), and osmotic solute concentration (Barat *et al.*, 2001).

Although, there have been previous reports on the effect of osmotic solution (type and/or concentration) on the physicochemical property and quality of many dried fruits such as mangoes (Sablani and Shafiur Rahman, 2003), jackfruit (Srinang *et al.*, 2015), pineapples (Paul *et al.*, 2014), apples (Chauhan *et al.*, 2011; Mandala *et al.*, 2005), red pitayas (Haj Najaf *et al.*, 2014), and lychees (Lakshmishri Roy, 2015). The physicochemical properties of the indigenous Ma-kiang fruit treated with osmotic dehydration followed by convective hot-air drying has not been investigated. The objective of this work was to determine the effect of different osmotic solutions on the quality and physicochemical properties of hot-air dried Ma-kiang.

## Methods

### Materials

Ma-kiang (*Cleistocalyx nervosum* var. *paniala*) fruits were provided by Rajamangala University of Technology Lanna Lampang, Lampang, Thailand. The fruits were washed, defective samples were removed, and the usable samples were kept at  $-20^{\circ}\text{C}$  until used for experiments. Commercial grade sucrose was bought from a local supermarket in Phayao, Thailand. Citric acid (CA) and calcium chloride ( $\text{CaCl}_2$ ) were purchased from the Union Science Co., Ltd., (Chiang Mai, Thailand).

### Osmotic dehydration procedure

In order to reduce the sour and astringent taste, Ma-kiang fruits were soaked in 4% and 20%  $\text{CaCl}_2$  solutions, respectively, and were kept at  $5^{\circ}\text{C}$  for 24 h. The fruits were then blanched in boiled water at  $90^{\circ}\text{C}$  for 10 min, dried using tissue paper and transferred to osmotic dehydration processing. The fruits were immersed in different osmotic solutions including 60°Bx sucrose solution containing 1% citric acid (60°Bx-CA), 60°Bx sucrose solution containing 1%  $\text{CaCl}_2$  (60°Bx- $\text{CaCl}_2$ ), 70°Bx sucrose solution containing 1% citric acid (70°Bx-CA) and 70°Bx sucrose solution containing 1%  $\text{CaCl}_2$  (70°Bx- $\text{CaCl}_2$ ) at a fruit/osmotic solution ratio of 1:3 (w/v). The 10% osmotic solution concentration was raised slightly daily until it reached the final concentration (60°Bx and 70°Bx). The concentration of osmotic solution was monitored using a handheld refractometer (Atago-Master-20M, Atago Co. Ltd, Tokyo, Japan). This process was conducted over 3 days at  $5^{\circ}\text{C}$ . After osmotic dehydration treatment, the samples were blanched at  $90^{\circ}\text{C}$  for 2 min and drained for 2 min to remove the residual osmotic solution. The osmo-dried samples were dried in a tray dryer at  $60^{\circ}\text{C}$  for 8 hr. All experiments were carried out in at least three replications.

### Physicochemical properties measurement

The color (CIE  $L^*$ ,  $a^*$  and  $b^*$ ) values of osmo-dried Ma-kiang fruits were determined using a Hunter LAB (ColorQuest XE, Hunter Associates Laboratory, Virginia, USA). Texture measurement was carried out with a Texture Analyzer (TA-XT Plus, Stable Micro Systems Ltd., Surrey, UK). The firmness and hardness of the samples were evaluated using a puncture probe and a knife blade, respectively. Moisture content (MC) was evaluated gravimetrically (AOAC methods 925.09, 1990) and  $a_w$  was measured at 25°C with a water activity meter (AquaLab 4 TE, Decagon Devices Inc., Pullman, WA, USA). The pH and total soluble solid (TSS) values were measured with a digital pH meter (Sg2-ELK, Mettler Toledo Co., Ltd., Greifensee, Switzerland) and a hand refractometer (Atago Master-20M, Atago Co. Ltd, Tokyo, Japan), respectively. Titratable acidity (TA) was measured using the titrimetric method (AOAC, 1984), TA value was expressed as % citric acid. To evaluate color and texture of the osmo-dried Ma-kiang fruits, five samples from each treatment were used. Ascorbic acid was determined using the colorimetric method (Klein and Perry, 1982) with minor modification. Absorbance was measured at 510 nm and the results were expressed as mg ascorbic acid per 100 g of dry weight (mg/100g DW). All determinations were performed in triplicate.

### Sensory evaluation

A 9-point hedonic scale preference test was employed to assess the differences between osmo-dried Ma-kiang treated with various osmotic solutions using the following scale: 1 = dislike extremely, 2 = dislike very much, 3 = dislike moderately, 4 = dislike slightly, 5 = neither like nor dislike, 6 = like slightly, 7 = like moderately, 8 = like very much, and 9 = like extremely (Resurreccion, 1998). The sensory attributes (color, appearance, texture, flavor, taste, and overall acceptability) were evaluated by a panel of 30 assessors. All samples were labeled with 3-digit numbers and distilled water was served between each sample to rinse the mouth.

### Statistical analysis

All results were expressed as mean  $\pm$  SD. The significance of the treatments was statistically evaluated by analysis of variance (ANOVA) at the 95% probability level. When significant ( $p \leq 0.05$ ), the means were compared using Duncan's New Multiple Range Test (DMRT).

## Results

### Physicochemical properties of osmo-dried Making

The osmo-dried Ma-kiang fruits were investigated for their physicochemical properties and compared with several pre-osmosed solutions. The sucrose solution (60°Bx and 70°Bx) mixed with additives (CA and  $\text{CaCl}_2$ ) was used as the osmotic solution. The list of color ( $L^*$ ,  $a^*$ , and  $b^*$ ), firmness and hardness value of osmo-dried Ma-kiang are given in Table 1. The color of Ma-kiang fruits derives from the anthocyanin pigments which provide the purplish-red color (Jansom *et al.*, 2008). The osmo-dried Ma-kiang color was determined as  $L^*$  value, from 25.15 to 26.54,

$a^*$  value from 9.44 to 10.95, and  $b^*$  value from 0.12 to 0.26. The results revealed that no significant difference in color ( $L^*$ ,  $a^*$ , and  $b^*$ ) value were obtained in each treatment ( $p>0.05$ ). But hardness and firmness were significantly different ( $p\leq 0.05$ ). Among all osmotic solution treatments, the 60°Bx-CaCl<sub>2</sub> sample showed higher firmness (50.44± 3.09 gf) than the 70°Bx-CaCl<sub>2</sub> (48.93 ± 2.89 gf), the 60°Bx-CA (45.98 ± 2.44 gf) and 70°Bx-CA (37.22<sup>d</sup> ± 3.05 gf), respectively ( $p\leq 0.05$ ).

The hardness ranged of 94.62-127.78N. The samples pre-osmosed in the 60°Bx-CaCl<sub>2</sub> showed the highest hardness ( $p\leq 0.05$ ), followed by 70°Bx-CaCl<sub>2</sub> and 60°Bx-CA. For 70°Bx-CA, hardness of 70°Bx-CA sample was lower than others ( $p\leq 0.05$ ) (Table 1).

**Table 1** Physical properties of osmo-dried Ma-kiang pre-treated with different osmotic solutions

Osmotic solution	Color			Texture	
	$L^*$	$a^*$	$b^*$	Firmness (gf)	Hardness (N)
60°Bx-CA	25.96 <sup>a</sup> ± 1.04	9.44 <sup>a</sup> ± 1.23	0.22 <sup>a</sup> ± 0.83	45.98 <sup>c</sup> ± 2.44	107.13 <sup>b</sup> ± 2.53
60°Bx-CaCl <sub>2</sub>	26.54 <sup>a</sup> ± 1.37	9.83 <sup>a</sup> ± 1.65	0.20 <sup>a</sup> ± 0.90	50.44 <sup>a</sup> ± 3.09	127.78 <sup>a</sup> ± 2.58
70°Bx-CA	26.03 <sup>a</sup> ± 0.72	10.95 <sup>a</sup> ± 1.71	0.12 <sup>a</sup> ± 0.92	37.22 <sup>d</sup> ± 3.05	94.62 <sup>c</sup> ± 2.54
70°Bx-CaCl <sub>2</sub>	25.15 <sup>a</sup> ± 0.70	9.46 <sup>a</sup> ± 1.92	0.26 <sup>a</sup> ± 0.64	48.93 <sup>b</sup> ± 2.89	108.78 <sup>b</sup> ± 1.70

Different letters in the same column indicates the mean values are significantly different ( $p\leq 0.05$ )

**Table 2** Chemical properties of osmo-dried Ma-kiang pre-treated with different osmotic solutions

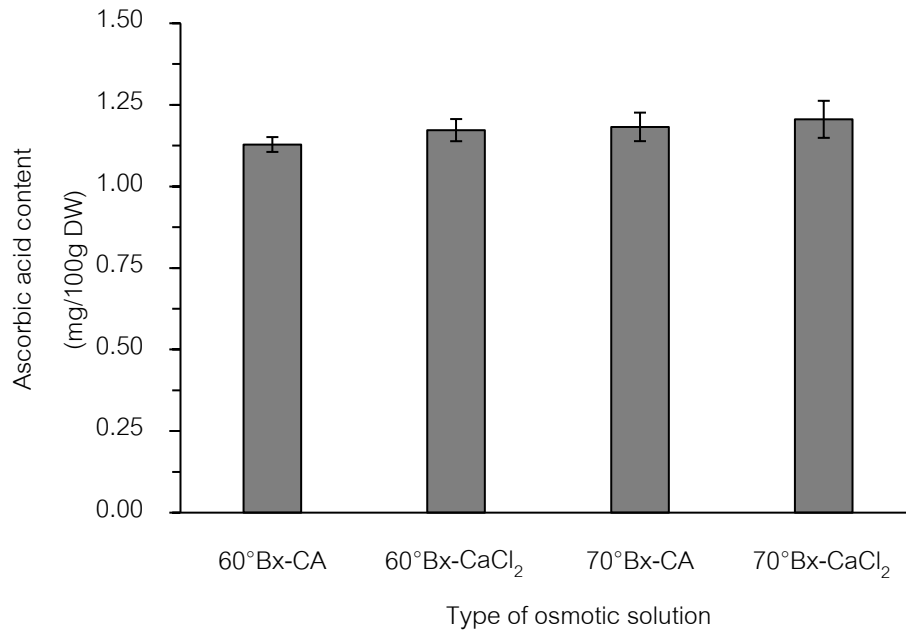
Osmotic solution	MC (% w.b.)	$a_w$	TSS (°Bx)	TA (%)	pH
60°Bx-CA	47.65 <sup>a</sup> ± 1.17	0.69 <sup>a</sup> ± 0.07	33.33 <sup>a</sup> ± 0.52	0.07 <sup>a</sup> ± 0.01	2.73 <sup>b</sup> ± 0.04
60°Bx-CaCl <sub>2</sub>	47.47 <sup>a</sup> ± 1.16	0.67 <sup>a</sup> ± 0.04	31.17 <sup>a</sup> ± 2.02	0.06 <sup>a</sup> ± 0.01	2.89 <sup>a</sup> ± 0.08
70°Bx-CA	33.71 <sup>b</sup> ± 1.33	0.64 <sup>a</sup> ± 0.02	31.83 <sup>a</sup> ± 3.04	0.07 <sup>a</sup> ± 0.01	2.74 <sup>b</sup> ± 0.06
70°Bx-CaCl <sub>2</sub>	32.61 <sup>b</sup> ± 0.76	0.63 <sup>a</sup> ± 0.02	30.50 <sup>a</sup> ± 3.66	0.07 <sup>a</sup> ± 0.01	2.89 <sup>a</sup> ± 0.09

Different letters in the same column indicates the mean values are significantly different ( $p\leq 0.05$ )

The values of MC,  $a_w$ , TSS, TA, and pH of osmo-dried Ma-kiang treated with different osmotic solutions are shown in Table 2. The MC values ranged from 32.61 to 47.65 (% w.b.) and the measured  $a_w$  values ranged between 0.63 and 0.69. This suggests that osmo-dried Ma-kiang products were considered safe for the microbial pathogens growing at which the critical point of  $a_w$  is less than 0.85 (Labuza and Altunakar, 2008). Meanwhile, the values of

TSS, TA, and pH were in the range from 30.50 to 33.33°Bx, 0.06 to 0.07 %, and 2.73 to 2.89, respectively. It can be seen that there were not significantly different  $a_w$ , TSS and TA values in all samples ( $p>0.05$ ) (Table 2).

The effect of different osmotic solutions on ascorbic acid content of osmo-dried Ma-kiang is shown in Figure 1. The average of ascorbic acid values of pre-osmosed fruits in 70°Bx-CaCl<sub>2</sub>, 70°Bx-CA, 60°Bx-CaCl<sub>2</sub> and 60°Bx-CA were 1.21, 1.18, 1.17 and 1.13 mg/100g DW, respectively. However, the different osmotic solutions did not significantly affect ascorbic acid content in all samples ( $p>0.05$ ).



**Figure 1** Ascorbic acid content of osmo-dried Ma-kiang pre-treated with different osmotic solutions

#### Sensory evaluation of osmo-dried Making

Sensory attributes of osmo-dried Ma-kiang were evaluated using a 9-point hedonic scale (where 1-9 represent extremely poor to excellent, respectively) by 30 panelists. The mean scores for each quality attribute of Ma-kiang undergoing different pre-osmotic dehydration conditions are listed in Table 3. The 70°Bx-CA sample had the highest overall liking score ( $p\leq 0.05$ ). However, samples infused in 70°Bx-CA treatment were not significantly better in the score of color, appearance, texture, flavor and taste than those samples infused in 70°Bx-CaCl<sub>2</sub> solution (Table 3).

**Table 3** Sensory evaluation of osmo-dried Ma-kiang pre-treated with different osmotic solutions

Osmotic solution	Sensory attribute					
	Color	Appearance	Texture	Flavor	Taste	Overall liking
60°Bx-CA	5.9 <sup>ab</sup> ±1.9	5.6 <sup>b</sup> ±1.6	5.5 <sup>bc</sup> ±1.5	5.5 <sup>ab</sup> ±1.8	6.2 <sup>bc</sup> ±1.6	6.1 <sup>cd</sup> ±1.6
60°Bx-CaCl <sub>2</sub>	5.6 <sup>b</sup> ±1.7	5.4 <sup>c</sup> ±1.7	5.3 <sup>c</sup> ±1.7	5.1 <sup>b</sup> ±1.8	5.7 <sup>c</sup> ±1.9	5.8 <sup>d</sup> ±1.5
70°Bx-CA	6.4 <sup>a</sup> ±2.0	6.3 <sup>a</sup> ±1.7	6.3 <sup>a</sup> ±1.7	6.2 <sup>a</sup> ±1.7	7.0 <sup>a</sup> ±1.4	7.0 <sup>a</sup> ±1.4
70°Bx-CaCl <sub>2</sub>	6.3 <sup>ab</sup> ±2.0	6.3 <sup>a</sup> ±2.0	6.4 <sup>a</sup> ±1.8	6.0 <sup>a</sup> ±2.2	6.5 <sup>ab</sup> ±2.0	6.9 <sup>bc</sup> ±1.7

Different letters in the same column indicates the mean values are significantly different ( $p \leq 0.05$ )

### Discussion

The osmo-dried fruits had slightly changed of the texture quality depending on the osmotic solution treatment. This demonstrated that the structure, hardness and firmness of osmo-dried Ma-kiang were significantly affected by kind of osmotic solution. This result corresponds with the hardness test which showed the maximum value of hardness could be obtained in the samples infused with the 60°Bx-CaCl<sub>2</sub> condition. This is due to the presence of CaCl<sub>2</sub> in the osmotic solution. It should be noted that the CaCl<sub>2</sub> can incorporated into the cell structure of fruit; consequently, the tissue and texture of the material could be preserved (Landim *et al.*, 2016; Ponting *et al.*, 1972). On the other hand, the 70°Bx-CA treatment showed the minimum firmness (37.22 gf) and hardness (94.62 N) values, compared to other samples, suggesting that a softer texture of osmo-dried Ma-kiang could be achieved with this osmotic solution. These samples can be preserved their shape and form of dried product due to the infused sugar in their structures. In this study, as sucrose concentration increased, the firmness and hardness of the osmo-dried samples decreased. Similar results were found in red pitayas (Haj Najaf *et al.*, 2014), strawberries (Castelló *et al.*, 2010) and apples (Lewicki and Lukaszuk, 2000).

The MC of osmo-dried Ma-kiang decreased with increasing concentration of sucrose. The osmo-dried Ma-kiang treated with sucrose solution 70°Bx had lower MC values than the fruit infused in the sucrose solution 60°Bx ( $p \leq 0.05$ ). This was probably because of the rate of water removal of the osmo-dried sample was strongly dependent on the concentration of osmotic solution (Chandra and Kumari, 2015). Therefore, the reduction of MC value would be expected with the increase in the osmotic syrup concentration. This result was in good agreement with previous works on osmo-dried banana slices (Jalali *et al.*, 2008). In addition, the lower pH values were found in the 60°Bx-CA and 70°Bx-CA treatments. The pH value of the osmo-dried fruit soaked in the sucrose solution combined with CA was lower than those samples soaked in the sucrose solution combined with CaCl<sub>2</sub> ( $p \leq 0.05$ ). This was due to differences in the types of osmotic agents.

It is well known that ascorbic acid is one of the most important bioactive compound which act as antioxidant in many fruits (Denardin *et al.*, 2015). They are usually found rich in the berry fruits. The ascorbic acid content varied from 1.13 to 1.21 mg/100g DW, but no statistically significant differences were observed ( $p>0.05$ ). This result did not correspond with Zhao *et al.* (2014) who reported that vitamin C content of osmo-frozen mangoes was reduced by increased osmotic concentration. This was attributed to the difference in the tissue texture between Ma-kiang and mango fruits. Berries, including Ma-kiang, have a tough skin, while other fruits have a softer texture; thus, this might be the reason why Ma-kiang fruit was less to leak vitamin C than mango pulp.

Results obtained from the sensory evaluation showed that the osmo-dried Ma-kiang were found to be a desirable dried fruit for assessors liking. Samples pre-osmosed in 70°Bx sucrose solution which is the higher sucrose concentration have been similarly reported by Falade *et al.* (2003), that they found the high acceptability in osmo-dried cashew apples when treated with the higher concentration of sucrose solution. According to the results, 70°Bx-CA treatment was recommended for processing osmo-dried Ma-kiang since it has better physical and chemical properties along with the highest score of overall liking in sensory quality.

## Conclusion

The results of this study clearly showed that all osmotic solutions may be used as pre-treatment of dried Ma-kiang fruit. Indeed, the sample prepared with the 70°Bx-CA solution resulted in better quality than the other treatments. The result of the sensory evaluation also revealed that the Ma-kiang infused with the 70°Bx-CA solution had the highest overall liking score. The quality of the osmo-dehydrated Ma-kiang is related not only to the physiochemical and organoleptic properties but also include some phytochemical properties which possibility are sensitive to processing conditions, particularly dehydration treatment. Thus, the effect of drying on bioactive compounds, such as anthocyanin, flavonoid, phenolic acid, and antioxidant activity of osmo-dried Ma-kiang will be examined in the next study.

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