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RESEARCH ARTICLE

Effects of Hot Air and Freeze Drying Methods on Physicochemical Properties of Citrus 'Hallabong' Powders

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Abstract Citrus 'Hallabong' powders were prepared using hot air and freeze drying methods, and their physicochemical and flow properties were measured. The yields of the powders from whole fruit and pressed cake by hot air drying method were 17.5 and 22.0%, while those by freeze drying method were 20.0 and 22.5%, respectively. Vitamin C was high in freeze-dried whole fruit powders (220.8-364.7 mg/100 g) compared with those in hot air-dried ones (80.1-114.6 mg/100 g). Browning index of freeze-dried powders was significantly lower than those of hot air-dried ones. Bulk densities, compaction densities, and Hausner ratios of the powders were significantly higher in freeze drying method compared with hot air drying method. Water solubilities and hygroscopicities of freeze-dried powders were higher than those of hot air-dried ones. In conclusion, 'Hallabong' powders can be made using freeze drying method with high quality in terms of vitamin C content, color, and water solubility.

Keywords: citrus 'Hallabong' powder, drying method, physicochemical property, flow property

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Introduction

Citrus 'Hallabong' is a hybrid between Kiyomi (*Citrus kiyomi*) and ponkan (*C. reticulata*), developed in Japan in 1972. Once 'Hallabong' was introduced in Korea in the early 1990s, it was called as a name of 'Dekopon' or 'Shiranuhi'. After then, 'Dekopon' was called 'Hallabong' named after Hallasan the mountain located in Jeju-do, where it is primarily grown since 1998 (1,2).

'Hallabong' is distinctive due to its sweet taste, large size, and the large protruding bump on the top of the fruit. Cultivation area and production of 'Hallabong' have been rapidly increased from 134 ha and 841 ton in 1998 to 1,188 ha and 22,199 ton in 2008, respectively because the consumer prefers its taste and flavor (2). 'Hallabong' is second to 'Satsuma' mandarin in production in Jeju. Overproduction of 'Hallabong' may cause the drop in the price of the fresh fruit. So, we need to find the method of processing the overproduced fruits.

There have been several studies about 'Hallabong' such as physicochemical properties for volatile flavor properties (1), quality standardization (2), and quality change during storage (3). However, the processing of 'Hallabong' has not been reported.

One of the processing methods to make food sub-materials is the drying for making a powder. The major factor to choose the drying method is the quality of dehydrated product. Air drying is the oldest process used to preserve foods. Advantage of air drying can extend a shelf life of a product as a result of the drying of foodstuffs by high temperature at a low expense. However, disadvantage is to cause the quality deterioration than that of the original foodstuff. Freeze drying is the process of removing the moisture by sublimation of a frozen product. Advantage of freeze drying can prevent the quality deterioration and microbiological reactions. And it can maintain the structure

and the shape of the original product by minimizing the volume reduction. Despite of these advantages, freeze drying is the most expensive process for manufacturing a dried product (4,5).

Food powders can be made after the drying process. In order to produce food powders, there is a need for information about the physical properties of powders because they affect their behavior during handling and processing. The changes in the particle shapes can occur during handling or storage as a result of moisture absorption, chemical reactions or mechanical attrition (6-8).

The objectives of this study were to prepare 'Hallabong' powders with whole fruit and pressed cake by hot air and freeze drying methods, and to measure their physicochemical and flow properties.

Materials and Methods

Materials Citrus 'Hallabong' was purchased from the local market in Jeju, Korea. It was stored at 4°C until used.

Preparation of 'Hallabong' powders 'Hallabong' powders were prepared by 2 different drying methods, hot air and freeze drying. 'Hallabong' was washed and cleaned. Then pressed cake and juice were obtained by the pulverization-screw press type juice extractor (#HCM-12,500; Hansung Pulverizing Machine Co., Ltd., Seoul, Korea). Whole fruit and pressed cake were dried by using hot air drying method at 60±1°C for 96 h. They were also frozen by a deep freezer at −70±1°C for 24 h and then dried for 72 h by a freeze dryer (PVTFD101A; Hucom Systems, Gwacheon, Korea).

The powders were made by a chopper (HR1396; Philips Co., Istanbul, Turkey) and sorted according to the particle size as 180-250, 250-425, and 425-850 μm using a standard sieve (Chunggyesanggongsa, Seoul, Korea). The powder samples were put in a container which was included a lithium chloride saturated solution (relative humidity: 11%) to adjust the same moisture content (9) and stored at the refrigerator (4°C) until used.

Measurement of physicochemical properties of the powders

***Moisture content*:** Moisture content was measured using air oven method at 105°C according to AOAC method (10).

***Soluble solids*:** The powders (5 g) were dissolved in a 45 mL distilled water, and the soluble solid was measured by using a hand-held refractometer (0-32%, Atago, Tokyo, Japan) and multiplied by the dilution ratio.

***Titratable acidity*:** The powders (1 g) was diluted to 100 mL with distilled water in a 250-mL beaker. A 0.5 mL of phenolphthalein indicator was added, and titration was performed with 0.1 N NaOH solution. Titratable acidity was calculated (10).

***Vitamin C*:** Vitamin C was extracted from the powders by the method of Rizzolo *et al.* (11). A portion of 0.5 g of the powders was added to 50 mL of 6% meta-phosphoric solution. The mixture was homogenized and centrifuged at 6,000×*g* at 4°C for 10 min. The supernatant was filtered through a syringe filter and Sep-Pak C18 cartridge. Then, vitamin C was measured by the HPLC system (Waters 2695; Waters Co., Milford, MA, USA) using the method of Albrecht *et al.* (12). The analytical column used was a μBondapak^{TM} NH_2 (3.9×300 mm, 10 μm, Waters Co.). The mobile phase used was 5 mM KH_2PO4 (pH 4.6) and acetonitrile (30:70). The flow rate and the wavelength of the analysis were 1 mL/min and 254 nm, respectively.

***Color and browning index*:** Color was measured using a Chroma meter (CR-300 Series; Minolta Co., Osaka, Japan) (13). The color was expressed using CIE Lab coordinates where L represents the lightness, a the redness or greenness, and b the blueness or yellowness. Total color difference (ΔE) was determined using the following equation:

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

where, the standard color value (L_0 96.98, a_0 0.19, b_0 1.95) was used.

Browning index was measured by the method of Kim and Lee (14). The powders (0.5 g) were suspended with 10 mL distilled water, and then the clear supernatant was obtained after the centrifugation at 5,000×*g* for 10 min. The optical density of the supernatant was measured immediately at 420 nm against distilled water as a blank using a UV-Vis spectrophotometer (MQX200 μQunat; Bio-Tek Instruments, Inc., Winooski, VT, USA).

Measurement of flow properties of the powders Flow properties of the powders such as bulk density, compaction density, and Hausner ratio were measured and calculated by using the method of Peleg (7) and Shin (15).

***Bulk density*:** The bulk density of the powder was calculated from its mass and volume. First, a 100-mL graduated cylinder was placed in the electronic balance (Type AX 200; Shimadzu Co., Tokyo, Japan), and a funnel (75×140 mm) was fixed in the stand above the cylinder. The powders were slowly put in the funnel until the weight of the powders became about 15 g in the cylinder. Then, the volume of the powders was read, and the bulk density of the powders was calculated.

***Compaction density and Hausner ratio*:** The cylinder with the powders was vibrated with Recipro Shaker (RS-1; Jeio Tech Co., Ltd., Seoul, Korea) for 1 min at 300×*g*. After that, the volume of the powders was read. The compaction

density of the powders was calculated from its mass and volume. The Hausner ratio was calculated as the ratio of compaction density and bulk density.

***Water solubility and swelling capacity*:** Water solubility and swelling capacity were measured by the methods of Dubois *et al.* (16) and Leach *et al.* (17). The powders (0.1 g) and distilled water (10 mL) were put into the 50-mL conical tube and mixed evenly. After the conical tube was placed into the water bath at 60°C for 30 min, it was cooled using cold water for 3 min. And then, it was centrifuged for 10 min at 5,000×*g*, and the supernatant was separated by decanting. The swelling capacity was calculated from the weight of precipitate. Meanwhile, the supernatant was dried at 105°C for 3 h, and the water solubility was calculated from the weight of the dried supernatant.

$$\text{Water solubility (\%)} = (B/A) \times 100$$
$$\text{Swelling capacity (g/g)} = C/(A-B)$$

where, A is the weight of the powders (g), B is the weight of the dried supernatant (g), and C is the weight of the precipitate (g).

***Hygroscopicity*:** Hygroscopicity was determined according to the method of Chung *et al.* (18). The powders (0.5 g) were put into a humidified container and placed in an incubator at 20°C. The weight of the powders was measured every hour for 7 h. Hygroscopicity of the powders was calculated as follows:

$$\text{Hygroscopicity (\%)} = [(A-B)/B] \times 100$$

where, A is the weight of the powders (g), and B is initial weight of the powders (g).

***SEM*:** A scanning electron microscope (JSM-6700F field emission gun-SEM; Jeol Ltd., Tokyo, Japan) was used to investigate the tissue characteristics of the powders after drying. The sample surface was coated with a thin layer of gold under high vacuum conditions before taking the micrographs. SEM micrographs were taken at an accelerating voltage of 5 kV and a magnification of ×500 (19).

Statistical analysis Experimental results are expressed as the mean±standard deviation (SD). The experimental data was analyzed by using the statistical package for social scientists (SPSS) version 12.0 (SPSS, Inc., Chicago, IL, USA). Significant difference from the respective controls for each experiment was tested using the Duncan's multiple range test. A $p<0.05$ was considered as statistically significant. All experiments were performed 3 times.

Results and Discussion

Chemical properties of the powders

***Yield of pressed cake, juice, and powders*:** 'Hallabong' (18.2 kg) was squeezed through a pulverization-screw press type juice extractor, and the yields of pressed cake and juice were 6.6 (35.9) and 11.6 kg (64.1%), respectively.

Whole fruit and pressed cake were dried by hot air and freeze drying methods. The yields of the powders from whole fruit and pressed cake by freeze drying method were 20.0 and 22.5%, respectively, while those were 17.5 and 22.0% by hot air drying method, respectively. Our result was similar to the report of Kim *et al.* (2), in that the yield of the powders of whole fruit by freeze drying was 20.0%.

***Titratable acidity, soluble solids, and vitamin C*:** Titratable acidity, soluble solids, and vitamin C in the powders with different drying methods are shown in Table 1. Titratable acidity was high in the powders of whole fruit (4.31-5.12%) compared with those in pressed cake (2.77-3.41%) because whole fruit contained more organic acids in the dried juice. Soluble solids of the powders from whole fruit were 80°Bx, while those of pressed cake were in the range of 55-60°Bx because whole fruit contained more soluble solids in the dried juice.

Vitamin C was high in freeze-dried whole fruit powders (220.8-364.7 mg/100 g) compared with those in hot air-dried ones (80.1-114.6 mg/100 g) because it was decomposed at high temperature in hot air drying method. Vitamin C was in the rage of 62.7-89.5 mg/100 g in freeze-dried pressed cake powders, but not detected in hot air-dried ones. Kayaa *et al*. (20) also reported that the destruction of vitamin C was increased with the increase of drying air temperature.

Meanwhile, vitamin C was decreased with the decrease of the particle size. It was known that as the surface area exposed to air during drying was increased, the loss of vitamin C increased (21).

***Color and browning index*:** The color of the powders with different drying methods is shown in Table 2. *L* values were high in freeze-dried powders compared with those in hot air-dried ones. However, *a* values showed opposite tendency. *b* values were higher in freeze-dried powders than hot air-dried ones. This could be due to the thermal degradation by high temperature in hot air drying. In fruits and vegetables, *L* and *a* values in color can be used for the evaluation of browning degree, and lower *L* and higher *a* values show that they are browning (22-24).

Browning index of freeze-dried powders was significantly lower than those of hot air-dried ones. High browning index of hot air-dried powders can be caused by degradation of color due to high drying temperature.

Flow properties of the powders

***Bulk density*:** The bulk densities of the powders with different drying methods are shown in Table 3. Bulk densities of the powders were significantly higher in hot air drying method compared with freeze drying method. This

Table 1. Titratable acidity, soluble-solids, and vitamin C of 'Hallabong' powders with different drying methods

Powder	Drying method	Particle size (μm)	Titratable acidity (%)	Soluble solids (°Bx)	Vitamin C (mg/100 g d.w.)
Whole fruit	Freeze	180-250	4.49±0.24$^{d1)}$	80	244.0±39.7^{c}
		250-425	4.40±0.12^{d}	80	220.8±36.1^{c}
		425-850	4.31±0.09^{d}	80	364.7±30.6^{d}
	Hot air	180-250	5.12±0.06^{e}	80	80.1±2.9^{ab}
		250-425	4.48±0.07^{d}	80	81.5±1.1^{ab}
		425-850	4.42±0.08^{d}	80	114.6±6.9^{b}
Pressed cake	Freeze	180-250	3.30±0.31^{c}	60	63.3±1.0^{a}
		250-425	2.99±0.04^{ab}	60	62.7±2.8^{a}
		425-850	2.77±0.03^{a}	58	89.5±3.0^{ab}
	Hot air	180-250	3.41±0.07^{c}	58	ND
		250-425	3.17±0.17^{bc}	58	ND
		425-850	3.00±0.13^{ab}	55	ND

$^{1)}$Mean±SD (n=3); $^{a-e)}$Values with different letters in the column are significantly different according to Duncan's multiple range test (p<0.05); ND, not detected

Table 2. Color and browning index of 'Hallabong' powders with different drying methods

Powder	Drying method	Particle size (μm)	Color				Browning index
			L	*a*	*b*	ΔE	
Whole fruit	Freeze	180-250	83.47±0.24$^{f1)}$	2.19±0.16^{bc}	68.71±0.19^{j}	68.1±0.2^{g}	0.363±0.017^{e}
		250-425	75.20±1.33^{d}	3.71±0.76^{d}	66.44±2.50^{i}	68.2±2.1^{g}	0.251±0.008^{c}
		425-850	74.86±2.57^{d}	6.83±0.97^{f}	64.35±0.65^{h}	66.6±0.9^{g}	0.227±0.003^{c}
	Hot air	180-250	78.42±1.61^{e}	3.22±0.98^{cd}	45.95±1.90^{f}	47.9±2.4^{bc}	0.501±0.015^{f}
		250-425	76.22±0.86^{de}	3.93±0.30^{d}	43.66±0.72^{e}	46.7±1.0^{b}	0.477±0.032^{f}
		425-850	68.58±1.26^{c}	6.88±0.69^{f}	40.46±1.23^{d}	48.3±1.8^{bc}	0.383±0.014^{e}
Pressed cake	Freeze	180-250	85.91±1.46^{f}	-1.26±0.15^{a}	49.20±0.28^{g}	48.6±0.6^{bc}	0.163±0.023^{b}
		250-425	83.49±0.24^{f}	1.62±0.25^{b}	51.29±0.37^{g}	51.2±0.4^{e}	0.127±0.005^{a}
		425-850	77.37±1.49^{de}	5.13±1.18^{e}	50.18±1.86^{g}	52.3±2.3	0.152±0.007^{ab}
	Hot air	180-250	70.56±0.43^{c}	7.34±0.18^{f}	36.62±0.07^{c}	44.2±0.3^{a}	0.289±0.013^{d}
		250-425	60.51±1.44^{b}	10.90±0.69^{g}	33.83±0.67^{b}	49.6±0.9^{cd}	0.318±0.038^{d}
		425-850	49.56±1.69^{a}	12.85±0.55^{h}	27.82±1.12^{a}	55.5±0.8^{f}	0.303±0.006^{d}

$^{1)}$Mean±SD (n=3); $^{a-j)}$Values with different letters in the column are significantly different according to Duncan's multiple range test (p<0.05).

Table 3. Flow properties of 'Hallabong' powders with different drying methods

Powder	Drying method	Particle size (μm)	Bulk density (g/mL)	Compaction density (g/mL)	Hausner ratio
Whole fruit	Freeze	180-250	0.382±0.003$^{e1)}$	0.437±0.005^{e}	1.143±0.004^{de}
		250-425	0.360±0.002^{d}	0.408±0.003^{d}	1.134±0.002^{cde}
		425-850	0.341±0.000^{c}	0.385±0.000^{c}	1.128±0.000^{bcde}
	Hot air	180-250	0.645±0.016^{k}	0.728±0.041^{j}	1.129±0.036^{bcde}
		250-425	0.618±0.014^{j}	0.683±0.001^{i}	1.106±0.026^{b}
		425-850	0.578±0.000^{i}	0.601±0.000^{h}	1.040±0.000^{a}
Pressed cake	Freeze	180-250	0.327±0.000^{b}	0.376±0.000^{bc}	1.150±0.000^{e}
		250-425	0.311±0.003^{a}	0.355±0.005^{ab}	1.142±0.002^{de}
		425-850	0.300±0.002^{a}	0.338±0.007^{a}	1.127±0.016^{bcde}
	Hot air	180-250	0.534±0.003^{h}	0.597±0.005^{h}	1.117±0.003^{bcd}
		250-425	0.512±0.007^{g}	0.569±0.010^{g}	1.111±0.004^{bc}
		425-850	0.483±0.003^{f}	0.534±0.004^{f}	1.105±0.002^{b}

$^{1)}$Mean±SD (n=3); $^{a-k)}$Values with different letters in the column are significantly different according to Duncan's multiple range test (p<0.05).

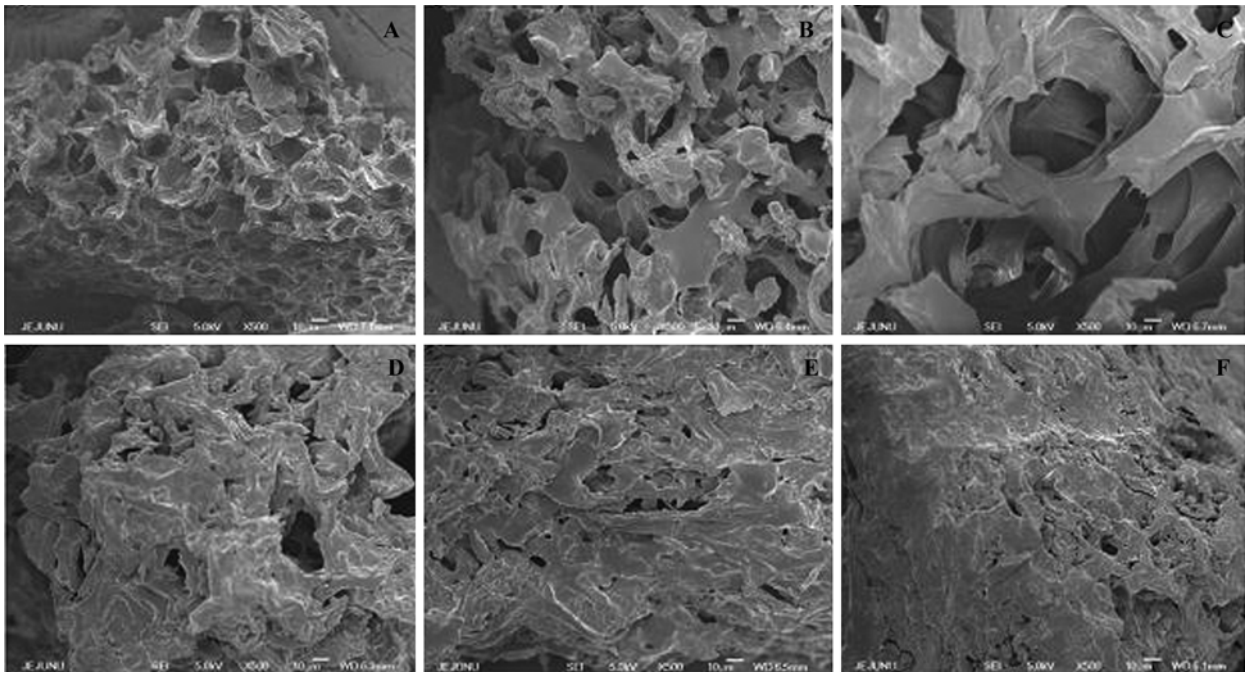

Fig. 1. Scanning electron micrograph of freeze-dried whole fruit powders (A, 180-250; B, 250-425; C, 425-850 μm) and hot air-dried ones (D, 180-250; E, 250-425; F, 425-850 μm).

was due to the difference in the pore size of the powders formed during a drying process. The porosities of hot air-dried powders become smaller (Fig. 1D, 1E, 1F) due to the surface hardening and the shrinkage, while those of freeze-dried powders becomes larger (Fig. 1A, 1B, 1C) because the particle shape of freeze-dried powders maintained the original form by the sublimation of the ice distributed uniformly within the particles by quick freezing (25).

With the same drying method, bulk densities of the powders were increased with the decrease of the particle size due to the decrease of the void volume between the particles in smaller particles (26,27). Peleg (7) reported that bulk density of most food powders was in the range of 0.3-0.8 g/mL. In this study, bulk density of 'Hallabong' powders was also in the range of 0.34-0.64 g/mL.

***Compaction density*:** Table 3 also shows the compaction densities of the powders with different drying methods. Compaction densities showed similar tendency with bulk densities. Compaction densities of the powders in hot air drying method were significantly higher than those of freeze drying method. This result would be that the structure of freeze-dried particles was not easily collapsed during tapping because the powders by freeze drying method maintained the original form. On the other hand, hot air-dried particles were filled with void spaces and could be easily collapsed during tapping (25).

Compaction densities of the powders were increased with the decrease of the particle size. As the particle size decreased, the smaller particle was able to fill the void space during tapping. Therefore, the powders with small particle size showed high compaction density (27).

***Hausner ratio*:** Hausner ratios of the powders with different drying methods are also shown in Table 3. Hausner ratios also showed similar tendency with bulk densities. Hausner ratios of freeze-dried powders were larger than those of hot air-dried ones. This result showed that the volume change by the external impact or vibration in freeze-dried powders was small due to small internal friction force of particles (28).

Hausner ratio was increased with the decrease of the particle size. This was because the decrease of the particle size of the powders increased the cohesiveness by the van der Waals forces and the inter-particle forces (29).

***Water solubility and swelling capacity*:** Water solubility and swelling capacity of the powders with different drying methods are shown in Table 4. Water solubilities of freeze-dried powders were higher than those of hot air-dried ones. In particular, water solubilities of whole fruit powders were significantly different depending on drying methods. Water solubilities of whole fruit powders were significantly higher than those of pressed cake powders. This was because the dried juices in whole fruit contained high soluble solids which have strong hygroscopicity (30). Water solubility of the powders was increased with decreasing the particle size. As the particle size is small, the surface area is large and the water is easy to transfer. Consequently, the water solubility of small particles was higher than that of large particles (9,14,31).

Swelling capacities of hot air-dried powders were higher than those of freeze dried ones. Swelling capacities of

Table 4. Water solubility and swelling capacity of 'Hallabong' powders with different drying methods

Powder	Drying method	Particle size (μm)	Water solubility (%)	Swelling capacity (g/g)
Whole fruit	Freeze	180-250	72.0±0.6$^{g1)}$	7.0±0.5^{a}
		250-425	66.5±3.5^{f}	9.5±0.5^{b}
		425-850	62.3±1.7^{e}	11.6±0.3^{cd}
	Hot air	180-250	49.2±3.6^{d}	10.8±1.0^{bc}
		250-425	47.8±3.4^{d}	12.3±1.0^{cd}
		425-850	45.5±2.5^{d}	13.0±1.2^{de}
Pressed cake	Freeze	180-250	33.2±0.8^{c}	14.3±0.6^{ef}
		250-425	31.6±0.6^{bc}	15.6±1.1^{fg}
		425-850	29.0±1.1^{b}	16.6±1.3^{g}
	Hot air	180-250	31.6±1.2^{bc}	21.0±1.4^{h}
		250-425	30.6±1.8^{bc}	24.1±0.9^{i}
		425-850	25.4±1.2^{a}	26.2±0.9^{j}

$^{1)}$Mean±SD (n=3); $^{a-j)}$Values with different letters in the column are significantly different according to Duncan's multiple range test (p<0.05).

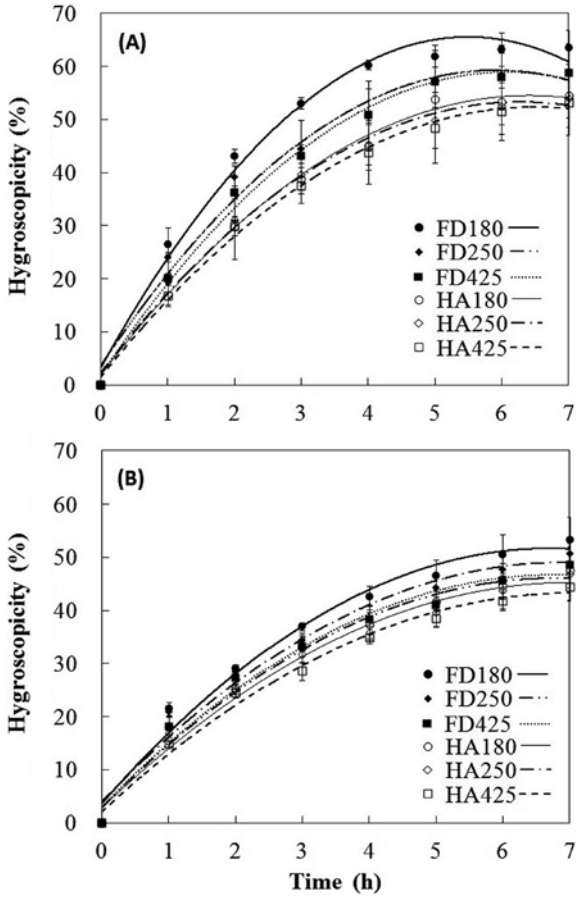

Fig. 2. Hygroscopicity of whole fruit powders (A) and pressed cake powders (B) with different drying methods and time (freeze drying FD180, 180-250; FD250, 250-425; FD425, 425-850 μm; hot air drying HA180, 180-250; HA250, 250-425; HA425, 425-850 μm).

pressed cake powders was significantly higher than those of whole fruit powders. This was because the insoluble components in pressed cake powders would be swelling immediately by absorbing water, while in whole fruit powders, soluble solids would be dissolved in water and then the insoluble components would be swelled by absorbing water (30).

***Hygroscopicity*:** The hygroscopicity of the powders with different drying methods are shown in Fig. 2. The hygroscopicities of freeze-dried powders were higher than those of hot air-dried ones. The time to reach 50% water absorption was 3-4 h in freeze-dried whole fruit powders compared with 5-6 h in hot air-dried ones. Water absorption rate was high in freeze-dried powder compared with hot air-dried ones. It was known that the hygroscopicity was high in freeze-dried products compared with hot air-dried ones because freeze-dried products had high porosity with small pores and can absorb more moisture through it than hot air-dried ones (25,32).

Meanwhile, whole fruit powders showed higher hygroscopicity than pressed cake ones because whole fruit powders contained more soluble solids which can absorb more moisture compared with pressed cake ones (33). Hygroscopicity of the powders was increased with decreasing the particle size because the absorbing surface area/unit weight was large as the decrease in the particle size (34).

In conclusion, 'Hallabong' powders can be made using freeze drying method with high quality of chemical and flow properties in terms of vitamin C content, color, density, and water solubility as food sub-materials.

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