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A THESIS
FOR THE DEGREE OF MASTER OF
ENGINEERING

Physicochemical Properties of Citrus Hallabong
Powders and Granules

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Powders and Granules

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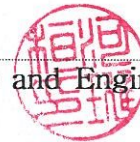
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요 약

제주산 한라봉의 과실과 착즙박을 열풍건조와 동결건조한 후 분말의 물리화학적 특성과 유동성을 측정하였다. 또한 분말을 이용하여 과립을 제조한 후 시판제품과 특성을 비교하였다.

건조 방법에 따른 한라봉 과실의 동결건조 수율은 20.0%, 열풍건조 수율은 17.5% 이었다. 또한 한라봉 착즙박의 동결건조 수율은 22.5%, 열풍건조 수율은 22.0% 이었다.

한라봉 과실의 적정 산도는 동결건조 분말이 4.31~4.49%, 열풍건조 분말이 4.42~5.12% 이었고, 착즙박의 적정 산도는 동결건조 분말이 2.77~3.30%, 열풍건조 분말이 3.00~3.41%를 나타내었다. 한라봉 과실의 당도는 동결건조 분말과 열풍건조 분말이 80 °Brix이었고, 착즙박은 동결건조 분말이 58~60 °Brix, 열풍건조 분말이 55~58 °Brix를 나타내었다. 한라봉 과실의 비타민 C 함량은 동결건조 분말(220.8~364.7 mg/100g)이 열풍건조 분말(80.1~114.6 mg/100g) 보다 높았다. 착즙박의 비타민 C 함량은 동결건조 분말이 62.7~89.5% mg/100g인 반면 열풍건조 분말에서는 검출되지 않았다. 색도는 L값과 b값이 한라봉 동결건조 과실과 착즙박 분말 모두 열풍건조 분말에 비해 높았으나, a값은 열풍건조 분말이 동결건조 분말에 비해 높았다. 갈변도는 한라봉 과실과 착즙박 모두 동결건조 분말이 열풍건조 분말에 비해 유의적으로 낮았다.

분말의 유동적 특성에서 겉보기 밀도, 다짐 밀도와 hausner ratio는 한라봉 과실과 착즙박 분말 모두 열풍건조가 동결건조보다 유의적으로 높았다. 용해성과 흡습성은 한라봉 과실과 착즙박 분말에서 동결건조가 열풍건조보다 높았다. 그러나 팽윤력은 한라봉 과실과 착즙박 분말에서 열풍건조가 동결건조보다 높았다.

물리화학적 특성과 유동적 특성이 우수한 한라봉 동결건조 착즙박 분말(입자크기 180~250 μm)을 이용하여 한라봉 과립을 제조하였다.

한라봉 과립의 배합비율은 동결건조 착즙박을 10%, 유동층건조 주스(10%) 분말을 70%, 첨가물을 6.92%로 고정하였고, 주스와 구연산 첨가량을 달리하여 세가지 종류의 과립 즉, J10C3(주스 10.0%, 구연산 3.08%), J08C4(주스 8.75%, 구연산 4.33%), J07C5(주스 7.50%, 구연산 5.58%)를 제조하였다.

한라봉 과립의 수분 함량은 5.08~5.44%이었고, 시판 제품은 2.39%이었다. 적정 산도는 한라봉 과립이 4.35~5.88%이었고, 시판 제품은 10.3%이었다. pH는 한라봉 과립이 2.54~2.70이었고, 시판 제품은 2.59이었다. 당도는 한라봉 과립이 90~98 °Brix이었고, 시판 제품은 100 °Brix이었다. 당산비는 한라봉 과립이 15.3~22.5이었으며, 시판 제품은 9.7이었다. 비타민 C 함량은 한라봉 과립이 1.22~1.60 g/100g이었고, 시판 제품은 9.31 g/100g이었다.

유동적 특성으로서, 겉보기 밀도는 J10C3, J08C4와 J07C5이 각각 0.541, 0.580, 0.660 g/mL으로서, J10C3는 시판 제품(0.549 g/mL)과 유사하였다. 다짐 밀도는 J10C3, J08C4와 J07C5이 각각 0.561, 0.603, 0.689 g/mL으로서, J08C4는 시판 제품(0.616 g/mL)과 유사하였다. Hausner ratio는 J10C3, J08C4와 J07C5이 각각 0.964, 0.962, 0.957으로 시판 제품(0.892)보다 모두 높았는데, 이로보아 시판제품이 한라봉 과립에 비해 유동성이 유의적으로 우수하였다. 용해성은 J10C3, J08C4와 J07C5이 각각 72.0, 66.7%, 66.6%으로 시판제품(75.6%)보다 낮았다. 팽윤력은 J10C3, J08C4와 J07C5이 각각 3.84, 5.42, 6.40 g/g으로 시판제품(3.47 g/g)보다 모두 높았다. 흡습성은 J10C3이 가장 높았으며, 다음으로 J08C4, 그 다음으로 J07C5 순이었고, 시판제품이 가장 낮았다.

한라봉 과립과 시판 제품의 관능검사 결과 color, sweetness와 overall acceptance는 유의적으로 차이가 없었으나, flavor와 bitterness는 시판 제품이 과립에 비해 선도호가 유의적으로 높았다.

결론적으로 한라봉 동결건조 착즙박과 유동층건조 주스를 이용하면 물리화학적 특성과 유동적 특성이 우수한 분말과 과립을 제조할 수 있을 것으로 기대되며, 비상품 한라봉을 가공함으로써 한라봉 생과의 적정가격을 유지하는데도 기여할 것으로 생각된다.

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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 both whole fruit and pressed cake powders were significantly higher in hot air drying method compared with freeze drying method. Water solubilities and hygroscopicities of freeze-dried whole fruit and pressed cake powders were higher than those of hot air-dried ones. In conclusion, Hallabong powders can be made using freeze drying method with good physicochemical and flow properties.

Three types (J10C3, J08C4, J07C5) of Hallabong granules were prepared with the powders of freeze dried pressed cakes and fluidized bed-air dried juices at different mixing ratios, and their physicochemical properties were compared with the targeted commercial product. The soluble solids of Hallabong granules were slightly lower than that of the commercial product. Hallabong granules were about two times lower in titratable acidity than that of the commercial product. pH of commercial product was in the middle of the granules even though titratable acidity of commercial product was two times

high. Vitamin C in Hallabong granules were about 6 times lower than that of commercial product. Bulk and compaction density of commercial product were in the middle of the Hallabong granules. However, hausner ratios of three granules were higher than that of commercial product. J10C3 was similar to commercial product in water solubility and swelling capacity. The granule similar to commercial product in terms of hygroscopicity was J07C5. J10C3 and J08C4 was not significantly different from the commercial product in color, sweetness, and overall acceptance. In conclusion, Hallabong granules we made were not significantly different compared with commercial product.

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 (Song *et al.*, 2005; Kim *et al.*, 2006).

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 (Kim *et al.*, 2006). 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 way of processing the overproduced fruits.

There have been several studies about Hallabong such as physicochemical properties for volatile flavor properties (Song *et al.*, 2005), quality standardization (Kim *et al.*, 2006), and quality change during storage (Lee *et al.*, 2007b). 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 very 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 (Garau *et al.*, 2006; Ratti, 2001).

Fluidized bed-air drying is a process to transfer heat and materials simultaneously. The air is sent in the fluidized bed-dryer and then the particle is floated by the drag. Because of the advantage to rapidly increase the heat and materials transfer, it uses widely in the food and petrochemical industries (Davidson and Harrison, 1971). Fluidized beds offer advantages such as: (i) the fluidity of the bed facilitating easy handling and transport of solids, (ii) high heat and mass transfer rates, (iii) perfect mixing of material in the bed, and (iv) the possibility of applying other sources of energy such as immersed heating coils, etc (Srinivasakannan and Balasubramanian, 2008). Fluidized bed-air drying is very effective to decrease loss of the nutrients and increase the drying velocity at low temperature. Particularly, it is very effective to dry the powder and it uses at the flour mill and the pharmaceutical industry (Lee, 2006).

Food powders can be made after the drying process. In order to produce the good quality of food powders, there is a need for information about their handling and processing characteristics. The physical properties of powders are important because they affect its behavior during storage, 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. The hygroscopicity can be affected by the type of food, ingredients, shape, and size, etc (Schubert, 1987; Peleg, 1983; Ko *et al.*, 1999).

The objective of this study was to measure the physicochemical and flow properties of Hallabong powders and granules prepared with hot air and freeze drying methods.

Part I

Physicochemical Properties of Citrus Hallabong Powders

1. Materials and methods

1.1. Materials and chemicals

Citrus “Hallabong” was purchased from the local market in Jeju, Korea. It was stored at 4°C until needed. Meta-phosphoric solution, acetonitrile, and monopotassium phosphate were obtained from Sigma Chemical Co. (St. Louis, MO, USA). Hydrochloric acid was obtained from Junsei Chemical Co. (Tokyo, Japan).

1.2. Preparation of Hallabong powders

Hallabong powders were prepared by two different drying methods as freeze and hot air drying as follows. 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., Korea). Whole fruit and pressed cake were dried by using hot air drying method at 60±1°C for 96 hr. They were also frozen by deep freezer at -70±1°C for 24 hr and then dried for 72 hr by freeze dryer (PVTFD101A, Hucom systems, Korea).

The powders were made by a chopper (HR1396, Philips Co., Istanbul, Turkey) and sorted according to the particle size as 180~250 µm, 250~425 µm, 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 (Kim *et al.*, 2007) and stored at the refrigerator (4°C) until used.

1.3. Measurement of chemical properties of the powders

1.3.1. Moisture content

Moisture content was measured using air oven method at 105°C (AOAC, 2000).

1.3.2. 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, Japan) and multiplied by the dilution ratio.

1.3.3. pH and titratable acidity

The pH value of the powders was determined by blending 1 g of the sample with 10 mL distilled water, using the pH meter (SevenEasy, Mettler Toledo, Switzerland) calibrated with standard buffers at pH 4 and 7.

Titratable acidity of the powders was measured as follows (KIFA, 2006). 1 g of the powders was diluted to 100 mL with distilled water in a 250 mL beaker. 0.5 mL of phenolphthalein indicator was added, and titration was performed with 0.1 N NaOH solution. Titratable acidity of the powders was calculated as follows:

Titratable acidity (% , as a citric acid) = $\{(A \times F \times 0.0064)/\text{sample weight (g)}\} \times 100$

A: consumptions (mL) of 0.1 N NaOH solution

F: a factor of 0.1 N NaOH solution

1.3.4. Vitamin C

First of all, vitamin C was extracted from the powders by the modified method of Rizzolo *et al.* (1984) as follows. 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 force at 4°C for 10 min. The supernatant was filtered through a syringe filter and Sep-Pak C18 cartridge.

The content of vitamin C in the sample was measured by the HPLC system (Waters 2695, MA, USA) using the method of Albrecht *et al.* (1990). The analytical column used was a μ BondapakTM NH₂ (3.9 x 300 mm, 10 μ m, Waters, Ireland). The mobile phase used was 5 mM KH₂PO₄ (pH 4.6) and acetonitrile (30:70). The flow rate and the wavelength of the analysis were 1 mL/min and 254 nm.

1.3.5. Color and browning index

Color was measured using a Chromameter (CR-300 Series, Minolta Co., Osaka, Japan) (ASTM Committee, 1975). 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 calculated using the following equation:

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

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

Browning index was measured by the modified method of Kim *et al.* (2009) as follows. 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 force for 10 min. The optical density of the supernatant was measured immediately at

420 nm against the distilled water as a blank using a UV-Vis spectrophotometer (MQX 200, μ Quant, Bio-Tek Instruments, Inc., Korea).

1.4. 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 modified method of Peleg (1983) and Shin (2009).

1.4.1. Bulk density

The bulk density of the powders was calculated from its mass and volume. First, a 100 mL mess cylinder was placed in the electronic balance (Type AX 200, Shimadzu Co., Japan), and a funnel (75 mm x 140 mm) was fixed in the stand above the cylinder as shown in Fig. 1. 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 as follows:

$$\text{Bulk density (g/mL)} = (A/B)$$

A: weight of the powders (g)

B: volume of the powders (mL)

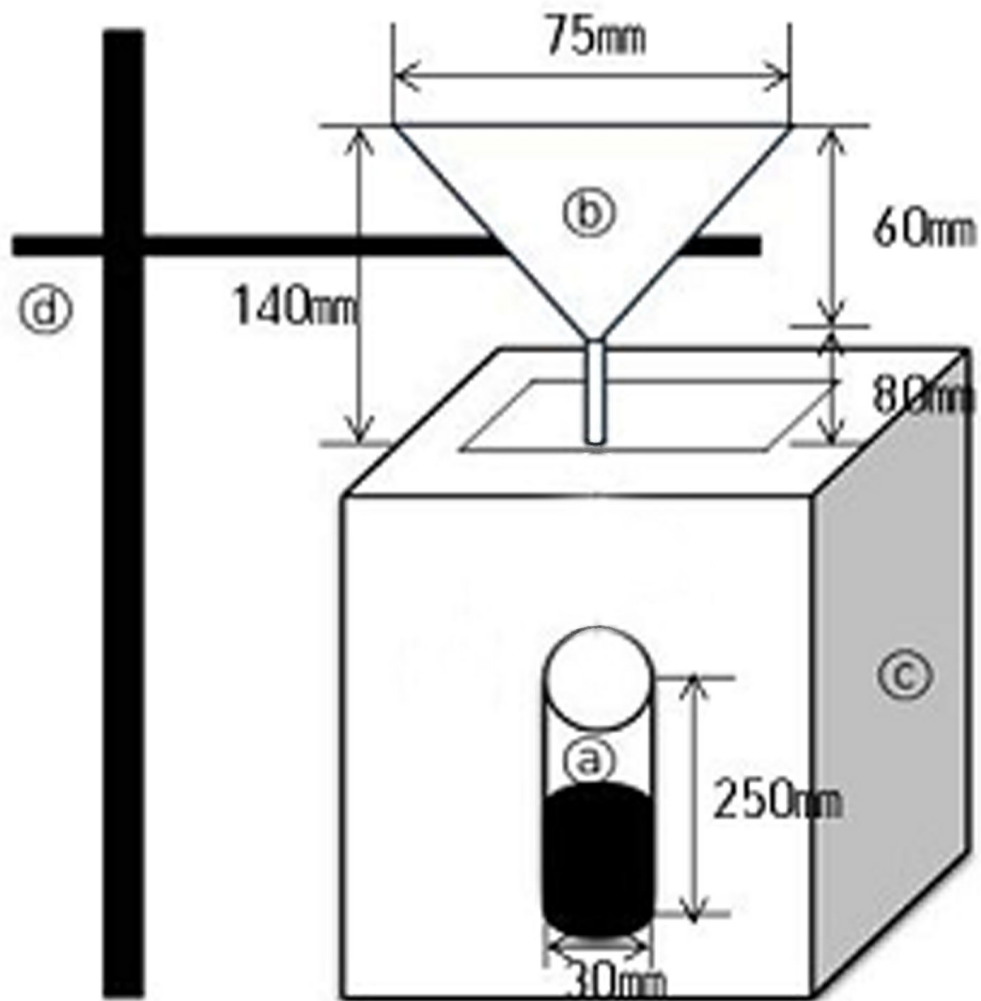


Fig.1. Apparatus for measuring the bulk density.

Ⓐ 100 mL mess cylinder, Ⓑ funnel, Ⓒ Electronic balance, Ⓓ stand

1.4.2. Compaction density

The cylinder with the powders measured the bulk density above was vibrated with Recipro Shaker (RS-1, Jeio Tec, Korea) for 1 min at 300 rpm. After that, the volume of the powders was read. The compaction density of the powders was calculated as follows:

$$\text{Compaction density (g/mL)} = (A/B)$$

A: weight of the powders (g)

B: volume of the powders (mL)

1.4.3. Hausner ratio

The "Hausner ratio" is defined as the ratio between compaction density and bulk density (Hausner, 1967). This "Hausner ratio" could be used as a flow property index of the powders (Gray and Beddow, 1968/9). The Hausner ratio was calculated as follows:

$$\text{Hausner ratio (\%)} = (A/B)$$

A: Bulk density of the powders (g/mL)

B: Compaction density of the powders (g/mL)

1.4.4. Water solubility and swelling capacity

Water solubility and swelling capacity were measured by the modified methods of Dubois *et al.* (1956) and Leach *et al.* (1959). 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 for 30 min at 60°C, it

was cooled using cold water for 3 min. And then, it was centrifuged for 10 min at 5,000 g force, 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 hr, and the water solubility was calculated from the weight of the supernatant.

$$\text{Water solubility (\%)} = (B/A) \times 100$$

A: weight of the powders (g)

B: weight of the dried supernatant (g)

$$\text{Swelling capacity (g/g)} = [C/\{A \times (100 - \% \text{water solubility})\}] \times 100$$

A: weight of the powders (g)

C: weight of the swollen powders (g)

1.4.5. Hygroscopicity

Hygroscopicity was determined according to the modified method of Chung *et al.* (2005). The powders (approximately 0.5 g) were put into a humidified container and placed in an incubator where becomes setting with 20°C. Moisture content of the powders was weighed every hour for 7 hr. Hygroscopicity of the powders was calculated as follows:

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

A: weight of the powders (g)

B: Initial weight of the powders (g)

1.5. Statistical analysis

Experimental results are expressed as the mean \pm 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 (DMRT). A p -value <0.05 was considered statistically significant. All experiments were performed three times.

2. Results and discussion

2.1. Preparation of Hallabong powders

Hallabong was squeezed through a pulverization-screw press type juice extractor. The yields of pressed cake and juice were 35.9% and 64.1%, respectively (Table 1). Korea Food Research Institute (1998) reported that the yield of juice from Satsuma mandarin (*C. unshiu* Marc.) was 70.5% using an experiment juice extractor, and the juice yield of Hallabong was low than that of Satsuma mandarin.

The yields of the powders from whole fruit and pressed cake by freeze-drying method were 20.0% and 22.5%, respectively, while by hot air-drying method those were 17.5% and 22.0%, respectively (Table 2). Our result was similar to the report of Kim *et al.* (2006), saying that the yield of the powders whole fruit by freeze drying was 20.0%.

Table 1. Yield of pressed cake and juice from Hallabong

	Weight (kg)	Yield (%)
Pressed cake	6.6	35.9
Juice	11.6	64.1
Total	18.2	100.0

Table 2. Yields of the powders from whole fruit and pressed cake from Hallabong

Drying method	Hallabong	Weight (kg)		Yield (%)
		Before	After	
Freeze-drying	Whole fruit	5.13	1.03	20.0
	Pressed cake	3.28	0.74	22.5
Hot air-drying	Whole fruit	5.08	0.89	17.5
	Pressed cake	3.27	0.72	22.0

2.2. Chemical properties of the powders

2.2.1. pH, titratable acidity, soluble-solids and vitamin C

pH of the powders was not significantly different regardless of drying methods and particle sizes. pH was in the range of 3.79~3.90.

Titratable acidity was high in the powders of whole fruit (4.3~5.12%) compared with those in pressed cake (2.77~3.41%) because the juices were included in whole fruit which contained more organic acids. However, it was not significantly different with drying methods.

Soluble-solids of the powders from whole fruit were 80 °Brix, while those of pressed cake were in the range of 55~60 °Brix (Table 3). It was not significantly different with drying methods and particle sizes.

Vitamin C was high in freeze-dried whole fruit powders (220.8~364.7 mg/100 g) compared with that 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 range 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.* (2010) also reported that the destruction of vitamin C increased with increasing drying air temperature.

Vitamin C was low with the decrease of the particle size as shown in Table 4. When the surface area which is exposed to air was increased, loss of vitamin C also increased. The loss of vitamin C was increased depending on the rate of the oxygen in the air (Erenturk *et al.*, 2005). Consequently, contents of the vitamin C can be decreased with decrease in particle size.

Table 3. pH, titratable acidity, soluble-solids, and vitamin C of Hallabong powders with different drying methods

Powder	Drying Method	Particle size (μm)	pH	Titratable acidity (%)	Soluble-solids (°Brix)	Vitamin C (mg/100g dry weight)
Whole fruit	Freeze	180~250	3.90±0.02 ^{e1)}	4.49±0.24 ^d	80	244.0±39.7 ^c
		250~425	3.79±0.02 ^a	4.40±0.12 ^d	80	220.8±36.1 ^c
		425~850	3.85±0.00 ^{cd}	4.31±0.09 ^d	80	364.7±30.6 ^d
	Hot air	180~250	3.83±0.02 ^{bc}	5.12±0.06 ^e	80	80.1±2.9 ^{ab}
		250~425	3.84±0.03 ^{cd}	4.48±0.07 ^d	80	81.5±1.1 ^{ab}
		425~850	3.79±0.03 ^{ab}	4.42±0.08 ^d	80	114.6±6.9 ^b
Pressed cake	Freeze	180~250	3.88±0.02 ^{de}	3.30±0.31 ^c	60	63.3±1.0 ^a
		250~425	3.89±0.03 ^e	2.99±0.04 ^{ab}	60	62.7±2.8 ^a
		425~850	3.91±0.00 ^e	2.77±0.03 ^a	58	89.5±3.0 ^{ab}
	Hot air	180~250	3.82±0.02 ^{abc}	3.41±0.07 ^c	58	ND ²⁾
		250~425	3.82±0.03 ^{abc}	3.17±0.17 ^{bc}	58	ND
		425~850	3.85±0.02 ^{cd}	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).

²⁾ ND: not detected

2.2.2 Color and browning index

Table 4 shows the color of the powders. L values were high in the freeze-dried powders compared with those in hot air-dried ones in both of whole fruit and pressed cake. However, a values showed opposite tendency. b values were higher in freeze-dried powders than hot air-dried ones. This can be happen 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 (Castaner *et al.*, 1999; Chung *et al.*, 2009; Wang *et al.*, 2010).

Browning index of freeze-dried powders was significantly lower than those of hot air-dried ones as shown in Table 4. High browning index of hot air-dried powders can be caused by degradation of color due to high drying temperature.

Table 4. Color and browning index of Hallabong powders with different drying methods

Powder	Drying Method	Particle size (μm)	Color				Browning Index
			L ²⁾	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 ^{de}	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 ^e	0.152±0.007 ^{ab}
	Hot air	180 ~ 250	70.56±0.43 ^e	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 \pm SD ($n = 3$).

^{a-j)} Values with different letters in the column are significantly different according to Duncan's multiple range test ($p < 0.05$)

2.3. Flow properties of the powders

2.3.1. Bulk density

Table 5 shows the bulk densities of the powders with different drying methods. Bulk densities of both whole fruit and pressed cake powders were significantly higher in hot air drying method compared with freeze drying method. This was due to the difference in the pores of the powders formed during a drying process. It was known that the porosity of hot air-dried powders become smaller due to the surface hardening and the shrinkage, while that of freeze-dried powders becomes larger 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 (Lee and Kim, 2001).

With the same drying method, bulk densities of the powders from whole fruit were significantly higher than those of pressed cake. This was due to the solid composition where the soluble-solids were 80 °Brix and 55~60 °Brix in the whole fruit and pressed cake, respectively (Table 4). Peleg (1983) also reported that the bulk density of the powders was affected by the solid composition.

With the same material and the same drying method, bulk densities were increased with the decrease of the particle size due to the decrease of the void volume between the particles (Lam *et al.*, 2008; Littlefield *et al.*, 2011). Peleg (1983) 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.

2.3.2. Compaction density

Table 5 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 from whole fruit and pressed cake 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 almost the original form. On the other hand, hot air-dried particles were filled with void spaces and could be easily collapsed during tapping (Lee and Kim, 2001).

With the same drying method, compaction densities of whole fruit powders were significantly higher than those of pressed cake powders. This result would also be because the chemical composition of powders is different between whole fruit and pressed cake.

Compaction densities of whole fruit and pressed cake 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 of small particle size showed high compaction density (Littlefield *et al.*, 2011).

2.3.3. Hausner ratio

Table 5 also shows Hausner ratios of the powders with different drying methods. Hausner ratios also showed similar tendency with bulk densities. Hausner ratios of the powders from freeze-dried whole fruit and pressed cake were smaller 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 and in a stable condition. Also, that was due to small internal friction force of particles (Lee *et al.*, 1993).

Hausner ratio was decreased with the decrease of the particle size. This was because the decrease of the particle size of the powders increased the cohesivity by the van der Waals forces and the interparticle forces (Abdullah and Geldart, 1999).

Table 5. 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 ^e	0.437±0.005 ^e	0.875±0.003 ^{ab}
		250~425	0.360±0.002 ^d	0.408±0.003 ^d	0.882±0.002 ^{abc}
		425~850	0.341±0.000 ^c	0.385±0.000 ^c	0.886±0.000 ^{abcd}
	Hot air	180~250	0.645±0.016 ^k	0.728±0.041 ^j	0.886±0.029 ^{abcd}
		250~425	0.618±0.014 ^j	0.683±0.001 ⁱ	0.904±0.021 ^d
		425~850	0.578±0.000 ⁱ	0.601±0.000 ^h	0.962±0.000 ^e
Pressed cake	Freeze	180~250	0.327±0.000 ^b	0.376±0.000 ^{bc}	0.870±0.000 ^a
		250~425	0.311±0.003 ^a	0.355±0.005 ^{ab}	0.876±0.001 ^{ab}
		425~850	0.300±0.002 ^a	0.338±0.007 ^a	0.887±0.013 ^{abcd}
	Hot air	180~250	0.534±0.003 ^h	0.597±0.005 ^h	0.895±0.002 ^{bcd}
		250~425	0.512±0.007 ^g	0.569±0.010 ^g	0.900±0.003 ^{cd}
		425~850	0.483±0.003 ^f	0.534±0.004 ^f	0.905±0.002 ^d

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)

2.3.4. Water solubility and swelling capacity

Table 6 shows water solubility and swelling capacity of the powders with different drying methods. Water solubilities of freeze-dried whole fruit and pressed cake powders were higher than those of hot air-dried ones. Specially, 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 dried juices included in whole fruit contained high soluble solids which have strong the hygroscopicity (Kim *et al.*, 2008).

Water solubility of whole fruit and pressed cake 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 (Kim *et al.*, 2007; Kim and Lee, 2009; Lee *et al.*, 2007a).

Swelling capacities of hot air-dried whole fruit and pressed cake powders were higher than those of freeze dried ones. Swelling capacity was decreased with decreasing the particle size (Kim *et al.*, 2007; Kim and Lee, 2009). This result was similar to the reports of Kim *et al.* (2007) and Kim *et al.* (2009).

Swelling capacity of pressed cake powders was significantly higher than that 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, the soluble solids would be dissolved in water, and then the insoluble components would be swelled by absorbing water (Kim *et al.*, 2008).

Table 6. 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)

2.3.5. Hygroscopicity

Figure 2 and Table. 7 show the hygroscopicity of the powders with different drying methods. The hygroscopicities of freeze-dried whole fruit and pressed cake powders were higher than those of hot air-dried ones. The time to reach 50% water absorption was 3~4 hr in freeze-dried whole fruit powders compared with 5~6 hr in hot air-dried one. Water absorption rate was high in freeze-dried powder compared with hot air-dried ones. However, in pressed cake powders there were no significant differences in water absorption rate with different drying methods. 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 (Lee and Kim, 2001; Tsami *et al.*, 1999).

Meanwhile, whole fruit powders showed higher hygroscopicity than pressed cake ones because whole fruit contained more sugars which can absorb more moisture compared with pressed cake (Kim *et al.*, 1996). Hygroscopicity of the powders was increased with decreasing the particle size because the absorbing surface area per unit weight was large as the decrease in the particle size (Kim, 1992).

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

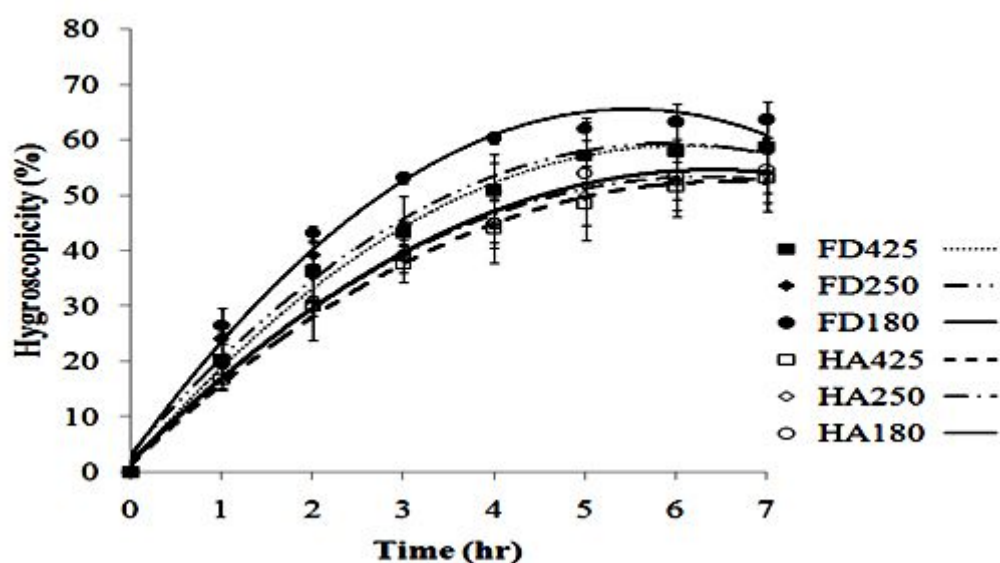
Table 7. Hygroscopicity with elapsed time of Hallabong powders with different drying methods

Powder	Drying Method	Particle size (μm)	Hygroscopicity (%)						
			1 hr	2 hr	3 hr	4 hr	5 hr	6 hr	7 hr
Whole fruit	Freeze	180~250	26.5±3.2 ^{e1)}	43.1±1.3 ^e	53.1±1.0 ^h	60.4±1.0 ^f	62.0±2.1 ^e	63.3±3.2 ^h	63.6±3.2 ^d
		250~425	24.0±0.8 ^{de}	39.2±2.2 ^d	44.5±5.3 ^g	50.7±6.6 ^e	57.1±4.7 ^e	58.5±4.2 ^{gh}	59.1±4.8 ^{cd}
		425~850	20.1±0.9 ^{bc}	36.3±1.3 ^d	43.2±2.1 ^{fg}	50.8±5.1 ^e	57.2±5.1 ^e	58.0±6.1 ^{fgh}	58.7±5.7 ^{cd}
	Hot air	180~250	20.1±5.0 ^{bc}	29.8±6.0 ^c	38.9±2.9 ^{def}	45.0±4.5 ^{de}	53.9±9.2 ^d	53.3±6.0 ^{efg}	54.6±5.9 ^{bc}
		250~425	19.5±0.2 ^{bc}	31.1±0.1 ^c	39.7±2.9 ^{ef}	45.2±3.8 ^{de}	49.9±5.3 ^d	52.6±3.4 ^{defg}	54.0±3.5 ^{bc}
		425~850	16.8±2.0 ^{ab}	30.0±1.8 ^c	37.6±3.4 ^{cde}	43.9±6.1 ^{cd}	48.5±6.7 ^{cd}	51.6±5.4 ^{cdefg}	53.2±6.1 ^{bc}
Pressed cake	Freeze	180~250	21.5±1.2 ^{cd}	29.2±0.5 ^{bc}	37.0±0.8 ^{cde}	42.7±1.9 ^{bcd}	46.7±3.0 ^{bcd}	50.5±3.8 ^{bcdef}	53.4±4.3 ^{bc}
		250~425	20.9±0.7 ^{cd}	28.5±0.2 ^{abc}	34.5±1.2 ^{bcd}	38.4±1.3 ^{abcd}	44.4±2.1 ^{abcd}	47.8±2.5 ^{abcde}	50.7±1.3 ^{abc}
		425~850	18.1±1.8 ^{abc}	27.3±2.5 ^{abc}	33.3±2.0 ^{bc}	38.4±2.8 ^{abcd}	41.1±0.9 ^{ab}	45.7±3.2 ^{abcd}	48.5±4.1 ^{ab}
	Hot air	180~250	18.2±0.5 ^{abc}	25.4±1.2 ^{ab}	33.2±0.8 ^{bc}	37.4±2.7 ^{abc}	41.7±0.9 ^{abc}	44.6±2.5 ^{abc}	47.7±4.2 ^{ab}
		250~425	17.0±0.5 ^{ab}	25.2±1.1 ^{ab}	31.8±1.6 ^{ab}	35.9±2.0 ^{ab}	39.7±2.7 ^{ab}	43.8±3.8 ^{ab}	46.9±4.9 ^{ab}
		425~850	14.8±0.5 ^a	24.5±0.9 ^a	28.6±1.7 ^a	35.0±0.6 ^a	38.5±1.6 ^a	41.9±1.6 ^a	44.5±2.6 ^a

¹⁾ Mean ± SD ($n = 3$).

^{a-h)} Values with different letters in the column are significantly different according to Duncan's multiple range test ($p < 0.05$)

(A) Whole fruit powders



(B) Pressed cake powders

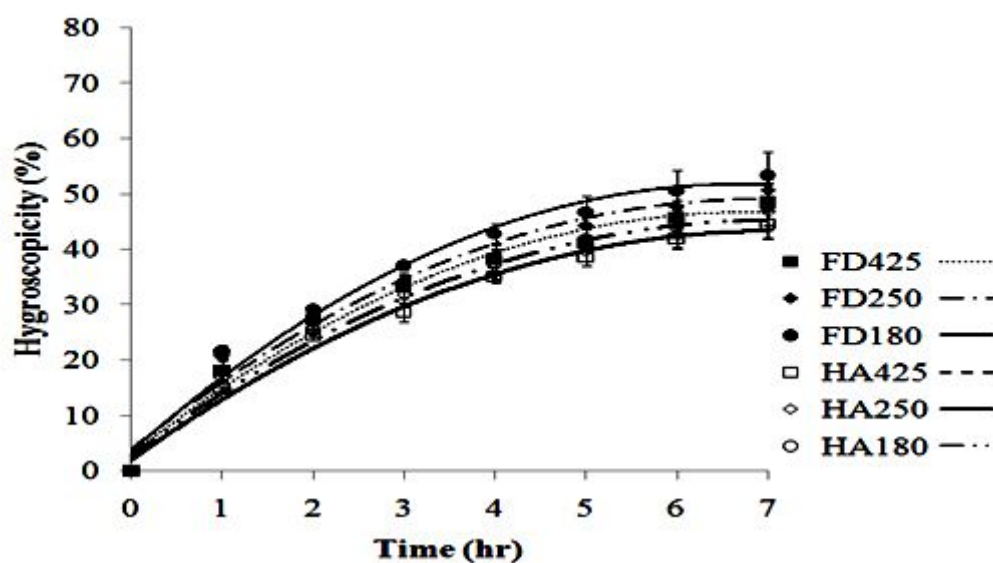


Fig. 2. Hygroscopicity with elapsed time of (A) whole fruit powders and (B) pressed cake powders with different drying methods (freeze drying FD425, 425~850 μm ; FD250, 250~425 μm ; FD180, 180~250 μm hot air drying HA425, 425~850 μm ; HA250, 250~425 μm ; HA180, 180~250 μm).

Part II

Physicochemical Properties of Citrus Hallabong Granules

1. Materials and methods

1.1. Materials and chemicals

Ethanol was obtained from Woori ethanol supplies Co. (Busan, Korea). Anhydrous dextrose was obtained from Daesang Co. (Seoul, Korea). Anhydrous citric acid was obtained from Jungbunzlauer Austria AG. (Vienna, Austria). XYLISORB 700 as xylitol was obtained from Roquette (Merville, France). Collagen peptide-sp was obtained from C&A Biotech (Chungbuk, Korea). L-Ascorbic acid was obtained from HEBEI Welcome Pharmaceutical Co. Ltd (Shijiazhuang, China). Calcium pantothenate, vitamin E-acetate, and riboflavin were obtained from BASF AG (Ludwigshafen, German).

1.2. Preparation of Hallabong granules

Three types of the granules were prepared with the powders of freeze dried pressed cakes and fluidized bed-air dried juices at different mixing ratios as shown in Table 8 and Fig. 3.

10% Fluidized bed-air dried juices were made as follows. 5 g of the juice and 90 g of anhydrous dextrose were mixed. And then 5 g of the juice was again added and dried at 40°C for 8 min in a fluidized bed-air dryer (Jeil Machine Co. LTD., Gyeonggi, Korea) The “fluidized bed air dried juice” sample was passed through a 425~850 μm sieve.

The granules were made as follows. Freeze dried pressed cake and 10%

fluidized bed-air dried juice were put into a beaker and mixed. Then, the additives such as citric acid, xylitol, vitamin C, collagen, calcium pantothenate, and vitamin E were added and were mixed. And then vitamin B₂ dissolved in 300 mL ethanol was added. This mixture was homogenized and hot air-dried for 12 hr at 40°C. Finally, this dried mixture was milled using a chopper (HR1396, Philips Co., Istanbul, Turkey) and sieved using a standard sieve.

Table 8. The mixing ratio for preparation of the granules

Sample name	J10C3	J08C4	J07C5
Materials	Mixing ratio (%)		
Freeze dried pressed cake	10.0	10.0	10.0
10% Fluidized bed-air dried juice	70.0	70.0	70.0
Juice	10.0	8.75	7.50
Citric acid	3.08	4.33	5.58
Other additives ¹⁾	6.92	6.92	6.92
Total	100	100	100

¹⁾ Xylitol (4.62%), vitamin C (2.00%), collagen (0.13%), calcium pantothenate (0.07%), vitamin E (0.07%), vitamin B2 (0.03%)

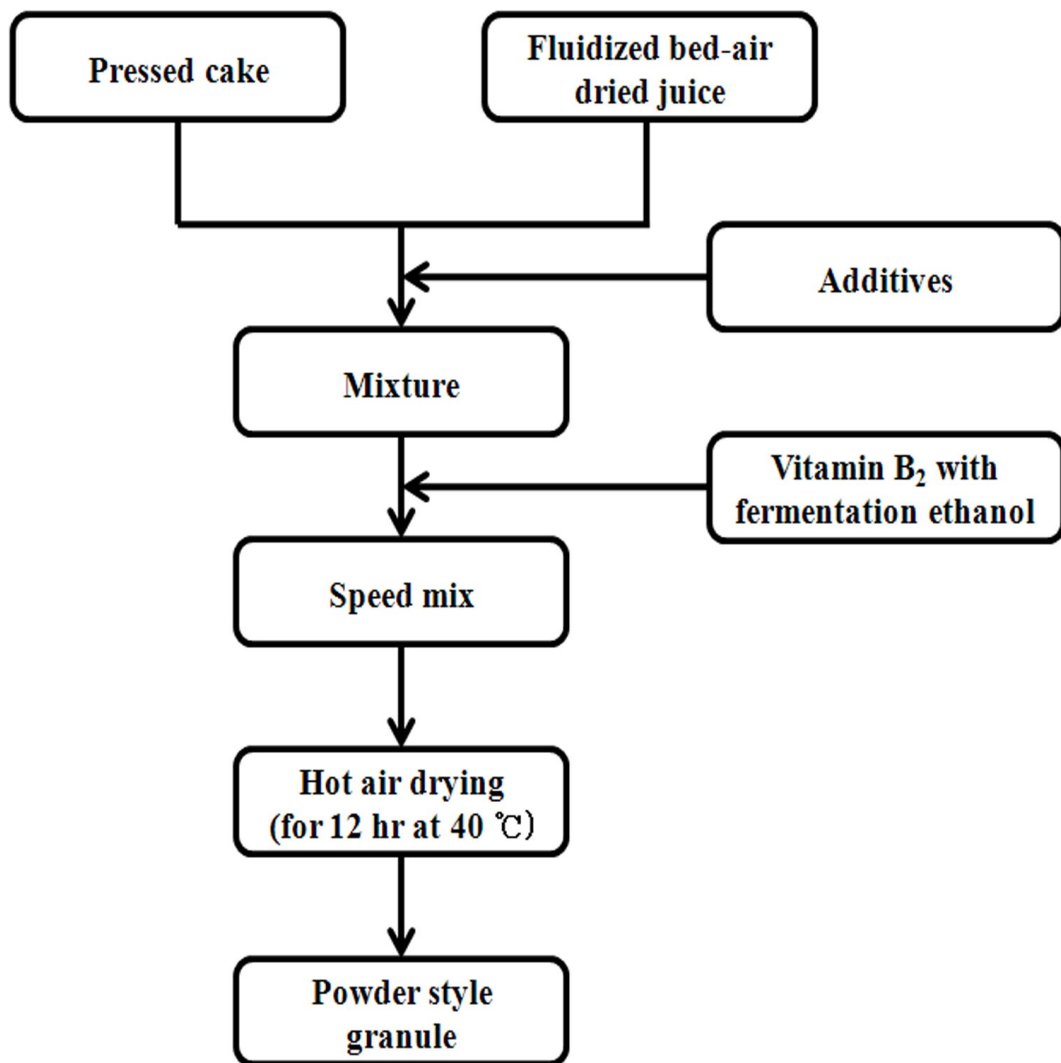


Fig. 3. Preparation method of the granules.

1.3. Measurement of chemical properties of the powders

1.3.1. Moisture content

The method was the same as in Part I .

1.3.2. Soluble-solids

The method was the same as in Part I .

1.3.3. pH and titratable acidity

The method was the same as in Part I .

1.3.4. Vitamin C

The method was the same as in Part I .

1.3.5. Color

The method was the same as in Part I .

1.4. Measurement of flow properties of the granules

1.4.1. Bulk density

The method was the same as in Part I .

1.4.2. Compaction density

The method was the same as in Part I .

1.4.3. Hausner ratio

The method was the same as in Part I .

1.4.4. Water solubility and swelling capacity

The method was the same as in Part I .

1.4.5. Hygroscopicity

The method was the same as in Part I .

1.5. Sensory evaluation of the granules

Sensory test was performed by the modified method of Eyiler and Oztan, 2011. Sensory test was carried out with 21 panelists (13 males and 8 females). Five-point hedonic scale (1 point = dislike extremely and 5 point = like extremely) was used. Samples for sensory test were commercial product, J10C3, and J08C4. Color, flavor, sweetness, bitterness, and overall acceptance were evaluated for each sample.

1.6. Statistical analysis

The method was the same as in Part I

2. Results and discussion

2.1. Physicochemical properties of Hallabong granule

In order to decide the particle size of the granules, commercial product was purchased and its particle size was measured as shown in Table 9. Particle size was in the range of 106~425 and not even. From the experimental result in Part 1, the flow properties were increased with the decrease in particle size. Therefore it was decided that the particle size of the granules in this experiment was above 150 μm .

Table 9. Particle distribution of a commercial product

Particle size (μm)	Weight (g)	Yield (%)
106 \leq	4.71	25.7
125 \sim 106	1.65	9.0
150 \sim 125	1.65	9.0
180 \sim 150	2.90	15.8
212 \sim 180	1.17	6.4
250 \sim 212	1.66	9.1
300 \sim 250	1.40	7.7
425 \sim 300	2.35	12.8
< 425	0.82	4.5
Total	18.31	100.0

2.1.1 Moisture content, titratable acidity, pH, soluble-solids, and vitamin C

Moisture contents were the same in three different granules made in this study, while that of commercial product was two times low as 2.39% (Table 10). Titratable acidity of the granule was increased with the increased of citric acid added as an additive. However, commercial product was about two times higher in titratable acidity than the granules prepared in this study. pH of commercial product was in the middle of the granules even though titratable acidity of commercial product was two times high. Soluble-solids of the granules were in the range of 90~98 °Brix, while that of commercial product was slightly higher than those of the granules. Vitamin C of commercial product was 9.31 g/100 g and 6 times higher than the granules we made.

Table 10. Moisture content, titratable acidity, pH, soluble-solids, and vitamin C of Hallabong granules

Granules	Moisture content (%)	Titratable acidity (%)	pH	Soluble-solids (°Brix)	Soluble solids/acidity	Vitamin C (g/100g dry weight)
J10C3	5.44±0.63 ^b	4.35±0.04 ^a	2.70±0.02 ^c	98 ^{c1)}	22.5±0.2 ^d	1.60±0.01 ^b
J08C4	5.08±0.31 ^b	4.73±0.02 ^b	2.57±0.01 ^a	94 ^b	19.9±0.1 ^c	1.52±0.02 ^b
J07C5	5.13±0.15 ^b	5.88±0.03 ^c	2.54±0.02 ^{ab}	90 ^a	15.3±0.1 ^b	1.22±0.01 ^b
Commercial product	2.39±0.35 ^a	10.32±0.15 ^d	2.59±0.02 ^b	100 ^d	9.7±0.2 ^a	9.31±0.62 ^{a2)}

¹⁾ Mean ± SD (*n* = 3).

²⁾ Particles > 150 μm

^{a-d)} Values with different letters in the column are significantly different according to Duncan's multiple range test (*p*<0.05).

2.1.2. Color

The granules we made were high in L values, low in a values, and high in b values compared with the commercial product (Table 11). This means that the granules we prepared was slightly lighter, more greenness, and slightly yellowness compared with commercial product. 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 (Castaner *et al.*, 1999).

Table 11. Color of Hallabong granules

Granules	Color value			
	L	a	b	ΔE
J10C3	92.75±0.55 ^{d1)}	-6.17±0.19 ^c	45.42±0.84 ^b	44.1±0.9 ^b
J08C4	92.10±0.03 ^{bc}	-6.92±0.14 ^a	47.35±0.51 ^c	46.1±0.5 ^c
J07C5	91.55±0.21 ^{ab}	-7.22±0.12 ^a	48.68±0.58 ^d	47.6±0.6 ^d
Commercial product	90.82±0.59 ^a	-0.17±0.20 ^d	43.86±0.32 ^a	42.4±0.4 ^a

¹⁾ Mean \pm SD ($n = 3$).

^{a-d)} Values with different letters in the column are significantly different according to Duncan's multiple range test ($p < 0.05$).

2.1.3. Flow properties

Bulk and compaction densities of the granules prepared in this study were in the order of $J10C3 < J08C4 < J07C5$ (Table 12). This may be due to the difference in the juice and citric acid added. Those properties of the commercial product were in the middle of the granules. It was known that the bulk properties of granules were determined by the physiochemical properties of the material, the geometry, size and surface characteristics of the individual particles (Peleg, 1983).

Hausner ratios as a flow property index of the granules (Gray and Beddow, 1968/9) were higher than that of the commercial product. This means that the flow properties of the granules were better than that of the commercial product.

2.1.4. Water solubility and swelling capacity

Water solubility of the granules was in the order of $J10C3 > J08C4 = J07C5$ (Table 13). However, swelling capacity was in the reverse order of $J10C3 < J08C4 < J07C5$. This might be caused by soluble components of the juice added. Meanwhile, J10C3 was similar to the commercial product in water solubility and swelling capacity.

2.1.5. Hygroscopicity

Hygroscopicity was in the order of $J10C3 > J08C4 > J07C5$ (Table 14). The granule J07C5 was similar to the commercial product in terms of hygroscopicity. By 1 hr, water absorption of commercial product was about 16.8%. However, J10C3 and J08C4 were about two times higher than the

commercial product. The difference of hygroscopicity among J10C3, J08C4 and J07C5 was due to the difference of carbohydrate content of the juice added as shown in Fig. 4.

Table 12. Flow properties of Hallabong granules

Granules	Bulk density (g/mL)	Compaction density (g/mL)	Hausner ratio
J10C3	0.541±0.005 ^{a1)}	0.561±0.005 ^a	0.964±0.000 ^b
J08C4	0.580±0.002 ^b	0.603±0.002 ^b	0.962±0.000 ^b
J07C5	0.660±0.013 ^c	0.689±0.014 ^c	0.957±0.001 ^b
Commercial product	0.549±0.003 ^a	0.616±0.009 ^b	0.892±0.017 ^a

¹⁾ Mean ± SD ($n = 3$).

^{a-c)} Values with different letters in the column are significantly different according to Duncan's multiple range test ($p < 0.05$).

Table 13. Water solubility and swelling capacity of Hallabong granules

Granules	Water solubility (%)	Swelling capacity (g/g)
J10C3	72.0±2.7 ^{b1)}	3.84±0.48 ^a
J08C4	66.7±0.3 ^a	5.42±0.56 ^b
J07C5	66.6±2.3 ^a	6.40±0.30 ^c
Commercial product	75.6±1.5 ^b	3.47±0.68 ^a

¹⁾ Mean ± SD ($n = 3$).

^{a-c)} Values with different letters in the column are significantly different according to Duncan's multiple range test ($p < 0.05$).

Table 14. Hygroscopicity with elapsed time of Hallabong granules

Granules	Hygroscopicity (%)						
	1 hr	2 hr	3 hr	4 hr	5 hr	6 hr	7 hr
J10C3	30.1±0.9 ^{b1)}	43.4±2.2 ^b	56.1±2.2 ^c	66.7±2.5 ^c	75.2±3.7 ^c	81.7±4.6 ^c	87.5±4.4 ^c
J08C4	27.2±1.4 ^b	39.2±1.9 ^b	47.0±2.2 ^b	58.2±2.5 ^b	66.3±2.4 ^b	74.8±2.6 ^b	80.6±3.2 ^b
J07C5	20.0±4.6 ^a	27.2±3.9 ^a	38.8±2.5 ^a	48.8±4.7 ^a	57.2±3.6 ^a	66.3±1.2 ^a	73.1±1.7 ^a
Commercial product	16.8±3.0 ^a	25.4±3.1 ^a	36.8±5.8 ^a	46.7±2.3 ^a	54.7±2.0 ^a	63.7±2.9 ^a	70.3±4.0 ^a

¹⁾ Mean ± SD ($n = 3$).

^{a-c)} Values with different letters in the column are significantly different according to Duncan's multiple range test ($p < 0.05$).

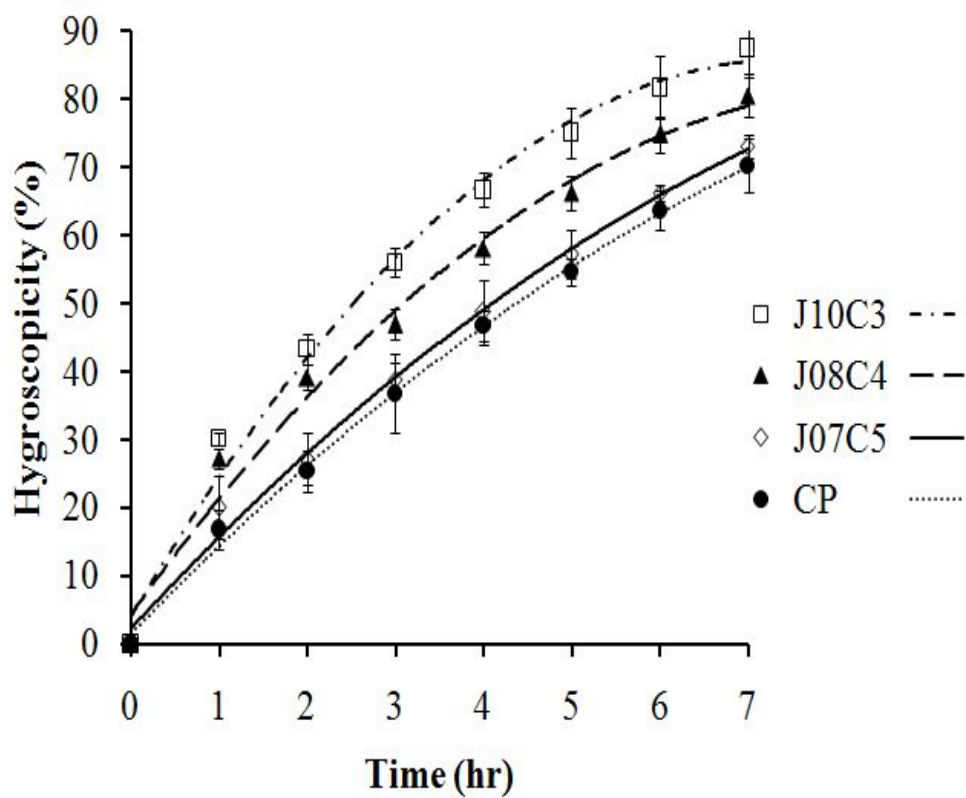


Fig. 4. Hygroscopicity with elapsed time of Hallabog granules (J10C3, J08C4 and J07C5) and Commercial product (CP).

2.2. Sensory evaluation of the granules

Table 15 and Fig. 5 show the results of sensory test for the granules (J10C3, J08C4) and the commercial product. The flavor and bitterness of the commercial product were more preferable than the granules made in this study. However, J10C3 and J08C4 were not significantly different from the commercial product in terms of color, sweetness, and overall acceptance.

Table 15. Sensory attributes of Hallabong granules

Granules	Color	Flavor	Sweetness	Bitterness	Overall acceptance
J10C3	3.57±0.75 ^{a1)}	2.29±0.85 ^a	3.43±0.87 ^a	2.86±1.01 ^a	3.00±1.00 ^a
J08C4	3.52±0.68 ^a	2.38±0.97 ^a	3.38±0.92 ^a	2.81±0.87 ^a	3.00±0.84 ^a
Commercial product	3.33±1.11 ^a	3.29±1.31 ^b	2.86±1.06 ^a	3.95±0.86 ^b	3.52±0.93 ^a

¹⁾ Mean ± SD ($n = 3$).

^{a-b)} Values with different letters in the column are significantly different according to Duncan's multiple range test ($p < 0.05$).

5-point hedonic scale (1 point = dislike extremely and 5 point = like extremely)

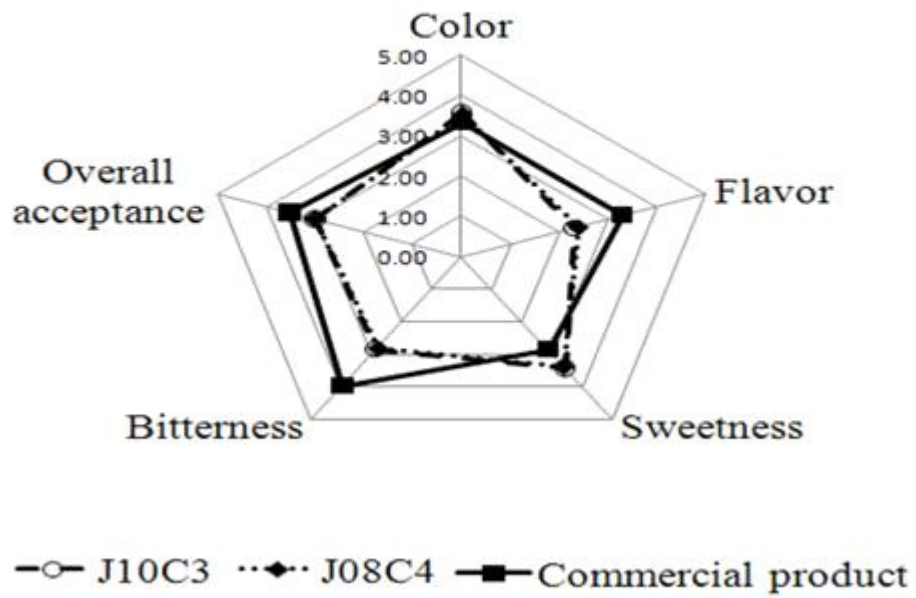


Fig. 5. Sensory properties of J10C3, J08C4, and commercial product.

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