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Physicochemical and Functional Characteristics of Spray-dried Persimmon-prebiotics Powder

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Physicochemical and Functional Characteristics of Spray-dried Persimmon-prebiotics Powder

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Abstract

Persimmons contain health beneficial components such as dietary fibers, vitamins, minerals, and polyphenols, which have antioxidant and antidiabetic effects as well as prevent cardiovascular and other various diseases. However, since most fruits contain 70–80% of moisture content, limitations such as storage, transportation loss could occur. Since most fruits are eaten after peeling, so waste issues, pesticide problems, and seasonality are other limitations. In addition, bioactive compounds such as vitamins and polyphenols could be degraded at high moisture content conditions. Spray drying is widely used for fruit powderization with produced powders having low moisture content and water activity, thus could overcome listed limitations. However, when fruits are powderized, stickiness could occur due to high amount of sugars which lead to low glass transition temperature. Thus, drying aids such as polysaccharides and proteins must be added when spray drying. Resistant maltodextrin (RMD) is a prebiotics dietary fiber that can improve the intestinal environment by becoming a nutrient for probiotics. Meanwhile, RMD is used alone, stickiness could occur, clogged to bag filter, resulting in process stop. Gum arabic, which has film-forming properties, results in stable feed solution, so it is usually used with maltodextrin when fruit powderization. The objective of this research is to examine physicochemical and functional properties of persimmon extract spray-dried powder with using resistant maltodextrin and gum arabic at a ratio of 7:3 (w/w) as drying aids (PE-RMGA). The feed solutions were prepared by adding 30% (w/w) drying aids which were maltodextrin or resistant maltodextrin and gum arabic to 10% (w/w) persimmon extract. The moisture content and water activity were 2.21%, and

0.158, respectively, which means microbiologically safe. Due to small particle size (14.4 μm), flowability and dispersibility might represented poor value. PE-RMGA showed GAB sorption model which is general sight of fruits caused by high sugar contents, and had hygroscopic properties, thereby caution is required when handling, storing of powders. Degree of encapsulation of total phenols and proanthocyanidins were 62% and 49%, respectively, but vitamin C was oxidized, it might be due to extraction process. Like other fruits, sucrose and glucose instead of sucrose made up most sugars, which work in combination with dietary fiber, so using a moderate amount will help control the glycemic index. Comparing sweetness with sucrose using electronic tongue, no significant difference was observed. Based on results, produced PE-RMGA had poor flowability according to HR and CI, and hygroscopic properties, so caution is required in handling and storage area and could be used as a natural sweetener which has antioxidant, antidiabetic, and neuroprotective characteristics if proper amounts are used. In addition, PE-RMGA contains not only components of persimmon but also prebiotics dietary fiber, it will have a more health functional role.

Key words: Persimmon, Resistant maltodextrin, Spray drying, Proximate composition, Powder yield, Particle size, Particle flowability, Powder reconstitution properties, Moisture sorption isotherm, Glass transition temperature, Total phenolics content, Proanthocyanidin content, DPPH/ABTS assay, HPLC, Electronic tongue

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Chapter 1.

Research background

1. Fruits

Fruit refers to edible food that runs on trees or herbaceous plants. It is very similar with vegetable. The food from the annual and grassy plants is a vegetable, and from the perennial and tree species is a fruit. Fruits can be classified by various criteria. Typically, the types can be distinguished by seasons (Table.1.1).

1.1. Composition

Apple, grape, pear, clementine, peach, and persimmon are fruits which are widely consumed in Korea. Fruits consist of about 80-90% water. The rest of the compounds are sugars, lipids and proteins, dietary fibers, phenolic compound and minerals such as vitamin C (Table.1.2). In mineral category, among fruits, persimmon contains high contents of potassium and calcium about 161 mg, and 8 mg per 100 g, respectively (Butt et al., 2015). However, peach contains more potassium and calcium, which represents 228 mg, and 10 mg per 100 g, respectively (Ministry of food and drug safety, 2019) (Table 1.2).

Table.1.1. Seasonal fruits from spring to winter (Foodnuri Information Service, 2021)

Season	**Fruits**
Spring	kiwi, strawberry, etc.
Summer	oriental melon, watermelon, grape, peach, etc.
Autumn	pear, apple, persimmon, etc.
Winter	tangerine, hanrabong, citron, lemon, etc.

Table.1.2. Proximate composition of several fruits popular in Korea (Butt et al., 2015; Chiteva & Wairagu, 2013; Lim & Rabeta, 2013; Ministry of food and drug safety, 2019)

Fruits	Proximate composition (%, w/w)						Mineral contents in ash (mg/100 g)			
	Water	Protein	Lipid	Carbohydrate		Ash	Ca	P	K	Vitamin C
				Sugar	Fiber					
Apple	87.1	0.2	0.4	9.7	2.4	0.2	4	10	39	5
Grape	86.9	0.5	0.1	12.0	0.2	0.3	4	29	108	-
Pear	89.4	0.6	0.1	9.1	0.7	0.1	2	11	171	4
Clementine	88.6	0.5	0.1	10.0	0.5	0.3	10	-	142	36
Peach	87.8	0.6	0.2	8.6	1.7	1.1	10	27	228	8
Persimmon	80.3	0.6	0.2	16.5	2.1	0.3	8	17	161	50

*- not detected

1.2. Health promoting properties

A primary metabolite is a kind of metabolite that is directly involved in normal growth, development, and reproduction. It commonly has a role in a physiological function in the organism. The example of primary metabolites is carbohydrates, proteins, lipids, and vitamins. Conversely, a secondary metabolite is not involved in those processes directly, but usually has an important ecological function (Rehab et al., 2018), and it generally help to resist fungi, bacteria, and plan virus infections (Molyneux et al., 2007). This includes steroids, essential oils, alkaloids, carotenoids, and polyphenols (Harborne et al., 1999; Molyneux et al., 2007).

Fruits are health supplement which play an important role in human health (Wargovich, 2000). For example, as shown in Figure 1.1, antioxidant and antidiabetic properties are dominant effects of fruits. Antioxidants such as vitamin C, carotenoids, and polyphenols prevent or reduce oxidative stress caused by free radicals, thereby protect the cell system from oxidative damage, reduce the risk of chronic diseases, and detoxification/treatment of carcinogens and even changing the path of tumor cells (Adom et al., 2003; Wargovich, 2000). According to Misbah et al, 2013; Rauf et al., 2019, some compounds, such as polyphenolic compound lowers the absorption of glucose, and then shows anti-diabetic effects by suppressing the activation of α-amylases and α-glucosidases (Demir et al., 2019; Taslimi & Gulcin, 2017), or inhibiting aggregation of human islet amyloid polypeptide (Jiao et al. 2013). Besides, especially carotenoids and vitamin C, is associated with reduced incidence and

severity of cataracts (Brown et al., 1999).

However, excessive production of oxidants can cause imbalance, resulting in oxidative stress (Adom et al., 2003), and can damage to polymers such as proteins, lipids, and DNA, resulting in increased risk of developing chronic diseases such as cancers cardiovascular diseases.

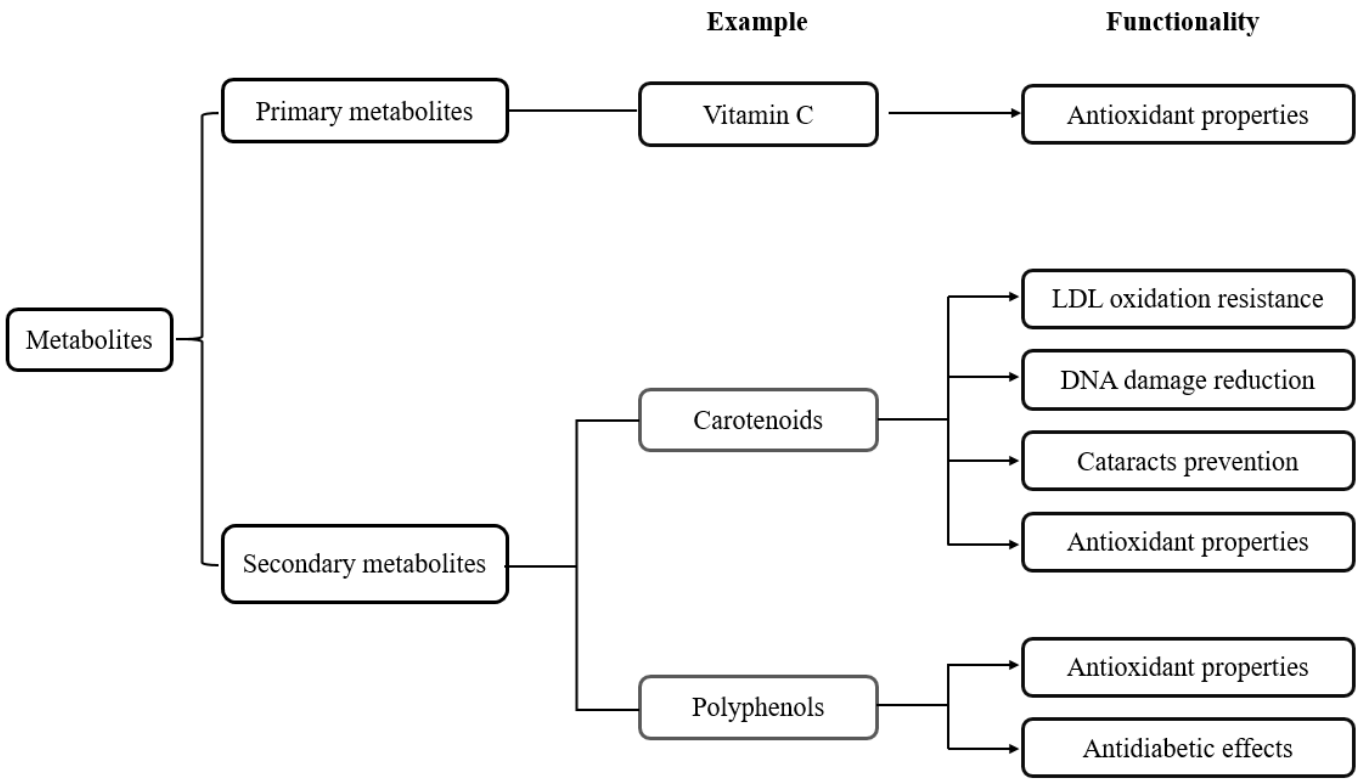


Fig. 1.1. Health beneficial effects of fruits

2. Persimmon

Persimmon is an edible fruit of a number of species of trees in the genus *Diospyros*, and it is one of the most consumed fruits in Korea and is called an Eastern fruit because it is difficult to see in the West. The consumption of persimmon is especially densely packed in Asia which is almost 90% (Esteban-Muñoz et al., 2021). There are more than 400 kinds of persimmons (Butt et al., 2015), including *D. kaki*, *D. oleifera*, *D. lotus*, and *D. virginiana* (Bibi et al., 2007). *D. kaki* is the most popular and promising specie due to health benefitting properties (Butt et al., 2015), and it is classified as sweet persimmon, daebongsi, and bansi (Fig. 1.2). In Korea, persimmons are mainly divided into astringent persimmons and sweet persimmons (Kim et al., 2018) that make the pulp soft. Persimmons have high sugar content than other fruits (Table. 1.2), and higher anti-diabetic effects than other fruits (Butt et al., 2015), resulting in a sweet taste while also showing anti-diabetic effect, and also high antioxidant function (Lee et al., 2012). It is a fruit that has long been used in the private sector for food and medicine, as mentioned in the Donguibogam, an encyclopedia that combines medical books of China and Korea under the orer of Heo Jun's ancestors.

(a) (b) (c)

Fig. 1.2. Appearance of *D. kaki* species. (a) sweet persimmon (*D. kaki* Fuyu), (b) daebong-si (*D. kaki* Hachiya), and (c) bansi (*D. kaki*) (Bburi kitchen, 2018)

2.1. Composition

2.1.1. Proximate composition of persimmon

Among persimmons, *D. kaki*, the oriental persimmon is the most widely cultivated (Morton, 1987). It has about 80% of water, which indicates that it contains less water and has a higher carbohydrate ratio than other fruits, this can be seen that the sweetness of persimmon is relatively higher than those of (Table. 1.2). The content of lipids, proteins, and ash is less than 1%. The major amino acids were proline (74.50 mg/100 g), glutamic acid (23.18 mg/100 g), and aspartic acid (21.89 mg/100 g), and in addition to Bansi, daebongsi, and yeonsi also showed the same order as above (Jeong et al., 2010). In case of fatty acids, palmitic acid, palmitoleic acid, and linolenic acid were identified as major fatty acids (Jeong et al., 2010). Ash is inorganic residue remaining after complete oxidation of organic matter in a food sample, and contains such as minerals. Sugar is about 12-15% and dietary fiber is about 2-3%, and among fibers, pectin is the main. (Butt et al., 2015; Yaqub et al., 2016), to sum up, persimmon consists mainly on water and carbohydrates (Table.1.3).

Cheongdo bansi (*D. kaki* Thunb) is a flat-shaped persimmon produced in Cheongdo area (Kim et al., 2010). Unlike ordinary *Diospyros kaki*, it has low moisture content of 74.5%, and high contents of sugar and fiber which are 20.6%, 3.4%, respectively (Table 1.3).

Table.1.3. Proximate composition of *D. kaki* persimmon (Butt et al., 2015) and *D. kaki* Thunb. cv. Cheongdo bansi (Kim et al., 2010)

Classification	Proximate composition (%, w/w)					
	Water	Protein	Lipid	Carbohydrate		Ash
				Sugar	Fiber	
Persimmon (*D. kaki*)	87.1	0.2	0.4	9.7	2.4	0.2
Cheongdo bansi (*D. kaki* Thunb. cv. Cheongdo bansi	86.9	0.5	0.1	12.0	0.2	0.3

2.1.2. Vitamins

Persimmon contains high content vitamin A (65 mg/100 g), and vitamin C about 50 mg per 100 g (Table 1.2), which is one-half of the recommended daily dose (Casanueva et al., 2005). This content is more than 10 times higher than that of apples. Vitamin C is a type of vitamin that causes scurvy in the human body when it is deficient (Casanueva et al., 2005). As a strong reducing agent, it is one of the essential ingredients for the human body, such as activation of synthetic enzymes in collagen as well as antioxidants. However, most of the iron contained in food is in the form of Fe^{3+}, which can react with vitamin C and cause a fenton reaction (Chen et al., 2017) (Fig 1.3). Vitamin C (ascorbic acid) can reduce ferric ions (Fe^{3+}) to ferrous ions (Fe^{2+}), then the ferrous ion reacts with oxygen, resulting in producing superoxide. Dismutation of superoxide leads to hydrogen peroxide and this reacts with ferrous ions to form hydroxyl radicals (Saha & Roy, 2017).

In summary, vitamin C is used as an antioxidant, but eating foods containing iron may act as an oxidizing agent.

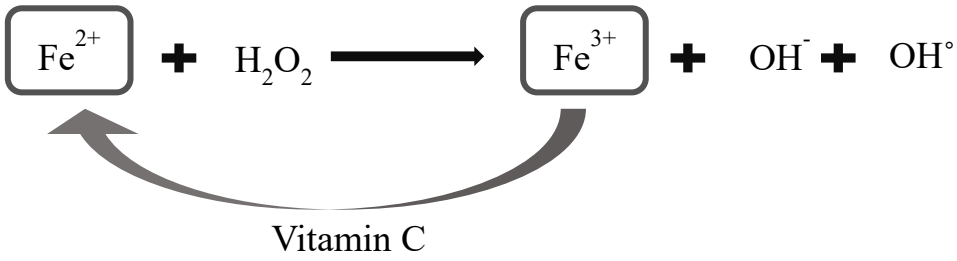


Fig. 1.3. Fenton reaction mediated by vitamin C (Saha & Roy, 2017)

2.1.3. Polyphenols

Persimmon fruit is a great source of polyphenols, which contains about 168 mg gallic acid equivalent (GAE)/100 g dry weight, which is about 4 times higher than apples and 8 times higher than tomatoes (Chen et al., 2008). Polyphenols are representative antioxidants. However, the higher the moisture content, the lower the stability of polyphenolic compound, and the lower the antioxidant properties (Dlamini et al., 2007).

Polyphenols are one of the phytochemicals. These are a group of naturally occurring organic compounds characterized by multiples of phenol units, aromatic alcohol compounds found in plants and structurally diverse (Quideau et al., 2011). Polyphenols are classified as flavonoids, and non-flavonoids (Manach et al., 2004).

(1) Flavonoids

Flavonoids comprises of fifteen carbons, with two aromatic rings connected by a three-carbon bridge (Beecher, 2003) (Fig. 1.4). Major flavonoids include flavones, flavonols, flavanols, flavanones, isoflavones, anthocyanidins, and proanthocyanidins (Manach et al., 2004; Yaqub et al., 2016).

(2) Non-flavonoids

The main non-flavonoids are the C6-C1 phenolic acids, most notably gallic acids, which are the precursor of hydrolysable tannins, caffeic acids, ferulic

acid (3-methoxy-4-hydroxycinammic acid), *p*-coumaric acid, and their conjugated derivatives, and the polyphenolic C6-C2-C6 stillbenes (Yaqub et al., 2016).

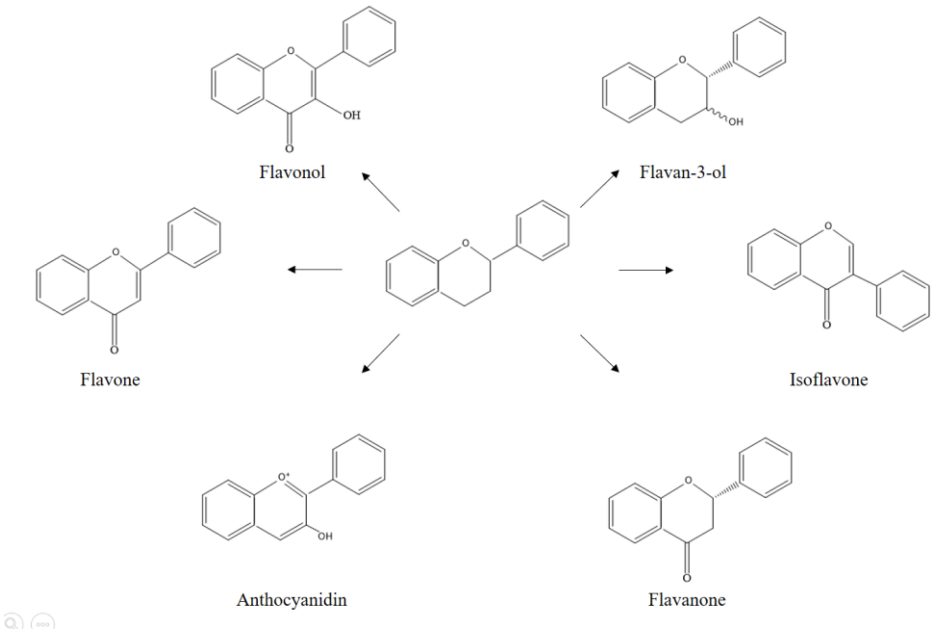
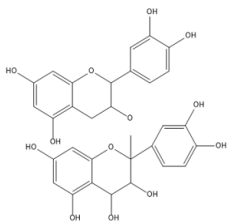
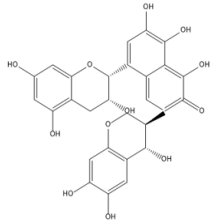
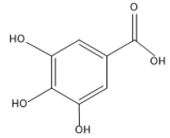
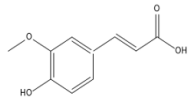
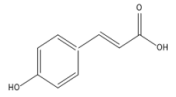
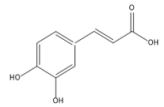


Fig.1.4. Generic structures of the major flavonoids (Crozier et al., 2006)

Table. 1.4. Several polyphenols commonly found in persimmon (Yaqub et al., 2016)

Classification		Structure	Classification		Structure
Flavonoid	Proanthocyanidin		Non-flavonoid	Hydrolysable tannin	
				Phenolic acids Gallic acid	
				Ferulic acid	
				p-Coumaric acid	
				Caffeic acid	

Among polyphenols, as shown in Table 1.4, persimmon contains mainly proanthocyanidins (flavonoids), and hydrolysable tannins, and phenolic acids (gallic acid, ferulic acid, *p*-coumaric acid, and caffeic acid) (non-flavonoids) (Yaqub et al., 2016). These polyphenolic compounds have antioxidant properties, and especially tannins have not only antidiabetic effects but also antioxidant properties (Asgar et al., 2013).

Tannins are astringent, polyphenolic biomolecules that bind to and precipitate proteins and other diverse organic compounds including amino acids and alkaloids (Beart et al., 1985). Tannins also have a role in plant defense systems because they possess severe astringent tastes (Beart et al., 1985). The astringency of persimmons mainly depends on the concentrations of water soluble tannins (Li et al., 2007). Soluble tannins which are soluble in water exhibit astringent taste, and non-flavonoids. However, the fruits become sweet and soft after artificial removal of astringency by CO_2 gas, alcohol vapor, or warm water treatment, or natural ripening. (Takashi et al., 1999). After these treatments or ripening, soluble tannins polymerize into insoluble, condensed tannin resulting in reduced astringency (Vázquez-Gutiérrez et al., 2013). Condensed tannins are polymers formed by the condensation of flavans (flavan-3-ols, or flavan-3,4-diols) (He et al., 2008). They are also called as proanthocyanidins because they yield anthocyanidins when depolymerized under oxidized conditions. Proanthocyanidins are particularly abundant in some fruits and juices (Santos-Buelga & Scalber, 2000). As mentioned,

polyphenolic compounds have antioxidant properties, for example, proanthocyanidins are potent antioxidants and show 50 and 20 times more activity than vitamin E and C, respectively (Kawase et al., 2003). Proanthocyanidins also has been reported to exhibit anti-obesity, anti-cancer, anti-inflammatory activities (Liu & White, 2012), and neuroprotective effects (Li et al., 2018).

2.2. Health functional properties of persimmon

(1) Antioxidant properties

Persimmon has considerable amount of polyphenolic compounds (Gu et al., 2008). These components have antioxidant properties more than vitamin C and vitamin E.

(2) Antidiabetic properties

Proanthocyandins has been reported as an anti-diabetic agent by inhibiting aggregation of human islet amyloid polypeptide (Jiao et al. 2013). In type 2 diabetes, this human islet amyloid peptide aggregates to form amyloid fibrils which are toxic to β-cells Inhibitors, and therefore, proanthocyanidins are potential therapeutic agents for type 2 diabetes (Campos et al., 2020; Marzban et al., 2003).

(3) Neuroprotective effects

Proanthocyanidins also have neuroprotective effects which are related to Alzheimer's disease (Li et al., 2004; Moreira et al., 2005; Stanciu et al., 2020), for example, previous study examined that grape seed proanthocyanidin could prevent Alzheimer's disease (Lian et al., 2016). Accumulation of insoluble amyloid beta (Aβ) induces the aggregation of peptide-forming amyloid fibrils, which have been demonstrated to be neurotoxic *in vitro* and *in vivo* (Li et al., 2004). Somewhat surprisingly, Alzheimer's disease and diabetes share several risk factors, such as, beta-amyloid deposition, higher cholesterol, and degeneration (Ristow, 2004).

(4) Prevention of cardiovascular disease

Substance called scopoletin contained in persimmons is one of the polyphenolic compounds and effective in preventing vascular diseases by acting to strengthen the walls of blood vessels. (Butt et al., 2015)

(5) Boosting immune system

Vitamin C is also high in quantity which not only helps strengthen immunity but also plays an excellent role in eliminating active oxygen (Zhao et al., 1989).

(6) Sweetening effect

Persimmons contain about 15% sugar that is higher than other fruits. The Glycemic index (GI) value is half of sugar (Murakami et al., 2006).

2.3. Cheongdo-Bansi (*Diospyros kaki* Thunb. cv. Cheongdo-Bansi)

Cheongdo-bansi is called bansi because unlike the long-shaped daebong, it is flat and it represents the astringent persimmon in Korea (Lim et al., 2015). Cheongdo bansi has excellent quality with tender skin and high sugar content, and the advantage of being easy to eat and processing because it is the only seedless persimmon in Korea (Kim et al., 2016).

As shown in Table 1.3, The persimmons produced in Cheongdo area are about 5-10% less water than other persimmons and have more sugars and fibers (Kim et al., 2010).

The characteristics of composition and health beneficial properties of persimmon are organized in Table 1.5. Cheongdo-bansi has functionality due to health beneficial components such as vitamins and dietary fibers, especially vitamin C (Hossain et al., 2018)., which is much more than other fruits such as strawberries and citrus fruits. In particular, the form of vitamin C is reductive type so that it is not easily destroyed when exposed to heat, water, or air (Lee et al., 2018). In addition, it is effective in preventing aging, fatigue, and cold. In addition, unlike other persimmons, it has the characteristic of having no seeds, and has the highest DPPH capacity (Rural Development Administration, 2014).

Table. 1.5. Composition and health beneficial effects of Cheongdo bansi (*D. kaki* Thunb. cv. Cheongdo-bansi) (Hossain et al., 2018)

Composition characteristics	**Health functional properties**
Low moisture content	High antioxidant properties
High sugar content	High antidiabetic effects
High dietary fiber content	Neuroprotective effects
High vitamin C content	Fatigue and cold prevention
No seeds (low waste)	

Table. 1.6. Advantages and limitations in fruit consumption and commercialization

Advantages	**Limitations**
Antioxidant properties	Seasonality
Antidiabetic properties	Low shelf life
Boosting immune system	Waste problem
Prevention of diseases	Pesticide

3. Limitations in fruit commercialization

Despite the various advantages, which are written on the Table 1.5 and Table 1.6, the intake of fruits is low. According to WHO, recommendation consumption of fruits and vegetables is more than 400 g, and the Korean Nutrition Society recommends 200 to 600 g of fruits (as of age 19 to 29). On the other hand, the results of the National Health and Nutrition Survey, which was conducted in 2016, showed that the consumption of fruits by Koreans (age 1 or older) was 191 g, less than the recommended amount by the WHO as well as the Korean Nutrition Society. The reasons for the limitation of fruits without any treatments are people have different preferences, fruits are seasonal foods and contains a lot of water, so expiration date is short, have waste problem and people's concern about pesticides (Table 1.6).

3.1. Seasonality

We can't eat fruit whenever we want. Since fruits are seasonal foods, they can only be produced for a certain period of time, thereby limiting their intake. For example, watermelon is hard to eat in the middle of winter because it is produced only in the summer (July-August), and on the contrary, clementines are generally produced from late fall to winter, making it difficult to eat in the summer. Persimmon is also a seasonal fruit produced between October and November, there is a limit to eating whenever we want to (Table 1.1). For this reason, prices are not constant and there is a seasonal variability.

3.2. Storage loss

Most fruits are made up of water (80%) and can be contaminated by microorganisms, making it difficult to maintain their existing state for a long time. If the existing form is not maintained and the taste, aroma, or appearance is changed, which can lead to garbage problems such as falling fruits and blemishes (Nguyen et al., 2020), and the intake of fruits can be low. Low shelf life can be controlled and suppressed by adjusting the temperature, pH, moisture, etc. Refrigerated/freezing system can reduce microbial contamination by lowering the temperature, but it is important to note that not all fruits have a better appearance and aroma just because they are kept at lower temperatures. For example, in the case of bananas, because they are tropical fruits, at refrigerated temperatures, the cell walls are destroyed and the digestive enzymes of the fruit are lost, and the fruit gradually turns black (Nguyen et al., 2003). It is also possible to suppress microorganisms through acidic methods such as pickles and onion pickles (Behera et al., 2020). In addition, microorganisms can be suppressed by controlling the moisture content, and in the case of fruits, it is considered appropriate to control the moisture content due to the high moisture content.

3.3. Transportation loss

Fruits not only have poor shelf life due to high moisture content, which leads to poor storage, but also heavy and bulky characteristics, which makes

transportation difficult. For instance, apple weighs up to 242 g, and the average banana and peach weigh about 126 g, 147 g respectively (FDA, 2017 “Raw fruits poster”). For persimmons, weighs about 168 g. The shape as well as the weight/volume is very important for transportation, as in the case of a larger volume, it is possible to transport less than square shape. Each fruit has a different shape and transportation becomes difficult when thought of with weight.

3.4. Waste issues

When eating fruits, it is common to peel them and remove the seeds from the inside. The discarded peel is about 20% of its original weight, and this discarded part contains more antioxidants such as polyphenols and ascorbic acid (Guo et al., 2003; Li et al., 2006). In addition, defect fruit is also problem, and this could be affected by a number of factors. After processing which is for removal pesticide, the non-edible parts of fruits constitute around 10 to 60% of the total weight, and of which, skin and peel are the main constituents of these wastes, representing more than 50% (Nguyen et al., 2020).

3.5. Pesticide residues

As explained in Section 2.4, the use of pesticides is inevitable to protect fruits. Most people worry about pesticide-based fruits because they can have an

adverse effect on the human body, such as carcinogenicity and neurotoxicity (Alavanja et al., 2004).

4. Fruit powderization

Fruits which deteriorate easily during storage have to be preserved. The storage period of fruits must be prolonged to enable offseason supply and maintain their nutritional value. Seasonality and low shelf life of fruits can be overcome by controlling moisture content. A typical method of controlling moisture content is the drying method. The advantages of a dried powder from liquid forms are lowering storage and transportation costs, and high stability of active substances (Krishnaiah et al., 2014).

4.1. Significances of fruit powderization

Fruits are health supplement because they have bioactive compounds such as vitamins and phytochemicals. The significances of fruit powderization are to overcome seasonality, to lengthen the shelf life and stability, to reduce volume for easy handling, and to overcome waste issues such as using peels with pulp or for using as coloring agents (Fig. 1.5, Table 1.7). Another approach is for recycling waste materials (Table 1.7). The fruit waste which constitutes injured fruit and inedible part of fruit, mostly disposed in the environment causing emission of harmful greenhouse gases (Nguyen et al., 2020). Nowadays the

fruit waste generated is more than 20% over the world, so if inedible part and injured fruit are converted into powders, it would make complete utilization of the bioactive compounds like polyphenols, anthocyanin, pectin etc (Li et al., 2006). In addition, as explained at Section 2.3.1, polyphenols are moisture sensitive, so when the water content is sufficiently high, polymerization occurs, resulting in lower antioxidant performance and loss of conventional structure (Fang & Bhandari, 2011). Encapsulation by using powderization can protect encapsulants from the harsh environments (e.g light, oxygen, pH, heat, etc) as well as to control their release (Gull et al., 2020). Also, these powders are beneficial environmentally, therapeutically and economically (Murakonda & Dwivedi, 2020).

By drying methods, persimmon seasonality can be overcome as well as low shell life. In addition, the proportion of discarded fruits such as leaves and peels can also be reduced. In addition to solving the problem, the persimmon powder containing antioxidant bioactive compounds such as polyphenols can be made and sweetening effect can also be obtained because it has high sugar content (Butt et al., 2015). Insoluble tannin has an anti-diabetic effect (Liu & White, 2012), so persimmon powder can be used as a natural sweetener, a healthy sweetener.

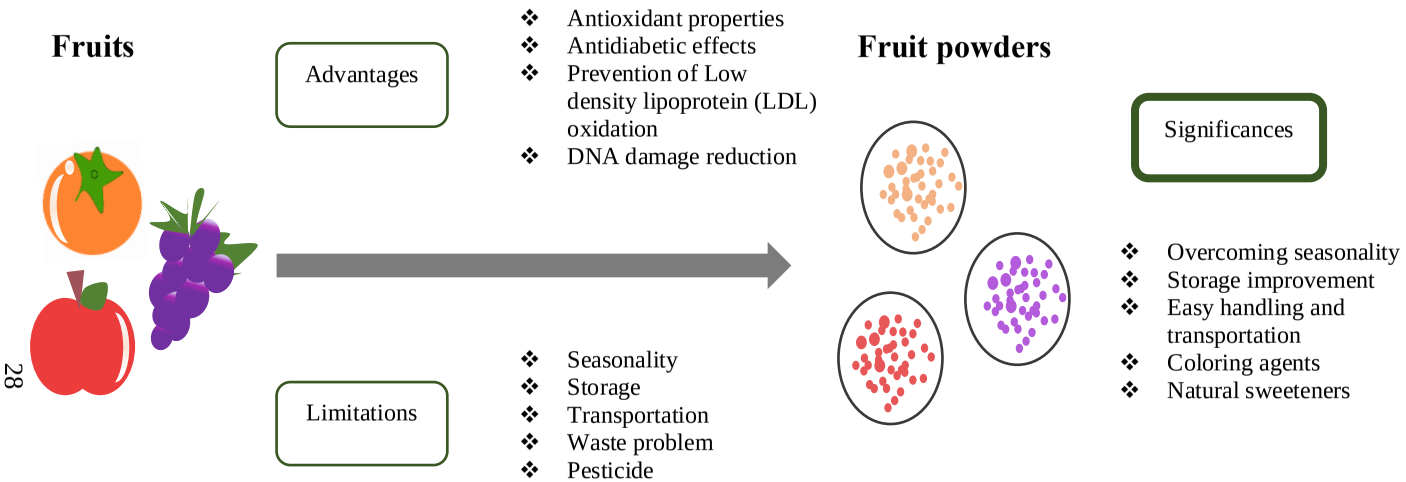


Fig.1.5. Significances of fruit powderization

Table.1.7. Significances of fruit powderization

Fruits	Objectives	Results	Significances	References
Acai	Effect of spray drying conditions (types of drying aids) of acai investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling)	Tonon et al., 2009
Acerola	Effect of spray drying conditions of acerola investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling)	Moreira et al., 2008
Apple	Effect of spray drying conditions of apple investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling)	Sarabandi et al., 2018
Bayberry	Effect of spray drying conditions of bayberry investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling)	Fang et al., 2011
Blackcurrant	1. Effect of spray drying conditions of blackcurrant investigation 2. Natural coloring agent 3. Health supplement (betacyanin)	Produced powders can be used as an excellent natural colorant	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling) 4. Waste materials recycling 5. Coloring agent (anthocyanins)	Archaina et al., 2018

Blueberry	1. Effect of spray drying conditions of blueberry investigation 2. Physical properties of the powder produced	Produced powders were affected by drying conditions	1. Bioactive compound retention 2. Longer shelf life and stability 3. Reduced volume (easy handling) 4. Waste materials (skins, pulp residues, seeds and stems for 15-55%) recycling	Waterhouse et al., 2017
Carambola	1. Convenience of consumption 2. Health supplement 3. Overcoming seasonality	Spray-dried powders have low storage and transportation costs as compared with the raw fruits	1. Bioactive compounds retention 2. Overcoming seasonality 3. Lowstorage and transportation costs as compoared with the raw fruits	Saikia et al., 2014
Cashew apple	Effect of spray drying conditions (drying aid ratio) of cashew apple investigation	Produced powders were affected by drying conditions	1. Easy transportation 2. Longer shelf life	Oliveira et al., 2009
Chokeberry	Effect of spray drying conditions (drying aid ratio) of chokeberry investigation	Produced powders were affected by drying conditions	1. Bioactive compound (anthocyanins) retention 2. Sensitive ingredients protection	Janiszewska-Turak et al., 2019
Cranberry	Effect of spray drying conditions of cranberry investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling)	Michalska-Ciechanowska et al., 2020
Goldenberry	Effect of spray drying conditions (types of drying aids) of goldenberry investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling)	Etzbach et al., 2020

			4. Low storage and transportation costs	
Grape	Effect of spray drying conditions (types of drying aids) of grape investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling)	Kuck et al., 2015 Farrag et al., 2018 Moreno et al., 2016
Jaboticaba peel extract	Effect of spray drying conditions of jaboticaba investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling) 4. Waste materials recycling 5. Coloring agent	Silva et al., 2013
Jamun fruit	1. Effect of spray drying condition (inlet temperature) of jamun juice investigation 2. Physical properties of the powder	1. Spray-dried jamun juice powders have functional benefits 2. Good quality powders with optimum moisture content and water activity can be produced by spray drying	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling)	Santhalakshmv et al., 2015
Jussara pulp	Effect of spray drying conditions (types of drying aids) of jussara investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling)	Santana et al., 2016

Khasi mandarin	1. Convenience of consumption 2. Health supplement 3. Overcoming seasonality	Spray-dried powders have low storage and transportation costs as compared with the raw fruits	1. Bioactive compounds retention 2. Overcoming seasonality 3. Low storage and transportation costs as compared with the raw fruits	Saikia et al., 2014
Mango seed kernel	Effect of spray drying conditions of mulberry investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Byproduct regeneration	Lim et al., 2019
Mulberry	Effect of spray drying conditions of mulberry investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling)	Fazaeli et al., 2012
Orange peel extract	Effect of spray drying conditions of orange investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling)	Shofinita & Langrish, 2014
Passion fruit	Effect of spray drying conditions of passion fruit investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling)	Carrillo-Navas et al., 2011
Pineapple	1. Increasing stability 2. Effect of spray drying conditions (inlet temperature, maltodextrin concentrations) of pineapple investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life (Goula & Adamopoulos, 2010)	Hashib et al., 2015 Jittanit et al., 2010

Pineapple peel	Effect of spray drying conditions of pineapple investigation	Produeced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling) 4. Exotic aroma and pleasant flavor 5. Byproduct (peel and core) regeneration	Lourenco et al., 2020
Pink guava	1. Increasing stability 2. Volume reduction (easy handling) 3. Effect of spray drying conditions of pink guava investigation	1. Produced powders were affected by drying conditions 2. Better quality of final product (enhanced shelf life)	Information on physical properties (moisture content, particle size, yield, color, bulk density, and flowability)	Shishire et al., 2014
Pitaya fruit	1. Natural coloring agent 2. Health supplement (betacyanin)	1. Produced powders can be used as an excellent natural colorant 2. Powders are rich in protein, fat, ash, fiber, and antioxidant besides containing natural coloring compounds betacyaninds	1. Natural coloring agent 2. Bioactive compound retention	Tze et al., 2012
	1. Increasing stability of natural pigments (Celi & Brooks, 2017; Downham & Collins, 2000)	1. Produced powders can be used as an excellent natural colorant 2. Produced powers were affected by drying conditions 3. Improvement the production of encapsulated pigments by natural wastes as wall materials 4. Application: candies, soft drinks, ice cream, and bakery products	1. Natural coloring agent 2. Bioactive compounds retention 3. Overcoming waste problem (wall materials) 4. Mucilages and gelatin usages (ecologically sustainable alternative) (Wang et al., 2012) 5. Potential applications as thickening	Utpott et al., 2020

			agent, stabilizer, or emulsifiers (Dick et al., 2019)	
Tamarillo	1. Health supplement (anthocyanins and carotenoids) 2. Effect of spray drying conditions (types of wall materials) of tamarillo investigation	1. Produced powders were affected by drying conditions 2. Bioactive compounds were effectively microencapsulated using different types of wall materials	Bioactive compounds retention	Ramakrishnan et al., 2018
Tamarind	Effect of spray drying conditions of tamarind investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling)	Bhusari et al., 2014
Sugarcane	Effect of spray drying conditions of sugarcane investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling)	Khuenpet et al., 2016
Watermelon	Effect of spray drying condition (inlet temperature) of watermelon investigation	Produced powders were affected by drying conditions	1. Bioactive compounds retention 2. Longer shelf life and stability 3. Reduced volume (easy handling)	Quek et al., 2007

4.2. Preparation of feed suspension for drying

Bioactive compounds can be protected by controlling the moisture content (Leyva-Corral et al., 2016). Due to the high moisture content in the sample such as fruits or fruit juices, drying is the typical encapsulation and powderization method (Gull et al., 2020). Feeds can be used in the form of juice, but the concentrated feed reduces the moisture content first before drying, so it can be preserved in a more microbial safe form than fruit itself or juice when storing before drying. Therefore, feed suspension, which was extracted first before drying, was used as a sample in anticipation of higher stability.

The feed suspension was made with three key points:

1) To solve the discard peel problem which is described in Section 3.4, the powder will be produced by grinding the persimmon including the peel and the pulp together. Among persimmons, cheongdo-bansi has no seed, so it is more convenient to make.

2) Most pesticides are washed by water, and processes involving heat can increase volatilization, hydrolysis or other chemical degradation and thus reduce residue levels (Łozowicka & Jankowska, 2016). It is possible to overcome the pesticide limits described in Section 3.5, because process of making feed suspension includes water extraction and heat treatment.

3) Persimmon contains a lot of polyphenolic compounds, but especially more in the peels than in pulp (Yaqub et al., 2016). As mentioned in the above

first key point, if peel is used with pulp to make feed suspension, it can not only overcome the waste problem but also make more functional powder.

4.3. Main technologies for fruit powderization

There are many different technologies for powderization and encapsulation where powder is produced directly and additional steps are required (Table 1.8), of which three main technologies (spray drying, freeze drying, and vacuum drying) are highly used due to advantage of generating powder directly.

Table. 1.8. Technologies of powderization (Celli et al., 2015)

Process	Advantages	Disadvantages
Methods from which a powder is directly obtained		
Spray drying	Cost-effective	Fine particle production
	For heat-sensitive compounds	Installation costs
	Fast and continuous	
	Easy to operate and scale up	
Freeze- or vacuum drying	High heat sensitive compounds retention	Refrigerated condition requirement
	High retention of volatiles	Long processing time (20 h)
	Rapid and nearly complete rehydration of the dried product	High energy use
Spray chilling/cooling	Cost effective	Lipophilic coating
	Operated continuously	Expulsion of bioactive during storage
	Easy to operate and scale up	
Methods that require additional step for the production of powder		
Fluid bed coating	Availability of different configurations	Powder type requirement at start step
	Secondary coating that offers an additional barrier	Long process time (2-12 h/batch)
		Requirement of excess of coating material
		Uneven coating and exposure of the core
Emulsification	For hydrophilic and hydrophobic compounds	Thermodynamically unstable product
	Easy to perform and scale up	Additional process for powder production
Coacervation	Easy to perform	Additional process for powder production
	Load is high (40-90%)	High cost
		Instability and leakages during storage
Liposome	Biodegradable and biologically inert	Low encapsulation yield
	Encapsulation of hydrophilic and phobic compounds	High cost
		Drying process requirement

4.3.1. Spray drying

Spray drying is a method in which the biopolymeric solution or suspension of liquid contacts hot air and is evaporated, dried, and turned into powder form (Fig. 1.6). There are three main steps in spray drying process: (1) atomization of the feed solution or suspension into a drying chamber, (2) drying of the droplets when they are formed, and (3) evaporation of the droplets and form solid particles (Shabde & Hoo. 2008). Advantages of spray drying process are ability to control particle size, easy to scale up, continuous production, short time operation and relatively low cost (Sosnik & Seremeta, 2015). The quality of spray dried product depends on spray drying conditions such as inlet temperature, outlet temperature, feed rate, air flow rate and atomizing pressure (Bhusari et al., 2014).

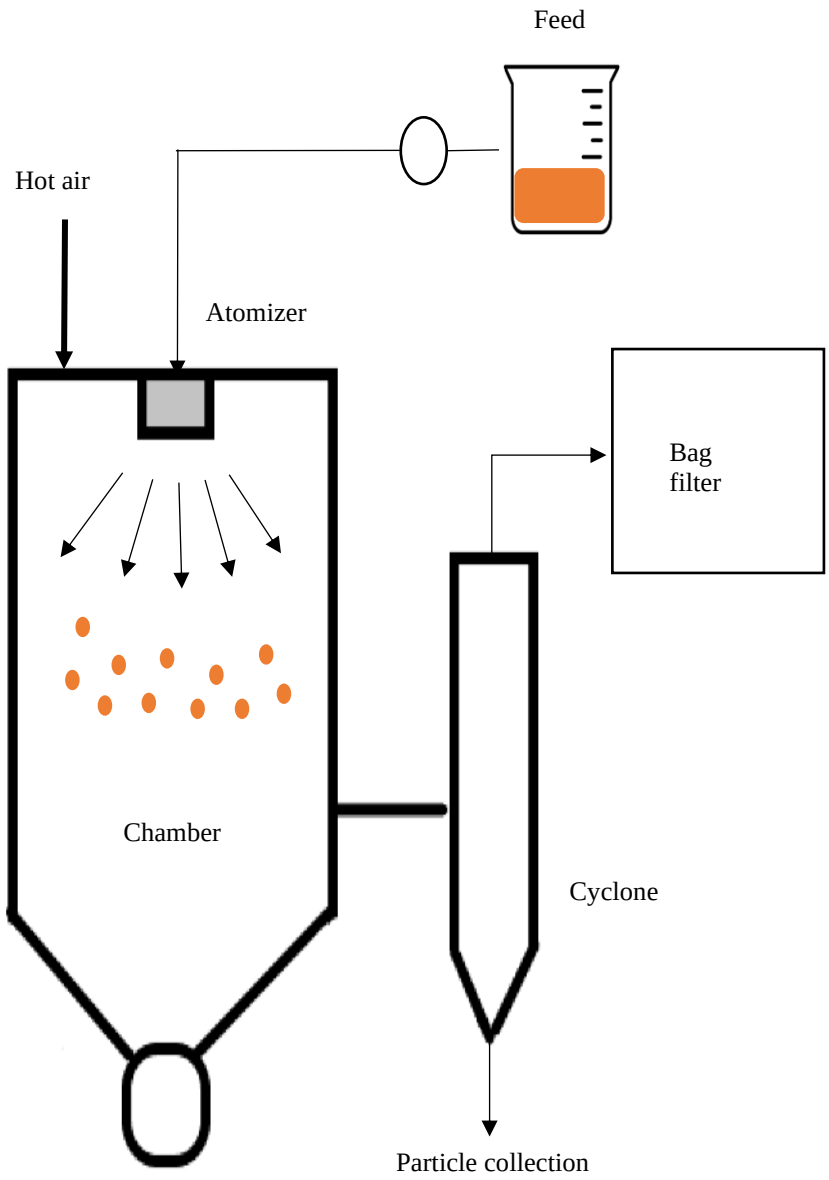


Fig.1.6. Schematic of spray-drying process

4.3.2. Freeze drying

Freeze drying is a low temperature dehydration that has several steps: freezing the product, lowering pressure, and then removing the ice by sublimation (Patel et al., 2010). Firstly, by decreasing the temperature about -40 ℃, and to avoid the liquid phase, it is definitely essential to lower the partial pressure of water, below the triple point pressure. The system slightly increases the temperature to dry or sublimate water or ice and continuously removes unfrozen water from the solute phase by vacuum conditions (Patel et al., 2010). Oxidation and phase transition do not occur due to operation at a low temperature, which is good for bioactive compound protection (Chávarri et al., 2012). It also has the advantages of high yield because there is no thermal damage and no stickiness problem (Chávarri et al., 2012). However, process cost is more expensive than that of the spray drying (Serna-Cock & Vallejo-Castillo, 2013).

4.3.3. Vacuum drying

Vacuum drying is generally used for the drying of substances which are heat sensitive or hygroscopic, and the principle is creating vacuum conditions to decrease the chamber pressure below the vapor pressure of the water, causing it to boil (Parikh et al., 2015). The complete product is placed in the vacuum chamber and subjected to a vacuum, which reduces the boiling point and this vaporises the water, even at low temperatures (Parikh et al., 2015). The pressure (vacuum) reaches all parts of the product, so that the moisture is

completely vaporised and discharged via the vacuum system. This method of drying can be a useful tool for solid products that are heat-sensitive (Parikh et al., 2015). Advantages of vacuum drying method is that reduced energy/operating costs, short cycle/drying times, drying process that protects materials and low required maintenance (Parikh et al., 2015).

4.4. Drying aids for fruit powderization by spray drying

The substrates which are spray dried can be divided into two groups: non-sticky and sticky food (Tan et al., 2011). Non-sticky materials including dairy products and solutions such as maltodextrin, gums, and proteins can be easily made by spray drying and remains free flowing (Tan et al., 2011). In case of sticky foods, when they spray-dried, they get stick to the drying chamber or may get transformed into sticky agglomerates which leads to bag filter and makes operating problems and low product yield (Bhandari & Howes, 2005). Stickiness often occurs when powder is made by spray drying. This phenomenon can be explained by particle-particle stickiness (cohesion) and particle-wall surface stickiness (adhesion) (Papadakis & Bahu, 1992). Stickiness properties (cohesion and adhesion) can coexist during the spray drying of sugar-rich food materials (Bhandari et al., 1997). Cohesion can be formed because of mobile or immobile liquid bridges, mechanical interlocking namely intermolecular and electrostatic forces and solid bridges (Boonyai et al., 2004). Adhesion of powders to the drying chamber can cause loose of powder quality when retained for a long time. The sticky foods are rich in sugar

and acid (Bhandari & Howes, 2005). Sugars and acids have low molar mass, high hygroscopicity, and low glass transition temperature (T_g) which contribute to the stickiness problem (Bhandari et al., 1997). At a temperature of higher than T_g + 20 ℃, particles tend to form a sticky surface, leading to powder stickiness (Bhandari et al., 1997). To reduce stickiness, addition of biopolymers which have high molar mass as drying aids to the feed solution thereby increasing T_g is a common way (Downton et al., 1982). Examples of biopolymer, which are used as a drying aid, are carbohydrates and proteins.

4.4.1. Polysaccharides

Use of polysaccharides such as cellulose, alginate, dextran, chitosan, maltodextrin, carrageenan, gum arabic, xanthan, and inulin can prevent stickiness during spray drying due to high molecular weight, lead to increasing T_g (Huang et al., 2017; Murugesan & Orsat, 2012). Among them, maltodextrin is most commonly used. Maltodextrin consists of D-glucose units connected in chains of variable length (Fig. 1.7). It is classified by dextrose equivalent (DE), which is a measure of the amount of reducing sugars present in a sugar product, expressed as a percentage on a dry basis relative to dextrose, and have a value of between 3 and 20 (Yusraini et al., 2012). Maltodextrin is characterized by its low price, low viscosity, and high solubility that easily soluble in water. (Rajabi et al., 2015). The drying aids normally used in the spray drying of fruits are maltodexrin and gum arabic mainly due to their high solubility and low viscosity, which are important for the process (Fazaeli et al., 2012).

Table. 1.9. Biopolymeric drying aids commonly used in spray drying (Costa et al., 2015; Huang et al., 2017)

Biopolymer	**Examples**
Carbohydrates	Cellulose, modified starch, maltodextrin, alginate, dextran, chitosan, gum arabic, xanthan gum, carrageenan, inulin, resistant maltodextrin, etc..
Proteins	Gluten, casein, albumins

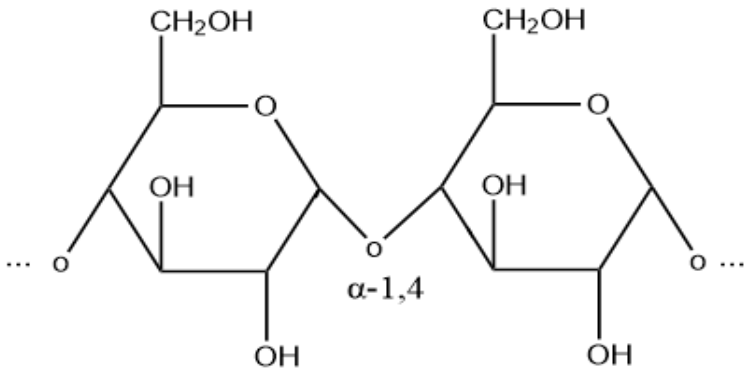


Fig. 1.7. Structure of maltodextrin (MD)

4.4.2. Proteins

Spray drying has the advantage of easy-scale up and rapid and continuous process, but in lab scale, there is a disadvantage of low yield even when using polysaccharides such as maltodextrin as a drying aid (Marante et al., 2020). When using proteins as drying aids, in addition to elevation of molar mass, modifying the surface properties is the alternative to minimize stickiness (Adhikari et al., 2009). When using proteins, it can modify the surface structure or produce semi-crystalline particles, which results reducing the hygroscopicity, degree of caking, the adhesion and increasing of powder production yield (Sarabandi et al., 2018). So proteins are used as a drying aid instead of polysaccharides, the drying yield is higher than those of, even if a small amount is used. Also, bioactive compounds could be protected by film forming properties of proteins, it may also alter the characteristics of the food surface itself, resulting in different characteristics from conventional foods (Adhikari et al., 2009). In addition, in case of whey protein isolate, it can cause allergenic eactions (Villa et al., 2018).

By supplementing the relatively low dry-yield caused by polysaccharide and at the same time adding a small amount of protein that does not deform the food surface as much as possible, (i.e. by using polysaccharide and protein as a dry-aid), the results were also good (Minemoto et al., 2002). Two or more encapsulating materials are mixed to amplify the retention of volatile compounds (Moser et al., 2017).

4.5. Powderization of persimmon extract by spray drying

For fruit powderization using spray drying, maltodextrin is the most commonly used as a drying aid, because it is cheap and can reduce stickiness (Krishnaiah et al., 2014). However, maltodextrin can cause allergic reactions and weight gain if consumed in large quantities (Hofman et al., 2016). There is also a limitation that glycemic index is between 85 and 105, so it can cause diabetes (Hofman et al., 2016). As interest in diabetes and obesity increases, interest in dietary fiber is increasing.

In addition, among fruits persimmon has a substantial amount of dietary fiber, and it is also beneficial to health, helping to prevent obesity and suppress constipation by helping to smooth bowel movements (Aleixandre & Miguel, 2008). Dietary fiber can be also used as a drying aid due to their high molar mass, basically. When using dietary fibers are used as a drying aid, it carries an appreciable amount of antioxidants, mainly polyphenolic compounds (Saura-calixto et al., 2011). Examples of what is primarily used as include inulin (Lourenco et al., 2020; Michalska-Ciechanowska et al., 2020; Paim et al., 2016). Inulin is a group of natural polysaccharides produced by many types of plants most commonly extracted from chicory industry. Both of them are widely used, but when used in large quantities, viscosities can be increased, making it difficult to feed smoothly, resulting in a low drying yield (Tonon et al., 2008). Resistant maltodextrin is an alternative drying aid which has prebiotics effects served as a food for the *bifidobacterium* as well as usage as dietary fiber (Astina & Sapwarobol, 2018). It dissolves well in water among

the dietary fiber, and unlike inulin and gum arabic, the viscosity is low and does not affect when feeding even when used in large quantities (Fastinger et al., 2013).

Table. 1.10. Differences between maltodextrin (MD) and resistant maltodextrin (RMD) (Pai et al., 2015)

	Maltodextrin	**Resistant maltodextrin**
Dextrose equivalent (DE)	3-20	8-12
Glycemic index (GI)	100	10
Structure	D-glucose units with α-1,4 and α-1,6 glycosidic bond	D-glucose units with α-1,4 α-1,6, α-1,2, and α-1,3 glycosidic bond
Characteristics	Good source of energy	Prebiotics dietary fiber

4.5.1. Resistant maltodextrin as a drying aid

Resistant maltodextrin (RMD) is a water soluble polysaccharide similar to maltodextrin (MD), and its DE averaged between 8-12 (Pai et al., 2015). The differences between MD and RMD are described at Table 1.10. MD consists of only (Fig. 1.7) α-1,4 bonds (approximately 95%) and α-1,6 bonds (approximately 5%), while resistant starch has α-1,4 bonds (about 63%), α-1,6 bonds (about 23%), and also α-1,2 and α-1,3 bonds (Fig. 1.8) (Astina & Sapwarobol, 2020). Due to the structure, it is indigestible in the small intestine but is fermented in the colon by colonic bacteria, resulting in short-chain fatty acid production (Astina & Sapwarobol, 2018). As a results it could be defined as a prebiotic dietary fiber, defining "non-digestible dietary fiber material that beneficially affects the host by selectively stimulating the growth and/or activity of one or a limited number of bacteria in the colon, and thus improves host health" (Śliżewska et al., 2012).

RMD is a promising potential drying aid with added health value due to not only its non-digestible prebiotic effects, in which it can reduce the rise in serum glucose after meals (Nomura et al., 1992), reduces built-in fat build-up (Ye et al., 2015; Hashizume et al., 2012), increase satiety when consumed (Ye et al., 2015; Guérin-Deremaux et al., 2011), and increase *bifidobacterium* in the gut (Ohkuma et al., 1990) which is probiotics, but also it is water soluble, low viscosity, and has low glycemic index (10% that of malotdextrin) (Goda et al., 2006).

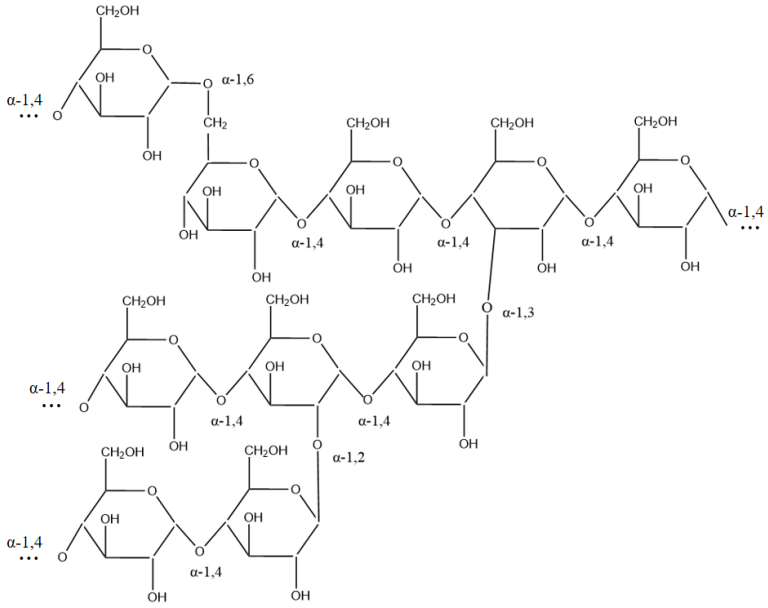


Fig. 1.8. Structure of resistant maltodextrin (RMD)

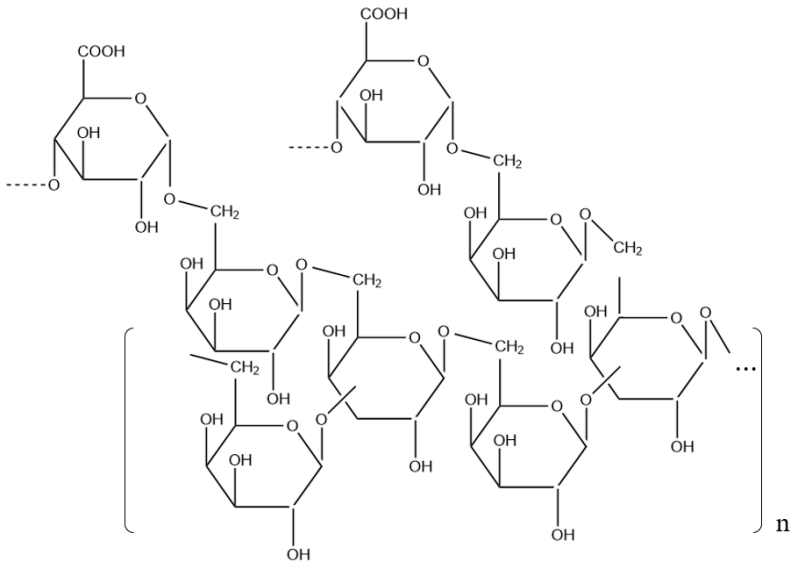


Fig. 1.9. Structure of gum arabic (GA) (Cissé et al., 2020)

4.5.2. Gum arabic as a drying aid

Gum arabic (GA), also known as acacia gum, is a natural gum consisting of the hardened sap of two species of the acacia tree (Gashua et al., 2016). It is a natural plant polysaccharide, and the structure is composed of the L-arabinose, L-rhamnose, and D-glucuronic acid, and 1,3-linked β-D-galactopyranosyl units (Fig. 1.9) (Cissé et al., 2020).

Gum arabic, along with maltodextrin, is also mainly used in spray drying due to their high solubility and low viscosity that are important properties of the spray-dried powder (Tran et al., 2018).

Due to its emulsifying and film forming properties (Turchiuli et al., 2004), the use of gum arabic can make initial emulsions more stable, and protect the active component during spray drying (Turchiuli et al., 2004). In addition, when gum arabic is used for spray drying in combination with maltodextrin and/or other polysaccharides, particles were obtained with controlled size distribution leading to higher powder yield cost effectively (Krishnaiah et al., 2014; Turchiuli et al., 2014), and can protect the active component during spray drying (Turchiuli et al., 2004).

5. Factors influencing the performance of spray drying

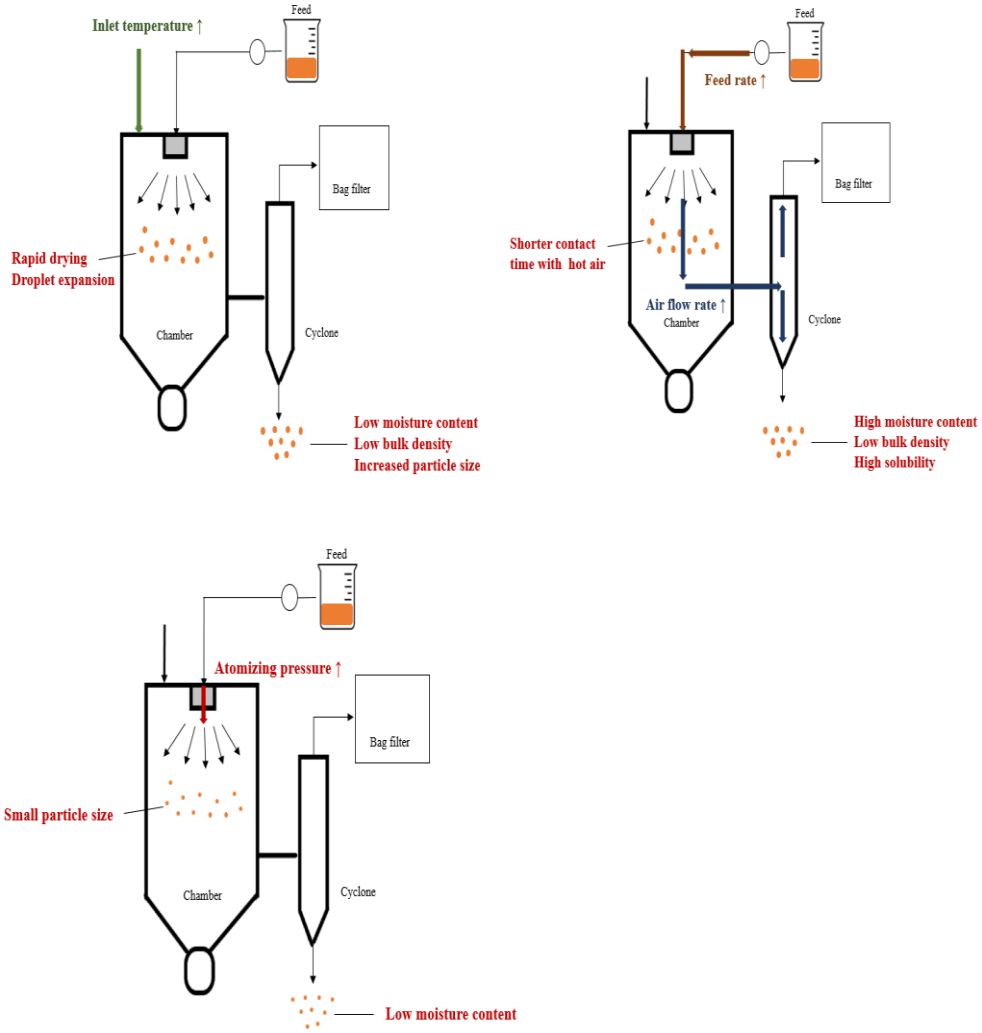


Fig. 1.10. Schematics illustrating the factors influencing the performance of spray drying

5.1. Inlet temperature

Inlet temperature affects diverse parts; moisture content, bulk density, particle size and morphology, drying yields, and stability of bioactive compounds.

When increasing the inlet air temperature, the residual moisture content of orange juice powder was reduced at a constant feed rate, air flow rate, and atomizing pressure (Chegini & Ghobadian, 2005; Goula & Adamopoulos, 2004; Quek et al., 2007). The reason why the faster heat-transfer between the product and drying air at higher inlet temperatures.

Additionally, high inlet temperature causes the reduced bulk density (Tonon et al., 2008). Increasing the inlet air temperature resulted in a rapid formation of dried layer on the droplet surface and this formation causes vapor-impermeable films on the droplet surface, followed by the formation of vapor bubbles and, consequently the droplet expansion (Chegini & Ghobadian, 2005).

The particle size is as well affected by inlet temperature. When using higher inlet temperatures, particles are produced with larger size and higher swelling (Buffo et al., 2001). Drying at higher temperatures results in faster drying rates, which leads to the early formation of a structure and that did not allow the particles to shrink during drying and makes hardened skin not to exit from the droplet (Buffo et al., 2001). Morphology also can be affected, when higher inlet temperatures, the smooth particle could be produced, and lower inlet temperatures, the skin remains moist so that the hollow particle can deflate and shriveled surface could be produced (Tonon et al., 2008).

In addition, drying yields are also affected by inlet temperatures, and the

degree depends on the sample. The increase of inlet temperatures often produces higher yield because of the greater efficiency of heat and mass transfer processes (Tonon et al., 2008). On the other hand, the increase of inlet temperature can reduce powder yield and the reason might be the melted powder and occurrence of cohesion (Chegini & Ghobadian, 2007).

Lastly, bioactive compounds retention could be affected by inlet temperature. Reduction of the compounds such as lycopene was likely due to the thermal degradation and oxidation (Goula & Adamopoulos, 2004). High inlet temperature causes fast dehydration, and these dehydrated products have large surface which is susceptible to oxidative decomposition during and storage. (Phisut, 2012).

5.2. Feed rate

The feed flow rate was negatively affected the moisture content. The example is the acai juice powder, higher flow rates made in a shorter contact time between the feed and drying air, less efficient heat transfer, thus caused the lower water evaporation, and lower drying yield (Tonon et al., 2008). Additionally, when increasing the feed rate, at constant atomizer pressure, more liquid was atomized into the drying chamber, thus time of drying was reduced and finally the drying was incorrect (Chegini & Ghobadian, 2007).

5.3. Air flow rate

The evaporation of droplets is decided by the speed and degree of the air movement. A lower drying air flow rate leads to an increase in the product halting time in drying chamber and enforces the circulatory effects (Goula & Adamopoulos, 2004). The increased residence time causes to the greater degree of moisture removal. In conclusion, an increase of the drying air flow rate, decrease the residence time of the product in the drying chamber and it leads to have higher moisture contents.

In addition, the effect of drying air flow rate on powder bulk density depends on its effect of moisture content due to the sticky nature of the product (Goula & Adamopoulos, 2004). When powders have higher moisture content, each particle leads to stick together and leave more interspaces between them and it consequently resulting the larger bulk volume. Therefore, the increase of air flow rate leads to high amount of moisture content in powders and low in powder bulk density.

The effect of drying air flow rate on powder solubility also depends on its effect on powder moisture content, as low moisture content seemed to be related with the fast dissolution (Goula & Adamopoulos, 2004). The reduction of air flow rate led to the increased moisture content of powder and decrease in powder solubility (Papadakis et al.,1998).

5.4. Atomizing pressure

The increased of atomizer pressure was spread the liquid into thin film layer and thus caused the smaller droplet and particle size (Chegini & Ghobadian, 2005). At higher atomizing pressure, smaller particle size is produced followed by larger surface, and lower moisture content by quicker drying so that the residual moisture content was decreased (Chegini & Ghobadian, 2005).

5.5. Drying aids

1) Types of drying aids

Among commercially used drying aids, major type materials are carbohydrates (maltodextrin, dextran, cellulose, hydrolyzed starch, and derived), gums (araxbic gums, agar, carrageenan), proteins (gluten, caseins, albumins, and peptides), lipids (wax, paraffin, and peptides), and biopolymers (Samantha et al., 2015). The molar mass of drying aids plays a major role in spray drying enhancement (Adhikari et al., 2009). This factor has a direct relationship with transition temperature, in which shorter chain molecules of drying aids have low transition temperature than longer chain of drying aids (Adhikari et al., 2009; Shrestha et al., 2008). Higher transition temperature powder has a high stability and low stickiness problem thus higher yield. Samborska et al. (2015) found that when using maltodextrin as a drying aid, increasing DE of maltodextrin improved significantly on product yield of dry raisin juice and honey bee. Cai and Corke (2000) has discovered that as the

molecular weight of maltodextrin decreased, the hygroscopicity of amaranthus betacyanin pigments increased. The authors mentioned that the molecular configuration of shorter chain maltodextrin has more hydrophilic groups. Using different type of drying aid also affects powder solubility (Fazaeli et al., 2012; Rajabi et al., 2015). Maltodextrin is easy to soluble in water, however starch is difficult to soluble. In addition, larger molar mass of maltodextrin dehydrates faster due to the higher surface area over volume ratio exposed to moisture (Fazaeli et al., 2012).

Particle size of powder was also affected the degree of polymerization of maltodextrin. The powder produced with maltodextrin 10 DE showed bigger particle size than 20 DE. This is related to the molecular size of each drying aid. High value of DE have shorter chain, thus the small size of the particles can be produced with maltodextrin 20 DE. Particle morphology as well as particle size can be influenced, for example, when spray dried with gum arabic, the produced powders showed the less amorphous behavior due to its larger molar mass (Yousefi et al., 2010).

2) Concentration of drying aids

The addition of high content of drying aid could increase the total solid in the feed and thus, reduce the moisture content of the particles. For example, Increase of maltodextrin concentration resulted in yield percentage (Shrestha et al., 2008). However, when low concentration of drying aid is used, the stickiness powder could be obtained (Tonon et al., 2008). On the other hand, if

the added maltodextrin was high enough, the resulted powders lost their original color, for instance, when added more than 10% maltodextrin, the particles lost their origin red-orange color (Tonon et al., 2008).

When increasing drying aid concentration, the high bulk density of produced powder could be obtained. In tomato juice powder and orange juice powder, the more added maltodextrin minimized the thermoplastic particles from sticking and the sticky or less free-flowing nature of a powder was associated with a high bulk density (Kwapińska & Zbiciński, 2005).

The higher concentration also leads to produce the larger particles, which may be related to the feed viscosity which exponentially increased with concentration (Jinapong et al., 2008). The higher concentration, the higher the liquid viscosity, and thus the larger the droplets formed during atomization (Jinapong et al., 2008).

Table. 1.11. Classifications of sweeteners

Type	Characteristics	Examples
Sugar	Composed of two monosaccharides, glucose and fructose Contribute to health issues such as tooth decay, diabetes, obesity, cardiovascular disease, high blood pressure, cancer, etc.	
Natural sweetener	Carbohydrates from vegetables, trees, seeds, roots, and nuts Received much attention due to increasing health concerns caused By over the consumption of sugar and some nonnutritive artificial alternative sweeteners	Honey, maple syrup, molasses, coconut sugar, xylitol, stevia, etc.
Artificial sweetener	Carbohydrate substitutes that replace natural sweeteners Low or no energy value Cost-effective availability with higher sweetening value Made of chemicals so many people have no preference Health issues include headaches, attention deficit disorders, etc.	Aspartame, neotame, saccharin, acesulfame potassium, sucralose, etc.

6. Sweetener

The basic tastes we can feel are a total of five things; sweetness, saltiness, sourness, bitterness, and umami (Ando, 2020). Among them, to obtain sweetness, sugar is commonly used, and nowadays, many alternative sweetener markets are developing (Tandel, 2011).

6.1. Sugars

Sucrose is a common sugar. It is a disaccharide, a molecule composed of two monosaccharides, glucose and fructose (Fig. 1.11). It is a substance that we consistently feel as a sample of sweetness and it is also a reference point for all sweeteners (Tandel, 2011). It is contained in most foods including juice, chocolate, candy, ice cream, etc. Once eating it, it's so sweet, however, consuming too much sugar can contribute to health problems such as tooth decay, diabetes, obesity, cardiovascular disease, high blood pressure, cancer, etc. (Tandel, 2011).

As people increasingly want and demand healthier sweets, they are conducting a lot of research on alternative sweeteners to replace sucrose (Morais et al., 2014; Saputro et al., 2017; Struck et al., 2014).

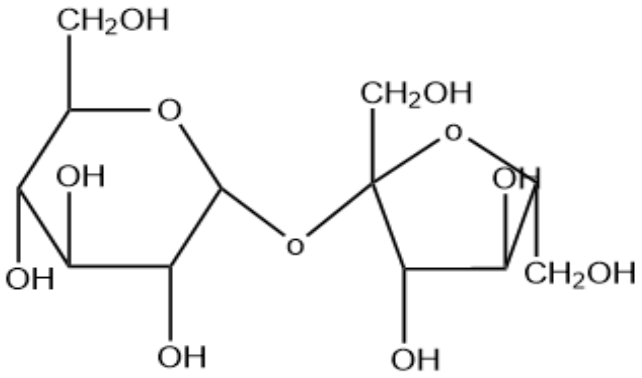


Fig. 1.11. Structure of sucrose

6.2. Natural sweeteners

Natural sweeteners have received much attention due to increasing health concerns caused by over the consumption of sugar as well as problems related to the safety of some nonnutritive artificial alternative sweeteners (Temps, 2018). Natural sweeteners, in comparison to nonnutritive artificial sweeteners, contain calories and nutrients, are metabolized, and change as they pass through the body.

Natural sweeteners are carbohydrates obtained from vegetables, trees, seeds, roots, and nuts. The commonly used natural sweeteners are honey, maple syrup, molasses, coconut sugar, xylitol, and stevia. Among them, stevia, steviol glucoside (stevioside) is extracted from the leaves of the plant Stevia rebaudiana Bertoni, a rhizomatous perennial shrub in the family of Asteraceae that originated in Paraguay and Brazil and it is the most popular natural sweetener.

Stevioside (stevia) have gained attention in food and beverages due to their properties of low-cost, highly stable, and it is as much as 300 times sweeter than table sugar, in general, ¼ teaspoon of powdered stevia extract, or 2 tablespoons whole-leaf stevia, equals about 1 cup of white sugar in sweetening (Misra et al., 2011). Despite it has therapeutic effects for diabetic patient, with the results of diabetes-induced rats, by injection of alloxan, where medium-polar leaf extract of S. rebaudiana (200 and 400 mg/kg) produced a significant decrease in the blood glucose, without producing condition of hypoglycemia after treatment (Misra et al., 2011), it also have properties of low in calories

(most natural sweeteners have a calorie value). Stevioside has a licorice-like, bitter taste. Most people who are accustomed to the sweetness of sugar do not prefer it (jung et al., 2021).

6.3. Artificial sweeteners

Artificial sweeteners comprise carbohydrate substitutes which replace natural sweeteners in food and beverages due to their cost-effective availability with higher sweetening value than natural sweeteners and very low or no energy (Neacsu & Madar, 2014). They are widely used in baking, candy, soft drinks, canned food, jams, pudding, jellies, powdered drink and dairy products. According to the FDA, the five main artificial sweeteners are aspartame, saccharin, acesulfame potassium, neotame, and sucralose (Neacsu & Madar, 2014).

Artificial sweeteners are made of chemicals so many people have no preference, and the use of artificial sweeteners has become controversial due to the health problems that are experienced by people who consume them (Temps, 2018). For example, aspartame, a representative non-caloric artificial sweetener, can lead to headaches, seizures, and attention deficit disorders (Neacsu & Madar, 2014). Some people experience constipation, swelling of certain body parts, and heart palpitations after consuming this artificial sweetener, and some cases, regular intake of artificial sweeteners has resulted in addiction (Neacsu & Madar, 2014).

6.4. Persimmon powder as a potential sweetener

Persimmon has high content of sugar, but low glycemic index (GI) (Pai et al., 2015). In addition, it contains some bioactive compounds such as vitamin C, polyphenolic compounds (Yaqub et al., 2016). Persimmon powder, made by persimmon extract using spray drying, can be a healthy natural sweetener due to not only high content of sugar, high antioxidant properties derived from vitamin C and phenolic compounds, and antidiabetic effects from proanthocyanidins, but also the role of resistant maltodextrin as a prebiotic dietary fiber.

7. Research significance

1) Spray-dried fruit powder can overcome the limitations associated with fruit commercialization such as seasonality, storage and transportation loss, waste issues, and pesticide residues.

2) Little information is available about persimmon powderization using resistant maltodextrin and applications as a natural potential sweetener.

3) Persimmon has high content of sugar (12-15%), and bioactive compounds such as vitamin C and polyphenolic compounds, so the produced persimmon powder can be a natural sweetener which also has antioxidant, and antidiabetic effects.

4) Bioactive compounds could easily be destroyed by many factors such as heat,

air, etc, however, drying aid can reduce the degradation of these compounds. As drying aids, resistant maltodextrin was used due to not only its low DE and properties of high solubility but also health beneficial effects as prebiotic dietary fiber.

5) Gum arabic has film forming properties, so the initial emulsion could be more stable, and it also has dietary fiber effects.

8. Preliminary test

When powderization using spray drying to overcome limitations of unprocessed fruits, the results of generated powder must meet the following parameters to an appropriate level: drying yield, moisture content, water activity. Powder drying yield should be at least 50% in increase the drying efficiency, and the produced powder's water activity and moisture content were considered as less than 0.3 (Tonon et al., 2009) and 5% (Ramakrishnan et al., 2018), respectively, which is good for fruit powder stability. The lower moisture content and water activity are desirable for food powders not only to extend the shelf life of the powders but also to prevent agglomeration and caking, which can result in wet powders, degradation of bioactive compounds, and hindrance of the flowability and dispersion. For these reasons, the drying conditions was chosen (Table. 1.12): inlet temperature, atomizing pressure, air flow rate, feeding rate, and outlet temperatures were 140 ℃, 100 kPa, 0.40 m^3/min, 270 mL/h, and 84 ± 4 ℃, respectively.

Table. 1.12. Effects of spray drying conditions on the drying yield (Y_s), moisture content (MC), and water activity (a_w) of resulting powders

Drying aid	TS (%)	T_{in} (℃)	T_{out} (℃)	r_r (mL/h)	r_a (m^3/min)	P_a (kPa)	Y_s (%)	MC (%)	a_w
MD	30	140	83 ± 5	380	0.30	100	-[a]	-[a]	-[a]
	40	140	83 ± 4	380	0.30	100	25	-[b]	-[b]
	50	140	87 ± 6	380	0.30	100	25	-[b]	-[b]
	60	140	89 ± 5	380	0.30	100	20	-[b]	0.20
MD	40	140	85 ± 4	240	0.30	100	34	1.50 ± 0.26	0.09
GA	40	140	84 ± 3	240	0.30	100	30	5.17 ± 0.04	0.26
MD+GA (1:5)	40	140	85 ± 4	240	0.30	100	40 ± 5	3.89 ± 0.15	0.20
MD+GA (1:1)	40	140	86 ± 3	240	0.30	100	36 ± 7	4.28 ± 0.55	0.21
MD+PE (10:1)	40	140	85 ± 4	240	0.30	100	30	3.13 ± 0.20	0.13
IN_{GR}	40	140	87 ± 3	240	0.30	100	-[a]	-[a]	-[a]
IN_{HP}	40	140	85 ± 3	240	0.30	100	-[a]	-[a]	-[a]
IN_{GR}+IN_{HP}	40	140	85 ± 5	240	0.30	100	-[a]	-[a]	-[a]
IN_{GR}+MD (1:1)	40	140	87 ± 4	240	0.30	100	8	2.58 ± 0.20	0.252
IN_{GR}+GA (1:1)	40	140	86 ± 4	240	0.30	100	38	3.79 ± 0.13	0.255
RMD	40	140	85 ± 5	240	0.30	100	42	4.84 ± 0.08	0.237
	40	140	85 ± 4	240	0.40	100	42	1.75 ± 0.23	0.232
	40	140	84 ± 4	270	0.40	100	44	2.00 ± 0.20	0.251
RMD+GA (7:3)	40	140	86 ± 4	270	0.40	100	56	2.21 ± 0.07	0.158

MD, maltodextrin; GA, gum arabic; PE, pectin; IN, inulin; RMD, resistant maltodextrin -[a] : not detected, -[b] : no experiment,

TS, Total solid; T_{in}, inlet temperature; T_{out}, outlet temperature; r_r, feed rate; r_a, air flow rate; P_a, atomizing pressure; Y_s, drying yield; MC, moisture content; a_w, water activity

In case of persimmon extract powder which is added with maltodextrin as a drying aid (PE-MD), powders were stuck to the wall of the cyclone without moving to the collector (Fig. 1.12 a) When the particles dry well, they are collected within a collector or accumulated in the area where the collector and cyclone meet. As shown in Fig. 1.12 (c), Persimmon extract powder which is added with resistant maltodextrin and gum arabic as drying aids (PE-RMGA), it is judged that the drying process was good when they did not stick to the cyclone wall and gathered like a whirl at the bottom of the cyclone. The water content of PE-MD is relatively lower than that of PE-RMGA (Fig. 2.4), each part of the particles has less weight, so the particles have not been able to sink to the collector. Unlike PE-MD, persimmon extract powder with resistant maltodextrin as a drying aid (PE-RM) has shown similar tendency to PE-RMGA (Fig. 1.12 b), which means that the drying was good, and on that basis PE-RM yield is higher than other cases, except for PE-RMGA (7:3) (Table. 2.2) The difference of dextrose equivalent (DE) value of MD (15~20) and RMD (12.3) might have affected the results. Higher DE maltodextrin with lower molar mass is known to lower the glass transition temperature, the particles would have higher hygroscopicity, sticked to chamber, and produce lower drying yield (Desobry et al., 1997). However, when spray dried with RMD, stickiness problem occurred, the air flow rate naturally decreased and blocked the bag filter, because the powder form was matrix type and rapid evaporation occurred. When rapid evaporation occurs, higher yields can be produced due to fast drying, however, fruit powders have high hygroscopicity (Moraga et al., 2012), RMD alone was used as a drying aid, maybe emulsion wasn’t stable

form, thereby could be turned into sticky powder and blocked the filter leading to poor blower performance.

GA is a complex heteropolysaccharide with a ramified structure and more hydrophilic groups, which results in a higher water binding capacity, also has film forming properties that form stable emulsions and could prevent stickiness.

Persimmon has high sugar contents, the T_g would be low due to low molar mass of sugars, and the resulting powder maybe sticky because of the lower sticky temperature. Also, as the rapid removal of water during the spray drying process produced amorphous material, in resultant of surface stickiness between adjacent powder particles. The caking problem occurred as the surface viscosity has reached a critical value and formation of inter-particle bonds between particle. Unless the air flow rate was manually increased, the drying yield is not fixed and gradually decreased, and if it was increased arbitrarily, it caused the reduction of the blower's life, so we used it mixed with GA. On the other hand, when the content of gum arabic was high, viscosity was also higher, the atomizer was not sprayed normally as shown in Fig. 1.13., and the solution was transformed in the form of icicles. Considering the drying yield, water activity, moisture content, and T_g which was measured immediately after spray drying, were observed (Table. 1.13). The ideal value was showed when RMD and GA were mixed at a ratio of 7:3, and this was selected as a final drying aid.

(a) (b) (c)

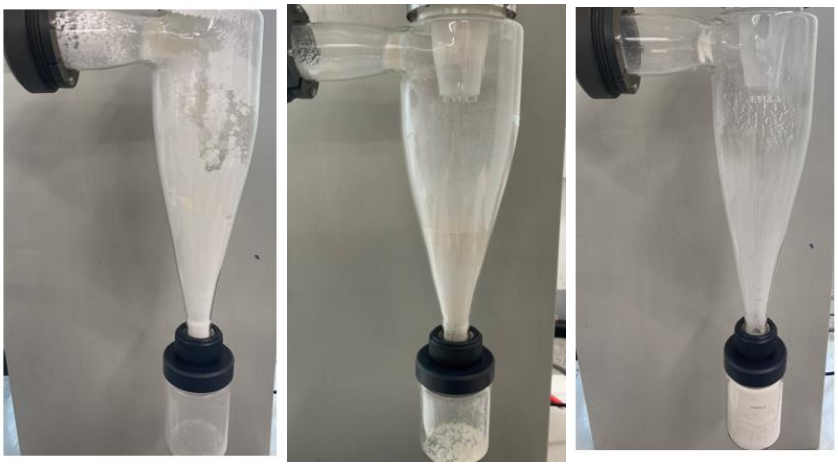


Fig. 1.12. Deposition of prebiotics-persimmon powders on the wall of cyclone and collector of spray dryer: (a) PE-MD, (b) PE-RM, and (c) PE-RMGA

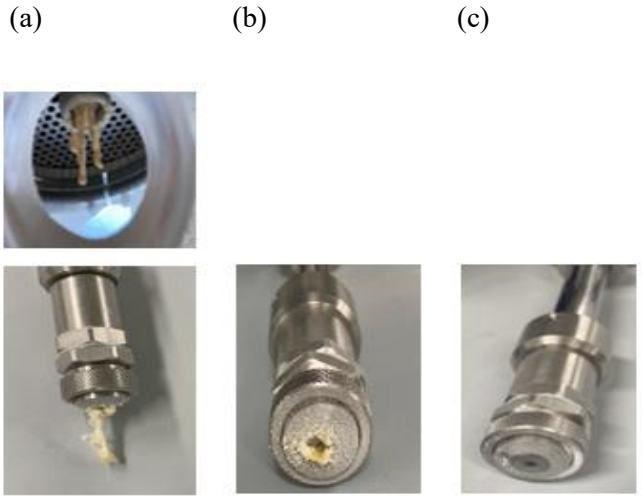


Fig. 1.13. Deposition of prebiotics-persimmon powders in atomizer: (a) PE-GA, (b) PE-RMGA, and (c) PE-RM

Table. 1.13. Drying yield (Y_s), moisture content (MC), and water activity (a_w) of prebiotics-persimmon powders prepared with different ratios of resistant maltodextrin (RMD) and gum arabic (GA).

Drying aid	TS (%)	T_{in} (℃)	T_{out} (℃)	r_r (mL/h)	r_a (m3/min)	P_a (kPa)	Y_s (%)	MC (%)	a_w
RMD + GA (10:0)	40	140	85 ± 5	270	0.40	100	44	2.00 ± 0.20	0.251
RMD + GA (9:1)	40	140	90 ± 4	270	0.40	100	44	2.00 ± 0.20	0.097
RMD + GA (8:2)	40	140	88 ± 6	270	0.40	100	46	3.06 ± 0.05	0.216
RMD + GA (7:3)	40	140	88 ± 5	270	0.40	100	56	2.21 ± 0.07	0.158
RMD + GA (6:4)	40	140	88 ± 6	270	0.40	100	44	3.50 ± 0.10	0.217
RMD + GA (5:5)	40	140	91 ± 7	270	0.40	100	40	2.93 ± 0.20	0.174

9. Research objectives

The aim of this study is to make persimmon powder for natural health promoting potential sweetener by spray drying using resistant maltodextrin and gum arabic as drying aids.

Physicochemical properties of persimmon powder by spray dried with resistant maltodextrin and gum arabic were examined in comparison with the persimmon powder by spray dried with maltodextrin.

Antioxidant properties, antidiabetic properties, and sweetening effects of persimmon powder by spray dried with resistant maltodextrin and gum arabic were examined in comparison with the standard materials (gallic acid, vitamin c, catechin, and sucrose).

The overall research stragety was illustrated in Fig. 1.14.

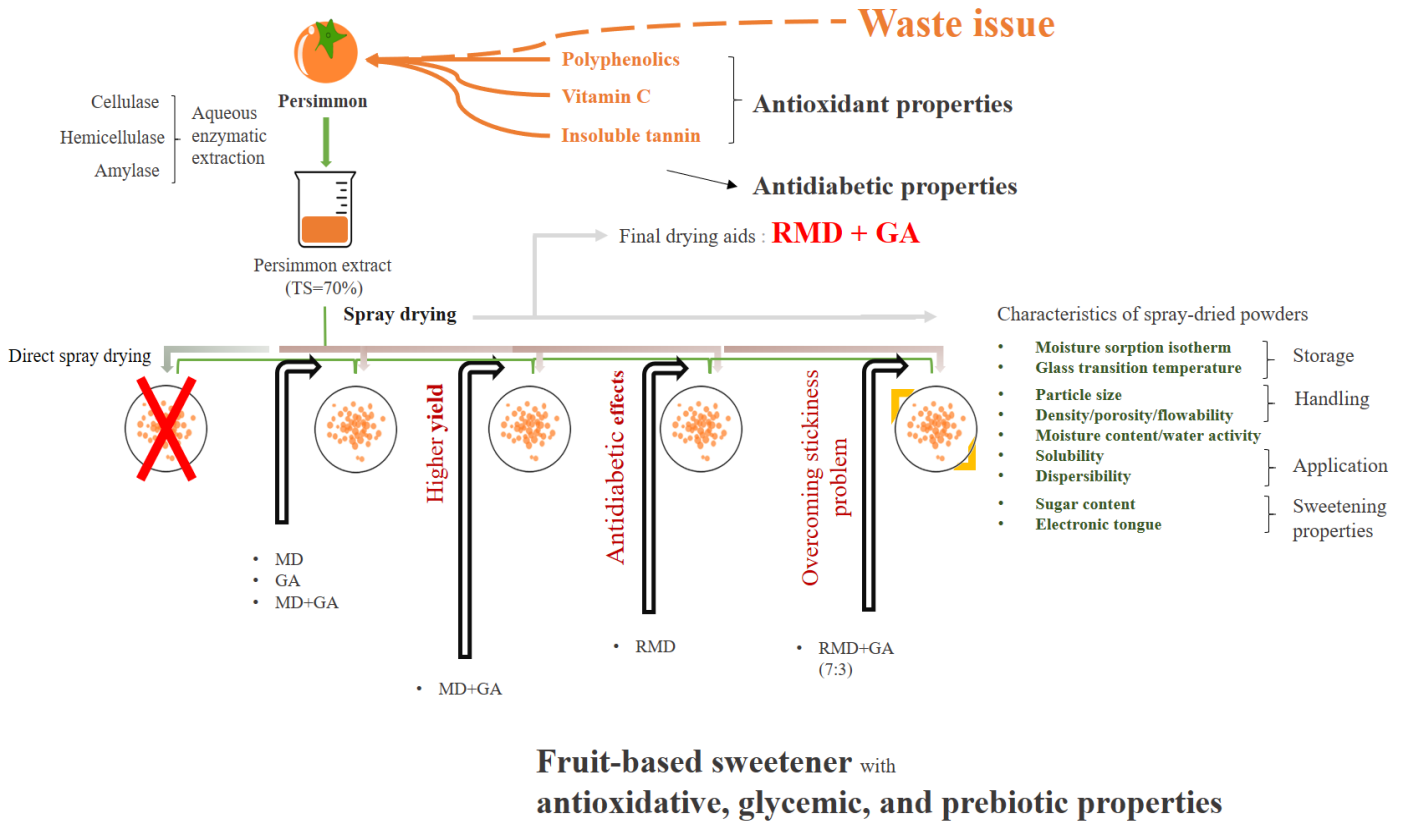


Fig. 1.14. Research strategy. RMD, resistant maltodextrin; GA, gum arabic.

Chapter 2

Physicochemical characteristics of persimmon powder produced by spray drying with a mixture of resistant maltodextrin and gum arabic

1. Introduction

Fruits mainly consist of water and carbohydrates about 80-90% and 9-18%, respectively (Ministry of food and drug safety, 2019). Due to high content of water, it has low shelf life, and most fruits have seasonality, so it's not possible to take whenever we want. Until the consumption stage, cultivated product lost is about 50% (Nguyen et al., 2020), and even at the consumption stage, despite the fact that the thrown peel contains more beneficial substances, the rate of thrown away is about 20% (Guo et al., 2003; Li et al., 2006).

Persimmon is an edeible fruit of a number of species of trees in the genus *Diospyros*. *Diospyros kaki* is the most representative species. Persimmons are one of the most consumed fruits in Korea, among species, Cheongdo bansi (*Diospyros kaki* Thunb. cv. Cheongdo-Bansi) contains less water and more sugars compared to *Diospyros kaki*, and is seedless (Kim et al., 2010).

Resistant maltodextrin (RMD) is a polysaccharide which consist of α-1,2, α-1,3 as well as α-1,4, and α-1,6 bonds (Astina & Sapwarobol, 2020). RMD is similar with maltodextrin (MD), however, GI of RMD is 10 times lower than MD (Goda et al., 2006), and it is a prebiotic dietary fiber (Burns et al., 2018;

Fernández-Raudales, et al., 2018) so when using RMD, beneficial effects such as antidiabietic and feeling satiety, growth of *bifidobacterium* could be obtained.

Gum arabic (GA) is composed of the L-arabinose, L-rhamnose, D-glucuronic acid, and 1,3-linked β-D-galactopyranosyl (Cisse et al., 2020). Because it has 1.5% to 3% content of protein (Anderson et al., 2009), it also has emulsifying properties, and thereby could generate higher yield when spray drying, but it could alter the original surface or lead to stickiness, when high amounts are used (Turchiuli et al., 2004).

Spray drying is a process that liquid solution or suspension contacts hot air and after evaporation, turned into solid particles. These process can control particle size, is easy to scale up, and it is cheap and fast process (Rajam & Anandharamakrishnan, 2015). Produced particles have low moisture content, that leads to higher stability. Limitation of unprocessed fruits such as seasonality, low storage could be overcome. In addition, the powder form also has an advantage in transportation because it can be packed in small packages rather than in the form of certain fruits and also the weight is reduced. However, when the liquid that have higher content of sugar meets hot air, stickiness can occur and the drying yield could be low due to low T_g of low molar mass components (Bhandari et al., 1997). Therefore, addition of drying aid is important to minimize the stickiness (Downton et al., 1982).

When RMD which has low viscosity, high solubility, and a role of prebiotic dietary fiber (Goda et al., 2006; Ohkuma et al., 1990) and GA with film forming properties (Turchiuli et al., 2004) are properly mixed at a non-viscous level,

produced by spray drying powders have high yield, and health benefitting properties as a dietary fiber. In addition, when using drying aid as mixture form, especially for mixture with GA (Sanatana et al., 2016) According to (Turchiuli et al., 2014), GA mixed with MD as drying aids, higher yield of spray drying was obtained than using GA or MD respectively. Only GA were used when spray drying, the yield could be high (Yousefi et al., 2010), however, depending on the amount, viscosity could be too high for feeding, and mixing with MD is cost effective with higher yield. On the other hand, GA and MD are the most commonly used when spray drying as drying aids each or mixed, however, little information is available about single use studies of RMD as a drying aid, and mixing RMD with GA when spray drying.

There are many factors that can affect the resulted particles in spray drying such as inlet temperature, feed rate, air flow rate, and atomizing pressure. By adjusting the factors, the higher drying yield could be produced and the produced powder have different properties such as particle size, density, reconstitution properties, etc.

Particle size is used to characterize the size distribution of particles in a given sample, and there are many different means to represent, for example, $d_{4,3}$ is a volume weighted mean and $d_{3,2}$ is a surface weighted mean. Density, mass of a unit volume of a material substance, is correlate with porosity and flowability (Abdullah & Geldart, 1999). When bulk density increases as pore space decreases, and flowability also increases. Reconstitution is the ability to return dehydrated particles to the liquid state by adding water, and it contains, for example, solubility and dispersibility (Fang et al., 2008). Solubility means that

the limit ability of ingredient which will dissolve and merge with the substance when it is put into. In short, the degree of the ingredient becoming a homogenous mass (molecular or ionic level) (Fang et al., 2008). Whereas, dispersibility just indicates the degree of dispersal. The ingredient doesn't merge with the substance it is put into, but it can be dispersed (spread out evenly) if handled according to a specific method (solute particle level) (Fang et al., 2008). When the powder meets the solvent, it is first dispersed and finally dissolved (Forny, et al., 2011). Powder stability could be affected by moisture content, water activity, temperature, etc. The lower moisture content and water activity are desirable for food powders to prevent agglomeration and caking, which can result in wet powders, degradation of bioactive compounds, and hindrance of the flowability and dispersion, to extend the shelf life of the powders. When solid particles absorb water, firstly they turned into glassy form, and finally to sticky or caking state (Boonyai et al., 2004).

In this study, the objective is to evaluate physicochemical properties of persimmon powder produced by spray drying wit a mixture of resistant maltodextrin and gum arabic as a drying aid.

2. Materials and methods

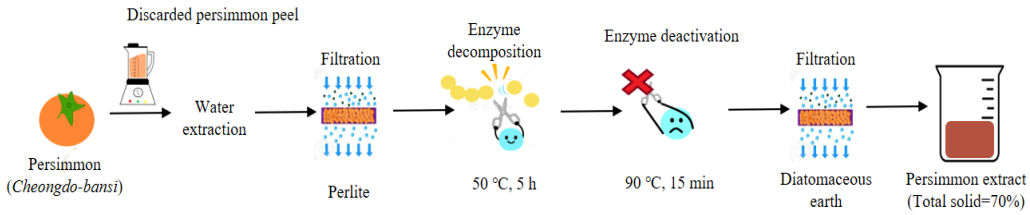
2.1. Materials

An aqueous extract of Cheongdo-bansi (*D. kaki* Thunb. cv. Cheongdo-Bansi) was obtained from Nature Farm (Seoul, Korea). Extraction was conducted as

follows. First, whole persimmon was washed to remove foreign substances and blended using a blender. Water extraction was conducted to facilitate stirring and extraction at room temperature and filtered it with perlite to get only water soluble substances. Then, enzyme decomposition such as cellulase, hemicellulose, and amylase was performed for 5 hours at 50 ℃ and then the enzyme was inactivated for 15 minutes at 90 ℃. Afterwards, the second filtering is implemented and concentrated to 70 brix (Fig. 2.1 a). According to Table 2.4, it consists of about 70% carbohydrates and 30% moisture.

For persimmon extract powderization, RMD (DE=12.3) (Matsutani Korea Ltd., Seongnam, Korea) was provided and MD (DE=15~20) and GA (Serimfood Co., Bucheon, Korea) were purchased.

(a)



(b)

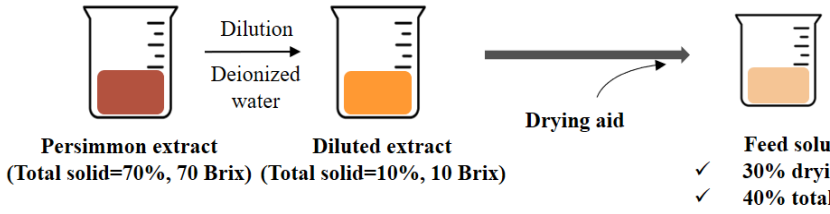


Fig. 2.1. Schematics of persimmon extraction and preparation of feed solutions. (a) Process of persimmon extraction (b) Preparation of feed solutions of 40% total solid (10% extract solid and 30% drying aid): extraction, dilution, and adding biopolymers as drying aid

2.2. Proximate composition analysis of persimmon extract

Proximate composition includes water, ash, crude proteins and lipids, and carbohydrates. According to general ingredient test method (AOAC, 2005), the compositions of extract were measured.

After measuring 2 g of the extract in crucible, it moved to 105 ℃ drying oven (Hanbaek Co., Bucheon, Korea) for 24 h until no change in weight. The sample moved to desiccator which contains silica gel to cool, and weigh it to calculate water contents.

To measure ash contents, 2 g of the extract was measured and heated at 550 ℃ until carbon disappear, cooled it down in a desiccator and weigh it to get the amount of ash.

Crude proteins were measured using Kjeldahl method which consists of disassembling and distillation and titration. 2 mg of samples were taken and put into a 300 mL Kjeldahl flask and added 1 g of a decomposition promoter and 20 mL of sulfuric acid to dissolve them. After cooling the decomposition solution, 100 mL of water was added and transferred to the 200 mL flask, filled the entire amount to 200 mL with water, and connected the Kjeldahl flask to the distillation part. 10 mL of 0.05 N sulphuric acid was poured into the flask and 2 drops of Brunswik solution were dropped and 25 mL of 30% sodium hydroxide solution was added. Then after distillation, 100 mL of the distilled solution was taken, titration was done until the Brunswik solution turned into green. The test was conducted separately in the same way for blank. Total nitrogen (%) was measured by 0.7003*(a-b)*100/sample (mg), which a means

the number of mL of 0.05 N sodium hydroxide solution required for neutralization in a blank test, and b means that the number of mL of that solution required for the test. Final crude proteins were obtained by multiplying total nitrogen and 6.25 (nitrogen coefficient).

Crude fats were measured using Soxhlet method. First, about 5 g sample was pre-dried, placed in a cylinder filter paper, and dried at 105 ℃ oven for 2 h, cooled in a desiccator and put ion an extraction tube of the inner slot extractor. After extraction, cooler and cylinder filter paper were removed from the extraction tube, and cooler was connected again, and heated. When all the ethers in the extraction flask were transferred to the extraction tube, the extraction flask was removed and evaporated ether completely. Placed at 105 ℃ oven for 1 h, and cooled in the desiccator. The amount of crude fat was calculated by (w_0-w_1) x 100/S, w_0 means weight of extraction flask (g), w_1 means weight of the extraction flask after extraction and drying (g), and S means the amount of sample (g).

The amount of total carbohydrates was calculated by subtracting all the values from 100.

2.3. Preparation of feed suspensions

Drying aids of 30% (w/w) were added into persimmon extract to make 40% (w/w) total solid of feed solution (Fig. 2.1 b). The solid ratio of the drying aid is taken into account yield (Table. 1.12, MD). Below this concentration, the high stickiness resulted in an insignificant process yield.

The two feed solutions were prepared: PE-RMGA and PE-MD. As drying aid mixture, 30% RMD and GA at a ratio of 7:3 were added to 10% persimmon extract (Table. 1.13), and 30% MD was added to 10% persimmon extract. As a control group, PE-MD was used instead of PE-GA, because the most commonly used drying aid for spray drying is MD. To dissolve completely, total solid of 40% (w/w) of persimmon extract with MD was stirred by using stirrer (Direct Driven Stirrer, Yhana Co., Kimpo, Korea) at 500 rpm for 10 min and 40% (w/w) persimmon extract with RMD and GA at a ratio of 7:3 was stirred as same with stirrer (Direct Driven Stirrer, Yhana Co., Kimpo, Korea) at 500 rpm for 10 min, and then performed by using a high speed homogenizer (IKA T25, IKA Co., Kolner, Germany) at 1000 rpm for 3 min at room temperature. On the other hand, spray drying with 30% GA as a drying aid resulted in the high viscosity, which prevented feedings from proceeding normally (Fig. 1.13 a), and the drying yield was as low as 30% (Table. 1.12), and as a result of using only RMD as a drying aid, the drying yield was about 42% (Table. 1.12), but it was stopped in the middle of the process and could not be used as a continuous process.

2.4. Spray drying

The feed solutions were fed into the spray dryer (Eyela SD-1000, EYELA Ltd., Tokyo, Japan) using the peristaltic pump. The type of atomizer was two fluid nozzle spray. The drying conditions such as inlet temperature, atomizing pressure, air flow rate, and feeding rate, were considered, and outlet

temperature was also adjusted between 80-90 ℃ by regulating other factors, and this parameter was measured every 5 min and averaged. When the outlet temperature is over than 95 ℃ during the process is difficult to handle. The drying yield should be at least 50% in increase the drying efficiency, and the produced powder's water activity and moisture content were considered as less than 0.3 (Tonon et al., 2009) and 5% (Ramakrishnan et al., 2018) respectively, which is good for fruit powder stability. The drying conditions was: inlet temperature, atomizing pressure, air flow rate, feeding rate, and outlet temperatures were 140 ℃, 100 kPa, 0.40 m^3/min, 270 mL/h, and 84 ± 4 ℃, respectively.

After spray drying, produced PE-MD and PE-RMGA were stored in 50 mL falcon tube enclosed with silica gel until experiment to prevent oxygen penetration and biological reactions at room temperature (Pedro et al., 2010; Alissa et al., 2020).

2.5. Determination of drying yields

The drying yield was defined as the ratio between the collected powder after spray drying and the amount of solids in the feed solution before spray drying (Broeckx et al., 2019).

$$\text{Drying yield (\%)} = \frac{Mass\ of\ collected\ powder\ (g)}{Mass\ of\ total\ solids\ in\ the\ feed\ solution\ before\ spray\ drying\ (g)} \times 100 \qquad (1)$$

2.6. Determination of water activity and moisture content

The water activity (α_w) of spray-dried powders were measured by using water activity meter (Decagon AquaLab, Meter Inc., Pullman, WA, USA) at 25 ℃.

The moisture contents of spray-dried powders were determined by drying at 105 ℃ in a drying oven (Hanbaek Co., Bucheon, Korea) for 24 h (AOAC, 2005). The moisture content (X) was calculated in a dry basis (%, db)

$$X\ (\%,\ \mathrm{db})\ =\ \frac{Mass\ of\ moisture\ (g)}{Mass\ of\ spray{-}dried\ powder\ (g) - Mass\ of\ moisture\ (g)} \times 100 \qquad (2)$$

2.7. Particle size analysis

The De Brouckere (volume-weighted) mean diameter ($d_{4,3}$), particle size distribution, and span value of spray-dried powders were checked by using laser diffraction particle size analyzer (1190, CILAS, Buffon, France). D(4,3) is weighted mean value by volume and is calculated that the sum of four squares of radius (d) divided by the sum of radius cubes.

$$d4{,}3 = \frac{\sum d^4}{\sum d^3} \qquad (3)$$

Span value was also calculated using following equation:

$$Span = \frac{d_{90} - d_{10}}{d_{50}} \qquad (4)$$

where d_{10}, d_{50}, and d_{90} are the diameters of the powders at 10%, 50%, and 90% cumulative volume in particle size distribution, respectively (Yu et al., 2019).

2.8. Particle morphology analysis

For understanding effects of particle surface, morphology, and structure, powders were observed by scanning electron microscope (SEM) (TM3030Plus Tabletop Microscope, Hitachi, Tokyo, Japan) at 5 kV, 100-2,000X magnification under high vacuum condition. When a particle sample is placed in the electron microscope, it is non-destructively bombarded by a finely focused beam (probe) of electrons. As the sample is irradiated by this stream of primary electrons a variety of interactions occur with the atoms in the sample. As a result, various forms of radiation are released from the sample which, when detected and processed, can be used to determined its constituent components.

2.9. Determination of powder density, porosity and flowability

Particle true density (ρ_p) was measured using a helium gas pycnometer (Accupyc II 1340, Micromeritics, Norcross, GA, USA) at 21 ± 1 ℃. Samples were occupied nearly 60% of volume in 10 cm^3 cell.

$$\rho_{\mathrm{p}} = \frac{Powder\ mass\ in\ the\ cell\ (g)}{Apparent\ volume\ of\ powder\ in\ the\ cell\ (cm^3)} \quad (5)$$

Loose bulk density (ρ_L) was measured by using 10 mL plastic graduated cylinder (Atalar & Yazici, 2019). Approximately 6 mL of volume in cylinder was filled with samples.

$$\rho_{\mathrm{L}} = \frac{Powder\ mass\ in\ cylinder\ (g)}{Powder\ volume\ in\ cylinder\ (cm^3)} \quad (6)$$

Tapped bulk density (ρ_T) was measured after 125 tapping at a height of 5 cm from the horizontal surface (Atalar & Yazici, 2019).

$$\rho_{\mathrm{T}} = \frac{Powder\ mass\ in\ cylinder\ after\ tapping\ (g)}{Powder\ volume\ in\ cylinder\ after\ tapping\ (cm^3)} \quad (7)$$

Porosity (ε) was calculated by using the relationship between ρ_T and ρ_p (Atalar & Yazici, 2019).

$$\varepsilon\ (\%) = \frac{(\rho_{\mathrm{p}} - \rho_{\mathrm{T}})}{\rho_{\mathrm{p}}} \times 100 \quad (8)$$

Carr index (CI) and Hausner ratio (HR) were used for flowability of powders calculated by using ρ_T and ρ_L Flowability is determined according to the criteria in Table 2.3 (Reddy et al., 2014).

$$\mathrm{CI}\ (\%) = \frac{(\rho_T - \rho_L)}{\rho_T} \times 100 \tag{9}$$

$$\mathrm{HR} = \frac{\rho_T}{\rho_L} \tag{10}$$

Table. 2.1. Specifications of flowability related to Carr index and Hausner ratio (Reddy et al., 2014)

Flowability	Carr index (%)	Hausner ratio
Excellent	0-10	1.00-1.11
Good	10-15	1.12-1.18
Fair	16-20	1.19-1.25
Possible	21-25	1.26-1.34
Poor	26-31	1.35-1.45
Very poor	32-37	1.46-1.59
Very very poor	>38	>1.60

2.10. Determination of reconstitution properties

Reconstitution properties such as dispersibility and solubility were measured at room temperature.

The dispersibility was measured according to Balde et al., 2017; Ji et al., 2016. One gram of powder was dispersed to 10 mL of distilled water in 50 mL beaker and stirred by a magnetic stirrer for 25 s at room temperature. The reconstituted powder passed through a 200 μm sieve. Then the 1.0 mL sieved solution was dried for 24 h at 105 ℃. After that, dispersibility (%) was calculated as follows.

$$\text{Dispersibility (\%)} = \frac{(10+a) * \%TS}{a * \frac{100-X}{100}} \times 100 \quad (11)$$

Where, a = amount of powder being used, X (%, db) = moisture content in the powder, and %TS = dry matter in percentage in the reconstituted powder after it has been passed through the sieve.

For the powder solubility, 1 g of powder was dispersed to 10 mL of distilled water in 50 mL beaker and stirred for 30 min at room temperature (Laokuldilok & Kanha, 2015). The suspension was centrifuged at 6000 rpm (Gyrozen 1236 MG, Gimpo, Korea) for 20 min. The supernatant was then dried for 24 h at 105 °C. After drying, the solubility was calculated by following this equation.

$$\text{Solubility (\%)} = \frac{Mass\ of\ dried\ supernatant\ (g) \times \frac{Mass\ of\ used\ powder\ (g) + mass\ of\ used\ distilled\ water\ (g)}{mass\ of\ centrifuged\ supernatant\ (g)}}{Mass\ of\ used\ powder\ (g)} \times 100 \quad (12)$$

2.11. Determination of moisture sorption isotherm

Moisture sorption isotherms of powders were measured by using gravimetric method (Yu et al., 2019). Saturated salt solutions (Table. 2.2) were placed in each glass desiccators and equilibrated at 25 °C in temperature-controlled chamber (HB-103LP; Hanbaek Scientific Co., Bucheon, Korea). For adsorption, approximately 2 g of powder was placed on the pre-weighed aluminum dished and moved to RH 0% desiccator. After equilibration, powders were moved to each desiccator from RH 0% to RH 94% and checked the difference of weight. In case of desorption, about 2 g of powder was placed on the RH 94% desiccator, and after equilibration, powders were moved to each desiccator from RH 94% to RH 0%. The desiccators with higher than 0.67 a_w, 30 g of thymol in 50 mL beaker was placed to prevent microbial growth (Sandoval et al., 2011).

For understanding the sorption mechanism and predicting absorbed moisture (X_{eq}) at given water activity and temperature, modeling of moisture sorption isotherms was conducted by using seven different types of empirical or semi-empirical models (Table 2.3) (Yu et al., 2019). The parameters of each models were got by using non-linear regression analysis using SPSS software (IBM SPSS Statistics 25, Chicago, IL, USA). The fitness of each model was evaluated by calculating the coefficient of determination (R^2), the standard error (SE), and the mean relative percentage deviation modulus (M_r) (Yu et al., 2019).

Table. 2.2. a_w of saturated salt solutions at 25 ℃ (Yu et al., 2019)

Saturated salt solution	a_w
Phosphorous pentoxide (P_4O_{10})	0.00
Lithium chloride (LiCl)	0.11
Potassium acetate (CH_3COOK)	0.23
Magnesium chloride ($MgCl_2$)	0.33
Potassium carbonate (K_2CO_3)	0.43
Magnesium nitrate ($Mg(NO_3)_2$)	0.53
Potassium iodide (KI)	0.69
Ammonium sulfate ($(NH_4)_2SO_4$)	0.80
Potassium nitrate (KNO_3)	0.94

Table. 2.3. Classifications of models for describing powder's moisture sorption isotherm (Yu et al., 2019)

Model	Mathematical expression
BET	$X_{eq} = X_0 C a_w / [(1 - aw)(1 - aw + Caw)]$
GAB	$X_{eq} = X_0 C K a_w / [(1 - Kaw)(1 - Kaw + CKaw)]$
Peleg	$X_{eq} = K_1 {a_w}^{n1} + K_2 {a_w}^{n2}$
Henderson	$X_{eq} = \left[-\ln\left(1 - \frac{a_w}{A}\right)\right]^{\frac{1}{B}}$
Smith	$X_{eq} = A + (Blog(1 - aw))$
Oswin	$X_{eq} = A(\frac{a_w}{1 - aw})^B$
Ferro-Fontan	$X_{eq} = [\frac{\gamma}{\ln(\frac{a}{a_w})}]^{1/r}$

X_{eq} , equilibrium moisture content (g water/g dry matter); X_0 , monolayer moisture content (g water/g dry matter); α_w, water activity; T, absolute temperature; C, K, A, B, r, γ, K_1, K_2, n_1, and n_2, model constants.

$$SE = \sqrt{\frac{\sum(Y_i - Ypi)^2}{df}} \quad (13)$$

$$M_{\mathrm{r}}(\%) = \frac{100}{N}\sum_{i=1}^{N}\frac{|Y_i - Y\mathrm{pi}|}{Y_i} \quad (14)$$

Where *df* is the degree of freedom of regression model, Y_{i} and *Ypi* are experimental and predicted values of absorbed moisture (X_{eq}), and *N* is the population of experimental data means, respectively. The sorption models of an R^2 value above 0.98 and the *Mr* value below 10% were selected for the reasonable fitness. A model with a lower SE value and a residual value closer to zero is regarded more suitable. (Yu et al., 2019).

2.12. Determination of glass transition temperature

Powders equilibrated at each a_{w} (0.0-0.33) in the desiccators were used to measure glass transition temperature (T_{g}) by using differential scanning calorimetry (DSC 250, TA Instrument, New Castle, DE, USA). When a_{w} was higher than 0.33, the powders were already glassy state (above T_{g}), it was impossible to measure T_{g}. Approximately 5 mg of powder was placed in T zero pan (T 181128), and sealed by T zero pan to maintain the machine without explosion when heated. Heating and cooling rate is 10 °C/min from -20 °C to 130 °C for two cycles. During the first heating treatment relaxation peak exist, in order to eliminate the hysteresis effects of thermal relaxations when

determining T_g, it is commonly repeated the heating cycle after sample has been cooled to the start temperature (Foster, 2002).

For understanding ratio of powder compositions and predicting T_g at given moisture content or water activity, Gordon-Taylor equation (Tonon et al., 2009) was applied by using non-linear regression analysis using SPSS software (IBM SPSS Statistics 25, Chicago, IL, USA).

$$T_g = \frac{w_s T_{gs} + k_{sw} w_w T_{gw}}{w_s + k_{sw} w_w} \quad (15)$$

where w_s and w_w are fraction of solid and water, k_{sw} is the constant value, and T_{gs} and T_{gw} are T_g of solid and water (Tonon et al., 2009).

2.13. Statistical analysis

All experiments were performed in triplicate, and the results were presented as mean ± standard deviation (SD). Statistical process of the experimental results was analyzed using SPSS 26 program (SPSS Inc., Chicago, IL, USA). Significant differences ($p \leq 0.05$) were determined by T-test using SPSS 26.

3. Results and discussion

3.1. Proximate composition of persimmon extract

Proximate composition of persimmon extract showed that contents of carbohydrates, crude proteins, crude lipids, ash, and water were 69.7%, 0.1%, 0.0%, 0.3%, 29.9%, respectively (Table. 2.4). While extraction, remained proteins and lipid would be removed due to some enzymes or heat treatment. As explained in section 2.1, extract was concentrated to 70 brix, and the composition was almost carbohydrates.

Table. 2.4. Proximate composition of persimmon extract

Composition	**(%, w/w)**
Total carbohydrates	69.7 ± 0.30
Crude proteins	0.1 ± 0.02
Crude fats	0.0 ± 0.01
Ash	0.3 ± 0.06
Moisture	29.9 ± 0.10

Experiments were performed in triplicate, and the results were presented as mean ± standard deviation.

3.2. Characteristics of spray-dried persimmon-prebiotics powders

Powder yields of PE-MD and PE-RMGA were 21% and 56% respectively (Fig. 2.2). When drying yield is higher than 50%, it is known as efficient spray drying (Liu et al., 2017). PE-RMGA had more than twice higher yield than of PE-MD, which means it is dried well compared to the PE-MD. MD is a hydrolyzed short chain starch that acts as a barrier against oxygen. However, when MD was used as wall material, the lowest efficiency was obtained, probably due to lack of emulsification and low film-forming capacity (Loksuwan, 2007). On the other hand, GA has great film-forming capacity, which allows it to better retain the encapsulated molecule (Krishnan et al., 2005, Mahdavi et al., 2016), but if too much amounts were used, viscosity of suspension was too high, making difficult to feed properly (Fig. 1.13), and produced low yield.

GA due to its emulsifying and film forming properties, the use of acacia gum, in combination with maltodextrin and/or inulin, allowed obtaining more stable initial emulsions with a controlled size distribution (~ 2 μm, monodispersed) which was preserved during spray drying. The spray drying powder yield was also improved (Turchiuli et al., 2014).

The moisture content of PE-MD and PE-RMGA are 1.56%, and 2.21%, and water activity were 0.09, 0.158, respectively. In general, moisture content and water activity of respectively less than 5% and 0.30 are recommended for fruit spray drying. Both PE-MD and PE-RMGA showed appropriate value, thus

microbiologically safe. Particle size of PE-MD (7.4 μm) could be responsible for the lower moisture content than PE-RMGA (14.4 μm) due to larger surface area exposed to hot air during spray drying (Tontul & Topuz, 2017).

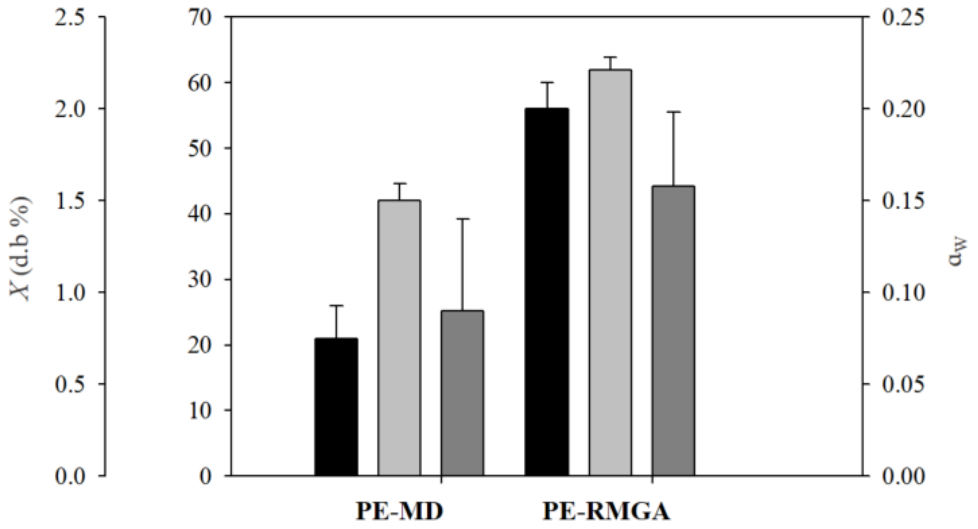


Fig. 2.2. Yield (Y_s,■), moisture content (X,), water activity (a_w,■) of PE-MD and PE-RMGA. PE-MD, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3. Experiments were performed in triplicate, and the results were presented as mean ± standard deviation.

Particle size of powder is important for handling, transportation, storage, flowability, and reconstitution properties. The particle size ($d_{4,3}$) of PE-MD and PE-RMGA showed 7.4 μm, and 14.4 μm, respectively (Fig. 2.4). GA has larger viscosity than MD or RMD, PE-RMGA showed larger particle size than PE-MD. According to Tontul and Topuz. (2017), higher viscosity feed produced the larger particle size. Span value indicates the width of the particle size distribution, the values were PE-MD (2.74) and PE-RMGA (2.09), respectively (Fig. 2.4). PE-MD showed a little broader size distribution (Fig. 2.3), due to structure of maltodextrin. Maltodextrin has a number of ramified hydrophilic groups that could be agglomerated during the handling, according to Zotarelli et al. (2017), for mango powders with maltodextrin produced fine and agglomerated particles. In addition, when spray dried with MD, matrix type of powders was produced, and because of stickiness and hygroscopic properties, the particles form agglomerates. On the other hand, GA has film forming properties, so each powder wouldn't collide and produce less agglomerate.

In terms of particle morphology, all of the particles showed spherical shapes of several sizes, which is typical of powders produced by spray drying (Ferrari et al., 2011). PE-MD particles tended to form agglomerate (Fig. 2.5 a), whereas PE-RMGA particles tended to form film (Fig. 2.5 b), and the particles did not stick together and existed alone. Although agglomerate did not form, large and small particles were each present.

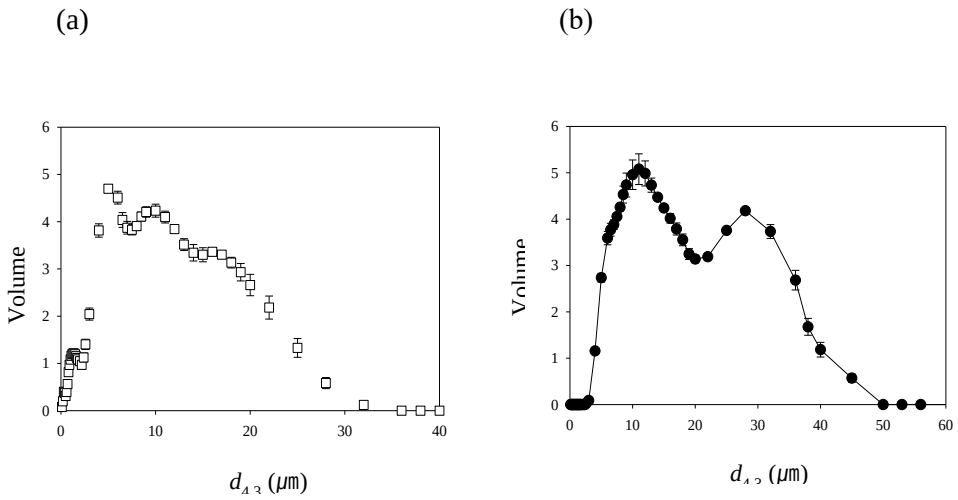


Fig. 2.3. Particle distribution of (a) PE-MD and (b) PE-RMGA. PE-MD, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3. Experiments were performed in triplicate, and the results were presented as mean ± standard deviation.

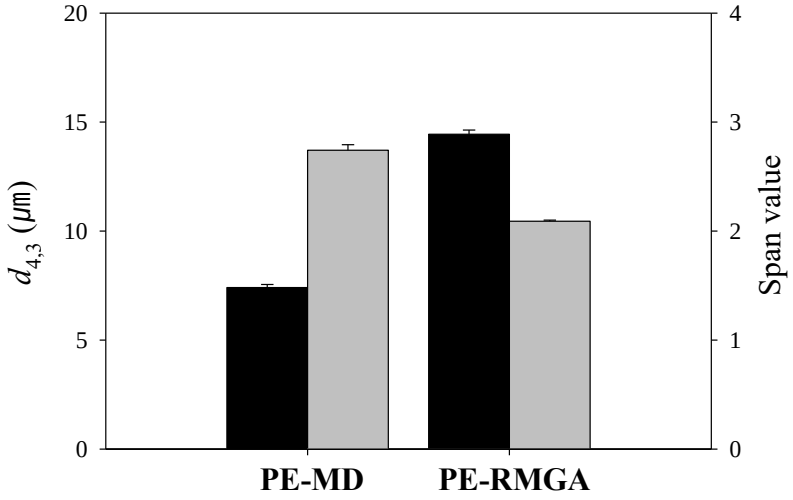


Fig. 2.4. Particle size ($d_{4,3}$, ■) and span value ( ) of PE-MD and PE-RMGA. Asterisks indicate significant differences between the values ($p \leq 0.05$). PE-MD, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3. Experiments were performed in triplicate, and the results were presented as mean ± standard deviation.

(a) (b)

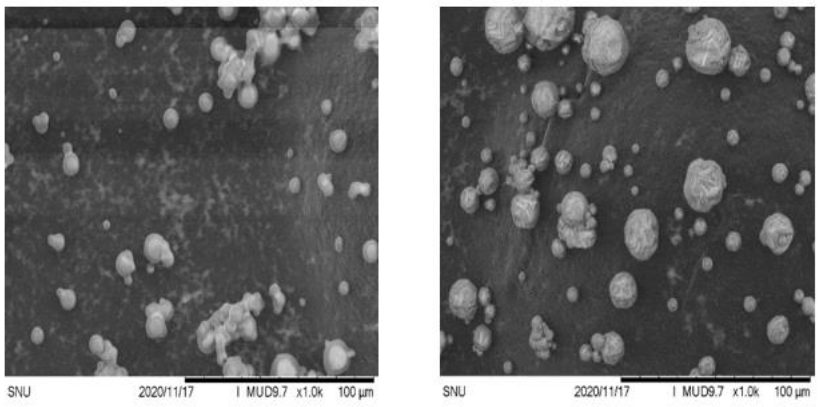


Fig. 2.5. Particle morphology (1000X) of (a) PE-MD and (b) PE-RMGA. PE-MD, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3.

Particle loose bulk density (ρ_L) of PE-MD was 0.35 g/cm^3, PE-RMGA was 0.51 g/cm^3 (Fig. 2.6). For PE-MD, porosity between particles was 56.2% (Fig. 2.7), so the ρ_L value measured low, and the difference ρ_L (0.35 g/cm^3) with tapped density (ρ_T) (0.63 g/cm^3) was big (Fig. 2.6). In case of PE-RMGA, the porosity (ε) was 50.4% (Fig. 2.7), so the ρ_L value was relatively high, and the difference between the ρ_L (0.51 g/cm^3) and ρ_T (0.70 g/cm^3) was small. Particle true densities (ρ_P) of PE-MD (1.44 g/cm^3), and PE-RMGA (1.41 g/cm^3) were similar (Fig. 2.6).

Spray drying inlet temperature was 140 ℃, at this temperature more agglomeration occurs, previous study indicated that porosity ε increased with increasing agglomerate mass (Deng et al., 2016) and the formation of these agglomerates may be due to the particles being dried at a slower rate, allowing stickiness for a longer time (Both et al., 2020).

Hausner ratio (HR) and carr index (CI) indicates powder flowability. In HR value, PE-MD and PE-RMGA calculated 1.80 and 1.37, and CI value resulted 44.44% and 27.14%, respectively (Fig. 2.8). Flowability of PE-MD was very very poor, and PE-RMGA was poor according to Table. 2.1. Both had poor flowability due to their small particle size, which were corresponding to large surface area, and strong the inter particle interactions, such as van der Waals, capillary and electrostatic forces with respect to particle weight (Fulchini et al., 2017). In case of PE-MD, their flowability was lower than that of PE-RMGA, because PE-MD had high hygroscopicity and porosity, so their particles tended to form agglomerates, rather free flowing properties.

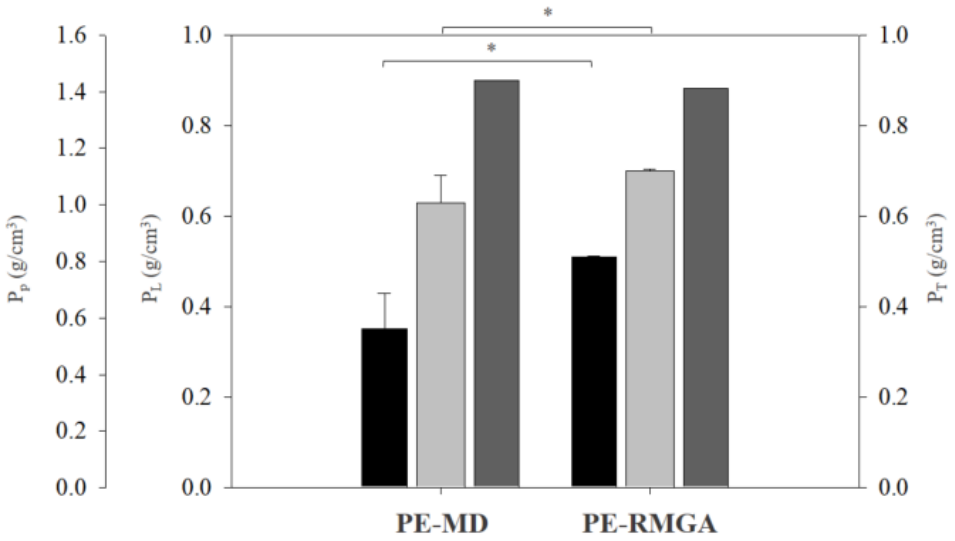


Fig. 2.6. Particle loose bulk density ($\rho_{L,}$ ■), tapped bulk density ($\rho_{T,}$ ■), true density ($\rho_{P,}$ ■) of PE-MD and PE-RMGA. Asterisks indicate significant differences between the values ($p \leq 0.05$). PE-MD, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3. Experiments were performed in triplicate, and the results were presented as mean ± standard deviation, and asterisks indicate significant differences between the values ($p \leq 0.05$).

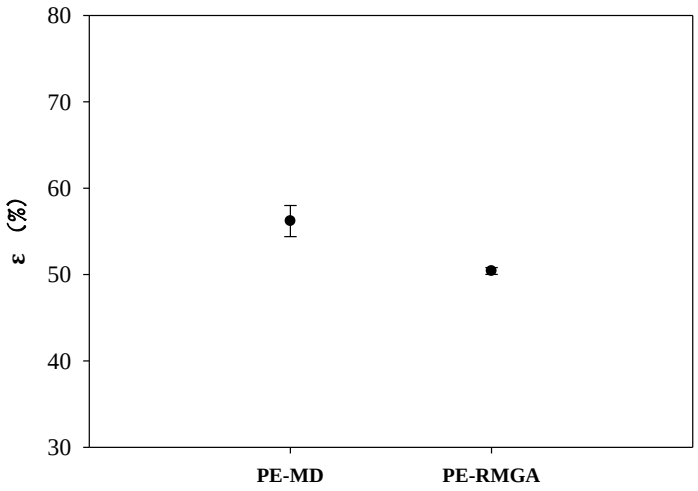


Fig. 2.7. Porosity (ε) of PE-MD and PE-RMGA. PE-MD, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3. Experiments were performed in triplicate, and the results were presented as mean ± standard deviation.

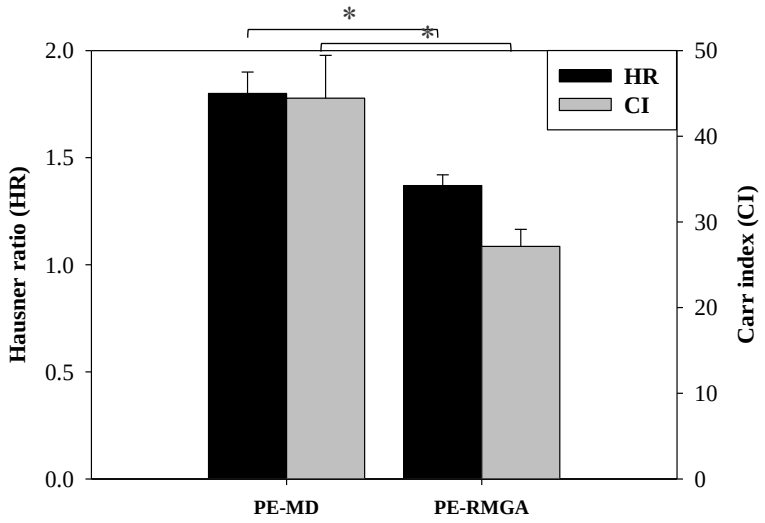


Fig. 2.8. Hausner ratio (HR) and Carr index (CI) values of PE-MD and PE-RMGA. Asterisks indicate significant differences between the values ($p \leq 0.05$). PE-MD, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3. Experiments were performed in triplicate, and the results were presented as mean ± standard deviation, and asterisks indicate significant differences between the values ($p \leq 0.05$).

In addition to PE-MD and PE-RMGA, sugar was also considered in case of dispersibility and solubility. The reason for this is that the sweetening ability and the ability to lower the glycemic index in functionality of PE-MD and PE-RMGA were compared with sucrose in Chapter 3, similar to that of reconstitution properties were compared with sucrose.

The initial dispersibility of sugar had the highest value of 85.33%, which over time the value of solubility was 79.33%. In comparison, dispersibility of PE-MD and PE-RMGA 55.02%, and 78.32%, and solubility were 86.67%, and 81.67% (Table. 2.5), respectively. The spray-dried powder had smaller particle size, high surface area of particles, also resulted in agglomeration at initial stage of reconstitution, and a higher dissolution rate due to the small particle size of spray-dried products resulting in higher porosity and poor flowability (Lin & Kao, 1989). For PE-MD and PE-RMGA powders, the small particle size initially tends to form agglomerates, which is considered to be low in dispersibility but high in final solubility due to wider surface area. Spray-dried powder was amorphous, while sucrose is crystal form. This difference is often attributed to the higher entropy and internal free energy of the metastable amorphous material, leading to enhanced solubility and chemical reactivity, relative to the thermodynamically more favorable and stable crystalline state.

Table. 2.5. Dispersibility and solubility of PE-MD, PE-RMGA, and sugar.

Powders	Dispersibility (%)	Solubility (%)
PE-MD	55.02 ± 0.82	86.67 ± 0.47
PE-RMGA	78.32 ± 0.82	81.67 ± 0.47
Sugar	85.33 ± 0.47	79.33 ± 0.47

PE-MD, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, spray-dried powder with 10% (w/w). Experiments were performed in triplicate, and the results were presented as mean ± standard deviation.

The Brunauer-Emmett-Teller (BET) and Guggenheim-Anderson-de Boer (GAB) model are the most widely used model in food systems, and it represents a fundamental of multi-layer sorption isotherms, whereas Smith, Peleg, Oswin, Henderson, and Ferro-Fontan models are empirical models (Andrade et al., 2011; Siripatrawan & Jantawat, 2006). The most suitable model of PE-MD and PE-RMGA based on R^2, SE M_r was GAB to apply in both adsorption and desorption (Table. 2.6). In adsorption, for PE-MD, except for Oswin model, other models had the R^2 value was greater than 0.98, but GAB only had less than 10% M_r value, and for PE-RMGA, most models had the R^2 value was lower than 0.98 except for BET, Peleg, and Ferro-Fontan models, but for M_r value, GAB only had less than 10%, which were 4.9%. In desorption, for PE-MD, the R^2 value was 0.98 or higher in case of GAB, Peleg, Smith, and Ferro-Fontan models, of which the value of GAB, and Peleg models were lower than 10% M_r value, and for PE-RMGA, GAB, Peleg, and Henderson models had the suitable value of R^2, and for GAB alone had the 9.725% of M_r value, which was less than 10%. The GAB and BET have similar equation and are the most common models used for the determination of the sorption isotherms for different materials, but BET model is suitable to predict up to 0.55 a_w, GAB model provides up to at least 0.9 a_w (Alamri et al., 2018). Other studies have shown that fruits were best suited to the GAB model (Maroulis et al., 1988; Pedro et al., 2010).

Table. 2.6. Parameters of sorption models

Model	Parameter	Adsorption		Desorption	
		PE-MD	PE-RG	PE-MD	PE-RG
BET	X_0	0.033	0.032	0.035	0.035
	C	2602203.431	11491688.77	31735963.7	466619292.3
	R^2	0.881	0.892	0.688	0.637
	SE	0.0497	0.0481	0.081	0.091
	M_r (%)	42.5460	39.5236	72.020	75.831
GAB	X_0	0.241	0.112	0.131	0.221
	C	0.497	1.304	3.579	1.742
	K	0.769	0.857	0.8	0.692
	R^2	0.998	1.000	0.993	0.993
	SE	0.010	0.011	0.016	0.001
	M_r (%)	9.672	4.914	8.237	9.725
Peleg	K_1	11.297	0.397	12.844	0.440
	K_2	0.439	0.196	0.392	0.366
	n_1	75.064	1.807	74.387	1.374
	n_2	2.176	1.087	1.234	23.902
	R^2	0.996	0.954	0.999	0.997
	SE	0.026	0.076	0.027	0.013
	M_r (%)	60.995	29.818	9.947	11.453
Henderson	A	1.848	1.865	2.180	2.164
	B	0.467	0.472	0.730	0.740
	R^2	0.992	0.986	0.975	0.989
	SE	0.019	0.023	0.026	0.018
	M_r (%)	95.771	85.255	22.549	14.307
Smith	A	-0.018	-0.014	0.025	0.024
	B	-0.413	-0.402	-0.394	-0.405
	R^2	0.993	0.997	0.984	0.973
	SE	0.017	0.016	0.019	0.024
	M_r (%)	69.641	20.808	11.662	17.689
Oswin	A	0.105	0.104	0.154	0.160
	B	0.574	0.569	0.432	0.425
	R^2	0.976	0.986	0.979	0.957
	SE	0.025	0.020	0.023	0.032
	M_r (%)	24.615	20.710	17.192	23.029
Ferro-Fontan	γ	0.444	0.074	0.247	0.371
	α	1.659	1110.34	1.448	3.302
	r	0.414	6.751	0.770	0.955
	R^2	0.998	0.947	0.986	0.989
	SE	0.518	0.177	0.120	0.089
	M_r (%)	67.838	205.921	36.442	38.257

When foods are spray dried, product's stability can be enhanced by reduction of the water activity, and moisture content, however, powdered products obtained from fruits are generally very hygroscopic (Santana et al., 2014), which may affect their physicochemical properties and even microbiological stability (Pombo et al., 2019). Hygroscopic property of persimmon powder was carried out by Moisture Sorption Isotherm (MSI), that described the relationship between the X_{eq} and its a_w, at 25 ℃. The sorption isotherm of PE-MD and PE-RMGA all showed type III behavior (Fig. 2.9 a and Fig. 2.10 a), which is characteristic of products rich in sugars (Pombo et al., 2019). Similar behavior was reported previously such as strawberry powder (Farahnaky et al., 2016), and orange juice powder (Oliveira et al., 2013). T_g is the point temperature which changes from the glassy state to the rubbery state (Ostrowska-Ligęza et al., 2014). Stickiness occurs above 10-20 ℃ higher than T_g (Ozmen et al., 2002; Silalai et al., 2010), so this parameter also could be used to predict powder stability during storage.

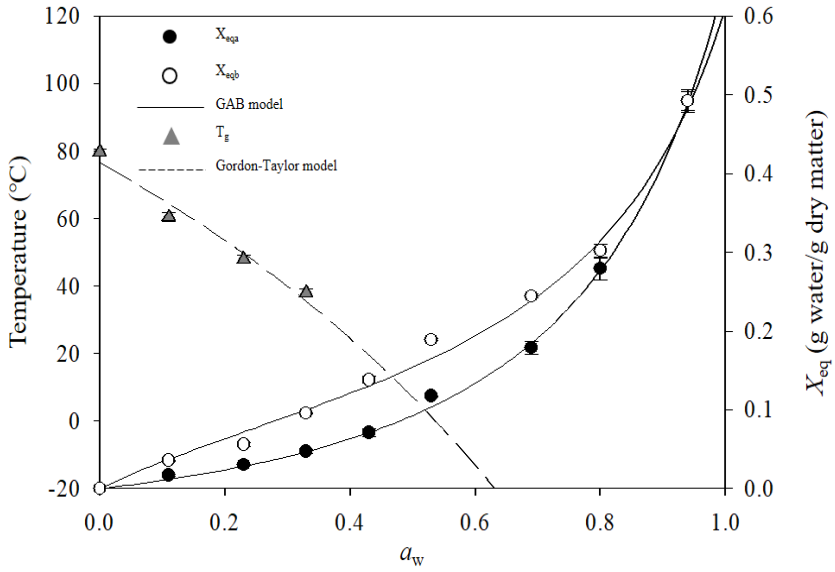
The adsorption isotherm of PE-MD presented linear behavior until 0.43 a_w, after this point, it showed exponential tendency with a progressive increase of the powder's moisture content (Fig. 2.9 a). PE-RMGA had similar tendency (Fig. 2.10 a). The T_g results of PE-MD and PE-RMGA at 25 ℃ were aw 0.40, and a_w 0.43, respectively. Above a_w 0.43, it was possible to see caking and stickiness occurred and even more liquidized than the same powder form as 0.43 or less (Fig. 2.9 b and Fig. 2.10 b).

There was a difference in the amount of water sorption depending on α_w, and it was attributed to hysteresis. When molecules get adsorbed layer by layer,

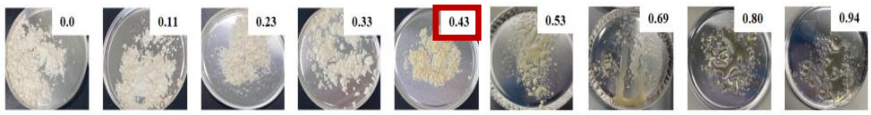
they fill higher energy sites near pore wall then low energy sites away from wall, molecules accumulated on two opposing walls get close enough to each other. When pressure reduction during desorption, because the molecules at low energy do not tend to leave their place, to pull the adsorbed molecules out of their sites, it must have higher gradient of chemical potential or equivalently pressure drop (Fig. 2.9 c and Fig. 2.10 c). This gap between equilibrium adsorption and desorption pressures is the hysteresis (Fig. 2.9 a and Fig. 2.10 a).

There was little sorption difference between PE-MD and PE-RMGA, both have GAB model and start point of glass transition 25 ℃ were about a_w 0.43, because that the persimmon extract itself had a very high sugar content of 70 brix, and when it is made as feed suspension, it contained high ratio of 30% drying aids which all had hydrophilic structures. According to these behaviors, the produced persimmon powder requires greater care, when handled and/or stored above 40% of RH, due to caking, liquefaction, degradation, and so on. It is recommended that the persimmon powder should be stored in a packaging impermeable to water and air, which does not allow the light to pass through.

(a)



(b)



(c)

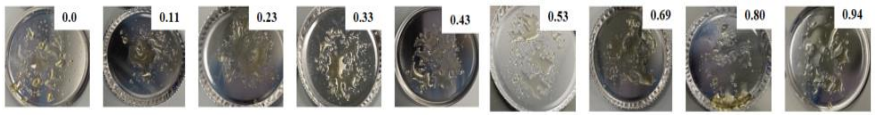
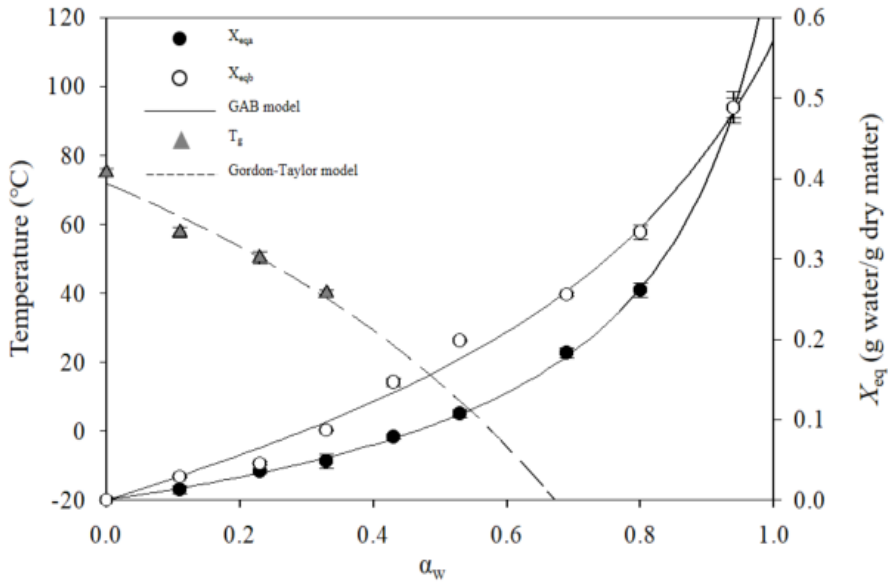
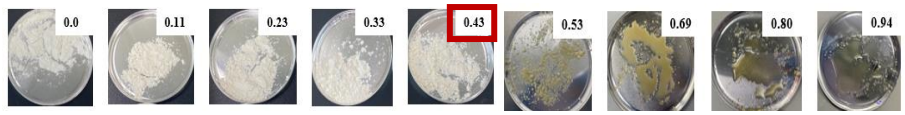


Fig. 2.9. (a) Glass transition temperature (T_g) with moisture sorption isotherm of PE-MD, (b) powder appearance of PE-MD in adsorption at each a_w, and (c) in desorption at each a_w PE-MD, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution. Experiments were performed in triplicate, and the results were presented as mean ± standard deviation.

(a)



(b)



(c)

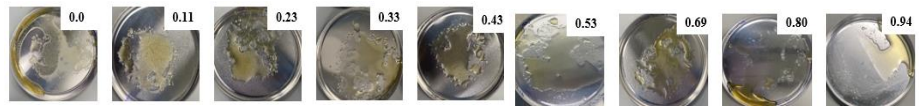


Fig. 2.10. (a) Glass transition temperature (T_g) with moisture sorption isotherm of PE-RMGA, (b) powder appearance of PE-RMGA in adsorption at each a_w, and (c) in desorption at each a_w. PE-RMGA, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3. Experiments were performed in triplicate, and the results were presented as mean ± standard deviation.

4. Conclusions

Limitation of fruits such as seasonality, storage loss, discarded waste issue, etc. were overcome by powderization. In addition, produced powder was much lighter than the fruit, it also had an advantage in handling.

RMD was used not only drying aid but also a prebiotics dietary fiber, and when mixed with GA at a ratio of 7:3, stickiness problem was overcome. The physicochemical properties such as moisture content, water activity, particle size and morphology, flowability, dispersibility, solubility, moisture sorption isotherm, and glass transition temperature were measured to check those properties and make sure suitability of PE-RMGA for use as a potential powder. Results of moisture content and water activity indicated that PE-RMGA was microbiologically safe. Particle size of PE-RMGA was about 14.4 μm, and had no agglomerates. PE-RMGA showed poor flowability with HR and CI were 1.37 and 27.14%, respectively, and lower dispersibility (73.32%) than sugar (85.33%), however, final solubilitdndy was 81.67%, which was higher than the sugar level (79.33%) due to small particle size, thus large surface area. When external forces such as moisture and pressure were applied to the powder, particles exhibited agglomerate formations due to the small particle size of PE-RMGA, resulted poor flowability and low dispersibility, but later solubilized better after 30 min because of the wide surface area. The moisture sorption isotherm showed that PE-RMGA had GAB model, contained high amount of sugar, resulted in hygroscopic properties and therefore difficult to store under above 43% RH conditions at room temperature.

Based on the results, the spray-dried powder had small size and poor flowability and thus could form agglomerates, resulted in a low dispersibility but high solubility due to its large surface area. It also had high hygroscopicity, caution is required in handling and storage area.

Chapter 3

Antioxidant and antidiabetic effects of persimmon-prebiotics powder and its application as a potential natural sweetener

1. Introduction

Fruits are health supplement because they contribute beneficial effects to human (Liu, 2003). Phytochemicals, which include carotenoids, and polyphenols help to resist virus or bacterial infections (Molyneux et al., 2007) resist LDL oxidation, reduce DNA damage (Seifried et al., 2003), and they contribute to antioxidant and antidiabetic properties. Among fruits, persimmon has antioxidant and antidiabetic effects due to polyphenolic compounds and vitamins (Butt et al., 2015; Lee et al., 2012). It contains high content of vitamin C (100 mg/100 g), and polyphenols (300-400 mg GAE/100 g).

Phenolic compounds divided into flavonoids, and non-flavonoids, and both of them and vitamin C have antioxidant properties. In addition to checking tannin, and vitamin C content, ABTS (2,2'-azino-bis (3-ethylbenzothiazoline-6-sulfonic acid) assays are the methods for the assessment of the antioxidant capacities.

Proanthocyanidins (condensed tannins) are a class of polyphenols, oligo- or polymers of flavan-3-ols, and these have antidiabetic properties by inhibition of α-amylase and α-glucosidase or inhibit aggregation of human islet amyloid

polypeptide and works as an anti-diabetic agent thus can prevent type-2 diabetes (Jiao et al. 2013).

The quality of food is determined by taste, aroma, touch, appearance, etc. Among them, the taste is felt through the sensation of the tongue. Electronic tongue (E-tongue) is a technology which is used to evaluate taste of foods, with partial sensitivity to different components. E-tongue mimics the function of the human tongue and discriminates between a variety of tastes such as sourness, saltiness, umami, sweetness, and aftertastes such as astringency, richness, etc. like human receptors, the sensors of an e-tongue detect the chemical compounds and generate electric signals as potentiometric variations (Fig. 3.1).

When the taste material is attached to the sensor surface, it could be detected by changing the potential difference (Zhang et al., 2015), so if the substance has no potential, it is difficult to measure. Artificial sweeteners are non-potential substances, it is impossible to measure, sugars and natural sweeteners are all possible. For similar reasons, pH sensitivity is important factors because it shows different taste results depending on pH.

In this study, the objective is to evaluate antioxidant properties by measuring tannin, vitamin C, flavonoid content, and ABTS assay, antidiabetic effects by assessment proanthocyanidins, and comparing sweetness with sugar in order to know whether it can be used as a potential natural sweetener.

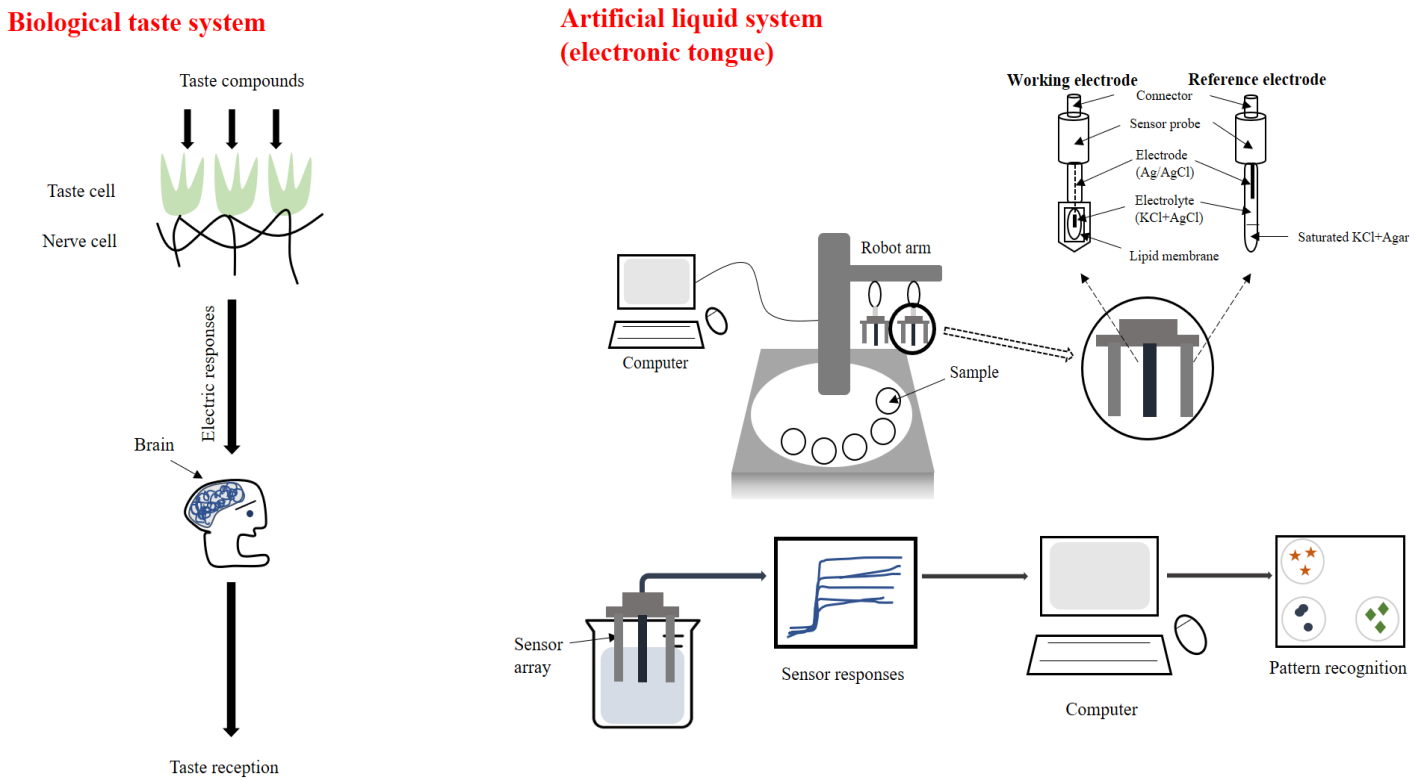


Fig. 3.1. Schematics of biological taste system and electronic tongue

2. Materials and methods

2.1. Materials

Persimmon powder was prepared by previously described in Section 2.1, 2.3, and 2.4 of Chapter 2. Gallic acid, 97.5-102.5% vitamin C, 99% and (+)-catechin hydrate, 98% standard were purchased from Sigma (Sigma Aldrich, Darmstadt, Germany). Vanillin, 99%, pure was purchased by Acros (Acros Organics, Massachusetts, USA).

2.2. Determination of total phenolic compounds

Total phenolics of persimmon extract, PE-MD and PE-RMGA were estimated by folin-ciocalteu's phenol reagent method (Singleton & Rossi, 1965) with some modifications. Standard material was gallic acid because it is inexpensive, easily soluble in water, recrystallized easily from water, readily dried, and stable in dry form. Before measuring sample, standard solution was prepared (Singleton et al., 1999). Approximately 50 mg of gallic acid were weighed and then dissolved in 50 mL flask with deionized distilled water (DDW), and diluted 100 mg/L gallic acid solution with DDW to 100 mg/L gallic acid. For drawing standard curve, 10, 30, 60, and 100 mg/L of gallic acid were prepared. After making standard solution, 2.6 mL of DDW (2.8 mL for blank) were prepared in 9 mL of test tube, and then 200 μL of properly diluted sample were added to each test tube and vortexed. In case of PE-ME and PE-RMGA,

filtrations were conducted using Acrodisc LC13 PVDF 0.45 μm (Gelman, USA) to examine the exact color without viscosity. As soon as 200 μL of folin-ciocalteu's phenol reagent were added to each test tube, vortexed, and waited 6 minutes. To finish the reaction, 2 mL of 7% Na_2Co_3 solution were added to each tube and vortexed again. After 90 minutes at room temperature (23 ℃), read absorbance using spectrophotometer (SPECTRONIC 200, Thermo Fisher Scientific, Middlesex County, MA, USA) at 750 nm versus prepared blank.

2.3. Determination of proanthocyanidin contents

Proanthocyanidin contents in persimmon extract, PE-MD, and PE-RMGA were measured by the vanillin-H_2SO_4 assay described by Cáceres-Mella et al. (2013) with some modifications. To 1 mL of sample solution (made by extract, PE-MD, and PE-RG) or reference standard solution, 2.5 mL of 1% (w/v) vanillin solution in methanol (sample) and 2.5 mL of a 1:3 v/v H_2SO_4/methonol were mixed. The mixture was incubated at 30 ℃ for 30 minutes and the absorbance at 500 nm was measured. A blank was prepared by substituting the vanillin solution in the reaction mix with methanol. The absorbance of the blank was subtracted from the absorbance of the corresponding vanillin-containing sample, read using spectrophotometer (UV-1800, Shimadzu Scientific Instruments Incorporated, Kyoto, Japan) at 500 nm versus prepared blank. The proanthocyanidin contents were expressed as mg catechin equivalent per 100 g (mg CTE/100 g).

2.4. Determination of vitamin C contents

Vitamin C content was measured according to Jeong et al. (2020). The HPLC system was Alliance e2695 (Waters, Milford, MA, USA). The stationary phase was an Agilent Zorbax Eclipse XDB-C18 column (5 μm of particle size, 4.6 mm i.d. x 250 mm; Agilent Technologies, Inc.). The flow rate was 0.8 mL/min, and the temperature was 30 ℃. Mobile phases were comprised of two solvents: 0.1% (v/v) formic acid in water (solvent A) and 0.1% (v/v) formic acid in acetonitrile (solvent B). The injection volume was 10 μL, and the detection wavelength was set to 254 nm. Vitamin C content of persimmon extract was quantified using the vitamin C standard curve.

2.5. DPPH/ABTS assay

To examine antioxidant properties of persimmon extract, PE-MD, and PE-RMGA, DPPH and ABTS assays were conducted according to Berg et al. (1999); Brand-Williams et al. (1995) using spectrophotometer (SPECTRONIC 200, Thermo Fisher Scientific, Middlesex County, MA, USA). The standard material was vitamin C. The principle of this method is to determine the radical scavenging activity of specific compounds which are allowed to react with DPPH and ABTS.

$$DPPH^{\bullet} + AH \rightarrow DPPH{:}H + A^{\bullet}$$

$$ABTS^{-} + BH \rightarrow ABTS{:}H + B^{-}$$

The reduction of ABTS is monitored using spectrophotometric techniques by the decrease of absorbance at 517 nm and 734 nm, respectively.

For $DPPH^{\bullet}$ scavenging activity, First, 0.1 mM of $DPPH^{\bullet}$ solution was prepared in 80% methanol (MeOH) (7.89 mg $DPPH^{\bullet}$/200 mL of 80% MeOH). Next, methanolic DPPH radical solution was waited for 20 minutes with stirring. The absorbance of $DPPH^{\bullet}$ solution was adjusted at 0.650 ± 0.020 with 80% methanol at 517 nm. Blank for the reference was the mixture of 0.1 mL of 80% MeOH and 2.9 mL of DPPH• radical solution. 0.05 mL of tested extracts or standard antioxidant solution (vitamin C solution) were added to 2.95 mL of methanolic $DPPH^{\bullet}$ solution. After that, the mixture was shaked and left to stand in the dark at 23 ℃ for 30 min, and decrease in absorbance of the resulting solution was measured by spectrophotometer at 517 nm at 30 min as the end-point.

ABTS assay was similar with DPPH assay. First of all, filtration was conducted with Acrodisc LC13 PVDF 0.45 μm (Gelman, USA) and diluted the $ABTS^{\bullet-}$ (stable oxidized free radical) solution between 0.650 ± 0.020 of the absorbance at 734 nm using phosphate buffered saline (PBS). After that, 980 μL of the radical solution were mixed with 20 μL of the sample, and the solution was incubated in 37 ℃ for 10 min. The vitamin C equivalents antioxidant capacity (VCEAC) was calculated by using the reduction in absorbance at 734 nm for 10 min.

For reference, PBS was made by 3 steps; 1) 10 mM (34.836 g/2 L) K_2HPO_4 and 150 mM (17.532 g/2 L) NaCl were prepared in 2 L volumetric flask using DDW similar with 1), 100 mM (6.8045 g/500 mL) KH_2PO_4 and 150

mM (4.383 g/500 mL) NaCl were prepared in 500 mL volumetric flask using ddH2O. 3) pH meter was used to calibrate. 4) pH meter probe was submerged into 2 L of K_2HPO_4/NaCl solution and stirring was performed. 5) pH of mixed solution (poured with mixing) which were KH_2PO_4/NaCl solution and 2 L of K_2HPO_4/NaCl solution was reached 7.40 (About 370 mL of KH_2PO_4/NaCl solution was poured), and solution was stored at 4 ℃.

The effect in % of inhibited reaction was expressed at 30 min, and radical scavenging activity inhibition was calculated:

$$\% \text{ Inhibition } = \frac{A_{reference} - A_{sample}}{A_{reference}} \times 100 \quad (16)$$

$A_{reference}$: the absorbance of the blank sample

A_{sample}: the absorbance of the tested sample at 30 min

2.6. Determination of sugar contents

To determine sugar content of persimmon extract, PE-MD, and PE-RMGA, high performance liquid chromatography (HPLC) was performed. Two system was organized; HPLC method with evaporative laser scattering detection (HPLC-ELSD), and HPLC method with refractive index detection (HPLC-RI), which are difference in detector. HPLC-ELSD value is more accurate than HPLC-RI for monosaccharides (Clement et al., 2006), and also HPLC-RI has

limitation when using gradient elution and changing the temperature. For HPLC-ELSD (Agilent 1100 series, Santa Clara, USA), standards such as arabionose, xylose, galactose, glucose, fructose, sucrose, maltose, and lactose were purchased from Sigma Aldrich (Sigma Aldrich, Darmstadt, Germany), and ELSD detector was from Alltech (Alltech, ELSD2000, USA). For sample preparation, persimmon extract was diluted about 50 times with DDW and filtered using syringe filter (Polyvinylidene fluoride (PVDF), 13MM, 0.45 μm), and samples were diluted (60 mg/ml) with DDW and filtered using syringe filter (PVDF, 13MM, 0.45 μm). Column system for HPLC-ELSD consisted of a 25 cm x 4.6 mm, 5 μm particle size packed with VG-50 4E (Shodex, Tokyo, Japan). Flow rate was 1 mL/min, gas flow was 1.9 L/min, column and ELSD operated at 60 ℃, and 90 ℃, respectively, and mobile phase was acetonitrile/distilled water/methnol. For HPLC-RI (Dionex Ultramate 3000, Thermo Scientific, MA, USA), RI detector (RI-71, Shodex, Tokyo, Japan) was used, and all standards (glucose, galactose, fructose, maltose hydrate, and maltotriose) were purchased from Sigma Aldrich (Sigma Aldrich, Darmstadt, Germany). Sample preparation was performed by diluted 50 times with DDW and filtered with syringe filter (PVDF, 13mm 0.45 μm), and the column system was TSKgel column (Tosoh, Tokyo, Japan) for consisted of 30 cm x 7.8 mm, 7 μm particle size, for size exclusion column. Operating condition were flow rate was 0.5 mL/min, column operated at 78 ℃, and mobile phase was distilled water.

2.7. Electronic tongue analysis

E-tongue (TS-5000Z, Insent, Tokyo, Japan) was used to compare the taste of sugar with persimmon extract, PE-MD, and PE-RMGA. The principle of e-tongue (Hayashi et al., 1999; Oohira et al., 1995) (1) measure reference potential (*V*r) in each reference solution. (2) measure electric potential (*V*s) in sample to check initial taste (Vs-Vr) (3) wash in reference solution (4) measure electric potential (*V*r') in reference solution again to confirm aftertaste (*V*r'-*V*r) (5) refresh sensor in alcohol solution (Fig. 3.2). Reference solution is composed of 30 mM KCl and 0.3 mM tartaric acid and is regarded as having almost no taste. For measuring, each taste has its own sensor, and a reference solution. In order to measure the sweetness, unlike other tastes, the measurement sensor is for a single-use and the solution for preconditioning must also be purchased separately. Other tastes except sweetness can be recycled and the preconditioning solution is also different. Sensors are made of lipid membranes, and the sugar can melt, so the sweetness is measured separately and the sensor is disposable. Another difference is that other taste are measured at once except for sweetness, however in case of sweetness, only one sensor should be attached separately and the measurement should be carried out using negative solution only.

Each extract, PE-MD, PE-RMGA, and sugar 10 g were mixed into 30 g of DDW to form 25% (w/w) solutions. Due to pH sensitivity of the machine, pH of extract, PE-MD, PE-RMGA, and sugar solution were measured and the results were about pH 4.8 for extract to PE-RMGA solution, and sugar solution

was pH 6.8. Based on sample pH (pH 4.8), the pH of the sugar solutions was set 3.8, 4.8, 5.8 and 6.8, and pH 6.8 sugar solution was control. E-tongue measurement was divided only for sweetness, and the other tastes due to characteristics of the device.

Before operating E-tongue except for sweetness, preparing solutions and preconditioning processes were conducted as shown in Table 3.1. For preconditioning, internal solution, which is for making similar to a human lipid membrane and reference solution, which is for controlled state of taste were made. Internal solution was made by following method: adding 248.2 g potassium chloride mixed with 900 mL DDW and stirred, transferred to a 1 L volumetric flask and filled to the 1 L with DDW, and finally added about 10 mg silver chloride and stirred for at least 8 h. Reference solution was made as follows: 0.045 g tartaric acid was dissolved in 900 mL of DDW, 2.24 g potassium chloride was mixed, stirred and filled to the 1 L volumetric flask with DDW.

For measuring, 3.33 M KCl solution, negatively charged membrane washing solution, positively charged membrane washing solution were made. The method for making KCl solution was similar with internal solution, 248.2 g potassium chloride was mixed with 900 mL DDW, stirred, and trasnfered to a 1 L volumetric flask and filled with DDW to the 1 L.

For negatively charge membrane washing solution, 300 mL ethanol was mixed with 500 mL DDW, and 100 mL of 1 M hydrochloric acid, stirred and transferred to a 1 L volumetric flask and filled to the 1 L mark with DDW.

Positively charged membrane washing solution was produced 7.46 g

potassium chloride with 500 mL DDW, and 300 mL ethanol. After that, 10 mL of 1 M potassium hydroxide solution was added, fully stirred together, and to a 1 L volumetric flask, and filled to the 1 L with DDW.

Each standard sample solution, salty-sample, sour sample, umami sample, bitter-(+)-sample, and bitter-(-)-sample solution were also made. Salty sample was made with 0.045 g tartaric acid, 900 mL DDW, and 22.37 g potassium chloride, stirred together, transferred and additionally quantified to 1 L with DDW. For sour sample, 0.45 g tartaric acid was mixed with 900 mL DDW and dissolved, 2.24 g potassium chloride was additionally stirred together and quantified to 1 L volumetric flask mark with DDW. Umami sample was made similar with salty sample, 0.045 g tartaric acid, 900 mL DDW, and 2.24 g potassium chloride were mixed, additional 1.87 g monosodium glutamate (MSG) was dissolved, and transferred to a 1 L volumetric flask and filled to the 1 L mark with DDW. Bitter-(+), bitter-(-), astringent sample solutions had same process with umami sample to adding 0.045 g tartaric acid, 900 mL DDW, 2.24 g potassium chloride. For bitter-(+)-sample, additionally, 0.04 g quinine hydrochloride was added and quantified to 1 L volumetric flask mark with DDW, bitter-(-)-sample, 100 μL iso-α-acid was additionally added, and also quantified to 1 L mark with DDW, and for astringent-sample, extra 0.50 g tannic acid was dissolved, and transferred to a 1 L volumetric flask and quantified with DDW.

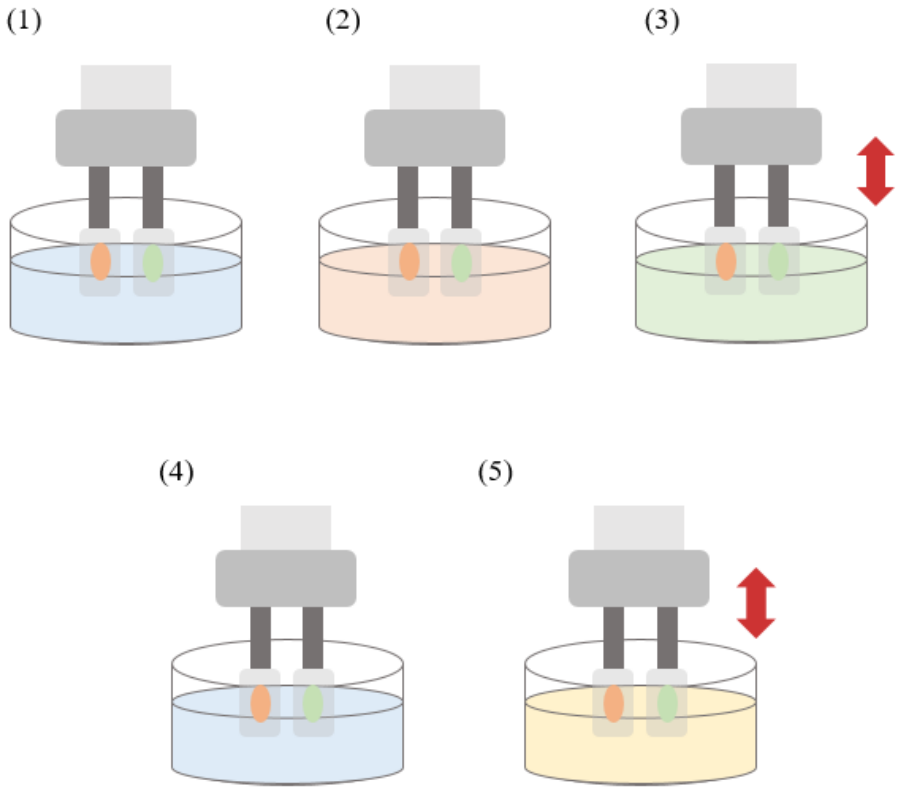


Fig. 3.2. Measurement flow of E-tongue. (1) measure reference potential (Vr) in reference solution, (2) measure electric potential (Vs) in sample (Vs-Vr = initial taste), (3) lightly wash together in reference solution, (4) measure electric potential (Vr') in reference solution again (Vr'-Vr = after taste), and (5) refresh sensor in alcohol solution

Table. 3.1. Comparisons of solutions used for E-tongue analysis

Classification of solution	Required materials
Reference solution	0.045 g tartaric acid 2.24 g potassium chloride 900 mL to 1 L DDW
Internal solution	248.2 g potassium chloride 10 mg silver chloride 900 mL to 1 L DDW
KCl solution	248.2 g potassium chloride 900 mL to 1 L DDW
Negatively charged membrane washing solution	300 mL ethanol 500 mL DDW 100 mL of 1 M hydrochloric acid solution
Positively charged membrane washing solution	7.46 g potassium chloride 500 mL DDW 300 mL ethanol 1 M potassium hydroxide solution
Salty sample	0.045 g tartaric acid 22.37 g potassium chloride 900 mL to 1 L DDW
Sour sample	0.45 g tartaric acid 2.24 g potassium chloride 900 mL to 1 L DDW
Umami sample	0.045 g tartaric acid 2.24 g potassium chloride 1.87 g monosodium glutamate 900 mL to 1 L DDW
Bitter-(+)-sample	0.045 g tartaric acid 2.24 g potassium chloride 0.04 g quinine hydrochloride 900 mL to 1 L DDW
Bitter-(-)-sample	0.045 g tartaric acid 2.24 g potassium chloride 100 μL iso-α-acid 900 mL to 1 L DDW
Astringent sample	0.045 g tartaric acid 2.24 g potassium chloride 0.50 g tannic acid 900 mL to 1 L DDW

Taste sensors for each taste (saltiness, sourness, umami, bitterness, and astringency, as shown in Fig. 3.3 (a), consist of sensor probe body, artificial lipid membrane, Ag/AgCl electrode, internal solution (3.33 M KCl potassium chloride with saturated silver chloride), and pin jack type electrode terminal. Before starting measurement, taste sensor preconditioning was performed; pin jack electrode terminal was removed from the sensor probe, and 200 μL of the internal solution was injected into the sensor probe body. Reference solution was poured into a beaker and taste sensor was soaked that the pin jack type electrode terminial faced up, covered with para-film, and waited about 24 hours. If the sensor probe was turned on its side or upside down, it might cause air bubbles resulting abnormal results.

Ceramic reference electrode was shown in Fig. 3.3 (b), comprised of a glass tube, Ag/AgCl electrode, internal solution (3.33 M potassium chloride with saturated silver chloride), and pin jack type electrode terminal. Likewise taste sensor, preconditioning was performed by removing pin jack type electrode terminal, injecting the internal solution into glass tube until the solution reaches about 5 mm from the upper end of the glass tube, poured into 3.33 M KCl solution, and covered with para-film about 24 hours.

After preconditioning, each taste sensors and ceramic reference electrodes were attached to the sensor head (Fig. 3.4), and measurement was conducted by moving sensor arm automatically first measuring between reference solutions and control sample groups, and finally replaced and measured between extract, PE-MD, PE-RMGA, sugar solution with different pH and reference solutions.

For sweetness measurement, same preconditioning was performed but using GL1 special solution which was substituted for 3.33 M KCl solution. After preconditioning, sweetness sensor and ceramic reference electrode was attached to the sensor head at positively charged membrane washing solution part. Sweetness sensor was measured only positively charged membrane washing solution part, and other tastes were measured using both positively and negatively charged membrane washing solutions (Fig. 3.4). In case of sweetness sensor (GL1 sensor), five cycle measurement was performed, but the first and second data were removed, and the remaining data were analyzed, other taste sensors were measured four times and the first cycle data was excluded.

(a) (b)

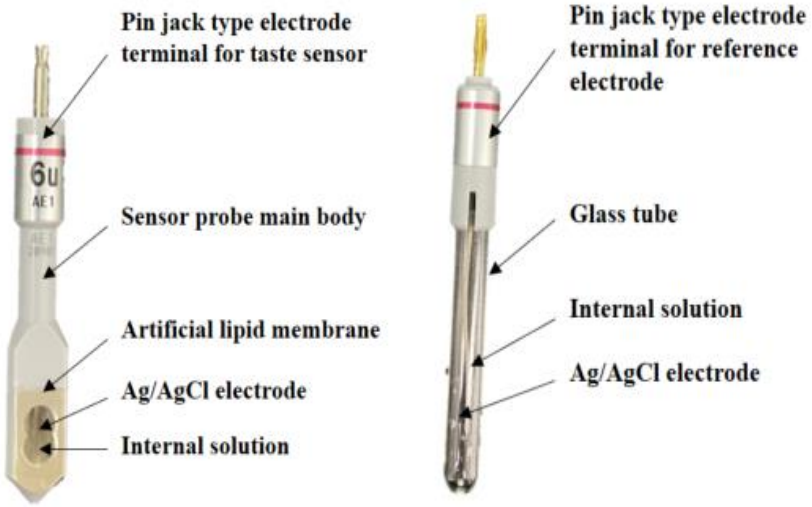


Fig. 3.3. (a) Taste sensor and (b) ceramic reference electrode for E-tongue

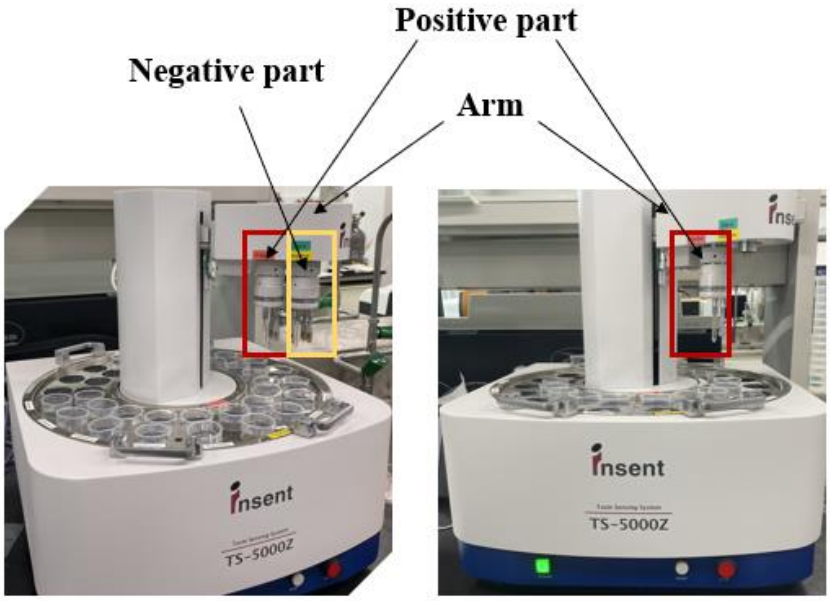


Fig. 3.4. Parts of E-tongue

2.8. Statistical analysis

All experiments were performed in triplicate, and the results were presented as mean ± standard deviation (SD). Statistical process of the experimental results was analyzed using SPSS 26 program (SPSS Inc., Chicago, IL, USA). Significant differences ($p \leq 0.05$) were determined by T-test using SPSS 26.

3. Results and discussion

The phenolic compounds are vital factors affecting radical scavenging and metal chelating activities and, hence, it is referred to as having antioxidant effects (Bei et al., 2005). The high correlation of total phenolic compound contents (TPC) and proanthocyanidin contents (PAC) (R^2=0.9998, and 0.9997, respectively) was found (Fig. 3.5). TPC of Cheongdo bansi is 402.1 ± 38.1 mg GAE/100 g dry weight (Jeong et al., 2018), as shown in Fig. 3.6, 169.39 ± 11.37 mg GAE/100 g dry weight of TPC was included in persimmon extract. The reduction of value might be due to heat sensitivity of flavonoid such as procyanidins (Chaaban et al., 2017; Ioannou et al., 2012). During extraction process, heating at 50 ℃ for 5 h and 90 ℃ for 15 min were conducted, so sensitive flavonoids might be destroyed. Other fruits such as apple (47.26 GAE/100 g dry weight), grape (93.88 ± 0.04 GAE/100 g dry weight), and tomato (21.24 ± 0.04) (Chen et al., 2008) have lower TPC that of persimmon extract. However, according to Zucoloto et al. (2015), Gold-Rush, a kind of apple, had 190 mg GAE/100 g dry weight, which value is higher than persimmon extract. Of course, it depends on the fruit, and even if it is the same fruit, the value of TPC may vary depending on the species. TPC of PE-MD and PE-RMGA were 62.11 ± 9.32, and 105.64 ± 13.20 mg GAE/100 g dry weight, respectively, and the retentions of TPC from extract were about 37%, and 62%, respectively. Spray-dried powder had low TPC than persimmon extract due to thermal treatment in which inlet temperature was 140 ℃ even if the process was short time, and when comparing PE-MD and PE-RMGA, stable emulsion

was produced by GA, so the produced powder might have more high retention of bioactive compound (PE-RMGA).

PAC of persimmon extract was 119.54 ± 1.30 mg CTE/100 g (Fig 3.6), which is about 70% of TPC. According to Denev et al. (2013), proanthocyanidins in persimmon are 1/2~1/3 of the total polyphenols, so PAC in persimmon extract is higher than average value. PAC of PE-MD and PE-RMGA were 38.32 ± 1.01, and 58.78 ± 2.02 mg CTE/100 g, respectively (Fig. 3.6), the percentage of each PAC in TPC were 62%, and 56%. Among flavonoids, proanthocyanidins have heat stability (Rauf et al., 2019), so these compounds might not be highly destroyed during extraction process. However, lower PAC of PE-MD compared to PE-RMGA but higher PAC compared to the TPC ratio observed. The reason for this is predicted that the bioactive compound could have been more protected due to lower water content of PE-MD. Retention of PAC from persimmon extract to PE-MD and PE-RMGA were 32%, and 49%, respectively. Similar with TPC, GA have film-forming properties, so that high encapsulation yield could be produced. In addition, according to Zhang et al. (2007), bioactive compound retention was better when the drying aids were mixed, so encapsulation yield of PE-RMGA might be better than PE-MD. Proanthocyanidin have 20 times higher antioxidant properties than vitamin C and 50 times higher than vitamin E (Yaqub et al., 2016), in conclusion, persimmon extract, PE-MD, and PE-RMGA have antioxidant, antidiabetic (Jiao et al., 2013) properties and neuroprotective effects (Li et al., 2018; Stanciu et al., 2020).

(a) (b)

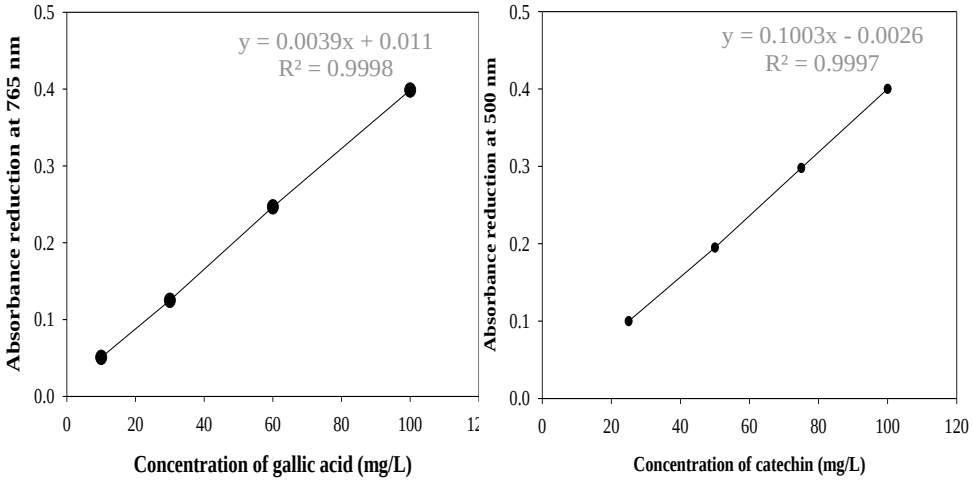


Fig. 3.5. Standard curve (a) gallic acid at 765 nm for TPC and (b) catechin at 500 nm for PAC. PE-MD, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3.

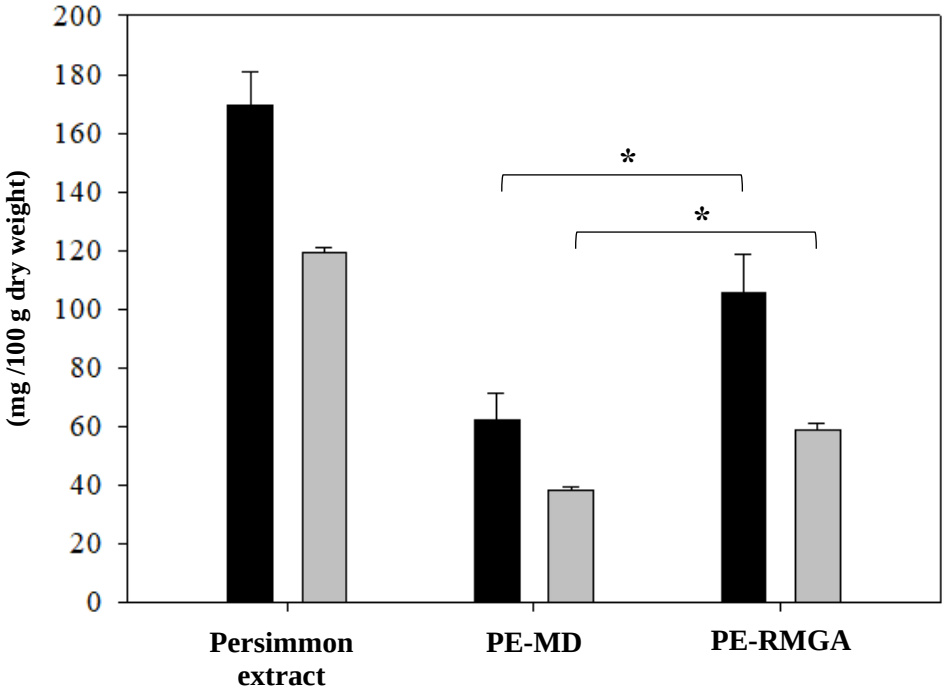


Fig. 3.6. TPC and PAC for persimmon extract, PE-MD and PE-RMGA. Asterisks indicate significant differences between the values ($p \leq 0.05$). TPC, total phenolic content; PAC, proanthocyanidin content; PE-MD, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3. Experiments were performed in triplicate, and the results were presented as mean ± standard deviation, and asterisks indicate significant differences between the values ($p \leq 0.05$).

Vitamin C content was investigated antioxidant properties by HPLC. Fig 3.7 indicates the results of the HPLC, (a) indicates vitamin C standard, (b) persimmon extract, and (c) indicates persimmon extract mixed with vitamin C standard. As shown in Fig. 3.7 (a) and (b), retention time values of standard and extract were found to be inconsistent. Mixing (a) and (b) together (Fig. 3.7 c), the spike input outcome showed that (a) and (c) were within the error range. In other words, extract was determined not to contain vitamin C, and the peak which located in front of was thought to be ghost peak. Usually, persimmon is rich in vitamin C, but vitamin C is so environmentally sensitive that it is destroyed by a heating, light, or even a little dip in water (Odriozola-Serrano et al., 2008). In the process of extractions of persimmon, the temperatures were 50 ℃ and 90 ℃, so it is believed that vitamin C of persimmon extracts had been eliminated.

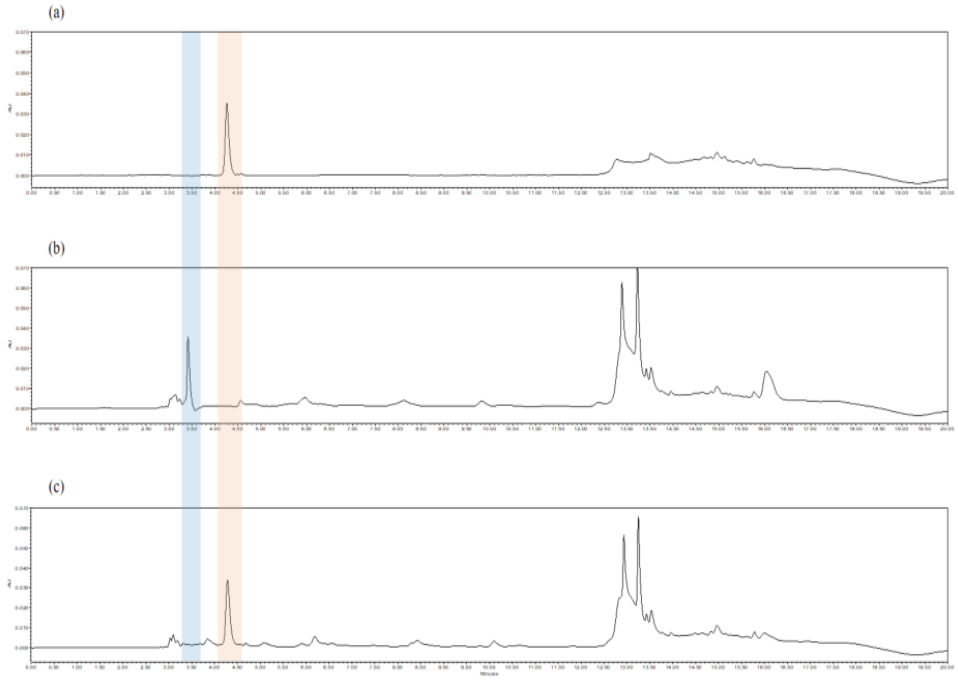


Fig. 3.7. Determination of vitamin C contents: (a) vitamin C standard, (b) persimmon extract, and (c) persimmon extract with vitamin C standard

DPPH/ABTS assays were for analyzing antioxidant effects by observing radical scavenging activity. Even after filtering samples were murky state so DPPH assay was impossible using spectrophotometer. Compared to ABTS assay, DPPH assay has some limitations (Kedare & Singh, 2011; Pekal & Pyrzynska, 2014): First, In DPPH, nitrogen (N) atoms are located in the middle of molecules, and unlike other antioxidants that react quickly with peroxy radicals, they may or may not react slowly to that (N) part. If the sample has small molecules, it is easy to access to DPPH, but if it is a large molecule, it is difficult to access the free radicals of DPPH. Both persimmon extract, PE-MD and PE-RMGA had high content of proanthocyanidin (Fig. 3.6), it is seem to have no DPPH results due to their high content of proanthocyanidin with large molecular weight. In addition, DPPH is pH sensitive, it can be lead to errors under acidic conditions.

ABTS assay was carried out in aqueous conditions. When applied to a variety of plant foods containing hydrophilic, lipophilic, and high-pigmented antioxidant compounds, ABTS assay is superior to DPPH assay (Floegel et al., 2011). R^2 value of this test was 0.9998 (Fig. 3.8), results of ABTS assay was reliable. Standard of ABTS assay was vitamin C. Persimmon extract contained about 139.42 ± 5.05 mg VCE/100 g dry weight (Table.3.2), PE-MD included about half the content (69.86 ± 13.15 mg VCE/100 g dry weight), and PE-RMGA was 82% content (113.75 ± 15.25 mg VCE/100 g dry weight) of persimmon extract. Previous study indicated that when using GA as a drying aid, the efficiency of the encapsulation process increased (Busch et al., 2017; Kuck & Noreña, 2016). Given that retention value of PE-RMGA was all higher

than PE-MD, it was considered to be the results of both poor emulsifying potential of MD (Samborska et al., 2021), and efficiency of mixing drying aids and film forming properties of GA.

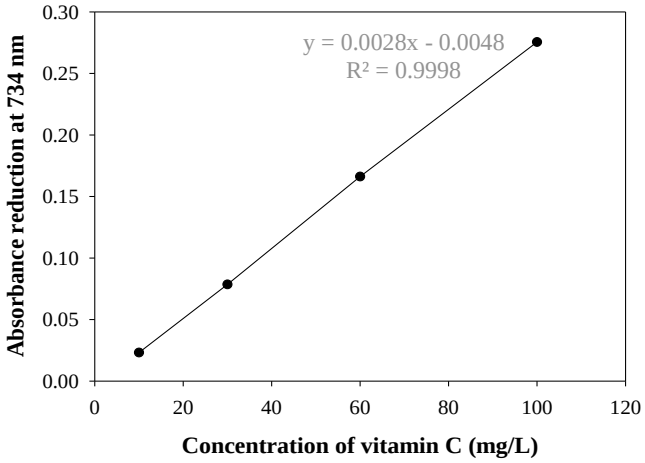


Fig. 3.8. Standard curve of vitamin C at 734 nm for ABTS assay

Table. 3.2. ABTS assay results of persimmon extract, PE-MD and PE-RMGA (mg VCE/100 g dry weight)

	mg vitamin C equivalent per 100 g (mg VCE/100 g dry weight)
PE	139.42 ± 5.05
PE-MD	69.86 ± 13.15
PE-RMGA	113.75 ± 15.25

PE, persimmon extract; PE-MD, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3. Experiments were performed in triplicate, and the results were presented as mean ± standard deviation.

HPLC-RI is the most popular method used for analysis of mono-, di- and polysaccharides from concentrated carbohydrate syrups (Givry et al., 2007), whereas HPLC-ELSD has been used for the simultaneous determination of fructose, sorbitol, glucose and sucrose in fruits (peach, apple, watermelon and cherry) (Ma et al., 2014), and for the qualitative and quantitative determination of glucose, fructose and sucrose in carbonated cola drinks and fruit juices (Li et al., 2007). Standard retention time of persimmon extract's HPLC-ELSD was shown in Table. 3.3 (a), and Fig. 3.9, and HPLC-RI was in Table. 3.3 (b). and Fig. 3.10. As shown in Fig. 3.11 and Table. 3.4, sugar of persimmon extract mainly consisted of fructose (48.8%), and glucose (47.2%). In case of HPLC-ELSD, total amount of sugar contents was about 55% in which 26.88 ± 0.19 g/100 g of fructose and, 26.02 ± 0.24 g/100 g of glucose. Proximate composition of persimmon extract (in Section 2.1) indicated about 70 g/100 g of composition was carbohydates, so 15% of the remaining components might be soluble dietary fiber such as pectins. HPLC-RI indicated sugar content was about 60.8%, and also could find that fructose and glucose were almost parts. Remaining would be also soluble dietary fiber, some organic acids, and minerals. On the other hand, there were little amount of sucrose, whereas most of components were glucose and fructose, which is corresponding to other fruits such as grapes (Orak et al., 2009).

Table. 3.3. Standard of sugars used for (a) HPLC-ELSD and (b) HPLC-RI

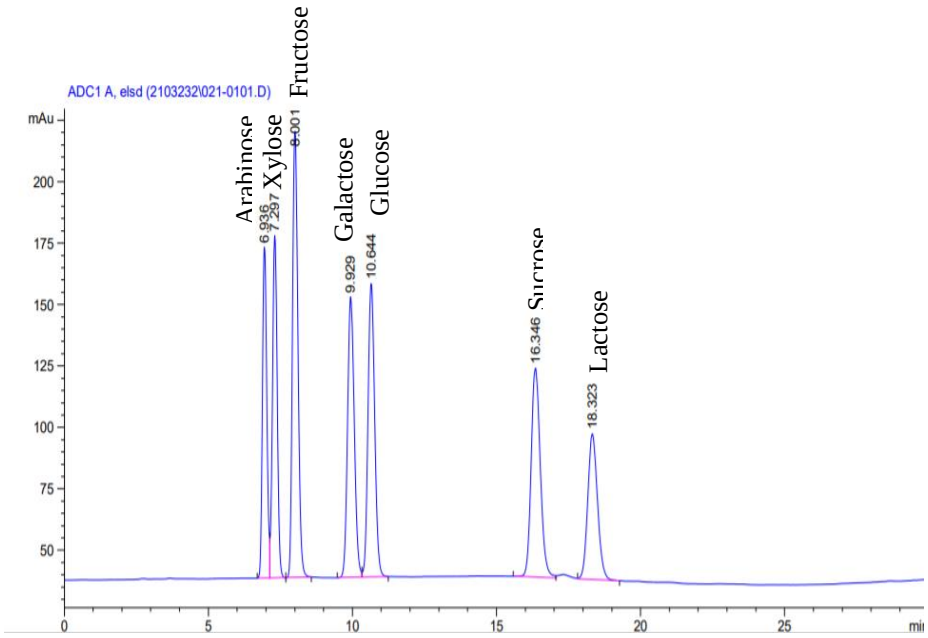
(a)

Peak name	Retention time (min)
Arabinose	6.939
Xylose	7.298
Fructose	8.001
Galactose	9.931
Glucose	10.645
Sucrose	16.346
Lactose	18.321
Maltose	19.588

(b)

Peak name	Retention time (min)
Glucose	16.445
Maltose	15.677
Maltotriose	15.053
Galactose	16.375
Fructose	16.878

(a)



(b)

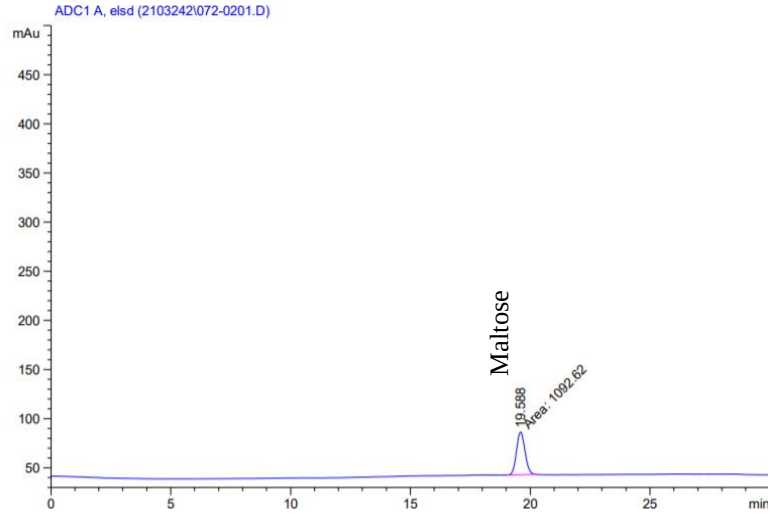
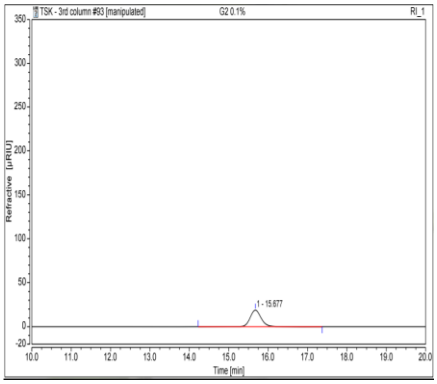
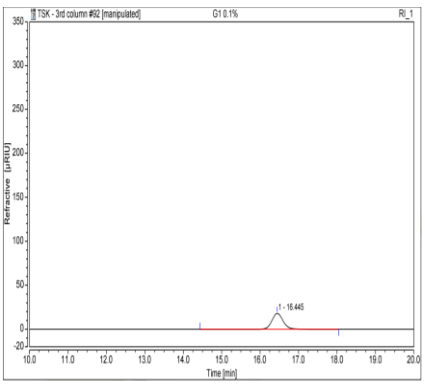


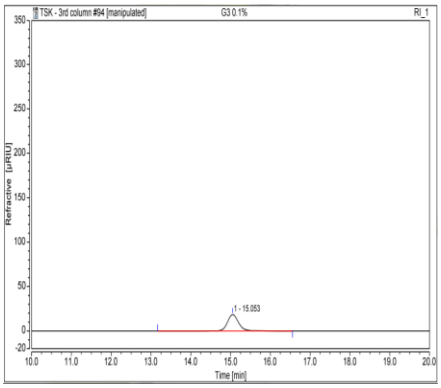
Fig. 3.9. Standard of sugars for HPLC-ELSD. (a) 7 mix standard and (b) maltose.

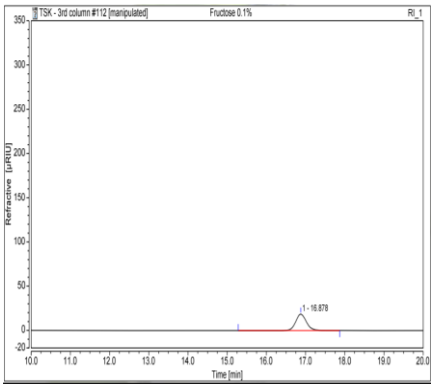
(a) (b)





(c) (d)





(e)

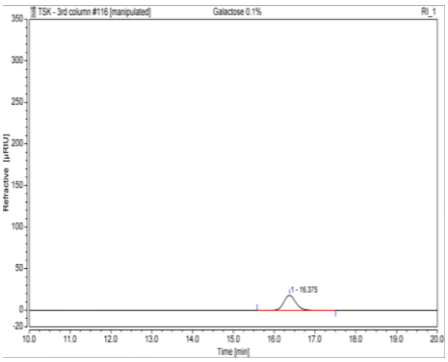
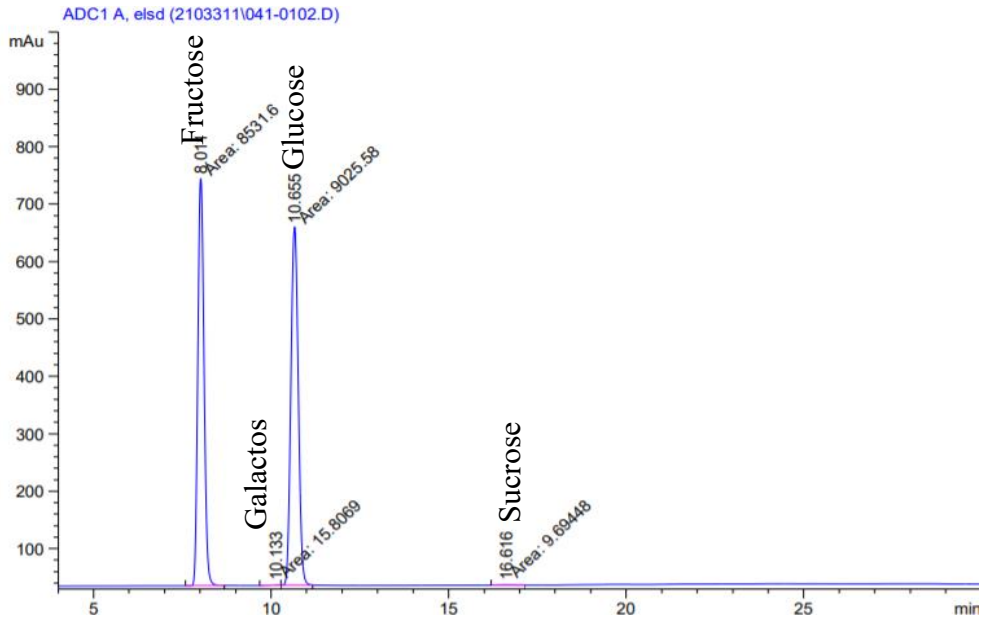


Fig. 3.10. Standard of sugars for HPLC-RI. (a) maltose, (b) glucose, (c) maltotriose, (d) fructose, and (e) galactose.

(a)



(b)

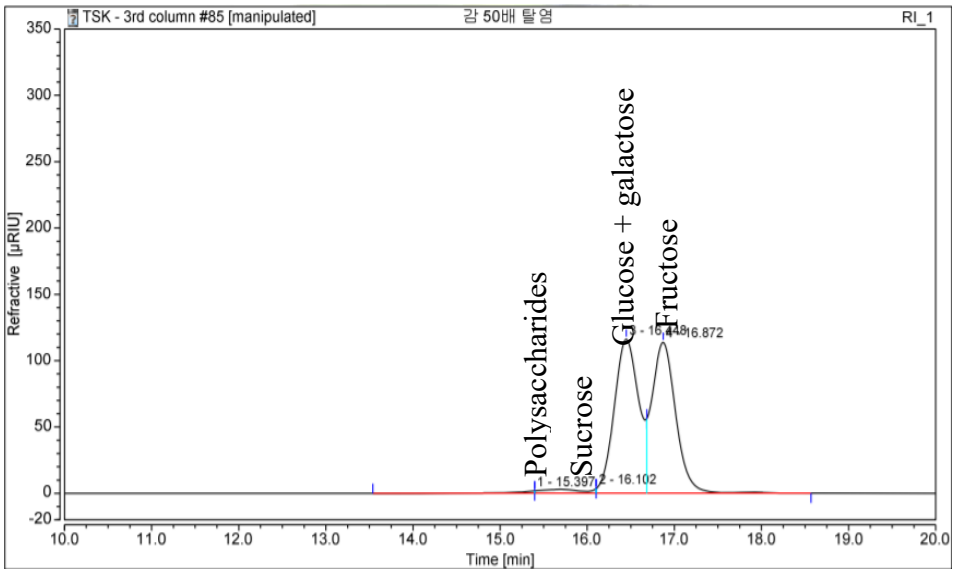


Fig. 3.11. Persimmon extract sugar content. (a) HPLC-ELSD and (b) HPLC-RI.

Table. 3.4. Persimmon extract sugar content. (a) HPLC-ELSD and (b) HPLC-RI

(a)

PE	Monosaccharides			Sucrose (%)
	Fructose (%)	Galactose (%)	Glucose (%)	
Total solid (g/100 g)	26.88 ± 0.16	1.2	26.02 ± 0.24	1.0
Retention time (min)	8.011	10.133	10.655	16.616

(b)

PE	Monosaccharides		Sucrose (%)	Polysaccharides (%)
	Fructose (%)	Glucose + galactose (%)		
Total solid (g/100 g)	29.55 ± 1.27	30.04 ± 1.40	1.24 ± 0.05	0.40 ± 0.02
Retention time (min)	16.873	16.450	16.105	15.395

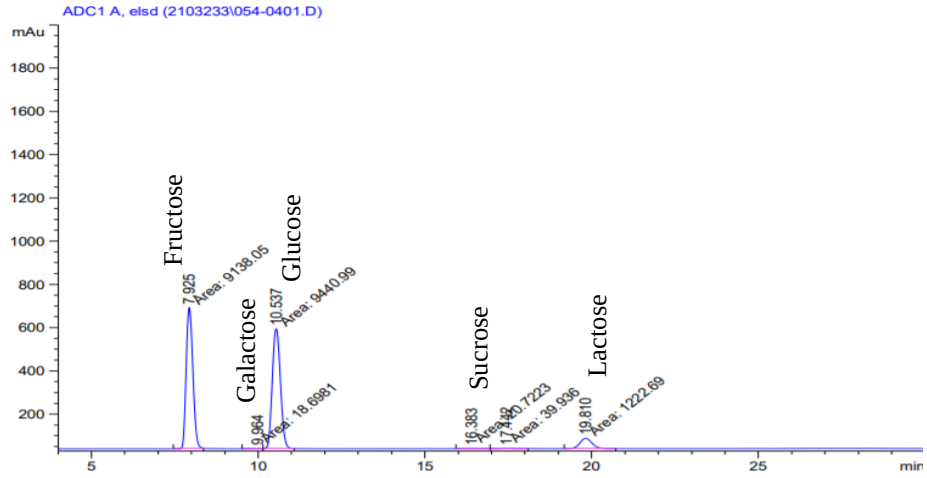
PE, persimmon extract. Experiments were performed in triplicate, and the results were presented as mean ± standard deviation.

HPLC-ELSD results showed that PE-MD consisted of 2.31 g/100 g of fructose and 2.21 g/100 g of glucose (total 4.52 g/100 g), and PE-RMGA contained mainly of 2.37 g/100 g of fructose and 2.10 g/100 g of glucose (total 4.47 g/100 g) (Fig. 3.12 & Table 3.5). This was about half the percentage of persimmon extract 10% (w/w) added to the feed solution, and assuming that persimmon extract contains approximately 85% sugar, the sugar contents of powders were reduced by 3.5–4.0 g. It might have been lost during the spray drying process or it may form a mixture because in case of fruits, dietary fibers are mixed with sugars together.

The elevation of blood glucose levels in the bloodstream is essential to meet the energy demands of the brain because glucose is the brain's only energy source and is used for muscular activity. Fructose is about 1.7 times sweeter than sucrose (Hanover & White, 1993), the GI index is about one sixth lower (Atkinson et al., 2008), does not raise blood glucose, and if proper amounts are absorbed (up to 90 g/day) it may aid glycemic control (Sánchez-Lozada et al., 2008) due to its effects of lowering HbA1c concentrations (Livesey & Taylor, 2008). However, it is probably misleading to conclude that this amount of fructose consumption is safe by examining only the effects of fructose on plasma triglycerides, weight, and HbA1c. Indeed, there is increasing evidence that high fructose intake can also raise blood pressure, decrease insulin sensitivity, lower glucose tolerance, increase apolipoprotein-B concentrations, and cause microvascular disease, glomerular hypertension, renal injury, fatty liver, systemic inflammation, endothelial dysfunction, oxidative stress, and activation of the renin angiotensin system (Brown et al., 2008; Glushakova et

al., 2008; Swarbrick et al., 2008). In addition, it cannot be the energy source to the brain, nor does it supply any energy to the muscle, and gets stored only as fat (Das, 2015; Parks et al., 2008). Therefore, persimmon extract and produced powders had beneficial effects such as antioxidants, but if too much consumption, they could cause increased fat and glucose level due to high levels of monosaccharides. Diabetes occurs when insulin function in the pancreas is not functioning properly when glucose is ingested. Methods to prevent this include insulin injection and enzyme treatment methods by suppressing elevation of glucose using dietary fiber or some enzymes (α-amylase or α-glucosidase) or inhibiting aggregation of human islet amyloid polypeptide (Jiao et al., 2013). It was also found that diabetic patients had a 65% increase in the risk of developing Alzhemier's disease and display lower basal cognitive skills including learning, memory, and perceptual speed (Arvanitakis et al., 2004). Pathogenic amyloids form when previously healthy proteins lose their normal structure and physiological functions. The original function of amyloid beta prevents Alzheimer's disease and diabetes. However, when aggregation occurs, the original function is lost, and the proanthocyanidin plays a role in preventing aggregation. So as mentioned before, with proanthocyanidins and a lot of fiber in persimmons compared to other fruits (Table. 1.2), persimmon extract, PE-MD and PE-RMGA could prevent problems that might be caused diabetes due to high monosaccharides contents. Especially for PE-RMGA, compared to PE-MD, it contained prebiotic dietary fiber, resistant maltodextrin, so it could be more beneficial and solve problems properly.

(a)



(b)

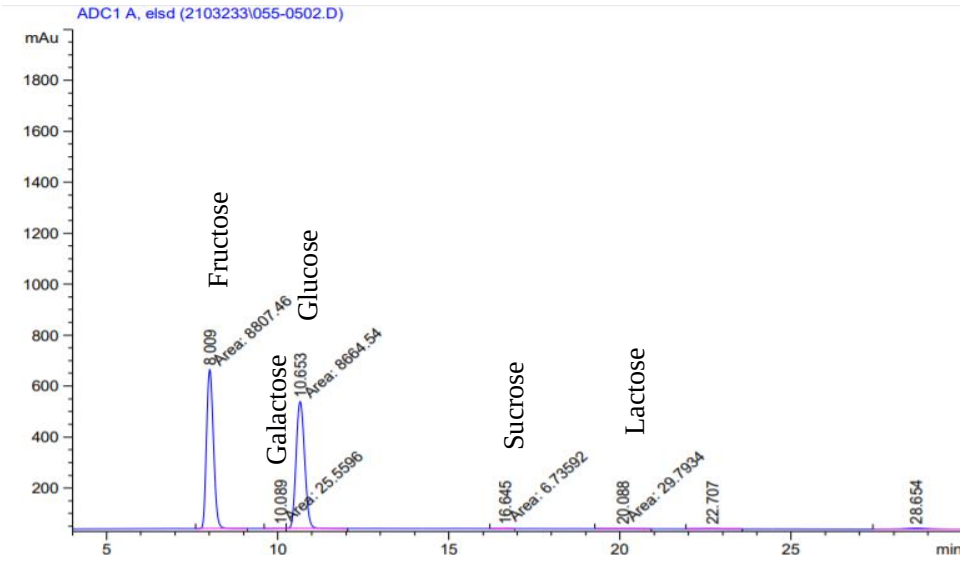


Fig. 3.12. Sugar content of. (a) PE-MD and (b) PE-RMGA by HPLC-ELSD

Table. 3.5. Sugar contents of PE-MD and PE-RMGA by HPLC-ELSD

PE-MD	Monosaccharides			Sucrose (%)
	Fructose (%)	Galactose (%)	Glucose (%)	
Total solid (g/100 g)	2.31 ± 0.08	2.21 ± 0.07	0.1	0.083
Retention time (min)	7.925	10.537	9.964	16.383
PE-RMGA	**Monosaccharides**			**Sucrose (%)**
	Fructose (%)	**Galactose (%)**	**Glucose (%)**	
Total solid (g/100 g)	2.37 ± 0.05	2.10 ± 0.09	0.1	0.083
Retention time (min)	8.009	10.653	10.069	16.645

PE-MD, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3. Experiments were performed in triplicate

Control was sucrose solution (pH 6.8), and Suc-pH5.8, Suc-pH4.8, Suc-pH3.8 indicated that sucrose solution with specific pH, and PE-DW was persimmon extract with DDW. All the samples were 25% (w/w) solution and sensor outputs of sourness to sweetness were expressed at Table. 3.6. Since tartaric acid and KCl were used for reference solution, sourness and saltiness should be considered when interpreting raw data measured by E-tongue. Due to the characteristics of the device, it is interpreted that if the value is below zero, the corresponding taste have “no taste”, and if is above zero, the taste will be felt. For example, if the value of umami is -5 mV, the taste of umami is not felt, and if +3 mV, the taste of umami can be felt. In addition, since tartaric acid and KCl were used for reference solution, sourness and saltiness should be interpreted in consideration, in the case of sourness, the reference value is -13, and saltiness is -6. In other words, sourness is interpreted that if a number is less than -13 mV, and saltiness is less than -6 mV, corresponding taste could not be felt, respectively. It is also interpreted that it is meaningless when having values below “1”.

According to Table. 3.6, all samples were interpreted as meaningless data except bitterness, umami, and sweetness, and for sourness at Suc-pH3.8 and PE-RMGA and PE-DW for richness. On the other hand, due to the device limit characteristics, umami is applied only to certain foods (Japanesse green tea), so it was excluded from the analysis, and richness was also excluded because it is aftertaste of umami. On the other hand, Suc-pH3.8 can be interpreted to be relatively felt sourness because pH was adjusted with HCl. In other words, the remaining flavors were bitterness and sweetness, and bitterness was difficult

to obtain absolute figures due to the limitation of the samples. The reason is that the samples had low conductivity, due to characteristics of sugars that does not dissociate in water, it is difficult to judge bitterness when conductivity value is low. Based on control (suc-pH6.8), when in hexagons (Fig. 3.13), it seemed that there was a differences in tastes, but it was meaningless data except for sweetness.

Table. 3.6. Relative value of taste sensor of E –tongue (mV)

	Sourness (mV)	Bitterness (mV)	Astringency (mV)	Aftertaste-B (mV)	Aftertaste-A (mV)	Umami (mV)	Richness (mV)	Saltiness (mV)	Sweetness (mV)
Control	-18.09	1.33	-12.13	0.06	-0.07	4.12	0.69	-18.10	25.01
Suc-pH 5.8	-16.04	-0.25	-12.23	-0.26	-0.05	3.97	0.66	-18.11	24.38
Suc-pH 4.8	-13.33	-2.21	-12.63	-0.28	-0.06	3.44	0.66	-18.40	23.67
Suc-pH 3.8	-5.51	-3.84	-12.53	-0.19	-0.03	1.43	0.64	-18.59	18.13
PE-MD	-16.71	2.39	-6.78	0.79	0.14	5.34	0.72	-12.40	16.05
PE-RMGA	-11.98	2.34	-3.75	0.80	0.38	0.62	2.22	-7.91	13.01
PE-DW	-17.08	0.83	-4.87	0.08	0.18	5.34	1.05	-8.85	20.21

Control, pH 6.8 sucrose solution (25%, w/w); Suc-pH 5.8, pH 5.8 sucrose solution (25%, w/w); Suc-pH 4.8, pH 4.8 sucrose solution (25%, w/w); Suc-pH 3.8, pH 3.8 sucrose solution (25%, w/w); PE-MD, solution (25%, w/w) of spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, solution (25%, w/w) of spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3; PE-DW, solution (25%, w/w) of persimmon extract with DDW.

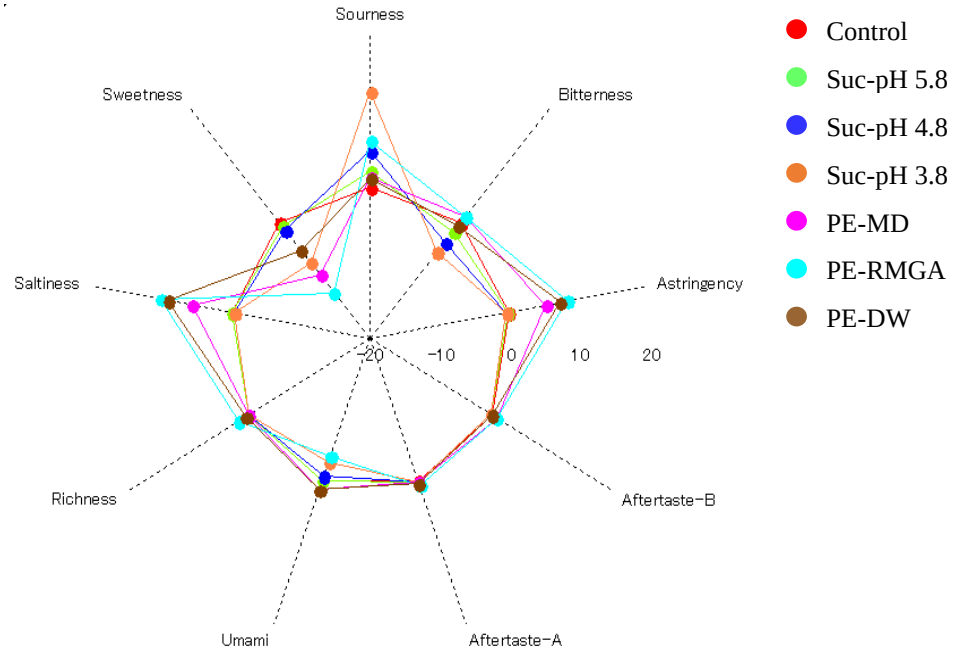


Fig. 3.13. Results of electronic tongue expressed as enneagon shape based on control. Control (●), suc-pH5.8 (●), suc-pH4.8 (●), suc-pH3.8 (●), PE-MD (●), PE-RMGA (●), PE-DW (●). Control, pH 6.8 sucrose solution (25%, w/w); Suc-pH 5.8, pH 5.8 sucrose solution (25%, w/w); Suc-pH 4.8, pH 4.8 sucrose solution (25%, w/w); Suc-pH 3.8, pH 3.8 sucrose solution (25%, w/w); PE-MD, solution (25%, w/w) of spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, solution (25%, w/w) of spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3; PE-DW, solution (25%, w/w) of persimmon extract with DDW.

The interpretation of the value was calculated as a logarithmic value, which was interpreted as 1.2 times difference in concentration if the value was twice different, and 10 times difference if value was 12.6 times different ($1.2^{12.6} \approx 10$). For example, in sweetness, if sample A had a value of 1 and sample B had a value of 4, this would be four times the numerical difference, however, $1.2^4 \approx 2.07$), so sample A can be expressed as having twice concentration of sweetness compared to B. Since the E-tongue is pH sensitive, the data were analyzed by dividing the control into two parts: normal aqueous solution (Suc-pH6.8) (Table. 3.7) and Suc-pH4.8 (Table. 3.8). With typical aqueous solutions under control, the values of electric response were control (25.01 mV), PE-MD (16.05 mV), PE-RMGA (13.01 mV), and PE-DW (20.24 mV) (Table. 3.7; Fig. 3.14). To compare sweetness concentrations, control (1.92), PE-MD (1.23) and PE-DW (1.56) were divided by the lowest PE-RMGA value (13.01 mV) respectively, and to express as intensity of taste (concentration), based on PE-RMGA, each 1.2^n value was taken respectively (control (1.42), PE-MD (1.25) and PE-DW (1.33)). In other words, the sweet taste concentration of a sugar solution was 1.42 times higher than that of PE-RMGA, (Fig. 3.14). This does not mean that it is actually 1.42 times sweeter, but based on the concentration value, for example, when the concentration of PE-RMGA is 100, the concentration value of control is 142. Similarly, calculating the value based on Suc-pH4.8, the taste scale of Suc-pH4.8 corresponded to 39% difference in concentration with PE-RMGA (Table. 3.8), which is human differential sensitivity for basic tastes on average. This means that when PE-RMGA has 100 sweet ingredients, it has 139 for Suc-pH4.8 (Fig. 3.15). Finally, PE-MD,

PE-RMGA, and PE-DW were all less sweet than sugar solutions, but they do not differ that much.

Table. 3.7. Electric response (mV), intensity of taste of Suc-pH 6.8, PE-MD, PE-RMGA and PE-DW

	Sweetness (mV)	**Translated value**	**Intensity of taste (concentration)**
Control	25.01	1.92(25.01/13.01)	$1.42(1.2^{1.92})$
PE-MD	16.05	1.23(16.05/13.01)	$1.25(1.2^{1.23})$
PE-RMGA	13.01	1	1
PE-DW	20.24	1.56(20.24/13.01)	$1.33(1.2^{1.56})$

Control, pH 6.8 sucrose solution (25%, w/w); PE-MD, pH 4.8 solution (25%, w/w) of spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, pH 4.8 solution (25%, w/w) of spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3; PE-DW, pH 4.8 solution (25%, w/w) of persimmon extract with DDW.

Table. 3.8. Electric response (mV), intensity of taste of Suc-pH 4.8, PE-MD, PE-RMGA and PE-DW

	Sweetness (mV)	**Translated value**	**Intensity of taste (concentration)**
Suc-pH4.8	23.67	1.82(23.67/13.01)	$1.39(1.2^{1.82})$
PE-MD	16.05	1.23(16.05/13.01)	$1.25(1.2^{1.23})$
PE-RMGA	13.01	1	1
PE-DW	20.24	1.56(20.24/13.01)	$1.33(1.2^{1.56})$

Suc-pH 4.8, pH 4.8 sucrose solution (25%, w/w); PE-MD, pH 4.8 solution (25%, w/w) of spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, pH 4.8 solution (25%, w/w) of spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3; PE-DW, pH 4.8 solution (25%, w/w) of persimmon extract with DDW.

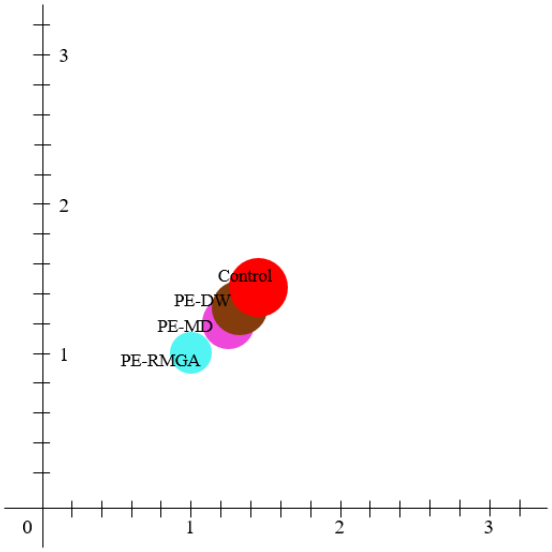


Fig. 3.14. Results of intensity of taste based on electric response (mV) on sweetness compared with control (Suc-pH6.8). Control, pH 6.8 sucrose solution (25%, w/w); PE-MD, pH 4.8 solution (25%, w/w) of spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, pH 4.8 solution (25%, w/w) of spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3; PE-DW, pH 4.8 solution (25%, w/w) of persimmon extract with DDW.

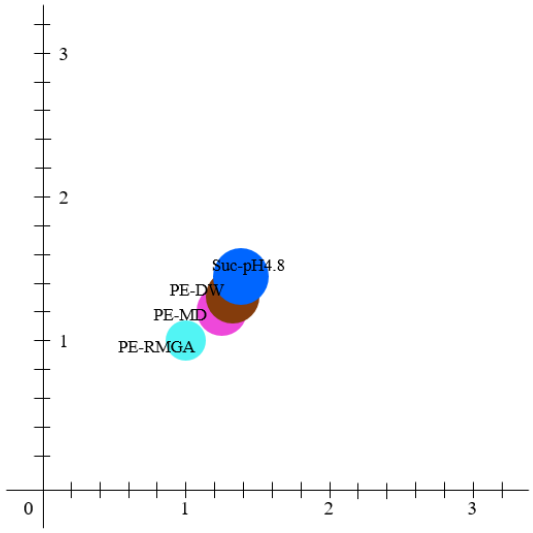


Fig. 3.15. Results of intensity of taste based on electric response (mV) on sweetness compared with Suc-pH4.8. Suc-pH 4.8, pH 4.8 sucrose solution (25%, w/w); PE-MD, pH 4.8 solution (25%, w/w) of spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) maltodextrin solution; PE-RMGA, pH 4.8 solution (25%, w/w) of spray-dried powder with 10% (w/w) persimmon extract and 30% (w/w) resistant maltodextrin and gum arabic solution with ratio of 7:3; PE-DW, pH 4.8 solution (25%, w/w) of persimmon extract with DDW.

4. Conclusions

PE-RMGA had 105.64 ± 13.20 mg GAE/100 g dry weight of total phenolics and 58.78 mg CTE/100 g dry weight of proanthocyanidin contents. Due to emulsifying properties of gum arabic, initial emulsion was stable, resulted in high encapsulation efficiency. No vitamin C content detected, maybe due to extraction process, sensitive vitamin C might be eliminated. According to the results of HPLC-ELSD, PE-RMGA consisted almost of fructose and glucose instead of sucrose. Eating less than 90 g of fructose a day can be beneficial to health, but eating more than that can be harmful to health and can be fatal to diabetics because of its high glucose content. However, it may be possible to lower the glucose intake level because RMD, a prebiotic diet fiber, was added as a drying aid. Compared with sugar water solutions, PE-RMGA was less sweet than that, with a concentration difference of around 40%, but did not show much difference. Until the consumption stage, the cultivated product losts are about 50% and the weight of the fruit peel is about 20%, however, since cheongdo-bansi has no seeds, the waste could be reduced by grinding the whole fruit and making by powderization. As mentioned at Chapter 2, poor flowability could cause handling problems, difficulty storing above RH 43% at room temperature, but could be used as a potential sweetener with health promoting properties such as anti-oxidant, anti-diabetic, and neuroprotective characteristics.

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Abstract in Korean

감에는 식이섬유, 비타민, 미네랄, 폴리페놀 등 건강에 이로운 성분이 들어 있어 항산화 및 항당뇨 효과는 물론 심혈관계 질환 등 각종 질병을 예방한다. 하지만, 대부분의 과일의 수분 함량은 70~80%에 달하기 때문에 보관, 운송 손실 등에서의 한계점을 가질 수 있고, 대부분의 과일은 껍질을 벗긴 후 먹기 때문에 쓰레기 문제, 농약 문제, 계절성 문제 등도 있다. 또한 비타민과 폴리페놀과 같은 생물 활성 화합물은 높은 수분 함량 조건에서 분해될 수 있기 때문에 수분함량을 조절하는 것은 생물 활성 화합물 파괴 방지를 위해 굉장히 중요하다. 분무 건조는 과일 분말화에 널리 쓰이고 있으며, 생산된 분말은 수분 함량 및 수분활성도가 낮기 때문에 앞서 열거된 한계를 극복할 수 있다. 하지만 과일에는 당분이 많아 유리 전이온도가 낮기 때문에 분말화될 때 고온에 의해 끈적임이 발생할 수 있고, 그렇기 때문에 분무 건조 시 다당류, 단백질 등 건조보조제를 추가해야 한다. 저항성 말토덱스트린은 프로바이오틱스의 영양소가 되어 장내 환경 개선에 도움을 줄 수 있는 프리바이오틱스 식이섬유이다. 한편, 저항성 말토덱스트린이 단독으로 사용될 경우, 끈적임이 발생할 수 있으며, 그로 인해 분무건조기의 필터가 막혀서 공정이 정지될 수 있다. 과일 분말을 만들 때 말토덱스트린과 함께 필름 형성 능력을 가진 구아검을 사용한다. 본 연구에서는 건조 보조제로써 7:3의 비율로 저항성 말토덱스트린과 구아검(30%, w/w)을 감 추출액(10%, w/w)에 넣은 후 분무건조 시키고 생성된 분말의 물리화학적(Chapter 2) 및 기능적 특성(Chpater 3)을 확인하는 것을 목적으로 하였다. 감 추출액(10%, w/w)에 말토덱스트린이나 저항성

말토덱스트린과 구아검 건조보조제(30%, w/w)를 첨가한 후 분무 건조시킨 결과 수분 함량과 수분활성도는 각각 2.21%, 0.158로 미생물학적으로 안정한 것으로 나타낸고 입자 크기(14.4 μm)가 작기 때문에 유동성과 분산성이 낮은 값을 나타났다. 생성된 분말은 일반적인 과일과 같은 GAB 흡착 모델을 보여주었으며, 흡습성이 강하다는 특성을 가지고 있어 분말 취급, 보관 시 주의가 요구된다. 총 페놀함량과 프로안토시아니딘 함량의 보존 정도는 높았지만 비타민C 함량은 검출되지 않았는데, 이는 추출 과정 중 제거됐을 수 있다. 당 함량은 자당 대신 대부분이 포도당과 과당으로 구성 되었고, 전자혀로 자당과의 단맛을 비교해도 큰 차이는 관찰되지 않았다. 다른 과일과 마찬가지로 설탕 대신 자당과 포도당이 대부분의 당을 구성하고 있었는데 이는 식이섬유와 함께 결합하여 작용되기 때문에 적당량을 이용하면 혈당 지수를 조절하는데 도움이 될 것이다. 결과적으로, 감 추출액에 저항성 말토덱스트린과 구아검을 혼합하여 분무 건조시킨 후 생성된 분말은 유동성과 습도 특성이 좋지 않아 취급 및 보관부위에 주의가 필요하지만 적정량을 사용할 경우 항산화, 항당뇨, 신경보호 특성을 가진 천연 감미료로 사용할 수 있다. 또한, 해당 분말은 감의 건강에 이로운 성분뿐만 아니라 프리바이오틱스 식이섬유를 함유하고 있어 보다 더 건강기능적인 역할을 할 것이다.

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생각보다 시원했던 2019년 여름, ‘한적하고 신기하다’ 평창에 처음 왔을 때 제가 느낀 감정입니다. 연구에 대한 호기심으로 시작됐던 낯선 지역에서의 생활이 생각보다 힘들었던 만큼, 연구에 대한 확신을 가지고 학위논문의 마지막 페이지를 적고 있다는 사실에 새삼 놀랍습니다. 랩실에서 가장 어리고 미숙했던 제가 학위논문을 완성할 수 있었던 이유는 당연히 많은 분들의 도움과 가르침 덕분이기에 부끄럽지만 소소한 감사의 말씀을 드리고 싶습니다.

먼저, 2년동안 한결 같은 가르침으로 많은 성장을 이끌어주신 정동화 지도교수님께 깊은 감사를 전해드리고 싶습니다. 연구 주제 선정부터 제 의견을 존중해주시고, 연구과정 중 수없이 발생했던 문제점을 어떻게 헤쳐 나가야할 지 알려주셨으며, 그리고 스스로의 채찍질에 스트레스 받는 성향의 저를 파악하시고 차근차근 설명해주신 교수님 덕분에 무사히 학위과정을 마칠 수 있었습니다. 교수님의 지도로 인해 연구에 대한 확신이 생겼습니다. 앞으로 연구자로서 막연한 연구 분야에도 자신 있게 과감하게 뛰어들고 문제가 생겼을 때 스스로 학습하고 이해할 수 있는 능력을 바탕으로 교수님의 큰 가르침에 보답하겠습니다.

또한, 바쁘신 가운데에도 두 번의 세미나를 통해 진심 어린 피드백을 주시고 학위논문 심사위원을 맡아주신 김도만 교수님, 김효진 교수님 정말 감사드립니다. 소중한 피드백을 주셔서 물음표로 남을 수 있었던 실험이 느낌표로 바뀌며 마무리를 지을 수 있었습니다.

석사 과정 중, 실험적으로 많은 도움을 주신 분들께도 감사의 인사를

드립니다. 전자혀 실험 관련해서 궁금한 점이 생길 때마다 연락을 드려도 언제나 친절하게 답변해주시고 온라인 미팅을 통해 손수 시험 방법까지 알려주시는 등 적극적으로 많은 가르침을 주신 일본의 Habara 박사님, 함께 항산화 실험을 진행함에 있어 많은 도움을 주신 경희대 김관중 박사님, 최승수 연구원 에게도 감사의 말씀을 드립니다. 지금은 함께 있지 않지만 6개월동안 많은 도움을 주신 동현 선배 감사드리고, 항상 웃는 얼굴로 격려해주시고 연구 방향이 틀어질 때마다 함께 고민해 주시던 제 영원한 사수 기철 오빠, 언제 어디서든 고민상담해주고 친동생처럼 아껴주던 승연 언니 정말 감사합니다.

짧은 석사과정 중 가장 많은 시간을 함께 보내며 저에게 정말 큰 힘이 되어주신 식품소재공학연구실 식구들, 학위논문을 작성함에 있어 많은 도움을 주고 실험적으로도 많이 도와주신 Andy 박사님, 입학부터 졸업까지 물심양면으로 정말 많은 도움을 주시고 막히는 부분이 있을 때마다 시원하게 해결 해주실 정도로 정말 못하는 것이 없으신 영원한 대장 지혜 언니, 처음부터 끝까지 막내라 생겼었던 고민을 시원하게 없애주고 오히려 편하게 대해주며 연구 중 막히는 부분이 생길 때마다 자기 일처럼 신경 써주던 다영 언니, 기쁠 때나 슬플 때, 힘들 때까지도 누구보다 크게 공감해주고 위로해주며 기숙사 도서관에서까지 함께 공부한 랩실 내 유일한 동갑 친구 현민, 쿠크다스 멘탈이지만 열정적이고 착한 성암 씨, 조용하지만 공감 능력이 뛰어나고 정말 성실한 대범 씨까지, 어떤 표현으로도 담을 수 없을 만큼 큰 감사의 인사를 드립니다. 그리고 첫 입학 면접 때부터 웃는 얼굴로 먼저 다가와주고 시작부터 논문학기까지 함께 한 정말 착한 영원한 동기 혜리, 만날 때마다 웃음을

준 숙, 많은 격려와 응원을 해준 주호 씨, 재원 오빠, 예닮 언니, 혜리, 정빈 씨, 단열이를 포함한 바이오식품산업학과 연구원 및 학생분들 진심으로 감사의 말을 전합니다.

평창이라는 환경 때문에 핑계를 대며 소원했던 친구들에게도 늦은 감사의 인사를 전합니다. 때로는 언니같이, 때로는 동생같이 서로가 서로에게 조언을 주며 언제 연락해도 어색하지 않고 반갑게 응답해주며 얘기를 들어주는 수연이와 예지, 무서운 선배에서 없어서는 안될 정도로 소중한 언니가 되어버린 항상 감동을 주는 하람 언니 언제나 고맙고 진심으로 고맙습니다. Lusionspet, 영원한 어벤저스이자 대학 동기인 주혜, 그리고 지우 에게도 감사합니다.

마지막으로 어떤 상황이든 존중해주고 지지해주시는 엄마, 아빠, 그리고 오빠 정말 사랑하고 감사드립니다. 제가 성장할 때마다 누구보다 기뻐해 주시고 좌절할 때에도 변함없는 사랑으로 믿어주신 부모님 덕분에 많은 성취를 할 수 있었습니다. 힘들다는 핑계로 연락에 소홀해도 묵묵히 기다려 주시고 아낌없이 사랑을 표현해 주신 부모님 덕분에 밝고 행복한 삶을 살아올 수 있었습니다. 더욱 성장된 모습으로, 이제는 든든한 딸이 되도록 쭉 최선을 다하겠습니다. 그리고 어렸을 때부터 하나밖에 없는 동생이라고 배려해주고 본인이 힘들 때도 동생 먼저 생각하는, 동생 바보로 불릴 정도로 한없이 착하고 배려심이 깊은 오빠 항상 감사합니다. 항상 믿어주고 지지해주며 사랑해주는 소중한 가족이 있었기에 힘든 상황에 굴하지 않고 무사히 학위과정을 마칠 수 있었습니다. 항상 감사하고 사랑합니다.

이외에도 응원해주신 많은 분들께 감사드립니다. 석사 2년이라는

과정이 짧았지만 상대성 이론을 경험한 것 마냥 오랜 기간을 함께 보낸 듯한 깊은 유대감에 한편으로는 아쉽기도 하여 발걸음이 가볍지만은 않습니다. 바람결에 툭 닿는 평창 소식이 그리워질 것 같은 느낌도 듭니다. 바이오식품산업 전공 교수님들과 학생, 그리고 연구원분들께도, 비행기, 비전을 가지고 행동에 옮기면, 졸업이라는 기적이 일어납니다. 진달래, 진짜로 달콤한 내일이 오기를, 모바일, 모든 것이 바라는 대로 일어나길 진심으로 염원하겠습니다.

연구에 대한 열정과 진심으로 적어 내린 감사의 마음을 바탕으로 새로운 시작, 힘차게 비상하겠습니다. 감사합니다.

2021년 8월,

이민정 올림