THESIS

EFFECT OF SPRAY DRYING ENCAPSULATION METHOD ON FLAVOR QUALITY OF ANDALIMAN (*Zanthoxylum acanthopodium* DC.) POWDER

Written as partial fulfillment of the academic requirements to obtain
the degree of *Sarjana Teknologi Pertanian Strata Satu*

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STATEMENT OF THESIS AUTHENTICITY

I, a student of Food Technology Department, Faculty of Science and Technology, Universitas Pelita Harapan,

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Hereby declare that my thesis, entitled **"EFFECT OF SPRAY DRYING ENCAPSULATION METHOD ON FLAVOR QUALITY OF ANDALIMAN (*Zanthoxylum acanthopodium* DC.) POWDER"**:

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Karawaci, December 18th, 2013

(Clarissa Akyla)

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APPROVAL BY THESIS SUPERVISORS

EFFECT OF SPRAY DRYING ENCAPSULATION METHOD ON FLAVOR QUALITY OF ANDALIMAN (*Zanthoxylum acanthopodium* DC.) POWDER

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APPROVAL BY THESIS EXAMINATION COMMITTEE

We the undersigned, certify that a thesis defense has been held on January 29th, 2014, as partial fulfillment of the academic requirement to obtain the degree of *Sarjana Teknologi Pertanian Strata Satu* in the Food Technology Department, Faculty of Science and Technology, Universitas Pelita Harapan, for the student:

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With the following title **"EFFECT OF SPRAY DRYING ENCAPSULATION METHOD ON FLAVOR QUALITY OF ANDALIMAN (*Zanthoxylum acanthopodium* DC.) POWDER"**, and that the thesis has been approved by the examination committee.

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ABSTRACT

Clarissa Akyla (03420100042)

EFFECT OF SPRAY DRYING ENCAPSULATION METHOD ON FLAVOR QUALITY OF ANDALIMAN (*Zanthoxylum acanthopodium* DC.) POWDER

(xv + 113 pages: 11 tables, 10 figures, and 28 appendices)

Andaliman is one of Indonesian traditional spices well known in North Sumatra. It possesses unique flavor with pleasant citrus-like aroma and a tongue-numbing trigeminal sensation. To preserve andaliman flavor and trigeminal characteristic in form of andaliman powder, application of a suitable spray drying encapsulation method is needed. Andaliman was firstly extracted by maceration using ethyl acetate: ethanol (1:1), and then the extract was subjected to spray drying. Treatments for andaliman extract spray drying were inlet temperature (135˚C, 150˚C, 165˚C), extract to carrier ratio (1:4, 1:6, 1:8), and maltodextrin to gum arabic ratio (3:2, 1:1, 2:3). The best condition for andaliman spray drying was determined through sensory evaluation, and was found to be andaliman powder produced at inlet temperature of 150˚C, with extract to carrier ratio of 1:8 and ratio of maltodextrin to gum arabic of 3:2. Quantitative Descriptive Analysis (QDA) result shown that flavor of andaliman powder was similar to those of fresh andaliman (citrus like aroma, sour floral citrusy aroma, warm citrus aroma, sour taste, bitter taste, and tongue-numbing trigeminal sensation), but with more pronounced taste, trigeminal sensation, citrus-like and sour floral citrusy aroma. The resulting andaliman powder had yield of 23.96%, moisture content of 5.06%, water activity of 0.122, powder solubility of 37.11%, yellow color, bulk density of 0.32 g/ml, and hygroscopicity of 24.80 g moisture/ 100 g dry solids. This research demonstrated the feasibility of andaliman powder production by using spray drying encapsulation method.

Keywords : Andaliman, encapsulation, QDA, spray drying, trigeminal sensation
References : 70 (1978-2013)

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The writer realizes that this thesis report is far from perfect and it may contain many mistakes, both the expected and unexpected ones. Therefore the author would like to deliver apology and humbly welcome any critics and/or suggestions that may improve this report. Hopefully this report could be useful and helpful for all the readers. God Bless You.

Karawaci, December 2013

Writer

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CHAPTER I

INTRODUCTION

1.1 Research Background

Flavor is a very important characteristic of food product since people prefer to eat foods with attractive flavor (Zuidam and Nedovic, 2010). Flavor does not only include compounds imparting taste and aroma, but is a complex combination of the olfactory, gustatory and trigeminal sensations (Jelen, 2012). New and interesting flavor ingredients are continuously being searched and identified, but in order to have a practical usage, it is important that the flavor ingredients do not only possess interesting organoleptic properties, but also efficient to be produced as well as remain stable during long period of storage and to processing condition that includes high temperature, humidity and extreme pH (Galopin *et al*., 2004).

Andaliman (*Zanthoxylum acanthopodium* DC.) is one of Indonesian traditional spices especially well known in North Sumatera. It has been commonly used in Batak cuisine, such as *arsik, saksang* and *sambal tinombur*. Wijaya (2001) have succeeded in isolation and identification of andaliman chemical composition including its volatile aroma constituents and potent odorant. Besides being used for its pleasant citrusy and warm peppery flavor, andaliman also has a unique characteristic of pungent sensation, commonly perceived as ‘tingling’ or numbing effect to the tongue. Several researches have also shown andaliman potential as antimicrobial and antioxidant, as well as its applications to preserve foods, such as for *tahu* and fish preservation (Parhusip, 2006; Chandra, 2010; Parhusip *et al.*, 2007). Nevertheless, very small amount of researches have been conducted on the unique flavor and trigeminal characteristic of andaliman, and its potential as a natural flavoring has yet to be fully studied.

Commonly, fresh andaliman is used in dishes preparation since it has stronger flavor compared to the dried ones. However, the fruit itself is very susceptible to deterioration and cannot withstand storage more than several days in its fresh condition. Traditional sun drying may extend andaliman shelf life, but due to the unstable flavor compounds, the strength of flavor will be greatly reduced in comparison with fresh andaliman. Wijaya (2000) isolated and identified the trigeminal active compound of andaliman which is a substituted amide named as sanshool (2E, 6Z, 8E, 10E-N-(2'-methylpropyl)-dodecatetraenamide). According to Galopin *et al.* (2004), the highly flavored and pungent sanshool compound isolated from *Zanthoxylum* is an unstable and unsaturated aliphatic amide, which tends to polymerize easily. Yang (2008) mentioned that the tingling compound in various Zanthoxylum species is an alkylamide compound, which decomposes easily under hydrolytic conditions and UV light. This causes them to be difficult to store over prolonged period and to incorporate to processes that promote instability. Therefore, to improve the potential of andaliman as a natural flavoring, a processing technology that can prolong andaliman shelf life while maintaining its flavor and trigeminal sensation characteristics is needed.

Flavor encapsulation technique has been widely used to convert flavor compounds which are majorly liquid at room temperature, into dry, free flowing powders that is more applicable for usage. There are many methods for encapsulation, but spray drying and extrusion are two major commercial processes that have been used a lot. More than 90% of encapsulated flavoring in the market is produced by spray-drying (Nusinnovitch, 1997). Besides changing the food product physical characteristics, encapsulation provides protection for the flavor solids during storage against light, heat, moisture, or air that can induce oxidation reactions, and can also provide a controlled or delayed release of flavor (Spanier *et al.*, 2001). Many spray drying applications have been utilized to encapsulate spice oleoresins, which despite their contribution to spice flavor, are very reactive and unstable to light, temperature and oxygen (Shaikh *et al.*, 2006; Khrisnan *et al.*, 2005; Soottitantawat[2] *et al.*, 2005; Bayram *et al.*, 2005).

1.2 Research Problem

Andaliman has a great potential as a natural flavoring since it possess unique flavor and trigeminal characteristics. However, so far the extraction of the trigeminal active compound and the flavor compound has been done separately in different studies. Moreover, the susceptibility of andaliman fruits in fresh condition and the instability of its flavor and trigeminal active compound have caused limitation in utilizing the fruit’s potential. Spray drying encapsulation has been used to produce several kinds of powder flavorings, as well as to increase the stability of the flavor compounds towards extreme processes and storage. However, the spray drying encapsulation method regarding the temperature and encapsulating agent type and concentration, in order to produce andaliman powder has yet to be studied. Therefore this research focuses on finding the suitable spray drying encapsulation method in order to produce andaliman powder with optimum flavor characteristics.

1.3 Research Objectives

1.3.1 General Objectives

The general objective of this research was to preserve the unique flavor and trigeminal characteristic of andaliman in form of andaliman powder, by application of spray drying encapsulation method.

1.3.2 Specific Objectives

The specific objectives of this research were:

1) To determine the suitable extraction solvent for andaliman flavor and trigeminal active compounds.
2) To determine the effect of spray drying inlet temperature and carrier agent composition towards the flavor qualities of andaliman powder.

CHAPTER II

LITERATURE REVIEW

2.1 Flavor

Sense of taste includes perception of non-volatile substances which when dissolved in water, oil, or saliva, can be detected by receptors in the taste buds on the tongue surface or other areas of mouth and throat. There are five taste qualities: salty, sweet, sour, bitter and umami. Aroma perception happens when volatile molecules are sensed by olfactory receptor on the nasal epithelium, located at the roof of nasal cavity (Kemp *et al.*, 2009). Flavor does not only include compounds imparting taste and aroma, but is a complex combination of the olfactory, gustatory and trigeminal sensations (Jelen, 2012).

Flavor has a very important role in determining consumer satisfaction and inducing further consumption of foods. However, most flavors are delicate and volatile; therefore there have been concerns in how to preserve them. Most aroma compounds available are produced through chemical synthesis or extraction, but synthetic flavor have been avoided due to their reputation as being toxic or harmful to health (Teixeira *et al.*, 2004). There has been increasing interests in study of flavor stability because of its influence to quality and acceptability of foods. Flavor stability is a complex system consisting of many variables, for example some flavor compounds are more stable in carbohydrates which are water soluble, while others are more stable in lipid-base coating (Madene *et al.*, 2006).

2.1.1 Trigeminal Sensation

Beside perception of the taste and smell system to chemical sensation, there is a generalized chemical sensitivity called as “chemesthesis”, which can be perceived in nose, mouth, eyes and over the whole body. Some common flavor experiences due to stimulation of trigeminal nerves includes the fizzy tingle of carbon dioxide in soda, the burn of hot peppers, the pungency of black pepper, ginger and cumin, nasal pungency of mustard and biting sensation of raw onions and garlic. Widely studied examples of trigeminal active compounds include capsaicin in chili pepper and piperine in black pepper. The noted characteristics of these compounds are their very low thresholds and long-lasting nature of stimulation (Lawless, 2005).

2.2 Andaliman

Andaliman is a wild spice from North Sumatera, Indonesia, also known traditionally as ‘*merica Batak’*. It has the characteristic of a fresh citrusy and warm sweet peppery odor. Conventionally, andaliman is used to cover the fishy odor of raw fish or meat (Wijaya *et al.*, 2001). Although it is not as spicy as chili or pepper, andaliman exhibits numbing effect to the tongue due to its unique flavor. The fruit is small and spherical, green in color when it is young and fresh, red when it is mature and black when it is dried. Aroma of fresh andaliman is stronger as compared to the dried ones.

Andaliman potential as an effective antimicrobial has been studied by Parhusip *et al.* (2003) and Parhusip (2006), also with its application in tofu preservation (Parhusip *et al.*, 2007). Andaliman crude extract showed significant antimicrobial ability against *P. aeruginosa, B. stearothermophilus, S. typhimurium* and *B. cereus.* Chandra (2010) has also studied application of andaliman extract for Indian mackerel (*Rastrelliger kanagurta)* preservation. However, research regarding andaliman is very limited and its potential as natural flavoring has not been widely acknowledged (Wijaya *et al.,* 2001).

Wijaya *et al.* (2001) conducted a research to analyze the potent odorant of andaliman maceration extract using GC-MS, GC/O and aroma extract dilution analysis (AEDA) methods. It was reported that the best extraction method was achieved by using maceration method, and the extract with the highest to lowest intensity was achieved by using diethyl ether, chloroform, ethanol and acetone, respectively. From the GC-MS of maceration extract using diethyl ether, 24 volatile components were detected and identified. Majority of the volatile components are oxygenated monoterpenes (α-pinene, β-myrcene, limonene), followed by hydrocarbon monoterpenes (α-terpineol, β-citronellol, geraniol) and others including aromatic and other monoterpenes. Two major components in the flavor extract of andaliman is geranyl acetate and limonene, which gives the sour floral citrusy and sweet citrusy aroma, respectively. Limonene was also found to be the major component in other Zanthoxylum family, including the Japanese pepper (*Zanthoxylum piperitum DC.*) which better known as sansho. From the AEDA (aroma extract dilution analysis) result, citronellal was reported as the component with the highest aroma intensity, with its characteristic of strong and warm citrusy aroma, followed by limonene with its citrus peel sweet aroma (Wijaya *et al.,* 2001).

As it belongs to *Zanthoxylum* sp., andaliman also possess a numbing trigeminal sensation produced by a compound namely (2E, 6Z, 8E, 10E-N-(2'-methylpropyl)-dodecatetraenamide) which can be found in the pericarp of the fruit. Many other species of Zanthoxylum have been also utilized as spices, including *Z. simulans* (Taiwan), *Z. sansho* (Japan), *Z. schinifolium* (Korea) and *Z. rhetsa* (India). Moderate concentration of andaliman extract was reported to have immunomodulator effect. Oral intake of andaliman also shown to has anti-diabetic, anti-hypertensive and relaxation actions, increases appetite and improves bowel evacuation. The scent simulation, however, reduced the appetite and indicated anti-obesity activity due to its ability to keep the nerve active (Wijaya, 2008).

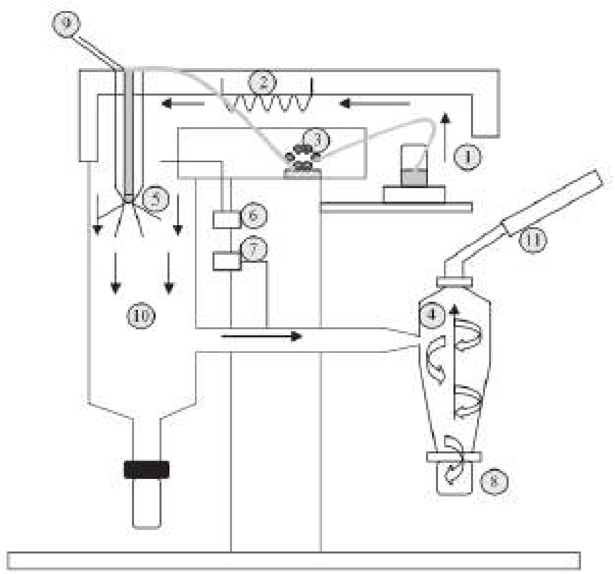
The highly flavored and pungent sanshool compound isolated from *Zanthoxylum* is found to be an unstable and unsaturated aliphatic amide, which tend to polymerize easily (Galopin *et al.*, 2004). Yang (2008) also mentioned that the tingling compound in various Zanthoxylum species is an alkylamide compound, which decomposes easily under hydrolytic conditions and UV light. This causes them to be difficult to store over prolonged period and to incorporate to processes that promote instability. Igarashi *et al.* (2012) have tried to chemically synthesize this compound through coupling in order to increase its stability. Wijaya (2000) extracted the trigeminal active compound in andaliman by maceration method, using ethyl acetate-ethanol (10:1) as solvent. From the study, it was reported that the trigeminal active component in andaliman is an amide.

Waziiroh (2012) studied the isolation of an anti-hyperglycemic in hot compound of dried Japanese pepper and fresh andaliman. From the result, it was reported that water-soluble extract of fresh andaliman fruit had the highest extraction yield, while for dried Japanese pepper the ethyl acetate soluble extract had higher extraction yield. From the TLC result, it can be assumed that the targeted hot compound is in the water-soluble extract of fresh andaliman, and in dried Japanese pepper the part of pungent compound have vaporized with water, therefore the extraction yield of dried Japanese pepper is mainly essential oil. The result suggested that the trigeminal active hot compound in fresh andaliman yield higher result when extracted using ethanol compared to water.

2.3 Spray Drying Encapsulation of Food Flavors

Encapsulation is a process to cover an active compound with a protective wall material to provide certain degree of barrier against evaporation, reaction, or migration in foods. Encapsulation of flavors have been done using many methods such as spray drying, spray chilling or cooling, extrusion, freeze drying, coacervation and molecular inclusion. Selecting the best method depends on the end product requirements of usage and the processing condition that is to be done in the manufacturing (Madene *et al.*, 2006).

Spray drying has been a very common technique in production of dry flavorings. The advantages of this process include availability of equipment, low cost, wide options of carrier, good retention of volatiles and stability of the finished product. In spray drying, infeed material (usually water, carrier and flavor in aqueous form) is atomized into a stream of hot air, causing the atomized particles to dry very rapidly while trapping the volatile flavor components inside the droplets. The powder produced is then collected thorough cyclone. Flavor retention can be maximized through usage of high infeed solids level, high viscosity infeed, optimum inlet (160-210˚C) and high outlet (> 100˚C) air temperatures as well as high molecular weight flavor molecules (Reineccius, 2004).



Note: (1) Inlet of the drying air, (2) heating system, (3) peristaltic feed pump, (4) cyclone, (5) atomizer, (6) inlet temperature controller, (7) outlet temperature controller, (8) final product collector, (9) compressed air, (10) drying chamber, (11) aspiration system

Figure 2.1 Spray drying device
Source: Fernandez-Perez *et al.* (2004)

The basic parts of any types of spray dryers are shown in Figure 2.1. Although there are many types of spray drying, all of them generally includes a part where the incoming air is heated, atomizing the material to be dried into the heated air, a chamber where drying will happen and a part to separate powder and air (such as a cyclone separator). Commonly the infeed material has been prepared before by dissolving the material and forming a coarse emulsion using high shear mixer. This infeed may be homogenized or not, and then it will be pumped into the atomizer. Atomization usually utilizes a single-fluid high-pressure spray nozzle or centrifugal wheel (Reineccius, 2004).

The atomization objective is to increase the surface area of the droplets, in order to optimize the evaporation process of water. After atomized, the droplets will undergoes contact with the heated air, and evaporation of 95% water will happen in just a few seconds. Most spray dryers in flavor industry used co-current design, where the product enters the dryer in the same direction as the drying air. This type of design provides rapid drying which is good to protect the flavor from much heat as in counter current design. The temperature of the spray dried product will not exceed the temperature of the exit air. When the water evaporated, the atomized droplets are cooled down and will only increases to the exit air temperature if the drying rate slows down. The last part consists of separation of the powder from the air (Reineccius, 2004).

Factors influencing retention of volatile flavor during spray drying includes the solid content of infeed, molecular weight and vapor pressure of the volatile compound, type and molecular weight of carrier material, atomization process, drying air velocity and mixing with atomized infeed, inlet and exit air temperature, relative humidity of the inlet air, infeed emulsion size and atomized droplet size as well as dryer feed temperature (Reineccius, 2004).

2.3.1 Encapsulation/ Carrier Materials

Spray drying encapsulation utilizes a carrier or an encapsulating agent, commonly hydrolyzed starches, modified starches, or gum arabic (Reineccius, 1999). The flavor carrier highly influences the shelf life of oxidizable flavor compounds. To be used as encapsulating agent of flavor compound, some requirements have to be met, including unreactivity with core material, easily handle form (low viscosity at high concentration), allow complete removal of solvent, maximum protection of the active ingredient against environmental factors, has a good emulsion stability and effective dispersion to control release of flavor (Madene *et al.* , 2006). There are four major groups of flavor carriers: mono and disaccharides (sucrose, lactose and glucose), hydrolyzed starches (maltodextrin and corn syrup solids), chemically modified (emulsifying) starches and gums. Mono and disaccharides have the advantages of being inexpensive and are often used together with chemically modified starch or gum Arabic to reduce cost. However, they rarely constitute the majority of carrier composition because they tend to undergoes caking or crystallization during storage due to their low glass transition temperature, poor flavor retention and not easily dried (Reineccius, 2004).

Hydrolyzed starch provides advantages of being inexpensive, bland flavor, very soluble (up to 75%) and low viscosity in solution. Higher dextrose equivalent (DE) gives even better protection to oxidation, but also increases the possibility of caking during storage. They are very suitable for hydrophilic volatiles, but they lack emulsifying ability and therefore may not provide sufficient protection for lipophilic volatiles. On the other hand, emulsifying starch has been partially hydrolyzed to give them lipophilic properties. However, they are highly cost and give poor protection to flavor oxidation if used alone (Reineccius, 2004).

In preparing infeed, carrier is firstly hydrated (heated or not) until it reaches desired solids level. There is an optimum infeed solids level for each carrier material in which the maximum solubility of the carrier is achieved;

increasing solids level positively affect the flavor retention, but it cause disadvantages of decreasing drying output. This optimum level is usually determined through experiments. The hydrated carrier is then mixed with the flavoring material to form an emulsion through high shear mixing or homogenization before entering the spray dryer (Reineccius, 2004).

Some carrier material become very viscous at relatively low solids content, and low solid content leads to poor flavor retention. Too high viscosity of infeed delays the formation of droplets during atomization, leading to flavor volatile loss during drying. Carriers which are good emulsifiers and good film former usually produce better flavor retention. Emulsification ability is especially important in retention of lipophilic volatiles but less important for hydrophilic volatiles (Reineccius, 2004).

2.3.1.1 Maltodextrin

Maltodextrin is made from partial hydrolysis of corn flour using acids or enzyme, resulting in polysaccharides consisting of D-glucose units linked by $\alpha(1\rightarrow4)$ glycosidic bonds. Dextrose equivalents (DE) value of maltodextrin shows their degree of hydrolysis, and is inversely related to its average molecular weight. The advantages of using maltodextrin are its bland flavor, low viscosity at high solids ratio and its availability in different molecular weight. However, they lack emulsifying ability and provide relatively low protection to volatile compounds. Maltodextrin has a good solubility in water and provides good encapsulating ability as its bind flavor, form a film layer and lower the oxygen permeability on the matrices surface (Madene *et al.*, 2006).

2.3.1.2 Gum Arabic

Gums are generally bland and tasteless but may have pronounced effect on taste of food such as decreasing sweetness attributed to its viscosity. Gum arabic is most commonly used gum as flavor encapsulating material. It is very versatile for many applications as it has good solubility, low viscosity and good emulsification characteristic, as well as good retention of volatile compounds. It is also suitable for lipid droplets encapsulation as it takes the roles of both surfactant and drying matrix, therefore preventing loss of volatiles in contact to air. Its application is limited, however, since it is more expensive than maltodextrin; therefore there is a need to search for suitable alternatives (Madene *et al.*, 2006).

Combination of gum arabic and maltodextrin as a carrier agent in application to microencapsulation of cardamom oil have shown increase in retention of flavor when gum arabic fraction was increased. Another study reported that in spray dried particles formed by the mixture of maltodextrin and gum arabic, the retention of volatile which is normally >80% depends on the spray drying inlet temperature, emulsion concentration and viscosity, as well as proportion of gum arabic to maltodextrin. In the study of 2-acetyl-1-pyrroline encapsulation by spray drying, it was reported that 70:30 combination of gum arabic to maltodextrin produce capsules with the best quality (Madene *et al.*, 2006).

2.3.2 Inlet and Exit Air Temperature

Inlet temperature highly affects the efficiency of the drying and the damages of flavor retention, while outlet temperature is related to the desired residual moisture content in the final powder product. High inlet temperature may allow rapid formation of semi permeable membrane on the droplet surface, but if too high may cause heat damage to the flavor compound, bubble growth in the droplets and also surface disruption leading to increased drying losses. Optimum inlet air temperature for flavor retention is reported to be 160-210˚C. Higher exit air temperature produce a lower relative humidity of the drying air, therefore allow more rapid drying, better flavor retention and reduced moisture content (Reineccius, 2004).

2.4 Physical Properties of Spray Dried Powder

Considerations in spray drying product performance requirements include particle size and shape, absolute and bulk densities, flowability, dispersibility, moisture content, appearance, flavor load, shelf-life, stability to caking, structural strength, release properties, initial emulsion stability and reconstituted stability **(**Reineccius, 2004).

2.4.1 Particle Densities

Absolute density is defined as weight of a given particle volume of powder, while bulk density is defined as the weight of a given total (or bed) volume of powder. Absolute density is measured by using helium pycnometry, while bulk density is determined by placing a known weight of powder in a graduated cylinder and then shaking it until the powder bed reaches certain volume. Higher inlet temperature and lower temperature differences usually resulted in powder with lower density, while higher infeed total solids will increase the absolute density. Bulk density is importance to determine how much weight of a material will fit into a packaging. It is influenced by the absolute density, particle shape and particle size (Reineccius, 2004).

2.4.2 Appearance

Appearance of spray dried flavoring in terms of color is usually influenced by the particle size. Small particles have a lighter color while larger or agglomerated particles are seen as darker and richer in color. Particle size is influenced by the temperature of drying. When drying occurs rapidly, larger particles will be produced due to early formation of structure which leads to incomplete shrinking and moisture removal. High total solids of infeed also cannot shrink properly and will produce larger particle size (Reineccius, 2004).

2.4.3 Moisture Content

Moisture content of the final product is mainly affected by the drying temperature. Low moisture can be achieved by increasing the inlet temperature and decreasing the temperature difference of inlet and outlet temperature (ΔT). High inlet temperature holds more moisture as they are released from the droplets, resulting in a drier powder. Spray dried powders usually contain 1-6% moisture (Reineccius, 2004).

2.4.4 Dispersibility

Dispersibility is important when reconstitution of the powder is going to be done. It is highly influenced by the particle size, particle density and types of carrier. Gum Arabic is difficult to disperse in cold water, while modified starches and starch hydrolysates are easier to disperse. In final application, dry blending of the flavors with other ingredients can reduce their dispersing problems (Reineccius, 2004).

2.4.5 Structural Strength

High density and lower load powders are preferred because particle that is high in flavor or low in density (hollow), are easily to crack or damaged. Usually spray dried powders contain 20% flavor, because higher loads of flavor may leads to fractural damage of the particle. This will cause exposure to oxygen and loss of flavor through evaporation, since there are fewer barriers to degradation.

2.5 Sensory Evaluation

Sensory evaluation is defined as a scientific method used to measure and interprets responses to products as perceived through the human senses. In product development, sensory evaluation help to determine important sensory attributes that may increase product acceptability, identify consumer segments, evaluate new concepts and measure sensory changes in products with altered processing or ingredients. In general, sensory evaluation can be divided into objective and subjective testing. Objective testing focus on evaluating product's sensory attributes commonly by a selected or trained panel, while subjective or affective test focus on measuring the consumer reaction to product's sensory attributes. Objective test can be divided into discriminative, ranking, scoring and descriptive test (Kemp *et al.*, 2009; Lawless and Hildegarde, 2010).

Table 2.1 Test methods of sensory evaluation

Class	Question of interest	Type of test	Panelist type
Discriminative	Are products perceptibly different in some way?	Analytic	Screened for sensory acuity, oriented to test method, in some cases are trained
Descriptive	How do samples differ in specific sensory characteristics?	Analytic	Screened for sensory acuity and motivation, trained or highly trained
Affective	How well are products liked or preferred?	Hedonic	Untrained

Source: Lawless and Hildegarde (2010)

2.5.1 Discriminative Test

Discrimination test is used to evaluate if there is a difference or similarity between two or more samples. Data is analyzed using statistical significance to determine whether the sample can be concluded as different or similar. This type of test usually performed in panel training, determining sensitivity threshold, investigating the effect of processing or ingredient changes and also as a preliminary assessments. In discrimination test, ‘no difference’ option can be either allowed or the panelists are forced to make a decision. When ‘no difference’ option is allowed, there are two common approaches to the data analysis; the ‘no difference’ responses are ignored or the ‘no difference’ response is split proportionally between the products (Kemp *et al.*, 2009).

Discriminative test can be divided to overall difference test and attribute specific test. Attribute specific test is used to determine if difference exists between two samples in terms of specific attribute such as sweetness, hardness or intensity of aroma. On the other hand, in overall difference test, assessors may use all available information to judge the sample. In some cases, this test can be specified to one attribute, but this will require the masking of other sample attributes, such as using colored light to mask the sample appearance. Several forms of overall difference test are triangle test, duo-trio, difference from control test, same-different test and A-not A test (Kemp *et al.*, 2009).

2.5.2 Rating (Scoring) Test

Ordinal scales are the most basic scale used to measure perceived intensity. The most commonly used ordinal scales are ranking and rating (scoring) scales. Rating scales, as compared to ranking, provide a continuous response.

Scoring test for intensity measurement requires panelist to score sample, either on line or category scales, for perceived intensity of a sensory attribute. This method measures the amount of difference between samples (Watts *et al.*, 1989).

The scale used may have a word or number for every scale category, or anchored only at the extremes. Using no numbers and only two words anchor provide a less complicated scale and also minimize bias. Words chosen to anchor the rating scale must meaningful and not ambiguous. They must be a general terms and not personal preferences word that may reflect different perception to different people. Use of intensity measures combined with word anchors that is understood by the subjects, can be a very successful procedure since optimal sensitivity and minimal variability is achieved (Watts *et al.*, 1989).

One of the scales that can be used for rating is line scale. Line scales, with endpoints of the scale labeled, are commonly utilized to quantify characteristics. Scoring test can be done on line scales or category scales. It measures the perceived intensity of a sensory characteristics and the amount of difference between samples. Line scale result is analyzed by converting panelists' marks to numerical scores. This is done by measuring the distance in cm from the lowest intensity point to the panelists' marks (Watts *et al.*, 1989).

Elimination of number from the scale and from the panelists' responses in scaling method, as compared to scoring method, eliminated two sources of bias, which are avoidance (or preference) for particular number with negative (or positive) connotations, as well as bias from the subjects who changed use of number over time. This can be a problem since it is unknown if the change is related to the sample's different or true bias (Stone and Sidel, 2004).

2.5.3 Descriptive Test

Descriptive test is conducted to generate a more precise sensory description of a product and also describe and quantify the sensory attributes between products, usually by a trained panelist (Kemp *et al.*, 2009). In descriptive test, it is important to carefully determine vocabulary description of attribute, characteristics, character notes and descriptions through discussion and agreement among the panel members. Intensity and quantification of each descriptive analysis must be ensured to reflect the level, scale, category and ratio of each parameter (Lawless and Heymann, 1998). According to Gacula (1997), the effectiveness of descriptive analysis depends on four factors which are the panelist training and experience, panel leader, analysis implementation and commitment of the panel.

According to Hootman (1992), quantitative descriptive analysis (QDA) principle is based on the panelist's capability to verbalize their perception on a product with a trustworthy consistency. According to Stone and Sidel (2004), humans are better at judging relative sensory differences rather than evaluating absolute differences. QDA is distinct from other descriptive methods since it allows panelists to make relative judgment with high degree of precision, as compared to other method such as spectrum analysis which forces panelists to finalize the absolute difference in products. QDA includes complete listing of sensory attributes as based on panel perceptions, order of occurrence, relative intensity measure of each attribute and statistical analysis of the responses. This method consists of formal panelist screening and training, sensory language development and usage, and repeated scoring on products in order to obtain a complete quantitative description (Hootman, 1992).

Panel leader is responsible to conduct all the screening process, organization and implementation of screening test and also selecting subject for training, but does not participate as a panelist in any screening or training. Panel leader can also assist the panel to clarify certain product attribute, obtain references and also determine until when the training will be completed (Hootman, 1992).

Panelist screening procedure includes three basic step including product usage and familiarity, discrimination ability and task comprehension. This step will select individuals that are significantly able to discriminate differences and eliminate those that have difficulty in following instructions or are inconsistent in their discrimination ability. Usually QDA is started with 25 individuals to obtain an expected 12 to 15 qualified panelists. These individuals will then be trained, with the objective is to develop a scorecard, containing sensory language that describes the panelists' perception of the products and has been agreed by the entire panel. This also includes explanation for each word and standardized procedure for product evaluation, and also scoring practice to familiarize the panelists with the scale. References may also used to generate sensory terminologies when there is confusion during training sessions. In some cases, discrimination trials may be conducted for the product which is going to be described (Hootman, 1992; Stone and Sidel, 2004).

The actual product evaluation will be conducted individually in separated booths, without reference standards. The quantitative analysis will be done using unstructured line scale. For QDA, a line scale that is 15.29 cm (6 in) length with sensory intensity words anchored 0.5 inch from each end should be used (Stone and Sidel, 2004). Since panelists are allowed to mark any part of the scale, the difference among products produced by QDA will be a relative measurement. According to Gacula (1997), spider web graph is commonly used to present the analysis result of QDA. Figure 2.2 shows the example of QDA's spider web.

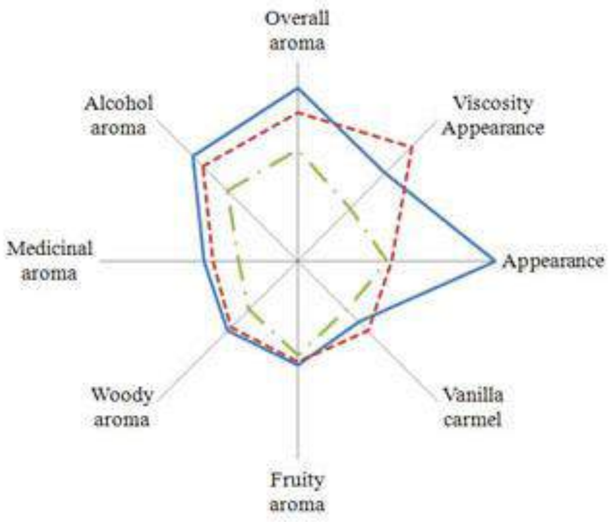


Figure 2.2 Example of spider web based on QDA result
Source: Stone and Sidel (2004)

CHAPTER III

RESEARCH METHODOLOGY

3.1 Materials and Equipment

Materials that were used in the research include fresh andaliman (*Zanthoxylum acanthopodium* DC.*)* (Appendix A) which was obtained from traditional market Pasar Senen in Jakarta, maltodextrin 10 DE and gum Arabic (PT. Prima Abadi), aroma standards including citral, citronellal, limonene, vanilline, geranyl acetate, β ocimene, α pinene, ethyl propionate and sour pungent (acetic acid) (International Flavor & Fragrance Inc.), ethanol and ethyl acetate food grade (CV. Anugrah Jaya Chemical), filter paper Whatman no.1, Na_2SO_4 and other materials for sensory analysis.

Equipment used in this research include analytical balance (*Ohaus Adventurer*), dry blender, refrigerator, freezer, pipette, spatula, tongs, dark bottles, funnel, spray dryer (*Buchi 190*), graduated cylinder, beaker glass, erlenmeyer flask, evaporating dish, Buchner vaccum filter, vacuum rotary evaporator (*Rotavapor Buchi R-210*), oven (*Memmert UNE 800*), hand refractometer (*Atago*), stirrer (*Heidolph RZR-1*), dessicator (*Wertheim*), chromameter (*Konica Minolta CR-400*), a_w meter (*Novasina MS1 Set-*a_w) and other equipments for physical and sensory analysis.

3.2 Research Methods

The research was conducted in two parts including the preliminary research and main research. The preliminary research was to determine the solvent that can obtain best extraction of andaliman flavor and its trigeminal active component. Meanwhile the main research was determination of the most effective spray drying condition, in terms of inlet temperature and carrier agent composition, to encapsulate flavor and trigeminal characteristic of andaliman powder, as well as powder characterization by physicochemical analysis and QDA (Quantitative Descriptive Analysis) to fresh andaliman and andaliman powder.

3.2.1 Preliminary Research

The preliminary research was done to determine the best solvent to extract andaliman flavor and trigeminal sensation characteristic. The extraction of andaliman trigeminal active compound was conducted following the maceration procedure conducted by Waziiroh (2012). In the research, it was reported that ethanol extract of fresh andaliman produced the highest yield of hot compound.

Another solvent used in this research was combination of ethyl acetate and ethanol in reference to the previous research conducted by Wijaya (2000). The ratio of ethyl acetate to ethanol was determined by trial and error. Extraction was also tested using water as the solvent, in consideration of polarity of the previously tested solvents and safety of the produced andaliman extract. There were three types of solvent to be tested in this preliminary research: water, ethanol and ethyl acetate: ethanol (1:1).

For the extraction, 100 gram andaliman fruits were crushed in dry blender for 10 seconds and then mixed with 220 ml solvent. The extraction was conducted using maceration method in room temperature for 48 hours. The mixture was then vacuum filtered using Whatman filter paper no.1, and the residue in the filter paper was discarded. The solvent was then removed from the extract using vacuum rotary evaporator at 55˚C. Procedure for andaliman extraction can be seen in Figure 3.1.

Andaliman fruits

↓

Crushed using dry blender (10 seconds)

↓

Added with solvent
(water, ethanol, ethyl acetate:ethanol=1:1)

↓

Maceration in room temperature for 48 hours

↓

Vacuum filtration using Whatman filter paper no.1

↓

Filtrate

↓

Removal of solvent by vacuum rotary evaporator (T=55˚C)

↓

Andaliman extract

Figure 3.1 Flowchart of andaliman extraction
Source: Waziiroh (2012) with modification

Determination of the best extract was done using sensory test by scaling method towards 20 panelists (with replication). The objective of this sensory test was to find andaliman extract that has the most similar taste to fresh andaliman. Three extract samples were presented at a time to the panelists in randomized order, while the fresh andaliman sample was presented to the panelists on separated session to avoid direct comparison. The questionnaire for andaliman extract scaling test can be seen in Appendix B.

The sensory data was statistically analyzed using one-way ANOVA and compared using Duncan Test in SPSS software. The research design was completely randomized design with one factor (types of solvent) with three levels (water, ethanol, ethyl acetate: ethanol (1:1)) and two replications. The mathematical model of completely randomized design was:

$$Y_{ij} = \mu + A_i + \varepsilon_{ij}$$

where:

Y_{ij} = Observation value of andaliman extract using solvent i and with replication j

μ = Real mean data

A_i = Effect of solvent on andaliman extract on level i

ε_{ij} = Error

The hypotheses of this research were:

H_0 = There is no effect of types of solvent on flavor and trigeminal sensation intensity of the andaliman extract

H_1 = There is effect of types of solvent on flavor and trigeminal sensation intensity of the andaliman extract

3.2.2 Main Research

The main research was divided into two parts which were the determination of spray drying condition to produce andaliman powder and powder characterization through physicochemical analyses and Quantitative Descriptive Analysis (QDA).

3.2.2.1 Determination of Andaliman Powder Spray Drying Condition

The factors used were spray drying inlet temperature, ratio of andaliman extract to carrier agent and ratio of maltodextrin to gum arabic used to formulate the spray drying infeed mixture. The spray drying procedure was based on the research previously conducted by Turchiuli *et al.* (2011), with modification on the inlet temperature, infeed solid content and carrier agent concentration. The infeed mixture was prepared by hydration of the carrier agent (maltodextrin and gum arabic mixture) in water, followed by the addition of andaliman extract and mechanical stirring using Heidolph stirrer for 10 minutes at speed 3.

The first step was the spray drying inlet temperature determination. For this purpose, mixture of andaliman extract and carrier agent in water (extract: carrier: water= 1:6:100 (w/w/v)) were prepared and spray dried using three different inlet temperature (135˚C, 150˚C and 165˚C). The carrier agents used were maltodextrin (MD) and gum arabic (GA) (3:2), in reference to Turchiuli *et al.* (2011). The flowchart of the spray drying procedure can be seen in Figure 3.2.

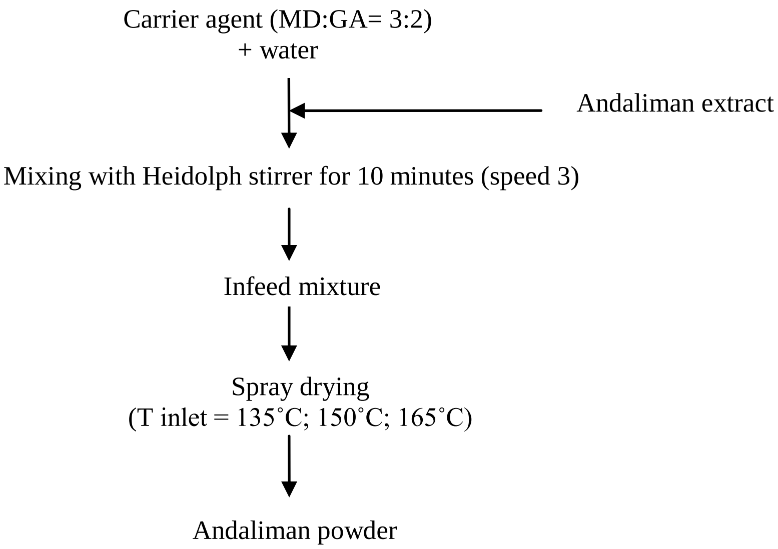


Figure 3.2 Flowchart of andaliman powder spray drying
Source: Turchiuli *et al.* (2011) with modification

Sensory test was conducted to determine the andaliman powder with the highest intensity of flavor and trigeminal sensation after spray drying application. Scaling method using line scales (Appendix C) was performed towards 20 panelists (with replication). The result of the sensory test was statistically analyzed using one-way ANOVA and compared by Duncan Test in SPSS software. The research design was completely randomized design with one factor (inlet temperature) with three levels (135˚C, 150˚C, 165˚C) and two replications.

The mathematical model of completely randomized design was:

$$Y_{ij} = \mu + A_i + \varepsilon_{ij}$$

where:

Y_{ij} = Observation value of andaliman powder using inlet temperature i and with replication j

μ = Real mean data

A_i = Effect of inlet temperature on andaliman powder on level i

ε_{ij} = Error

The hypotheses of this research were:

H_0 = There is no effect of inlet temperature on flavor and trigeminal sensation intensity of the andaliman powder

H_1 = There is effect of inlet temperature on flavor and trigeminal sensation intensity of the andaliman powder

The chosen inlet temperature was then used to determine the andaliman extract to carrier agent ratio and maltodextrin to gum arabic ratio that can obtain the andaliman powder with highest flavor and trigeminal sensation retention. There were nine treatments of spray drying, consisting from three ratio of andaliman extract to carrier agent (1:4, 1:6 and 1:8) and three ratio of maltodextrin to gum arabic (3:2, 1:1 and 2:3). All infeed formulations were made using 100 ml water (w/v).

The resulting powder were subjected to sensory analysis using scaling test towards 20 panelists, similar to the one conducted for the inlet temperature determination (Appendix C). The result of the sensory test was statistically analyzed using two-way ANOVA in SPSS software. The research design used was completely randomized design with two factors 3 x 3. The variables used were the ratio of andaliman extract to carrier agent and ratio of maltodextrin (MD) to gum arabic (GA). The mathematical model of completely randomized design with two factors was as follows:

$$Y_{ijk} = \mu + A_i + B_j + AB_{ij} + \varepsilon_{ijk}$$

where:

Y_{ij} = Observation value of andaliman powder produced using ratio of andaliman extract to carrier agent i and ratio of maltodextrin to gum arabic j, with k replication

μ = Real mean data

A_i = Effect of extract: carrier agent on level i (i = 1:4, 1:6, 1:8)

B_j = Effect of MD: GA on level j (j = 3:2, 1:1, 2:3)

AB_{ij} = Effect of interaction between andaliman extract: carrier agent on level i and MD: GA on level j

ε_{ijk} = Error

The hypotheses of this research were:

H_0 =

1) There is no effect of andaliman extract to carrier agent ratio on the flavor and trigeminal sensation intensity of andaliman powder.
2) There is no effect of maltodextrin to gum arabic ratio on the flavor and trigeminal sensation intensity of andaliman powder.
3) There is no effect of interaction between andaliman extract to carrier agent ratio and maltodextrin to gum arabic ratio on the flavor and trigeminal sensation intensity of andaliman powder.

H_1 =

1) There is effect of andaliman extract to carrier agent ratio on the flavor and trigeminal sensation intensity of andaliman powder.
2) There is effect of maltodextrin to gum arabic ratio on the flavor and trigeminal sensation intensity of andaliman powder.
3) There is effect of interaction between andaliman extract to carrier agent ratio and maltodextrin to gum arabic ratio on the flavor and trigeminal sensation intensity of andaliman powder.

3.2.2.2 Andaliman Powder Characterization

The best andaliman powder selected was analyzed for its powder properties and characteristics including powder yield, moisture content, water activity, color, solubility, bulk density and hygroscopicity. Descriptive analysis using QDA method was also conducted for the fresh andaliman and andaliman powder produced by spray drying.

3.3 Analysis Procedures

3.3.1 Rating Test with Line Scale (Stone and Sidel, 2004 and Watts *et al.*, 1989, with modification)

Determination of the best extraction and best spray drying condition were done using scaling test towards 20 panelists (with replication). Preparation of the fresh andaliman sample was done by crushing it using dry blender, while the andaliman extracts and andaliman powder were diluted in water (2%) for this purpose. Fresh andaliman and three solvent extracts were assessed for five parameters, including pungent (pepper-like aroma), sour (citrus-like) aroma, bitter taste, sour taste and trigeminal sensation (tongue-numbing) (Appendix B). Meanwhile the andaliman powder samples were assessed for the intensity of aroma, taste and trigeminal sensation (Appendix C).

Panelists were instructed to place fresh andaliman sample on their tongue for 30 seconds and then spit out, while the extract and powder solution were presented using dropping pipette to standardize the amount of sample assessed (3 drops for each assessment). Panelists were then asked to place a mark on an unstructured 15 cm line scale with intensity words (none to strong) on each end, according to the perceived intensity of each parameter. The trigeminal sensation parameter were assessed 30 seconds after the sample were placed on tongue to provide time for the trigeminal sensation to develop. Mineral water and crackers were provided to neutralize panelists' tongue as well as to reduce carry over and fatigue.

3.3.2 Andaliman Powder Yield

The yield of the andaliman powder was calculated from the weight of andaliman powder produced as compared to the initial weight of andaliman fruits used. The formula was:

$$\text{Yield (\%)} = \frac{\text{total weight of andaliman powder (g)}}{\text{total weight of initial andaliman fruits (g)}} \text{ x } 100\%$$

3.3.3 Moisture Content (AOAC, 2000)

The method used in moisture content determination was AOAC direct gravimetric method with slight modification. Empty dish were placed in the oven at 105˚C for 3 hours for drying, and then transferred to the desiccators. After cooling down, the dish was weighed. Two grams of sample was weighed into the dish. The dish containing sample was then placed in the oven at105˚C for 3 hours. The dish and sample were weighed again. The moisture content was calculated using the formula:

$$\text{Moisture (\%)} = \frac{(\text{weight of sample before drying (g)} - \text{weight of sample after drying (g)})\text{x}100\%}{\text{weight of sample before drying (g)}}$$

3.3.4 Water Activity (Kanpairo *et al.*, 2012)

Water activity of the andaliman powder was measured using the water activity meter (a_w meter). Sample was placed in a_w meter for about 10 minutes, and the displayed reading was noted down after it has shown stable value. Water activity value ranges from 0.00 (absolute dry) to 1.00 (pure water).

3.3.5 Powder Solubility (Kanpairo *et al.*, 2012)

About 0.5 grams of andaliman powder (W_1) was placed into a beaker glass, followed with addition of 5 ml distilled water at 25˚C. The mixture was gently stirred using a spatula until all the particles were dissolved thoroughly. The solution was then filtered through Whatman filter paper no.1 which has been weighed before (W_2). Then the filter paper was dried in oven (100˚) for 4 hours, cooled down in a desiccator and then re-weighed (W_3). The powder solubility was calculated using the formula:

$$\% \text{ Solubility} = 100 - \frac{\{[W_3 - W_2]\ X\ 100\}}{W_1}$$

3.3.6 Color (Hutching, 1999)

The color analysis of the andaliman powder was measured by Hunter method using Chromameter CR-400/410 instrument. Powder sample was placed into the sample container. Chromameter was turned on and calibrated. Sample was then shot and the displayed L, a and b values were noted down. The values were reported as L (lightness), a (+a is red, -a is green) and b (+b is yellow, -b is blue). L value ranges from 0 (black) to 100 (white), a value indicates red to green color with +a from 0 to 100 for red color and –a from 0 to -80 for green color, while b value indicates blue to yellow color with +b from 0 to 70 for yellow color and –b from 0 to -70 indicating blue color. The measurement was done in two replications for each sample. From the L, a and b value obtained, ˚Hue was calculated using the formula:

$$^{\circ}\text{Hue} = \text{arc tan}\left(\frac{b}{a}\right)$$

3.3.7 Bulk Density (Kanpairo *et al.*, 2012, with modification)

Bulk density was measured by gently placing 2.0 ± 0.1 grams of andaliman powder sample into a dried 10 ml cylinder and then the cylinder was gently tapped for 3 times. The volume of the powder was recorded and the bulk density was calculated using following formula:

$$\text{Bulk density} = \frac{\text{Weight of andaliman powder (g)}}{\text{Volume of andaliman powder (ml)}}$$

3.3.8 Hygroscopicity (Cai and Corke, 2000, with modification)

To measure hygroscopicity, approximately 2 grams of andaliman powder sample was placed at room temperature (25˚C) inside an airtight desiccator filled with Na_2SO_4 saturated solution (RH = 81%). The sample was left for 1 week and the hygroscopic moisture was weighed and expressed as grams of moisture per 100 grams of dry solids (g/ 100 g).

3.3.9 Quantitative Descriptive Analysis (QDA) (ASTM, 1981)

QDA was performed in order to analyze the flavor characteristics of the andaliman powder produced in comparison to fresh andaliman. The QDA analysis was conducted by panelists that have passed the selection and have participated in the training procedures. The QDA procedure includes panelist selection, panelist training and quantitative analysis.

3.3.9.1 Panelist selection (ASTM, 1981)

Panelist selection was firstly conducted in order to select panelists with good sensory sensitivity. The selection was done to 25 students of Food Technology Universitas Pelita Harapan who were in their 7th semester. These candidates were selected because they have already obtained the basic of sensory evaluation in class. Preliminary screening was done by asking the panelists to fill questionnaire regarding time spare, willingness and commitment of each panelists to participate in the training continuously. Medical history and food habits of each panelist were also observed through the questionnaire. Furthermore, panelists were asked to do scaling exercise in order to obtain information regarding panelists' scaling accuracy (Appendix D).

The next part of panelist selection was triangle test for basic taste and aroma. The objective was to know the ability of each panelist in differing taste and aroma, as well as to measure their sensitivity towards the characteristics of each flavor. Standard solutions that were prepared in testing the basic taste include sucrose (sweet), citric acid (sour), NaCl (salty) and caffeine (bitter) as mentioned in Table 3.1. The panelist candidates will be asked to taste the presented sample and then select one that is different between the three samples.

Table 3.1 Standard solution concentration used in triangle test

Ingredients	Concentration		
Sucrose	1 %	2%	4%
Citric acid	0.035 %	0.07%	0.14%
Salt (NaCl)	0.1 %	0.2%	0.4%
Caffeine	0.035 %	0.07%	0.14%

Source: ASTM (1981)

Triangle test for aroma was also conducted in order to measure the ability of panelist candidates in differentiating characteristics of similar aroma properties. The standard odorants that were prepared include citral, citronellal, limonene, vanilline, geranyl acetate, β ocimene, α pinene, ethyl propionate and sour pungent (acetic acid). Triangle test for aroma was similar with triangle test for basic taste. The candidates were asked to choose a different sample between the three odorants presented. The questionnaire for triangle test of taste and aroma can be seen in Appendix E and F, respectively. Panelists that have answered at least 16 questions correctly out of 24 for either taste or aroma test were selected to participate in the training procedures.

3.3.9.2 Panelist training (ASTM, 1981)

Training procedure was necessary to develop the sensitivity of the selected panelists and also to maintain decision consistency, in order to prepare the panelists as 'trained panel'. Selected panelists received trainings related to terminology, definition and evaluation procedures, as well as to increase their skills in discriminating flavor intensity. This was done through scaling and ranking test for taste and aroma samples with different intensity. Trainings were conducted in a conference style room (Moskowitz *et al.*, 2012). Sessions were led by the panel leader, whom should provide appropriate training products and reference material, but should not participate in the product description analysis. Training was conducted for a period of approximately two months, consisting of eight sessions with each session conducted for one to two hours. The training activities for each session were shown in Table 3.2.

Table 3.2 Panelist's training schedule for Quantitative Descriptive Analysis

Session	Training activity
1	Introduction to QDA training, scaling and ranking test, sniffing and tasting technique Training session scheduling
2	Introduction to andaliman sample and discussion of sample's flavor attributes
3	Introduction of sample's taste attributes, development of taste vocabulary
4	Ranking and scaling test for taste
5	Introduction of andaliman related odorants and development of aroma vocabulary
6	Ranking and scaling test for aroma
7	Focus group for qualitative flavor analysis of andaliman
8	Ranking and scaling test for taste and aroma attributes obtained from qualitative test

Panelists were firstly introduced to fresh andaliman sample and powder, as well as terminologies of flavor that may possibly occur in fresh andaliman sample and andaliman powder. Then panelists were then familiarized with rating and scaling method for taste and aroma. Panelists were also introduced to common terminology of aroma that possibly occurs in andaliman. Flavor terminology training objective was to standardize the concept of terminology that can be communicated between panelists (Lawless & Heymann, 1998). Then, panelists were trained for rating and scaling test for taste and aroma. The rating and scaling test were done in replicates until the panelists were considered as able to make correct and consistent judgment. The questionnaire for scaling test of taste and aroma can be seen in Appendix G and H, respectively, while questionnaire for ranking test of taste and aroma can be seen in Appendix I and J, respectively.

The next step was a qualitative flavor analysis in order to obtain sensory descriptions subjectively, and then followed with scoring sheet arrangement for quantitative analysis. Qualitative analysis was done in a focus group, guided by a group leader chosen among the panelists. Panel leader task were to provide information regarding the overall attributes known to be related to andaliman sample without influencing the discussion result.

3.3.9.3 Quantitative analysis (ASTM, 1981)

Intensity determination of sensory perception were done by the trained panelists using unstructured line scales (15.29 cm or 6 inches), anchored 12.7 mm (1/2 in.) from each end by a pair of terms which explain the attribute being tested. The samples presented in the test were the andaliman powder, along with the fresh andaliman sample that has been crushed in order to compare the flavor characteristics of the two samples. The trained panel filled the QDA scoresheet for taste and aroma (Appendix K and L, respectively) in sensory laboratory under defined conditions (temperature and light) in separated individual cabins.

Panelists were asked to place a vertical mark across the line at a point which best reflect the magnitude of perceived intensity of tested attribute. Each sample evaluation was done in four replications. Data analysis was done by converting panelists' marks to numerical scores. This is done by measuring the distance in cm from the left (lowest) anchor point to the panelists' marks (Watts *et al.*, 1989). The QDA results were analyzed using t-test and visualized in form of spider web.

CHAPTER IV

RESULTS AND DISCUSSION

Andaliman (*Zanthoxylum acanthopodium* DC.) used in this research were obtained from Pasar Senen, Jakarta. The condition of the andaliman obtained consisted of mostly fresh and young green to dark-green fruits, mixed with small amount of mature red fruits. Andaliman were bought on the day they were received by the supplier from their plantation, cleaned and separated from leaves and twigs, and then stored in freezer overnight before being used for extraction on the next day. Fresh andaliman in this research refers to andaliman fruits which have been frozen for maximum three days.

Figure 4.1 Andaliman after cleaned and separated from leaves and twigs (scales in centimeter)

4.1 Preliminary Research

In the preliminary research, andaliman extraction was conducted by maceration method on 100 gram andaliman fruits, using three different solvents (water, ethanol, ethyl acetate: ethanol= 1:1). The yield for the andaliman extraction can be seen in Table 4.1.

Table 4.1 Yield of andaliman extraction

Solvent	Yield (%)
Water	4.07
Ethyl acetate: ethanol (1:1)	4.22
Ethanol	3.83

Ethyl acetate: ethanol (1:1) provided combination of polar and semi-polar solvent characteristic, and therefore may result in higher yield of extraction since it extracts both polar and semi-polar compounds. In this research, however, it can be observed that there was no significant difference between the extraction yields ($p>0.05$) (Appendix N). Previous study by Waziiroh (2012) reported that yield of andaliman extraction by ethanol was 3.027%.

The andaliman extracts were subjected to scaling test by 20 panelists. Fresh andaliman and three solvent extracts were assessed for five parameters, including pungent (pepper-like aroma), sour (citrus-like) aroma, bitter taste, sour taste and trigeminal sensation (tongue-numbing). The result of the scaling test can be seen in Appendix O and the result of the statistical analysis can be seen in Appendix P. Based on Appendix P, for pungent aroma, sour aroma and trigeminal sensation parameter, both ethyl acetate: ethanol (1:1) and ethanol extract were shown to be not significantly different from andaliman. As for sour taste parameter, only ethyl acetate: ethanol (1:1) extraction showed no significant difference compared to fresh andaliman. However, for bitter taste parameter, both ethyl acetate: ethanol (1:1) and ethanol extract shown significant difference to the fresh andaliman.

Figure 4.2 visualized the scaling result of andaliman extract and fresh andaliman in a spider web. The fresh andaliman as the control was observed to have highly pungent and sour aroma, low bitter taste, relatively sour taste and strong trigeminal sensation. According to Wijaya *et al.* (2001), andaliman has the characteristic of citrusy and peppery aroma. The result of GC-MS analysis in the study of andaliman volatile component by Wijaya *et al.* (2001) showed that there are two major flavor compounds in andaliman, geranyl acetate and limonene, with sour floral citrusy aroma and sweet, citrus-peel-like aroma, respectively. Limonene was also reported as major flavor component in other Zanthoxylum species, including the Japanese pepper (*Zanthoxylum piperitum* DC) or *sansho*, *Zanthoxylum simulans* and *Zanthoxylum bungeanum.* From the GC/O analysis in the same study, it was reported that andaliman flavor is dominated by a strong and fresh citrus aroma, while from the AEDA (aroma extract dilution analysis) result, it was reported that the potent odorant giving andaliman its characteristic flavor is citronellal with its strong and warm citrusy aroma. Andaliman also possess a numbing trigeminal sensation produced by a compound namely (2E, 6Z, 8E, 10E-N-(2'-methylpropyl)-dodecatetraenamide) which can be found in the pericarp of the fruit (Wijaya, 2008).

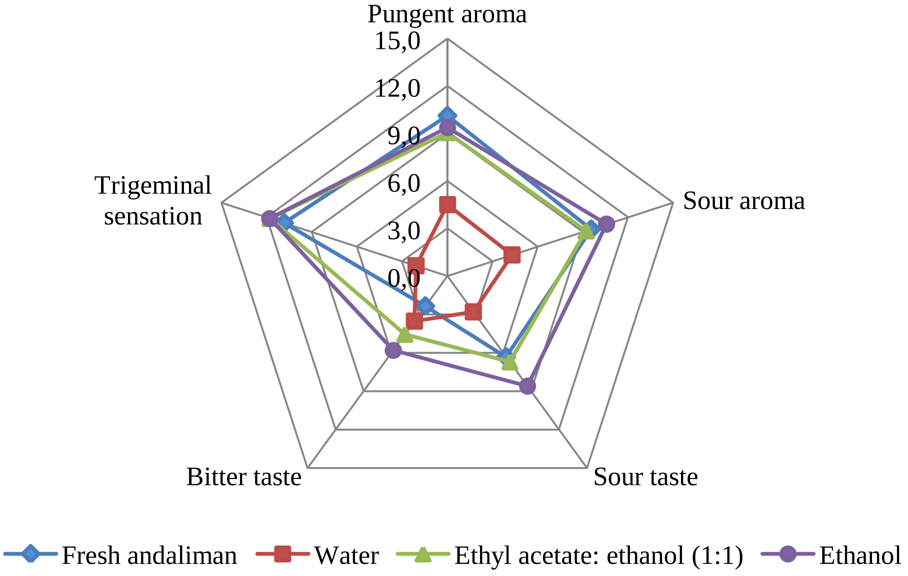


Figure 4.2 Spider web of fresh andaliman and andaliman extracts flavor

Based on Figure 4.2, it can be seen that water extraction resulted in very low intensity of flavor and trigeminal sensation compared to fresh andaliman and other extracts. Previous studies by Kumar and Paridhavi (2011) and Bhattacharya and Zaman (2009) reported that water extraction of *Zanthoxylum nitidum* stem and *Zanthoxylum limonella* fruits mainly extracted carbohydrates and proteins, besides several bitter taste compounds namely saponins and glycosides. This may explains the low overall flavor intensity of andaliman water extract in this study, except for its bitter taste intensity which was higher compared to fresh andaliman.

Except for water extract, other andaliman extracts flavor profile was comparable to that of fresh andaliman. Both ethyl acetate: ethanol (1:1) and ethanol extract showed the characteristic of sour and pungent aroma, as well as trigeminal sensation, which was higher than fresh andaliman. However, it can be observed that ethanol extract resulted in significantly higher bitter taste, while bitter taste was observed at low intensity in the fresh andaliman.

Bhattacharya and Zaman (2009) studied the organoleptic properties of *Zanthoxylum nitidum*, one of the large Zanthoxylum genus found in India reportedly used for medicinal purposes. The fresh stem bark of *Z. nitidum* has the characteristic of aromatic and bitter taste. It was further reported that ethanol extraction of *Zanthoxylum nitidum* stem bark resulted in extract containing alkaloids and flavonoids, compounds which are known to have astringent and bitter taste profile. Since andaliman is from the same Zanthoxylum genus, high intensity of bitter taste in andaliman ethyl acetate: ethanol (1:1) and ethanol extract may be resulted from stems that was not removed properly during andaliman cleaning. The stem barks that were extracted together with the fruits therefore cause the andaliman extracts to have prominent bitter taste.

Several other studies also have demonstrated presence of bitter tasting compounds in *Zanthoxylum* fruits extract. Study by Alphonso and Saraf (2012) on secondary metabolites of a medicinal plant from Zanthoxylum genus, *Zanthoxylum rhetsa* (Roxb.) DC reported that ethanolic extract of the fruits showed presence of glycosides, flavonoids, bitter principles, coumarins and terpenoids. Kumar and Paridhavi (2011) studied the physiochemical properties of *Zanthoxylum limonella* Alston, another genus of Zanthoxylum that has bitter taste characteristic and commonly used as traditional medicine in India. Beside several alkaloids reported to be isolated from its stem bark, ethanol extract of its fruit also reported to result in extract containing alkaloids, tannins and flavonoids.

Meanwhile, Babu *et al.* (2007) reported identification of flavone glycosides from *Zanthoxylum acanthopodium* DC fruits, namely herbacetin-7,8,4'-trimethyl ether (tambulin), 8-O-glucosyl-gossypetin-7,4'-dimethyl ether (tambuletin) and herbacetin-8,4'-dimethyl ether, as well as a newly identified flavone glycosides characterized as 7-O-α-D-glucosyl-3,8-dihidroxy-2-(3-hidroxy-4-methoxyphenyl)-5-methoxy-4*H*-1-benzopyran-4-one. According to Drewnowski and Carneros (2000), despite their functional properties, bioactive compounds such as plant based phenols and polyphenols, flavonoids, isoflavones, terpenes and glucosinolates are bitter and astringent.

Bitter taste has an extremely low threshold, and foods that taste bitter tend to be rejected by consumers. The high intensity of bitter taste in ethanolic extract of andaliman therefore needs further study regarding its consumer acceptance level. Based on the scaling test result, it can be seen that ethyl acetate: ethanol (1:1) extraction produced andaliman extract with the most similar flavor profile compared to fresh andaliman. Therefore ethyl acetate: ethanol (1:1) was chosen as the solvent for andaliman extraction.

4.2 Main Research

4.2.1 Effect of Spray Drying Inlet Temperature

The first part of the main research was to select an inlet temperature for spray drying of andaliman powder that can maintain its flavor and trigeminal characteristic. Carrier agent consisting of maltodextrin and gum arabic in 3:2 ratio was used, with the ratio of andaliman extract to carrier agent was 1:6. The infeed mixture (65 ml) was then spray dried at inlet temperature of 135˚C, 150˚C and 165˚C, with other parameters of spray drying maintained constant (flow of spray gas at 600L/ h, aspirator rate at 100% and spray pump at 30%). The spraying time noted from the spray drying process can be seen in Appendix Q while outlet temperature, powder yield and moisture content were shown in Table 4.2.

Table 4.2 Outlet temperature, powder yield and moisture content of andaliman powder spray dried with different inlet temperatures

Inlet temperature (˚C)	Outlet temperature (˚C)	Powder yield (g)	Moisture content (%)
135	76-79	3.1145^{a}	8.07 ± 1.25^{a}
150	86-91	3.0974^{a}	7.56 ± 1.57^{a}
165	96-100	3.3447^{a}	7.58 ± 1.48^{a}

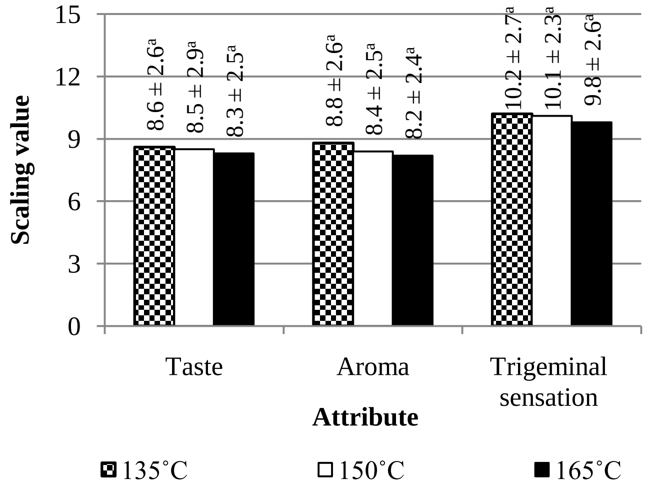
Note: Values with different superscripts indicate significant differences between treatments

According to Cai and Corke (2000) and Tee *et al.* (2012) in study of spray drying of *Amaranthus* betacyanin and *Piper betle L.* (*Sirih*) leaves extract, respectively, the heat and mass transfer between droplets and heating air is more efficient at higher temperature, leading to higher process yield. Based on the statistical analysis of yield (Appendix R), however, there was no significant difference between yield of andaliman powder spray dried at different inlet temperature ($p>0.05$).

While inlet temperature is highly related to the efficiency of the drying and the damages of flavor encapsulation, outlet temperature is related to the desired residual moisture content in the final powder product. Higher exit air temperature produce a lower relative humidity of the drying air, therefore allow more rapid drying and reduced moisture content. Cai and Corke (2000) also mentioned that increased drying rate is related to lower moisture content.

Moisture content of powder is a crucial factor in justifying the suitability of drying treatment. Moisture content is inversely correlated with spray drying inlet temperature (Pagala and Popuri, 2013). High inlet temperature holds more moisture as they are released from the droplets, resulting in a drier powder (Reineccius, 2004). According to Tee *et al.* (2012), at higher inlet drying temperature, greater temperature gradient exists between the droplets and hot drying air, causing greater rate of heat transfer to the droplet and provide driving force for moisture to evaporate. However, based on the statistical analysis of the moisture content data (Appendix R), there was no significant difference between the moisture content of andaliman powder spray dried at different inlet temperature ($p>0.05$).

The spray dried andaliman powder was then subjected to scaling test by 20 panelists (with replication). Three samples of andaliman powder sprayed with different spray drying inlet temperature were diluted in water (2%) and assessed for the aroma, taste and trigeminal sensation intensity. The score of the scaling test can be seen in Appendix S and the result of the statistical analysis can be seen in Appendix T. Figure 4.3 shows the scaling test result of andaliman powder with three different inlet temperature.



Note: Scaling value shows intensity of perceived attributes (0 = none; 15 = strong)
Values with different superscripts indicate significant differences between treatments for each of the tested attribute

Figure 4.3 Scaling value of andaliman powder spray dried with different inlet temperatures

As can be seen in Figure 4.3, there was no significant difference in taste, aroma and trigeminal sensation between andaliman powder produced with inlet temperature at 135˚C, 150˚C and 165˚C ($p>0.05$). According to Reineccius (2004), influence of drying air temperature can be less significant when less volatile flavors are being dried at higher concentration. In order to provide further understanding regarding the effect of inlet temperature and its relationship with other spray drying parameter, several trial and error attempts were done with different carrier agent content in the infeed mixture, which was then spray dried at inlet temperature 150˚C and 165˚C. It was observed from independent sensory evaluation (done by a panel of food technologists) that at inlet temperature of 165˚C, decrease in carrier agent content (extract: carrier= 1:4) caused more pronounced decrease in flavor and trigeminal sensation compared to that at inlet temperature of 150˚C.

According to Madene *et al*. (2006), flavor encapsulation by spray drying method is mainly limited by the loss of low-boiling point aromatics at higher drying temperature. Cai and Corke (2000) also reported that spray drying at inlet temperature of 165˚C caused more pigment losses (3.85%) than at inlet temperature of 150˚C (2.77%). Higher drying air temperature was also reported to decrease the bulk density of the powder, which further decrease the pigment retention during storage of the powder.

Pagala and Popuri (2013) reported that spray drying of ginger oil at higher temperature decrease the average particle size, which may result in poor storage stability of the powder. However, it was also reported that at lower inlet temperature (below 140˚C), resulting powder has poor quality with large particle and low flow ability. Study of *Pad Thai* powder spray drying by Auppathak (2010) selected inlet/ outlet temperature 150/ 90 as the most suitable spray drying temperature to produce *Pad Thai* powder with less left over in the machine and lower product loss. Based on references from previous studies, inlet temperature of 150˚C was selected as the suitable inlet temperature for andaliman powder spray drying.

4.2.2 Effect of Carrier Agent Concentration and Composition

The next part of the main research was determination of ratio of andaliman extract to carrier agent and ratio of maltodextrin (MD) to gum arabic (GA) to produce andaliman powder with desirable flavor and trigeminal sensation. Infeed mixture (100 ml) was spray dried at inlet temperature of 150˚C, with other parameters of spray drying maintained constant (flow of spray gas at 600L/ h, aspirator rate at 100% and spray pump at 30%). Outlet temperature, yield and spraying time noted from the spray drying process can be seen in Appendix U.

Table 4.3 Effect of different MD: GA towards andaliman powder moisture content at each extract to carrier ratio

Extract: carrier	Moisture content		
	MD: GA		
	3:2	1:1	2:3
1:4	6.2 ± 0.6[a]	6.1 ± 0.2[a]	9.9 ± 0.1[b]
1:6	8.1 ± 0.5[b]	6.0 ± 0.2[a]	8.0 ± 0.3[b]
1:8	6.2 ± 0.4[a]	6.0 ± 0.6[a]	6.2 ± 0.1[a]

Note: Values with different superscripts indicate significant differences between MD: GA ratio for each extract to carrier ratio

According to Cai and Corke (2000), higher feed solid content increase the powder yield, but decrease the drying rate. It was also mentioned in the study that decreased drying rate caused increase in powder moisture content. According to statistical analysis of moisture content in Appendix V, there was significant effect of interaction between extract: carrier ratio and maltodextrin: gum arabic ratio to moisture content of andaliman powder ($p<0.05$). Moisture content of andaliman powder for different carrier agent treatments were shown in Table 4.3 and 4.4.

Table 4.4 Effect of different extract: carrier towards andaliman powder moisture content at each MD to GA ratio

MD:GA	Moisture content		
	Extract: carrier		
	1:4	1:6	1:8
3:2	6.2 ± 0.6[a]	8.1 ± 0.5[a]	6.2 ± 0.4[a]
1:1	6.1 ± 0.2[a]	6.0 ± 0.2[a]	6.0 ± 0.6[a]
2:3	9.9 ± 0.1[c]	8.0 ± 0.3[b]	6.2 ± 0.1[a]

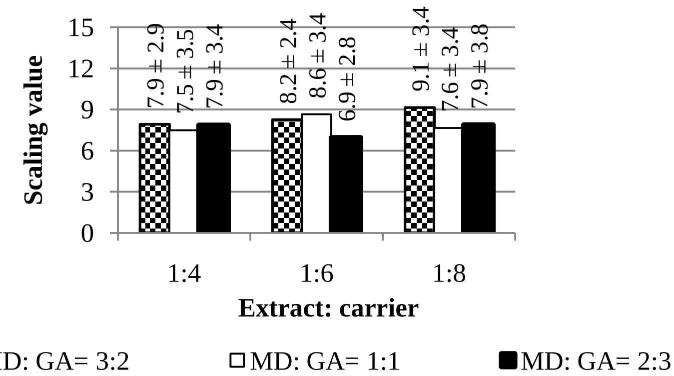
Note: Values with different superscripts indicate significant differences between extract: carrier ratio for each MD to GA ratio

It was observable in Table 4.3 that almost at every level of extract: carrier used, ratio of MD: GA= 1:1 resulted in the lowest moisture content compared to other MD: GA ratio. According to Kanpairo (2012), in the study of tuna flavor powder spray drying, moisture content decrease as the concentration of maltodextrin increase due to its hygroscopic properties. The result is in accordance with this study as it can be seen at ratio MD: GA= 2:3, where maltodextrin fraction is the smallest compared to other ratio, the moisture content of andaliman powder tend to be higher.

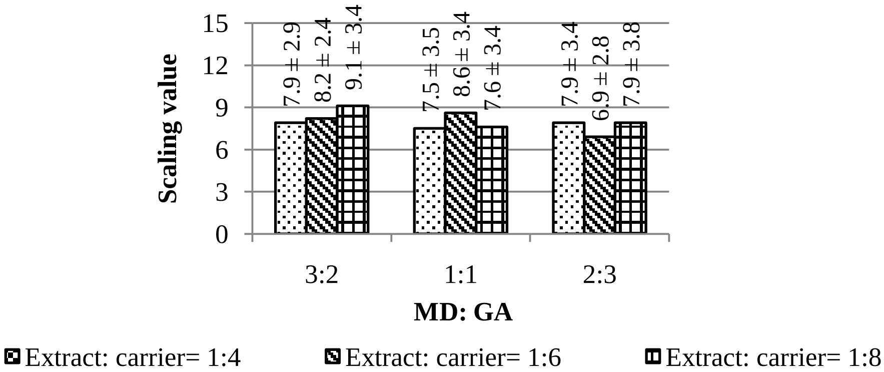
Moreover, it can be observed in Table 4.4 that the effect of extract to carrier ratio was only significant at MD: GA level of 2:3. At this level of MD: GA, the extract to carrier ratio of 1:8 resulted in the lowest powder moisture content compared to other extract: carrier ratio. It was highly possible that this was also due to higher fraction of maltodextrin in the carrier agent.

The spray dried andaliman powder was then subjected to scaling test by 20 panelists (with replication). Andaliman powder were diluted in water (2%) and assessed for the aroma, taste and trigeminal sensation intensity. The score of the scaling test can be seen in Appendix W and the result of the statistical analysis can be seen in Appendix X. Figure 4.4 and 4.5 show the scaling value of andaliman powder taste and aroma, respectively.

As can be seen in Appendix X, there was no significant effect of interaction between extract: carrier agent ratio to maltodextrin: gum arabic ratio, as well as no significant effect of extract to carrier agent ratio and maltodextrin to gum arabic ratio on taste and aroma parameter ($p>0.05$). According to study conducted by Cai and Corke (2000) on betacyanin spray drying using maltodextrin as carrier agent, higher solid content in the feed material decrease the betacyanin loss during spray drying. Moreover, according to Madene *et al.* (2006), maltodextrin as carrier agent lacks emulsifying ability and has low retention of volatile compounds. Meanwhile, gum arabic, despite being more expensive than maltodextrin, has emulsification property and good retention of volatile compounds. According to Reineccius (2004), carriers which are good emulsifiers and good film former usually produce better flavor retention. It was also reported that in encapsulation using blend of gum arabic and maltodextrin, trend of increased retention was observed with increasing fraction of gum arabic. This was, however, not observed in andaliman powder spray drying.



(a)

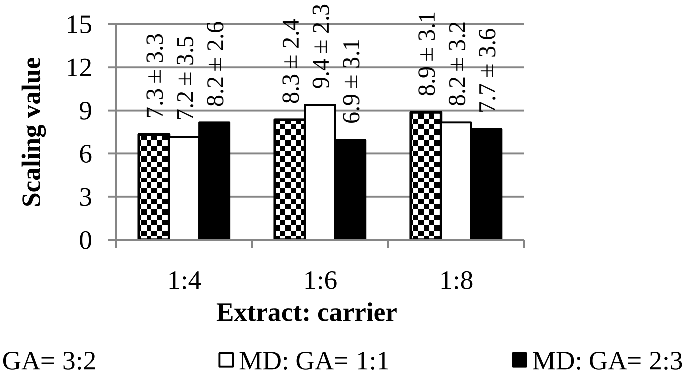


(b)

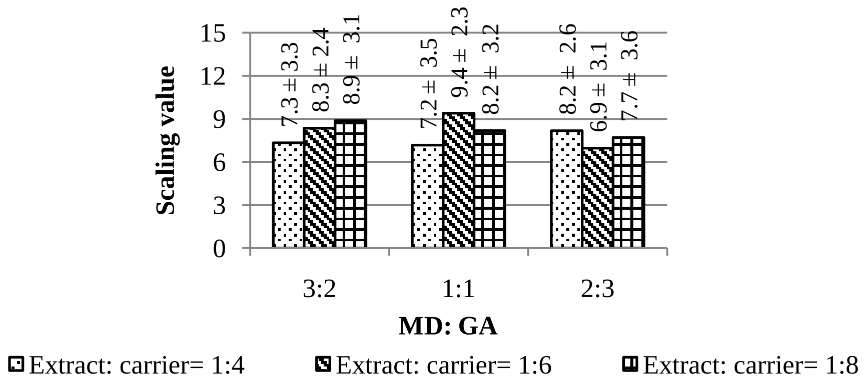
Note: Scaling value shows intensity of aroma (0 = none; 15 = strong)

Figure 4.4 (a) Effect of extract: carrier at each level of MD: GA, and (b) Effect of MD: GA at each level of extract: carrier; towards scaling value of andaliman powder aroma

Reineccius (2004) reported that beside infeed solid content, molecular weight and vapor pressure of flavor compounds also influence flavor retention during spray drying. Larger flavor molecules have slower diffusion rate and drying surface become impermeable to them more quickly during moisture evaporation. Vapor pressure also controls the rate of flavor loss before the droplet drying takes place and surface become semi permeable. This may explains why there was no significant difference in taste and aroma intensity between treatments with different composition of carrier agent. Larger and less volatile flavor compounds may not lost to as much extent compared to the small, very volatile ones. Therefore, there might be changes in andaliman flavor profile before and after spray drying process, but changes in flavor intensity between treatments of spray drying may be not as much significant.



(a)



(b)

Note: Scaling value shows intensity of taste (0 = none; 15 = strong)

Figure 4.5 (a) Effect of extract: carrier at each level of MD: GA, and (b) Effect of MD: GA at each level of extract: carrier; towards scaling value of andaliman powder taste

On the other hand, there was significant effect of interaction between extract: carrier and MD: GA towards trigeminal sensation parameter (Appendix X). Since the effect of extract: carrier and MD: GA interaction was significant only on trigeminal sensation parameter, this parameter was used to select the best treatment for andaliman powder spray drying. In order to observe the significance of each factor's effect independently, estimated marginal means of andaliman powder trigeminal sensation scaling value were shown in Table 4.5 and 4.6.

Table 4.5 Effect of different extract: carrier towards andaliman powder trigeminal sensation scaling value at each MD to GA ratio

MD:GA	Trigeminal scaling value		
	Extract: carrier		
	1:4	1:6	1:8
3:2	7.6 ± 4.0[a]	9.3 ± 2.7[a]	11.6 ± 2.0[b]
1:1	10.3 ± 2.8[a]	11.2 ± 2.2[a]	9.7 ± 3.0[a]
2:3	9.2 ± 2.4[a]	8.4 ± 3.4[a]	10.5 ± 2.5[a]

Note: Scaling value shows intensity of perceived attributes (0 = none; 15 = strong)
Values with different superscripts indicate significant differences between extract: carrier treatments for each MD to GA ratio

As can be seen in Table 4.5, there was significant effect of extract: ratio to trigeminal sensation at MD: GA level of 3:2, while at MD: GA= 1:1 and 2:3, no significant difference observed on effect of extract: carrier ratio towards the scaling value of trigeminal sensation. At MD: GA= 3:2, treatment of extract: carrier= 1:8 was observed to give the best retention of trigeminal sensation. According to Reineccius (2004), higher infeed solids accelerate the formation of semi permeable membrane on the drying particle surface during drying. Cai and Corke (2000) also reported that higher carrier agent concentration increased the betacyanin retention during storage, possibly due to the decrease of surface betacyanin in the capsule. Higher in feed solids also decrease total surface area by producing larger particle size (Bhandari *et al.*, 1992). It can be said that at constant amount of flavoring material, increasing carrier solids provide better encapsulation and protection for the encapsulated materials. Therefore, extract: carrier= 1:8 was firstly selected as the suitable spray drying treatment to maintain andaliman trigeminal sensation.

Table 4.6 Effect of different MD: GA towards andaliman powder trigeminal sensation scaling value at each extract to carrier ratio

Extract: carrier	MD: GA		
	3:2	1:1	2:3
1:4	7.6 ± 4.0[a]	10.3 ± 2.8[b]	9.2 ± 2.4[ab]
1:6	9.3 ± 2.7[a]	11.2 ± 2.2[b]	8.4 ± 3.4[a]
1:8	11.6 ± 2.0[a]	9.7 ± 3.0[a]	10.5 ± 2.5[a]

Note: Scaling value shows intensity of perceived attributes (0 = none; 15 = strong)
Values with different superscripts indicate significant differences between MD: GA treatments for each extract to carrier ratio

In Table 4.6 it can be observed that at extract: carrier= 1:8, there was no significant difference in effect of MD: GA towards trigeminal sensation. Mixture of gum arabic and maltodextrin has been reported as high solid carriers with acceptable viscosity in spray drying encapsulation of cardamom oil (Sankarikutty *et al.* 1988). Reineccius (1991) reported that in encapsulation of a mixture of ethyl propionate, ethyl butyrate, orange oil, cinnamic aldehyde and benzaldehyde using gum arabic and maltodextrin, it was observed that retention increased as the gum arabic fraction was increased. Meanwhile, in the study of flavor release from spray-dried maltodextrin/ gum arabic matrices by Yoshii *et al.* (2001), it was reported that the release of ethyl butyrate during storage decreased as the concentration of maltodextrin in the feed mixture increased.

However, according to Reineccius (2004), despite its good emulsifying and encapsulating characteristic, gum arabic has relatively high price, almost ten times to that of maltodextrin. As a result, optimum flavor retention with less gum arabic fraction in the carrier agent was more desirable. Madene *et al.* (2006) also reported that although gum arabic has good solubility, low viscosity, good retention of volatile compounds and emulsification characteristic that makes it very versatile for almost all encapsulation method, its application in food industry is limited because of its expensive price and fluctuating availability. On the other hand, maltodextrin as carrier agent has the ability to form matrices which is important in wall forming systems. Maltodextrin is also a good compromise between cost and effectiveness, regardless of its lack of emulsifying ability. In the study of aroma encapsulation by Cai and Corke (2000), blend of maltodextrin and gum arabic in the ratio of 3:2 was used and resulted in powder with good aroma retention (17%). Hence, in consideration to encapsulation quality and efficiency, ratio of extract: carrier 1:8 and ratio of maltodextrin to gum arabic 3:2 were chosen for andaliman powder spray drying.

4.3 Andaliman Powder Characterization

Characterization of andaliman powder resulting from the determined spray drying conditions was conducted through physicochemical analysis and Quantitative Descriptive Analysis method. In the QDA, andaliman powder flavor characteristics are compared to those of fresh andaliman in order to measure the flavor and trigeminal sensation of the andaliman powder after undergoing spray drying encapsulation process.

4.3.1 Physicochemical Characterization of Andaliman Powder

Knowing physicochemical properties of a food product is essential to understand its characteristics toward process conditions. These properties influence the treatment the food receive during processing as well as an indicator for other properties and food quality (Rao and Das, 2003). Parameters used in the physicochemical characterization of andaliman powder were powder yield, moisture content, water activity, powder solubility, color, bulk density and hygroscopicity (Appendix Y). The result of the physicochemical analyses of andaliman powder can be seen in Table 4.7.

In the study of *Amaranthus* betacyanin powder spray drying by Cai and Corke (2000), the betacyanin powder dried at inlet temperature of 150˚C with maltodextrin (20%) as carrier agent has moisture content of 6.80%, bulk density of 0.67 g/ml and hygroscopic moisture of 46.0 g/ 100 g dry solids. While in the study by Tee *et al.* (2012) on spray drying of *Piper betle* L. (*Sirih*) leaves extract, the powder dried at inlet temperature of 160˚C with maltodextrin as the carrier agent had moisture content of 6.99%, 10.53 g powder yield and 28.88% hygroscopicity. In the spray drying encapsulation of ginger oil studied by Pagala and Popuri (2013), inlet temperature of 150˚C using gum arabic as carrier agent, resulted in powder with moisture content of 5.90% and bulk density of 0.79 g/ml.

Table 4.7 Physicochemical properties of andaliman powder

Parameters (unit)	Result
Yield (%)	23.96 ± 1.11
Moisture content (%)	5.06 ± 0.18
Water activity (a_w)	0.122 ± 0.008
Solubility (%)	37.11 ± 7.02
Color (˚hue)	Yellow (105.16 ± 0.94)
Bulk density (g/ ml)	0.32 ± 0.01
Hygroscopicity (g moisture/ 100 g dry solids)	24.80 ± 0.40

As can be seen in Table 4.7, andaliman powder yield relative to the initial andaliman fruits used is 23.96%. In the study of tuna flavor powder spray drying by Kanpairo *et al.* (2012), the powder yield was ranging from 11.2 to 23.3% relative to the infeed solid content. From infeed solid content of three gram andaliman extract and 24 gram carrier agent, the average yield of andaliman powder is 17 gram (or 63% relative to infeed solid content), which was higher compared to the *Sirih* leaves extract powder and tuna flavor powder yield.

Moisture content of powder is a crucial factor in justifying the suitability of drying treatment. It is mainly affected by the drying temperature and usually ranged between 1-6% for spray dried powders (Reineccius, 2004). Moisture content of andaliman powder is 5.06%, lower compared to the betacyanin powder and ginger oil powder dried at the same inlet temperature, and also lower than the *Sirih* leaves extract powder dried at higher inlet temperature. According to Pagala and Popuri (2013), desirable moisture content for spray dried spice powder is around 3-5% since higher moisture content cause the powder to be susceptible to microbial growth during long term storage, while lower moisture content may cause powder dusting. The moisture content of andaliman powder is relatively low compared to other powder dried at inlet temperature as low as 150˚C however; this parameter should be optimized if the andaliman powder was desired to be stored over a long period. Lower moisture can be achieved by increasing the inlet temperature and decreasing the temperature difference of inlet and outlet temperature (ΔT) (Reineccius, 2004).

Water activity of powder is important parameter in spray dried powder as it affects the powder shelf life. Food with a_w less than 0.6 is considered as microbiologically stable (Kanpairo *et al.*, 2012). In the study of tuna flavor powder by Kanpairo *et al.* (2012), the resulting powder has water activity ranging from 0.33 to 0.48. Meanwhile in the study of aroma (consisting of ester, aldehyde, and lactone) encapsulation using maltodextrin and gum arabic, water activity of resulting powder ranged from 0.1 to 0.13 (Turchiuli, 2011). Andaliman powder which has water activity of 0.122, therefore, is considerably low in water activity and may have a stable shelf life.

The solubility of andaliman powder is 37.11%, which is much lower compared to the solubility of tuna flavor powder (60.87 to 70.12%). According to

Kanpairo *et al.* (2012), solubility is one of the most important physicochemical properties of powder, as a good solubility indicates potential application to food system by providing better appearance and smoother mouthfeel after reconstitution. Solubility is highly influenced by particle size, density and types of carrier. Gum arabic is difficult to disperse in cold water, while modified starches and starch hydrolysates are easier to disperse (Reineccius, 2004). According to Kanpairo *et al.* (2012), solubility of powder can be increased by increasing the maltodextrin concentration.

Table 4.8 Visual color interpretation of chromameter ˚Hue

˚hue	Color
342-18	Red purple
18-54	Red
54-90	Yellow red
90-126	Yellow
126-162	Yellow green
162-198	Green
198-234	Blue green
234-270	Blue
270-306	Blue purple

Source: Hutching (1999)

From the calculation of color measurement, the ˚Hue of andaliman powder is 105.16. According to ˚Hue color interpretation by Hutching (1999) in Table 4.8, andaliman powder color is yellow. Fresh andaliman is green in color when it is young and fresh, red when it is mature and black when it is dried. According to Reineccius (2004), appearance of spray dried flavoring powder in terms of color is usually influenced by its particle size. Small particles have lighter color while larger or agglomerated particles are seen as darker and richer in color. L (lightness) value ranges from 0 (black) to 100 (white). The L value of andaliman powder is 86.73, which means that the powder color is almost white. This implies that the particle size of andaliman powder was relatively small. Particle size is reported to be influenced by the temperature of drying. At higher inlet temperature, drying occurs rapidly, causing larger particles to be produced due to early formation of structure which leads to incomplete shrinking and moisture removal.

Andaliman powder bulk density is 0.32 g/ ml, lower than that of ginger oil powder (0.79 g/ ml) and betacyanin powder (0.67 g/ ml). Study of aroma encapsulation by Turchiuli (2011) also reported bulk density of spray dried aroma powder ranging from 0.49 to 0.51 g/ cm^3. According to Micha (2008), particles which are less cohesive (free flowing) tend to occupy relatively small volume, and therefore have high bulk density. On the other hand, cohesive powder tends to produce 'open structure' which resulted in lower bulk density. Powder subjected to compaction such as by tapping will increase its bulk density. According to Kanpairo *et al.* (2012), high bulk density powder usually means that the particles tend to stick or settle close to each other. On the other hand, Cai and Corke (2000) mentioned that the lower bulk density implies that there are more occluded air within the powder, causing the powder to have higher possibility of oxidative degradation during storage.

According to Tee *et al.* (2012), powder is more hygroscopic when it has low moisture content. This was, however, not true with andaliman powder as it has both moisture content and hygroscopicity value lower than those of betacyanin powder and *Sirih* leaves extract powder. According to Cai and Corke (2000), increasing the maltodextrin concentration reduces the hygroscopicity of the produced powder. This shows that andaliman powder may have good stability over prolonged storage.

4.3.2 Quantitative Descriptive Analysis (QDA) of Fresh Andaliman and Andaliman Powder

Total number of panelists that passed the QDA selection was 14 panelists (8 females and 6 males). They had to attend 8 training sessions, approximately one to two hours each session. The training sessions objective were to increase panelist's sensitivity and consistency in differing intensity of flavor attributes, as well as to develop a scorecard for the sample QDA.

Based on the result of panelists discussion in focus group, flavor of andaliman fruits was composed of sour taste and bitter taste, with aroma of citrus-like, warm citrusy and sour floral citrusy, as well as a tongue-numbing trigeminal sensation. Since the objective of the QDA was to compare the flavor of andaliman powder to fresh andaliman, panelists were asked to judge both the flavor of andaliman powder and fresh andaliman during QDA sample evaluation. For sample preparation, fresh andaliman sample were crushed using blender, while andaliman powder were diluted in water to 14.5%. The amount of sample assessed was standardized to 1 gram of crushed fresh andaliman and 0.5 ml (4 drops) of diluted andaliman powder.

Sample evaluation was conducted independently in standard sensory booth with proper lightning and environment. The sample evaluation was done in four replications by each panelist. The result of the fresh andaliman and andaliman powder QDA evaluation can be seen in Appendix Z and the statistical analysis of the QDA result can be seen in Appendix AA. It was observed that andaliman powder was significantly higher in citrus like aroma, sour floral citrusy aroma, sour taste, bitter taste and trigeminal sensation compared to fresh andaliman. Figure 4.6 shown the result of QDA visualized in a spider web.

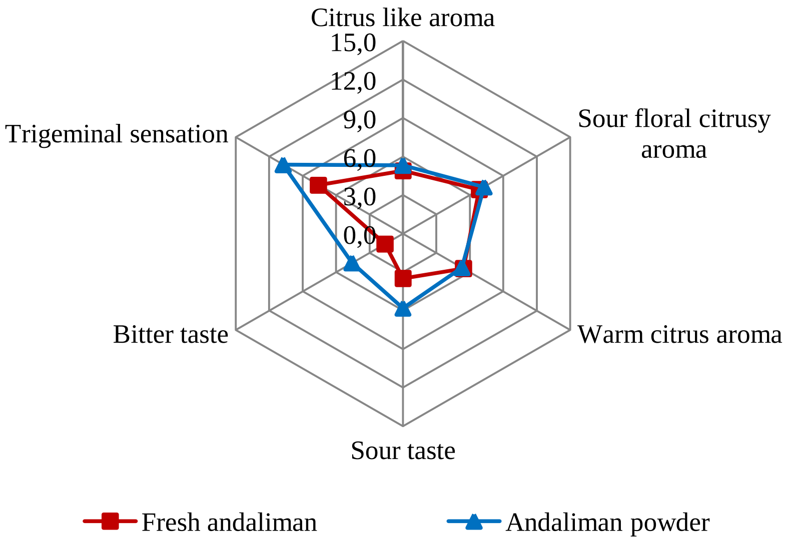


Figure 4.6 Spider web of andaliman powder and fresh andaliman flavor

From Figure 4.6, it can be seen that andaliman powder has similar flavor characteristics with fresh andaliman, but with higher perceived intensity. According to Wijaya *et al.* (2001), two major components andaliman flavor extract are geranyl acetate and limonene, which give the sour floral citrusy and sweet citrusy aroma, respectively. While from the AEDA result, citronellal was reported as the component with the highest aroma intensity, with its characteristic of strong and warm citrusy aroma. In terms of aroma, the significant difference were likely to be desirable, since the sour floral citrusy aroma and citrus like aroma in powder andaliman were significantly higher compared to andaliman powder. This may be resulted from the concentration process that happens in conjunction with the andaliman extraction. The amount of aroma compounds was likely to be higher in the andaliman extract than in the initial fresh andaliman used. This also showed the effectiveness of the spray drying process which has successfully encapsulated the volatile aroma compound inside the powder particles and maintained the flavor characteristics.

In terms of taste, spray dried andaliman powder showed more pronounced bitter and sour taste compared to fresh andaliman. The taste of fresh andaliman itself were not distinctive, just slightly sour and a very slight bitter taste. Sour taste is related to the citrusy aroma characteristic of andaliman, therefore the intensification of sour taste perception may be related with the increase of the aroma intensity in andaliman powder.

On the other hand, it was not certain if the bitter taste intensification was solely caused by andaliman extraction process or due to other factors, since the increase of bitter taste in andaliman powder was more prominent compared to the increase of other flavor attributes. According to Yang (2008) and Galopin *et al.* (2004), the tingling (trigeminal active) sanshool compound isolated from various Zanthoxylum species was an alkylamide, which tends to polymerize and decomposes easily under hydrolytic conditions. Therefore, there were possibilities that the bitter taste intensification were also caused by polymerization of the trigeminal active compound due to heat and other hydrolytic conditions during andaliman powder processing. This, however, requires further study on the characteristic of the trigeminal active compound in andaliman.

It was also observed from the spider web that the andaliman powder has higher intensity of trigeminal sensation compared to fresh andaliman. This again showed the effectiveness of the implemented spray drying conditions in producing andaliman powder with flavor characteristics comparable to those of fresh andaliman.

CHAPTER V

CONCLUSION AND SUGGESTIONS

5.1 Conclusion

Andaliman extract with similar flavor and trigeminal characteristics to fresh andaliman was obtained by maceration method using ethyl acetate: ethanol (1:1) as solvent. The extraction yield was 4.22% relative to the initial andaliman fruits used.

Andaliman powder with similar flavor attributes to fresh andaliman could be obtained by spray drying the selected extract with maltodextrin: gum arabic combination (3:2) in ratio of 1 to 8, using 150˚C as the inlet temperature. QDA result shown that sour and bitter taste, trigeminal sensation, citrus like and sour floral citrusy aroma attributes were more pronounced in powder andaliman compared to fresh andaliman. The obtained andaliman powder had 23.96% powder yield, moisture content of 5.06%, water activity of 0.122, powder solubility of 37.11%, yellow color, bulk density of 0.32 g/ml, and hygroscopicity of 24.80 g moisture/ 100 g dry solids.

5.2 Suggestions

The potency of andaliman as natural flavoring should be further studied since the uniqueness of andaliman flavor has yet to be known by many people. Further research regarding the andaliman powder spray drying should be developed since there were still many attributes to be improved such as the powder physicochemical characteristics, as well as the bitter taste intensification and related consumer acceptance. Process optimization such as using Response Surface Methodology (RSM) is recommended in order to optimize the spray drying condition and therefore obtain andaliman powder that has better flavor characteristics and high powder quality. More comprehensive flavor analysis, such as by GC-MS or GC-O method, as well as utilization of trained and experienced panelists along the study, are also suggested in order to gain objective and subjective sensory profile of andaliman powder.

Solutions for technical problems, such as the time-consuming manual cleaning of andaliman fruits as well as andaliman frozen storage, should be considered if the research were to be applied on larger scale. Furthermore, research on andaliman powder application in cooking may be conducted to study the flavor changes of andaliman powder during cooking process.

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APPENDICES