Comparison of Several Processing Methods in Preserving the Flavor Properties of *Andaliman* (*Zanthoxylum acanthopodium* DC.) Fruit

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Highlights:

- Oven drying at 54 °C for 8 h was found to be the most suitable method to preserve *andaliman* fruit without significant changes in its flavor quality.
- Spray drying at an inlet temperature of 150 °C was able to preserve the flavor quality of andaliman fruit extract.
- A 1:8 ratio of extract to carrier agent with a maltodextrin to gum arabic ratio of 3:2 produced encapsulated *andaliman* powder that resembled the flavor of fresh *andaliman* fruit.

Abstract. *Andaliman* (*Zanthoxylum acanthopodium* DC.) is a plant endemic to North Sumatra. Its fruit has a very specific citrus-like aroma, gives a tingling sensation and is commonly used for seasoning in Bataknese traditional cuisine. To extend the shelf life and preserve the quality of *andaliman* fruit, post-harvest handling is needed. Seven drying methods were applied and compared, i.e. sun, air, fluidized bed, oven, far infrared, freeze, and spray drying. Considering the physicochemical and sensory properties and efficiency, oven drying was selected for further study. The optimum condition for drying was temperature at 54 °C for 8 h resulting in 0.67 desirability level based on the response surface method (RSM). Spray drying encapsulation of *andaliman* fruit extract was performed at an inlet temperature of 150 °C with an extract to carrier ratio of 1:8. Maltodextrin (MD) and gum arabic (GA) with a ratio of 3:2 was chosen as the carrier agent. The quantitative descriptive analysis (QDA) result showed that the encapsulated *andaliman* powder had a flavor that resembled fresh *andaliman* with a more pronounced taste, a citrus-like trigeminal sensation, and a sour floral citrusy aroma.

Keywords: *andaliman; drying; flavor; RSM; Zanthoxylum acanthopodium DC.*

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1 Introduction

Andaliman (*Z. acanthopodium* DC.) is a spice that is endemic to North Sumatra and has a typical warm-citrusy aroma and causes a tingling-numbing sensation. The citrusy aroma of *andaliman* is caused by citronellal and limonene while the trigeminal sensation is due to the presence of 2E, 6Z, 8E, 10E-N-(2'methylpropyl)-dodecatetraenamide, also known as sanshool. A similar compound is also found in *Z. piperitum* (Japanese pepper) and *Z. bungeanum* (Szechuan pepper) [1].

The typical trigeminal sensation caused by *andaliman* is attractive for the flavor industry and brings economic value due to the saturation in the flavor market driving the development of exotic and unique flavors. Moreover, scientific studies have proved that *andaliman* possesses health benefits, which potentially fulfill consumer interest in natural food ingredients with health benefits [1]. However, *andaliman* is a very perishable commodity. Its high moisture content induces fungal decay during storage at room temperature within a few days; its color changes to black and it is covered by white-fungal hyphae within two weeks [1]. Therefore, proper post-harvest handling is needed. Drying by reducing the moisture content is a common practice to preserve spices and herbs. One of the critical factors in the drying process is temperature. Herbs and spices usually contain flavor compounds that are vulnerable to high temperatures. For this reason, several drying methods toward *andaliman* fruit were compared to determine the method that produces preserved *andaliman* with optimum flavor quality.

Nowadays, flavor ingredients in the form of extracts need to be developed in order to enhance wide application in the food system. Flavor encapsulation by spray drying is widely used to produce food ingredients and to protect the flavor from damage, which is usually caused by light, heat, moisture or air exposure and also to provide a more controlled release of the flavor [2]. More than 90% of encapsulated flavorings are produced using this method [3], particularly related to spice oleoresins, which are usually very reactive and unstable toward light, temperature and oxygen [4]. The present research aimed to obtain a suitable method to produce encapsulated *andaliman* powder that preserves the fresh flavor quality of *andaliman*.

2 Methods

2.1 *Andaliman* Fruits Drying

Fresh *andaliman* (FA) fruits were obtained from North Sumatra. Six processing methods were applied to the samples. Far infrared drying (FIR) was done at 60

°C for 7 h [5], oven drying (OD) was done at 60 °C for 5 h [6], fluidized bed drying (FBD) was done at 40 °C [7] for 6 h and 50 min, freeze-drying (FD) was done at -52 °C and 1.8 Pa for 69 h.

Sun-drying (SD) was done in a green-house facility for 28 h. Air drying (AD) was conducted under shade for ±142 h. After drying the physicochemical properties (moisture content, water activity, color (Hunter method), bulk density, and hygroscopicity) and sensory quality were evaluated.

2.1.1 Multiple Comparison Test

A multiple comparison test was conducted to estimate the difference between the dried *andaliman* samples from the six drying methods compared to a fresh *andaliman* sample. Native users of *andaliman* who worked as cooks in North Sumatra were purposively sampled as the panelists. The aim was to select panelists who were familiar and had good sensitivity toward *andaliman* flavor quality. The multiple comparison test was done using an intensity rating test for aroma and trigeminal sensation attributes assessed by 15 panelists.

The measurement scale used in testing was a 15-cm line scale. FA was used as the standard and determined with a value of 7.5. The results of the assessment were further analyzed by ANOVA (analysis of variance) and Duncan's multiple comparison test with the SPSS 20 software.

2.1.2 Drying Condition Optimization

Optimization of the selected method was performed with temperature (T) and time (t) as the main factors. The treatment levels were based on the conditions used in an initial research (T_b < 50 °C < T_a; t_b < 5 h < t_a). Before optimization, the upper (a) and lower (b) limits of the two factors were determined to decide whether the response obtained was significantly different from the process factors.

The optimization was carried out using RSM with the Design Expert 7 (DX-7) software (Stat-Ease, Minneapolis, USA), measuring the following parameters: moisture content, water activity, aroma, and trigeminal sensation intensity. Sensory evaluation was conducted using the balanced incomplete block (BIB) method [8] to avoid bias caused by satiation of the panelists.

The effect and regression coefficients of individual linear, quadratic, and interaction terms were determined. Model determination for the response was measured using the D-optimal design. Verification was done after obtaining the optimum drying method.

2.2 Encapsulation of *Andaliman* Fruit Extract by Spray Drying

2.2.1 Extraction

One hundred grams of FA were crushed in a dry blender for 10 s and mixed with 220 ml of EtOAc and EtOH (1:1). Maceration was done at room temperature for 48 h, after which the mixture was vacuum filtered using Whatman filter paper no. 1. The solvent from the filtrate was removed using a vacuum rotary evaporator at 55 °C.

2.2.2 Spray Drying Condition

The spray drying (PD) inlet temperature, the ratio of *andaliman* fruit extract to carrier agent and the ratio of maltodextrin (MD) to gum arabic (GA) were determined as the factors of the PD process. The infeed mixture was prepared by hydration of the carrier agent in water. The extract was added and mechanically stirred using a Heidolph stirrer for 10 min at speed 3. The first step was done to determine the PD inlet temperature.

The mixture of extract and carrier agent in water (with extract to carrier to water ratio at 1:6:100 (w/w/v)) were prepared and spray dried using three different inlet temperatures (135 °C, 150 °C and 165 °C). MD and GA at a ratio of 3:2 was used as carrier agent. The selected inlet temperature was used to determine the *andaliman* extract to carrier agent ratio and the MD to GA ratio that could obtain *andaliman* powder with the highest flavor and trigeminal sensation retention.

Three ratios of *andaliman* extract to carrier agent (1:4, 1:6, and 1:8) and three ratios of MD to GA (3:2, 1:1, 2:3) were applied. All formulas were prepared using 100 ml water (w/v). All encapsulated *andaliman* powder samples were subjected to sensory analysis using a rating rest with a line scale assessed by 20 panelists. Physicochemical characteristics were evaluated, including yield, moisture content, water activity, color (Hunter method), bulk density, hygroscopicity, and powder solubility.

2.2.3 Quantitative descriptive analysis (QDA)

QDA [9] was performed to analyze the flavor characteristics of *andaliman* powder compared to FA by the panelists that passed the selection and participated in the training procedure (Table 1), followed by quantitative analysis.

Intensity determination of sensory perception was done by the 14 trained panelists using unstructured line scales (15.29 cm) anchored 12.7 mm from each end by a pair of terms that explain the attribute being tested. The QDA results were analyzed using t-test and visualized with a spider web.

Table 1 Training procedures for quantitative descriptive analysis.

Session	Training procedure
1	Introduction to QDA training, scaling and ranking test, sniffing and tasting technique
2	Introduction to *andaliman* sample and discussion of sample flavor attributes
3	Introduction of sample 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 discussion for qualitative flavor analysis of *andaliman*
8	Ranking and scaling test for taste and aroma attributes obtained from qualitative test

3 Result and Discussion

3.1 Physicochemical Characteristic

Table 2 shows the physicochemical properties of the dried *andaliman* samples from the various methods. Compared to the acceptable maximum moisture content of white pepper in SNI 0004:2013 of 13% (b/b) [10], the dried *andaliman* samples produced with FBD and AD were not acceptable. In line with that, the water activity (Aw) of the dried *andaliman* from both methods also did not qualify. The recommended Aw for dried spices such as black pepper, onion powder, cinnamon, white salt and red pepper is 0.351-0.587 [11].

The color, bulk density, hygroscopicity and solubility of the powder are not mandatory properties for dried spices, but they do determine consumer acceptance. Based on Hutching [12], the visual color interpretation of the chromameter °hue of the dried *andaliman* fruit was green (162-198), while that of the spray-dried *andaliman* was yellow (90-126).

The L value of the *andaliman* powder indicates that it was almost white. Despite all dried *andaliman* samples being within the same °hue range, they had different L values. The FD samples had the brightest color among the samples, while the FBD and AD samples had the darkest color, which did not represent FA.

The moisture content and Aw of the samples affected the hygroscopicity of the dried *andaliman*. The samples from the AD and FBD methods had a hygroscopicity of -1.42 and -3.58 g moisture/100 g dry solid, respectively, indicating that the samples tended to release moisture.

Table 2 Physicochemical properties of *andaliman* from several processing methods.

Parameters	Processing Methods						
	FIR	**OD**	**FD**	**FBD**	**AD**	**SD**	**PD**
Yield (%)	25.33 ± 0.55^{b}	24.04 ± 0.45^{ab}	26.72 ± 0.23^{c}	31.39 ± 0.21^{e}	28.40 ± 0.14^{d}	26.76 ± 0.53^{c}	23.96 ± 1.10^{a}
Moisture content (%)	7.95 ± 0.07^{c}	5.00 ± 0.00^{b}	3.50 ± 0.00^{a}	15.00 ± 0.00^{e}	13.00 ± 0.00^{d}	8.05 ± 0.07^{c}	5.06 ± 0.11^{b}
Water activity	0.567 ± 0.030^{c}	0.448 ± 0.021^{b}	0.447 ± 0.010^{b}	0.677 ± 0.028^{d}	0.759 ± 0.001^{e}	0.560 ± 0.018^{c}	0.123 ± 0.010^{a}
Color (°hue)	180.04 ± 2.17^{bc}	178.52 ± 0.05^{b}	179.32 ± 0.06^{bc}	181.39 ± 0.01^{c}	181.31 ± 0.02^{c}	181.54 ± 0.02^{c}	105.17 ± 1.14^{a}
Color (L)	38.74 ± 0.06^{c}	36.52 ± 0.42^{b}	43.45 ± 0.11^{d}	36.14 ± 1.35^{b}	30.92 ± 0.11^{a}	36.30 ± 0.13^{b}	86.73 ± 0.28^{e}
Bulk density (g/ml)	0.38 ± 0.00^{c}	0.42 ± 0.00^{d}	0.32 ± 0.00^{b}	0.27 ± 0.03^{a}	0.32 ± 0.01^{b}	0.44 ± 0.01^{d}	0.32 ± 0.01^{b}
Hygroscopicity (g moisture/ 100 g dry solids)	2.98 ± 0.01^{c}	7.14 ± 0.03^{d}	8.83 ± 0.09^{e}	-0.90 ± 1.01^{b}	-3.85 ± 0.10^{a}	2.94 ± 0.05^{c}	24.80 ± 0.35^{f}
Solubility (%)	-	-	-	-	-	-	37.11 ± 7.02

Note: FIR: far infrared drying; OD: oven drying; FD: freeze drying; FDB: fluidized bed drying; AD: air drying; SD: sun drying; PD: spray drying. Mean ± SD of two replicates (n = 2) for each attribute with different letters are significantly different (P < 0.05) using Duncan's multiple comparison test.

3.2 Sensory Quality of Dried *Andaliman* Fruit

Figure 1 shows that the samples produced by the FD method had the highest trigeminal sensation intensity, followed by the samples produced by the OD and FIR method. FIR had the score of aroma intensity closest to FA, followed by OD and FD. The AD and FBD samples exhibited the lowest aroma intensity and AD had the lowest trigeminal sensation intensity. Sriwichai, *et al.* [13] found that the essential oil from AD *Z. myriacanthum* had a higher preference score than fresh *Z. myriacanthum*. The essential oil from the OD sample also had a higher preference score, yet odor active values (OAV) for the major aroma compounds, i.e. α-terpene, L-linalool, decanal and dodecanol, were not detected. The undesired flavor of AD may have been caused by the high relative humidity in the area where the *andaliman* was dried.

To choose the method to further optimize, all values of the physicochemical and sensory properties were rated. Overall, FD was the best drying method, but OD was not significantly different in retaining the flavor intensity of *andaliman* and is considered more economically affordable. OD was therefore chosen for further optimization.

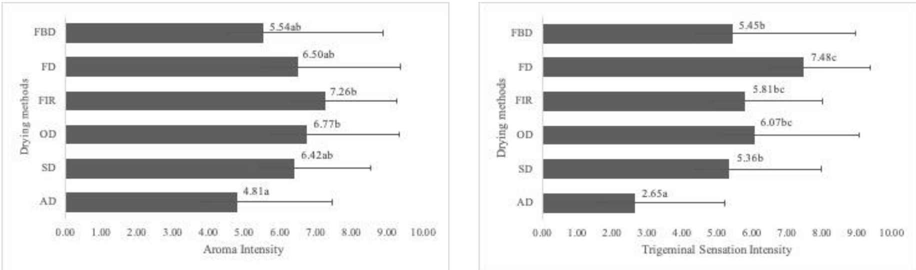

Figure 1 Comparison of aroma and tingling sensation intensity between dried *andaliman* samples from different drying methods.

* The mean scores (n = 15) for each attribute with different letters were significantly different ($P < 0.05$) using Duncan's multiple comparison test.
** FIR: far infrared drying; OD: oven drying; FD: freeze-drying; FDB: fluidized bed drying; AD: air drying; SD: sun-drying.

3.3 Optimization of *Andaliman* Fruit Drying

The upper and lower limits for drying temperature and time were determined, i.e. 80 °C > T > 45 °C and 5h < t < 8 h. Sixteen drying conditions were suggested by DX-7. The responses were recorded and processed, after which DX-7 generated cubic, quadratic, mean, and linear models for moisture content, Aw, aroma and trigeminal sensation, respectively.

All the models were significant, except for the aroma attribute model (Table 3). The equations that could describe the effect of drying temperature and time on (1) moisture content, (2) Aw, (3) aroma, and (4) trigeminal sensation can be seen below.

$$\begin{aligned} MC = {} & 503.19523 - 19.52602T - 0.43624t + 0.012746Tt + \\ & 0.25150T2 - (9.20886 \times 10\text{-}5)\ T2t - (1.06478 \times 10\text{-}3)\ T3 \end{aligned} \quad (1)$$

$$\begin{aligned} Aw = {} & 9.04218 - 0.34124T - (2.27252 \times 10\text{-}3)\ t + \\ & (2.90834 \times 10\text{-}5)\ Tt + (4.61301 \times 10\text{-}3)\ T2 - \\ & (2.15775 \times 10\text{-}5)\ T3 \end{aligned} \quad (2)$$

$$A = 6.59375 \quad (3)$$

$$TS = 8.49940 - 0.065144T + (3.50996 \times 103)t \quad (4)$$

where MC is moisture content, Aw is water activity, A is aroma intensity, TS is trigeminal sensation intensity, T is temperature (°C), t is time (min). The response plots show that the application of higher temperature and longer time lowered the moisture content and the Aw score, as indicated by the blue area (Figure 2).

The drying process usually reduces the flavor intensity of spices because of the increase of temperature and time. However, in this study, the different drying conditions did not significantly affect the aroma. Thirty types of volatile compounds were detected in the extract from FD *andaliman* fruit, including geranyl acetate as the major compound, and citronellal and limonene, which are the aroma impact compounds accounting for the fresh citrus aroma [14]. This may occur due to the intensification of the aroma impact compounds of *andaliman* or the development of new compounds during the process, resulting in a stronger aroma.

Aromatic profiling of the *Z. myriacanthum* essential oils from dried fruit showed the changes in chemical composition and OAV from the different drying methods compared to fresh fruit [13]. Further study is needed to prove the impact of the drying process on the aroma compounds.

Table 3 Analysis variance of mean, linear, quadratic, and interaction terms and coefficients for the prediction model of oven-dried *andaliman* fruit.

	Moisture content (%)	**Aw**	**Aroma**	**Trigeminal sensation**
Model	0.0015 (significant)	< 0.0001 (significant)	-	0.0169 (significant)
Lack of fit	0.9705 (not significant)	0.8610 (not significant)	0.0121 (significant)	0.2696 (not significant)
Adj R-squared	0.7833	0.9291	-	0.3838
Predicted R-Squared	0.6217	0.8633	-1.1378	0.2163
Adequate Precision	8.734	15.917	-	6.187

Note: $P < 0.05$ is significant at $\alpha = 0.05$; lack of fit is not significant at $P > 0.05$.

Andaliman's trigeminal sensation compound, known as sanshool [15], was unstable at high temperatures and degraded by 30% in vegetable oil at 160 °C for one hour [16]. In line with this study, the trigeminal sensation intensity of the dried *andaliman* fruit decreased along with the increase of temperature. Therefore, it was necessary to determine the optimum drying temperature and time to retain the trigeminal sensation. To achieve this, the optimization goal of the drying process was determined (Table 4).

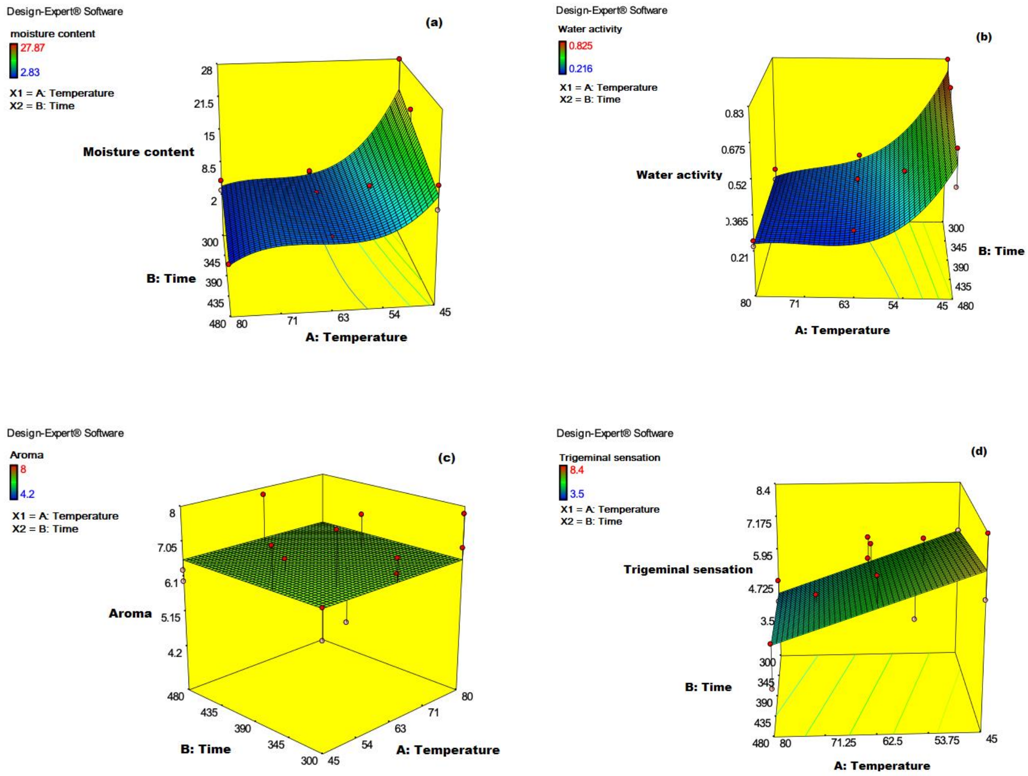

Figure 2 Response surface plot of (a) moisture content, (b) water activity, (c) aroma, and (d) trigeminal sensation as a function of time and temperature.

Table 4 Criteria for numerical optimization of oven drying process conditions and predictive solution generated by DX-7.

Drying criteria	Unit	Optimization goal	Lower limit	Upper limit	Predictive Solution	Verification		
						95% CI low	95% CI high	Actual
Temperature	°C	In range	45	80	54			
Time	min	In range	300	480	480			
MC	%	In range	6.00	12.00	6.50	0.00	13.69	8.23 ± 0.25
aW	-	minimize	0.216	0.825	0.327	0.23	0.420	0.383 ± 0.01
A	-	maximize	4.2	8.0	6.59	5.98	7.21	10.21 ± 2.78
TS	-	maximize	3.5	8.4	6.65	5.59	7.70	12.45± 1.31

Note: The actual values were mean ± SD of three replicates (n = 3) for moisture content (MC) and Aw, fifteen replicates (n = 15) for aroma (A) and trigeminal sensation (TS).

Drying at 54 °C for 480 min was selected, with level of desirability at 0.67 out of 1. The higher desirability indicates the conformity of the drying conditions to produce optimum aroma and trigeminal sensation. The actual responses in the verification were higher compared to the predicted scores (Table 4). Thus, the suggested drying conditions were acceptable and can potentially be used to substitute fresh *andaliman* with dried ones as food additives on a daily-basis as well as raw or intermediate materials to develop functional flavor ingredients.

3.4 Encapsulation of *Andaliman* Extract by Spray Drying

To determine the inlet temperature, *andaliman* extract and carrier agent at a ratio of 1:6 were used. The carrier agent consisted of MD and GA at a 3:2 ratio. The infeed mixture (65 ml) was spray dried at inlet temperatures of 135, 150, and 165 °C. The other parameters of PD were kept constant, i.e. outlet temperature at 150 °C, spray gas flow at 600 L/h, aspirator rate at 100% and spray pump at 30%. Figure 3 reveals that there was no significant difference between the different inlet temperatures for all measured parameters. Several trials using varying carrier agent content in the infeed mixture were done and showed that at 165 °C a decrease in carrier agent content (extract to carrier ratio at 1:4) caused a more pronounced decrease in flavor and trigeminal sensation compared to inlet temperature at 150 °C. This temperature was selected as the inlet temperature for spray drying.

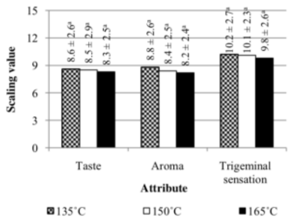

Figure 3 Scaling value of spray-dried *andaliman* powder at different inlet temperatures (0 = none; 15 = strong). (Note: Values with different superscripts indicate significant differences between treatments for each of the tested attributes).

Figure 4 shows that there was no significant difference in aroma and taste value due to the ratio of extract and carrier agent as well as the ratio of the carrier agent itself. However, there was a significant difference between the samples towards the trigeminal sensation. Table 5 shows that the extract to carrier level at an MD to GA ratio of 3:2 was the highest and the only significantly different between the treatments. Higher infeed solids could accelerate the formation of semi-permeable membrane on the drying particle surface [17] and a higher carrier agent concentration was able to increase the compound retention of the semi-permeable membrane on the drying particle surface [18]. A constant amount of flavoring material and increase of carrier solids provided better encapsulation and protection of the core material. Thus, an extract to carrier ratio of 1:8 was selected to retain the trigeminal sensation.

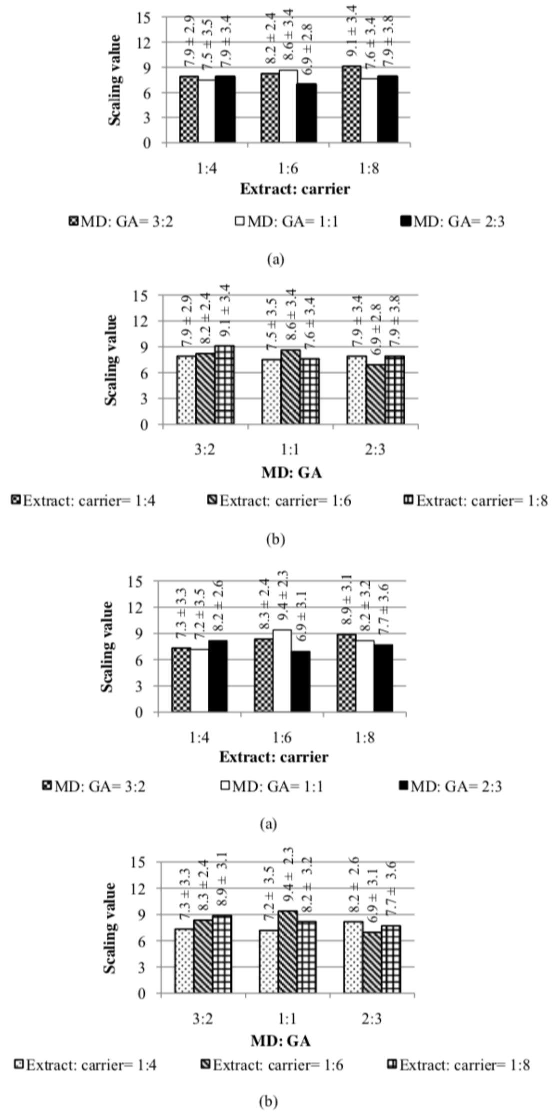

Figure 4 Left: (a) Effect of the extract to carrier ratio at each MD to GA ratio; (b) effect of the MD to GA ratio at each extract to carrier ratio towards the scaling value of *andaliman* powder aroma. Right: (a) Effect of the extract to carrier ratio at each MD to GA ratio; and (b) effect of the MD to GA ratio at each extract to carrier ratio towards the scaling value of *andaliman* powder taste.

Meanwhile, Table 6 shows that extract to carrier ratio at 1:8 had no significant influence on the effect of the MD to GA ratio towards the trigeminal sensation. Mixture of the carrier agents is commonly applied in PD encapsulation. Yoshii, *et al.* [19] reported that the flavor release of ethyl butyrate during storage decreased as the concentration of MD increased. GA has good emulsifying and encapsulating characteristics, but it is relatively more expensive than MD [17]. MD is a good compromise between cost and effectiveness, regardless of its lack of emulsifying ability. Cai & Corke [18] have shown that a mixture with a MA to GA ratio of 3:2 is able to produce good aroma retention (17%). Therefore, an extract to carrier ratio of 1:8 with a MD to GA ratio of 3:2 were chosen for *andaliman* powder PD.

Table 5 Effect of different MD to GA ratios for each extract to carrier ratio and different extract to carrier ratios toward the trigeminal sensation scaling value.

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

Note: The scaling value (mean ± SD) shows the intensity of the perceived attributes (0 = none; 15 = strong) from 20 panelists. Values with different superscripts indicate significant differences between treatments.

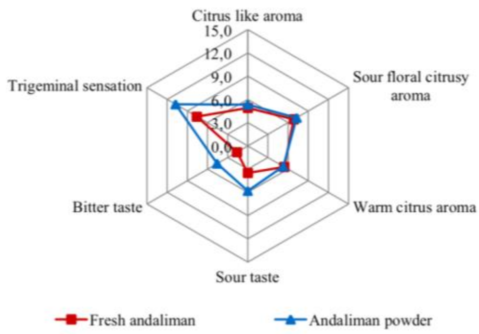

Figure 5 Visualized QDA of *andaliman* powder and fresh andaliman flavor profile and intensity from 14 panelists and four (4) replications for each panelist.

The characterization of the *andaliman* powder was compared to FA through QDA. Figure 5 shows that the encapsulation of the extract in the selected conditions produced *andaliman* powder with similar flavor characteristics but with higher perceived intensity compared to fresh *andaliman*. The encapsulation process was effective in preserving the aroma compounds inside the powder particles. *Andaliman* powder had a more pronounced bitter and sour taste compared to FA. The sour taste could be a perception of the citrusy aroma of *andaliman* and its intensification may be related to the increase of citrusy aroma intensity. The bitter taste intensification may be caused by the polymerization of the trigeminal-active compounds. Yang [20] reported that sanshool tends to polymerize and decompose easily under hydrolytic conditions. Further study is needed to prove the changes in the non-volatile compounds of *andaliman* during processing.

4 Conclusion

Oven drying at 54 °C for 8 h is suggested as the most appropriate processing method to produce dried *andaliman* fruit with optimum flavor quality. Spray drying at an inlet temperature of 150 °C, using an extract to carrier ratio of 1:8 with a MD to GA ratio of 3:2 was selected to produce encapsulated *andaliman* powder. QDA showed that the *andaliman* powder had a similar flavor to FA with more pronounced intensity. Quantitative measurement of sanshool and aroma compounds from every drying method and the correlation to the sensory attributes is suggested for further study to give more insight.

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