

Optimization of Salt Addition in the Making of Sunti Acid Process Using *Responde Surface Methodology* (RSM)

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Abstract— Syringe acid is a typical Acehnese seasoning made of starfruit. Syringe acid is used in a variety of Acehnese specialties such as tamarind (tamarind), time cranks (stir-fried fish), pay ungkoet (Pepes), and comma (stir-fried wooden fish). Commercial injectable acids vary in color, texture and contain calcium oxalate. The color of commercial injectable acid tends to be dark with a soft texture and calcium oxalate which has an effect on health. In this study, optimization of salt concentration and salting time was used to produce color, texture and reduce calcium oxalate.

Keywords— Acid Sunti, Respons Survace Methodology, Salt, Optimations.

I. INTRODUCTION

Belimbing wuluh (Averrhoa bilimbi) is one of the plants that grow in the whole region of Indonesia. Aceh is one area that has good growth potential and almost every house has star fruit. Traditional starfruit is used traditionally for itching, swelling, skin eruption and coughing. In India, the fruit is used for obesity. In India, the fruit is used for obesity (1). *Averrhoa bilimbi* extract showed antifungal activity against A. Niger and C. *albicans* (2), antimicrobial (3). *Averrhoa bilimbi* contains high levels of oxalate (8,57-10,32g/100g) and ascorbic acid (60.95 mg/100g) (4).

Syringe acid is a typical Acehnese seasoning made from a *Averrhoa bilimbi* with a process of grinding and salting. The characteristic features of sunti acid are reddish brown color, flat oval shape, soft texture and dry appearance (5). Utilization of injectable acid as a cooking ingredient has been used by all regions in Aceh. Syringe acids have the characteristics of being sour, salty, and umami (6). Syringe acid can contribute to the taste of umami foods which are added to the Sour syringe acid (6). Conventional syringes are processed naturally under sunlight.

Conventional injectable acids produced by the community today vary in terms of color and texture. It is suspected that color and texture variations in acid injection salting and drying factors. Observations in the field of salt producers and salting time in the process of making Sodium acid vary. This causes researchers to improve the salting process to get optimal results. Injectable acid with gel color can cause consumer disinterest in the product. Composition of the salt determines the characteristics of the produced syringe acid (6). Salt in ingredients that can accelerate *chlorophyll* forms *pheophytin*. The method of drying using a cabinet dryer produces higher levels compared to natural syringe acid using sunlight. Surface response methodology is a technique to get optimal results under the conditions given to analyze problems in the form of response variables influenced by independent variables and get optimal responses. Optimization of salt concentration and salting time in the process of making Sodium Acid has not been done. The overall objective of this study was to optimize salt concentration and salting time to produce color, texture, and decrease in calcium oxalate.

II. METHOD

A. Place, Tools and Materials

The research was carried out in the Biochemistry laboratory, Laboratory of Agricultural Product Processing of Agricultural Product Technology Department. The process of making syringe in the Unsyiah Agricultural Technology Laboratory.

The raw materials used in this study were *Averrhoa bilimbi* (obtained from the Pidie area), local salt (obtained from the Beureunun market). Chemicals with purity pro-analysis (p.a) obtained from CV. Krida Tama Persada Malang.

The tools used for the manufacture of injectable acids are Memmert OVG 6 type cabinet dryers, TXT 32 brand texture analyzers, Konica Minolta brand color readers, Hanna Instrument brand pH meters, Shimadzu LC 20A brand HPLC, and Scanning Electron Microscopy (SEM) brands JSM T-100, JEOL, Japan.

B. Characterization of Commercial Sunti Acid

The physicochemical characterization of commercial injectable acid was obtained from traditional markets in the Pidie, Lhokseumawe and Aceh Besar regions. The sensory evaluation uses the Just About Right scale (JAR) follows the Maximo *et al.*, (8) and the best product determination follows the method Massey *et al.*,(9).

C. Sensory Evaluation

Commercial injection acids have a variety of colors and textures that are complemented by the use of salt. The use of the JAR scale to determine the optimal attributes of the product. 50 panelists are used for product evaluation. Samples, labeled with three-digit randomization. Data were analyzed by ANOVA with samples and panelists as groups in the Tukey Test model (P < 0.05) used for each product for higher or higher colors and textures, faster and whiter. Determination of



color intensity and texture level using the Kruskal-Willis test (p < 0.05).

D. Making Sunti Acid

Starfruit is sorted and separated by size. Then weighed as much as 2000 g and boiled at 600 C for 5 minutes for the heating process. Starfruit was dried using a cabinet dryer at 350 C for 8 hours. Furthermore, starfruit was given salt with salt concentration and salting time according to the design of the experimental design. Re-drying using a 350 C temperature dryer for 8 hours and the second salt with a concentration of half of the initial level. Re-drying using a 35° C temperature dryer for 8 hours and stored at room temperature.

E. Analysis Method

The salt and acid levels follow the Khodman method (11), the water content using the official method of AOAC (12), the calcium oxalate content follows the Iwuoha *et al.*, method (13), levels of citric C follow the Sidarmadji (11) method, pH using Sudarmadji *et al.* (11), total acid follows the method of Sudarmadji *et al.*, (11), organic acid levels using the HPLC method (14), color observations following Yau's method, (15), texture observations following the method of Zoulias, (16). Scanning Electron Microscopy (SEM) (Manual Instruction type FEI EnspectS50).

F. Experimental Design

Optimization of mechanical injection of syringe for the response of color, texture, and calcium oxalate levels with the independent variables salt concentration and salting time using the surface response method (RSM). The two variables studied in this study were salt concentration and salting time. The middle point of the study design was taken from the salt concentration and the length of salting the preliminary study.

Code -1, 0 and 1 are symbols that show the value of each variable. Code -1 shows the minimum value, code 0 shows the optimum value and code 1 shows the maximum value. In this experiment X_1 is a salt concentration variable with code -1 (1%), 0 (1.75%) and 1 (2.5%), code X_2 is the length of salting with code -1 (5 hours), 0 (9.50 hours) and 1 (14 hours). All treatments consisted of 13 saline processes where each process condition followed a central composite design trial design (Table 1). The data obtained is analyzed using the Design Expert software version 7.1. to estimate the responses. The behavior of the sytem was explained by the following quadratic equation:

$$y = \beta_0 + \sum_{i=1}^3 \beta_i X_i + \sum_{i=1}^3 \beta_{ii} X_i^2 + \sum_{i=1}^2 \sum_{j=2}^3 \beta_{i,j} X_i X_j + \varepsilon....(1)$$

Where Y is the observation response, $\beta 0$ is the intercept, βi is linear coefficients, βi iis quadratic coefficients, βi j is treatment interaction coefficients, Xi is treatment codefor (i) factor and Xj is treatment code for (j) factor.

TABLE 1. Composite Design of the Second Order Center with Two Factors

	Para	Respon			
Std	Konsentrasi garam (%)	Lama Penggaraman (Jam)	Warna (L)	Tekstur (Gram)	Kadar Oksalat (mg/100 g)
1	1.00	5			
2	2.50	5.00			
3	1.00	14.00			
4	2.50	14.00			
5	0.69	9.20			
6	2.81	9.50			
7	1.75	3.14			
8	1.75	15.45			
9	1.75	9.50			
10	1.75	9.50			
11	1.75	9.50			
12	1.75	9.50			
13	1.75	9.50			

Statistic analysis

Optimization using Design Expert software (Version 7.1, Stat-Ease Inc., Minneapolis, MN). All analysis were carried out in three replications and the results were given \pm standard deviation. The two-way model of variance (ANOVA) with the type of commercial injection acid, the treatment and interaction applied to the analytic data obtained using the MINITAB version 14 software program (Minitab inc.). The Tukey test is used to determine interactions. Statistically significant differences were determined at the level of p <0.05

III. RESULTS AND DISCUSSION

A. Characterization of Commercial Suntic Acid

Commercial sunti acids have physicochemical content that from region to region. The physicochemical varies composition includes salinity, water content, calcium oxalate, vitamin C content, pH, total acid, color L a b, texture (Table 2). The physicochemical properties of commercial injections have a significant effect. This is due to the process of salting, drying and the climate where raw materials grow. Observation in the field of the process of making an injection acid wherein, pidie syringe acid uses about 2% of the amount of material with a 12 hour salting time and 8 hours of drying time. Lhokseumawe syringe acid uses 3% salt with 14 hours salting time and 9 hours drying time. Furthermore, Aceh Besar syringe uses 3.5% salt with 14 hours salting time and 9 hours long drying. The place to grow raw materials (Averrhoa bilimbi) is different from each region. Lhokseumawe has a soil pH of 6, Pidie has a soil pH of 5.5 and Aceh with a large soil pH of 7.10. The pH of the soil affects the nutrients in the soil which form the chemical content in the fruit. It was suspected that salting and drying time affected the physicochemical properties of commercial injectable acids. The use of high salt will produce a high-salt end product. The higher the salt and the longer the drying affects the color. The higher the salt the faster the conversion rate of chlorophyll forms pheophytin (brown) by the enzyme chlorophyllase. Furthermore, heating will release magnesium contained in chlorophyll to form pheophytin. Repeated heating will cause the release of the phytol group in chlorophyllide to form pyropheophytin (brown).



The commercial texture of sunti acid is closely related to salt and drying. Allegedly the higher the salt is thought to cause the higher the thermostatic pressure in attracting water in the material and the salt solution will enter the material. The longer salting the faster the tissue cells damage in the material so that the water in the material will be easily free. High salt levels cause increased hardness and thickness of fruit flesh because high levels of NaCl occur in Ca²⁺ uptake which plays a role in maintaining hardness and improving the texture of the material. Furthermore, the longer the drying is thought to cause changes in the cell wall of the material and cause more water loss. stated that enzymatically changes in texture from soft to soft are related to the degradation of the polysaccharide component by the enzyme *pectinolytic* and *cellulolytic* and the hydrolysis of *polygalactonate* acid (17). Differences in chemical composition are influenced by the process and place of growing both internal and external raw materials.The salting process and the different maturity level of the fruit will produce different chemical compositions (18). Commercial sunti acid composition table 2.

NO	Donomotor	Acid Sunti	Acid Sunti	Acid Sunti
INU	rarameter	Lhokseumawe	Pidie	A. Besar
1	Salinity (%)	35,41±0,58 ^b	30,50±0,68°	44,71±0,55 ^a
2	Water content (%)	36,96±0,01°	41,57±0,02 ^b	48,80±0,01 ^a
3	Ca- Oxalate content (mg/100g)	50,99±1,67 ^b	48,63±1,1°	57,16±0,40 ^a
4	Vitamin C content (%)	19,1±2,54 ^b	23,5±2,54 ^a	14,7±2,54°
5	pН	$2,4\pm0,05^{a}$	$2,7\pm0,05^{a}$	$2,6\pm0,10^{a}$
6	Acid content (%)	$5,76\pm0,64^{a}$	$6,08{\pm}0,87^{a}$	$5,44\pm0,64^{a}$
7	Color L (<i>Lightness</i>)	38,1±0,60 ^b	43,1±0,20 ^a	32,5±0,20 ^c
8	Color a+ (redness)	3,9±0,57°	$8,6\pm0,40^{a}$	5,9±0,57 ^b
9	Color b+ (yellowness)	7,7±0,84 ^b	11,8±0.28 ^a	4,14±0,44°
10	Hardness (g)	732,2±3,92 ^b	542,3±1,85°	656,9±6,26
11	Springiness (mm)	8,45±1,22 ^a	6,92±2,46°	$7,02\pm3,23^{b}$

TABLE 2. Physical Physicochemical Composition of Commercial Suntic Acid.

Description: Different notations on the same line show significant differences at $\alpha=0.05$

B. Sensory Evaluation

The results of the Kruskal-Wallis test showed a significant effect (p < 0.05) on color, texture and overall attributes. Thus indicating that the panelists could feel the difference in the intensity of the attributes of the products presented. The results of variance showed significant differences in color, texture and overall attributes. The mean significance values of Kruskal-Willis and sensory attributes are presented in Table 3.

TABLE 3. Mean significance value and mean value of commercial sensory acid sunti attributes

NO	Code / Region	Color	Texture	Overall		
1	231 (Lhokseumawe)	2,20±0,53°	3,20±0,63°	3,00±0,63 ^b		
2	232 (Pidie)	$4,10\pm0,41^{b}$	$4,00\pm0,40^{b}$	$4,72\pm0,45^{a}$		
3 233 (Aceh Besar) $6,92\pm0,27^{a}$ $5,04\pm0,44^{a}$ $1,02\pm0,89^{c}$						
Remarks: p <0.05 indicates significant.						

different notations in the same column show difference at $\alpha = 0.05$.

The results of the variance analysis showed that there were significant differences (p <0.05) between the three

samples for the hedonic score for the whole. The value of all attributes between dislikes really likes on a hedonic scale. To add Lhokseumawe syringe, I don't like it. Reflecting the fact that 50% of the panelists set a dislike score on the Lhokseumawe sample. Whereas for Pidie sunti acid likes or approaches very like it. Reflecting that 80% of panelists determined that they were as fond of Pidie syringe. In contrast, only 19-23 panelists gave disliking scores on the overall attributes of Aceh Besar's injectable acid.

Panelists considered that the color of the Lhokseumawe syringe sample was dark (faded brown) and gave comments such as dislike. Pidie syringe acid attributes reddish brown color and gives a fondness for Pidie syringe. On the other hand, sam sunti Aceh Besar has a whitish brown color attribute and gives a value of dislike. What's interesting is that more than 25% of the panelists gave the score too dark and too bright for the color of the injectable acid product. Scores for the texture of the hardness of the panelists gave a slightly harsh value to the Lhokseumawe Sodium Acid product and gave a value of dislike. Panelists give a soft Pidie syringe acid score and give a liking value. Instead, the panelists gave the Acehnese injection acid score Large soft and gave a value of dislike. More than 50% of panelists gave a near-true score for commercial injectable acid products. Thus, color and texture can have a negative impact on the overall preference of the product.

C. Determining the Best Product

Determination of the best products using the Multiple attribute method Zeleny (10). The parameters used are the levels of salts, calcium oxalate levels, and textures. The expected value on all parameters is the lowest. While the highest expected color value parameter. From the results of calculations that have been done, the best results from a number of compositions are Pidie syringe acid at 30.50% salinity, calcium oxalate 48.67 mg/100g, hardness 542.3 g, and color 38.1.

D. Experimental Design

The preliminary study aims to obtain the upper limit and lower limit to be inputted to the RSM. The formulation of the use of salt for injecting acid in the preliminary research study can be seen in Table 4 below:

	Formulation		Dependent Variable			
NO	Salt concentration (%)	Salting time (hour)	Color (L)	Texture (Gram)	Calsium oxalate (mg/100 g)	
1	1	8	23,7±0,15	686,6±5,61	53,7±0,17	
2	1	12	27,8±0,15	668,7±6,56	49,3±0,35	
3	1,5	8	48,0±0,40	516,9±2,89	40,0±0,51	
4	1,5	12	44,3±0,53	557,3±1,20	40,5±76	
5	2	8	55,7±0,21	954,0±3,13	47,0±0,36	
6	2	12	60,9±0,79	992,7±2,71	49,2±0,25	
7	2,5	8	31,9±0,57	1120,8±9,80	50,8±0,20	
8	2,5	12	18,3±0,25	1053,5±2,37	47,9±0,74	
9	3	8	16,3±0,15	1332,2±2,57	51,1±0,17	
10	3	12	14,9±0,30	1460,6±0,42	52,1±0,36	

TABLE 4. Preliminary Research, Formulation of Salt Concentration and Salting Time



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Description: Form Value Average \pm standard deviation of 3 replications.

Based on the results of preliminary research salt concentration formulation and duration of acidification of the injection syringe process were found in the treatment of 1.5% salt concentration and 8-hour salting time which had a brightness value (L) of 42,3±0,66, texture 549,7±0,44g and calcium oxalate levels 40±0,66mg/100g. The formulation was chosen because when compared to the treatment of 1.5% salt concentration and 12 hour salting there was an increase in brightness of 6, a decrease in the hardness of 42.9 g and a decrease in calcium oxalate levels of 5 mg/100 g of material. Increasing the value of brightness and texture is much brighter than the brightness and texture of other treatments. When the salt concentration of 2% and the duration of 8 hours of salivation were significantly reduced by 17,7 brightness levels, the texture increased by 444g, and calcium oxalate 7 mg/100g. Therefore the formulation of salt concentration of 1,5, and the duration of salting 8 hours will be used as a center point in the optimization of the formulation in the main research RSM.

The best formula in the preliminary study was 1,5% salt concentration and 8 hours salting time. Because of that the lower limit and upper limit factor concentration are 1% (-1) and 2,5% (+5), respectively. At the time of salting the lower limit and the upper limit each 5 hours (-1) and 12 (+1). Table 5 The independent variable values for the RSM design response.

TABLE 5. Color response, texture and calcium oxalate Composite Center of the Kedu Order with two factors.

	Para	ameter	Respon		
No Std	Konsentrasi garam (%)	Lama Penggaraman (Jam)	Warna (L)	Tekstur (Gram)	Kadar Oksalat (mg/100 g)
1	1	5	24,8	818,1	59,1
2	2,5	5	16,7	951	51,7
3	1	14	26,1	749,3	51,7
4	2,5	14	15,3	953,8	59,2
5	0,69	9,20	29,4	974	60,7
6	2,81	9,50	20,6	971,1	59,6
7	1,75	3,14	28	790,8	56,1
8	1,75	15,45	30	612,3	60
9	1,75	9,50	41,1	491,3	41,1
10	1,75	9,50	44,5	530,6	40,2
11	1,75	9,50	42,1	533,1	41,8
12	1,75	9,50	40,3	498	40,2
13	1.75	9.50	45.2	452.9	38.4

a. Color response modeling and analysis

The color response analysis shows that the quadratic model is the suggested model. In the summary statistical model, the quadratic model has the highest adjusted R2 and predicted R2 compared to the linear and 2FI models, which are 0.8670 and 0.5239, respectively. The quadratic model is known to have a low PRESS (Prediction Error Sum of Squares) value of 63.30. Fig. 1. Showing the model produced by the color response is a quadratic model. This model has a value of R^2 0.9224 which is close to 1. The closer it is to one, shows that the correlation between the observed values and predictive values is more appropriate (Saniah, 2008).



The quadratic equation obtained for the level of color (brightness) of the syringe acid is:

 $\begin{array}{l} Y =- \ 46,21056 + \ 61,31693 \ X_1 + \ 8,29672 \ X_2 - \ 0,20000 \ X_1 \ast X_2 \\ - \ 18,46889 \ X_1^{\ 2} - \ 0,41426 \ X_2^{\ 2}. \end{array}$

Where Y: brightness (L), X_1 : Salt concentration (%), X_1 : Length of salting (hours)

b. Texture response modeling and analysis

The results of the color response analysis, indicate that the quadratic model is the suggested model. In the summary statistical model, the quadratic model has the highest adjusted R^2 and predicted R^2 compared to the linear and 2FI models, which are 0.9418 and 0.6939, respectively. Figure 2. shows the model produced by the color response is a quadratic model. This model has a value of R^2 0.9503 which is close to 1.



The quadratic equation obtained for the sunti acid level is: $Y = + 2382,58 - 1509,54 X_1 - 119,39930 X_2 + 5,30370 X_1 * X_2 + 432,77333 X_1^2 + 5,33012 X_2^2.$

Description Y = Hardness (g), X_1 = Salt Concentration (%), X_2 = Duration (hour).

c. Calcium oxalate response modelling and analysis

The results of the color response analysis indicate that the quadratic model is the recommended model. In the summary statistic model, the quadratic model has the highest adjusted value R^2 and predicted R^2 compared to the linear model and 2FI of 0.9791 and 0.9537 respectively. The quadratic model is known to have a low PRESS (Prediction Error Sum of Squares) value of 41.50.

Figure 3 shows the model produced by color response is a quadratic model. This model has an R^2 value of 0.9878 which is close to 1.



The quadratic equation obtained for the reduction of calcium oxalate acid is :

 $\begin{array}{l} Y=+\ 141,\!92712-66,\!64224\ X_1\!-\!9,\!22186\ X_2\!+1,\!10370X_1\!\!*\!X_2\\ +\ 15,\!97556\ X_1^{\ 2}\!+2,\!49\ X_2^{\ 2} \end{array}$

Where: Y = Calcium oxalate (mg/g), X₁ = Salt Concentration (%), X₂ = Length of Salting (hours).

The effect of salt concentration and salting duration on the response can be seen in Figures 1, 2 and 3. The color response will continue to decrease the brightness value (darkness) in response to high salt concentration and salting duration. On the contrary, the higher the concentration of salt and the longer the salting duration, lead to brighter color and harder texture. If the salt concentration is lower by 1,75% and the salting duration shorter than 9,50 hours, a brighter color and softer texture will be produced. This may be because of the optimal relationship between salt concentration and the length of salting has been reached.

Salt can cause faster *chlorophyll* conversion rates to form pheophytin (brown color). The higher salt is thought to decrease the *chlorophyll* activity in the material caused by the increased activity of the enzyme chlorophyllase. *Chlorophyllase* is an enzyme that can catalyze the degradation of chlorophyll. The change in color from bright green to brown that occurs during the process is caused by the degradation of *chlorophyll* to form a pheophytin due to the release of magnesium in chlorophyll replaced by hydrogen formed pheophorbides and chlorine. The addition of salt increases alkalinity which reduces the level of chlorophyll degradation. Solution to inhibiting the conversion of chlorophyll to pheophytin during heating by using 1.2% salt has a greater effect in maintaining and preserving its color (19). A number of studies have evaluated the color kinetics and degradation in fruits and vegetables such as broccoli (20), peas (21), Spinach (22). Other factors that change its color are low pH, the temperature, the presence of salt, the enzymes and the active ions that affect the stability of chlorophyll. *Chlorophyll* has better stability at high pH than low pH.

The length of salting can increase the hardness of the material. Allegedly, the longer the material in the salt solution the longer the salt attracts water content so that the faster the cell wall is damaged which leads to the lower the water content in the material that can cause physical changes. The longer the salting duration the softer texture caused by changes in tissue cells material, the bigger the damage of the material in the heating process the more accelerate the softening of texture. In the study of explained the level of material texture depends on the salt concentration which at 2,7% salt concentration on the third day showed a change in non-soft texture, but on contrary1 2,7% concentration on the third day showed a softer texture (23). The salt reduction can affect water holding capacity, texture, sensorial attributes, stability, and life (24).

The decrease in calcium oxalate contained in the material is caused by the reaction of salt and calcium oxalate bound so that the particles of oxalic acid are bound in the chemical circuit of salt. That salt was able to reduce calcium oxalate levels by 60,78% in walur tubers (25). Treatment soaking in the water with 10% of salt for 120 minutes can reduce calcium oxalate levels by 51,5% in talas benang (26). The optimal concentration can cause a decrease in calcium oxalate levels. Dissolved salt will change into sodium ions and chloride ions, and will bind with calcium oxalate to form sodium oxalate and the deposits of water-soluble calcium oxalate dichloride. *d. Optimal point response and verification*

The value of the stationary point was obtained from the analysis carried out under optimal conditions includes salt concentration of 1,77% and salting time of 10,03 hours, weight of material 2000 g, and the result of the analysis produced colors, textures and calcium oxalate levels under optimal conditions where L 42,47, texture 499.02 g, and 40,05 mg / 100g, respectively. This condition is optimal for getting the color level, texture, and decrease in calcium oxalate. Verification was done by comparing the response analysis of the research with the response value predicted by Design Expert. The level of color accuracy, texture and calcium oxalate were 98,66%, 99,66%, and 97,56%, respectively, while the difference values were 0,34%, 1,33, and 2,44% respectively. All results were obtained from real experiments, showing a validation of the Response Method of Surface Methodology. The verification and desirability value of 0.92 showed accurate results. The optimal and actual verification is presented in Table 6.

	Salt concen tration	Salting Duration	Color (L)	Texture (g)	Ca- Oksalat (Mg/10 0g)	Desirab ility
Predic tion	1,77	10,03	42,47 69	499,027	40,059	0,92
Actua 1	1,77	10,03	43,1	497,3	40,7	-
Accuracy Level (%)		98,67	99,66	97,56		
Difference Value (%)		1,33	0,34	2,44		

TABLE 6. Optimal Verification Selected From Expert Design Models

E. Phytochemical Analysis of Sunti Acid Optimization and Commercial Sunti Acid

Characterization of optimal injectable acid and the best commercial injectable acid stage 1. Parameters that were analyzed for salinity: levels of oxalate, citric acid, malic acid, lactic acid, color, and texture. The chemical compositions of optimal sunti acid and commercial sunti acid are presented in Table 7.



Lactic Acid (ppm)

Malac Acit (ppm)

Citric Acid (ppm)

Color L (Lightness)

Texture (Hardness) (g)

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TABLE 7. Physical Physicochemical Composition of Optimal Sunti Acid and					
Commercial Sunti Acid					
Domenter	Acid sunti	Acid Sunti			
Parameter	Optimal	Komersial			
Salt content (%)	$28,46\pm0,28^{a}$	30,50±0,28 ^b			
Ca-oxalate Level (mg/100g)	40,7±0,98 ^b	48,67±1,68 ^a			

130,435

0.222

247,92

43,1±0,20b

497,3±1,70^b

4.87

5.868

99,252

45,1±0,66^a

542,3±1,8ª

Based on Table 7 the salt content of the commercial sunti acid is 30.50 ± 0.28 higher than the optimal sunti acid 28.46 ± 0.28 . This is because, in the process of making commercial sunti acid, the use of salt is higher than the optimal sunti acid. The use of high concentration salt in the substance will produce high salt content. On the contrary, the lower the use of salt in the processing of sliced acids will result in lower salt content. (27) Reported that high salt concentrations caused an increase in salt content in the material. Increased salt levels in commercial sunti acids were influenced by the amount of salt and the repeated addition of salts.

The highest calcium oxalate content was found in commercial acids 30.50 ± 0.28 while the lowest was found in optimal sunti 28.46 ± 0.28 . In the study (28) States that explains that by boiling it at 800C for 30 minutes on taro tubers can reduce the content of calcium oxalate by 70%. Calcium oxalate compound is difficult to dissolve in water with solubility at 200C only 0,00067 g / g H2O at 900C, calcium oxalate solubility was obtained at 0.001 g / g H2O. The salting process can reduce calcium oxalate levels. In study, soaking in salt solutions can reduce the level of calcium oxalate 40,37% sante tuber (25). The NaCl solution can reduce calcium oxalate levels due to the ionization of NaCl in water into Na⁺ ions and Cl⁻ ions. The Na⁺ ion will bind to oxalate to form sodium oxalate compounds and chloride residues which are easily soluble in water. Calcium oxalate crystals have a negative effect on the body if consumed in excess which can cause gout and kidney stones.

Analysis of organic acid by HPLC method with wavelength 215 nm on optimal sunti acid and commercial sunti acid. The highest lactic acid content was found in optimal sunti acid 130.435 ppm and the lowest was found in commercial sunti syringe 4.87 ppm. This might be influenced by the length of salting. The longer the salting the more the content loses in the ingredients along with the release of liquid in the material by salt. Furthermore, the longer the storage is carried out, the greater the possibility of losing lactic acid content which is influenced by oxygen and the temperature of the storage space.

Table 5. The increase in citric acid is higher in optimal sunti acid, which is 247.92 ppm and the lowest is commercial sunti acid 99.252 ppm. This is determined by salting. The higher and the longer the caring, the more material will be more diverse with the liquid in the material. The highest malic acid level in the commercial acid sunti is 5.686 ppm and the lowest at optimal acid is 0.222. This is a decision from the time of harvest. Many starfruits from Pidie contains Pidie Asam, which is harvested in the rainy season. (31) explain

which fruits are harvested during the rainy season, increase the chemical composition higher than that harvested in the dry season. Age and season affect the chemical content of the fruit. Delay in harvest time reduces the reserves of chemicals caused by an increase in secondary crops.

The highest brightness level of commercial injection acid is L 45 \pm 0.66 rather than optimal, which is L 43.1 \pm 0.20. This is because the concentration of commercial salt is higher than optimal and the drying time. Optimal injection acid is lower, which is 1,75%, while commercial is higher, which is 2%. The higher the salt the faster the conversion rate of *chlorophyll* forms *pheophytin*. Addition of chloride salts such as sodium, magnesium or calcium decreases *pheophytinization* activity because the electrostatic coating of salt occurs. The heating process will cause magnesium in *chlorophyll* to be released to form a derivative of *pheophorbide*, the release of Mg in *pheophorbide* causes a change in color from green to brownish. *Pheophorbide* formed when heated again, will release the *phytol* group found in chlorophyllide will become darker.

The highest level of hardness of injectable acid in commercial injection acid is 542.3 ± 1.85 g while the lowest in optimal injection acid is $497,3\pm 1,70g$. The optimal level of violence is softer than commercial. Explaining the character of the main textures of high-quality injectable acid which has an ideal hardness value of close to 500g (soft) (32). The difference in the level of optimal and commercial injectable acid texture is due to the high salt concentration and duration of drying. The optimal injection acid is drying 3 times while commercial injectable acid is 4 times. The use of high salt is inhibited in Ca²⁺ uptake. Ca²⁺ ion plays a role in maintaining hardness and improving the texture of the material. The effect of Ca²⁺ fruit hardness occurs because of cross-linking between Ca²⁺ ions and pectin (polygalacturonate acid residues) on the cell wall and middle cell so that the cell membrane stabilizes. The formation of crosslinking is influenced by the activity of the enzyme pectin methylesterase which can break up the methyl group in pectin compounds so that there is a free carboxyl group that can bind to Ca^{2+} ions (33). long drying times can produce unstable emulsions with poor texture and low water binding capacity. Short reduction in salt content and drying can affect water holding capacity, texture, sensorial attributes, stability and shelf life in ingredients (24).

F. Scanning Eletron Microscopy (SEM)

The optimization of SEM on the surface of the injection syringe showed that the salt particles found on the surface of the injectable acid were less optimal, with a smaller size and not clumping than the commercial injectable acid more salt particles, large and clot as shown in Figure at a enlargement of 2500 x. This is because the optimal injection of saline acid is lower than commercial use. The lower the salt used, the lower the salt content in the ingredients.

Figure 4 shows the difference in particle size of salt because the optimal injection of salt acid using salt is lower at 1.75% than commercial 2%. Giving salt repeatedly causes the salt to be higher and the buildup of salt in the material. Asan



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sunti is optimal for giving salt 3 times with a smaller amount than commercially giving salt 4 times and more.

Figure 5 shows different calcium oxalate crystals. Optimal injection acid is a less visible oxalate crystal "Fig. 4 (a)" of more commercial oxalate crystals "Fig. 4 (b)". This is because of the optimal acid through the boiling process at a temperature of 800C for 5 minutes and the use of salt in reducing calcium oxalate. Whereas in Pidie sunti acid reduction of calcium oxalate with the use of salt.

According to Iwuoha et al., (1995) by boiling at 90° C for 40 minutes it can reduce calcium oxalate on average by 70%. Boiling treatment at 80° C in 8% NaCl solution with boiling time for 25 minutes, where the calcium oxalate content left in porang tuber was 0.55% in other words the decline reached 90.9%(34).

Based on the results of scanning electron microscopy test the form of oxalate crystals in the starfruit is in the form of raphida (needle). Various forms of raphida are found divided in a single raphida form with a pointed tip "Fig. 5 (a)" in the form of a file "Fig. 5 (b)". The most common morphologies of calsium oxalate crystals in plants are shown in Figure 6.



Fig. 4. Observation of Salt Crystals on Optimal Suntic Acid Surface (a) and Commercial Sunti Acid (b) enlargement of 2500 x

These morphologies include block-like rhombohedral or prismatic crystals present as single or multiple crystals per cell, large elongate rectangular styloids that occur as single crystals per cell, bundles of needleshaped (acicular) raphide crystals, masses of small angular crystals referred to as crystal sand, and multifaceted conglomerate crystals called druses (often single but also multiple per cell) (29). Calcium oxalate crystals vary and are generally described in the form of raphida, druse, styloid, prism, and sand crystals (36).



Fig. 5. Presence of Oxalate Crystals in Optimal Suntic Acid (a) Commercia Suntic Acid (b) with enlargement of 250 x





Fig. 6. The overall form of oxalate crystal (a) and oxalate crystal rod (b) in sunti acid enlargement of 2000 and 2500 x

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Calcium oxalate crystals are solid, erganistic materials. Formed as the end result of metabolism, there are also formed due to the occurrence of solids of liquid food reserves so that they are in the form of granules. These crystals are quite numerous in the cortex, parenchyma, phloem and xylem parenchyma can also be found in vacuoles or plasma cells.

IV. CONCLUSION

- A. Formulation of the best treatment operation is 1.77% salt concentration and 10.03 hours salting time with a brightness response rate of 42.45, texture (hardness) 499.04 and calcium oxalate 40 mg / 100g. The actual response of the optimum formulation has a brightness value of 45.1, 497.3 hardness and calcium oxalate level 28.4 mg / 100g.
- *B.* Optimization of the injection syringe produces the same optimal injection acid with the best stage 1 injectable acid that consumers like.
- *C.* Optimum physicochemical comparison of injectable acid with commercial injection acid shows optimal injection acid is better than commercial injectable acid.

REFERENCES

- Mokhtar, I.S., and Aziz, N.A., "Antimicrobial properties of Averrhoa bilimbi extracts at different maturity stages" Journal of Medical Microbiology & Diagnosis, vol 5, issue 3, pp. 233. 2016
- [2] Rana, S.M., Billah, M.M., Hossain, M.S., Saifuddin, Islan, S.K., Banik, S., Naim, Z., and Raju, G.S., "Susceptibility of microorganism to selected medicinal plants in Bangladesh" Asian Pacific Journal of Tropical Biomedicine, vol 4, issue 11. pp. 911-917. 2014.
- [3] Kumar, K.A., Anupama, S.K., and Latha, J.N., 3013. "A review on phytochemical constituents and biological assays of Averrhoa bilimbi". International Journal of Pharmacy and Pharmaceutical Science Research, vol 3, issue 4, pp. 136-139.
- [4] Vera, L.A., Lima, D.G., Mélo A.E., and Lueci, D.A. 2001. "Physicochemical Characteristics Of Bilimbi (*Averrhoa bilimbi* L)". Journal Bras. Frutic. Jaboticabal – SP, vol. 23, issue 2, pp. 421 - 423.
- [5] Hayati, R., Soewarnot, T., Soekarto, end Nuraida, L. 2002. "Study of Salting and Drying of Wuluh Starfruit (Averrhoa bilimbi) in Making Asam Sunti from Aceh". Agripet, vol. 3 issue 1, pp. 29-36.
- [6] Eren, X.C., Wijaya, H., Farida, N.D. 2017. "Charaterization of Aroma Compounds in Indonesian Traditional Seasoning (Asam Sunti) Made From Averrhoa bilimbi L". Journal of Food and Agriculture, vol. 29, issue 5, pp.378-386.
- [7] Nilawati, dan Nurlela. 1999. "The influence of blanching and salt use on starfruit to produce asam sunti products". Bull. Litbang Industri, vol. 26, pp. 7-12.
- [8] Maximo, G., Rutenbeck, S., Lana P., Anna, V.A., Resurrection and Howard, R.M. 2007. The Just About Right Intensity Scale, Functional Analyses and Relation to Hedonic. Journal of Sensory Studies, vol 22, hlm. 192-211
- [9] Massey, L. K., Roman-Smith, H., end Sutton, R.A.L., 1993. "Effect of dietary oxalate and calcium on urinary oxalate and risk of formation of calcium oxalate kidney stones". Journal of the American Dietetic Association, vol. 93, issue 8, pp. 901-906.
- [10] Zeleny, M. 1982. "Multiple Criteria Decision Making". Mc Graw Hill Book Company. New York
- [11] Sudarmadji, S., B. Haryono, dan Suhardi. 2007. Prosedur Analysis for Food and Agriculture. Liberty. Yogyakarta.
- [12] AOAC, 2005. Official Methods of Analysis. Association of Official Analytical Chemists. Benjamin Franklin Station, Washington
- [13] Iwuoha, C.I., and Kalu, F.A., 1995. "Calcium Oxalate And Physico-Chemical Properties of Cocoyam (*Colocasia esculenta* and *Xanthosoma sagittifolium*) Tuber Flours As Affected By Processing". Food Chemistry, vol. 54, issue 1, pp. 61–66.

- [14] Nour, V. I., Trandafir, and Lonica. 2010. HPLC Organic Acid Analysis In Different Citrus Juice Under Reversed Phase Conditions. Not. Bot. Hort. Agroboth. Cluj. Artikel.
- [15] Yau, E.W., Rosnah, S., Noraziah, M., Chin, N.L., Osman H., 2010. "Physico-chemical Composition of the Red Seedless Watermelons (Citrullus lanatus)". International Food Research Journal, vol. 17, issue 2, pp. 327-334.
- [16] Zoulias, E.I., Oreopoulou, V., Tzia, C., 2002. "Textural properties of low fat *cookies* Containing Carbohydrate or Protein Based Fat Replacers. Journal of Food Engineering, vol. 55, pp. 337–342.
- [17] Papageorge R.M., McFeeters, M.F., and Fleming, H.P., 2003. "Factors Influencing Texture Retention of Salt-free, Acidified Red Bell Peppers during Storage. Journal of Agricultural and Food Chemistry, vol. 51, issue 5, pp 1460-1463.
- [18] Muzaifa, M., 2018. "Changes in the chemical components of belimbing wuluh starfruit (averrhoa bilimbi) during making sunti acid". Journal of agricultural technology". vol. 22, issue 1. pp 37-42.
 [19] Hudson, M.A., Sharples, V.J., & Gregory, M.E., 1974. "Quality of home
- [19] Hudson, M.A., Sharples, V.J., & Gregory, M.E., 1974. "Quality of home frozen vegetables. II. Effect of blanching and or cooling in various solutions on conversion of chlorophyll". Journal of Food Technology, vol. 9, pp 105–108.
- [20] Weemas, C.A., Ooms, V., Van Loey, A.M., & Hendrickx, M.E., 1999. "Kinetics Of Chlorophyll Degradation And Colour Loss In Heated Broccoli Juice". Journal of Agriculture and Food Chemistry, vol. 47, issue 6, pp 2404–2409.
- [21] Ryan, S.T., & Tong, C.H., 2000. "Degradation kinetics of chlorophyll in peas as a function of pH". Journal of Food Science, vol. 65, issue 8, pp 1296–1302.
- [22] Canjura, F.L, Schwartz, S.J., & Nunes, R.V., 1991. "Degradation kinetics of chlorophylls and chlorophyllides". Journal of Food Science, vol. 56, issue 6, pp. 1639–1643.
- [23] Miguel, A., Cervantes, B., Dartl, Lund, and Norman, F.O., 1983. "Effects of Salt Concentration and Freezing on Mozzarella Cheese Texture". Journal Dairy Sci., vol. 66, issue 2, pp. 204-213.
- [24] Clemens, R., 2012. "Food science and sodium". Food Technology, vol. 66, issue 1, pp. 9.
- [25] Chotimah, S.T.F. 2013. "Reduction of calcium oxalate by boiling using nacl solution and supplement to improve the quality of the santé (*Alocasia Macrorrhiza*) Sebagai Bahan Pangan". Journal of Chemical and Industrial Technology, vol. 2, issue 2, pp 76-83.
- [26] Syafrizal, M., Muharfiza, and Lestari, S., 2015. "Reduction of oxalate levels in Banten local's taro through immersionin salt water". PROS SEM NAS MASY BIODIV INDON, Vol. 1, issue, 7, pp. 1707-1710.
- [27] Ruusunen, M., & Puolanne, E., 2005. "Reducing sodium intake from meat products". Meat Science, vol 70, issue 3, pp 531–541.
- [28] Savage, G.P., & Dubois, M., 2006. "The effect of soaking and cooking on the oxalate content of taro leaves". International Journal of Food Sciences and Nutrition, volume 57 issue, 5/6, pp 376-381.
- [29] Fransisca, P.P., Nurwantoro and Pramono, Y.B., 2011. "Total lactid and bakteria, lactid acid levels, and yogurt drink with adding extract bit (*Beta vulgaris* L.)". J. Food Technol. Vol 5, issue 4, hlm. 154-156.
- [30] Gad, A.S., Kholif A.M., and Sayed, A.F., 2010. "Evaluation of the nutritional value of functional yogurt resulting from combination of date palm syrup and skim milk". J. Food Technol., vol. 5, issue 4, pp. 250-259.
- [31] Lennox, A., Ragoonath, J., 1990. "Carambola and bilimbi". Fruits, Paris, vol. 45, issue 5, hlm. 497-501,
- [32] Irhami. Study of Post Harvest Treatment Belimbing Wuluh (Averrhoa bilimbi, L) as Raw Material for Making Sunti Acid Powder Using Kabu Dryingt (Pray Dryer). M.S Thesis. Departemen Post Harvest Technology. Institute Pertanian Bogor. Bogor. 2012.
- [33] Guzman, I.L. and M.D. Barret. 2000. Comparison of calsium chloride and calsium lactate effectiveness in maintaining shelf stability and quality of fresh-cut cantaloupes. Postharvest Biology and Technology, vol. 19, issue 1, pp 61-72.
- [34] Nyoman S.W., and Rasmito, A., 2018. "Decrease of calcium oxalate levels in porang umbi (*Amorphopallus oncophillus*) with heating process in the solution of nacl". Journal of Chemical Engineering, vol 13, issue 1, pp 1-4.
- [35] Vincent, R., Franceschi and Nakata, A.P., 2005. "Calcium Oxalate in Plants Formation and Function". Annu.Rev. Plat Biol., vol. 56. Pp. 43-45.



[36] Ilarslan, H., Palmer, R.G., Imsande J., end Horner, H.T., 1997. "Quantitative determination of calcium oxalate and oxalate in developing seeds of soybean (*Leguminosae*)". American Journal of Botany, vol. 84, issue 9, pp 1042-1046.