

## Study of Ultrasound Extraction and Stability on the Physicochemical Characteristics of Pure-Bred Purple Sweet Potato Extract of Unpad Collection

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### ABSTRACT

The purpose of this study was to obtain a solution of anthocyanin extract from blanched sun-dry purple sweet potato with the right ultrasound time to produce the best natural dye solution with the best physical and chemical characteristics. The method that used is the Experimental Method using a Randomized Block Design (RBD). The experiment consisted of four treatments and each was repeated three times. The next step is to determine the best ultrasound time (30,60,90,120 minutes). That the duration variation method of ultrasound extraction had a significantly different effect on each total anthocyanin, but did not have a significant effect on pH, yield, and moisture content. The treatment of ultrasound for 120 minutes produced the best characteristics with anthocyanin total 185.20 mg / g, color intensity L \* (brightness) 28.98, a \* (redness) 63.90, b\* (yellowness) 50.18, water content 85.39%, pH 2.93, and the yield of 28.66%. with the treatment of temperature stability and the best pH is at a temperature of 60°C and pH 3.

**Keywords:** Anthocyanin Pigment, Blanching, Stability, Ultrasound.

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### INTRODUCTION

Color is one of the factors that influence consumer acceptance of a food product. Most food products on the market, both food and beverages use dyes from synthetic materials that are added to make food products appear more attractive. Synthetic dyes in food products is rapidly used in food and beverages, and is often misused. For example excessive usage, if consumed in excess of the threshold value (set by the government through the Ministry of Health) one of which can cause several diseases such as cancer, stroke, and heart disease (Ernie, 1986)<sup>1</sup>.

Anthocyanin is a natural pigment that gives red, blue and violet colors. Anthocyanin sources are found in fruits, vegetables, flowers, and various types of plants. Christie et al. (2010) stated that Anthocyanins are water soluble and safe for consumption, so they are commonly used as natural dyes for food and beverage products.

Sweet potato plants (*Ipomoea batatas* L.) are food crops and ubiquitous groups originally from Latin America (Martin and Leonard, 1967)<sup>2</sup>. In Indonesia, these plants are favored by farmers because they are easy to manage and resistant to drought, besides that they can grow on various types of soil and have the best harvest age for 130 days (Herlin et al. 2012). Sweet potatoes can grow well and become fruitful if the climate requirements are appropriate during their growth. The growth temperature ranges from 10°C - 40°C and the optimum temperature is 21°C - 27°C (Jedeng, 2011)<sup>3</sup>.

Blanching is a process carried out on food before canning or freeze-drying. Blanching is a heating process in food using temperatures below 100°C. Blanching aims to deactivate enzymes that allow changes in color, texture, taste of food ingredients. But the purpose of blanching also varies depending on the material to be used and the purpose of the next process (Muchtadi et al, 2013)<sup>4</sup>.

To get optimal results, blanching should be carried out at controlled temperatures and times, cooling immediately without delaying processing. The right blanching treatment can bring many benefits, such as, avoid unwanted changes, reduce microbial content, maintain color, soften the tissue, assist the release of cellular gases in the tissue so as to prevent corrosion and improve the texture of dried food (Winarno, 2004)<sup>5</sup>.

The ultrasonic method is a method that uses ultrasonic waves, namely acoustic waves with frequencies greater than 16-20 kHz. They are non-destructive and non-invasive, so they can be easily adapted to various applications. According to Kuldiloke (2012)<sup>6</sup>, one of the benefits of the ultrasonic extraction method is to speed up the extraction process. This is evidenced by the study of Cameron and Wang (2014)<sup>6</sup> about the extraction of corn starch which stated that the yield of corn starch obtained from the ultrasonic process for 2 minutes was around 55.2-67.8%, almost the same as the yield obtained from heating with water for 1 hour which is 53.4%.

Things that affect the ultrasonic ability to cause cavitation effects applied to food products include ultrasonic

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Table 1 : Results of anthocyanin solution pigment color value in purebred purple sweet potato analysis

Treatment	L*	a*	b*
30 minutes	16.50 ± 0.26 <sup>d</sup>	47.78 ± 0.32 <sup>d</sup>	27.72 ± 0.50 <sup>d</sup>
60 minutes	18.45 ± 0.31 <sup>c</sup>	49.72 ± 0.55 <sup>c</sup>	30.39 ± 0.15 <sup>c</sup>
90 minutes	20.70 ± 0.40 <sup>b</sup>	56.45 ± 0.32 <sup>b</sup>	35.85 ± 0.08 <sup>b</sup>
120 minutes	28.98 ± 0.24 <sup>a</sup>	63.90 ± 0.18 <sup>a</sup>	50.18 ± 0.20 <sup>a</sup>

Description: The same letter shows no significant difference at the level of 5% according to the Duncan test

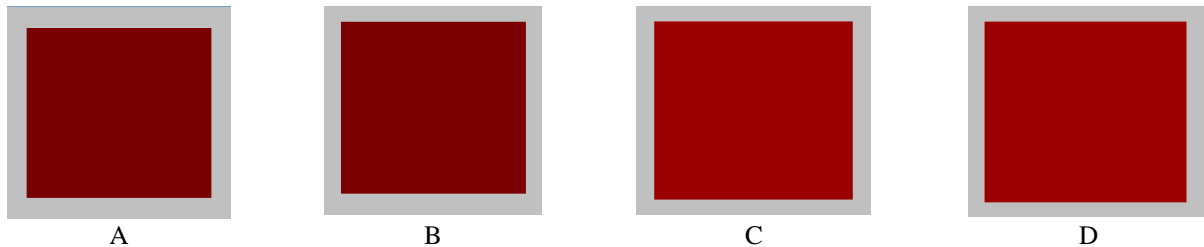


Figure 1 : Purple Sweet Potato Anthocyanin Pigment Solution

Information : A = 30 minutes Ultrasound; B = 60 minutes Ultrasound; C = 90 minutes Ultrasound; D = 120 minutes Ultrasound

Table 2 : Results Of Water Content Analysis of Purebred Purple Sweet Potato Anthocyanin Pigments.

Treatment	Water Content (%wb)
30 minutes	88.57 ± 1.13 <sup>a</sup>
60 minutes	87.31 ± 0.51 <sup>b</sup>
90 minutes	86.57 ± 0.46 <sup>bc</sup>
120 minutes	85.39 ± 0.56 <sup>d</sup>

Description: The same letter shows no significant difference at the level of 5% according to the Duncan test

Table 3 : Results of total anthocyanin analysis of purebred purple sweet potato anthocyanin pigment solution.

Treatment	Total Anthocyanin (mg/100 gram)db
30 minutes	65.64 ± 0.18 <sup>d</sup>
60 minutes	73.16 ± 0.81 <sup>c</sup>
90 minutes	126.93 ± 0.97 <sup>b</sup>
120 minutes	185.20 ± 0.37 <sup>a</sup>

Description: The same letter shows no significant difference at the level of 5% according to the Duncan test

characteristics such as frequency, intensity, amplitude, power, product characteristics (such as viscosity, surface tension) and surrounding conditions such as temperature and pressure. (Lestario et al., 2005)<sup>7</sup>.

## MATERIALS AND METHODS

### Research Materials and Tools

The materials used in this experiment were local yam variety of purple sweet potato, with a 4.5-month harvest age obtained from Ciparanje Jatinangor. The supporting raw materials used were distilled water, tartaric acid, and maltodextrin. The chemicals used for the analysis were distilled water, potassium chloride buffer solution (0.025 M) pH 1, sodium acetate buffer solution (0.4 M) pH 4.5, and concentrated HCl. Tools used include: analytical

scales, gas stoves, basins, cages, knives, cutting boards, vacuum ovens, filter cloth, silicone plastic molds, oven blowers. Tools used for analysis: filter paper, desiccator, analytical scales, rotary evaporator, uv-vis spectrophotometer, aluminum cup, magnetic stirrer, a set of centrifugation devices, PSA, crucible tong, cuvette, stirring rod, 100 ml beaker glass, 5 ml measuring pipette, 10 and 25 ml volumetric flasks, glass vials, batch ultrasound, freeze dryer, vacuum dryer and spray dryer.

### Research Method

The research conducted consists of two phases, preliminary phase and main phase.

#### Preliminary Phase

Preliminary research conducted is as follows:

The harvested purple sweet potato extracted by ultrasonic method with different treatment time, 30, 60, 90 and 120 minutes. Chemistry (water content and total anthocyanin).

#### Main Phase Research

The stages of making coloring preparations in detail are as follows:

##### Sorting and Peeling

Sorting and peeling aims to remove parts of blanched purebred purple sweet potato that has been damaged and separates parts that cannot be eaten like tuber skin.

##### Washing

Washing is done by flowing clean water, aiming to clean the sweet potato flesh from the dirt that is still attached like soil and the skin debris left behind after the stripping process.

##### Size Reduction

Reduction of the size of purple sweet potato was carried out using a knife, slicing was carried out longitudinally with a thickness of ± 1 cm sweet potato slices.

##### Blanching

Blanching aims to deactivate enzymes that allow changes in color, texture, taste of food ingredients.

##### Drying

The blanched purple sweet potato is then dried using a blow-dryer for 15 hours at a temperature of 50°C.

##### Ultrasound Extraction

Table 4 : Results of pH analysis of purebred purple sweet potato anthocyanin pigments.

Treatment	pH
30 minutes	2.87 ± 0.03 <sup>a</sup>
60 minutes	2.88 ± 0.03 <sup>a</sup>
90 minutes	2.98 ± 0.04 <sup>a</sup>
120 minutes	2.93 ± 0.11 <sup>a</sup>

Description: The same letter shows no significant difference at the level of 5% according to the Duncan test

Table 5: Results of yield analysis of purebred purple sweet potato anthocyanin pigment.

Treatment	Yield (%)
30 minutes	31.04 ± 0.54 <sup>a</sup>
60 minutes	30.37 ± 0.18 <sup>bc</sup>
90 minutes	29.47 ± 0.44 <sup>c</sup>
120 minutes	28.66 ± 0.28 <sup>d</sup>

Description: The same letter shows no significant difference at the level of 5% according to the Duncan test

Sweet potato is mixed with extracting solution, that is distilled water and 1% tartaric acid with a ratio of ingredients and extracting solution is 1: 5. Extraction is carried out aimed at removing pigment compounds from the tissue material. Manufacture of blanched pure-bred purple sweet potato anthocyanin pigment with four ultrasound treatments, namely 30 minutes, 60 minutes, 90 minutes, and 120 minutes.

#### Filtering

The filtering of purple sweet potato is done to separate the pigment compounds produced during extraction with starch which is still mixed with sweet potato pulp. Filtering is done with filter cloth. After the filtering process, the filtrate in the form of pigment and sediment in the form of starch is obtained.

#### Sedimentation

The filtration process has not been able to separate the starch as a whole which is still mixed in the pigment filtrate so that it needs to be settled for 12 hours at room temperature.

#### Decantation

Decantation aims to separate the constituent mixture in the form of liquid and solids. Decantation is done by pouring liquid into another container carefully so that the solids separate from the liquid. The principle of decantation is the difference in the form of matter in a mixture, which is between solids and liquids so that by using decantation techniques, liquids can be separated from the mixture.

#### Centrifugation

Centrifugation aims to separate the liquid pigments and solids in the form of starch because of the centrifugal force. The principle of centrifugation is that the material is inserted into a centrifuge tube and then rotated in a centrifuge device. When the material is rotated, the force that plays the most role is the centrifugal force so that the particles will separate and split according to the specific gravity of each particle. Centrifugation was carried out at a speed of 5000 rpm for 15 minutes at room temperature.

#### Concentration

Concentration aims to increase the concentration of pigment compounds by evaporating as much as 50% of the solvent by vacuum using a rotary evaporator. The concentration process is carried out at a temperature of 50°C to  $V = \frac{1}{2}$  Initial volume and produces concentrated anthocyanin extract. Testing the anthocyanin concentration of pigment extract with the pH-Differential-Lambert Beer method to determine the best ultrasound time refers to Modifications of Giusti and Wrolstad (2001)<sup>9</sup>, and supported by testing pH with a pH meter referring to AOAC (1984)<sup>10</sup>, color intensity of pigment extract by CIELAB method refers to Yam and Papadakis (2003)<sup>11</sup>.

## RESULTS AND DISCUSSION

#### Color

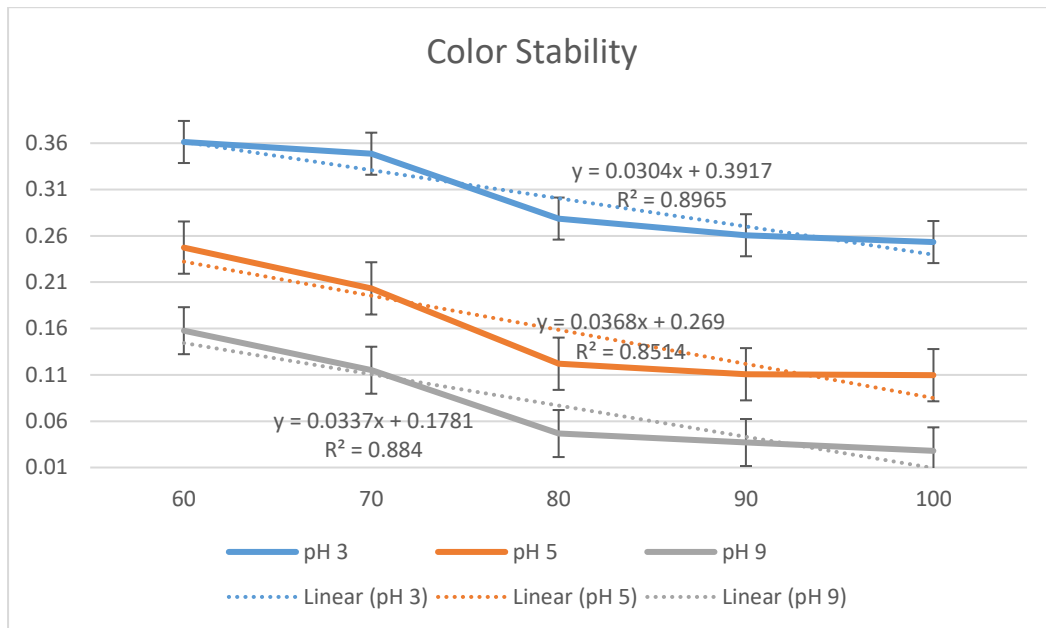
Based on the results of the statistical test in the Appendix, it shows that the effect of the ultrasound extraction duration on the anthocyanin pigment solution of sun-dry blanched purple sweet potato has a significant effect on the color produced. The results of statistical tests can be seen in Table 1.

According to the Duncan test at the level of 5%, the treatment of ultrasound extraction for 30, 60, 90, and 120 minutes gave significantly different results for the value of  $L^*$  of anthocyanin pigments. The brightness value ( $L^*$ ) shows the brightness intensity, where the higher the  $L^*$  value indicates the higher the brightness intensity. The highest  $L^*$  value for the 120 minute ultrasound treatment was 28.98 and the lowest on the 30 minute ultrasound was 16.50, the change in the resulting value was significant. This shows that the ultrasound extraction time of anthocyanin pigments influences the brightness intensity of the pigments produced.

The value of  $a^*$  is the red color intensity produced against an anthocyanin pigment solution. The higher the value of  $a^*$  indicates the intensity of red is higher. Based on Duncan's test at the level of 5%, the highest  $a^*$  value on the 120-minute ultrasound treatment was 63.90 and the lowest on the 30-minute ultrasound treatment was 47.78. The longer the ultrasound treatment shows the intensity of red (the value of  $a^*$ ) increases, the decrease that occurs is significant or each treatment gives a significantly different effect.

The value of  $b^*$  is the intensity of the yellow color produced against anthocyanin pigments. The higher the value of  $b^*$  indicates the higher the intensity of yellow. Based on the Duncan test at 5% level, the mean value of  $b^*$  on 30 minutes ultrasound was significantly different from the value of  $b^*$  on ultrasound 60,90 and 120 minutes. The highest  $b^*$  value on 120 minute ultrasound and lowest  $b^*$  value on 30 minutes ultrasound. The longer the ultrasound time shows the intensity of the yellow color increases. The color of the anthocyanin pigment solution can be seen in Figure 1.

Temperature rise in the processing through storage can cause changes and damage to anthocyanin that occur through the stages of hydrolysis on anthocyanin glycosidic bonds which produce unstabil aglycones and the opening



Graphic 1 : Graphic of color stability against ph

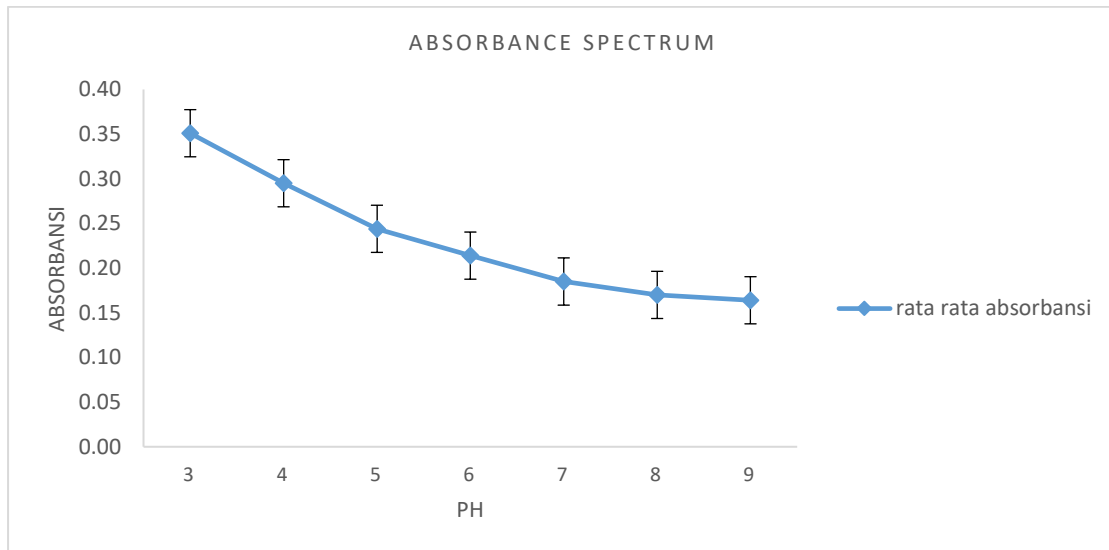


Figure 2 : Graphic of color stability against ph

of aglycone rings so that carbinol and chalcone groups are not colored According to Markakis (1982)<sup>12</sup>.

One of the factors that most influence the occurrence of anthocyanin degradation is temperature, where heating at a certain temperature can change the structure of the anthocyanin reaction in the equilibrium reaction from the cation form of flavium (red) to chalcone (colorless). This is in accordance with the results of the study that the higher the drying temperature shows the increasing anthocyanin degradation which is characterized by a decrease in the value of a \* and an increase in the value of b \*. The yellow color indicates the pigment is getting closer to the chalcon shape.

**Water Content**

Based on the results of statistical tests showed that the effect of the duration of ultrasound extraction on anthocyanin pigment solutions had a significant effect on

the water content produced. The results of statistical tests can be seen in Table 2.

Based on Table 2, the water content of the anthocyanin pigment solution on 30 minutes ultrasound was significantly different from ultrasound 60 minutes, 90 minutes, and 120 minutes, but 60 minutes ultrasound was not significantly different from the 90 minute ultrasound treatment. The highest water content of anthocyanin pigment solution on 30 minutes ultrasound was 88.57% while the lowest water content on 120 minutes ultrasound was 85.39%. The moisture content of anthocyanin pigments from purple sweet potato has decreased with the longer duration of ultrasound extraction. The water content decreases due to the evaporation of water in the higher ultrasound devices which is higher than the lower drying temperature.

**Total Anthocyanin**

Table 8 : Best treatment for purebred purple sweet potato anthocyanin pigment determination.

Observation Criteria	Scor	Purple Sweet Potato Anthocyanin Pigments			
		30 minutes Ultrasound	60 minutes Ultrasound	90 minutes Ultrasound	120 minutes Ultrasound
Brightness (L*)	4	16.50 d	18.45 c	20.70 b	28.98 a
Redness (a*)	7	47.78 d	49.72 c	56.45 b	63.90 a
Yellowness (b*)	2	27,72 d	30,39 c	35,85 b	50,18 a
Water Content (% b/b)	3	88.57 a	87.31 b	86.57 bc	85.39 d
Total Anthocyanin (mg/100g)	8	65.64 d	73.16 c	126.93 b	185.20 a
pH	6	2.87 a	2.88 a	2.98 a	2.93 a
Yield (%)	1	31.04 a	30.37 bc	29.47 c	28.66 d
Total		9	6	6	33

Description: The same letter shows no significant difference at the level of 5% according to the Duncan test

Based on the results of the statistical test in Appendix 3, it was shown that the length of ultrasound extraction to anthocyanin pigment solutions had a significantly different effect on the total anthocyanin produced. The statistical test results of ultrasound extraction time on pigment solutions can be seen in Table 3.

Based on Table 3, the highest total anthocyanin solution of anthocyanin pigment on 120 minutes ultrasound was 185.20% while the lowest total anthocyanin on 30 minutes ultrasound was 65.64%. This shows that the duration of ultrasound extraction has a significant effect on the total anthocyanin of anthocyanin pigment solution.

The purple sweet potato anthocyanin solution ranges from 65.64 to 185.20%. The anthocyanin pigment solution between treatments for the duration of ultrasound extraction has a high total anthocyanin. This is influenced by the nature of the anthocyanin pigment which dissolves well in water and the addition of acids used during the extraction process. Anthocyanin pigments have polar molecules that are more soluble in polar solvents such as water than non-polar solvents (Vargas and Lopez, 2003)<sup>13</sup>

#### pH

Based on the results of the statistical test in the Appendix, the duration of ultrasound extraction did not significantly affect the pH of the anthocyanin pigment solution. The statistical test results of ultrasound extraction time on the pH of anthocyanin pigment solutions can be seen in Table 4.

Based on Table 4, the pH of the pigment solution has almost the same average, this is due to the use of the same amount of tartaric acid at the ultrasound extraction time of 30 minutes to 120 minutes which is 2.87 - 2.93. Based on Table 4, the pH value of purple sweet potato anthocyanin pigment dissolved in distilled water has a range of 2.87 - 2.98. The pH value is within that range because in the study the tested anthocyanin pigments were dissolved in distilled water so that no other factors could affect pH stability.

According to Markakis (1982)<sup>12</sup>, the range of pH 2-4 has a small effect on the speed of anthocyanin destruction during

the heating process under the absence of oxygen, but the presence of large amounts of oxygen can accelerate the degradation of anthocyanins in the pH range 2-4. Anthocyanin has stability at low pH (acid), which is around 2-3. The pH value of the encapsulated anthocyanin pigments produced in this study ranged from 2-3 which indicates a pH range for anthocyanin stability.

The stability of the pH value is caused by the role of tartrate acid added to anthocyanin extract, because tartrate acid is a good acid in maintaining pH stability so that the resulting anthocyanin pigment is also quite stable. The stability of the anthocyanin pigment pH is also supported by previous discussion that the color of the pigment produced in all treatments is still red which indicates that the pigment is still in the form of a stable cation flavium.

#### Yield

Based on the results of the statistical test in Appendix 3, it was shown that the duration of ultrasound extraction of anthocyanin pigment solutions had a significantly different effect on the yield produced. The results of statistical tests can be seen in Table 5.

Based on Table 34, based on the results of the study showed that the treatment of ultrasound extraction for 30 minutes and 120 minutes was significantly different, but the ultrasound extraction treatment for 60 minutes was not significantly different from the ultrasound 90 minute extraction treatment. The highest yield for ultrasound extraction treatment for 30 is 31.04% and the lowest yield in ultrasound 120 minutes extraction treatment is 28.66%. This is caused by the heat generated from the ultrasound extraction process.

Ultrasonic has a faster and more perfect ability in the extraction process compared to the Yellow Pumpkin Carotenoid Extraction method by the Ultrasonic Wave Method according to Wahyuni (2015)<sup>14</sup>. Maceration and soxhlet. The mechanical effects caused by ultrasonic waves can increase the ability to penetrate solvents into material cells, thereby increasing the number of cell components that diffuse into solvents according to

Table 9: The Best Treatment Antioxidant Activity.

Treatment	Antioxidant Activity Mean IC <sub>50</sub> (ppm)
120 minutes Ultrasound	79,66 ± 0.56

Brennan (2006)<sup>15</sup>.

#### *Color Stability Against Temperature*

Based on the results of the research on the stability of red color from purple sweet potatoes the temperature between 60° - 100°C at pH 3 has an absorbance between 0.253 - 0.361. Figure 1 shows that the lower the temperature used, the higher the absorbance value. There was a decrease in the absorbance level of the treatment using high temperatures, even the absorbance of pigments at a temperature of 80°C had a color change, indicating anthocyanin damage. This shows that the purple sweet potato color extract is more stable at lower temperatures than at high temperatures. The absorbance value at 80°C decreased significantly indicating that the effect of color stability on the temperature of the anthocyanin pigment solution had no significant effect on the pH produced.

#### *Color Stability against pH*

Based on the results of the research on the stability of red color from purple sweet potato pH 3,4,5,6,7,8,9 has an absorbance between 0.16 - 0.36 with wavelength  $\lambda$ . The statistical test in Figure 2 shows that the effect of color stability on the pH of the anthocyanin pigment solution has no significant effect on the pH produced.

Figure 2 shows that the lower the pH value, the higher the absorbance value. There was a decrease in the stability of the color extract against the pH treatment, even the absorbance of the pigment at pH 5 had a color change, indicating anthocyanin damage. This shows that the color extract of purple sweet potato is more stable in acidic pH than at alkaline pH. pH 5 and above causes damage to the anthocyanin pigment which color changes to colorless (color bleaching occurs) this is consistent with the research of Hanum (2000)<sup>16</sup>, that the condition of black glutinous rice concentrate at pH 5.5 shows a greater decrease in pigment levels or at least stable compared to the pH conditions below which are pH 3.5 and 4.5.

#### *Best Treatment Determination*

The best treatment is determined based on observational criteria which include brightness intensity with L \* value, red intensity with a \* value, yellow intensity with value b \*, water content, yield, pH, particle size and anthocyanin concentration. The matrix for determining the best treatment of anthocyanin pigments from purple sweet potato solution can be seen in Table 8.

Based on Table 8, the determination of the best treatment based on color analysis is seen from the treatment which gives a significantly different effect on the values of L \* and a \*. The higher the L \* value, the brighter the color produced, and the higher the value of a \* the resulting color is getting red, and the smaller the value of b \* the more color it produces. The results of the statistical test showed that all treatments gave significantly different effects on the value of L \*, the value of a \* and the value of b \* anthocyanin pigments. Therefore the treatment of

ultrasound duration for 120 minutes is included in the best treatment. Determination of the best treatment based on water content was determined based on the standard of the water content of the powder product. The results showed that the treatment of ultrasound duration for 120 minutes resulted in water content of 85.39%, total anthocyanin 185.20 mg / 100 g, pH 2.93.

Based on table 9, the anthocyanin concentration was determined based on the highest concentration of anthocyanin pigments. The observations showed that the 120-minute ultrasound extraction treatment produced the highest total anthocyanin of 185.20 mg / 100g. Therefore, the treatment of ultrasound extraction treatment for 120 minutes was included in the best treatment.

The pH value of anthocyanin pigments is determined based on pH which is in accordance with anthocyanin stability standards. The results of the observation showed that all treatments gave no significant effect. The pH range of encapsulated anthocyanin pigments ranges from 3.01-3.04. This value is in accordance with the anthocyanin pH standard, which ranges from 2-3. Therefore, all treatments are included in the best treatment.

The yield was determined based on the highest yield of the encapsulated anthocyanin pigment. The results of the observation showed that the treatment of ultrasound extraction for 30 minutes produced the highest yield of 31.04%. Therefore, the treatment of ultrasound extraction for 30 minutes is included in the best treatment because it can increase the economic value if the purple sweet potato anthocyanin pigment is commercialized.

## CONCLUSION

That the duration of ultrasound extraction method had a significantly different effect on each anthocyanin total, but did not have a significant effect on pH, yield, moisture content. At this stage the best method of ultrasound duration is 120 minutes of ultrasound with the highest total anthocyanin, antioxidant activity, and color.

## ACKNOWLEDGEMENTS

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## CONFLICT OF INTEREST

We have no conflict of interest.

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