



Microencapsulation of Anthocyanin Extract from Roselle (*Hibiscus sabdariffa* L.) Calyx using the Spray Drying Method

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Abstract— Roselle (*Hibiscus sabdariffa* L.) is one of the plants that is rich in anthocyanins. Anthocyanin pigments are usually unstable; therefore, one method to increase their stability is microencapsulation using various coating agents. This research aimed to determine the optimal ratio of maltodextrin and whey protein isolate (WPI) as a coating agent for producing roselle extract microcapsules and to assess the effects of pH and temperature on microcapsule stability. Dried roselle calyx was initially extracted using combinations of ethanol, distilled water, and citric acid. The roselle extract had a yield of 73.48%, a moisture content of 39.44%, a pH of 2.53, a total anthocyanin content of 38.85 mg/L, and a color intensity with a wavelength peak at 522 nm and an absorbance of 2.414. The roselle extract was then encapsulated using maltodextrin and whey protein isolate as coating agents at different ratios (1:0, 1:3, 1:1, 3:1, and 1:0) using the spray drying method. The chosen ratio of maltodextrin and whey protein isolate was 1:0, with color intensity (absorbance at 520 nm) of 0.453 ± 0.01 , yield of $66.60 \pm 4.04\%$, moisture content of $11.04 \pm 0.56\%$, solubility of $81.40 \pm 5.39\%$, total anthocyanin content of 10.33 ± 0.25 mg/L, and encapsulation efficiency of $92.12 \pm 2.63\%$. Furthermore, the stability of the selected microcapsules at different pH (2, 4, 6, 8, and 10) and temperatures (25°C, 40°C, 55°C, 70°C, and 85°C) was assessed based on total anthocyanin content and color intensity. Results showed that the highest stability of roselle extract microcapsules was obtained at pH 2-6 and temperature below 70°C.

Keywords— maltodextrin; microencapsulation; roselle extract; spray drying; whey protein isolate

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I. INTRODUCTION

Color is one of the important factors in consumer interest in food products. The use of synthetic colorants is relatively common in food products because they are more stable than natural colorants. In addition to stability, synthetic colorants tend to have higher color intensity and uniformity, and lower cost [1]. However, the use of synthetic colorants in food products can cause adverse effects on human health because they have the potential to be carcinogenic and allergenic, therefore, for long-term consumption, they might not be safe [2]. Along with increasing public awareness of health, the use of natural colorants is believed to be safer for health. One natural pigment with potential as an alternative to synthetic colorants is anthocyanin. Anthocyanins are water-soluble flavonoids that possess attractive colors ranging from blue, red, and purple, which can be found in flowers, leaves, and fruits [3]. Anthocyanins are also known for their potential health benefits, such as their use as an antidiabetic agent and their role

in preventing cardiovascular diseases and neurological disorders [4].

One of the plants known to be rich in anthocyanins is roselle (*Hibiscus sabdariffa*). Roselle is a flower that has been cultivated and widely distributed worldwide, including Indonesia. Anthocyanins from roselle calyx can be extracted using solvents to yield a concentrated extract [5]. Previous studies have reported that roselle calyx contains higher amounts of anthocyanin compared to cabbage (*Brassica oleracea*), beets (*Beta vulgaris*) [6], Malabar melastome (*Melastoma malabathricum*), and sweet potatoes (*Ipomoea batatas*) [7]. However, the main disadvantage of anthocyanins is their low stability, which is influenced by pH, light, temperature, enzymes, formation of complexes with metal ions, and the presence of co-pigments [8,9]. Moreover, a previous study reported that aqueous or ethanolic anthocyanin extracts are more stable at low temperatures, in low light, and at low pH [10].

One method to improve the stability of anthocyanins is encapsulation [11]. Microencapsulation is carried out using a coating material that encapsulates the core material or active substance, thereby reducing degradation of the active compound [12]. Microencapsulation can be done using several methods, the most widely used method is spray drying. In spray drying, the core material is either dissolved or suspended in a polymer solution, which is then pumped to a nozzle and atomized into fine droplets. These droplets are vaporized upon contact with hot gas, leaving the core material encapsulated in the matrix and forming dry particles [13].

The type and ratio of coating agent are important factors that influence the physical and chemical properties of the microcapsules obtained. An ideal coating material should stabilize microcapsules, exhibit high encapsulation efficiency, be soluble, and protect the active substance. However, with all the criteria mentioned, it is difficult to achieve with a single coating agent; therefore, most studies use a combination of coating agents [14]. Therefore, a combination of coating agents for microencapsulation could compensate for the limitations of a single coating agent and improve product quality and stability [15].

Maltodextrin is commonly used in spray-drying encapsulation because it provides a substantial barrier that prevents oxidation and other external factors [16]. Previous studies have successfully encapsulated roselle anthocyanin extract using maltodextrin combined with different coating materials, such as gum Arabic [17], trehalose [13], and whey protein concentrate (WPC) [18]. The combination of maltodextrin and whey protein isolate for microencapsulation of roselle anthocyanin extract has not been investigated. Previous research reported that whey protein isolate can increase the stability of anthocyanins by forming hydrogen-bonded complexes [19]. A combination of whey protein isolate and starch has also been used for probiotic microencapsulation and incorporated into low-pH beverages [20]. However, the combination of maltodextrin and whey protein isolate has been applied to other extracts. Purple corn anthocyanin has been shown to enhance stability during high-temperature storage [21]. This combination has also been reported to effectively retain bioactive compounds in purple yams [22].

Furthermore, the stability of roselle extract microcapsules at different temperatures and pH values is essential to investigate, as the anthocyanin content is influenced by both. Therefore, this research was conducted to determine the optimal ratio of maltodextrin and whey protein isolate (WPI) as a coating material for producing roselle extract microcapsules and to assess the effects of pH and temperature on the stability of roselle extract microcapsules.

II. MATERIAL AND METHODS

A. Material

The materials used in this experiment were dried roselle calyx obtained from a farm in Magelang, Indonesia, maltodextrin obtained from the shop "Indoplant" (Yogyakarta, Indonesia), whey protein isolate "Provon 292", ethanol 96% (Merck, pa), distilled water, citric acid (Merck), potassium chloride (KCl) (Merck), sodium acetate (CH₃COONa) (Merck), sodium chloride (NaCl) (Merck), acetic acid (CH₃COOH) (Merck), and disodium hydrogen phosphate (Na₂HPO₄) (Merck).

B. Methods

Extraction of Anthocyanin Pigment

The extraction process started by crushing the dried roselle calyx with an herb grinder (Fomac) and sieving with a 35-mesh sieve to reduce the size of the material. Afterwards, the maceration process was conducted with a combination of distilled water and 96% ethanol (50:50), with the addition of 2% citric acid. The ratio of roselle and solvent used is 1:6 (w/v).

The maceration process lasted 24 hours. The first 2 hours of maceration were performed with a shaker at 195 rpm and room temperature (25 °C). Next, the mixture was stored in a dark room for 22 hours at 25°C. During maceration, the mixture in the Erlenmeyer flask was covered with aluminum foil. After 24 hours, the extract was filtered through Whatman no. 1 filter paper using a Buchner vacuum pump. The filtrate was then concentrated to remove the solvent using a rotary evaporator at 60°C under 200 mbar [23,24]. The concentrated roselle extract was analyzed for its yield [25], moisture content [25], total anthocyanin content [23], [26], color intensity [27], and pH [28].

Research Stage I

The first research stage was conducted to determine the optimal ratio of maltodextrin and WPI for coating roselle extract microcapsules. Firstly, the coating material, i.e., maltodextrin and WPI, was mixed in the ratio of 0:1, 1:3, 1:1, 3:1, and 1:0. The roselle concentrated extract was then mixed with the coating material at a ratio of 1:3. These mixtures were then dissolved in 200 mL of distilled water using a magnetic stirrer for 10 minutes at room temperature. Afterwards, the feed solution was fed to the spray dryer (Buchi) at an inlet temperature of 150°C and a pressure of 1 bar [29,30].

TABLE 1
 FORMULATION OF THE FEED SOLUTION

Composition	Ratio of coating material				
	0:1	1:3	1:1	3:1	1:0
Maltodextrin (g)	0	3.75	7.5	11.25	0
Whey Protein Isolate (g)	15	11.25	7.5	3.75	15
Roselle concentrated extract (g)	5	5	5	5	5
Distilled water (mL)	200	200	200	200	200

The formulation of the feed solution is shown in **Table 1**. The microencapsulated roselle extract was analyzed for its total anthocyanin content [23], [26], yield [25], moisture content [25], solubility in water [31], color intensity [27], and encapsulation efficiency [26]

Research Stage II

The second research stage was conducted to determine the effects of pH and temperature on the stability of the selected roselle extract microcapsule from the first-stage research. The pH values used in this experiment were 2, 4, 6, 8, and 10, while the temperatures were 25°C, 40°C, 55°C, 70°C, and 85°C.

About 0.1 g of selected roselle extract microcapsules was dissolved in 10 ml distilled water. The solution was heated for 1 hour according to the heating treatment. The pH stability test was performed by dissolving the selected roselle extract microcapsule in a buffer solution at various pH values [32]. The stability of the extract microcapsule at different temperatures and pH was determined by analyzing color intensity [27] and total anthocyanin [23], [26].

Moisture Content Analysis [25]

Moisture content was analyzed using the oven-drying method. About 2 grams of the sample were placed on the evaporating dish and dried in the oven at 105°C for 6 hours. Afterwards, the sample was placed on the desiccator for 15 minutes. The sample was weighed until it reached a constant weight. The formula for moisture content measurement is as follows:

$$\text{Moisture Content}(\%) = \frac{D-(E-C)}{D} \times 100\% \quad \text{eq (1)}$$

Where:

C = Weight of empty evaporating dish after drying (g)

D = Weight of sample before drying (g)

E = Weight of evaporating dish + sample after drying (g)

Total Anthocyanin Content Analysis [23], [26]

Determination of anthocyanin concentration was performed using the pH-differential method. About 0.1 gram of microcapsules was weighed and mixed with 1 ml of distilled water. Afterwards, 10 ml of 96% ethanol was added to extract the anthocyanin. The filtrate was collected and analyzed. Two buffer solutions were used to dilute the solution, which are pH 1 and pH 4.5. The sample was placed into 10 ml of each buffer, and the absorbance of each sample was measured by a UV-Vis spectrophotometer at 520 nm and 700 nm, using distilled water as a blank. Total anthocyanin content (mg/L) expressed as cyanidin-3-glucoside, with the formula expressed below:

$$\text{Total Anthocyanin Content (mg/L)} = \frac{A \times MW \times DF \times 10^3}{\epsilon \times l} \quad \text{eq (2)}$$

Where:

A = $(A_{520\text{nm}} - A_{700\text{nm}})_{\text{pH } 1.0} - (A_{520\text{nm}} - A_{700\text{nm}})_{\text{pH } 4.5}$

MW (molecular weight) = 449.2 g/mol for cyanidin-3-glucoside

DF = dilution factor

ϵ = the molar absorptivity of cyanidin-3-glucoside (26,900 L mol⁻¹ cm⁻¹)

l = cell path length in (1 cm)

10³ = factor for conversion from g to mg

Solubility in Water Analysis [31]

The solubility test was performed by dissolving 0.1 g of microcapsules in 10 ml of water. After that, the solution was centrifuged at 3500 rpm for 10 minutes. About 4 ml of supernatant obtained was transferred to an evaporation dish and put in an oven at 100°C for 5 hours. The sample was weighed until it was constant, and the result was calculated with the formula below:

$$\text{Solubility}(\%) = 1 - \frac{C-B}{A} \times 100\% \quad \text{eq (3)}$$

Where:

A = Weight of initial sample (g)

B = Weight of empty evaporating dish (g)

C = Weight after drying (g)

Color Intensity [27]

Color intensity was determined by measuring absorbance on a UV-Vis spectrophotometer. About 0.1 gram of the sample was weighed and mixed with 10 ml of distilled water. To obtain the absorbance at the peak, the sample absorbance was measured at various wavelengths (400-700 nm).

Encapsulation Efficiency [26]

An encapsulation efficiency (EE) test was conducted to determine the effectiveness of the microencapsulation process. Encapsulation efficiency was calculated as the percentage of total anthocyanin content (TAC) relative to surface anthocyanin content (SAC) using the pH differential method. TAC was calculated as stated in subchapter 3.4.3. For SAC, 0.1 g of microcapsules was weighed and mixed with 10 ml of 96% ethanol. The solution was centrifuged for 10 minutes at 6000 rpm, and the supernatant was collected and analyzed. The encapsulation efficiency of the microcapsule was calculated by using the equation below:

$$EE(\%) = \left(\frac{TAC - SAC}{TAC} \right) \times 100 \quad \text{eq (4)}$$

Where:

TAC = Total Anthocyanin Content (mg/L)

SAC = Surface Anthocyanin Content (mg/L)

Experimental Design and Data Analysis

The experimental design used in the first and second research stages is a completely randomized one-factor design. For the first research stage, the factor was the ratio of maltodextrin and whey protein isolate as a coating material. The maltodextrin-to-whey protein isolate ratios were 0:1, 1:3, 2:2, 3:1, and 1:0. Each ratio was replicated 3 times, yielding a total of 15 samples. For the second research stage, the factors used were temperature and pH, analyzed separately as single factors. The temperatures used are 25°C, 40°C, 55°C, 70°C, and 85°C, meanwhile the pH

used are 2, 4, 6, 8, and 10. Each factor was also replicated three times, resulting in a total of 15 samples. The data obtained were processed using Microsoft Excel and statistically analyzed using One-Way ANOVA with a confidence level of 95% in SPSS software version 25.0. The post-hoc test used was the Duncan test.

III. RESULT AND DISCUSSION

Characteristics of Roselle Extract

The characteristics of the roselle extract in this research are shown in **Table 2**. Table 2 shows that, in general, the results obtained in this research were quite comparable to those of the previous study.

TABLE 2
 ANALYSIS RESULTS OF ROSELLE EXTRACT

Parameter	Amount	Previous study results
Yield (%)	73.48	75.10 [33]
Moisture Content (%)	39.44±0.25	-
pH	2.53±0.01	2-2.5 [34]
Total Anthocyanin Content (mg/L)	38.85±0.13	35.24 [35]
Color Intensity Peak (Wavelength (nm) and Absorbance)	522 nm, 2.414±0.01	526nm, 1.400 [27]

The pH of roselle extract obtained in this research was 2.53±0.01. Roselle extract is known for its sour taste and low pH value. The pH value is an important factor that affects the anthocyanin extraction. At lower pH, anthocyanin yield was the highest [36]. The main organic acids in roselle are citric acid, malic acid, and others, resulting in a low pH of approximately 2-2.5 [34].

The total anthocyanin content of roselle extract obtained in this research was 38.85±0.13mg/L. The level of extracted anthocyanins is affected by the solvents used [35]. In this research, the combination of ethanol and water was used for extraction because its polarity is similar to that of anthocyanins; thus, extraction with this combination is more effective [37]. The highest extraction efficiency was also reported in previous research using a combination of ethanol and water as the solvent [38]. Moreover, citric acid used in the extraction could increase the color intensity, as well as the yield and extraction efficiency [36].

The color intensity of the roselle extract was measured using a spectrophotometer at specific wavelengths and absorbance values. **Table 2** shows that the roselle extract obtained in this study exhibited the highest peak at 522 nm, with an absorbance of 2.414±0.01. The main anthocyanins present in roselle calyx are cyanidin (red pigment) and delphinidin (violet pigment) [39], with the maximum wavelength of anthocyanin found at around 530 nm [40]

Effect of Different Ratios of Maltodextrin and WPI on Characteristics of Microencapsulated Roselle Extract

Yield

The results of the statistical analysis in **Table 3** show that the ratio of maltodextrin to WPI had no significant effect ($p>0.05$) on the yield of microcapsules produced, with a range of 58.67-66.60%.

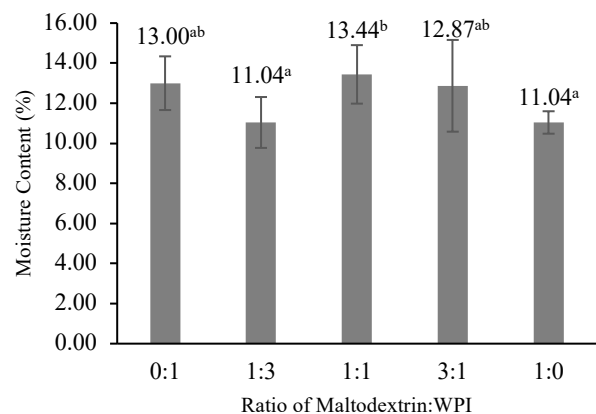
TABLE 3
 YIELD OF ROSELLE EXTRACT MICROCAPSULES

Ratio of Maltodextrin:WPI	Yield (%)
0:1	61.59 ± 5.73
1:3	60.94 ± 1.27
1:1	58.67 ± 8.61
1:3	60.83 ± 4.02
1:0	66.60 ± 4.04

This result is comparable to other previous studies using different coating agents for encapsulation. It was stated that microencapsulated roselle with various concentrations of maltodextrin as a coating agent had a yield of 56.46-72.75% [17]; meanwhile, a combination of maltodextrin:WPI for encapsulating pumpkin seed oil produced a yield of 65.6% [41].

Moisture Content

Moisture content analysis was carried out because it indicates the quality of microencapsulated powder and its storage stability [42]. Statistical analysis using One-Way ANOVA (**Figure 1**) showed that the ratio of maltodextrin and WPI had a significant effect ($p\leq 0.05$) on the moisture content of the roselle extract microcapsules produced.



Note: Different letter notation indicates a significant difference ($p\leq 0.05$)

Fig. 1 Effect of coating ratio on the moisture content of microencapsulated roselle extract

In general, spray-dried products have a moisture content of less than 5%. However, it is affected by spray-drying conditions, such as the types of carriers. Both maltodextrin and WPI are hydrophilic, making them readily bind to water molecules in the surrounding environment [43].

Furthermore, the moisture content of microencapsulated roselle extract obtained in this research was relatively higher compared

to previous studies on spray drying of roselle's anthocyanin extract using maltodextrin (6.5-12.1%) [26] or roselle calyx juice using maltodextrin (5.74-6.14%) [44].

Solubility in Water

Solubility is an important property of microcapsules; a high-quality powder is expected to have high solubility. Maltodextrin, as a coating agent, is known to have good water solubility, while WPI functions as an emulsifier that can help melt suspended substances in the aqueous phase. The use of WPI can accelerate the dissolution of microcapsules when applied to foodstuffs [45].

The results of the statistical analysis in **Table 4** showed that the ratio of maltodextrin to WPI had no significant effect ($p > 0.05$) on the solubility in water of the microcapsules produced, with a range of 78.63-84.30%.

TABLE 5
 SOLUBILITY IN WATER OF ROSELLE EXTRACT
 MICROCAPSULES

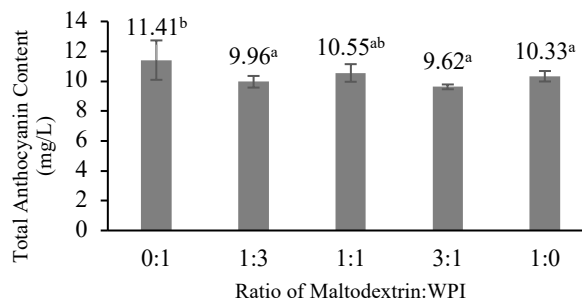
Ratio of Maltodextrin:WPI	Solubility in Water (%)
0:1	78.63±0.42
1:3	80.71±4.21
1:1	78.65±0.21
3:1	84.30±3.41
1:0	81.40±5.39

The solubility of roselle extract microcapsules in this research was higher compared to spray-dried lycopene-rich tomato extract using combination of maltodextrin, WPI, and modified starch (68.4-77.2%) [45], spray-dried roselle extract using combination of maltodextrin and xanthan gum (70%) [46], and comparable to red *melinjo* peel extract microcapsule using WPI (87.25%) [47]. However, the solubility is lower than that of roselle microcapsules with maltodextrin as a coating agent (93.71-97.85%) [26]. The lower solubility observed in this research compared to previous studies may be due to WPI denaturation during spray drying. Heat-induced aggregation has been reported to reduce solubility [48].

Total Anthocyanin Content

The statistical analysis results using One-Way ANOVA showed that the ratio of maltodextrin and WPI had a significant effect ($p \leq 0.05$) on the total anthocyanin content of the microcapsules produced. These results are shown in **Figure 2**. In general, **Figure 2** shows that the total anthocyanin content of the microcapsules is lower than that of the roselle extract without encapsulation (**Table 2**).

Furthermore, **Figure 2** shows that the microcapsule with WPI as the coating agent has the highest total anthocyanin content among the treatments, at 11.41 ± 1.05 mg/L. This result is supported by a previous study that reported that WPI could reduce anthocyanin loss under low-pH conditions [49].



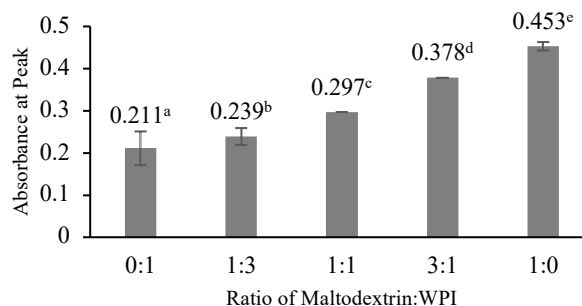
Note: Different letter notation indicates a significant difference ($p \leq 0.05$)
 Fig. 2 Effect of coating ratio on the total anthocyanin content of microencapsulated roselle extract

Statistically, in terms of total anthocyanin content, the use of WPI itself as a coating agent gave similar results to the combination of maltodextrin and WPI at a ratio of 1:1. This is because maltodextrin also provides a good protective effect towards the stability of anthocyanin pigment. The flavylium cation compounds in anthocyanin form a complex with carbohydrate from maltodextrin. Thus, making the anthocyanin more stable [50].

Compared with previous studies that encapsulated anthocyanins from roselle extract, the total anthocyanin content obtained in this research is lower, *i.e.*, 31.17 mg/L [51] and 26.58 mg/L [52]. Both studies used maltodextrin as the coating agent. This might be due to the high maltodextrin concentration (above 20%) that was added during the encapsulation process [53].

Color Intensity

Based on spectrophotometric measurements, all treatments reached their maximum absorbance at 520 nm, the wavelength of the red pigment [54]. The statistical analysis results using One-Way ANOVA showed that the ratio of maltodextrin and WPI had a significant effect ($p \leq 0.05$) on the color intensity of the microcapsules produced. The absorbance at the peak and the results of the statistical analysis are shown in **Figure 3**.



Note: Different letter notation indicates a significant difference ($p \leq 0.05$)
 Fig. 3 Effect of coating ratio on the color intensity peak of microencapsulated roselle extract

Figure 3 shows that the highest color intensity was achieved with the roselle extract encapsulated using maltodextrin only.

This result is in accordance with the previous study, which stated that the highest color intensity in anthocyanin powder from red dragon fruit was obtained when maltodextrin was used as a coating agent, compared to when maltodextrin was combined with gum arabic [51].

When maltodextrin is combined with WPI, color intensity decreases. This might be because WPI has a yellowish-cream color, which affects the final color of roselle extract after microencapsulation. Moreover, a previous study reported that the use of maltodextrin in microencapsulated red cabbage extract increased the color intensity of the microcapsules, whereas WPI decreased it [32].

Encapsulation Efficiency

Encapsulation efficiency is an essential parameter for microcapsules because it indicates the coating material's ability to retain the encapsulated material [26]. Encapsulation efficiency is also positively correlated with the shelf life of anthocyanin microcapsules, and maltodextrin has been reported as one of the best encapsulating matrices for preserving anthocyanin [13]. The statistical analysis results using One-Way ANOVA showed that the ratio of maltodextrin to WPI had no significant effect ($p > 0.05$) on the encapsulation efficiency of the produced microcapsules. The result is shown in **Table 5**.

TABLE 5
 ENCAPSULATION EFFICIENCY OF ROSELLE EXTRACT
 MICROCAPSULES

Ratio of Maltodextrin:WPI	Encapsulation Efficiency (%)
0:1	95.32±1.69
1:3	99.07±0.69
1:1	95.42±3.62
1:3	94.92±3.10
1:0	92.12±2.36

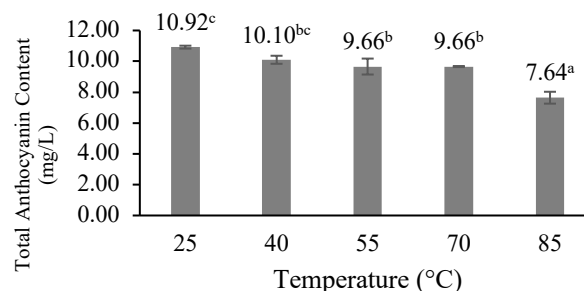
The results of microcapsules produced from all samples were high, indicating the coating agent was effective in encapsulating the anthocyanin from roselle extract. This result is comparable to a previous study, *i.e.*, anthocyanin of roselle extract encapsulated using combination of maltodextrin and gum Arabic (>96.7%) [26], butterfly pea flower (*Clitoria ternatea* L.) extract encapsulated using maltodextrin and WPI (90.07-94.14%) [55], and higher than roselle extract encapsulated using combination of maltodextrin and trehalose (66.65-76.53 %) [13].

Based on the analysis results, the maltodextrin:WPI combination of 1:0 was used for the research stage II. This combination yielded the highest color intensity and the lowest moisture content. The selected microcapsules were then analyzed in the following research stage to assess the effect of temperature on the stability of total anthocyanin content and color intensity.

Effect of Temperature on the Stability of Microcapsules

Total Anthocyanin Content

The statistical analysis results using One-Way ANOVA showed that the temperature had a significant effect ($p \leq 0.05$) on the total anthocyanin content of the microcapsules. This result is shown in **Figure 4**.



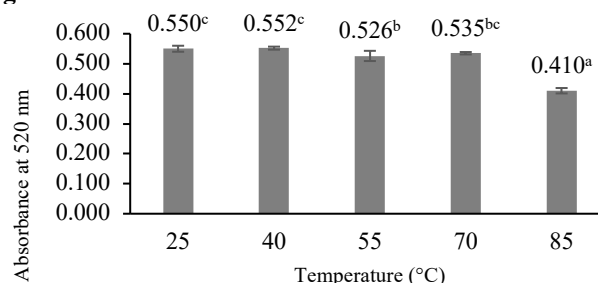
Note: Different letter notation indicates a significant difference ($p \leq 0.05$)
 Fig. 4 Effect of temperature on the total anthocyanin content stability of microencapsulated roselle extract

Figure 4 shows that higher temperatures decrease anthocyanin content, starting at 40 °C. However, the decrease in total anthocyanin content remained stable between 40 and 70 °C. A significant difference was seen after heating at 85°C. Thermal degradation of anthocyanin extract occurs above 80 °C [5].

High temperatures cause anthocyanins to break down. As the temperature increases, the anthocyanin structure is damaged. The hydrolysis of the glycosidic bonds of anthocyanin happened at position 3, resulting in the production of labile aglycones. The aglycone rings then open, forming a colorless carbinol and a chalcone group [23]. However, previous research also reported that maltodextrin was the most effective encapsulating material in improving the stability of spray-dried *camu-camu* (*Myrciaria dubia* (H.B.K.) McVaugh) extract [5].

Color Intensity

The statistical analysis results using One-Way ANOVA showed that the temperature had a significant effect ($p \leq 0.05$) on the color intensity of the microcapsules. This result is shown in **Figure 5**.



Note: Different letter notation indicates a significant difference ($p \leq 0.05$)
 Fig. 5 Effect of temperature on the color intensity (absorbance at 520 nm) of microencapsulated roselle extract

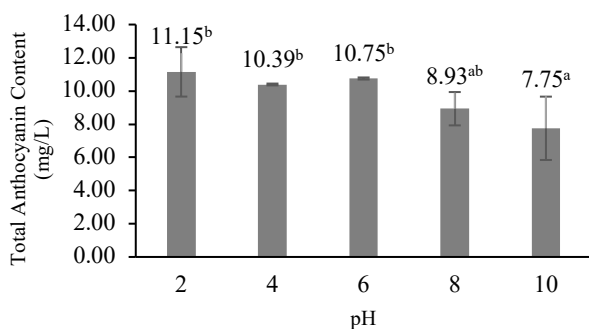
Figure 5 shows that, in the temperature ranges 25-40°C and 55-70°C, color intensity was not significantly different. However, at 85°C, the absorbance decreases significantly. High temperatures cause decomposition and structural changes in anthocyanins, leading to discoloration [13]. The findings of this research are consistent with previous research, which demonstrated that the thermal degradation of anthocyanins occurs at approximately 80°C, as evidenced by a decline in their absorbance [5].

Based on **Figure 4**, which shows that at 85 °C, total anthocyanin content also decreases significantly. This shows that the hydrolysis of anthocyanin is the primary cause of pigment color loss, resulting in lower absorbance observed.

Effect of pH on the Stability of Microcapsules

Total Anthocyanin Content

The statistical analysis results using One-Way ANOVA showed that pH had a significant effect ($p \leq 0.05$) on the total anthocyanin content of the microcapsules. This result is shown in **Figure 6**.



Note: Different letter notation indicates a significant difference ($p \leq 0.05$)
 Fig. 6 Effect of pH on the total anthocyanin content stability of microencapsulated roselle extract

Figure 6 shows that total anthocyanin content was stable at pH 2-6 and started to decrease at pH 8-10. The anthocyanin from roselle is more stable at acidic pH (<5.0) and unstable at alkaline pH[5]. This result is also consistent with previous studies, which reported that roselle extract encapsulated in a maltodextrin and trehalose matrix is stable at pH 2.6-5 [13]. At acidic pH, the structure of anthocyanin is in the flavylium cation form, which is red in color and highly stable at low pH [27], [36].

Color Intensity

To observe color changes, indicated by shifts in peak absorbance wavelength, the absorbance of microencapsulated roselle extract at different pH levels was measured over 400-700 nm. This result is shown in **Figure 7**. Moreover, the statistical analysis results using One-Way ANOVA showed that pH had a significant effect ($p \leq 0.05$) on the color intensity of the microcapsules. This result is shown in **Figure 8**.

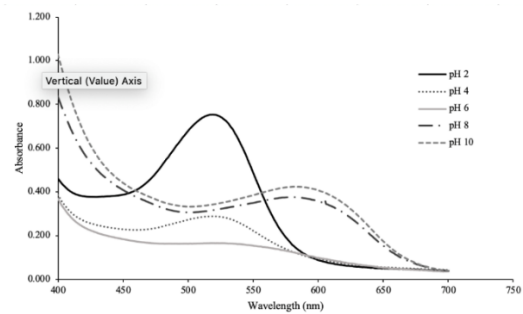
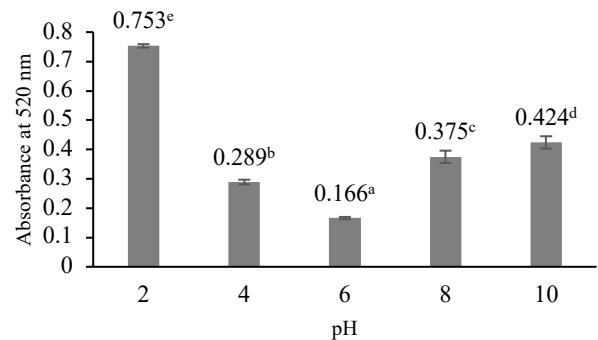


Fig. 7 Effect of pH on the wavelength at peak absorbance of microencapsulated roselle extract



Note: Different letter notation indicates a significant difference ($p \leq 0.05$)

Fig. 8 Effect of pH on the color intensity (absorbance at 520 nm) of microencapsulated roselle extract

Figure 7 shows that the pH values of roselle extract microcapsules at 2, 4, and 6 have the highest absorbance at 520 nm, which is the wavelength of anthocyanin. This also shows that the samples at pH 2, 4, and 6 are still red in color; however, the absorbance decreased as the pH increased, as shown in **Figure 8**. Meanwhile, at pH 8 and 10, roselle extract microcapsules exhibit the highest absorbance at 582 nm, because the microcapsules change to a more yellowish-green color.

This result is consistent with a previous study, which reported that pH changes cause a transformation in the chemical structure of anthocyanin. At pH 2-3, anthocyanins are mainly present as yellow salt ions, resulting in a red color. At pH 4-6, their structures gradually transform into quinoid, resulting in a decrease in red color. At pH 7-9, their structures transform into colorless pseudo-bases, and the color gradually turns blue. When the pH is greater than 9, anthocyanins degrade in a strongly alkaline environment, and the color changes to yellow-green [56].

IV. CONCLUSION

The best ratio of maltodextrin to whey protein isolate as a coating agent for roselle extract microencapsulation using the spray-drying method was 1:0, resulting in the highest color intensity and yield. The best roselle microcapsules have a solubility of $81.40 \pm 5.39\%$, a total anthocyanin content of

10.33 ± 0.25 mg/L, and an encapsulation efficiency of 92.12 ± 2.63%.

Temperature and pH affected the stability of the microcapsules, expressed in total anthocyanin content and color intensity. The roselle extract microcapsules have the highest stability at temperatures lower than 70°C and a pH range of 2-6.

Even though this research shows that maltodextrin is the most effective coating material for encapsulating anthocyanins from roselle extract, the stability of the microcapsules under other conditions, such as exposure to light and variations in storage time, should be further investigated. With more comprehensive data, the scale-up process for producing roselle extract microcapsules could be more feasible for future applications.

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CONFLICT OF INTEREST

The authors declare no conflict of interest to disclose.

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