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Formulation of Low-Calorie Roselle Syrup Using Sorbitol and Carboxymethyl Cellulose

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ABSTRACT

Roselle flowers can be used as an ingredient in herbal drinks with health benefits. Roselle flowers contain several active components, such as anthocyanins, flavonoids, phenolic acids, and some organic acids. Roselle flowers have health benefits such as anti-hypertensive, anti-diabetic, and diuretic. In addition, roselle flowers also function as a sour taste and natural dye. The use of roselle flowers as a low-calorie syrup product has the potential to be developed. A sweetener (sorbitol) and thickening agent (Carboxymethyl Cellulose) can be applied to this low-calorie syrup product. Sorbitol is a sugar alcohol (polyol) that contains low calories of 2.6 kcal/g with a sweetness level of 0,6 times compared of sucrose. CMC is water soluble, colorless, non-toxic, and acts as a thickener, stabilizer, and water binder. This study aims to determine the best formulation of CMC based on physicochemical and sensory characteristics of low-calorie roselle syrup. Ratio of sorbitol and water used was 1:1. The addition of CMC increased the total dissolved solids, viscosity, and acidity and decreased the brightness and redness of roselle syrup. With the addition of sorbitol and 0.5% CMC thickener, rosella syrup produced the highest score on the sensory test (overall), which was 3,8 out of 5 (like). This formula produced viscosity (19.33 cP), total dissolved solids (33 .68), lightness value (3.46), a*value (8.06), pH (3.00), antioxidant activity (21.51%), and glucose level (11.07 mg/ml).

Keywords : syrup, roselle, *Carboxymethyl Cellulose*.

1. Introduction

Syrup is one of the sweetened drinks whose most significant component is a sweetener. It has a viscous and concentrated form, so it must be diluted in water first before consumption. The main syrup-making ingredients are sweeteners and solvents, but they can be added with thickeners, preservatives, essences (aroma givers), and dyes (Wulandari et al., 2018). Syrup has a high sugar content of at least 65%, commonly consumed by children to adolescents, while the elderly and diabetics reduce or even do not consume sugary drinks. Excess consumption of sugary drinks can lead to dental caries, obesity, and even provoke diabetic diseases. Diabetes can be triggered due to a lack of physical activity and an unattended diet, such as consuming sugary foods and drinks.

One of the plants that can act as an antidiabetic in syrup is rosella extract produced from rosella petals (*Hibiscus Sabdariffa* L.). The plant is known to cure hypertension, diabetes, and diuretics because it contains carbohydrates, sucrose, proteins, fats, polyphenols of the anthocyanin group, alkaloids, flavonoids, phenolic acids, and some organic acids on its petals (Carvajal-Zarrabal et al., 2009; Mahadevan et al., 2009; Amperawati et al., 2019; Yuniati et al., 2021). This is evidenced by the research of Andraini & Yolanda (2014) on mice affected by diabetes, and rosella can work as antidiabetics by lowering blood glucose levels, increasing insulin secretion, and increasing insulin secretion insulin resistance. In addition, rosella extract functions as a red dye as well as a giver of sour taste and flavor, such as tea leaves (Ananta et al., 2019). Anthocyanin compounds in rosella are used as antioxidants and purplish-red dyes that are water-soluble, unstable to heat, and have a sour taste (Boas et al., 2014, Amperawati et al., 2019). In processing and storing products, anthocyanins can experience a decrease caused by temperature, pH, light, oxygen, enzymes, and aqueous solutions (Laleh et al., 2006; Mashudi, 2012; Fernandes et al., 2014).

One of the forming components of the viscosity of syrup is a thickener. Thickeners play an important role in increasing viscosity and providing stability to syrup. CMC is widely used in the food industry in the form of sodium carboxymethyl cellulose salt which can function as a water binder, thickener, and stabilizer (Kamal, 2010). CMC is hydrophilic, inert, soluble in cold water and hot water but insoluble in organic solutions (alcohol, ethyl, acetate, esters, ketones, ethers, and others), stable to fats, has no color, no odor, is non-toxic and resistant to hot temperatures (>300°C) (Kamal, 2010). The stability of CMC exists in the range of pH 7-8.5, if the pH is too low (< 3) it can cause the viscosity to increase and a precipitate is formed whereas, if the pH is > 10 the viscosity of the syrup will decrease slightly (Imeson, 1992). CMC is widely used in various industrial fields because it is easy to get, the price is relatively cheap, it is easily soluble in water. This study aims to determine the effect of thickener concentration levels on physical, chemical, and sensory characteristics of rosella syrup, as well as determine the best formulation of rosella syrup based on sensory characteristics.

2. Methods

Formulation of Roselle Syrup

The main ingredient used is rosella flowers. First of all, fresh rosella flowers are separated from the seeds so that fresh rosella petals are obtained. Then, prepare the pot and pour water, sorbitol, salt as well as a CMC thickener into it. Then, it is heated to a temperature of up to 100 ^{o}C and the stove is turned off. Fresh rosella petals are inserted and stirred continuously for 15 minutes. After finishing stirring, filtering is carried out so that rosella syrup is produced. Then, rosella syrup is put in bottles that are pre-sterilized with the oven at a temperature of 121 ^{o}C for 15 minutes. Formulation of roselle syrup can be seen in Table 1. In this study, CMC thickening agents with a concentration of 0%, 0.5%, 1%, 1.5% were used. Storage is carried out in glass bottles and stored at room temperature.

Tabel 1. Formulation of Rosella Syrup

Ingredients	**Formulation**			
	F1	**F2**	**F3**	**F4**
Water (g)	125	125	125	125
Sorbitol (g)	125	125	125	125
CMC (%)	0	0,5	1	1,5
Salt (g)	0,125	0,125	0,125	0,125
Rosella flower (g)	50	50	50	50

Physicochemical Analyses

Physicochemical analyses of roselle syrup measured were total dissolved solid, viscosity, colour intensity, pH value, antioxidant activity, and glucose level. Total dissolved solids show water-soluble components such as glucose, sucrose, fructose, organic acids, and water-soluble proteins (Farikha et al., 2013). This analysis was performed using a digital refractometer with the total result of dissolved solids units of °brix.

Color measurements on the product are carried out to determine the influence of the thickener concentration on the syrup. The instrument used is a chromameter that measures the color of the sample on the surface. The results of this color measurement are brightness/lightness (L*), redness/redness (a*), and yellowness(b*). Brightness has a maximum value of 100 and a minimum value of 0. The larger the value indicates that the sample has a higher brightness level and vice versa. The color intensity of the positive a* value indicates the sample has a color in the red direction, and the negative indicates the color toward green. Meanwhile, a positive b* value indicates a color of yellow and a negative b* shows a color of blue (Sinaga, 2019).

pH value analysis is carried out to measure the degree of acidity or alkalinity of a solution using a pH meter instrument. In the pH meter, there are two electrodes, namely a glass electrode and a reference electrode, where at the end of the glass electrode, there is a bulb that functions as a place for positive ion exchange (H^+), which can cause potential differences between the two electrodes. However, if the difference in the potential of the glass electrode is more favorable than the reference electrode, the pH of <7 means that the solution is acidic and vice versa.

Antioxidant activity can be analyzed by the DPPH method (1,1-diphenyl-2-picrylhydrazil), a free radical compound, when mixed with the DPPH solution. The antioxidant compounds from rosella will bind to DPPH* (diphenylpicrylhydrazyl) so that it becomes DPPH-H (diphenylpicrylhydrazine). The process changes color from dark purple to pale yellow, which means free radical compounds are reduced by antioxidant compounds (Mohamed, 2016).

The analysis of glucose levels using the phenol-sulfuric acid method uses an 80% solution of phenols and sulfuric acid. This method is often used for testing carbohydrates expressed in percent glucose. The syrup is first diluted in water. The dilution results are added an 80% phenol solution, and then a sulfuric acid solution is added. The sample that was reddish turned yellowish. Samples containing oligosaccharides or polysaccharides will undergo hydrolysis by sulfuric acid into monosaccharides (glucose). Monosaccharides will react with phenols and sulfuric acid. It produces heat that can hydrate glucose into hydroxymethyl furfural compounds. The compounds will react with phenols to have an orange color yellowish (Nielsen, 2009).

Afterward, the sample was cooled in a bath at 25°C for 10 minutes for the solution to stabilize. Then an absorption measurement is carried out at a wavelength of 490 nm.

3. Results and Discussion

3.1. Physical Characteristics

3.1.1. Total Dissolved Solid

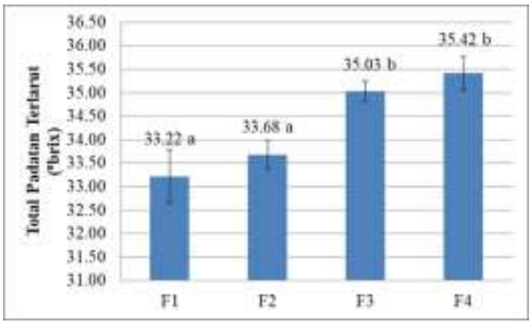

Figure 1. Total Dissolve Solid of Roselle Syrup

The digital refractometer works with the principle of refraction of light by passing light into the solution. The more solutes in the syrup, the greater the refractive index to produce higher °brix. Comprehensive testing of dissolved solids on rosella syrup, resulting in values ranging from 32.22-35.42 °brix. In the four samples, there was an increase in total dissolved solids as the CMC concentration increased. It happens because CMC is a polysaccharide with a hydroxyl group (OH), so it can bind to components more strongly and cause the syrup to stabilize. It increases the total dissolved solids in the syrup and the concentration of thickeners (Fahrul et al., 2020). In addition, sorbitol can also increase the concentration of soluble material, especially in F1 samples (without adding thickeners). It is because sorbitol is hydrophilic and contains a hydroxyl group (OH) that can bind free water so that the soluble components increase the viscosity of a solution (Suryadri et al., 2020). Rosella flowers contain several organic acids, such as citric acid and malic acid, which can dissolve, thus increasing the total dissolved solids (Yuniati et al., 2021).

3.1.2. Viscosity

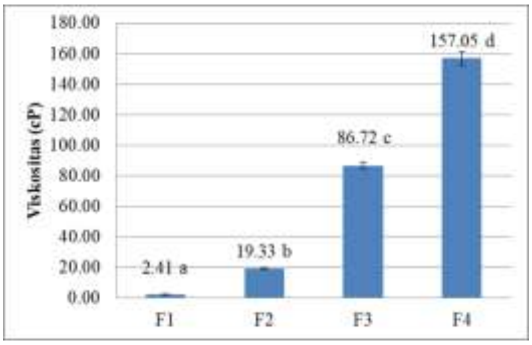

Figure 2. Viscosity of roselle syrup

The viscosity of the four syrup samples showed a significant difference, where syrup samples with the addition of CMC thickeners could increase viscosity. The syrup that has the lowest viscosity is rosella syrup without the addition of CMC (F1), which is 2.41 ± 0.45 cP, followed by an F2 sample (rosella syrup with the addition of CMC 0.5%), F3 (rosella syrup with the addition of CMC 1%), and the highest viscosity with the addition of CMC 1.5% (F4) of 157.05 ± 4.35 cP. It is in accordance with CMC, which is hydrophilic and can form crosslinks with solvent molecules to form a structure that is increasingly viscous to rigid (Kamal, 2010). There is an interaction between CMC and water, where sodium (Na+) in water will be released and replaced by hydrogen (H+), thus forming HCMC and resulting in increased viscosity (Bochek et al., 2002). Meanwhile, for F1 samples, the viscosity of the syrup is obtained due to the presence of hydrophilic sorbitol so that it can bind free water around it; hence the viscosity increases.

3.1.3. Color intensity

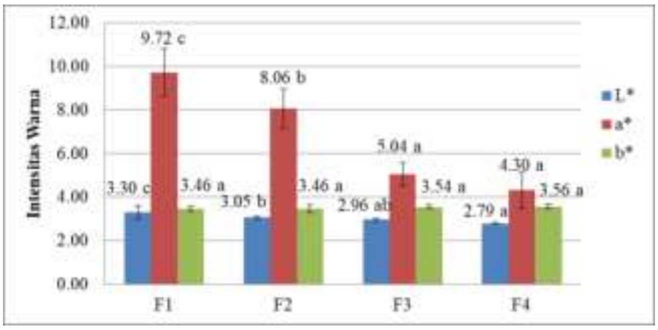

Figure 3. Color intensity of roselle syrup

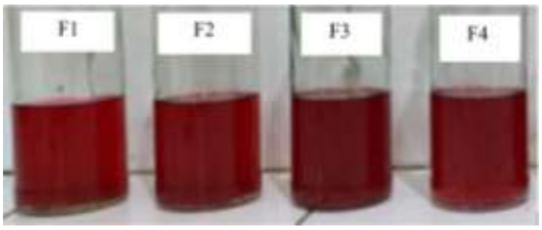

Figure 3. Roselle syrup

The highest brightness level (L*) of rosella syrup was produced by F1 (rosella syrup without thickeners) of 3.3 ± 0.3, and the syrup sample that had the lowest brightness was F4 (rosella syrup with a 1.5 CMC thickener) of 2.79 ± 0.04. The addition of a CMC thickener can result in further decreased brightness. This follows Deviarni and Warastuti (2017), which state that the sugar (glucose) content contained in CMC when there is heating, then caramelization will occur, causing the color of the sample to get darker or cloudier.

The color intensity of the lowest a* value of rosella syrup was at F4 (rosella syrup with a 1.5% CMC thickener) of 4.3 ± 0.83 and the highest a* value of 9.72 ± 1.07 in F1 (rosella syrup without thickeners). Adding a CMC thickener causes the a* value to get lower, which means the syrup has a darker red color. The red color in rosella syrup is caused by anthocyanins in roselle, which are water-soluble pigments (Amperawati et al., 2019). Meanwhile, the red color is getting darker because CMC contains glucose which, when heated, is caramelized so that the color of the syrup is darker (Deviarni and Warastuti, 2017). In addition, CMC works as a thickener that can bind various components, one of which is anthocyanins (Fahrul et al., 2020). With the increase of CMC, more anthocyanin compounds are bound, so the syrup gets redder. For color intensity, the value of b* does not differ markedly at a 95% confidence level and has a stable value ranging from 3.46-3.56.

3.2. Chemical Characteristic

3.2.1. pH Value

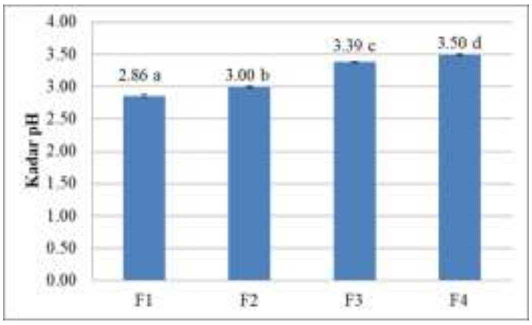

Figure 4. pH Values of Roselle Syrup

The results of the pH values in the four samples were significantly different, the sample that had the lowest pH value was rosella syrup without thickeners (F1) of 2.86 ± 0.02, and the highest pH value was rosella syrup with the addition of a 1.5% CMC thickener (F4) of 3.5 ± 0.02. It can be seen that the increase in the concentration of CMC thickeners resulted in an increased pH value. The increase in pH is due to CMC thickeners, where CMC processing is made through the alkalization and esterification stages, so it is alkaline (Latif et al., 2007). In addition, CMC also contains hydrocolloids that have a carboxyl group that is easily hydrolyzed to cause an increase in the pH value (Manoi, 2006).

3.2.2. Antioxidant Activity

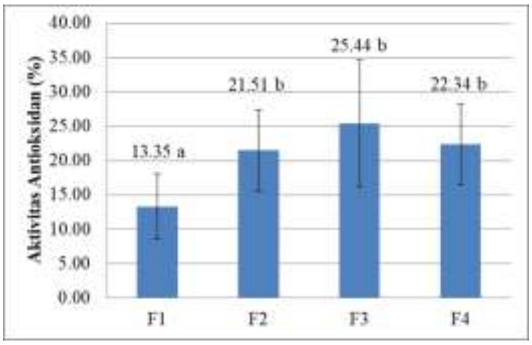

Figure 5. Antioxidant Activity

In the food industry, antioxidants are compounds used to extend the shelf life of a product because it can inhibit the formation of free radicals by donating electrons (Mohamed, 2016). Rosella contains antioxidant compounds consisting of anthocyanins, alkaloids, flavonoids, phenolic acids, citric acid, and malic acid (Carvajal-Zarrabal et al., 2009; Yuniati et al., 2021). One of the antioxidant compounds in the roselle that play a crucial role is anthocyanins, a group of polyphenols. Anthocyanins are proven to suppress oxidative stress from the effects of free radicals on insulin resistance and increase insulin resistance by reducing blood glucose and plasma insulin levels performed in experimental animals (Andraini & Yolanda, 2014; Sarbini et al., 2019).

The test results showed a significant difference between syrup samples with the addition of thickeners and without the addition of thickeners. Rosella syrup without the addition of CMC (F1) produces the lowest antioxidants at 13.35%, while rosella syrup with the addition of CMC produces antioxidants ranging from 21.51-25.44%. This indicates that using CMC thickeners can bind antioxidant compounds because CMC has a hydroxyl group, so the addition of CMC can bind components more strongly (Fahrul et al., 2020). However, there is no noticeable difference between antioxidant activity and the addition of CMC concentrations. The chart shows an increase in antioxidants from F2 by 21.51 ± 5.95% to 25.44 ± 9.25% in the F3 sample. Then there was a decrease in F4 to 22.34 ± 5.83%. This decrease in antioxidants is thought to be due to antioxidant compounds in rosella, especially anthocyanins, being unstable to pH, temperature, light, oxygen, enzymes, and aqueous solutions (Fernandes et al., 2014). Anthocyanin molecules can be damaged by an increase in pH, temperature, light exposure and storage at room temperature (Laleh et al., 2006; Mashudi, 2012). The stability of anthocyanins is related to several double bonds on their chemical structure that can be degraded through extraction, processing, and storage processes (Fernandes et al., 2014).

3.2.3. Glucose level

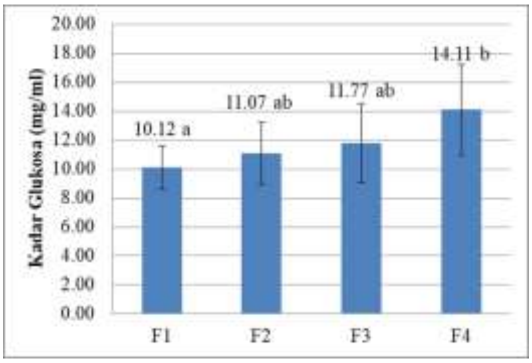

Figure 6. Glucose level of Roselle Syrup

The glucose levels of all samples showed no significant difference, however there was an increase in glucose levels along with the addition of CMC. The lowest glucose content is rosella F1 syrup with a yield of 10.12 ± 1.48 mg / ml and the highest glucose level in rosella F4 syrup of 14.11 ± 3.14 mg / ml. The increase in glucose levels might caused by CMC which has a polymer chain structure consisting of cellulose molecular units and has many glucose components, so that it can increase glucose levels in rosella syrup (Rahmaningtyas et al., 2016). In addition, the presence of sorbitol as a sweetener does not contribute glucose in syrup because sorbitol is a sugar alcohol that does not have a carbonyl group such as aldehydes, ketones, and carboxylic acids due to the process of making sorbitol from glucose that is hydrogenated with high pressure, sorbitol is not classified as reduction sugars such as monosaccharides (glucose, fructose, galactose) and disaccharides (lactose, maltose) (Suseno et al., 2008). On sample F1 (without the addition of thickeners),

3.3. Sensorical Analysis

Table 2. The results or sensorical analysis

	Color	Aroma	Sweetness	Sourness	Bitterness	*Overall*
F1	3,27 ± 0,45[a]	3,1 ± 0,61[a]	3,1 ± 0,71[a]	3,47 ± 0,68[a]	1,87 ± 0,63[a]	3,34 ± 1,20[a]
F2	3,37 ± 0,49[a]	3,03 ± 0,67[a]	3,13 ± 0,51[a]	3,53 ± 0,51[a]	1,97 ± 0,67[a]	3,80 ± 0,80[ab]
F3	3,53 ± 0,63[a]	3,07 ± 0,45[a]	3,2 ± 0,76[a]	3,63 ± 0,76[a]	1,73 ± 0,64[a]	3,41 ± 0,80[a]
F4	3,6 ± 0,72[ab]	3,03 ± 0,49 [a]	3,4 ± 0,67 [a]	3,5 ± 0,73[a]	1,83 ± 0,75[a]	3,38 ± 1,19[a]

Keterangan :

- F1 = sirup rosella without addition of CMC
- F2 = Sirup rosella with the addition of CMC 0,5%
- F3 = Sirup rosella with the addition of CMC 1%
- F4 = Sirup rosella with the addition of CMC 1,5%

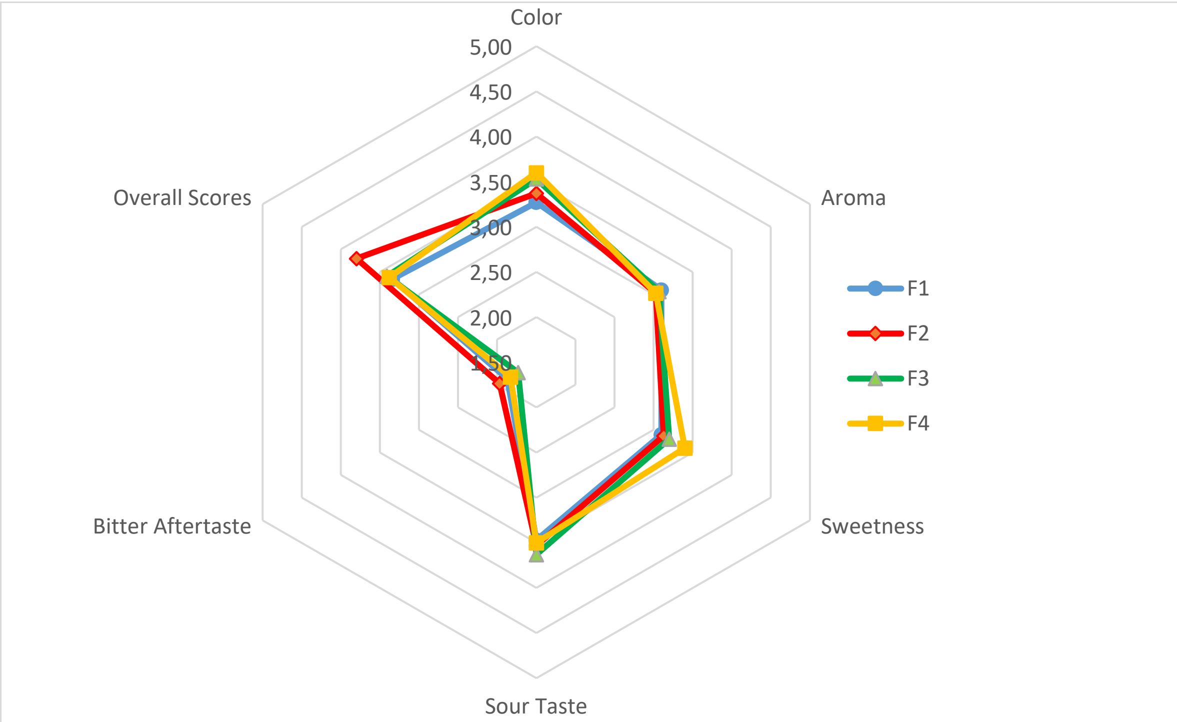

Figure 7. The Results of Sensorical Analysis

3.3.1. Color

The results of this descriptive test study showed that the treatment of CMC thickener concentrations had a significant effect on the color of rosella syrup. The four samples produced color values ranging from 3.27 - 3.6 which means they are slightly red in color. The red color in rosella syrup comes from the anthocyanin content in rosella flowers which is a water-soluble pigment that can produce a purplish-red color, as well as an antioxidant (Boas et al., 2014; Amperawati et al., 2019).

3.3.3. Aroma

The results showed that the treatment of CMC thickener concentrations had no significant effect on the aroma of rosella syrup. From the descriptive rating test of the aroma of rosella syrup, the average value ranges from 3.03 - 3.1, meaning that the syrup is slightly scented with rosella. Samples F2 (syrup with the addition of CMC 0.5%) and F4 (syrup with the addition of CMC 1.5%) had the lowest value, while the F1 sample (syrup without the addition of CMC thickener) had the highest value. Rosella has an acidic aroma derived from acidic compound components such as citric acid and malic acid (Yuniati et al., 2021). However, the aroma of rosella in the syrup only smells slightly sour. It is due to the mixing of water with the syrup to reduce the aroma of rosella.

3.3.2. Sweetness

Dari hasil uji deskriptif tidak ditunjukkan adanya perbedaannyata antara konsentrasi pengental CMC dengan rasa manis pada sirup. Pada keempat sampel dihasilkan nilai berkisar 3,1 - 3,4 yang berarti sirup memiliki rasa sedikit manis, dimana sampel F4 (penambahan pengental CMC sebanyak 1,5%) memiliki nilai paling tinggi. Sirup memiliki rasa sedikit manis dikarenakan gula yang dipakai adalah sorbitol yang memiliki tingkat kemanisan 60% dari sukrosa dan kalori sebanyak 1,6 kkal/g (National Center for Biotechnology Information, 2022). Selain itu, rasa manis juga disumbang oleh adanya pengental CMC yang memiliki unit molekul selulosa pada strukturnya, dimana selulosa merupakan polisakarida yang memiliki banyak komponen glukosa (Rahmaningtyas *et al*., 2016).

3.3.3. Sour Taste

From the results of descriptive tests, there is no significant difference between the concentration of CMC thickeners and the sweetness in syrup. In the four samples, values ranging from 3.1 - 3.4 were produced, which means that the syrup has a slightly sweet taste, whereas the F4 sample (adding a CMC thickener of 1.5%) has the highest value. The syrup has a slightly sweet taste because the sugar used is sorbitol which has a sweetness level of 60% of sucrose and calories as much as 1.6 kcal / g (National Center for Biotechnology Information, 2022). The sweet taste is also contributed by the presence of a CMC thickener with a cellulose molecular unit in its structure. In contrast, cellulose is a polysaccharide with many glucose components (Rahmaningtyas et al., 2016). The results showed no significant influence between the treatment of CMC thickener concentrations and the sour taste. The four samples produced values ranging from 3.47 - 3.63, meaning that the syrup tasted slightly sour. The average results of the panelists showed that the F3 sample (syrup with the addition of a CMC thickener of 1%) had the highest yield.

In contrast, the F1 sample (syrup without adding a CMC thickener) produced the lowest value. The sour taste in syrup is caused by citric acid, malic acid, tartaric acid, and oxalic acid in the rosella (Mahadevan et al., 2009; Yuniati et al., 2021). In addition, some anthocyanins give a refreshing sour taste and a red color to the syrup (Amperawati et al., 2019).

3.3.4. Bitter *Aftertaste*

The study results showed no significant difference between adding a concentration of CMC thickeners and a bitter aftertaste in rosella syrup. The average results of the panelists after bitter taste in rosella syrup ranges from 1.73 - 1.97, which means rosella syrup is not bitter. According to Purbowati et al. (2020), rosella has a high acid content that can cause a bitter taste. His research on rosella tea resulted in the fact that panelists did not favor rosella tea in a ratio of 10:100 to water because of its sour and bitter taste. The theory is not following the results of the study. This can be caused by differences in the manufacturing steps. In the research of Purbowati et al. (2020), the manufacturing step begins with drying and sifting rosella into a powder, then mixing it with water. This method can cause more compounds in rosella to dissolve more so that it produces high acids and causes a bitter taste. Meanwhile, making rosella syrup in this final project by stirring rosella flowers in hot conditions is filtered so that the soluble compounds are not optimal. Thus, the bitter aftertaste in rosella syrup is not felt but rather a more pronounced sour taste.

3.3.5. Overall Scores

Overall, the panelist assesses the overall attributes (color, aroma, sweet taste, sour taste, after-taste bitter). In the results obtained, it is known that the F2 sample, namely rosella syrup with the addition of a CMC thickener of 0.5%, produced the highest average value of 3.80 ± 0.80.

3.4. Correlation Among Parameters

Tabel 3. Correlation among Parameters (Pearson Correlation Coeffisient)

Parameter	Total Dissolved Solid	Lightness (L*)	a* Value	b* Value	Viscosity	pH value	Glucose Level	Antioxidant Activity
Total Dissolved Solid	1	-0,569**	-0,840**	0,347	0,893**	0,925**	0,440*	0,378
Lightness (L*)	-0,569**	1	0,683**	-0,105	-0,724**	-0,747**	-0,444*	-0,494*
a* Value	-0,840**	0,683**	1	-0,331	-0,887**	-0,933**	-0,564**	-0,406*
b* Value	0,347	-0,105	-0,331	1	0,327	0,332	-0,006	0,371
Viscosity	0,893**	-0,724**	-0,887**	0,327	1	0,958**	0,522**	0,368
pH Value	0,925**	-0,747**	-0,933**	0,332	0,958**	1	0,479*	0,466*
Glucose Level	0,440*	-0,444*	-0,564**	-0,006	0,522**	0,479*	1	0,047
Antioxidant Activity	0,378	-0,494*	-0,406*	0,371	0,368	0,466*	0,047	1

In the results of the correlation test, it was found that there was an inversely proportional relationship between color intensity (L* and a*) and total dissolved solids, viscosity, and antioxidant activity. The lower the L* and a* values in rosella syrup, the higher the total dissolved solids, viscosity, and antioxidant activity in the syrup. CMC thickeners have a hydroxyl group (OH) that can bind components more strongly and make the syrup stable (Fahrul et al., 2020). Things such causes the syrup to become thicker and the total solids dissolved in the syrup increase. Some of its components are anthocyanins, organic acids, water, carbohydrates, and proteins that break down into simpler compounds (Rahmaningtyas et al., 2016; Yuniati et al., 2021). More dissolved anthocyanin compounds produce a redder syrup color and increase antioxidant activity.

Meanwhile, the intensity of the colors L* and a* is directly proportional, which means the lower redness, the brightness level of rosella syrup also decreases. The red color in rosella syrup is obtained from the content of easily water-soluble anthocyanins (Amperawati et al., 2019). CMC thickeners can cause a decrease in color to become darker because CMC can bind various components, one of which is anthocyanins (Fahrul et al., 2020). So that the increasingly bound anthocyanin content causes the color of the syrup to become redder and the brightness level to decrease.

4. Conclusions

4 The effect of CMC thickeners on the physical and chemical characteristics of rosella syrup is an increase in total dissolved solids, viscosity, pH value and glucose levels. Still, it lowers the brightness and redness levels of rosella syrup.

Descriptive sensory tests show that rosella syrup has a slightly red color, a slightly smelled rosella aroma, a slightly sweet, slightly sour taste, and no bitter taste. Rosella syrup with the addition of a CMC thickener of 0.5% is the best formulation based on sensory tests (overalls) with an average value of 3.07 ± 0.64 (likes) and antioxidant activity of 21.51 ± 5.95%.

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