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Effervescent formulation based on variation of nanocapsules matrix type and roselle (*Hibiscus sabdariffa*) nanocapsules percentage

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Abstract

Roselle flowers are usually consumed in the form of fresh steeping. To increase the flexibility of using roselle, effervescent roselle-based products are made which of course more practical, efficient and attractive. Unfortunately, the bioactive compound in roselle extracts is not stable to environmental influences so nanoencapsulation technology needs to be done. The encapsulation matrix type gives a unique effect on each core. The purposes of this study were to determine the effects of variation in the matrix types and percentage of roselle nanocapsules on the physical and chemical effervescent properties. The research design used in this study was a Complete Random Design with 2 factors and 3 replications. The first factor comprised matrix types (A): maltodextrin (A1) and Arabic gum (A2). The second factor was roselle nanocapsule percentages (B): 10% (B1), 15% (B2) and 20% (B3). Observed variables in this study were total phenol, anthocyanin, vitamin C, antioxidant activity and dissolving time. The Arabic gum nano encapsulated matrix type gave a better value of Vitamin C, antioxidant activity and dissolving time than maltodextrin, namely 0.149±0.049 µg/3 g, 34.02±4.52%, 2.93±0.80 mins. The percentage of roselle nanocapsules by 20% gave the highest vitamin C value, namely 0.188±0.032 µg /3 g. The best treatment combination was obtained from the treatment (A2B3) Arabic gum nanocapsules using the third percentage (20%) with the characteristics: total phenol content 4.17 µg /3 g, anthocyanin 0.070 µg /3 g, vitamin C 0.188 µg /3 g when it dissolves in 1.25 mins.

1. Introduction

Roselle has been known as a fiber-producing plant. However, along with the increasing popularity of the slogan back to nature among the public, Rosella's prestige was also raised. The flexibility in using roselle is currently still limited considering the bioactive compounds in plants that have high added value. This is because the roselle raw material used is still in the form of powder and micro-scale extract encapsulation (Selim *et al*., 2004).

This bioactive content has resulted in roselle being widely used as traditional medicine in Southeast Asia (Tsai *et al*., 2002), food coloring (Piyarat *et al*., 2014), antioxidants (Purbowati *et al*., 2019), and flavoring (Ruangsri *et al*., 2008). Currently, there are many roselle-based products such as tea (Olatunji *et al*., 2014), cupcakes (Abdel-Moemin, 2016), syrup, jam (Arueya *and* Akomolafe, 2014) and jelly (Kharismawati *et al*., 2015). In the health and beauty sector, rosella can be used as a raw material for making toothpaste (Mahmud, 2018) and soap. Based on the foregoing, it is necessary to strive for an alternative form of encapsulation at the nanoscale, so that the high added value of bioactive components can be maintained along with the benefits it generates.

Roselle extract which was still in liquid form causes problems in its use in industries, transportation and also relatively short shelf life. Anthocyanin, a bioactive compound in roselle strongly affected by pH, solvent, temperature, oxygen, enzymes, light (Chumsri *et al*., 2008). To diminish some degradation effects of the

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compound is very common to use encapsulation technique namely microencapsulation. But, according to Mardiah *et al*. (2014), effervescent rosella with formulations using microencapsulation still has a weakness, namely brittle with relatively few bioactive compounds. For these reasons, it was necessary to apply more advanced encapsulation technology that was nanoencapsulation technology on roselle extract. So that its utilization was more optimal.

Nanoencapsulation is a technology for packaging substances in small sizes by utilizing techniques such as nanocomposites, nanoemulsification, and nanostructuring so that functional products can be produced that can release core parts in a controlled manner (Sekhon, 2010). The principle of nanoencapsulation is the mixing of the water phase, the core substance phase and the coating material phase until a stable emulsion is formed, then the process of attaching the coating material to the surface of the core material and the process of reducing the particle size (Dubey, 2009). According to Purbowati *et al*. (2016), nano encapsulated roselle extract had better stability to temperature, time and pH than liquid extract forms. In this research, nanoencapsulation used β-cyclodextrin, capable of trapping 94% of the bioactive compounds therein.

Nano encapsulant matrix selection was a crucial stage in the nanoencapsulation process. Different matrix will provide different protection to the core. Encapsulant must be food grade and GRAS (Generally Recognized as Safe). The compounds can be used as a retaining material. The encapsulant matrix was starch, Arabic gum, methylcellulose, gelatin, whey protein, sugar syrup, β cyclodextrin, disaccharides, pullulan and sodium caseinate (Wandrey *et al*. 2010; Naufalin and Rukmini, 2013; Purbowati *et al*., 2016). The effect of encapsulant agents is unique, depending on the nature of the protected core material. For this reason, it is necessary to examine the compatibility between the core material and anchoring material. The use of Arabic gum as a coating material can protect, even volatile compounds, from oxidation and evaporation (Kanakdande *et al*., 2007). Gum arabic has a high viscosity which reaches 38.0 cP, so the wall layer that is formed will better protect the core material because the skin layer (shell) is stronger, so it is able to protect the volatile core material when the drying process goes well (Sugindro *et al*., 2008). Gum Arabic is also suitable for encapsulation during the drying process because it can protect against oxidation and evaporation and prevent moisture absorption. (Wandrey *et al.,* 2010). Carbohydrates such as starch, maltodextrin are good coatings because they have low viscosity at high solids and have high solubility properties (Balasubramani *et al*., 2014)

On the other hand, the development of knowledge and technology in the field of food encourages the development of products with the right formulation to process natural ingredients into a form of preparation that is easily accepted by the consumer. In addition to other quality parameters that must still be met. Thus, it is expected to increase people's interest in consuming.

The effervescent tablet formulation of roselle was developed to make easier for the consumer. This was done to make it more efficient to use. Effervescent has several advantages, namely that it was more easily and quickly absorbed by the body so that the therapeutic effect was more pronounced, optimal compatibility increases body fluid intake and more practical to use (Harrera-Arellano *et al*., 2007). Roselle has a sour taste. So, it was necessary to determine the exact level of roselle percentage. The combination of roselle percentage and matrix type gives rise to good effervescent characteristics that are acceptable to consumers.

This study aims to determine the effect of variations in nanoencapsulation matrix type and roselle nanocapsule percentage on the physical and chemical effervescent tablet's characteristics.

2. Materials and methods

2.1 Materials

Roselle flower (Bought in Beringharjo Market Yogyakarta), Arabic gum, maltodextrin, artificial sweeteners of aspartame and sorbitol, sodium bicarbonate, citric acid, malic acid, polyethylene glycol, ascorbic acid standard, gallic acid standard, ethanol, maltodextrin, buffer sodium acetate, acetate buffer, reagent folin, distilled water, polyethylene glycol, ethanol (PA), DPPH solution and other chemicals for analysis.

2.2 Roselle extraction method

The roselle extraction method was based on Purbowati *et al*. (2018). Approximately 10 g of the dried roselle petals powder is mixed with 100 mL aquadest. The extraction was done in microwave power extraction 250 W and 5 mins of extraction for each unit combinations. The microwave used for extraction was Electrolux EMM 2007X.

The slurry was radiated in a microwave oven at regular intervals (one-minute radiation and two mins off) to keep the temperature not rising above the boiling point. Roselle extract was filtered and concentrated with a vacuum evaporator at 70°C, 44 cmHg for 15 mins.

2.2 Roselle extract nanoencapsulation

The roselle extraction method was based on Purbowati (2016). Maltodextrin/Gum Arabic mixed with aquadest with the ratio of the nanoencapsulation matrix (g): water (mL) = 1:5 (w/v). This solution was stirred for 30 mins. Roselle concentrate was added to the solution with the ratio of the solution: extract = 20: 1 (v/v). These solutions were homogenized with OSK 7313 Homogenizer at 40°C for 30 mins until an emulsion is formed. Subsequently, the emulsion particles were reduced into nanoparticles using a 22,000-rpm dispersing machine (Ultra-Turrax) for 30 mins. This nano-sized emulsion was then dried using a spray drier with an inlet temperature of 120°C and a pressure of 4.25 bar.

2.3 Roselle effervescent formulation

The ingredients used were sweetener aspartame and sorbitol, citric acid, malic acid, sodium bicarbonate, polyethylene glycol, and roselle nanocapsule powder. The total base of the material made as much as 3 g with the treatment of the formulation composition is as follows in Table 1.

Table 1. The Ingredients of roselle nanocapsule effervescent

Ingredients	Formula A (%)	Formula B (%)	Formula C (%)
Aspartam	0.06	0.06	0.06
Natrium Bikarbonat	40	40	40
Citric acid	30	30	30
Mallic acid	8.6	8.6	8.6
PEG	0.03	0.03	0.03
Roselle nanocapsule	10	15	20
Sorbitol	11.31	6.31	1.31
Sum	100	100	100

The process of making effervescent drinks was done by mixing all the ingredients well and cast them into a tablet with a tablet casting device with 3 g of the total weight for each. The effervescent tablets were packed using aluminum foil for further analysis.

2.4 Anthocyanin

The total anthocyanin content was determined using following procedure (Fuleki and Francis, 1968): *a*pproximately 1 mL of sample solution was added with 1 mL of acidic ethanol (95% ethanol + N HCL (85:15 v/v)). The sample was stored overnight at 40°C. The absorbance was measured using a 535 nm wavelength.

2.5 Total phenol

The total phenol content was determined using the Folin-Ciocalteu method (Chew *et al*., 2009). About 0.4 mL of sample solution was added with 1.5 mL of Folin-Ciocalteu reagent (10% v/v). After being incubated for 5 mins, it was mixed with 1.5 mL of 7.5% (w/v) of Na_2CO_3 solution. After 90 mins of incubation at room temperature and darkroom, its absorbance was measured using 765 nm. Gallic acid was used as a standard. The result was presented as mg Gallic Acid Equivalent (GAE)/g materials.

2.6 Antioxidant capacity

Capacity antioxidant test using the DPPH method (Blois, 2005). 100 mL of sample was added with 3 mL of methanol. This solution was adding with 1 mL of DPPH and keep in the dark for 15 mins at room temperature. The decrease of DPPH absorbance measure with spectrophotometry using λ 517 nm. As the standard was used gallic acid.

2.7 Experimental design and data analysis

The research design used in this study was a completely randomized design with two factors and three replications. The first factor comprises the type of nano encapsulant matrix (A): maltodextrin (A1) and Arabic gum (A2). The second factor was the percentage of nanocapsule powder (B): 10% (B1) which comprises 15% (B2) and 20% (B3).

Test data were analysed by ANOVA at the level of 5%. If there is a real influence, continue with Duncan's multiple area tests.

3. Results and discussion

From the results of the data processing, the nanoencapsulation matrix type had a significant effect on total phenol, anthocyanin, vitamin C, and dissolved time. While the percentage of roselle nanocapsules had a significant effect on phenol, anthocyanin, vitamin c variables, and had no significant effect on dissolved time. Meanwhile, the interaction between nanoencapsulant matrix type and the percentage of nanocapsules added had a significant effect on total phenol and anthocyanins. And had no significant effect on vitamin C and dissolving time.

3.1 Total phenols

The type of matrix and the percentage of nanocapsules were significantly affected the total phenol in effervescent tablets. Table 2 shows that the combination of Arabic gum with a concentration of 20% roselle nanocapsules gave the highest phenol content of 4.1740 µg/3 g. Maltodextrin coated with the same percentage had a total phenol value of 2.9075 µg/3 g. This meant that Arabic gum was better at coating roselle

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phenol compounds. Gum Arabic has a high viscosity which reaches 38.0 cP, so the wall layer that is formed will better protect the core material because the skin layer (shell) was stronger, so it can protect the volatile core material when the drying process goes well (Sugindro *et al.*, 2008). This condition as a result of exposure to heat energy received by maltodextrin is greater than Arabic gum, due to the nature of phenolic compounds which are unstable at high temperatures. This was in line with research by Purbowati *et al*. (2016)

Table 2. Effect of combination treatment of encapsulant types and concentrations of rosella nanocapsules on the amount of total phenol and anthocyanin in effervescent

Treatment	Total phenol	Anthocyanin
A1B1	$1.96±0.20^{a}$	$0.012±0.007^{e}$
A1B2	$2.38±0.11^{cd}$	$0.028±0.006^{d}$
A1B3	$2.90±0.68^{bc}$	$0.058±0.011^{b}$
A2B1	$2.06±0.34^{d}$	$0.007±0.007^{e}$
A2B2	$3.35±0.21^{b}$	$0.039±0.008^{c}$
A2B3	$4.17±0.37^{a}$	$0.070±0.002^{a}$

A1B1 = maltodextrin with concentration 10%, A1B2 = maltodextrin with concentration 15%, A1B3 = maltodextrin with concentration 20%, A2B1 = arabic gum with concentration 10%, A2B2 = arabic gum with concentration 15%, A2B3 = arabic gum with concentration 20%
Values are expressed as mean±standard deviation of four independent data. Values with different superscript in the same column are significantly different in *Duncan*'s test (P <0.05).

3.2 Anthocyanin

The effect of variation in the percentage of Roselle nanocapsules, matrix type, and interaction both of them to anthocyanin was significantly different. Table 2 shows that the increasing nanocapsule percentage will increase the anthocyanin content in effervescent tablets. Nanoparticles formulation is needed to increase the bioavailability of active compounds penetration (Nurkhasanah *et al*., 2015), the higher the percentage of rosella nanocapsules added, the number of bioactive compounds in it will be even higher. Arabic gum is better at coating rosella bioactive material. This is in line with Khasanah *et al*. (2015). Maltodextrin can form a good matrix network, but its viscosity was lower than Arabic gum and made the drying process last relative short. Gardjito *et al*. (2006) stated that the simpler molecular structure of maltodextrin makes it easy for water to evaporate during the drying process. Anthocyanin, a bioactive compound in roselle strongly affected by pH, solvent, temperature, oxygen, enzymes, light (Chumsri *et al*., 2008). As a result, maltodextrin compared to gum arabic is not good enough to protect rosella bioactive compounds, namely anthocyanins. The highest anthocyanin value was in combination with A2B3, which is Arabic gum with 20% of roselle.

3.3 Vitamin C

The different types of the matrix have a significantly different effect on vitamin C content in effervescent. This result can be seen in Table 3. This difference is due to the different chemical structures of maltodextrin and Arabic gum, besides the ability of the coating material to protect the bioactive compounds therein is also different. According to Khasanah *et al*. (2015) maltodextrin did not have a good emulsifying ability. This results in a thinner layer of protection and does not have the ability to properly cover the core material. The matrix must be able to form films, be easily biodegradable, have low viscosity and hygroscopic. The total phenol in rosella 70% is anthocyanin (Purbowati *et al*., 2016). Anthocyanins are water-soluble dyes and compatible with water-based encapsulants matrices such as maltodextrin and Arabic gum. In addition, Arabic gum molecular weight was greater than maltodextrin (Gardjito *et al*., 2006). Mean that the solubility of Arabic gum is higher than maltodextrin. This results in the Arabic gum solution being thicker than the maltodextrin solution. So that when drying use a spray dryer using an inlet temperature of 140°C caused the total levels of vitamin C in Arabic gum to be higher. Although spray drying is a fast process, a change in the spray drying temperature can affect the concentration of vitamin C.

Table 3. The effect of variation type nanocapsules matrix on vitamin C, antioxidant activity and time dissolve in effervescent.

Type nanocapsules matrix	Vitamin C (μg/3 g)	Antioxidant activity (%)	Dissolution Time (mins)
Maltodextrin	$0.118±0.053^{b}$	$26.85±8.56^{b}$	$1.25±0.17^{b}$
Arabic gum	$0.149±0.049^{a}$	$34.02±4.52^{a}$	$2.93±0.80^{a}$

Values are expressed as mean±standard deviation of four independent data. Values with different superscript in the same column are significantly different in *Duncan*'s test (P <0.05).

Table 4. The effect of variation nanocapsule concentration on vitamin C in effervescent.

Nanocapsule concentration (%)	Vitamin C (μg/3 g)
10	$0.079±0.032^{c}$
15	$0.135±0.017^{b}$
20	$0.188±0.032^{a}$

Values are expressed as mean±standard deviation of four independent data. Values with different superscript in the same column are significantly different in *Duncan*'s test (P <0.05).

Table 4 shows that the increasing nanocapsule percentage will increase the vitamin C content in effervescent tablets. The use of the encapsulant matrix served as a wall to coat the active ingredients. The more nano-encapsulation matrix was added, the more vitamin C was trapped.

3.4 Antioxidant activity

The antioxidant properties of the nanocapsules were positively correlated with the content of bioactive compounds therein. Roselle flower contains bioactive compounds, namely phenol 19.45±0.32 mg/g, vitamin C 20.47±0.34 mg/g, anthocyanin 13.51±0.03 mg/g. According to Purbowati *et al*. (2016), the antioxidant activity of rosella flowers was positively correlated with the total content of phenol and vitamin C.

Table 3 shows that the type of matrix had a significant effect on the antioxidant activity of the effervescent tablet. The antioxidant activity of effervescent tablets using Arabic gum is 34.02% higher than effervescent tablets using the maltodextrin matrix which is 26.85%. This is because the content of vitamin C and the total phenols in the nanocapsules made from Arabic gum was greater than nanocapsules that used maltodextrin. Christian and Jackson (2009) stated that the role of antioxidant activity in rosella was not only by anthocyanins as a phenolic group but also the content of vitamin C. The higher levels of anthocyanin in effervescent tablets, the higher antioxidant activity. In this study, effervescent tablets which have formulas with high vitamin C and anthocyanin content will also have a high antioxidant capacity, namely in formulas using Arabic gum with a value of 34.02%.

3.5 Dissolving time

The variation of the matrix significantly affects the effervescent dissolving time. Effervescent tablets are designed to break in contact with liquid such as water or juice, often causing the tablet to dissolve into a solution. The time it takes from the first contact with the water surface until it is completely dissolved is called dissolving time. However, if all the ingredients used are the same, in this research the difference in the effervescent dissolving time is determined by the difference in the matrix used. The nanocapsules used should have high solubility in the commonly used solvent, cold water. The results of the dissolving time test showed in Table 3 that maltodextrin had a lower solubility time of 1.25 mins faster than the solubility time of matrix Arabic gum which was 2.93 mins.

According to Mardiah *et al*. (2014), the addition of maltodextrin fillers in large effervescent tablets greatly affects the hardness value of effervescent tablets made. Fragility is a picture of the bonding strength of the particles forming the tablets. The more fragile the tablet, the more resistant it is to erosion. As a result, when the maltodextrin's tablets are put into the water the dissolution time is shorter than using Arabic gum fillers.

The solubility of effervescent tablets is influenced by the presence of crushing agents in the form of acidic substances (malic acid and citric acid) and base sources (sodium bicarbonate). Both types of this material, when met with water, will produce CO_2 gas which gives a refreshing effect. In addition to these factors, the amount of coating agent and the method of mixing also affect the dissolution time of the tablet.

4. Conclusion

The Arabic gum gave better value on vitamin C, antioxidant activity and dissolving time than maltodextrin, namely 0.149±0.04 9 µg/3g, 34.02±4.52%, 2.93±0.80 mins. The percentage of rosella nanocapsules by 20% gave the highest vitamin C value, namely 0.188±0.032 µg/3 g. The best treatment combination was obtained from the treatment using (A2B3) Arabic gum matrix and 20% of roselle nanocapsules with the characteristics: total phenol content 4.17 µg/3 g, anthocyanin 0.070 µg/3 g, vitamin C 0.188 µg/3 g, when it dissolves in 1.25 mins.

Conflict of interest

The authors declare no conflict of interest.

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