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Spray Drying of Rosella (*Hibiscus sabdariffa L.*) Powder: Effect of Shelf Life on Physicochemical Properties and Cyanidin 3-O—glucoside

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Abstract. The effects of inlet temperatures of 120 and 150°C and shelf life levels at 3, 6, 9, 12, and 15 days in temperature 30°C on the physicochemical properties, anthocyanin and *Cyanidin 3-O—glucoside* of spray-dried rosella powder were studied. A study was conducted using Armfield spray dryer FT30MKII to produce spray-dried rosella powders using 10% maltodextrin concentrations as the encapsulating agent. Moisture content, water activity, and solubility of powder were significantly affected by inlet temperature. However, an increase in the level of inlet temperature did not substantially affect the L^* , a^* , b^* , hue, and chroma values. An increase in drying temperature decreased the anthocyanin and *Cyanidin 3-O—glucoside* activity of the powder.

1. Introduction

Drying an antioxidant-rich extract will promote improved shelf life and decreased microbial degradation due to reduced water content. Also, a decrease in weight by eliminating water would make it possible for the product to be shipped at a substantially reduced rate, which might raise the probability that the result would be made available widely. The production of a powdered product may also increase the ease with which this antioxidant-rich powder is added to consumer food products. Rosella extract (*Hibiscus sabdariffa L.*) high in antioxidants was selected as sample material. Anthocyanins, the largest group of natural pigments in plants, are the primary antioxidants to a rosella. They are responsible for many attractive plants, such as flowers, fruit (especially berries), and vegetables [1].

Spray-drying microencapsulation is a typical process for converting an encapsulating agent (core material) into powder and a homogenized mixture of the encapsulating agent (wall material). Maltodextrin and solvent substances will wrap around the material at the time of embedding quickly surrounding the core material with sprays and high temperatures for solvent volatilization [2]. Spray drying techniques are the most common due to their high quality, efficiency, and low-cost microencapsulation [3]. A vital factor in the efficiency of the encapsulation process is the drying conditions of the spray, such as the temperature of incoming air [4]. Some studies have shown that the temperature of incoming air spray dryers can affect the chemical and physical properties of microcapsules [4–6]. However, there is little research on the impact that incoming air temperature and shelf life can have on the physicochemical and cyanidin 3-O-glucoside microcapsules rosella (*Hibiscus*



sabdariffa L.). Therefore, it is desirable to maximize incoming air and shelf life's temperature to achieve the best physical, chemical, and functional qualities of the rosella microcapsules.

Microencapsulation is the process by which bioactive materials are coated to form small multi-function capsules [7]. This approach is generally used to increase consumer acceptance by applying water-insoluble aromatics to water foods, minimizing unwanted tastes, and odors [8]. Microencapsulation can also prevent and improve the active element's stability by protecting the active ingredient from the adverse effects of moisture, acid, heat, oxygen, and material interaction [9]. Another benefit of microencapsulation is the setting of release points or release parameters [10]. We can more precisely determine whether the active component is made accessible to the target by controlled release microencapsulation. It releases the active ingredient from the embedded matrix if it is most useful for the desired application.

The research aims: 1) to produce rosella powder in the form of microencapsulated with differences inlet temperature and shelf-life; 2) physicochemical analysis which includes water content, water activity, and solubility of rosella powder based on differences inlet temperature of 120°C and 150°C; 3) analysis of anthocyanins and cyanidin-3-glucoside on the influence of differences in temperature inlet 120°C and 150°C and shelf life for 15 days.

2. Methodology

2.1. Materials

Rosella was purchased from the local market in Makassar, Indonesia. Rosella is thoroughly washed under tap water to extract adhesive dust and used to produce spray-dried rosella powder. Pro-quality chemicals analysis grade: potassium chloride and sodium acetate (buffer solution), hydrochloric acid supplied by Merck Millipore (Burlington, Massachusetts, United States), maltodextrin from sigma aldrich.

2.2. Preparation of rosella powder using a spray dryer

Early stages by extracting rosella flowers by maceration method using water solvents. Rosella flowers dried with the oven at a temperature of 40°C for 6 hours. Then mashed and continued with maceration extraction for 24 hours. The filtrate is filtered and centrifuged at 3500 rpm for 15 min at 10°C. Furthermore, rosella extracts 350 mL mixed with maltodextrin concentration 10%, homogenized and inserted into a spray dryer with an inlet temperature of 120°C speed of 3 rpm. The extract is dried with an inlet temperature of 150°C at the same process.

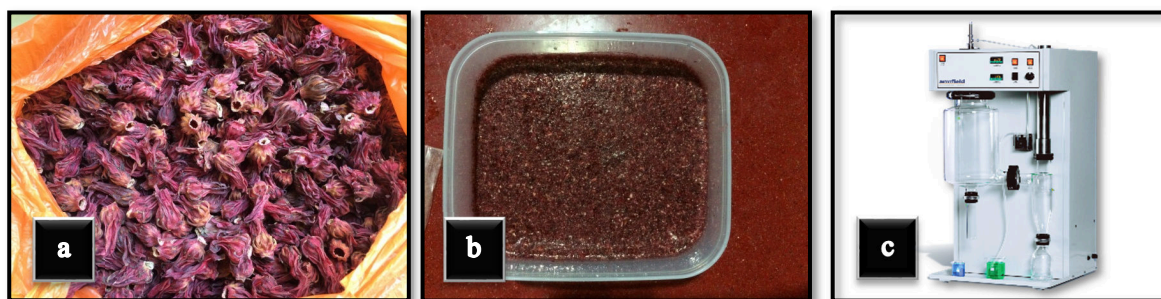


Figure 1. Preparation of rosella powder: a) rosella, b) rosella extract; and c) Armfield spray dryer model FT30MKII

2.3. Analysis of physicochemical extract rosella during storage

Rosella powder was obtained from the process of spray drying with an inlet temperature of 120°C and 150°C, packaged using HDPE (high-density polyethylene) plastic bottles, placed in the stabilizer for 15 days, with part-time measurement every three days, which includes analysis of water content, water activity, and solubility. Moisture content, water activity, dry ash, and solubility determinations were determined by standard research methods [11].

2.4. Determination of total anthocyanin content and cyanidin-3-glucoside

The pH-differential method was used to measure the total amount of anthocyanin (TAC) [12]. Anthocyanin pigments are subjected to reversible structural transformations, with a pH change manifest through distinct spectra of absorption. Oxonium colored form predominates at pH 1.0, and hemiketal colorless form at pH 4.5. The pH differential method is based on this reaction, allowing accurate and rapid measurement of total anthocyanins in the presence of polymerized degraded pigments and other compounds of interference. In short, transfer 1 mL of extracted solution to 10 mL of volumetric flask to prepare two sample dilutions, one with potassium chloride buffer, pH 1.0, and the other with sodium acetate buffer, pH 4.5. Equilibrate these dilutions for 15 min. Measure absorbance of each dilution at 510 and 700 nm (to be corrected for each dilution haze) against a distilled water cell. All measurements should be taken between 15 min and 1 hour after sample preparation as time is longer. Standing times tend to increase the observed readings. Absorbance readings are performed against water blanks. The samples to be measured should be clear and do not contain haze or sediment; however, some colloidal materials may be suspended in the sample, resulting in light scattering and cloudy appearance (moisture). This light dispersion must be corrected by reading at a wavelength where the sample is not absorbed, i.e., 700 nm. The diluted sample (A) absorption is calculated as follows:

$$A = (A_{510} - A_{700})_{\text{pH } 1.0} - (A_{510} - A_{700})_{\text{pH } 4.5}$$

In the original sample, calculate the monomeric anthocyanin pigment concentration using the following formula:

$$\text{Monomeric anthocyanin pigment (mg/L)} = (A \times \text{MW} \times \text{DF} \times 1000) / (\epsilon \times l)$$

And the total anthocyanin content of the sample was converted to mg/100 g. MW is the molecular weight, DF is the dilution factor, and the molar absorption factor ϵ , pigment content is calculated as cyanidin-3-glucoside and MW = 449.2, ϵ = 26,900.

2.5. Colour profile determined by chromameter

The color analysis of the sample was performed with a duplicate of Minolta Chroma CR-400 (Minolta Co, Osaka, Japan) and Hunter method (as L, a, and b value). The parameter of the value L, a, b will be visible where the rosella powder surface amount luminosity (L), angle of color ($^{\circ}$ h), and chromaticity (C). On a value (+) is the red color, and the value (-) on a green color show; on a value (+) in b, the yellow color is indicated; on a value (-), the blue color is displayed. The color specifications are then measured on the chart [13].

3. Results and Discussion

3.1. Rosella powder physical properties during storage

The powder's moisture content ranged from 6.34 to 10.28% for all inlet air temperatures during the retention period. Microcapsules sprayed at 120°C showed significantly higher moisture content than other treatments. The moisture content was considerably reduced at a temperature of 150°C inlet air. Since a moisture content of 6% or less is optimal to minimize microbial contamination and oxidation of lipids, an air inlet temperature greater than 100°C would be preferable [14]. The powder's moisture content was lower at higher inlet air temperatures, probably as the air inlet temperature increased and drying capacity increased.

The value of a_w rosella powder decreases when the temperature of the inlet temperature used is higher. The inlet temperature used in making rosella powder ranges from 120°C and 150°C, with a_w values ranging from 0.23 to 0.48 in table 1. Spray drying particles can absorb atmospheric water readily in the surrounding air. At this point, the powder's surface becomes sticky, resulting in the caking of the powder [2]. Hygroscopicity increased due to a drop in water content. The powder's high hygroscopicity indicates the need to investigate further the relationship between water content and stability, and shelf life.

Rosella powder with an inlet temperature of 150°C tends to dissolve more quickly than rosella powder with an inlet temperature of 120°C. Rosella powder with an inlet temperature of 150°C has a

low moisture content that makes the powder more hygroscopic and quickly absorbs water. The solubility of the powder in water is also higher. Solubility is an essential requirement for using proteins as effective functional ingredients in various high-humidity foods, such as emulsions, foams, and beverages. To understand how the high-dispersion drying technique can influence rosella powder solubility.

Table 1. Analysis of physicochemical rosella powder during storage

Temperature (°C)	Time (Days)	Water content (%)	Water activity (aw)	Solubility (%)
120	0	8.74	0.37	93.6
	3	8.80	0.37	93.6
	6	8.96	0.4	93.6
	9	9.09	0.41	93.2
	12	9.92	0.43	93.1
	15	10.28	0.48	92.8
150	0	6.34	0.23	95.5
	3	6.58	0.23	95.5
	6	7.51	0.29	95.5
	9	7.61	0.31	95.3
	12	7.64	0.31	95.2
	15	8.06	0.34	95.2

3.2. Determination of anthocyanin and cyanidin-3-glucoside

Susceptible compounds are anthocyanin and are unstable during processing and storage. The function of the internal properties (pH, chemical structure, and concentration of anthocyanin) of the available enzymes and other color-inducing substances, ion metals, sugars, and processing conditions (heating intensity and duration, storage time and temperature, oxygen and light) is useful in their stability [15]. Anthocyanin content on the first day of storage at inlet temperatures of 120°C and 150°C is 0.176 mg/ml and 0.137 mg/ml. Anthocyanin levels in rosella powder indicate anthocyanin instability during a 15-day hold. The longer the storage of rosella powder, the lower the anthocyanin levels. This is due to the presence of degraded pigments with reactions to changes in flavium cation into kalkan.

On the first day, rosella powder storage with an inlet temperature of 120°C obtained cyanidin levels of 28.1 mg/L; after storage for 15 days, cyanidin levels decreased to 18.7 mg/L. The decrease in storage occurred due to several factors, namely the influence of light through the packaging used to pack rosella powder. Sunlight through the packaging and temperature causes an oxidation reaction; there is a disconnection of covalent bonds resulting in sugar molecules' release. Anthocyanins found in rosella are *delphinidin*, *cyanidin*, *petunidin*, *myricetin*, *pelargonidin*, and *malvidin*. In rosella flowers, *Delphinidin 3-sambubiosid* is the primary type of anthocyanin is about 85% of the total anthocyanins and is a compound that gives red color and *cyanidin 3-sambubiosid* that contributes pink color.

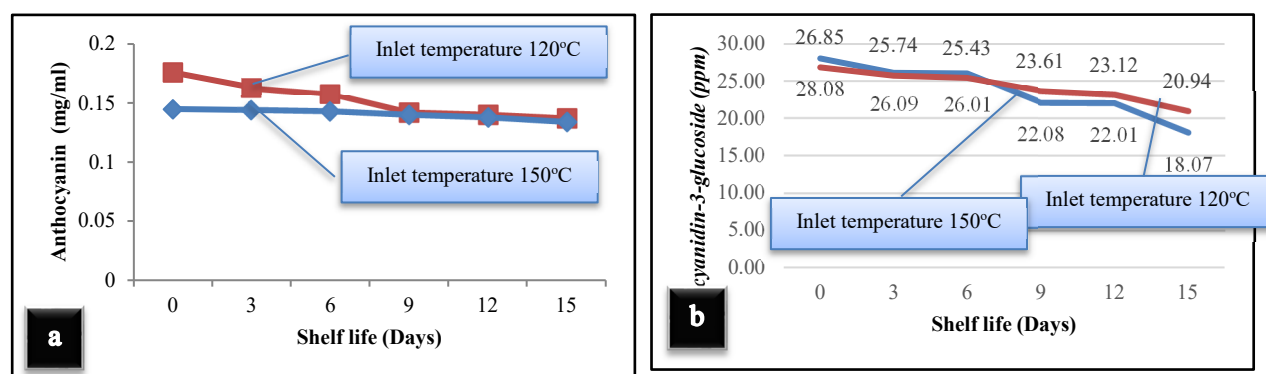


Figure 2. Determination antioxidant: a) anthocyanin b) cyanidin-3-glucoside

3.3. Color

The powder's color is a crucial factor in consumer acceptance and represents the original food product. Even if food powder (in other products) is very applicable with considerable health benefits but does not have attractive eye-view aspects, it may not appeal to the employers. Table 2 shows rosella powder color values obtained by various treatments. Intensifying temperatures, the L* rosella powder value increased. Similarly, [16] the highest lightness value was observed at the highest inlet drying temperature of spray-dried tomato powders indicating less darkness due to pigment-oxidation. The lightness of rosella powders decreased by increasing the inlet drying temperature due to their high sugar content, which causes browning of powders [30].

Table 2. The color characteristic of rosella powder

Parameters	Inlet temperature											
	120°C						150°C					
	0	3	6	9	12	15	0	3	6	9	12	15
*L	23	23.1	23.3	23.9	23.9	28.4	24.9	23.3	23	22.9	22.5	22.4
*a	18	18.1	18.4	18.7	19.1	20.1	19.2	17.9	17.6	17.5	17.4	17.1
*b	3.9	3.9	3.8	3.8	3.6	3.6	4.3	3.9	3.9	3.9	3.8	3.4
°Hue	21	21	20	20	19	18	22	21	22	22	22	20
Color	R	R	R	R	R	RP	R	R	R	R	R	R

Description:

R = Red

RP = Red Purple

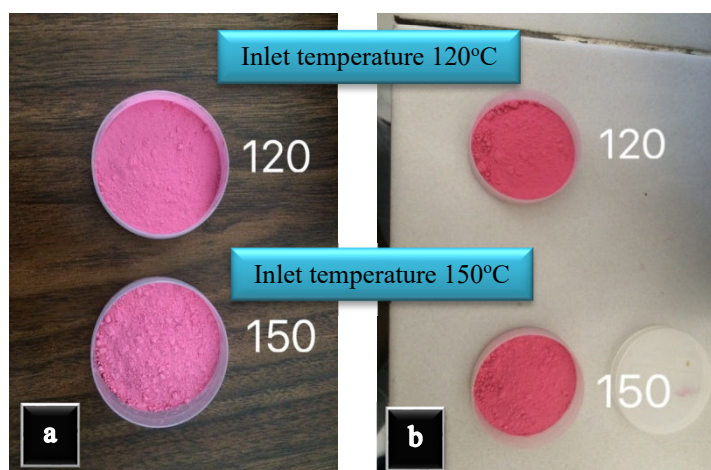


Figure 3. Colour profile during storage: a) shelf life 6 days; b) shelf life 12 days

4. Conclusion

The results showed that the inlet temperature influences the chemical and physical properties of spray-dried powders significantly. As the inlet temperature and shelf life increased, the powder's humidity and solubility increased while the time required for reconstitution increased. The lightness and hue of the spray-dried powders were reduced, and the loss of anthocyanin and cyanidin-3-glucoside could be correlated at higher inlet temperatures. The inlet temperature also decreased as the sugar content increased. In general, the spray-dried powders are the best colorimetric, reasonably low humidity content and water activity at an inlet temperature of 150°C with a shelf-life of 3 days, as well as good anthocyanin and cyanidin-3-glucoside content. The physical and chemical properties of powders are essential for the production of high-quality rosella powders. A further investigation is required to address surface stickiness and powder hygroscopicity about the shelf life of the product.

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