







Ohmic Heating Process for the Extraction of Betacyanin Pigment from Red Dragon Fruit Peel as a Natural Food Colorant Ingredient

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<https://doi.org/10.18280/ijht.420333>

ABSTRACT

Received: 16 March 2024

Revised: 22 May 2024

Accepted: 4 June 2024

Available online: 27 June 2024

Keywords:

betacyanin, extraction, ohmic heating, solvent, temperature

The usage of natural food colorants in food processing is increasing nowadays. Red dragon fruit peel is rich in betacyanin pigment. The peel, which is generally categorized as food waste, can be extracted and used as a natural food colorant that produces a red-violet color. Pigment extraction using ohmic heating can shorten the extraction time. Ohmic heating has a very fast heating rate and can avoid excessive thermal damage to pigments. This study aims to determine the effect of solvent type and extraction temperature on chemical and physical characteristics, as well as process parameters (heating time, electrical conductivity, and ohmic heating efficiency) of extracts that have been extracted using the ohmic heating method. The study used a 2-factor group randomized design, namely solvent type (distilled water, citric acid 0.2%, and NaCl 0.2%) and extraction temperature (50, 60, 70, 80, and 90°C). The results showed that the interaction between solvent type and extraction temperature significantly affected betacyanin content. Each factor (solvent type and extraction temperature) had a significant effect on the heating time, electrical conductivity, and extraction yield. NaCl 0.2% solvent can increase electrical conductivity so that the heating time is faster and pigments can be extracted from the cells. Ohmic heating can be used for the extraction process of betacyanin pigment from red dragon fruit peel, which is heat-labile.

1. INTRODUCTION

The color of food products is one of factors consumers consider when choosing a food product. Generally, color used in the manufacture of food products consist of two types, namely natural and synthetic food colorant. The use of synthetic food colorant that exceeds the ADI (Acceptable Daily Intake) value can endanger health, such as reducing brain motor activity, the formation of free radicals in the human body, and the occurrence of DNA damage in the colon [1]. The increasing human awareness of health has led to the increasing use of natural coloring in food products. Sources of natural food coloring are from plants and animals. Examples of plant sources of natural food coloring include: red dragon fruit, beet tubers, strawberries, tomatoes, grapes, turmeric, saffron, and vegetables [2-4].

Red dragon fruit is a source of natural food colorant that contains betacyanin pigment. Java, Kalimantan, and South Sulawesi are the producing areas of red dragon fruits [5]. Indonesia uses 4,300 ha of land (mainly in Banyuwangi, East Java, and East Kalimantan) for dragon fruit cultivation. From 2,300 ha in Banyuwangi, the yield of dragon fruit is 117,700 tons. Generally, the weight of dragon fruit peel is 35% of the total weight of the fruit, so if calculated from the number of crops per year, it is known that dragon fruit peel waste in Indonesia reaches 82,000 tons per year [6]. Generally, people

only eat the flesh of red dragon fruit, while the peel is discarded. Red dragon fruit peel contains betacyanin pigments and antioxidants so that it can be used as a natural colorant in food [7].

Red dragon fruit peel needs to be extracted first so that the betacyanin pigment can be separated from other components. Extraction using conventional methods such as maceration, distillation, and soxhlet requires a large amount of solvent and requires a longer extraction time [4]. On the other hand, non-conventional extraction methods require shorter extraction times and smaller amounts of solvent. Unconventional extraction methods are new and widely developed extraction methods in research. Types of unconventional extraction methods include: ultrasonic, high pressure, PEF (Pulse Electric Field), and ohmic heating. The ohmic heating extraction method has many advantages, including: faster and more even heating that can produce products with minimal changes in structure, nutrients, and organoleptic properties [8]. In ohmic heating technology, the electrical resistance contained in food materials is utilized to produce heat. In this process, electrical energy is converted into heat energy. The advantage of using ohmic heating technology is that it can avoid damage to substances that are labile to heating, such as pigments and vitamins [9, 10].

Several studies have shown that pigment extraction using the ohmic heating method can achieve the highest yield of

betasianin at 70.58%, while the conventional method can achieve the highest yield of 57.80%. On the other hand, the energy efficiency of ohmic heating is higher (37-67%) than using conventional extraction methods (13-17%) [11]. In research on the extraction of anthocyanin pigments from black rice bran, extraction using ohmic heating produced extracts containing higher total anthocyanins (10,818.5 µg/g) than steaming-assisted solvent extraction (7,713.4 µg/g) [9]. The ohmic heating method for the extraction of anthocyanin pigments in colored potatoes requires a shorter time and lower energy consumption than conventional extraction [12].

The solvent generally used for betacyanin pigment extraction is a polar solvent, namely water. The problem with using water as a solvent in ohmic heating extraction is that water has low electrical conductivity. The conductivity of pure water is 0.055 µS/cm [13], so the presence of ionic compounds is needed to increase electrical conductivity and extraction products such as salts and acids [14]. The addition of NaCl (sodium chloride) to the solvent can increase the electrical conductivity in the ohmic heating process [15]. Food conductivity can be affected by acid, salt, or water content [16]. The presence or concentration of some types of electrolytes such as NaCl can change the conductivity system in ohmic heating [17]. Ionic compounds such as acids and salts will dissociate in solution into H⁺, Na⁺, and Cl⁻. These ions will increase the electrical conductivity of the food matrix [18]. Conversely, the addition of citric acid to the solvent can reduce the pH value, causing an increase in the amount of betalain pigment extracted [17]. Citric acid acts as a chelating agent and increases betalain stability, as it neutralizes some of the electrophilic centers of betalain compounds. Changes in pH can enhance betalain regeneration and prevent pigment degradation during thermal processes [19]. It is necessary to evaluate the use of water, citric acid, and NaCl solvents and extraction temperature on the characteristics of betacyanin pigments from red dragon fruit peels.

2. METHOD

2.1 Materials

The material used in the study was red dragon fruit peel which is the rest of household consumption. Materials used for extraction include: Distilled water (Brataco), citric acid (PUDAK), and NaCl (JIC). Materials used for chemical analysis include: distilled water (Brataco), citric acid monohydrate (Merck), sodium hydrogen phosphate (PUDAK), and buffer pH 6.86.

2.2 Extraction with ohmic heating

The first step is to wash the red dragon fruit peel with running water to remove impurities. The volume of water used to wash 150 g of red dragon fruit peel is 500 mL. Then remove the scales from the red dragon fruit peel. Puree the red dragon fruit peel with a chopper for 1.5 minutes. Put the red dragon fruit peel pulp into solvent and stir until well mixed. Pulp: solvent ratio is 1:5 (w/v). The mixture of red dragon fruit peel pulp and solvent was put into an ohmic heating device (titanium electrode, maximum voltage and frequency are 220 V and 250 Hz. Electrode a is long, while electrode b is circular, with a diameter of 4.7 cm and a height of 2.5 cm. Distance between electrodes 2 cm). The extraction process was carried

out using ohmic heating with a voltage of 60 V and a frequency of 100 Hz. Illustration of ohmic heating assisted extraction system can be seen in Figure 1.

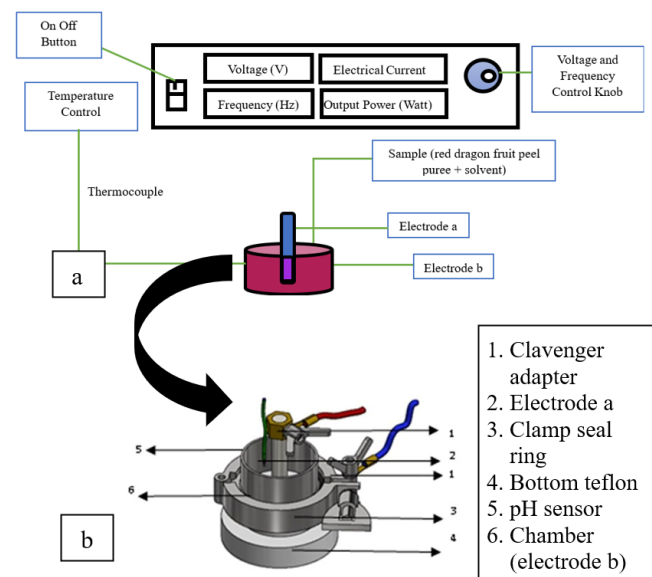


Figure 1. a) Schematic of ohmic heating-assisted extraction system. b) Schematic of electrode

The types of solvents used for the extraction process include: distilled water, 0.2% citric acid, and 0.2% sodium chloride (NaCl). The extraction temperatures used included: 50°C, 60°C, 70°C, 80°C, and 90°C. Furthermore, the filtration process was carried out using filter cloth and filter paper to separate the dragon fruit peel pulp from the extract. In addition, a pressing process is also carried out on the pulp contained in the filter cloth to remove the remaining extract.

This study used a two-factor Randomized Group Design, solvent type (distilled water, citric acid 0.2%, and NaCl 0.2%) and extraction temperature (50°C, 60°C, 70°C, 80°C, and 90°C). The study was repeated triplicate. Data were analyzed using ANOVA (Analysis of Variance). For the pH change parameter, a paired t-test was conducted between the pH of the sample before and after the extraction process.

2.3 Heating time [11]

The heating time is measured from when the ohmic heating is turned on until it reaches the extraction temperature.

2.4 Electrical conductivity [20]

Electrical conductivity was calculated with:

$$\sigma = \frac{IL}{AV} \quad (1)$$

where, σ =electrical conductivity (Siemens per meter or S/m); L=distance between the electrodes (m); A=cross sectional area of the electrode (m²); I=electrical current (A).

2.5 Ohmic heating efficiency [11]

Ohmic heating efficiency is calculated using the following equation:

$$\text{Ohmic heating efficiency} = \frac{Q}{\Sigma \Delta V I t} \times 100 \% \quad (2)$$

where, m=mass of sample (red dragon fruit peel and water) (kg); C_p =specific heat of sample (J/kg °C); T_i =initial temperature of the sample (°C); T_f =final temperature of the sample (°C); V=Voltage (V); I=electrical current (A); t=heating time (s).

2.6 Extraction yield [21]

The extract was dried using an oven until a constant weight was obtained.

$$\text{Extraction yield (\%)} = (W_2/W_1) \times 100 \quad (3)$$

where, W_2 =weight of dried pigment extracts; W_1 =weight of dragon fruit peel.

2.7 Determination of betacyanin content [1, 22]

Samples were weighed and diluted with McIlvaine buffer (pH 6.5; citrate-phosphate) to achieve an absorbance value of 1.0 ± 0.1 . McIlvaine buffer was prepared from 0.1 M citric acid (29.65 ml) and 0.2 M disodium hydrogen phosphate/ Na_2HPO_4 (70.35 ml).

The diluted sample was filtered before measurement with a spectrophotometer at a wavelength of 537 nm and a correction value at 600 nm. Calculation of betacyanin content using the following equation:

$$\text{BC (g/kg)} = (A \times V \times \text{DF} \times \text{MW} \times 1000) / (\epsilon \times L \times W) \quad (4)$$

where, BC=Betacyanin Content; A=Maximum absorbance value (537 nm) corrected by absorbance at wavelength 600 nm; V=Volume of extract (mL); DF=Dilution Factor; MW=Molecular weight of betanin (550 g/mol); ϵ =Molar extinction coefficient of betanin ($65,000 \text{ L mol}^{-1} \text{ cm}^{-1}$); L=Cuvette thickness (1 cm); W=Weight of extracted material (fresh state) (g).

2.8 pH change [23]

The pH of the sample was measured before and after the extraction process was carried out. pH was measured using a pH meter.

2.9 Color [11]

Color (L, a, b) is measured using a colorimeter/color reader [7]. Press the target button on the color reader, bring the color reader lens close to the sample. Press the button for sample color measurement.

$$\begin{aligned} L & (+=\text{lighter}, -=\text{darker}) \\ a & (+=\text{red}, -=\text{green}) \\ b & (+=\text{yellow}, -=\text{blue}) \end{aligned} \quad (5)$$

ΔE were calculated with equation:

$$\Delta E = ((L_0 - L_i)^2 + (a_0 - a_i)^2 + (b_0 - b_i)^2)^{1/2} \quad (6)$$

where, L_0 , a_0 , b_0 =sample color before extraction process; L_i , a_i , b_i =sample color after extraction process.

2.10 Data analysis

Data analysis used Minitab 17 and Microsoft Excel 2017 software. If there is a significant effect, the data is further tested using the Tukey test.

3. RESULTS AND DISCUSSION

3.1 Heating time

The interaction between solvent type and extraction temperature had no significant effect on the heating time, while each factor (solvent type and extraction temperature) had a significant effect on the heating time. Distilled water solvent type has the highest heating time compared to other solvent types (Figure 2). On distilled water solvents, the heating time between temperatures 50-90°C is 63 s, 87 s, 118 s, 139 s, and 154 s. This is because water has low electrical conductivity (Figure 3). The conductivity of pure water is $0.055 \mu\text{S/cm}$ [13]. Citric acid 0.2% and NaCl 0.2% solvent have higher electrical conductivity than distilled water. This is because ionic compounds such as acids and salts will dissociate in solution into H^+ , Na^+ , and Cl^- . These ions will increase the electrical conductivity of the food matrix [18]. Varghese et al. [14] in their research also said that acids and salts can increase electrical conductivity.

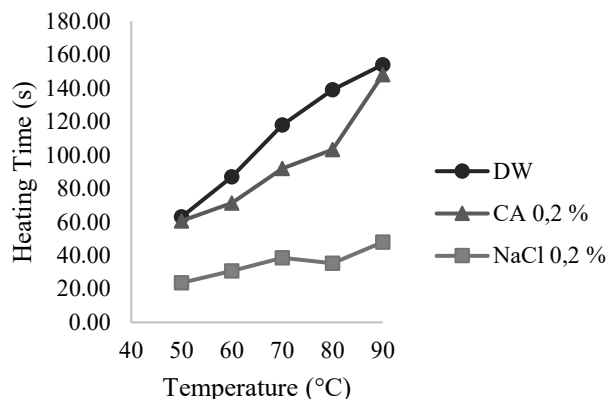


Figure 2. Heating time analysis (DW=Distilled Water; CA 0.2%=Citric Acid 0.2%; NaCl 0.2%=Sodium Chloride 0.2%)

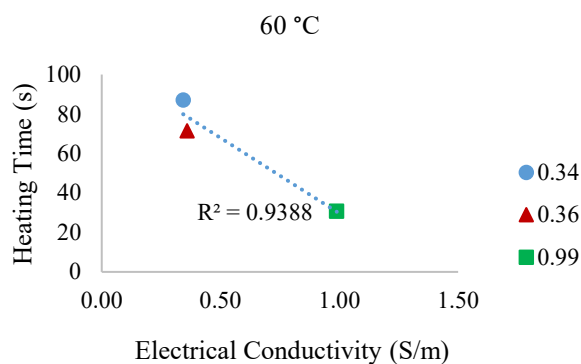


Figure 3. Correlation between heating time and electrical conductivity at temperature 60 °C (DW=Distilled Water; CA 0.2% = Citric Acid 0.2%; NaCl 0.2%=Sodium Chloride 0.2%)

The higher the extraction temperature, the longer the time used to reach that temperature. This is in line with research conducted by Al-Hilphy et al. [24] about the extraction of phenol compounds from wheat bran. In his research, the higher the extraction temperature, the longer the heating time.

The example correlation between heating time and electrical conductivity at temperature 60°C can be seen in Figure 3. It can be seen that the value of R^2 was 0,9388. The higher the heating time, the lower the electrical conductivity value. This indicates that heating time and electrical conductivity have a strong negative correlation relationship.

3.2 Electrical conductivity

The interaction between solvent type and extraction temperature had no significant effect on the electrical conductivity, while each factor (solvent type and extraction temperature) had a significant effect on electrical conductivity.

The electrical conductivity value increases with temperature (Figure 4). When the temperature of the sample increases, the resistance to ion movement will decrease. As a result, the movement of ions in the sample will be faster so that it can result in strengthening the electrical conductivity [25, 26].

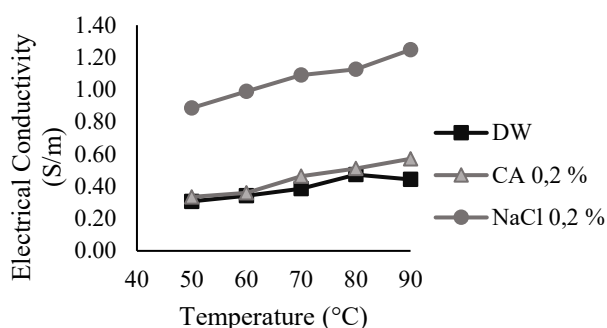


Figure 4. Electrical conductivity analysis (DW=Distilled Water; CA 0.2%=Citric Acid 0.2%; NaCl 0.2%=Sodium Chloride 0.2%)

NaCl solvent type has the highest electrical conductivity value. Electrical conductivity of NaCl solvents at temperatures of 50-90°C includes 0.89 S/m, 0.99 S/m, 1.09 S/m, 1.13 S/m, and 1.25 S/m. This is because the change in electrical conductivity value is due to changes in the ion content of a solution. The electrical conductivity of a solution will increase with increasing salt concentration [27].

Foodstuffs containing ions such as salts and acids cause the conduction of electric current. Electrical conductivity (σ) is a measure of how well a material accommodates the movement of electric charges. When an electrolyte is in an electric field, the ions in the electrolyte move towards the electrode with the opposite charge and generate heat due to the movement of the ions. In addition, the ions moving in it collide with each other. This creates resistance to the movement of the ions and increases their kinetic energy, thereby heating the material [28].

3.3 Ohmic heating efficiency

The interaction between solvent type and extraction temperature had no significant effect on the ohmic heating efficiency, while factor extraction temperature had a significant effect on ohmic heating efficiency. The highest

ohmic heating efficiency was found in the 60°C NaCl solvent treatment, which amounted to 65.78% (Figure 5). Ohmic heating efficiency increases with the extraction temperature but decreases at 90°C. This may be due to increasing extraction temperatures, so the electrical barrier on the heated material also increases. Therefore, the energy needed to maintain the same level of warming is also increasing, thereby reducing efficiency. The extraction temperature is an important parameter that affects energy consumption [29].

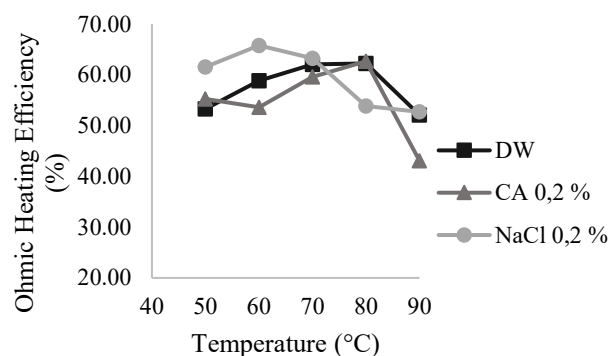


Figure 5. Ohmic heating efficiency analysis (DW=Distilled Water; CA 0.2%=Citric Acid 0.2%; NaCl 0.2%=Sodium Chloride 0.2%)

3.4 Extraction yield

The interaction between solvent type and extraction temperature had no significant effect on extraction yield, while each factor (solvent type and extraction temperature) had a significant effect on extraction yield. The highest yield was found in the 60°C distilled water solvent treatment, which amounted to 4.5%.

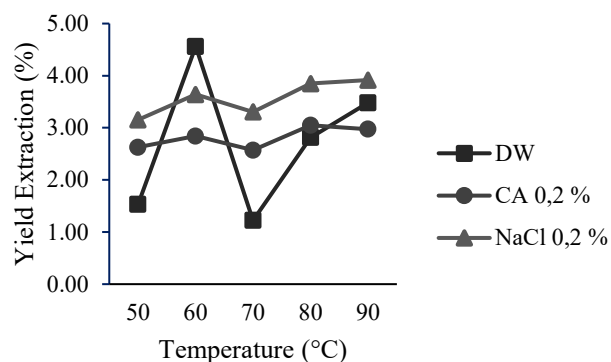


Figure 6. Extraction yield analysis (DW=Distilled Water; CA 0.2%=Citric Acid 0.2%; NaCl 0.2%=Sodium Chloride 0.2%)

In Figure 6, generally the higher the temperature, the higher the extraction yield. This can be because during the ohmic heating process there is rapid heating and electroporation occurs. Heating causes softening of the tissue and electroporation causes the formation of holes in the cells, causing the release of betacyanin compounds from the cells [20, 30]. This is in line with the research of Coelho et al. [31] on the extraction of phenol compounds in tomatoes using ohmic heating. Rapid heating and electroporation can increase

the release of phenol compounds. This is because cellular heat stress causes structural damage, thus increasing the release of phenol compounds.

3.5 Betacyanin content

The interaction between solvent type and extraction temperature had a significant effect on the betacyanin content of the extract (Table 1). The highest betacyanin content was found in the treatment of distilled water solvent type and 60°C extraction temperature, which amounted to 68.80 g/kg and was significantly different from other treatments.

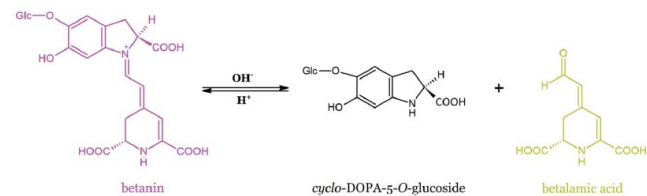
Table 1. Betacyanin content of extract

Treatment	Betacyanin Content (g/kg)
Distilled water, 50°C	42.76 ± 1.23 bc
Distilled water, 60°C	68.80 ± 2.50 a
Distilled water, 70°C	33.26 ± 3.10 de
Distilled water, 80°C	34.86 ± 0.36 bcde
Distilled water, 90°C	34.19 ± 3.58 cde
Citric acid 0.2 %, 50°C	40.09 ± 2.77 bcd
Citric acid 0.2 %, 60°C	43.51 ± 4.57 b
Citric acid 0.2 %, 70°C	35.69 ± 2.00 bcde
Citric acid 0.2 %, 80°C	36.06 ± 5.51 bcde
Citric acid 0.2 %, 90°C	28.25 ± 2.99 e
NaCl 0.2 %, 50°C	37.01 ± 0.75 bcde
NaCl 0.2 %, 60°C	43.95 ± 2.78 b
NaCl 0.2 %, 70°C	34.95 ± 2.33 bcde
NaCl 0.2 %, 80°C	43.07 ± 1.11 bc
NaCl 0.2 %, 90°C	36.86 ± 5.55 bcde

The displayed value was mean ± standard deviation (n=3). The values followed by different letters showed a significant difference (p<0.05).

For other treatments, it can be seen that betacyanin content increased up to a temperature of 60°C, and generally decreased above a temperature of 60°C (Figure 3). However, the decrease in betacyanin content in various types of solvents and extraction temperatures of 50-80°C was not significantly different. This is because the extraction process using the ohmic heating method can heat food ingredients at a fast rate so as to avoid excessive thermal damage to labile substances such as pigments [9]. Extraction using ohmic heating takes place faster than conventional extraction. This can be caused by increasing the permeability of the cell membrane in the sample faster than conventional heating, so that the mass transfer rate of extracted substances from the sample to the extraction medium also takes place faster [20].

In conventional extraction methods, the extraction process above 60°C causes betalain pigments (including betacyanin) to undergo hydrolytic degradation due to heat exposure [32]. Betacyanin is a heat-labile pigment. Increased temperature causes betanin and isobetanin (a group of betacyanin) to be broken down into betalamic acid and cyclo-DOPA (Figure 7) [22, 33].



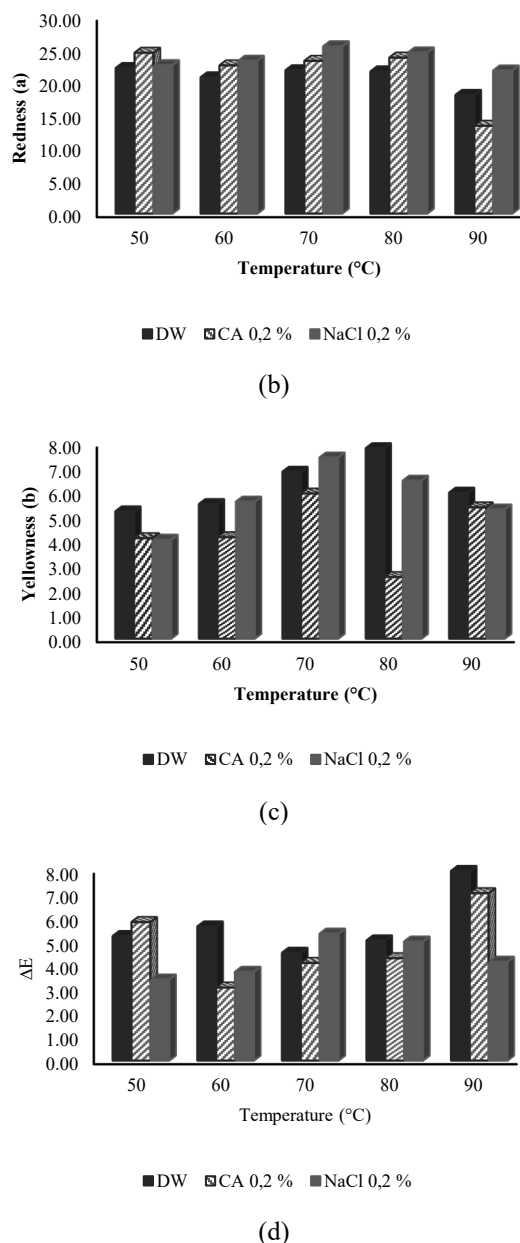


Figure 9. Color analysis of the extract: a. Lightness (L), b. Redness (a), c. Yellowness (b), d. ΔE

4. CONCLUSIONS

The results showed that the interaction between solvent type and extraction temperature significantly affected betacyanin content. Each factor (solvent type and extraction temperature) had a significant effect on the heating time, electrical conductivity, and extraction yield. Ohmic heating can be used for the extraction of betacyanin pigment from red dragon fruit peel. The use of ohmic heating can shorten the extraction time because the heating rate is fast. The presence of acids and salts used as solvents can increase electrical conductivity and shorten extraction time. Needs further research of betacyanin extraction using ohmic heating in pilot plant scale.

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NOMENCLATURE

I	electrical current, A
L	distance between the electrodes, m
A	cross sectional area of the electrode, m ²
V	voltage (V)
m	mass of sample (red dragon fruit peel and water) (kg)
T _i	initial temperature of the sample (°C)
T _f	final temperature of the sample (°C)
t	heating time, s
W ₁	weight of dragon fruit peel, g
W ₂	weight of dried pigment extracts, g

BC	Betacyanin Content, g/kg	L_i, a_i, b_i	sample color after extraction process
A	maximum absorbance value (537 nm) corrected by absorbance at wavelength (600 nm)	σ	electrical conductivity, S/m
V	volume of extract, mL	ϵ	Molar extinction coefficient of betanin (65,000 L mol ⁻¹ cm ⁻¹)
DF	Dilution Factor	Subscripts	
MW	molecular weight of betanin (550 g/mol)	DW	Distilled Water
L	cuvette thickness (1 cm)	CA 0.2%	Citric Acid 0.2%
W	weight of extracted material (fresh state) (g)	NaCl 0.2%	Sodium Chloride 0.2%
L_0, a_0, b_0	sample color before extraction process		