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Food and Drug Safety
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ARTICLE

Development Technology of Robusta Coffee Beans Decaffeinated with Controlled Maceration Using an Automatic Reactor

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ABSTRACT

Decaffeinated coffee has developed into a commercial product of high economic value. Health reasons factor into consumer preference for decaffeinated coffee. The decaffeination process generally uses simple tools and methods, so the process becomes less effective and takes a longer time. Decaffeination of controlled maceration method with automatic reactor can optimize the decaffeination process or decreasing of caffeine content inside the beans. This research aims to determine the characteristics of lightness, caffeine content, pH, sensory and the best treatment from various time and temperature treatments of robusta coffee bean decaffeination. The time treatments used are 2 hours, 4 hours, and 6 hours. While temperature treatments used are 45°C, 60°C, and 75°C. The results of this research show that by increasing the length of time and temperature used in the decaffeination process, the lightness and caffeine content were decreased but the pH of decaffeinated robusta coffee was increased. Decaffeinated robusta coffee with a treatment time of 6 hours and a temperature of 75°C has the highest pH value (7,01), the lowest lightness value (38,1) and caffeine content (0,33%). The best treatment of decaffeinated robusta coffee beans by controlled maceration method using an automatic reactor is the 2-hour treatment and the temperature treatment is 75°C.

Keywords: Decaffeinated Coffee; Controlled Maceration; Caffeine Content

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ARTICLE INFO
Received:30 March 2025 | Revised: Day 17 June 2025 | Accepted: 10 July 2025 | Published Online:20 July 2025
DOI: https://doi.org/10.55121/fds.v2i2.410

CITATION
Afriliana, A., Yunifa, N.R., Diniyah, N., et al., 2025. Development Technology of Robusta Coffee Beans Decaffeinated with Controlled Maceration Using an Automatic Reactor. Food and Drug Safety. 2(2): 33–44. DOI: https://doi.org/10.55121/fds.v2i2.410

1. Introduction

The current production of low-caffeine coffee, which is expanding, has led to its higher economic value than coffee with high caffeine content [1]. Increasingly, consumers are choosing low-caffeine coffee for health reasons [2]. This is because many individuals are aware of the negative effects of caffeine, and the industry has been producing low-caffeine coffee. Low to medium doses of caffeine may cause increased alertness, energy, and ability to concentrate, while higher doses may have negative effects such as anxiety, restlessness, insomnia, and increased heart rate [3]. The fact that caffeine has both positive and negative effects, it is necessary to decaffeinate coffee according to established guidelines.

One of the steps that can be taken to reduce the negative effects of caffeine is the decaffeination process. Decaffeination is a method to reduce the caffeine content in coffee beans. This process is carried out using organic, non-organic materials, and enzymes. The decaffeination process produces coffee with a caffeine content of 0.1–0.3% [4].

The decaffeination process is generally carried out by extraction and using organic solvents. Dimethyl ether, alcohol, acetone, chloroform, and water are some of the solvents that can be used to reduce caffeine levels. During decaffeination, the main flavor components can be removed, especially when using less specific solvents [5]. The choice of solvent in the decaffeination process is very important for coffee flavor. In this study, the direct decaffeination process utilizes water as the solvent instead of organic solvents. This choice is due to the fact that organic solvents can leave significant residues on the coffee beans and impart an unwanted solvent like aroma [6]. Caffeine is soluble in water, and its solubility increases with increasing water temperature. The solubility of caffeine in water is 0.6% at 0°C (32°F), 13% at 25°C (77°F) & 66.7% at 100°C (212°F) [7]. Temperature affects the solubility of caffeine in water and caffeine is easily soluble in hot water but slightly soluble in cold water [8]. The water solvent was chosen because of its high level of polarity, its easy availability and relative safety [9].

Decaffeination can be done through various extraction methods, including maceration, soxhletation, percolation, and reflux methods. Maceration is one of the commonly used cold extraction methods for decaffeination. This method is done by soaking and using room temperature (20–25°C). Maceration at room temperature causes the extraction process to be less complete so that the caffeine compound becomes less dissolved. Therefore, temperature modification is needed to optimize the extraction process. Increasing the temperature used will make the diffusion process that takes place greater, so that the extraction process will run faster [10]. The temperatures used in the decaffeination process in this study are 45°C, 60°C, and 75°C. Research on the decaffeination of robusta coffee beans using a controlled maceration method using water solvent in an automatic reactor with variations in temperature and extraction time needs to be done. The results of this study are expected to simplify and optimize the results in the robusta coffee decaffeination process.

2. Preparation of Decaffeinated Coffee with Water

The decaffeination process begins with measuring 1 kg of dry robusta coffee green beans, then steaming using water at a temperature of 100°C for 3 hours. The next step is maceration with water, with the temperatures used are 45°C, 60°C, and 75°C. Maceration is carried out with a variation in the length of time, which is 2 hours, 4 hours, and 6 hours. Meanwhile, the control sample was not treated with steaming or the decaffeination process. The next step is drying using natural sunlight.

3. Materials and Methods

3.1. Tools and Materials

Tools used for the coffee decaffeination process are an automatic reactor with a capacity of 5 kg, digital scales, a grinder (FOMAC), 60 mesh sieve, a basin, a pan, a stove, a spatula, and a pan. The tools used for analysis are glassware (Pyrex), UV-Vis spectrophotometry (Shimadzu UV-1800), pH meter (PH818), analytical balance (KERN), color reader (Konica Minolta CR-20), hot plate (Labtech

Daihan MSD 20D), magnetic stirrer (IKA 3672000), waterbath (Labtech), small spoon, small glass, and tray. The materials used in the study were green bean robusta coffee, water, $CaCO_3$, distilled water solvent, chloroform, filter paper, caffeine standard, label paper, aluminum foil and tissue.

3.2. Research Designs

The method used in the research is ANOVA and there are two factors, namely length of time (A) and temperature (B). The length of time consists of 2 hours (A1), 4 hours (A2), and 6 hours (A3); and temperatures of 45℃ (B1), 60℃ (B2), and 75℃ (B3). Each treatment was repeated twice.

3.3. Roasting

Decaffeinated robusta coffee beans are roasted using a temperature of 170°C and can be stopped when the first crack of coffee beans occurs or about 15 minutes of roasting process. The next step is mashed coffee beans using a grinder to make it medium fine size. The last step is sieving using a 60-mesh sieve so that the size of the coffee becomes uniform.

3.4. Determination of Lightness (L*)

The tool used for color measurement is a color reader. Preparation of materials for color measurement, namely decaffeinated robusta coffee beans. There is one measurement system on the screen when activating the device and then placing the color reader on white paper for the color target (Lt, at, bt). After that, placing the device on the sample with 5 different points and the values of L*, a*, and b* are known. The L* value indicates the brightness level with values ranging from 0 for black to 100 for white. The a* value indicates the color parameter between red (+) to green (-) and the b* value indicates the color parameter between yellow (+) to blue (-).

3.5. Determination of Caffeine Content

Preparation of Caffeine Standard Curve

The caffeine standard was weighed as much as 50 mg and put into a 50 mL measuring flask to make a mother solution with a concentration of 1 mg/mL. The addition of distilled water up to the limit mark and the solution was homogenized so that the caffeine book solution was obtained. Making a standard curve is the second stage to measure caffeine levels. Taking 2.5 mL of the parent solution and put into a 25 mL volumetric flask. Dilution with the addition of distilled water until the limit mark and homogenized, so as to obtain a standard solution of 100 mg/L (100 ppm). Furthermore, taking 0.05; 0.15, 0.3, 0.6, 0.9 mL of 100 ppm caffeine standard solution and each dilution into 5 mL of distilled water. The concentration of the standard solution obtained is 0.5, 1.5, 3, 6, 9 mg/L respectively [11].

Test for Caffeine Content of Decaffeinated Robusta Coffee

The caffeine quantitative test was carried out by measuring 1 g of decaffeinated robusta coffee and putting it into a beaker glass. Next, add 150 mL of hot distilled water to dissolve the material with stirring. The coffee solution was filtered through a funnel using filter paper into an Erlenmeyer, then 1.5 g of calcium carbonate ($CaCO_3$) and coffee solution were put into a separating funnel with a capacity of 150 mL. Furthermore, the extraction was carried out 4 times each with the addition of 25 mL of chloroform. The filtrate was taken and the extract (chloroform phase) was evaporated using a rotary evaporator until the chloroform evaporated completely. The solvent-free caffeine extract was put into a 100 mL volumetric flask, diluted with distilled water until the limit mark and homogenized. Next, the levels were determined by UV-Vis spectrophotometry at a wavelength of 275 nm [11].

3.6. Determination of pH

The pH meter was calibrated by putting the electrode in a pH 7 buffer solution then rinsed with distilled water and dried using tissue paper. The sample to be used is measured as much as 5 g, mashed using a pestle and mortar and then mixed with 5 mL of distilled water (1:1). The calibrated electrode is inserted into the sample. The pH value will be seen on the pH meter a few moments after the pH meter shows a constant number [12].

3.7. Determination of Sensory Hedonic

The hedonic test involved 25 untrained panelists. The panelists were then given explanations to recognize certain properties related to the hedonic test. In an effort to avoid the influence of panelists' opinions on the samples, the presenter presented the samples randomly by providing a five-digit label. Panelists were asked to test the quality level of the product's sensory attributes including color, taste, and aroma. Panelists gave a score with a range of 1 to 7, that is, very dislike (1), dislike (2), somewhat dislike (3), neutral (4), somewhat like (5), like (6), and very like (7) [13].

3.8. Determination of Effetiveness (De Garmo)

This process involves assigning a weighted value (BN) to each parameter, measured on a relative scale between 0 and 1. The normalized weight varies based on the importance of each parameter, which results as a consequence of a particular treatment. The variables being tested are divided into two categories, Group A which includes parameters with increasing means indicating better performance, and Group B which consists of parameters with decreasing means indicating better results (De Garmo, 1994 at Nafi et al., 2015). The effectiveness value (NE) of each variable is determined using the formula:

$$\textit{Weight Value (NB)} = \frac{\textit{Treatment Score}}{\textit{Total Weight}} \quad (1)$$

$$\textit{Effectiveness value (NE)} = \frac{Np - Ntj}{Ntb - Ntj} \quad (2)$$

$$\textit{Product Value (NP)} = NE \times NB \quad (3)$$

where: *NE* = Effectiveness Value; *NB* = Weight Value; *Np* = Treatment Value; *Ntj* = Worst Value; *Ntb* = Best Value.

4. Results and Discussion

4.1. Lightness of Decaffeinated Robusta Coffee

The lightness value of robusta coffee beans decaffeinated by controlled maceration method using automatic reactor is shown in **Figure 1**. Based on **Figure 1** shows that the lightness of decaffeinated robusta coffee beans decreases with increasing temperature and maceration time. The control sample (without treatment) had a lightness value of 51.23 and the lightness value of the coffee bean color decreased to 38.10 at the end of the process. The difference in treatment time and temperature in the controlled maceration decaffeination process using an automatic reactor had a significant effect ($p < 0.05$) on lightness (**Table 1**). The decaffeination process using water solvents causes the lightness value of coffee beans to decrease. The higher the process temperature used will produce coffee beans with a smaller brightness value [14]. The heat applied during the decaffeination process can break peptide bonds between amino acids in proteins or hydrogen bonds between acids and other compounds [15]. The breaking of peptide bonds or hydrogen bonds and the longer the decaffeination time, the more amino acids free in the coffee beans [16]. The increase in free amino acids and reducing sugars will cause the color of coffee beans to darken, marked by a decrease in brightness value.

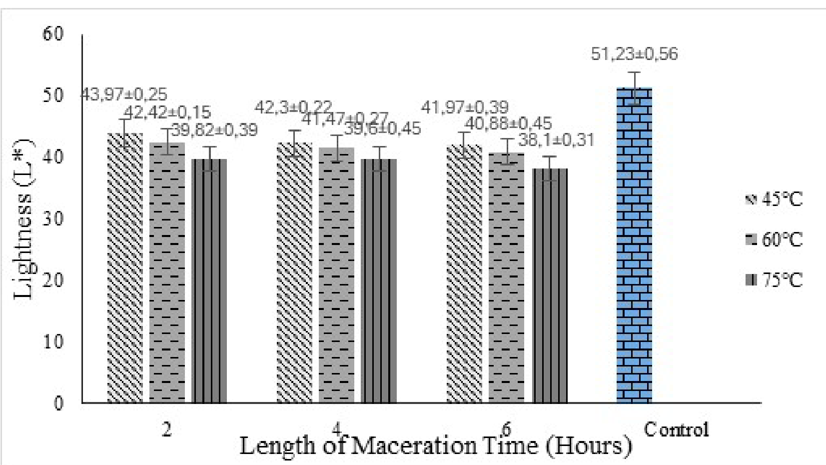

Figure 1. Lightness of decaffeinated robusta coffee beans with controlled maceration.

Table 1. Anova test results for lightness parameters.

Lightness Value				
Temperature (°C)	**Extraction Time (h)**			
	2 Hours	**4 Hours**	**6 Hours**	**Control**
45°C	43.97±0.25^{f}	42.3±0.22^{e}	41.97±0.39^{de}	51.23±0.56
60°C	42.42±0,15^{e}	41.47±0.27^{cd}	40.88±0.45^{c}	
75°C	39.82±0.39^{b}	39.6±0.45^{b}	38.1±0.31^{a}	

Note: Similar letters indicate no significant differences between samples/levels, Duncan's test with a 5% significance level.

4.2. pH Value of Decaffeinated Robusta Coffee

The degree of acidity (pH) is one of the important factors that need to be considered in coffee. The pH value contained in coffee is formed from the acid content in it such as formic acid, acetic acid, oxalic acid, citric acid, lactic acid, malic acid and quinic acid. Robusta coffee has an acidic pH of 5.25–5.40 [17]. The pH value of robusta coffee beans decaffeinated by controlled maceration method using an automatic reactor (**Figure 2**).

Based on **Figure 2**, the pH value of decaffeinated robusta coffee beans has a range of 6.25–7.01. The highest pH value is owned by robusta coffee beans (A3B3) treated for a length of time of 6 hours and a temperature of 75°C, with a value of 7.01. The difference in treatment time and temperature in the controlled maceration decaffeination process using an automatic reactor had a significant effect ($p < 0.05$) on pH value (**Table 2**). The increase in pH value in coffee beans is caused by several organic acids dissolving during the decaffeination process [18]. This is because during the decaffeination process, the coffee beans experience enlarged pores so that water can easily dissolve and evaporate several acidic compounds in the coffee beans. Chlorogenic acid released from caffeine is followed by the decomposition of chlorogenic acid into organic quinate compounds and dissolves in water so that the pH in robusta coffee beans will increase during the decaffeination process [19]. The longer the maceration extraction process, the higher the pH will increase. The acids contained in coffee beans play a role in the formation of the sour taste of coffee [14]. Decreasing acid levels will cause a decrease in the taste of coffee. Coffee acidity that is too low or too high can disrupt the overall balance of coffee flavor.

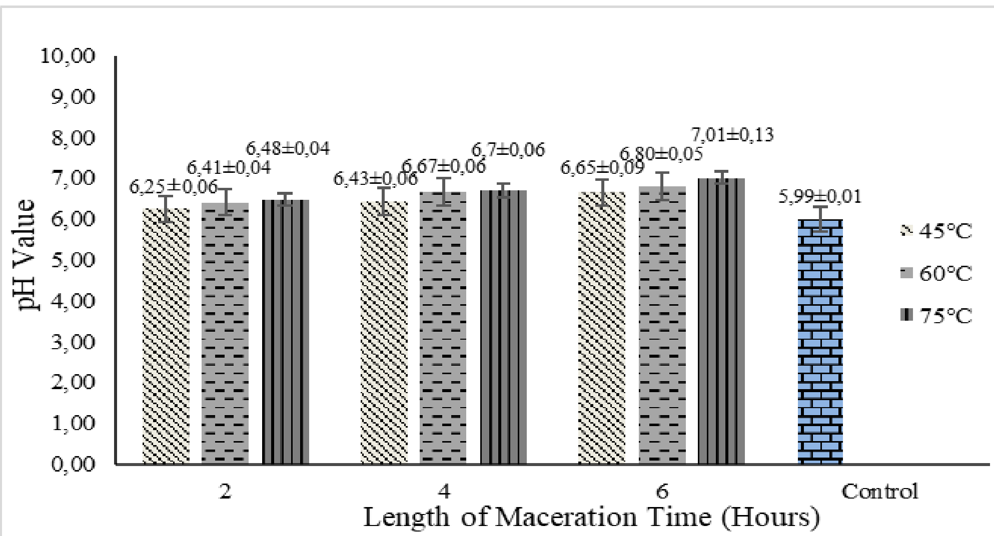

Figure 2. pH value of decaffeinated robusta coffee beans with controlled maceration.

Note: Similar letters indicate no significant differences between samples/levels, Duncan's test with a 5% significance level.

Table 2. Anova test results for pH value parameters.

pH Value				
Temperature (°C)	Extraction Time (h)			
	2 Hours	4 Hours	6 Hours	Control
45°C	6.25±0.06[a]	6.43±0.06[ab]	6.65±0.09[bc]	5.99±0.01
60°C	6.41±0.04[ab]	6.67±0.06[abc]	6.8±0.05[cd]	
75°C	6.48±0.04[abc]	6.7±0.06[bc]	7.01±0.13[d]	

4.3. Caffeine Content of Decaffeinated Robusta Coffee

Caffeine (1,3,7-trimethylxanthine) is a secondary metabolite compound of the alkaloid group found in almost all coffee. The calibration curve equation can be determined by making a standard caffeine solution with a range of concentrations. The calibration curve equation was determined by preparing a standard caffeine solution with a concentration range of 5–9 ppm and measuring its absorbance using UV/Vis spectrophotometry at λ 275 nm, resulting in the regression equation $y = 0.0227x + 0.0002$. The caffeine content of deccafeinated robusta coffee beans can be determined using the calibration curve (**Figure 3**). Based on statistical analysis using Two-Way ANOVA (**Table 3**) with a significance level of a 5%, there is a significant effect ($p \leq 0{,}05$) on the differences in treatment duration and temperature during the controlled maceration decaffeination process using an automatic reactor—results of analysis of the content of caffeine in shown in **Figure 4**. In the research results, the sample with the highest caffeine content was the control (without treatment) which was 1.64% and the lowest caffeine content was obtained by sample A3B3 (time 6 hours and temperature 75°C) with a caffeine content of 0.33%. Ground coffee that has a caffeine content of 0.1–0.3% can be categorized as low caffeine coffee [4].

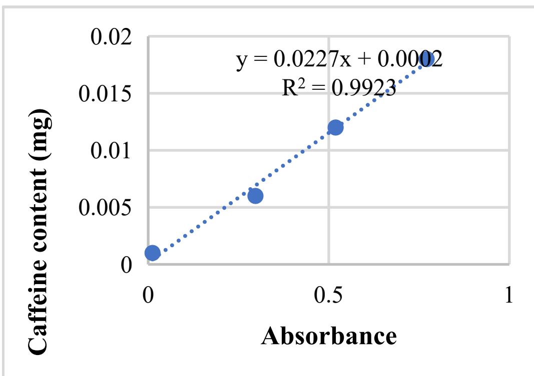

Figure 3. The calibration curve of caffein standart.

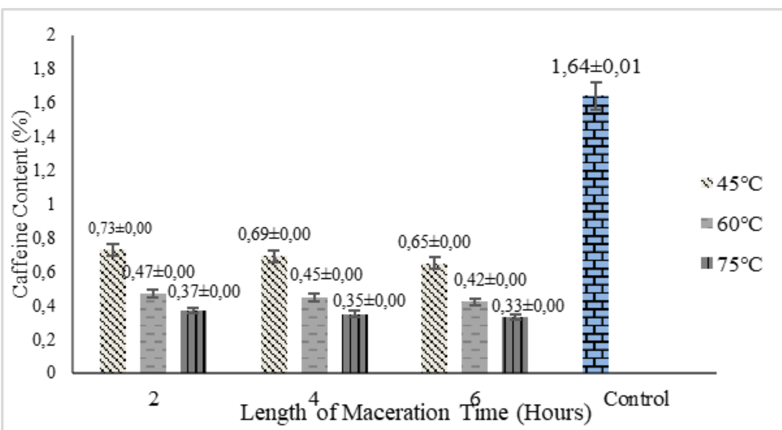

Figure 4. Caffeine content of decaffeinated robusta coffee beans with controlled maceration.

Table 3. Anova test results for caffeine content parameter.

Caffeine Content (%)				
Temperature (°C)	**Extraction Time (h)**			
	2 Hours	**4 Hours**	**6 Hours**	**Control**
45°C	0.73±0.001[h]	0.69±0.002[g]	0.65±0.002[f]	
60°C	0.47±0.001[e]	0.45±0.002[d]	0.42±0.00[c]	1.64±0.01
75°C	0.37±0.001[b]	0.35±0.00[a]	0.33±0.00[a]	

Note: Similar letters indicate no significant differences between samples/levels, Duncan's test with a 5% significance level.

The caffeine content decreases with increasing temperature used because heat can break down the bonds of complex caffeine compounds which occur more quickly. The higher the extraction temperature used can result in lower levels of caffeine produced [20]. The higher the temperature, the looser the mass density of both water as a solvent and coffee bean solids so that they have a larger empty space between molecules so that the higher the temperature, the more caffeine is extracted [21]. The maceration method is carried out by soaking the sample in a solvent for a certain period of time. Maceration time that is too short causes the extracted compound to not be maximized. Extraction time that is too short will cause the compound not to be extracted properly [10]. This is in accordance with the results of the caffeine content obtained in the 2-hour treatment sample which had higher results than the 4-hour and 6-hour treatment.

4.4. Sensory Hedonic

4.4.1. Color

Color is the first parameter used by panelists to determine the preference value. Color can provide information on the characteristics of a food product and become the appearance of a product to increase its appeal. The level of coffee roasting has different color changes and the level of color affects the taste of the coffee [22]. The color preference value of decaffeinated robusta coffee can be seen in **Figure 5**. The results of the chi-square statistical analysis with a significance of α 5% showed a significant effect ($p \leq 0.05$) on the color preference value of decaffeinated robusta coffee. The highest color preference value was owned by the control sample (without treatment) with a value of 5,76. The difference in color of each treatment can be caused by temperature and time in the decaffeination process. The roasting process can also result in uneven color. In the roasting process, a maillard reaction occurs which forms melanoidin compounds as protein derivative compounds that affect the color of the coffee brew [23].

4.4.2. Aroma

Aroma is an important factor that determines the quality of food products besides taste and color. The aroma character produced by each food product varies according to the type of ingredients used. The aroma of coffee appears due to the evaporation of volatile compounds during coffee brewing and is captured by the human sense of smell [24]. The aroma preference value of decaffeinated robusta coffee can be seen in **Figure 6**.

The results of the chi-square statistical analysis with a significance of α 5% showed a significant effect ($p \leq 0.05$) on the aroma preference value of decaffeinated robusta coffee. Based on **Figure 6**, it shows that the aroma value of decaffeinated robusta coffee produced ranged from 4.2 to 4.92. The highest value was obtained by the sample with a time of 2 hours and a temperature of 45 °C of 4.92. The decrease in flavor value was caused by most of the compounds that were precursors of aroma and taste that dissolved due to the high heat treatment during the decaffeination process so that the coffee that had been decaffeinated would taste bland [14]. Trigonelline is an aroma precursor in coffee and the roasting process will produce volatile compounds [25].

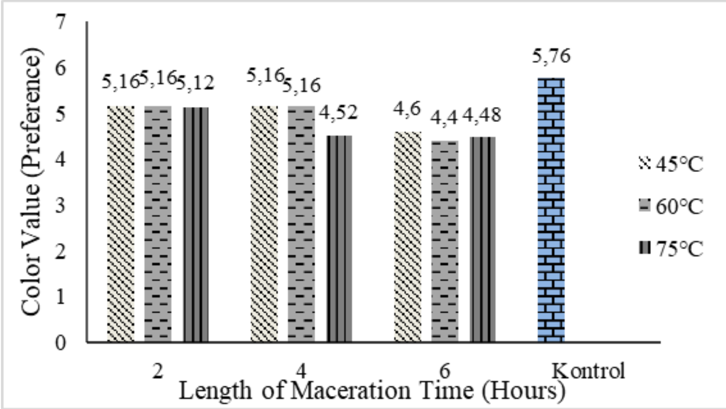

Figure 5. Color preference value of decaffeinated robusta coffee beans with controlled maceration.

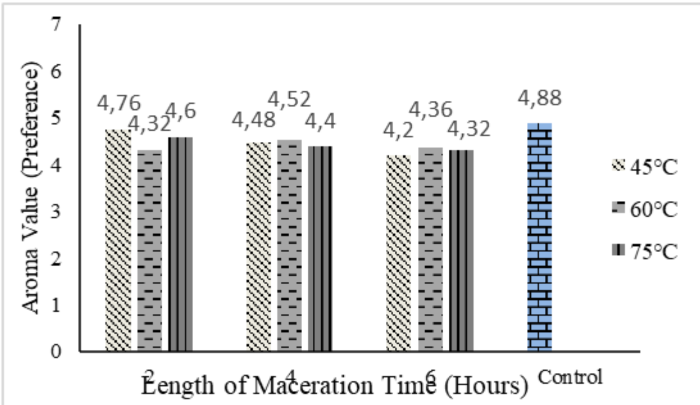

Figure 6. Aroma preference value of decaffeinated robusta coffee beans with controlled maceration.

4.4.3. Flavor

One of the important indicators that determines the quality of food products and can also influence the final decision of consumers to accept or reject a product is taste. Chemical compounds, temperature, and cooking time affect the taste of a product [26]. The taste preference value of decaffeinated robusta coffee can be seen in **Figure 7**.

The results of the chi-square statistical analysis with a significance of α 5% showed a significant effect ($p \leq 0.05$) on the taste preference value of decaffeinated robusta coffee. The highest value was obtained by sample A2B1, which used a time of 4 hours and a temperature of 45°C with a value of 4.2. The decaffeination process will take place quickly with the use of higher temperatures, but high temperatures result in a decrease in taste [4]. Caffeine compounds contribute a bitter taste of between 10–30% of coffee brews. Caffeine affects the taste of coffee brews, especially bitterness [27]. Therefore, the use of a temperature of 45 °C has a taste that is preferred by panelists compared to the use of temperatures of 50°C and 75°C which have a taste that is not too bitter but the bitter taste is still felt. In addition, roasting also affects the taste of coffee because roasting can increase the bitter taste.

4.4.4. Overall

Overall is an assessment used to reflect the overall assessment of the sample felt by the panelists individually [28]. The overall preference assessment of the panelists is an assessment of all observed parameters, including the aroma, taste and color of the decaffeinated robusta coffee brew. The overall preference value of decaffeinated robusta coffee can be seen in **Figure 8**. The results of the chi-square statistical analysis with a significance of α 5% did not show any significant effect ($p \leq 0.05$) on the overall preference value of decaffeinated robusta coffee. The higher the overall value, the more the coffee will meet the expected criteria [27]. In this study, the overall attribute assessment received the highest value in sample A2B1 with a treatment time of 4 hours and a temperature of 45°C.

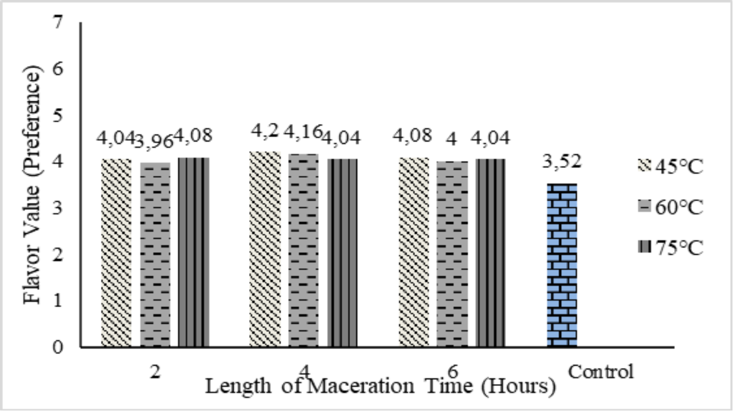

Figure 7. Flavor preference value of decaffeinated robusta coffee beans with controlled maceration.

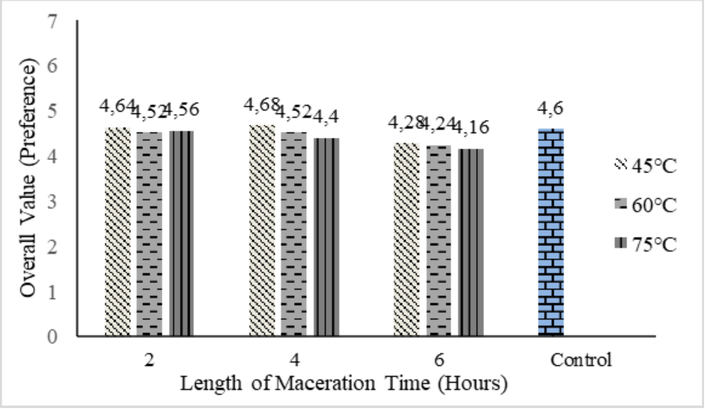

Figure 8. Overall preference value of decaffeinated robusta coffee beans with controlled maceration.

4.5. Effectiveness Value (De Garmo)

The total treatment value (NP) produced for decaffeinated robusta coffee can be seen in **Figure 9.** Based on the calculation of the treatment value (NP) for the brightness, caffeine content, pH and sensory parameters of decaffeinated robusta coffee in **Figure 9**, it can be seen that sample A2B1 (treatment time 2 hours and temperature 75°C) is the best treatment with a treatment value of 0.75. The characteristics of the test results from the treatment time 4 hours and temperature 45°C in the decaffeination process are brightness 39.82; pH 6.48; caffeine content 0.37%; aroma (hedonic) 4.6; color (hedonic) 5.12; taste (hedonic) 4.08; and overall (hedonic) 4,56.

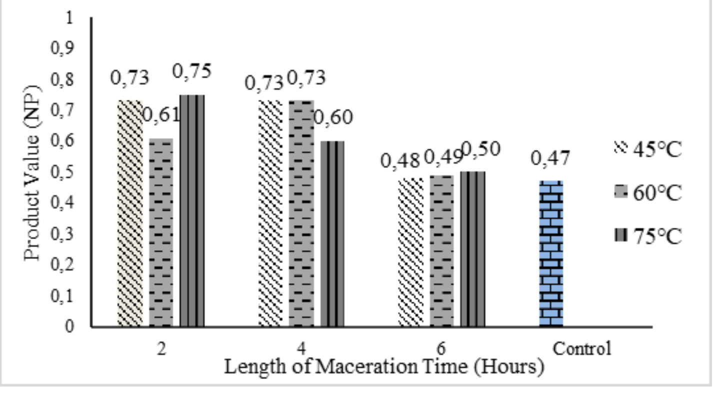

Figure 9. Effectiveness value of decaffeinated robusta coffee beans with controlled maceration.

5. Conclusions

Water-based decaffeination methods are known as a safe and environmentally sustainable approach, as they do not involve hazardous chemicals and preserve the integrity of the natural components in coffee beans. However, the use of an automatic reactor in this process requires a relatively long time, resulting in a significant increase in electricity consumption. On an industrial scale, this limitation can reduce production process efficiency. Therefore, using alternative solvents such as ethyl acetate is a strategic consideration to accelerate the decaffeination process while maintaining the quality and safety of the final product per applicable regulatory standards. Increasing the length of time and temperature used in the decaffeination process with controlled maceration using an automatic reactor, the brightness value and caffeine content will decrease, while the pH value will increase. In the sample treatment time of 6 hours and temperature of 75℃, the lowest brightness value (38.1), the lowest caffeine content (0.33%), and the highest pH value (7.01) were obtained. The decaffeination process of robusta coffee beans affects the level of panelists' preference for various parameters. The use of a treatment time of 4 hours and a temperature of 45℃ was most preferred by panelists from the taste parameters (4.2) and overall (4.68). While decaffeinated robusta coffee was preferred by panelists in the treatment time of 2 hours and 45℃ in terms of aroma (4.88), and without treatment in terms of color (5.76). The best treatment of decaffeinated robusta coffee beans using the controlled maceration method using an automatic reactor was in the treatment time of 2 hours and a temperature of 75℃ with brightness characteristics of 39.82; pH 6.48; caffeine content 0.37%; aroma (hedonic) 4.6; color (hedonic) 5.12; taste (hedonic) 4.08; and overall (hedonic) 4.56.

Author Contributions

Conceptualization, Design the experiments, writing, and editing paper, A.A.; Analysis some data, writing original draft paper, E.H., and N.R.; Review paper, and checking data, N. D., G.C.,and M. B.; Supply material for research (arabica decaf), H. K. All authors have read and agreed to the published version of the manuscript.

Funding

This research was supported by the research and community service institution (LP2M), University of Jember, through internal research funding, no 7554/UN25/KP/2024.

Institutional Review Board Statement

Ethical review and approval were waived for this study due to "Not applicable" for studies not involving humans or animals.

Informed Consent Statement

Not applicable.

Data Availability Statement

The data is unavailable due to privacy

Acknowledgments

We would like to say thank you so much for the research support from the University of Jember. We also thank to National Institute of Materials Science (NIMS), Japan, for the convenience of the research discussion.

Conflicts of Interest

The authors declare no conflict of interest

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