

Effect of Ethyl Acetate Concentration on Decaffeination of Robusta Coffee From Lombok, Indonesia

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ABSTRACT

Decaffeination is an innovative process in coffee processing to reduce caffeine content while maintaining coffee quality. This study aimed to observe the effect of various concentrations of ethyl acetate solvent on the decaffeination process of Robusta (*Coffea canephora*) from Lombok and its impact on coffee quality. The decaffeination process was carried out by soaking coffee beans at 50°C for 36 hours with the observed parameters consisting of water content, protein content, ash content, pH, total titratable acidity, and caffeine content. Data were analyzed using ANOVA to see the significance between treatments. The results of this study showed that different concentrations of ethyl acetate had a significant effect on the parameters of water content, protein content, total titratable acidity and protein content while not significant for ash content and pH. A 10% ethyl acetate concentration was identified as the most effective treatment, reducing caffeine content to 0.36%, while preserving protein content (10.47%), total titratable acidity (2.13%), moisture content (9.56%), ash content (3.31%), and pH (5.84). The results of this study indicate that the potential use of ethyl acetate as a solvent in the decaffeination process can provide an alternative added value to coffee products.

Key words: Coffee quality evaluation; ethyl acetate decaffeination; solvent-based decaffeination; robusta coffee decaffeination; robusta Lombok coffee.

1 INTRODUCTION

Caffeine is a naturally occurring alkaloid compound that is found in coffee. Robusta coffee contains two times more caffeine (1.6-2.4%) than arabica coffee (0.9-1.2%) (Kuncoro et al., 2017). Caffeine is found in the cell membrane of coffee beans, where the cell membrane contains 40% fat, 52% protein, and 8% carbohydrates. The effects of caffeine in humans include stimulant effects on the central nervous system, increased concentration, and the alleviation of sensations of drowsiness (Mendoza et al., 2023). However, it is important to consider that excessive caffeine consumption can lead to adverse effects including heart palpitations, gastric disorders, and tremors. To address the health implications of caffeine intake, a potential solution is to reduce caffeine levels in coffee by decaffeinating coffee beans (Khairun; Andriansyah; Emma, 2021). Caffeine levels in coffee can be reduced by breaking down the components contained in the membrane. Proteolytic enzymes facilitate the dissolution of caffeine by hydrolysing proteins and softening and breaking components in cells (Rosalinda; Febriananda; Nurjanah, 2021).

Decaffeination is one of the methods employed to reduce caffeine levels found in coffee beans (Utama et al., 2022). The decaffeination process of coffee beans can be achieved through soaking or a combination of steaming and dissolving. The utilisation of various organic and non-organic solvents in the solvent-based decaffeination

of robusta coffee is a notable feature. The predominant decaffeination process in the industry is solvent-based. The most commonly used solvents in this process include acetone, chloroform, benzene, ethyl acetate, ethyl alcohol, ethyl ether, methylene chloride, dichloromethane, and trichloroethylene. The main advantage of solvent-based decaffeination is its high selectivity, which preserves the coffee's distinctive taste and aroma. The most common solvent employed in this process is ethyl acetate (Shofinita et al., 2024). This solvent is characterized by its colorlessness and its capacity to carry a distinctive aroma. The utilisation of ethyl acetate as a solvent in the decaffeination process offers several advantages, including its volatility, non-hygroscopic properties, and low toxicity (Putri et al., 2017). The utilisation of ethyl acetate as a solvent in the decaffeination process has been shown to facilitate the dissolution of caffeine compounds present in coffee beans (Artini; Astuti; Warditiani, 2013). Shofinita et al. (2024) have demonstrated that the solvent-based decaffeination process on coffee beans can maintain the flavor, aroma, and color of coffee.

The findings of Wijaya and Yuwono (2015) indicated that robusta coffee decaffeination using ethyl acetate at a concentration of 15% was the most effective treatment, reducing the caffeine content in Robusta coffee from 2.30% to 0.14%. Another study conducted by Widyotomo et al. (2009) examined the decaffeination of Robusta coffee in a single

column reactor with a solvent concentration of 10% ethyl acetate, resulting in a reduction in caffeine levels from 2.35% to 0.3%, with a dissolution time treatment of 8-10 hours at 90-100 °C or 12 hours at 60-70 °C. Furthermore, Afriliana et al. (2019) reported that the decaffeination process using ethyl acetate in robusta coffee can reduce caffeine to 0.3%.

Robusta coffee plants have been shown to grow at altitudes in the range of 450-850 meters above sea level. Lombok Island has been identified as a region with potential for coffee production in Indonesia, with some plantations situated within the Mount Rinjani Geopark area (Zainuri et al., 2023). To the best of our knowledge, research related to the decaffeination process of robusta lombok coffee using ethyl acetate solvent has never been conducted. The present study aimed to observe the effect of various concentrations of ethyl acetate solvent on the decaffeination process of robusta (*Coffea canephora*) from Lombok and its impact on coffee quality.

2 MATERIAL AND METHODS

2.1 Materials and Tools

The materials used in this study were green bean robusta from several regions in North Lombok (500-1500 meters above sea level), distilled water, ethyl acetate, 0.1N HCl, H_3BO_3 , H_2SO_4 concentrated, BCG-MR indicator, phenolphthalein indicator 1%, calcium carbonate ($CaCO_3$), chloroform, K_2SO_4 , NaOH 0.01 N, NaOH 40%, sodium carbonate (Na_2CO_3) and copper (II) sulfate ($CuSO_4$).

The tools used in this study include the following: a steamer, a 40-mesh sieve, a blender, weighing bottle, cabinet dryer, porcelain cup, separating funnel, desiccator, Erlenmeyer flask, 50-ml and 100-ml beakers, filter paper, Kjeldahl flask, 100-ml volumetric flask, micro water bath, oven, pH meter, knife, UV-Vis spectrophotometer, furnace and analytical balance.

2.2 Decaffeination Process

The procedure for producing decaffeinated robusta coffee follows the modified method of Utama et al. (2022), which comprises several key stages. Initially, the robusta coffee beans utilised in each treatment were weighed to within 125 g. The coffee beans were then subjected to steaming at approximately 100°C for 30 minutes, with the objective of softening the bean tissue to facilitate the subsequent soaking process. Subsequently, the coffee beans were soaked in a micro waterbath at $\pm 50^\circ C$ for 36 hours with the addition of an ethyl acetate solution. Following this, the coffee beans were washed with running water to remove the dissolved caffeine. The coffee beans were then drained to reduce the residual washing water before being dried using a cabinet dryer at 60°C for 5 hours.

2.3 Analysis Method

This study consisted of 6 concentration treatments, namely 0%, 5%, 10%, 15%, 20% and 25%. The parameters observed were caffeine content (Maramis; Citraningtyas; Wehantouw, 2013), protein content (Association of Official Analytical Chemists - AOAC 2001), total titratable acid (Kasim et al., 2020), ash content (Wiyono, 2019), moisture content (Fauzi et al., 2017), and pH value (Saputri et al., 2020). The parameters were analyzed due to their critical role in determining coffee quality. Controls included untreated coffee samples to benchmark changes induced by the decaffeination process.

2.4 Statistical Analysis

Observation data were analyzed by the Analysis of Variance (ANOVA) with *Duncan's Multiple Range Test* (DMRT) at 5% significance level using Co-stat software (version 6.4, CoHort Software).

3 RESULTS

3.1 Caffeine Content

Caffeine is an alkaloid compound naturally found in coffee beans, tea leaves, and cocoa beans. It is a naturally occurring methylxanthine compound (1,3,7-trimethylxanthine) and is classified as a xanthine derivative, which belongs to the alkaloid group (Alawiyah; Rohman; Dan Purwandari, 2020). Additionally, caffeine plays a significant role in the development of flavor in coffee. Caffeine is a compound that function in the generation of stamina and relieve pain such as mild headaches when consumed in low doses. Meanwhile, consuming caffeine in high doses can have an impact on bone and calcium absorption, affect the work of the heart and change behavior (Nofrida et al., 2024) Based on the results of the study, soaking using ethyl acetate solution can reduce caffeine levels in robusta coffee. The results of soaking using ethyl acetate solution on the caffeine content of decaffeinated robusta coffee can be seen in Figure 1.

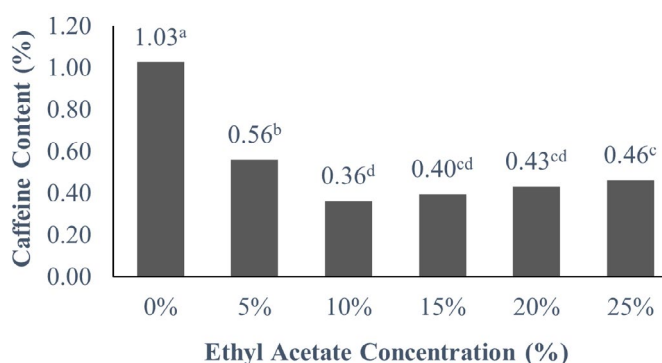


Figure 1: Effect of ethyl acetate concentration on caffeine content of decaffeinated robusta Lombok coffee.

3.2 Protein Content

Most proteins are enzymes or enzyme subunits and are one of the ingredients in coffee beans. Protein is found in the cell membrane by 52%, fat 40% and carbohydrates 8% (Rosmawati, 2013). Based on the results of the study, soaking using ethyl acetate solution had a significant effect on robusta coffee protein content. The results of soaking using ethyl acetate solution on the protein content of decaffeinated robusta coffee can be seen in Figure 2.

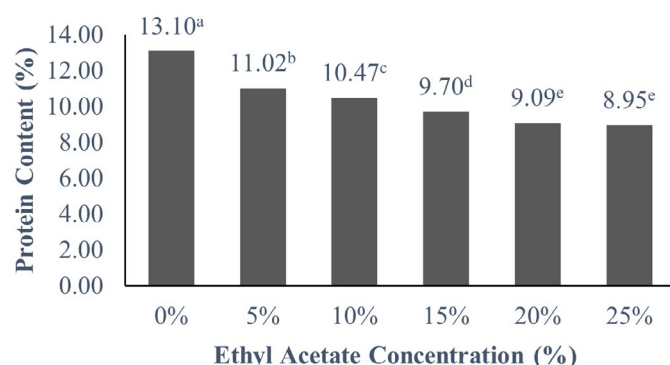


Figure 2: Effect of ethyl acetate concentration on protein content of decaffeinated robusta Lombok coffee.

3.3 Total Titratable Acid

Total titratable acid is organic acids such as citric, malic, lactic, and titrate formed during the fermentation or soaking process in coffee. Robusta coffee contains 0.5-3.5% organic acids, the types of organic acids found in coffee beans are short carbon chain organic acids that can dissolve in water (Kasim et al., 2020). The main organic acids found in coffee beans are oxalic acid, formic acid, lactic acid, acetic acid and citric acid (Towaha; Rubiyo, 2016).

Based on the results of the study, soaking using ethyl acetate solution significantly affects the total titratable acid content of decaffeinated robusta coffee. The results of soaking using ethyl acetate on the total acid content of robusta coffee can be seen in Figure 3.

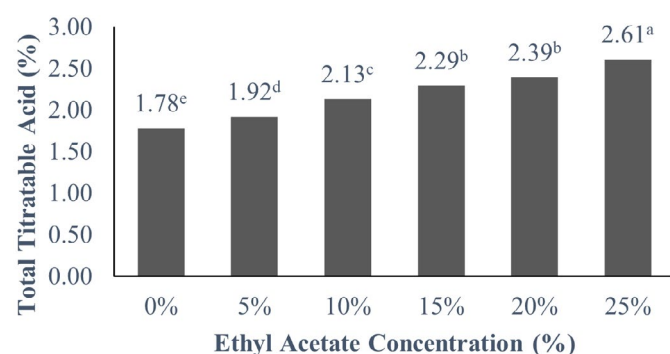


Figure 3: Effect of ethyl acetate concentration on total titratable acid of decaffeinated robusta Lombok coffee.

3.4 Moisture Content

Moisture content is the percentage of water content of a material that can be expressed based on wet weight (*wet basis*) or based on dry weight (*dry basis*). Based on the results of the study, soaking using ethyl acetate solution was able to reduce the water content of robusta coffee beans. The results of soaking using ethyl acetate solution on the water content of decaffeinated robusta coffee can be seen in Figure 4.

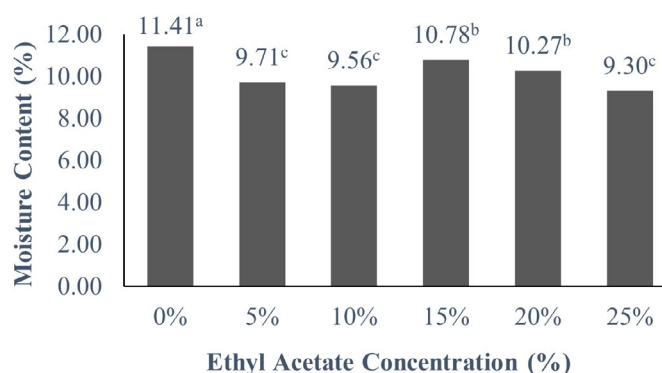


Figure 4: Effect of ethyl acetate concentration on moisture content of decaffeinated robusta Lombok coffee.

3.5 Ash Content

Ash content is the amount of inorganic or mineral components contained in a food. In general, food consists of 96% organic matter and water, while the rest are mineral elements. Based on the results of the study, soaking using ethyl acetate solution did not significantly affect the ash content of decaffeinated robusta coffee. The results of soaking using ethyl acetate on the ash content of robusta coffee can be seen in Figure 5.

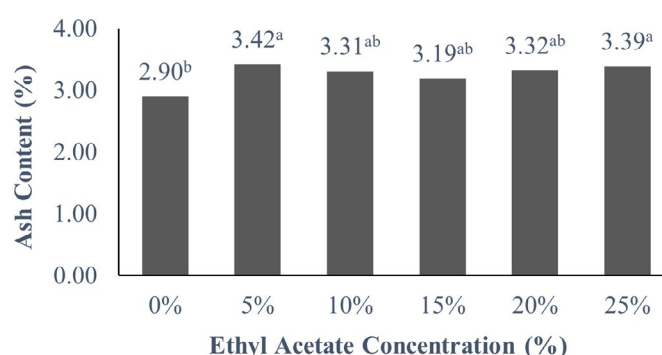


Figure 5: Effect of ethyl acetate concentration on ash content of decaffeinated robusta Lombok coffee.

3.6 Acidity level (pH)

pH is a test conducted to determine the degree of acidity or basicity of a solution. pH is defined as the logarithm of hydrogen ion activity. Based on the results of the study,

soaking using ethyl acetate solution did not significantly affect the pH value of decaffeinated robusta coffee. The results of soaking using ethyl acetate on the pH value of robusta coffee can be seen in Figure 6.

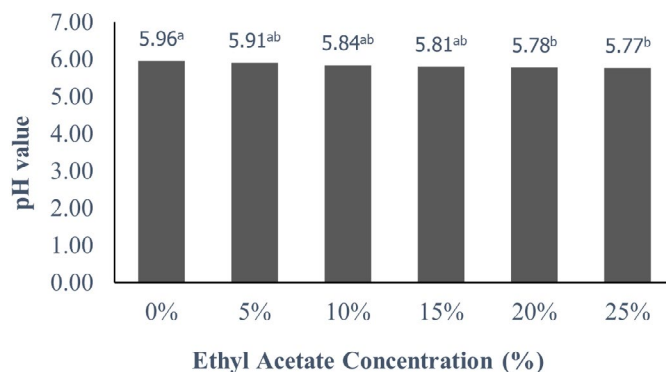


Figure 6: Effect of ethyl acetate concentration on pH of decaffeinated robusta Lombok coffee.

4 DISCUSSION

4.1 Caffeine Content

Figure 1 demonstrates that soaking using ethyl acetate in the decaffeination process leads to a decrease in caffeine levels in robusta coffee beans. According to Saputri et al. (2022), indicate that the decaffeination process employing 10% ethyl acetate concentration results in a substantial decline in caffeine levels, from 2.26% to 0.36%. This reduction can be attributed to the capacity of ethyl acetate to disrupt the molecular structure of caffeine compounds present in coffee beans, thereby facilitating their dissolution and removal during the decaffeination process.

According to Wijaya and Yuwono (2015), the percentage of caffeine content decreases with the length of steaming time and the high concentration of ethyl acetate in the decaffeination process. The longer steaming causes softening of the tissue in the coffee beans, making it easier for ethyl acetate to dissolve caffeine. In this case, caffeine including alkaloids is then in free condition and extracted using organic solvents. Based on research by Nuraeni et al. (2015), the heating process and high temperature can reduce the caffeine levels in coffee, where the higher the temperature and duration of heating, the caffeine content contained in coffee beans will be increasingly decomposed and evaporated. In addition, Afriliana et al. (2019) reported that the application of heat energy has been demonstrated to disrupt the bonds between caffeine and its molecular environment, thereby facilitating its dissolution in ethyl acetate. The physical changes that occur during the steaming of coffee beans, such as volume expansion, serve as the initial phase in the process of softening the coffee bean tissue and reducing the distance between cells. This increased

accessibility facilitates the diffusion of ethyl acetate molecules into the coffee beans, thereby accelerating the dissolution of the caffeine compound.

Suharman and Gafar (2017) explained that steaming or boiling enlarges pore structures in coffee beans and breaks the bonds between caffeine and chlorogenic acid. During steaming, hot steam penetrates the coffee beans, freeing caffeine molecules and facilitating their absorption into solvents. Similarly, Oktadina et al. (2013) found that soaking and washing reduce caffeine levels by removing caffeine detached from chlorogenic acid. This aligns with the data in Figure 1, showing a significant decrease in caffeine content in robusta coffee beans treated with 0% ethyl acetate concentration.

4.2 Protein Content

Figure 2 shows that immersion using ethyl acetate decreased the protein content along with the increase in ethyl acetate concentration used in the decaffeination process. Based on Saputri et al. (2022), using the same type of coffee, the protein content of green bean robusta variety is 14.09%. The soaking process using ethyl acetate caused a decrease in coffee protein content, which is 8.95% in the 25% ethyl acetate concentration treatment and the highest level was in the 0% ethyl acetate soaking treatment, which is 13.10%.

The decrease in protein content in robusta coffee beans due to decaffeination using ethyl acetate solvent may be because the solvent can selectively extract protein from coffee beans. In addition, increasing the concentration of ethyl acetate in the decaffeination process can lead to the precipitation of proteins from the solution. If the protein precipitates along with ethyl acetate, it can lead to a decrease in the protein content in decaffeinated coffee beans.

The decrease in protein content in robusta coffee beans is also thought to be caused by external factors other than the variables studied, namely the steaming and soaking processes during the decaffeination process. The soaking and heating process causes softening of coffee beans by the amount of water that enters the cell structure, resulting in the breaking of bonds in the protein structure (Afriliana et al., 2019).

4.3 Total Titratable Acid

The acidity of coffee is determined by its total titratable acid and pH. This acidity is a significant factor in the perception of coffee flavor, and is one of the primary parameters that affect its quality. The acid composition of coffee affects the sensation of acidity to a certain degree. However, it can also separately or collectively impact the bitterness, astringency, aroma, and color characteristics of the coffee beverage (Da Costa et al., 2024). Figure 3 shows that the addition of ethyl acetate in the soaking process has a significantly different effect on the total titratable acid content of robusta coffee beans. The level of

total titratable acid increased along with the addition of ethyl acetate concentration, where the total acid value obtained ranged from 1.78%-2.61%. The increase in total titratable acid is because ethyl acetate used as a solvent in the decaffeination process can extract and dissolve several other compounds that contribute to coffee acidity such as chlorogenic acid. Along with caffeine, chlorogenic acid can also be dissolved in ethyl acetate and eliminated in coffee beans.

The increase in total titratable acid in robusta coffee beans is thought to be caused by external factors other than the variable being tested, namely the length of soaking during the decaffeination process. The length of soaking time causes lactic acid and organic acids contained in coffee beans to increase so that the value of total titratable acid in coffee increases.

Based on research by Fauzi et al. (2017), the increase in total acid is closely related to the increased activity of lactic acid bacteria. During their growth, these bacteria use carbohydrate sources (sugar) and convert them into organic acids, where the accumulation of acids produced causes an increase in total acid. Therefore, the total acid content of decaffeinated robusta coffee increased.

4.4 Moisture Content

Figure 4 shows that there is a significant decrease in the water content of robusta coffee beans after decaffeination. However, the addition of ethyl acetate in the soaking process did not have a very significant effect on the water content of robusta coffee beans. The lowest water content was found in the 25% ethyl acetate concentration treatment, which amounted to 9.30% and the highest water content was found in the 0% ethyl acetate concentration treatment, which amounted to 11.41%.

The moisture content of coffee beans tends to fluctuate as the concentration of ethyl acetate used in the soaking process increases. During the decaffeination process, coffee beans may also absorb small amounts of ethyl acetate along with caffeine. When the coffee beans are removed from the ethyl acetate solution and dried, it is possible that some of the remaining ethyl acetate is still trapped inside the coffee beans. The concentration of ethyl acetate remaining in the coffee beans may affect the moisture content in the coffee beans.

Moisture content in coffee is generally related to the moisture of the coffee beans and can be influenced by other factors such as drying methods or storage of coffee beans (Pertiwi et al., 2024). The drying method using a cabinet dryer in the decaffeination process of coffee beans can cause fluctuations in the moisture content of the coffee beans, which may be caused by the coffee beans not drying evenly. According to SNI 01-2907-2008, the water content contained in coffee beans should not exceed 12%. This shows that the water content produced in this study has met the established quality standards.

4.5 Ash content

A high ash content may indicate the presence of impurities, while a low ash content may suggest incomplete roasting or substandard quality. Ash content is indicative of the mineral composition of coffee beans, with higher ash content often correlating with higher mineral content. Variations in raw material origin, such as bean maturity and environmental conditions, can lead to differences in ash content (Zainuri et al., 2023). Figure 5 shows that the ash content of robusta coffee beans increased after the decaffeination process and tended to fluctuate as the ethyl acetate concentration increased. This shows that the addition of ethyl acetate in the soaking process did not have a significantly different effect on the ash content of robusta coffee beans. The ash content produced in this decaffeinated robusta coffee ranged from 2.90%-3.42%. The increase in ash content may be due to the fact that ethyl acetate can remain on the coffee beans after the decaffeination process is complete. Ethyl acetate is an organic compound that can burn and produce ash residue when exposed to high heat.

The drying process of coffee beans using a *cabinet dryer* causes the ethyl acetate remaining in the coffee beans due to the decaffeination process to decompose and leave ash residue. However, the addition of ethyl acetate concentration in the coffee decaffeination process did not have a significant effect on the ash content of coffee. Ash content in coffee is generally related to minerals and other inorganic components present in coffee beans.

In the decaffeination process, soaking coffee using ethyl acetate usually does not react with mineral or inorganic components in coffee beans. Therefore, the use of ethyl acetate in the decaffeination process is unlikely to significantly affect the ash content of coffee. Rosalinda, Febriananda and Nurjanah (2021) stated that soaking coffee beans can dissolve water-soluble mineral components, calcium, magnesium, sodium, potassium, chlorine, bicarbonate and sulfate. In addition, the washing process can clean the remains of soluble minerals so that the remaining mineral components are less.

4.6 Acidity level (pH)

In general, high-quality coffee beverages exhibit higher pH values and lower acidity levels. The pH level of coffee is indicative of potential changes in the coffee fruit, such as undesirable fermentation occurring during the pre- or post-harvest phases. These changes can result in defects, leading to a decrease in pH and, consequently, a decline in beverage quality. While acidity is crucial to coffee quality, too strong or dominant acidity can be unpleasant and undesirable for the flavor profile of the beverage (Da Costa et al., 2024).

Figure 6 shows that the addition of ethyl acetate in the soaking process did not give a significantly different effect on the pH value of robusta coffee beans. The pH value of robusta coffee beans decreased with the higher concentration of ethyl acetate added. The pH value produced in this decaffeinated robusta coffee ranged from 5.77-5.96 or acidic. This relationship is inversely proportional to the total titratable acid value, which has been observed to increase with increasing ethyl acetate concentration.

The resulting decrease in pH value due to the decaffeination process can be caused by non-volatile acids contained in coffee beans dissolved into the solvent (ethyl acetate). Ethyl acetate has weak acidic properties that have the ability to acidify the solution and reduce its pH value. The ethyl acetate solvent used in the decaffeination process can cause a decrease in the pH of the coffee solution. This is in accordance with the research of Saloko et al. (2024), the decreasing pH value is influenced by the accumulation of organic acids and an increase in the number of protons H^+ as a result of bacterial metabolism due to the decomposition of amino acids, thus forming acids from lactic acid bacteria and acetic acid.

5 CONCLUSIONS

The caffeine content in decaffeinated robusta coffee beans decreased significantly after soaking in ethyl acetate. A concentration of 10% ethyl acetate effectively reduced the caffeine content to 0.36%. The optimal treatment was soaking robusta coffee beans in 10% ethyl acetate solution, resulting in caffeine content of 0.36%, protein content of 10.47%, total titratable acidity of 2.13%, moisture content of 9.56%, ash content of 3.31%, and pH value of 5.84. The findings of this study suggest that the utilization of ethyl acetate as a solvent during the decaffeination process may offer a novel added value to coffee products.

6 AUTHOR'S CONTRIBUTION

Conceptual Idea: Utama, Q.D.; Zainuri; Paramartha, D.N.A.; Methodology design: Utama, Q.D.; Zainuri; Paramartha, D.N.A.; Data collection: Nurhasyah; Data analysis and interpretation: Utama, Q.D.; Zainuri; Paramartha, D.N.A.; Nofrida, R.; Afriansyah, D., and Writing and editing: Utama, Q.D.; Zainuri; Paramartha, D.N.A.; Rahayu, N.; Pertiwi, M.G.P.; Anggraeni, I.M.D.

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