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The Effect of Coffee Bean Particle Size and Citric Acid Concentration on the Caffeine Content of Arabica Ground Coffee

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A B S T R A C T

Arabica coffee is a coffee commodity that is widely found in Indonesia because it is considered superior in terms of taste. Arabica coffee contains various biochemical compounds, one of which is caffeine, which is known to overcome drowsiness and reduce physical fatigue. However, caffeine use can also lead to negative side effects, such as anxiety, increased blood pressure, and decreased fine motor skills. This study addresses the public demand for lower-caffeine coffee to minimize these side effects. The research focuses on reducing caffeine levels by examining coffee bean size and solvent concentration during decaffeination. The method used was a completely randomized design (CRD) factorial pattern with two factors and two replications. The first factor was coffee bean particle size (4 mesh, 6 mesh, and 8 mesh) and the second factor was the maceration process with citric acid solution concentration (1%, 3%, and 5%). Data were analyzed using Analysis of Variance (ANOVA) and Duncan's Multiple Range Test (DMRT) at the 5% level. The results showed that the treatment of 8 mesh coffee bean size with 5% citric acid solution concentration produced the best caffeine content (1.324 mg/g). In addition, the characteristic values of moisture content (3.436%), ash content (2.065%), and phenol content (29.472 mg GAE/g), were obtained. And organoleptic test results with attributes of fragrant-very fragrant aroma (3.55), blackish-brown-black color (3.65), and bitter-very bitter taste (3.5).

1. INTRODUCTION

1.1. Research Background

Caffeine (1,3,7-trimethyl-xanthine) is a purine alkaloid derived from a secondary metabolite of coffee plants that begins with the biosynthesis of xanthosine monophosphate. In the metabolite pathway, the methylation subsequence step occurs in the presence of different N methyltransferases, with methionine being the methyl donor. Catabolism of purine groups in caffeine consists of degradation via a succession of demethylation processes that are broken down into carbon dioxide and ammonia [1].

Among the various sources of caffeine, coffee beans are one of the best-known and most widely consumed. Previous research has shown that caffeine levels in Arabica coffee beans can be affected by various factors, including the maturity level of the beans when harvested and post-harvest processing. A quantitative study using the UV-Vis spectrophotometer method found that each gram of roasted Arabica coffee contains caffeine levels that vary based on the maturity level of the beans. Young Arabica coffee contains 11.15 mg of caffeine per gram (1.151%), half-aged or medium-aged Arabica coffee contains 12.85 mg of caffeine per gram (1.285%), and aged Arabica coffee contains 12.01 mg of caffeine per gram (1.201%) [2]

Caffeine has an acute effect on blood pressure, especially in patients with hypertension. This increase in blood pressure occurs through biological mechanisms including caffeine binding to adenosine receptors, activating the sympathetic nervous system by increasing the concentration of catecholamines in plasma, stimulating the adrenal glands, and increasing cortisol production. This results in vasoconstriction and increases total peripheral resistance, which will cause blood pressure to rise [3]. Thus, a

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solutive method is needed to reduce caffeine levels in coffee beans for people with caffeine intolerance.

One of the factors that influence efforts to reduce caffeine levels in coffee is the size of the coffee beans and the solvent used. The size of the coffee beans determines how effectively the solvent can extract the caffeine. Smaller size, larger surface area allows the solvent to more easily penetrate and absorb the caffeine components present in the beans. This is due to the increased contact area between the solvent and the coffee beans which accelerates the diffusion process. The higher diffusion rate allows the solvent to extract the caffeine more efficiently.

Studies on the interaction of caffeine with various chemical compounds have provided important insights into the mechanism of caffeine sorption and separation from complex matrices such as coffee beans. Caffeine adsorption occurs in the pH range of 2-7, with more efficient sorption occurring in acidic environments. This suggests that acidic conditions can increase caffeine adsorption efficiency. [4]

Previous research [3] revealed that caffeine can form hydrogen bonds with citric acid. Caffeine, which has three potential sites to engage in hydrogen bonding as a bond receiver, can interact with citric acid. This reaction between caffeine and citric acid can be understood as an interaction between a weak base (caffeine) and a weak acid (citric acid). Citric acid can release hydrogen ions (H+) into caffeine, resulting in a salt compound that is more soluble in water than pure caffeine.

The formation of these caffeine-citrate compounds has important implications in the decaffeination or extraction of caffeine from coffee beans. The more water-soluble caffeine citrate compound compared to pure caffeine facilitates the process of separating caffeine from coffee beans, thereby increasing the efficiency and effectiveness of the decaffeination process. From this description, this study will examine the effect of coffee bean size and citric acid concentration on caffeine content in ground arabica coffee.

1.2. Literature Review

Decaffeination is the process of removing caffeine from coffee beans or tea leaves to reduce the caffeine content in the product to low or even undetectable levels. This process allows consumers who are sensitive to caffeine or who wish to avoid the stimulant effects of caffeine to still enjoy beverage products without worrying about its effects [5]

In the decaffeination process of coffee beans, various factors play an important role in supporting extraction efficiency, including the size of the extracted material as well as the solvent concentration. Coffee bean size has a significant impact on the speed of the decaffeination process using ethyl acetate solvent. The smaller the size of the coffee beans, the faster the decaffeination process takes place. However, the use of synthetic solvents such as ethyl acetate often raises concerns related to environmental and health issues [6].

Smaller coffee beans have a shorter caffeine diffusion distance, allowing the caffeine to dissolve more efficiently in the solvent [6]. Thus, coffee bean size is an important factor in optimizing the caffeine extraction process. The dimensional size of the extract material directly affects the extraction efficiency of the active substances. Smaller dimensions of the extract material tend to increase the contact surface area between the extract material and the solvent, thus allowing the solvent to more efficiently extract the desired components from the material [7].

Caffeine is an organic compound that has electrophilic properties, so it is able to bind acids through the formation of amide bonds [8]. Increased acidity during the extraction process has the potential to significantly increase solvent penetration into the plant matrix. An acidic environment can help dissolve or weaken structural components such as cellulose, hemicellulose, and pectin in plant cell walls, making it easier for solvents to access and interact with intracellular contents [9]. The solubility of alkaloids increases under acidic conditions, so more of the desired compound can be obtained from the same amount of plant material [10].

Caffeine has the ability to react or bind. Caffeine adsorption occurs in the pH range of 2-7, with caffeine absorption being more efficient in acidic environments [4]. Caffeine has the ability to form hydrogen bonds with citric acid. This reaction between caffeine and citric acid can be represented as a reaction between a weak base (caffeine) and a weak acid (citric acid), forming a salt compound that is more soluble in water than pure caffeine. This facilitates the process of decaffeination or extraction of caffeine from coffee beans [11].

The addition of organic acids to a certain extent can improve the efficiency of extracting caffeine from coffee beans [3]. It was also shown that decaffeination of green tea leaves was successfully carried out using the microwave-assisted extraction (MAE) method with citric acid pre-treatment. The presence of citric acid plays an important role in enhancing the extraction process by softening the plant cell walls and increasing the solubility of caffeine in the solvent used. This combination of cell wall softening and increased solubility makes the extraction process more efficient, resulting in more effective decaffeination. This study showed that the use of citric acid can significantly increase the extraction efficiency of caffeine from green tea leaves, making it a potential solvent for industrial applications [12].

1.3. Research Objective

This study aims to evaluate the effect of coffee bean size and citric acid concentration on the caffeine of ground arabica coffee contained in it.

2. MATERIALS AND METHODS

2.1. Materials and Tools

The main material used in this research is arabica coffee (*Coffea arabica*) and citric acid. Materials used for analysis include anhydrous caffeine, MERCK chloroform, Na_2CO_3 , Folin Ciocalteu reagent, etanol, and distilled water. The equipment used in the research includes oven, roasting machine, grinder, volumetric glasses, and trays. The equipment used for analysis includes: spectrophotometer UV-Vis, digital scale, memmert oven, hot plate, water bath, separatory funnel, Petri dishes, volumetric flasks, volumetric pipettes, volumetric flask, erlenmeyer flasks, beakers, dropper pipettes, and clamps, coffee filter, Whatman No. 1 filter paper.

2.2. Design Experiment and Analysist

This study used Completely Randomized Design (CRD) with 2 factors with factor I being coffee bean size (4 mesh or 4.76 mm, 6 mesh or 3.36mm, and 8 mesh or 2.38) and factor II being citric acid concentration solution (1%, 3%, and 5%). Each treatment combination was carried out 2 replicates. The data obtained were then analyzed using ANOVA (Analysis of Variance). If there were significant differences between treatments, it was followed by DMRT (Duncan's Multiple Range Test) at the 5% level

2.3. Implementation of Research

2.3.1 Coffee Processing

Arabica coffee beans are sorted to remove damaged beans. The sorted seeds were ground and filtered using a grinder and mesh 4, 6, and 8 to obtain a uniform size. Each sample was soaked in 1%, 3%, and 5% citric acid solution for 4 hours. After decaffeination, the seeds were rinsed to stop the process.

The coffee beans were then roasted for 10 minutes at 190°C. The roasted beans were ground into powder and sieved with a 60 mesh. The powder samples were stored in clean, tightly sealed containers to prevent deterioration before laboratory testing.

2.4 Observation Procedure

2.4.1. Chemical analysis

Analysis parameters include a content analysis water using the gravimetric method, ash content using the gravimetric method, caffeine content using the UV-Vis spectrophotometric method, and phenol content using the UV-Vis spectrophotometric method.

2.4.2 Organoleptic test

The scoring test is a testing method that uses 20 semi-trained panelists to assess the appearance of the sample based on the intensity of the attributes or properties being assessed. Ground coffee is brewed with the ratio of coffee used is 1:15 where 10 grams of coffee in 150 ml of water.

3. RESULT AND DISCUSSION

3.1. Chemical Analysis

3.1.1 Moisture content

The results of the analysis of variance showed that there was no significant interaction between coffee bean size and concentration of citric acid solution, but coffee bean size had a significant effect on the water content of ground coffee. The average value of water content of ground coffee with coffee bean treatment is presented in Table 1.

Table 1. Average moisture content of ground coffee with coffee bean particle size treatments

Bean size	Moisture content (%) ± SD
4 mesh	4.439 ± 0.100 [b]
6 mesh	3.816 ± 0.343 [ab]
8 mesh	3.398 ± 0.130 [a]

Note: Average values accompanied by different letters indicate significant differences at $p \leq 0.05$

The results of observations in table 1. show that there are significant differences from the treatment of coffee bean size on the results of ground coffee water content. The smaller the size of the coffee beans will significantly reduce the water content. The 8 mesh coffee bean size reduction treatment produces the lowest water content of 3.398%, while the 4 mesh coffee bean size reduction treatment produces the highest water content of 4.439%.

In the drying process, materials with a smaller size have a larger surface area to volume ratio, so more surface area of the material is directly exposed to heat. This increase in surface area allows the heat to reach all parts of the material more efficiently and evenly. As a result, the water molecules in the material can absorb heat energy faster and evaporate more easily. This accelerates the process of reducing the water content in the material. Therefore, small-sized materials experience faster and more effective drying compared to larger materials, which require more time for the heat to penetrate into the interior and evaporate the water thoroughly.

The absence of significant differences in the observed moisture content is due to the inability of citric acid to significantly alter the cellulose structure in the plant cell wall. Citric acid, may be effective in breaking down other components, but the concentration of citric acid solution used does not have the necessary chemical properties to effectively degrade cellulose.

A certain concentration of citric acid does not damage cellulose, on the contrary, citric acid solutions can be used to improve properties and expand applications of cellulose through various modification techniques [13]. Citric acid up to 30% concentration tends to have no significant effect on cellulose. However citric acid can affect cellulose under certain concentrations and conditions (e.g., by heating or in combination with other catalysts) [14].

3.1.2 Ash content

The results of the analysis of variance showed that there was no significant interaction between coffee bean size and concentration of citric acid solution, but the concentration of citric acid had a significant effect on the ash content of ground coffee. The average value of ash content of ground coffee with coffee bean treatment is presented in Table 2 below.

Tabel 2. Average value of ash content of ground coffee with coffee bean size treatment

Citric acid concentration	Ash content (%) ± SD
1%	1.830 ± 0.044 [c]
3%	1.642 ± 0.031 [b]
5%	1.530 ± 0.037 [a]

Note: Average values accompanied by different letters indicate significant differences at $p \leq 0.05$

The observation results in Table 2 show that there is a significant difference in the concentration of citric acid used. The treatment of 5% citric acid solution concentration has the lowest ash content value of 1.530% while the treatment of 4 mesh coffee bean size and 1% citric acid solution concentration has the highest ash content value of 1.830%.

The effect of citric acid on ash content is more of a side effect of changes in the chemical form of minerals and the processing that follows. This is in line with Wibowo *et al.* [6] citric acid can cause a decrease in pH which can cause changes in minerals from colloidal form to dissolved form so that minerals are more easily dissolved during the soaking process. The dissolution of these minerals in citric acid solution causes the remaining mineral content in the food to decrease so that the ash content in the food produced also decreases, and does not affect the quality of the food products produced [6]. Research by Pohling *et al.* [15] added that citric acid shows a significant effect on the demineralization process of a material. The higher the concentration level of citric

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acid used, it will increase the demineralization efficiency to a certain extent [15].

3.1.3 *Caffeine content*

The results of the analysis of variance showed that there was a significant interaction between Arabica coffee bean size reduction and citric acid concentration and each treatment had a significant effect on caffeine content. The average value of caffeine content of arabica coffee ground is presented in Table 3.

The observation results in Table 3. show that there is a significant difference between the treatment of seed size and concentration of citric acid solution on the caffeine content of ground coffee. The average caffeine content of coffee groud has a range between 1.324-4.881 mg/g. The treatment of 8 mesh coffee bean size and 5% citric acid concentration had the lowest caffeine content value of 1.324 mg/g while the treatment of 4 mesh coffee bean size and 1% citric acid concentration had the highest caffeine content value of 4.881 mg/g.

Tabel 3. Average value of caffeine content of ground coffee

Treatment		Caffeine content (mg/g) ± SD
Bean size	**Citric acid concentration**	
4 mesh	1%	4.881 ± 0.409 [e]
4 mesh	3%	4.160± 0.045 [d]
4 mesh	5%	4.046 ± 0.031 [d]
6 mesh	1%	3.822 ± 0.199 [d]
6 mesh	3%	3.274 ± 0.332 [c]
6 mesh	5%	3.119 ± 0.268 [c]
8 mesh	1%	1.971 ± 0.118 [b]
8 mesh	3%	1.456 ± 0.159 [a]
8 mesh	5%	1.324 ± 0.118 [a]

Note: Average values accompanied by different letters indicate significant differences at $p \leq 0.05$

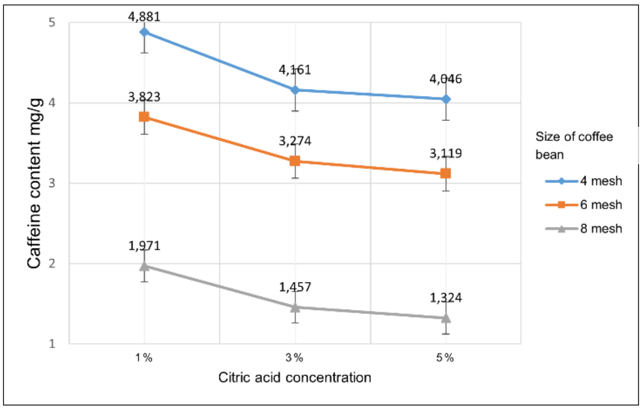

Figure 1. Relationship between coffee bean particle size and citric acid solution concentration treatments

Figure 1. shows that the relationship between the treatment of coffee bean size and the concentration of citric acid solution interacts with each other and each treatment has a significant effect on caffeine yield. The smaller the coffee beans, the lower the caffeine content, while increasing the concentration of citric acid solution will significantly reduce the caffeine content.

This decrease in caffeine content is due to the increased surface area to volume ratio resulting from the smaller particle or sample size. Smaller size contributes to a larger surface area, potentially increasing the efficiency of the extraction process. This is in line with Febrianto and Zhu's [16] statement that an increase in surface area due to smaller bean size can facilitate more effective caffeine extraction during the decaffeination process, resulting in lower caffeine levels in coffee beans.

The addition of citric acid concentration in the decaffeination process has the potential to increase the solubility of caffeine through reactions that occur during soaking. Increasing the acid concentration in the decaffeination process can increase the extraction efficiency to a certain extent. The reaction between citric acid and caffeine can increase the solubility of caffeine in solvents, facilitating the process of caffeine removal from coffee beans. Nonetheless, the effect of increasing acid concentration on extraction efficiency may have limits, where an excess of organic acids may no longer significantly improve caffeine extraction efficiency [3].

Caffeine in its reaction with citric acid, can deprotonate labile carboxylic acids to form nucleophilic carboxylates that can unlock reactive functionalities such as epoxy [17]. This process results in the formation of caffeine citrate. The reaction between caffeine and citric acid produces caffeine citrate salt in solution [17]. Citric acid acts as a weak acid that can release hydrogen ions (H+). Caffeine, as a weak base, reacts with the hydrogen ions released by citric acid. This process results in the formation of salt compounds between citric acid and caffeine. These salt compounds are generally more soluble in water than pure caffeine [18].

3.1.4 *Phenolic content*

The results of the analysis of variance showed that there was no significant interaction ($p \geq 0.05$) between coffee bean particle size and citric acid concentration. However, reducing the particle size of Arabica coffee beans has a significant effect on the phenol content of ground coffee. The average phenol content of the coffee powder for different particle size treatments is presented in Table 4.

Table 4. Average value of phenolic content of ground coffee

Bean size	Phenolic content (mg GAE/g) ± SD
4 mesh	48.434 ± 0.757 [a]
6 mesh	38.334 ± 0.416 [b]
8 mesh	30.374 ± 0.784 [c]

Note: Average values accompanied by different letters indicate significant differences at $p \leq 0.05$

The results in Table 4 show significant differences in total phenol content due to the particle size treatments. Smaller coffee bean sizes significantly reduce phenol content. The average phenol content of ground coffee ranges from 30.374 to 48.434 mg GAE/g. The 8 mesh particle size treatment results in the lowest phenol content of 30.374 mg GAE/g, while the 4 mesh particle size treatment yields the highest total phenol content of 48.434 mg GAE/g.

This indicates that smaller coffee bean sizes tend to produce lower total phenol content in coffee powder. Smaller coffee beans affect the total phenol content due to the roasting process. Coffee bean size can influence the evaporation rate of volatile compounds [19]. The smaller coffee beans have a significant impact on the roasting process. Smaller beans generally have a larger surface area per unit mass compared to larger beans, allowing for faster and more efficient heat transfer during roasting. Consequently, smaller coffee beans require lower roasting temperatures and shorter times to achieve the same roast level as larger beans.

The citric acid concentrations used in this experiment have not reached an optimal level for effectively extracting phenolic components from ground coffee. This has resulted in no significant differences observed in the coffee powder after treatment with citric acid solutions. The success of phenol extraction is highly influenced by the acid concentration used, and the concentrations chosen in this study were not sufficient to maximize phenol release from the coffee matrix.

Citric acid concentrations up to 5% did not show significant effects on food materials. Their findings indicate that 1%, 3%, and 5% citric acid concentrations do not cause differences in total phenol content due to the stability of phenolic compounds in weak acids up to 5%. The citric acid concentrations used were not effective in breaking down or extracting phenolic compounds [20]

The most effective citric acid concentration for phenol extraction is 10%. Their research showed that at a 10% citric acid concentration, the acid optimally breaks the complex bonds binding phenols in the source material, resulting in higher phenol extraction compared to lower acid concentrations [21].

3.2 Organoleptic Test

In a scoring test, this value reflects the level of the attribute that corresponds to the level description included in the test.

Table 5. Scoring test of arabica ground coffee

Parameters	Numeric scale
Color	1= yellow 2= yellowish-brown 3= dark brown 4= black 5= very black (intense)
Aroma	1= odorless 2= slightly aromatic 3= aromatic 4= very aromatic 5= extremely aromatic
Taste	1= not bitter 2= slightly bitter 3= bitter 4= very bitter 5= extremely bitter

The results of organoleptic testing using the scoring method are presented in Table 6.

Table 6. Organoleptic test results of scoring method

Organoleptik	**Hasil**
Color	S
Aroma	S
Taste	NS

Notes: NS = Non-Significant, S = Significant at the 5% level.

Table 7. The average value of the scoring test

Treatment		**Color**	**Aroma**	**Bitterness**
Bean Size	**Citric acid concentration**			
4 mesh	1%	2.20[a]	2.55[a]	3.65[a]
	3%	2.30[a]	2.55[a]	3.60[a]
	5%	2.35[a]	2.55[a]	3.65[a]
6 mesh	1%	3.20[b]	3.05[ab]	3.60[a]
	3%	3.35[b]	3.15[ab]	3.45[a]
	5%	3.30[b]	3.05[ab]	3.50[a]
8 mesh	1%	3.50[b]	355[b]	3.55[a]
	3%	3.50[b]	3.65[b]	3.50[a]
	5%	3.55[b]	3.65[b]	3.55[a]

Note: Average values accompanied by different letters indicate significant differences at $p \leq 0.05$

3.2.1 Color

The results of the analysis of variance showed that there were significant differences ($p \leq 0.05$) in the panelists' attribute scores on the samples. The observations in Table 7 show that coffee bean size significantly affects the color attribute. The smaller size of the coffee bean causes the color score value of the panelists towards the color parameter of the brewed ground coffee to increase. The color of Arabica ground coffee is influenced by the speed of heat propagation in the roasting medium.

The Maillard reaction causes significant changes in the sensory attributes of coffee beans. The melanoidins produced affect the color, aroma, and overall flavor profile of the brewed coffee. The Maillard reaction not only leads to the formation of melanoidins but also plays a role in the transformation of precursor compounds, resulting in a variety of flavor and aroma compounds [22]

3.2.2 Aroma

The results of the analysis of variance showed that there were significant differences ($p \leq 0.05$) by the panelists' attribute scores on the samples. The smaller the size of the coffee beans, the higher the aroma score of the panelists towards the aroma attribute of the ground coffee brew. Smaller coffee bean size reaches a higher roasting level than larger coffee bean sizes, so that the compounds formed in creating a distinctive coffee aroma are stronger [23]

Smaller coffee beans achieve higher roasting levels compared to larger coffee beans, resulting in stronger compounds that contribute to the distinctive coffee aroma. Reduction in phenolic compounds in roasted coffee beans can produce a more intense aroma due to complex interactions and reactions during the roasting process. While medium-roasted coffee beans may contain a variety of phenolic compounds and have prominent aromatic properties, the content of these phenolic compounds tends to decrease with increased roasting levels, especially with more intense roasting [24].

3.2.3 Taste

The results of the analysis of variance showed that there were significant differences by the panelists' attribute scores on the samples. Table 7. Shows no significant difference in flavor parameters. Coffee flavor is influenced by various chemical

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compounds. According to a study on volatile compounds, composition, and thermal behavior of coffee beans, coffee flavor is a combination of several constituents of volatile compounds such as acids, aldehydes, ketones, and phenolic compounds, along with non-volatile components such as sugars, proteins, amino acids, organic acids, and caffeine [25]

The bitterness of coffee is due to the presence of chlorogenic acid and chlorogenic acid lactones [26]. The bitter flavor is not only derived from caffeine, but chlorogenic acid degradation products account for about 60-70% of the bitter taste in coffee, and chlorogenic acid lactones in light to medium roasting [27].

4. CONCLUSION

The results showed that coffee bean size and citric acid concentration had a significant effect on caffeine content in Arabica ground coffee. The treatment that produced the lowest caffeine content was the treatment of 8 mesh coffee bean size and 5% citric acid solution concentration (1.456 mg/g), then obtained the value of water content (3.183%), ash content (2.073%), phenol content (29.472 mg GAE/g), and organoleptic test results with the attributes of fragrant-very fragrant aroma (3.55), blackish brown-black color (3.65), and bitter-very bitter taste (3.5).

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