Optimization of Defective Coffee Beans Decaffeination Using Palm Oil

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Abstract. Defective coffee beans amount to 15-20% of the total produced coffee beans. The defective coffee bean contains caffeine, which can negatively affect the human body, such as increased heart rate, and thus sensitive to consumption by some people. This study aims to optimize the decaffeination process of defective coffee beans. The extraction of aroma and flavor compounds was done by maceration, and the decaffeination was carried out using palm oil as a solvent. The type of beans (green and roasted beans), the decaffeination contact time, and the ratio between coffee bean extract and solvent were varied in this study. The caffeine content was quantified, and the organoleptic and color tests were done on the concentrated coffee extracts. It was found that the higher the amount of solvent volume in decaffeination, the higher the caffeine decrease. In addition, the longer the green beans' decaffeination time, the lower the caffeine decrease. Decaffeination using green coffee beans resulted in a greater reduction of caffeine (6.515-48.241%) than roasted coffee beans (8.495-24.272%). The optimum operating condition of green coffee bean decaffeination was the coffee bean extract and solvent ratio of 1:5.82 and the decaffeination time of 26.5 minutes. The organoleptic test result shows that decaffeinated coffee flavor had the same preferability as the commercial coffee flavor and was thus able to compete in the market.

Keywords: Defective Coffee Beans, Palm Oil, Decaffeination, Coffee Extract

INTRODUCTION

Defective coffee beans are coffee beans that have a low quality, which may be caused by a carry out of foreign matter (stones, sticks, and husk), black, broken, and brown beans, moldy beans, and insect-damaged beans (Kath, 2021). These beans contain chemical compounds, such as caffeine, chlorogenic acid (Ramalakshmi et al., 2007), and caffeol (Campanha et al., 2010), in which amounts do not differ significantly from superior quality coffee beans. The application of defective coffee beans is currently limited to producing jelly candies, body scrubs, and brewed coffee drinks. However, the caffeine content of defective coffee beans (0.9%) is still considered high enough, which might cause side effects on human health. Higher caffeine intake may lead to arrhythmias, central nervous system stimulation, heart rate increase, diastolic blood pressure increase, fetal development of pregnant woman imbalance, and breast milk production of nursing mothers decrease (Reyes et al., 2018). Thus, decaffeination can be done prior to consumption by several groups of people.

Decaffeination, in general, can be done by three methods: water decaffeination, supercritical decaffeination, and solvent decaffeination. Water decaffeination has the of water-soluble possibility aroma compounds loss, such as carbohydrates and chlorogenic acid (Pietsch et al., 2017). Supercritical decaffeination uses supercritical CO₂ as the solvent, which requires high installation and maintenance costs (Zabot et al., 2019). The solvent decaffeination method can be done by direct and indirect solvent decaffeination. Direct solvent decaffeination is done by pre-wetting, decaffeination, and steaming, which might cause flavor and aroma loss due to pre-wetting. Indirect solvent decaffeination needs a longer process than the other method, which consists of immersion, drying, decaffeination, flavor, and aroma return. The study of decaffeination of defective coffee beans is still limited as they mainly concern the decaffeination of superior quality coffee beans. Moreover, using palm oil as a decaffeination solvent is rarely used. This study used an indirect solvent decaffeination method was used since this method can minimize the loss of flavor and aroma compounds and is more straightforward than the water and supercritical method.

Oil can be applied as a decaffeination

solvent because it contains fat, effectively dissolving caffeine (Gottesman et al., 1985). Several solvents used in previous studies are ethanol, methanol, chloroform, benzene, and corn oil (Kartasasmita et al., 2012; Widagdyo et al., 2013). The dissolution of caffeine using oil as the solvent can occur due to the oleic acid content in the oil itself, which is more selective to caffeine than other acids (Hossain et al., 2011). Palm oil was chosen as the decaffeination solvent in this study because of the food-grade attribute, ease of resources, and high production. The oleic acid content in palm cooking oil is up to 42.5%, supporting decaffeination. In addition, the dissolution of caffeine using oil as the solvent can occur due to oleic acid content in the oil itself, which is more selective to caffeine than other acids (Hossain et al., 2011). Moreover, palm oil which is immiscible in water could prevent the carryover of the caffeine solvent.

This study aims to optimize the decaffeination process of defective green and roasted coffee beans using palm oil as a solvent. The caffeine content was quantified, and the organoleptic and color tests were done on the concentrated extract. The time and ratio between coffee bean extract and solvent were varied during decaffeination.

MATERIALS & METHODS

Materials

Materials used in this study are defective green coffee beans and defective roasted coffee beans (both from West Java, Indonesia), commercial palm oil (East Java, Indonesia), pure caffeine from coffee bean caffeine powder as standard (Soho Nootropics, Supplement Logistics LLC Arizona, USA), and commercial espresso concentrate. The defective coffee beans tend to be cracked, broken, black beans, and hollow coffee beans. The purity of the palm oil was 100%.

Coffee Beans Extraction

Aroma and flavor extraction for green and roasted coffee beans was done using the maceration method and water as solvent. Maceration was carried out using a threeneck boiling flask at 80° C for 6 h. The mixtures were centrifuged, and the extract was collected. The ratio between coffee beans and the solvent used is 1:3.35. This value is obtained from previous research by Wulandari et al. (2019.) Water as solvent was heated using a heating mantle until its temperature reached 80°C, which resulted in the highest increase in caffeine solubility (Kartasasmita & Addyantina, 2012), and the extraction was done for 6 hours. The flowchart of this process is shown in Figure 1. If after maceration, decaffeination, or caffeine content testing is not immediately carried out, the coffee extract will be cooled first to maintain the quality of the extract.

Decaffeination

Decaffeination was done by liquid-liquid extraction, with caffeine dissolved in the coffee extract as a solute and palm oil as a solvent. Coffee extracts and palm oil were put in an Erlenmeyer flask. Then the decaffeination was done using an orbital shaker (Oregon KJ201BD, imported by PT Golden Pratama) with a speed of 140 rpm at room temperature. The time and ratio between coffee bean extract and solvent were varied during decaffeination, and the time varied from 24, 30, 45, 60 to 66 minutes while the ratio varied from 1:0.17, 1:1, 1:3, 1:5, 1:5.82. The flowchart of this process is shown in Figure 2.

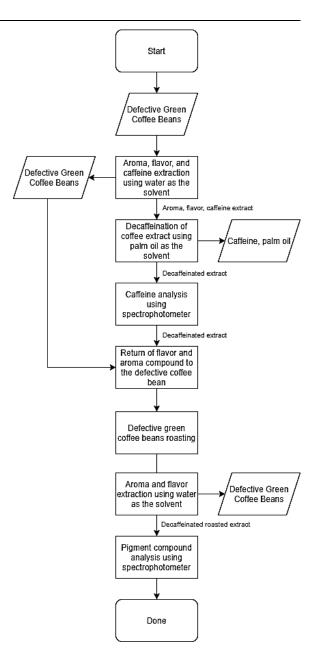


Fig. 1: Flow diagram of the decaffeination process of defective green beans

Return of Flavor and Aroma Compounds other than Caffeine

An additional step in processing green coffee beans as the raw material was done to return the flavor and aroma compounds (other than caffeine) to the extracted coffee beans. This is because the presence of flavor and aroma compounds are affected by extraction process (Shofinita et al., 2023). Extracted coffee beans were soaked in coffee extract without caffeine for 3 hours to produce flavor and aroma as a roasted coffee bean in the roasting process (Kartasasmita et al., 2012). If the coffee extract does not soak immediately into the coffee beans after decaffeination, the coffee beans will be frozen in the freezer.

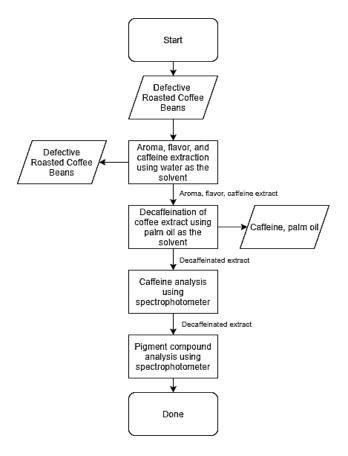


Fig. 2: Flow diagram of the decaffeination process of defective roasted beans

Coffee Bean Roasting

Green coffee beans were roasted in the oven at 210°C for 20-30 minutes until they reached a state based on the color and time of the bean cracking parameter. This step would produce a medium-roasted coffee bean with a balanced aroma and flavor (Lyman et al., 2003).

Caffeine Analysis

Caffeine in the decaffeinated coffee extract was analyzed using a spectro-

photometer (Shimadzu Europe, UV mini-1240). A calibration curve was made using 250 mg of pure caffeine powder dissolved in hot water. To find the maximum wavelength of caffeine, a standard solution of 6 mg/L was analyzed over a range of 250-300 nm. Caffeine was then analyzed using the maximum wavelength known from the previous step. Then, the standard solution was made into a calibration curve with concentrations of 1; 3; 6; 9; 12 mg/L. To find the maximum wavelength of caffeine, a standard solution of 6 mg/L was analyzed over a range of 250-300 nm. Caffeine was analyzed maximum then using the wavelength known from the previous step.

Organoleptic Analysis

Organoleptic analysis was done on the product of decaffeinated, non-decaffeinated, and commercial coffee extract. These samples are shown in Figure 3.



Fig. 3: Decaffeinated coffee extract (A), nondecaffeinated coffee extract (B), and commercial coffee extract (C).

Analysis was done on the green coffee beans because they exhibited a greater caffeine reduction due to decaffeination than roasted coffee beans. Analysis was done by mixing the coffee extract and water with a ratio of 1:2. For the sensory evaluation. There were 24 panelists with sensory criteria, both people who regularly consume coffee and those who do not, the panelists' age ranged from 21 to 65 years, with an even gender distribution. Parameters analyzed in this sensory analysis were bitterness, acidity, aftertaste, body, aroma intensity, aroma quality, overall taste, comparison between all samples, and level of preference for all samples. The Specialty Coffee Association of America (SCAA) method is used in this evaluation. This method is based on a quantitative descriptive sensory analysis of the beverage, which panelists perform. In this method, coffee beans are scored based on the primary attributes that comprise the sensory profile of coffee: fragrance/aroma, uniformity, clean cup, sweetness, flavor, acidity, body, aftertaste, balance, and the overall impression of the coffee. CHN Spec Colorimeter was used for the color test to determine the L*, a, b, chroma, and hue.

Optimization using Response Surface Method

Optimization was done using Minitab 2017 to get an optimum operational condition in the decaffeination process using green and roasted coffee beans. The variables considered were decaffeination time (minimum limit of 30 and maximum limit of 60) and solvent-to-coffee extract volume ratio (minimum limit of 1 and maximum limit of 5). A full second-order factorial design for two factors (decaffeination time and solvent to extract ratio (SER)) was carried out to determine the significant factor for decaffeination. Using the Response Surface Method (RSM) with a Central Composite (CCD) model produced Design 13 variations. Experimental experimental uncertainty was calculated using the standard deviation formula for each raw material variation.

RESULTS AND DISCUSSION

Decaffeination of Green and Roasted Coffee Beans

Decaffeination of Green Coffee Beans

In order to optimize the condition for decaffeination, the reduction of caffeine content was observed in Figure 4. The reduction of caffeine content was in the range of 6.515%-48.241%. Caffeine could dissolve in palm oil because of its ability to dissolve in organic solvents. Palm oil has a characteristic as an organic solvent with fat content, which effectively dissolves caffeine (Gottesman et al., 1985). This could be caused by caffeine and palm oil polarity. Palm oil has a close polarity to one of the caffeine solvents, chloroform, so it can be estimated that caffeine also dissolves well in palm oil. One of the fat contents in palm oil is oleic acid, which comprises most of the fatty acid in palm oil and is up to 42.5% weight. Previous research by Hossai et al. (2011) stated that oleic fatty acid interacts more with caffeine than with other fatty acids. Therefore, when contacted with palm oil, fatty acid content as caffeine solvent in palm oil could reduce caffeine content in the coffee extract.

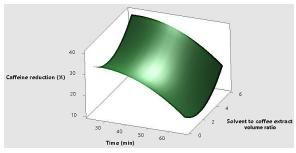


Fig. 4: Surface plot of caffeine reduction after decaffeination of green coffee beans

The surface plot in Figure 4 shows that the decaffeination time and the solvent-tocoffee extract volume ratio (SER) affected the caffeine reduction. Figure 4 shows that the higher the ratio of SER, the higher the caffeine reduction, and thus the higher the caffeine dissolved in a solvent. This could be seen from the increased surface of the plot surface along with the increase of solvent volume. Widagdyo et al. (2013) also obtained a similar trend using corn oil as a solvent. Caffeine reduction could occur because of the caffeine mass transfer from coffee extract to palm oil. The mass transfer in decaffeination using the liquid-liquid extraction method involves diffusion, which occurs from a higher concentration to a lower concentration. During this process, the solutes from the feed phase are transferred to the solvent phase. Both phases are separated by an interface and a double film (one of each phase). The mass transfer occurs exclusively in the double stationary film by the molecular diffusion mechanism. In the bulk of both phases, the concentration of caffeine is considered uniform as a consequence of perfect mixing. A higher solvent volume may result in an increase in the concentration gradient due to the difference in caffeine solubility, where the solubility of caffeine is higher in water compared to the solubility of caffeine in oil. This may cause an increase in the mass transfer rate of caffeine, so a larger caffeine reduction occurs in a higher SER.

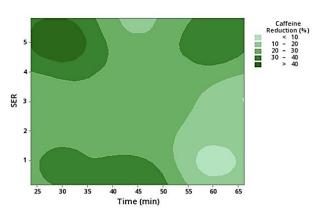


Fig. 5: Contour plot of caffeine reduction after decaffeination of green coffee beans

The optimum conditions obtained were a SER of 5.828:1 and a time of decaffeination of 26.545 minutes. This can be seen in Figure 5, where the highest caffeine reduction was obtained on the upper left side of the plot. The RSM-CCD equation for caffeine reduction as a function of solvent to coffee extract volume ratio (SER) during decaffeination of green coffee beans is described in Equation 1 as follows:

 $Caffeine\ reduction\ (\%) = 26.3 + 0.59 *$ DT - 4.73 * SER - 0.0111 * DT * DT + 0.97 *SER * SER(1)*DT: Decaffeination Time (minutes)*SER: Solvent-Extract Ratio

This equation can estimate the percentage of caffeine reduction during the decaffeination of green coffee beans using decaffeination time and solvent-extract ratio.

Decaffeination of Roasted Coffee Beans

The effect of decaffeination time and SER on caffeine reduction can be seen in Figure 6. Overall, the decaffeination of roasted coffee beans could reduce caffeine from 8.495% to 24.272%. It could be caused by volatile compounds, which inhibit the contact between caffeine and palm cooking oil. Therefore, this method obtained lower caffeine reduction than green coffee beans decaffeinated.

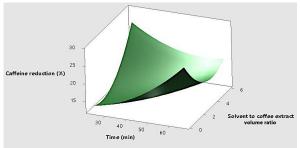
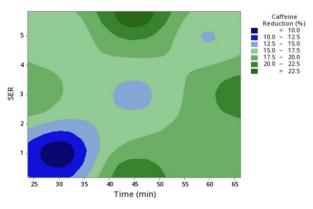
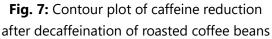


Fig. 6: Surface plot of caffeine reduction after decaffeination of roasted coffee beans

It was found that the higher the SER used, the higher the caffeine reduction obtained due to the increase in the caffeine concentration gradient between coffee extract and palm oil. This leads to the caffeine diffusion from the coffee extract into the palm oil to reduce the gradient of the caffeine content (McCabe et al., 2005).

At the coffee extract to solvent ratios of higher caffeine reduction 1:1 and 1:5, occurred the decaffeination as time increased. The longer the contact of caffeine and palm oil, the higher the caffeine content diffused to palm oil since the liquid-liquid extraction is affected by the time of solute and solvent contact. This result corresponded with the decaffeination process using corn oil as a solvent by Widagdyo et al. (2013).





Like the decaffeination of green coffee beans, the optimum operating condition was obtained at an SER of 5.828:1 and a decaffeination time of 45 minutes, resulting in a caffeine reduction of 24.27%. This can be seen in Figure 7, where the highest caffeine reduction was obtained on th

e upper central side of the plot. The equation from RSM-CCD for the response of roasted coffee bean decaffeination as a function of solvent-to-coffee extract volume ratio (SER) can be seen in Equation 2 as follows:

Caffeine reduction (%) = 14.4 - 0.168 * DT + 0.42 * SER + 0.00593 * DT * DT + 0.659 * SER * SER - 0.0884 * DT * SER (2) *DT: Decaffeination Time (minutes) *SER: Solvent-Extract Ratio

This equation can estimate the percentage of caffeine reduction during the decaffeination of roasted coffee beans using decaffeination time and solvent-extract ratio.

Decaffeinated Coffee Flavor Product Analysis

Using the optimum condition (green coffee beans as raw material, SER of 5.82:1, and decaffeination time of 26 minutes), a decaffeinated coffee extract was produced and analyzed for color and sensory.

Colorimeter Analysis

The values of L, a*, b*, hue, and chroma of each flavor sample were evaluated and shown in Table 1. Table 1 shows the color analysis for decaffeinated coffee extract (A), control or non-decaffeinated coffee extract (B), and commercial coffee extract (C).

Table 1. Colorimeter analysis result

	Α	В	С
L	2.79	2.88	2.74
a*	283.44	325.56	331.42
b*	15.11	19.37	19
Chroma	285.52	326.14	331.96
Hue	3.03	3.41	3.28

A: Decaffeinated Extract

B: Non-decaffeinated Extract

C: Commercial Coffee Extract

The different values among the samples were found due to the melanoidin compound as the coffee pigment that differs for each sample. Melanoidin, produced during the roasting process, is affected by the operating condition of the stage since a small difference in time and temperature used for roasting the coffee bean can significantly impact the coffee bean profile produced. According to Barbosa (2019), reducing sugar that promotes melanoidin formation contributes to the color of the coffee extract produced.

Regarding roasting decaffeinated green coffee beans, some things could be improved in maintaining a consistent roast profile of the beans. This circumstance may be due to several processes done to the green coffee beans, resulting in a ranging bean color and making the bean's color changes during the complex roasting process to notice. Additional from stress the earlier decaffeination process impacts the structure of green coffee beans. Having a more brittle cell structure, the green coffee beans release moisture easily thus the roasting process was faster. The first crack in the roasting process for decaffeinated and nondecaffeinated beans came simultaneously despite the decaffeinated beans' weight being lighter. This first crack look from decaffeinated coffee beans was different from the nondecaffeinated ones, leading to a conclusion that the decaffeination process influences the roasting profile of the roasted, decaffeinated coffee beans.

Table 2. Color perception based on ΔE^* (A= decaffeinated extract,

B = non-decaffeinated extract,

	C=	commercial	coffee	extract)
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Sample	ΔΕ*	Perception
A-B	42.3	Color tends to be
		different
A-C	48.1	Color tends to be
		different
B-C	5.9	Can be distinguished
		by glance

 ΔE^* calculation was done to know how each coffee sample differs, and the result can be seen in Table 2. It can be known that the decaffeinated extract was slightly darker than the non-decaffeinated one. This might happen due to the different roasting profiles between both beans before extraction.

Organoleptic Analysis

Figure 6 shows the comparison of organoleptic properties for 3 products: decaffeinated extract (A), non-decaffeinated extract (B), and commercial coffee extract (C)). Two-tailed type t statistical tests were performed to test whether a significant difference exists among the decaffeinated (A), non-decaffeinated (B), and commercial coffee extracts (C). Based on the statistical test, there is a significant difference in the acidity parameter between the three extracts, except for the bitterness and the overall taste. There is also a significant difference in the aftertaste, thickness, color intensity, and aroma intensity between samples A-B and A-C, aroma quality for samples A-B, and preference level for samples A-C.

Based on the data in Figure 8, the most bitter flavor was found in non-decaffeinated, commercial, and decaffeinated coffee extracts, respectively. As the decaffeinated extract was the least bitter, it supports the data that this product had the least amount of caffeine than the other samples due to the decaffeination process. While different caffeine content in the non-decaffeinated and commercial extracts might be produced since different coffee varieties and operating conditions of the flavor were used.

According to the acidity parameter, the non-decaffeinated flavor had the most acid profile, followed by the commercial and decaffeinated extracts. The lower acidity parameter in the decaffeinated extract was due to the presence of organic acid compounds that may be dissolved in the solvent during the decaffeination process. Since the non-decaffeinated extract obtained the most bitter and acidic coffee extract, this extract had the longest aftertaste in the mouth. However, non-decaffeinated extract's thickness or body was lower than the commercial extract. The body of the coffee extract is affected by the roasting condition. The higher the roasting degree of the coffee bean, the higher the body of coffee extract produced (Ngugi et al., 2021). The body difference in the flavors might owe to the coffee bean's protein, fiber, and fat, the degree of roasting, and the method used to process the sample (Fibrianto et al., 2018; Ngugi et al., 2021). According to the overall taste, the decaffeinated flavor had the highest score, followed by the commercial and non-decaffeinated flavors. The phenomenon could be achieved even though the decaffeinated extract was not as strong as non-decaffeinated and thus may have a balanced taste.



Fig. 8: The organoleptic properties of coffee extracts (A= decaffeinated extract, B= nondecaffeinated extract, C= commercial coffee extract)

The non-decaffeinated extract was

superior in aroma parameters, followed by the commercial and decaffeinated extracts. The aroma quality of the non-decaffeinated extract was better because of the strong roasted coffee aroma and high intensity of the aroma. The high aroma intensity of the non-decaffeinated and commercial flavors were obtained because the chemical compounds were more maintained and did not involve more processing than the decaffeinated extract.

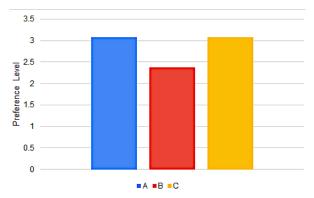


Fig. 9: The preference level of each coffee extract (A= decaffeinated extract, B= non-decaffeinated extract, C= commercial coffee extract)

decaffeinated Both and nondecaffeinated extracts were made using the defective coffee beans, yet it did not affect the panelists' acceptance of coffee in terms of aroma. The score for the preference level in Figure 9 showed that the decaffeinated extract had quite a similar preference level to commercial extract. Thus, the the decaffeinated extract might compete with the commercial extract on the market.

CONCLUSIONS

It can be concluded from this study that the higher SER, the higher the decrease in caffeine content. The caffeine decrease in green coffee beans was relatively higher (6.515%-48.241%) than in roasted coffee beans (8.495%-24.272%). It was also found that the decaffeinated coffee extract had a lower level of bitterness, acidity, body, color and aroma intensity, and aroma quality compared to non-decaffeinated coffee and commercial coffee extracts. Yet, it had the same level of preferability as commercial coffee extract so it may be able to compete in market. The optimum the operating conditions coffee for green bean decaffeination were a solvent-to-extract ratio of 5.82:1 for 26.5 minutes.

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