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Metabolite Profile of arabica Coffee Cascara from Typica Cultivar in Bandung with Different Drying Processes

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ABSTRACT

Arabica coffee bean (*Coffea arabica* L.) is Indonesia's leading commodity, with Bandung Regency as the largest producer in West Java. The exocarp and mesocarp of coffee fruits known as cascara have potential health benefits as a beverage. Cascara is usually dried by direct sunlight before consumption, but this process is prone to weather disturbances and contamination. The use of dehydrators is more controlled to maintain metabolite content, but there has been no research on cascara from coffee cultivars in Bandung Regency dried by this method. This study aims to obtain the metabolite profile of Typica arabica coffee cultivar cascara dried using direct sun and dehydrator. The cascara samples were taken from Gunung Puntang Coffee Plantation, Bandung Regency, and extracted using maceration method with 70% ethanol p.a. solvent. The metabolite content was analyzed by Gas Chromatography-Mass Spectrometry (GC-MS), which specifically detects volatile metabolites. Non-volatile compounds were not within the scope of this study. The results showed that direct sun-dried cascara had 6 metabolites, while dehydrator-dried had 15 metabolites. Both samples were dominated by caffeine (sun-dried cascara: 46.61%, dehydrator cascara: 24.44%). Five metabolites were found in both samples, with 1 unique metabolite in solar cascara and 10 unique metabolites in dehydrator cascara. This study showed differences in metabolite content in Typica cultivar cascara with different drying methods.

INTRODUCTION

Coffee beans are one of the important plantation commodities in Indonesia, with the fourth highest production level in the world and second in Asia (International Coffee Organization, 2023). Coffee production in Indonesia in 2022 reached 774.96 thousand tons, consisting of robusta coffee (*Coffea canephora*) and arabica coffee (*Coffea arabica*) (Badan Pusat Statistik, 2023). Arabica coffee production is predicted to increase by 30% in 2025 due to its higher economic value (Kaido & Takashino, 2023). Increased coffee production has the potential to increase coffee waste. Valorization of coffee waste, such as cascara, is needed to make it economically valuable and environmentally friendly.

Coffee bean production generates 90% of agricultural waste, including cascara, which is composed of the exocarp and mesocarp of coffee fruits (Iriondo-DeHond et al., 2020). Cascara makes up about 50% of the coffee harvest volume, but is often discarded without further processing, causing environmental problems (Jiménez-Zamora et al., 2015). The utilization of cascara in Indonesia is generally still limited to low-value products such as compost (Komaria et al., 2020). Cascara has the potential to become a food ingredient with high nutritional content and bioactive properties (Gemechu, 2020; Bondam et al., 2022). The high moisture content of fresh cascara limits its storage and transportability, so drying is required. Traditional sun drying is susceptible to weather changes and contamination (Dong et al., 2017). The use of a dehydrator can preserve aroma and flavor-forming volatile compounds (Dudek et al., 2022).

Volatile metabolites are directly linked to aroma, taste, and consumer acceptance of cascara-based beverages (Sales et al., 2023). Cascara tea products dried with a dehydrator have high sensory quality (Indrayani et al., 2022). However, there has been no analysis of volatile metabolite profiles in dehydrator-dried cascara. Aspects of coffee cultivars also need to be studied for their influence on the metabolite profile of cascara. Different coffee species and cultivars can produce different phenol and antioxidant contents (Heeger et al., 2017; Murlida et al., 2021). Lestari et al. (2023) have examined the Gayo cultivar and established the cascara metabolite profile of the coffee. However, there has been no research on the cascara metabolite profile of coffee cultivars in West Java, especially in Bandung Regency. Bandung Regency is the main producer of coffee in West Java, especially in the southern mountainous area which produces a lot of arabica coffee (Fithriyyah et al., 2020). One of the centers of coffee plantations in Bandung Regency is in the Gunung Puntang area, Campakamulya Village, Cimaung District. Gunung Puntang Coffee Plantation cultivates various cultivars of arabica coffee, including the Typica cultivar. Cascara from this plantation is separated from coffee beans for sale and processing by other parties, but its compound content is not yet known. Research is needed on the metabolite profile of arabica coffee cascara in cultivars grown in Bandung Regency, especially the Typica cultivar, which is dried using direct sunlight and a dehydrator.

MATERIALS AND METHODS

This research was conducted from December 2023 - June 2024. The cascara samples were obtained from Gunung Puntang Coffee Plantation, Campakamulya Village, Cimaung District, Bandung Regency, West Java. Arabica coffee fruits of Typica and Sunda cultivars were cleaned using running water to eliminate any dirt and other foreign materials. Peeling of the cascara (exocarp and mesocarp of the coffee fruit) was done using a coffee skin peeling machine to separate the cascara and coffee beans. Other parts that were still attached to the cascara, such as fruit stalks and remaining seeds, were separated manually. A total of 1 kilogram of cascara for each drying process was then stored in a closed container at -20°C until the drying process was carried out.

The drying and extraction of the samples were carried out at the Environmental Research Laboratory, Indonesia University of Education. The cascara samples were dried using two drying processes until reaching a constant dry weight. Sun drying was conducted based on research of Hu et al. (2023) with modifications. The samples were spread evenly on plastic trays placed under unobstructed sun exposure with a daily duration of 10 hours (07.00-17.00 UTC+07:00), until the constant dry weight was reached after the total of 60 hours. Dehydrator drying was carried out using a food dehydrator (ARD-PM99, 300W, 220V, operating temperature 35-60°C) for a total of 36 hours until the dry weight was constant. The drying procedure was carried out to the modified method from Indrayani et al. (2022). Cascara was spread evenly on each dehydrator tray, then the temperature was maintained at 40°C using the device settings until a constant weight was obtained. Each dried sample was pulverized into fine powder using a blender, then filtered using a 100-mesh sieve. The powder was then stored in an airtight container and dark conditions.

The dry powder of cascara was extracted using the maceration method. The extraction process with maceration was carried out according to the modified method from Lestari et al. (2023) and Utami et al. (2022). The solvent used was analytical grade 70% ethanol. A total of 20 grams of cascara powder was put into an Erlenmeyer flask, then 200 mL of 70% ethanol was added. The solution was stirred, then the flask was tightly closed and wrapped in aluminum foil to avoid light for a duration of 7 x 24 hours. The process of stirring the maceration mixture using a 150-rpm shaker was carried out every 24 hours. The filtration process was carried out using filter paper (Whatman No. 1). The filtrate was evaporated using a 40°C water bath (Sibata WB-K1) to obtain a concentrated extract with a paste-like consistency. The extract was then stored in a sealed bottle at 4°C until the GC-MS analysis process was carried out.

Metabolite analysis was carried out at the Forensic Laboratory of National Police Criminal Investigation Center (Bareskrim Polri), Bogor Regency, West Java, using a GC-MS equipment (Agilent 5973). The column used was Agilent type 190915-433UI. The carrier gas was helium with a flow rate of 1 mL/minute, and 1 µL of cascara ethanolic extract was injected into the device. Metabolite identification was carried out by comparing the similarity of the obtained data with the WILEY 9TH library and the National Institute of Standards and Technology (NIST) 2.0, with a fixed minimum limit of 80% similarity index.

RESULTS AND DISCUSSION

The GC-MS analysis roduced chromatograms with peaks indicating the compounds detected in cascara cultivar Typica extracts from direct sun drying (Figure 1) and dehydrator (Figure 2). A total of 16 metabolites were identified (Figure 3), with most compounds classified as fatty acids, yet caffeine alkaloid compounds dominated the percentage area in both samples. The two types of drying resulted in differences in the number and area of compounds identified, with five compounds found in both samples. Dehydrator drying resulted in a greater number of metabolites (15 compounds) than direct sun drying (6 compounds).

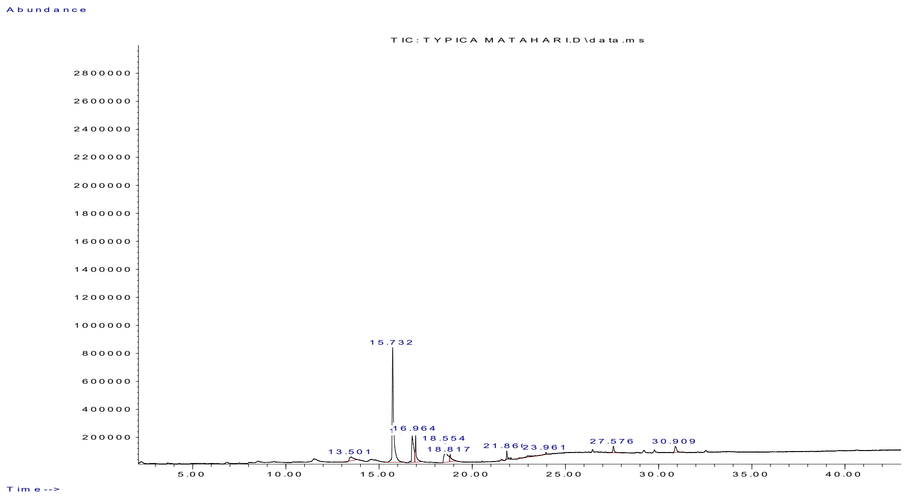

Figure 1. GC-MS chromatogram from the extract of direct sun-drying cascara Typica cultivar

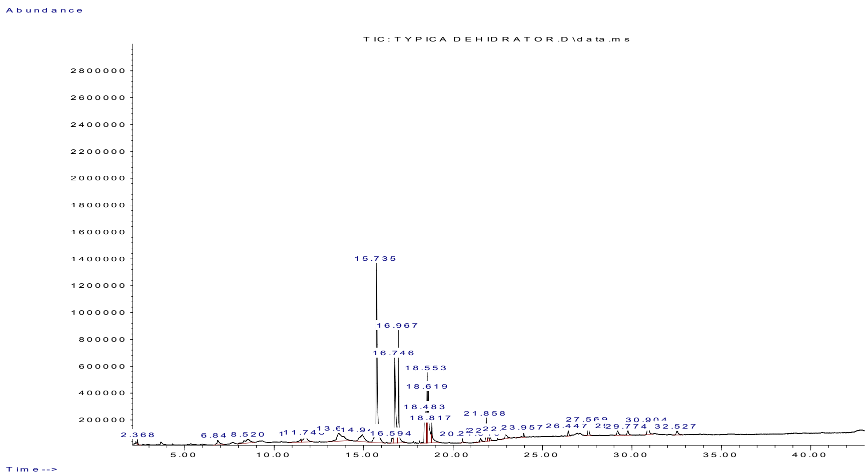

Figure 2. GC-MS chromatogram from the extract of dehydrator-drying cascara Typica cultivar

Compound	Group	Area (%) Typica Direct Sun	Area (%) Typica Dehydrator
2,3-Dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one	Pyran	0	1.29
Caffeine	Alkaloid	46.61	24.44
n-Hexadecanoic acid	Fatty acid	14.75	14.82
Hexadecanoic acid ethyl ester	Fatty acid	8.1	8.24
9,12-Octadecadienoic acid (Z,Z)-	Fatty acid	0	7.25
Linoleic acid ethyl ester	Fatty acid	12.95	5.32
9,12,15-octadecatrienoic acid (Z,Z,Z)- ethyl ester	Fatty acid	0	7.04
Eicosanoic acid	Fatty acid	3.61	0
Octadecanoic acid ethyl ester	Fatty acid	0	2.67
Eicosanoic acid ethyl ester	Fatty acid	0	0.53
Methyl ester bicyclo[2.2.1]heptane-2-acetic acid, alpha-oxo-exo-3-(2-oxo-1-phenylethenyl)-	Fatty acid	0	2.33
2-Methyl-Z,Z-3,13-octadecadienol	Fatty alcohol	0	1.07
γ-Tocopherol	Phenolic	0	0.63
α-Tocopherol	Phenolic	2.42	1.52
Campesterol	Sterol	0	0.83
γ-Sitosterol	Sterol	0	2.09

Figure 3. Heatmap of metabolites in Cascara Typica cultivar extracts dried by direct sun and dehydrator

This research evaluated the effect of drying methods on the volatile metabolite profile of cascara of Typica cultivar. The main findings showed that cascara samples dried using direct sun contained fewer metabolites than those dried with a dehydrator (Figure 4). Other studies have also reported that direct sun drying could lead to nutrient and bioactive chemical loss, affecting the overall metabolite profile of food (Mróz et al., 2024). The adverse effects of sun drying vary due to several thermal factors. Dehydrator drying minimizes the impact of these external factors by offering a controlled drying environment, thus better preserving metabolites. This controlled method can produce more consistent and high-quality cascara, maintaining its sensory qualities and market value. Jain & Tiwari (2003) highlighted that several external factors like solar radiation intensity, temperature of the surrounding, wind speed, and relative humidity in air can lead to inconsistencies in sun-dried products' quality.

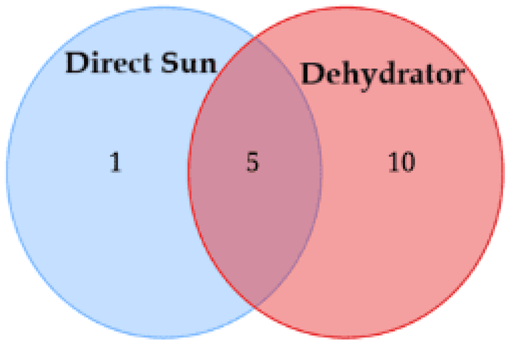

Figure 4. Comparison of metabolite counts in cascara Typica cultivar from direct-sun and dehydrator drying

The study also identified seven lipid compounds, including five fatty acids and two sterols, that were present only in dehydrator-dried samples. This is consistent with findings from other studies that report reduced lipid components in sun-dried foods (Tenyang et al., 2020; Zioud et al., 2023). Sun drying may reduce cascara's health benefits due to the loss of these bioactive lipids. Dehydrators, by avoiding UV exposure, preserve these beneficial components, making cascara more suitable for health-focused food applications. Csapó et al. (2019) noted that sun drying can damage free fatty acids, which are vulnerable to UV radiation, leading to oxidation. Research by Mróz et al. (2024) similarly found a decrease in the metabolite profile, especially lipids, when comparing sun drying to dehydrator drying in the microalgae Arthrospira platensis.

The fatty acid compound group was found in all samples and was the main constituent of metabolite components because it had the largest number of compounds. The dominance of fatty acid compounds was also reported in the Gayo cascara cultivar (Lestari et al., 2023). This is inseparable from the nature of fatty acids, which are very common and varied metabolites in plants because they have various functions, including structural, protective, and storage (Avato & Tava, 2022). Discoveries related to fatty acids as the main component in cascara are important to understand its nutritional value and health potential, so as to maximize the benefits of cascara products. Kachroo & Kachroo (2009) assert that fatty acids are widely found in plants because they are important for metabolism, through their role in inducing plant defense modes, modulating immunity, and functioning as components of membrane lipids. Xiao et al. (2021) utilized the properties of fatty acids commonly stored in seeds to build plant seed identification profiles.

One of the fatty acid compounds found in sun drying and dehydrator is 9,12-octadecadienoic acid (Z,Z)- otherwise known as linoleic acid. There is also 9,12,15-octadecatrienoic acid ethyl ester (Z,Z,Z)- or linolenic acid ethyl ester which is the esterification product of linolenic acid and only found in dehydrator dried cascara. The presence of essential fatty acids has also previously been detected in arabica coffee cascara from local cultivars in Panama (Bobková et al., 2022). Linoleic acid and linolenic acid are essential fatty acids known as omega-3 and omega-6, respectively, and must be provided through the diet because they are unable to be synthesized by humans, yet their presence is vital to carry out normal physiological conditions (Kaur et al., 2014). Cascara containing essential fatty acids can be utilized to meet nutritional needs, especially for individuals who do not get enough from their diet. Tallima & El Ridi (1971) revealed that essential fatty acid deficiency can have a malnutrition effect because these fatty acids are related to the synthesis of arachidonic acid compounds which are very important structurally and functionally for humans. It is important to balance the consumption of essential fatty acids with other nutrients, because Mariamenatu & Abdu (2021) reported that excessive consumption of essential fatty acids can cause inflammatory and allergic disorders and disorders in the nervous system.

In this study, no polyphenols were found. A number of previous studies have reported the presence of various types of polyphenols in cascara including chlorogenic acid, gallic acid, rutin, and tannins, but these studies used certain analytical methods to detect the presence of polyphenols, namely the Folin-Ciocalteu method and high-performance liquid chromatography (HPLC) (Arpi et al., 2021; Heeger et al., 2017; Murlida et al., 2021). The complex structure of the compound and the large molecular size limit the ability of polyphenols to evaporate easily, making the compound classified as non-volatile (Aronson & Ebeler, 2004; Heo et al., 2020). GC-MS analysis carried out to compile metabolite profiles in this study is not suitable for detecting the presence of polyphenols in cascara because GC-MS tools can only detect volatile compounds. Additional analytical methods are needed to determine the content of polyphenols which can supplement the metabolite profile information generated in this study. Research by Pua et al. (2021) did not detect polyphenols in cascara through GC-MS analysis, but additional data from HPLC analysis was able to produce polyphenol content data, with chlorogenic acid having the highest concentration. Rohloff (2015) stated that polyphenol analysis using GC-MS can be carried out through the extract derivatization stage to convert non-volatile compounds into stable volatile forms so that they can be detected by the GC-MS system.

Caffeine is a compound found in both cascara samples and dominates the metabolite profile of each sample. Caffeine is an alkaloid compound commonly found in plants of the genus Coffea, including in the exocarp and mesocarp of the fruits that make up cascara (Arpi et al., 2021). This compound is thought to function as a form of pest defense due to the toxic nature of the compound to insects (Lo & Hwang, 2024). Caffeine is also a central nervous system stimulant that can improve alertness and cognitive function (Wood et al., 2014). Cascara's caffeine content provides further benefits regarding its potential application as an alternative food source for processed coffee with lower caffeine levels than coffee bean products. This opens cascara to consumers who are sensitive to high levels of caffeine or who are reducing caffeine dependence, in the form of alternative coffee products in the form of brewing from dried cascara. The presence of caffeine as a dominating compound in coffee cascara samples in this study is in line with the results reported in the study of Pua et al. (2021) who used samples of C. arabica coffee fruit cascara from various countries.

The percentage of caffeine areas in both drying methods showed different values, with the direct sun-dried cascara having higher area (46.61%) than dehydrator-dried cascara (24.44%). This percentage area does not directly represent the concentration of compounds in the sample, as a standard of pure compound is required for comparison so that mass estimates can be determined (Lovestead & Urness, 2019). Therefore, the percentage area of caffeine and other compounds in the sample does not indicate the weight of the compound but rather indicates the dominance of the compound in the analyzed sample relative to the total of all compounds in one detection cycle. The relative caffeine content of cascara is important to consider in the development of cascara products, especially those intended for specific health benefits. For example, cascara dominated by a very high percentage of caffeine from direct sun drying may be more appropriate for products designed to increase energy and alertness, while cascara with a more balanced metabolite profile from dehydrator drying may be more beneficial for products focused on overall wellness.

CONCLUSIONS

Based on the results, it can be concluded that direct sun drying obtained fewer metabolites than dehydrator drying. Cascara from sun drying contained six metabolites classified as fatty acids, phenolics, and alkaloids. Dehydrator drying resulted in 15 metabolites classified as fatty acids, sterols, phenolics, fatty alcohols, alkaloids, and pyrans. Both had five metabolites in common, with caffeine as the most dominant metabolite.

Further research is needed to identify the metabolites of cascara using drying methods that do not involve heat, such as freeze drying. Studies are also required to understand the mechanisms by which various drying variables influence the metabolites in Cascara. Additionally, alternative metabolite analysis methods are necessary to detect the presence of non-volatile compounds in cascara, complementing the findings from GC-MS analysis.

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