Pelita Perkebunan 37(3) 2021, 239–254
DOI: 10.22302/iccri.jur.pelitaperkebunan.v37i3.464
ISSN: 0215-0212 / e-ISSN: 2406-9574

Sensory Properties and Volatile Compound Profiles of Anaerobic Fermented Gayo Arabica Coffee Beans

Budi Mulyara[1,2*)], Supriyadi[2)], Yudi Rahmadian[3)], and Onne Akbar Nur Ichsan[4)]

[1)]Study Program of Estate Crop Product Technology, Sekolah Tinggi Ilmu Pertanian Agrobisnis Perkebunan, Jl. Williem Iskandar, Medan Estate, Indonesia
[2)]Study Program of Estate Crop Product Technology, Faculty of Agricultural Technology, Universitas Gadjah Mada, Jl. Flora No. 1, Bulaksumur, Yogyakarta, Indonesia
[3)]Department of Animal Product Technology, Faculty of Animal Science, Andalas University, Kampus Limau Manis, Padang, Indonesia
[4)]Faculty of Technology and Food Industry, Universitas Slamet Riyadi, Jl. Sumpah Pemuda No. 18, Surakarta, Indonesia
[*)]Corresponding author: budimulyara@stipap.ac.id
Received: 10 May 2021 / Accepted: 6 October 2021

Abstract

Coffee is a popular beverage that is consumed worldwide, meanwhile flavor is an important aspect of excellent coffee. Fermentation was applied in the washed coffee processing to degrade the mucilage layer and improve the flavor quality. Fermentation in non-washed Gayo Arabica coffee processing has not been widely reported. This study aimed to analyze the effect of anaerobic fermentation on Gayo Arabica coffee processing to obtain coffee with the high sensorial score. Coffee cherries were processed and fermented for 0–48 hours. Sensorial score, volatile, and non volatile compounds were observed on roasted beans. A significant increase in sensory test score 85.58 was obtained in the pulped natural process which was fermented. Concentrations of citric, malic, succinic, lactic, and acetic acids were varied between the treatments. Volatile analysis using SPME-GCMS produced 14 compounds with an odor active value (OAV) >1 consisting of aldehydes, furans, ketones, phenols, pyrazines, and terpenes. β-damascenone was a compound that had the highest OAV with honey-like, fruity, apple, and rose aroma characteristics that dominate pulped natural and black honey. Fermentation for 48 hours in the pulped natural process is suitable for use in Gayo with humid condition and high altitude.

Keywords: *Coffea arabica*, SPME-GCMS, volatile compounds, HPLC, non-volatile components, coffee processing

INTRODUCTION

Coffee is a very popular drink that is consumed worldwide. Indonesia's coffee export volume reaches 280,000 tons from a total production of 685,787 tons (BPS, 2018). Gayo Arabica coffee is coffee grown in three regencies of Aceh province, namely Aceh Tengah, Bener Meriah, and Gayo Lues with an area of 99,624 ha (DGEC, 2017). Gayo Arabica coffee planting area is growing very fast with a range of planting altitude generally between 950–1450 m above sea level. Coffee varieties planted in Gayo are very diverse such as S 288, S 795, Ateng Jaluk, Timtim, Borbor, Ateng Super, P 88, and BP 52 A. The Gayo Arabica coffee varieties are recommended by the Government in the cultivation and it is also selected by farmers in the region (Hulupi *et al*., 2013).

Nowadays, coffee production, trade, and consumption are in the 'third wave' era where the coffee not only acts as a commodity but has also been transformed into a special product (Samoggia & Riedel, 2018). This trend is in line with the high demand for high-quality coffee (Giacalone *et al.*, 2016). A good coffee flavor has been described by a pleasant sensation, a balanced combination of flavor, body, and aroma without any faults. The flavor is an important parameter and a sensory guarantee for consumers (Mori *et al.*, 2003). The sensory characteristics and chemical composition of coffee have been the target of research in recent years. Therefore, it remains a new and creative processing of coffee beans to fulfil the requirements of the high-quality coffee by consumers with new desired flavor.

One of factors which influencing the flavor and aroma of coffee beans is variations in the post-harvest processing methods (Sunarharum *et al*., 2014). The post-harvest coffee processing consists of wet processing (wash) and dry processing (Avallone *et al*., 2001). In the wash processing, spontaneous fermentation is applied and this process is known to have high acidity and a superior quality compared to other processes (Joët *et al*., 2010; Sunarharum *et al.,* 2014). However, dry process is different from the wash process. In that process, the coffee bean is still covered by thick mucilage and it is not removed through the fermentation process like the wash process. Thus, pulp and mucilage of coffee bean will be directly dried under sun drying (Knopp *et al*., 2006; Silva *et al.*, 2008).

The dry coffee processing has been reported to have characteristics that are not better than the washing processing (Mazzafera & Padilha, 2004), such as the presence of bitter taste (Evangelista *et al.*, 2014b), the production of propionic acid and isobutyrate (Bressani *et al.*, 2018), even the appearance of a medicinal or medicinal taste (Clarke & Macrae, 1985), as well as high levels of defective beans that produce earthy and musty characters (Tadesse *et al*., 2015). In addition, the dry process could also produce undesirable characteristics, sour, toasted, and bitter (Lee *et al.*, 2015), with a moderate level of body (Duarte *et al*., 2010). Although the wash processing produce better coffee qualities than the dry process, it needs a lot of water in the washing step. This is not an effective way and also needs more handling. Therefore, a new approach is needed in the coffee processing with the zero water and produce a high coffee qualities.

Improvement in coffee bean process can be done with a controlled fermentation treatment. Anaerobic fermentation is suggested to improve the quality of coffee beans (var. *Catuai amarelo*, *Ouro amarelo*, *Mundo novo*) generated from the dry processing. This approach is potential to produce new desired flavors and create zero waste water with easy handling in the coffee processing (Martinez *et al.*, 2017; Ribeiro *et al.*, 2017; Bressani *et al.*, 2018). During anaerobe fermentation, microorganisms utilize substrates for metabolic processes and produce compounds such as organic acids that may contribute in the flavor development. Therefore, this study aimed to evaluate the effect of anaerobic fermentation on sensory properties, profile of volatile compounds and non-volatile characteristics in the dry processing Gayo Arabica coffee. This study is considered to be useful for processing the coffee beans and provide an alternative way to produce the high quality coffee beans in Indonesia and worldwide.

MATERIALS AND METHODS

Fully ripe of Arabica coffee cherries (Ateng Super variety), which grown at an altitude of 1800 m above sea level (asl.) in Uning Bertih Village, Bener Meriah Regency, Aceh, Sumatra, Indonesia, was harvested manually (handpicked).

Sample Preparation

The coffee cherries were sorted manually to separate branches, leaves, and impurities then was finished by flotation using a water tube. The sinked coffee cherries as superior cherries were separated and used as research materials. The sample was divided into three groups i.e: Group A, the superior cherry was put into a plastic bag that is tightly closed for two days and immersed in water as an anaerob fermentation treatment. After that, the cherries were sun dried for two days, and kept at temperature of 19°C–45°C until the moisture <17% before dehulling process. Group B, the superior cherry was put into a plastic bag that was tightly closed for two days for anaerobic fermentation and pulped using pulper machine (Vis-pulper) before dried. The dried parchment was then dehulled and roasted. Group C, the superior cherries was directly pulped using pulper machine (Vis-pulper). Depulped cherries was put into a plastic bag that was tighty closed for anaerobic fermentation and kept for two days before dried, hulled and roasted. The condition of drying, hulling, and roasting process for all treatments were similar. Each plastic bag contained about 13 kg of superior cherries. Each group was repeated three times. Furthermore, group A, B, and C were named as dry, pulp natural and black honey treatments, respectively. The green bean was then roasted using

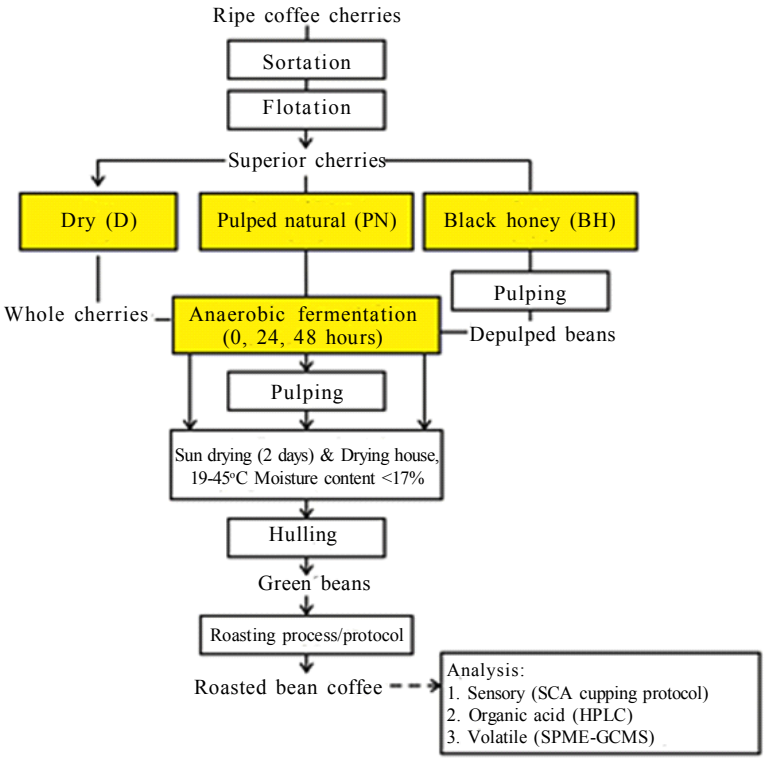

Figure 1. Flowchart of non-washed coffee processing

a roasting machine (Latina 800-N) at temperature of 200°C for 10 min. Roasted coffee beans were ground using a coffee grinder (Latina 600-N) with an average particle size of 20 mesh for further analysis.

Sensory Analysis

Sensory analysis of ground roasted beans was repeated three times and performed by three Q-Graders using the Specialty Coffee Association (SCA) cupping protocol. Coffee powder from each sample was weighed as much as 8.25 g and put into a ceramic glass. Coffee was brewed with water at 93°C until the volume reaches 150 mL. Brewing was done as much as five cups of each sample. Taste attributes consist of aroma, taste, acidity, body, balance, aftertaste, uniformity, sweetness, clean cup, and overall impression. The fragrance was evaluated by sniffing the ground coffee before brewing, while other attributes were evaluated by sipping. The aroma was detected between 0–4 minutes of brewing time, while taste, aftertaste, acidity, and body were analyzed between 10–20 minutes of brewing time. Sweetness, clean cup, and overall were done between 20–35 minutes of brewing time. Each attribute value was written on the score sheet by giving a value between 6 to 10 with an interval of 0.25. The total score of all attributes was ranged from 60 to 100.

Organic Acid Analysis

Organic acids were analyzed following the method of Ribeiro *et al.* (2010) using HPLC system (Shimadzu LC-20, autosampler). The organic acid analysis was done for citric acid, malic acid, lactic acid, acetic acid, and succinic acid. As much as 1.5 g of sample was added 10 mL of aqua pro in 15 mL volume falcon tube. The sample was shaken and left for 10 minutes. The pH of the supernatant was adjusted to 2 using 2 mL perchloric acid 0.2 M added into a falcon tube. The sample was then centrifuged at 10,000 G at 4°C for 10 minutes. The supernatant was filtered through a 0.45 µm cellulose filter. The filtrate was stored at 4°C before being injected to the HPLC system (Shimadzu LC-20, autosampler). The used column was AMINEX HPX-87H (300 × 7.8 mm) with a mobile phase of 0.1 M perchloric acid. The flow rate was 0.6 $mL.min^{-1}$ with an oven temperature of 30°C. The detector used PDA (Photometric Diode Array) with a wavelength of 215 nm. Identification and quantification of organic acids was done by comparing to calibration curve of authentic standards.

Sample Extraction

Volatile compounds extraction of roasted bean followed the method of Ribeiro *et al.* (2010) with slight modification. Samples extraction were done for control or without fermentation and fermentation for 48 hours. A total of 3 g of the roasted bean was ground using a coffee grinder (flat burr, Latina 600-N) with particle size approaching 20 mesh and put in vials 22 mL (Agilent). Samples were heated using a static water bath at 90°C for 30 minutes together with SPME fiber exposure. The fiber used was DVB/CAR/PDMS with a thickness of 50 µm (Supelco, Bellefonte, USA). 2,4,6-trimethyl pyridine 0.01% was added as an internal standard of 0.4 µL in each sample.

Volatile Compounds Analysis

Analysis of volatile compounds based on the method of Caporaso *et al.* (2018) with slight modification. GC-MS used was Agilent 7890A and Agilent 5975C equipped with DB-WAX (30 m x 250 mm x 0.25 mm)

column. Helium used as carrier gas at flow rate of 0.8 mL.min^{-1}. A splitless injetor was used. Injector and interface temperature were 250^{o}C and 230^{o}C respectively. The column was heated at 40^{o}C for two minutes then increased to 170^{o}C at 3^{o}C per minute, and rampped to 250^{o}C at a rate of 8^{o}C per minute. The GC-MS was set for measuring mass-to-charge (m/z) 29-550 atomic mass unit (amu) and identification of the compound refers to the National Institute of Standards and Technology (NIST) database. The C_{10}-C_{30} alkane series standard was used to determine the linear retention index value of the compounds identified. The relative percentage was calculated semi-quantitatively in parts per billion (ppb) units.

Statistical Analysis

Sensory and organic acid data analysis were done using SPSS (IBM Statistics 22) on one-way analysis of variance (ANOVA) with the statistical significance of differences ($p<0.05$). Duncan's multiple range test (DMRT) was used for specific differences means, while volatile compound analysis was analyzed through principal component analysis (PCA) using Minitab 18 software.

RESULTS AND DISCUSSION

Fermentation Condition

The pH value profile of the dry, pulped natural, and black honey samples are shown in Figure 2. The average pH value of the coffee beans before fermentation (0 hour) ranged from 6.5 to 6.7. The pH value in the samples then decreased significantly after 48 hours fermentation with a range value of 4-5.7. This was due to the fermentation process of the samples. Velmourougane (2013) found that during the fermentation of Arabica coffee (S 795 variety), there was a decrease in pH value from 5.43 to 4.71 which was fermented for 24 hours. During fermentation, biochemical reactions take place in different compounds. Generally, decreasing in pH value during fermentation was caused by degradation of organic compounds in mucilage into simple sugars by microorganisms which then produce acidic compounds in the fermented coffee. Furthermore, Avallone *et al.* (2001) stated that acidic compounds that reduces pH value in the range of 6.5 to 4.1 could affect the coffee flavor. The main microorganisms that dominated might be come from lactic acid bacteria (LAB) and some species of yeast such as *Saccharomyces* sp. and *Pichia* sp. with its main products such as organic acids and alcohol (Pereira *et al.*, 2017). Those three groups of microorganisms have been reported to contribute positively to the sensory characteristics of Arabica coffee. However, there is still a need for further research in this study to determine the group of microorganisms that grow during fermentation.

Fermentation temperatures of dry, pulped natural, and black honey samples were below 22^{o}C, this was due to the immersion of fermented coffee beans in the anaerobic condition. The temperature during fermentation process was around 13.3^{o}C–20.6^{o}C. Fermentation temperature was an important parameter that needs attention. Bertrand *et al.* (2012) stated that the acetic acid was easily accumulated in warm environmental temperatures like above 22^{o}C. However, pH values between temperature of 14.6^{o}C and 28.2°C did not show a positive correlation with the accumulation of acetic acid. In addition, Yusianto *et al.* (2007) stated that delayed pulping of coffee fruit at temperatures below 40^{o}C did not reduce the score of several flavor attributes.

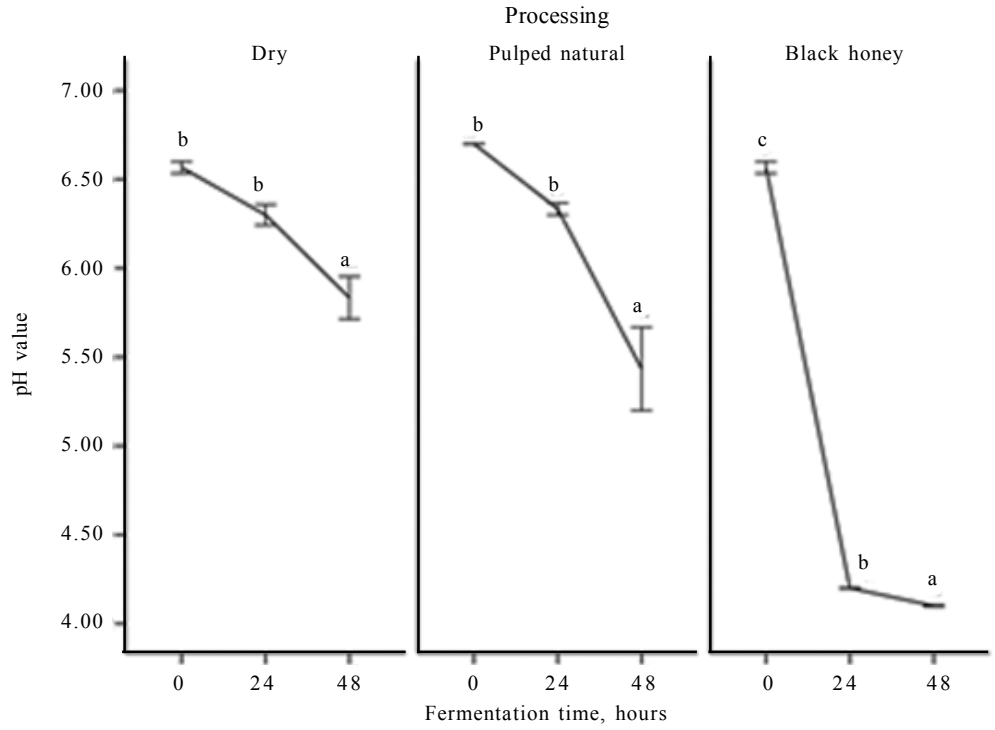

Figure 2. pH value profile of the dry, pulped natural, and black honey samples in aerobic fermentation. Means followed by the same letter for each attribute are not significantly different at p = 0.05 according to Duncan test

Sensory Analysis

The results of sensory analysis of dry, pulped natural, and black honey samples showed that there were significant differences in the attributes of aroma, flavor, aftertaste, acidity, body, balance, and overall, as well as the final score (Table 1). All of the treatments resulted in a final sensory score above 80 points. Those indicating all of the treatments were classified as specialty coffee (Ribeiro *et al.*, 2017). The fermentation treatment in the dry affected the aroma, flavor, acidity, and balance attributes. As well the fermentation treatment in the pulped natural also affected the aftertaste, acidity, overall, and final score attributes. The fermentation treatment in the black honey process affected the attributes of the body and overall. The highest sensory test scores and significantly increased were in pulped natural for 24 and 48 hours of fermentation.

Previous research related to the storage of coffee fruit in plastic containers at a temperature of 40°C before the pulping process has been done by Yusianto *et al.* (2007). The flavor attribute score decreased on fragrance, aroma, flavor, aftertaste, acidity, body, balance, overall, and total score. However, the storage of coffee fruit at temperatures below 40°C did not reduce the score of fragrance/aroma, flavor, aftertaste, acidity, body, balance, and overall. Dry process, pulped natural, and black honey have a temperature of no more than 22°C for 48 hours (data not shown). Temperature is one of many factors that supported the succession of microorganisms.

Each specific temperature range would be very suitable for the growth of certain microorganisms, so that it also affected the metabolites that would be produced. Ahmed *et al*. (2006) stated that lactic acid bacteria (LAB) was a mesophilic microorganism that

could grow in a temperature range of 20–30°C, and would be inhibited at 39°C. LAB produces lactic acid very slowly even at a temperature of 20°C so that the microorganisms that dominate the process in this study was predicted from the yeast group. Masoud *et al*. (2005), Masoud & Jespersen (2006), Silva *et al.* (2013), and Pereira *et al*. (2014) stated that the activity of microorganisms that degrade mucilage during fermentation could produce metabolites that could be diffused into coffee beans, then some chemical reactions occured and affected the coffee flavor.

The aroma characteristics of dry, pulped natural, and black honey roasted at medium level (210°C, 12 minutes) are shown in Table 2. The dry sample was dominated by chocolate and fruity characters. Chocolate and fruity characters were also the dominant characters found by Evangelista *et al.* (2016b) in the dry process of Brazilian Arabica coffee without the addition of a starter.

The pulped natural sample without fermentation was dominated by the character of nutty, caramelly, tobacco, while the other characters were floral, spicy, pineapple, and greenish. Unlike the case with the pulped

Table 1. Effect of various fermentation time on the score of cup test attributes in dry, pulped natural, and black honey samples

Attributes	Fermentation time (hours)	Processing		
		Dry	Pulped natural	Black honey
Aroma	0	8.08 ± 0.14[b]	8.08 ± 0.14	7.75 ± 0.00
	24	7.75 ± 0.43[b]	8.16 ± 0.28	8.00 ± 0.00
	48	7.25 ± 0.25[a]	8.16 ± 0.28	8.00 ± 0.00
Flavor	0	7.58 ± 0,14[a]	7.75 ± 0.00	7.66 ± 0.14
	24	8.00 ± 0.00[b]	7.91 ± 0.28	7.91 ± 0.14
	48	8.00 ± 0.00[b]	7.83 ± 0.14	7.66 ± 0.57
Aftertaste	0	7.50 ± 0.25	7.75 ± 0.00 [a]	7.58 ± 0.14
	24	7.58 ± 0.14	8.00 ± 0.00 [b]	7.66 ± 0.28
	48	7.58 ± 0.14	7.83 ± 0.14 [ab]	7.75 ± 0.00
Acidity	0	7.41 ± 0.14 [a]	7.25 ± 0.00 [a]	7.50 ± 0.25
	24	7.91 ± 0.14 [b]	7.58 ± 0.14 [b]	7.58 ± 0.14
	48	8.00 ± 0.00 [b]	7.66 ± 0.28 [b]	7.41 ± 0.38
Body	0	7.83 ± 0.28	8.00 ± 0.00	7.66 ± 0.14[a]
	24	7.75 ± 0.00	8.00 ± 0.00	8.00 ± 0.00[b]
	48	8.00 ± 0.00	8.08 ± 0.14	8.00 ± 0.00[b]
Balance	0	7.50 ± 0.00[a]	8.00 ± 0.00	7.83 ± 0.28
	24	7.91 ± 0.14[b]	8.00 ± 0.00	8.00 ± 0.00
	48	7.83 ± 0.14[b]	8.00 ± 0.00	7.66 ± 0.14
Uniformity	0	10 ± 0.00	10 ± 0.00	10 ± 0.00
	24	10 ± 0.00	10 ± 0.00	10 ± 0.00
	48	10 ± 0.00	10 ± 0.00	10 ± 0.00
Cleancup	0	10 ± 0.00	10 ± 0.00	10 ± 0.00
	24	10 ± 0.00	10 ± 0.00	10 ± 0.00
	48	10 ± 0.00	10 ± 0.00	10 ± 0.00
Sweetness	0	10 ± 0.00	10 ± 0.00	10 ± 0.00
	24	10 ± 0.00	10 ± 0.00	10 ± 0.00
	48	10 ± 0.00	10 ± 0.00	10 ± 0.00
Overall	0	7.41 ± 0.14	7.58 ± 0.14 [a]	7.66 ± 0.14 [a]
	24	7.66 ± 0.28	7.91 ± 0.28 [b]	7.50 ± 0.00 [a]
	48	7.66 ± 0.14	8.00 ± 0.00 [b]	7.91 ± 0.14 [b]
Final score	0	83.33 ± 0.28	84.41 ± 0.28 [a]	83.66 ± 0.62
	24	84.58 ± 1.04	85.58 ± 1.01 [b]	84.66 ± 0.28
	48	84.33 ± 0.14	85.58 ± 1.01 [b]	84.41 ± 1.01

Notes: Data are presented as mean ± standard deviation. Means followed by same letter for each attribute are not significantly different at $p < 0.05$ according to Duncan test. The final score classification was divided into four, there are not-specialty (less than 80), good & specialty (80–84.99), very good & specialty (85–89.99) , and extraordinary (90-100).

Table 2. The aroma characteristics of dry, pulped natural, and black honey at three variations of fermentation time

Treatments	Aroma
D-0	Dark chocolate, flat, nutty, silky, sweet melon, citrussy, chocolate, fruity, sour, boring, roasted peanut
D-24	Fruity, nutty, chocolate, rich, full body, sweet lemon, floral, slightly sour, astringency, roasted peanut, spicy
D-48	Jackfruit, floral, dry, bright nutty, sour, fruity, slightly tarty, boring, roasted peanut, citrussy, silky
PN-0	Nutty, caramelly, floral, spicy, pineapple, tobacco, greenish
PN-24	Butter nut, pineapple, floral, sweet orange, fruity, spicy, nutty, chocolate, tarty, creamy
PN-48	Roasted peanut, butter nut, floral, heavy body,fruity, caramelly, nutty, smoky, tobacco, tarty, herby, tea-like
BH-0	Nutty, chocolate, flat, tobacco, herby, greenish, bitter, floral
BH-24	Sweet, buttery, floral, juicy, bit honey, nutty, dry, spicy, woody, tobacco, creamy, silky, herby, greenish
BH.48	Sweet floral, nutty, chocolate, sunkist, fruity, tobacco, spicy, lemony

Notes: D (dry process), PN (pulped natural), BH (black honey), 0 (without fermentation), 24 (24 hours fermentation), 48 (48 hours fermentation).

natural sample which was fermented for 24 hours, some of the characters that found were fruity, sweet orange, spicy, butternut, tarty, and creamy. Likewise with pulped natural fermented for 48 hours, there were found herby and tea-like characters. The dominant caramelly character in pulped natural has also been found in previous studies conducted by Evangelista *et al.* (2016) in a semi-dry process (black honey) of Brazilian Arabica coffee (var. Acaia) without anaerobic fermentation.

Black honey without fermentation was dominated by the character of nutty, chocolate, and herby, but also found flat, tobacco, greenish, bitter. Bitter and herby taste observed in without fermetation coffee allegedly due to the presence of *Saccharomyces cerevisiae* and *Pichia guilliermondii* that accidentally grow on coffee beans (Evangelista *et al*., 2014). However, these allegations need to be answered through further research for the purposes of identifying the types of microbes that grow during the process. When treated with 24 hours fermentation, the aroma characters began to vary, such as juicy, honey, spicy, woody, creamy characters, and in the 48 hours fermentation, the sunkist and lemony characters were found. The complex aroma characteristic in the black honey treatment could be suspected because of the high population of yeast that grows when drying coffee beans.

Organic acids

The content of organic acids in dry, pulped natural, and black honey is shown in Table 3. Malic acid, citric acid, and lactic acid dominated the roasted beans. The malic acid and citric acid from all of the treatments were more than 1 $mg.g^{-1}$. The presence of malic and citric acids may provide delight acidity characters of the coffee (Avallone *et al.*, 2002; Schwan *et al*., 2012; and Evangelista *et al.*, 2016b). Sivetz (1963) stated that the content of citric acid and malic acid which was more than 1 $mg.mL^{-1}$ could provide good acidity in brewed coffee. Indonesian green bean Arabica coffee can produce lactic acid as much as 1,07 $mg.g^{-1}$, even with the presence of a starter and the addition of glucose as a substrate during fermentation, the amount of lactic acid can increase to 5.2 times (Wang *et al*., 2019). Sensory qualities of citric acid, malic acid, and lactic acid were burst tart, smooth tart, and sharp tart, respectively (Neta *et al*., 2007). Those acids have a low threshold so they could give good acidity.

Balzer (2001) and Ginz *et al.* (2000) stated that citric acid, malic acid, and acetic acid were produced by the degradation of carbohydrates in the initial roasting stage, the amount of that acids decreased as the temperature of the roasting gets higher. Sucrose is the main organic acid precursor that contains between 3–8% of the green

Table 3. Organic acids content of roasted bean of dry, pulped natural, and black honey in different fermentation time

Processing	Fermentation time (hours)	Organic acids (mg.g^{-1} dry bean)				
		Citric	Malic	Succinic	Lactic	Acetic
Dry	0	3.03 ± 0.78	5.68 ± 1.75	0.05 ± 0.00	3.69 ± 0.22	0.80 ± 0.43
	24	2.35 ± 0.31	7.18 ± 1.54	0.05 ± 0.00	2.31 ± 1.56	0.44 ± 0.19
	48	2.56 ± 1.03	9.36 ± 1.15	0.47 ± 0.59	3.37 ± 0.16	0.62 ± 0.19
Pulped natural	0	2.57 ± 0.48	10.01 ± 1.42	1.26 ± 0.45	3.34 ± 1.10	0.53 ± 0.20
	24	2.78 ± 0.06	11.66 ± 1.56	1.01 ± 0.92	4.05 ± 1.69	0.68 ± 0.37
	48	2.54 ± 0.50	10.69 ± 4.49	1.36 ± 0.92	3.23 ± 0.89	0.58 ± 0.13
Black honey	0	2.64 ± 0.51	11.70 ± 0.55	1.39 ± 0.49	3.47 ± 0.44	0.51 ± 0.12
	24	2.41 ± 0.55	13.95 ± 0.39	1.26 ± 0.31	5.38 ± 0.39	0,64 ± 0.16
	48	2.91 ± 0.11	10.41 ± 2.25	1.32 ± 0.36	3.71 ± 0.21	0.62 ± 0.23

Note: Data are presented as mean ± standard deviation.

bean. Sucrose is first converted to fructose and glucose in the caramelization process during roasting. Most organic acids are non-volatile compounds that contribute to the acidic or sour taste of food including brewed coffee. Neta *et al*. (2007) stated that the perception of sour taste was chemically and physically complex. Acidic taste is not related to hydrogen ions (pH), and it could not be explained completely with titrated acid, buffering capacity, molar concentration, physical and chemical structure.

Volatile compounds

Identification of volatile compounds in roasted beans of dry, pulped natural, and black honey was done for two fermentation time, 0 hour and 48 hours. The classes and concentration of volatile compounds were shown in Figure 3. There were several factors affecting the composition of volatile compounds, such as green bean, geographical conditions, postharvest, environmental conditions during harvest, defective coffee beans, and level of maturity (Toledo *et al.*, 2016). Acid, furan, and alcohol were the three classes of compounds that dominated all the samples. Some compounds that have high concentrations were acetic acid, 2-methyl-butanal, 3-methyl-butanoic acid, 2-furanmethanol, furfuryl acetate, furfural, acetylfuran, 5-methyl-furfural, 4-vinylguaiacol, methylpyrazine, and 3-ethyl-2,5-dimethyl-pyrazine. High acid and aldehyde compounds have been reported also as compounds that distinguished Arabica and Robusta coffee in roasted beans (Caporaso *et al.*, 2018).

Coffee dry produced several compounds that were not produced by pulped natural and black honey. These compounds were formic acid, ethanol, 3-hexanol, isopropyl alcohol, coffee furanone, a-angelica lactone, 3-hexanone, pyrazine, ethylpyrazine, 2-ethyl-3-methyl-pyrazine, 2-methylpyridine, 2-formyl-1 -methyl pyrrole, and 2-methyl-1,3-dithiane (data not shown). These compounds were key compounds that distinguished the dry sample with other samples. Dry coffee has a high content of acetic acid while pulp natural and black honey were dominated by furan, phenol, and pyrazine. Acetic acid contributes to the scent of pungent and vinegar while furan, phenol, and pyrazine provide complex scents such as sweet, buttery, bread, clove, savory, roasted, and nutty.

Flament (2001) stated that volatile carboxylic acids were unexpected acids because it produced negative scents such as vinegar and sweaty. Although some types of acids contributed positively such as the appearance of cheese, cream, and chocolate. Ginz *et al.* (2000) said that the concentration of acetic acid and formic acid would increase when the coffee was roasted in medium levels,

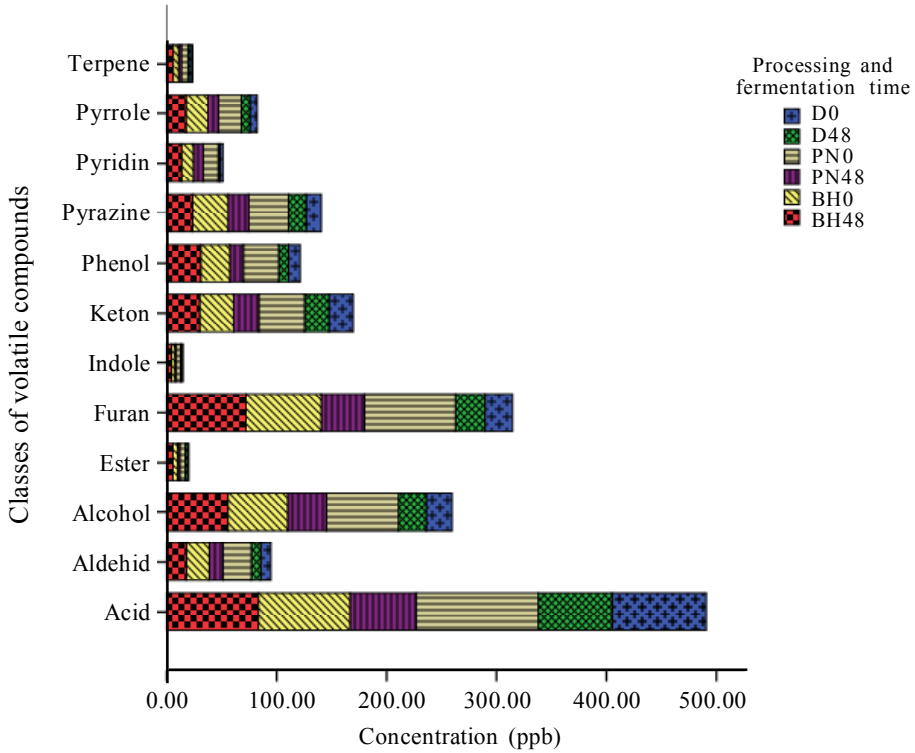

Figure 3. Classes of volatile compounds identified in anaerob fermentation of dry (D), pulped natural (PN), and black honey (BH)

but it would be degraded for the next. Organic acids could also be derived from carbohydrate degradation as a precursor. The high acetic acid in dry was also described by sour scent by panelists in sensory analysis, where, the sour scent was only found in the dry sample.

The compounds that contributed to the aroma in the dry, pulped natural, and black honey processes could be broken down based on an odor active value greater than 1 (Table 4). Amanpour & Selli (2016) stated that the odor active value (>1) of a compound could contribute to the aroma of coffee. Odor active value is the potential value of compounds that contribute to the aroma which is calculated from the ratio between the concentration of the compound in coffee with the threshold odor of the compound (Kulapichitr *et al.*, 2019). β-damascenone, 4-vinylguaiacol, and 2-methylbutanal were the volatile compounds that most powerfull contributed on the coffee aroma. The odor active value of those compunds were 11.506, 79.76, and 57.98 respectively.

The compounds that contributed to the aroma were processed using principal component analysis (PCA). Based on principal component analysis, the first component or PC 1 was sufficient to classify volatile compounds and the treatment of 96.4% of the total variance (Figure 4, Figure 5). Based on the plot score, almost all compounds are in the negative PC 2 quadrant. The negative PC 2 quadrant has separated β-damascenone (terpene) and coffee furanone (furan). β-damascenone is a compound that has the highest odor active value of all treatments on pulped natural and black honey while coffee furanone is a compound that gives character to the dry sample. β-damascenone compounds have

aroma characteristics such as honey-like, fruity, apple, and rose aromas, whereas coffee furanone has the characteristics of bready and sweet aroma. The fruity and rose scents of the β-damascenone compound are also shown by the presence of the dominant fruity and floral scents in the dry, pulped natural, and black honey samples. Likewise, the bready and sweet aroma of the coffee furanone compound approaches the dominant chocolate aroma in the dry sample. Blank *et al.* (1992) and Grosch (1998) stated that β-damascenone was a compound that has a great effect on the aroma of roasted coffee grounds with a low threshold.

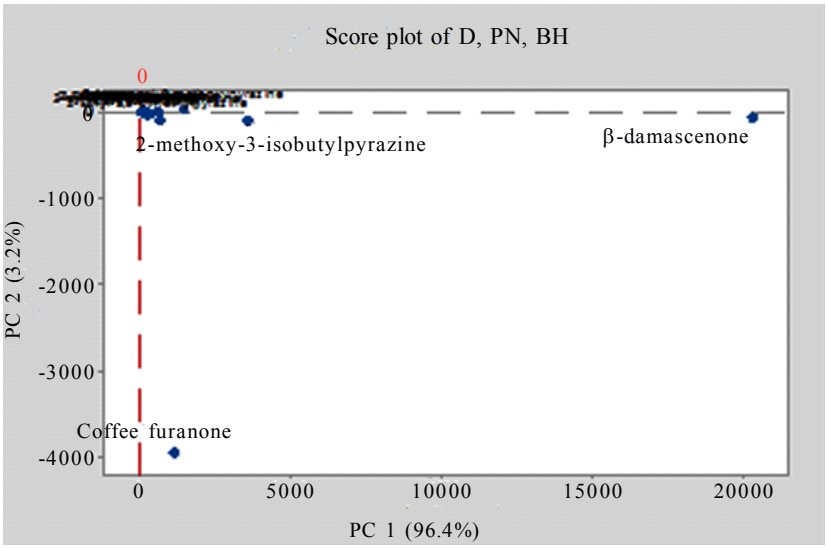

Figure 4. Score plot for distinguishing aroma compounds in the fermentation process from the first two main components (PC1 and PC2)

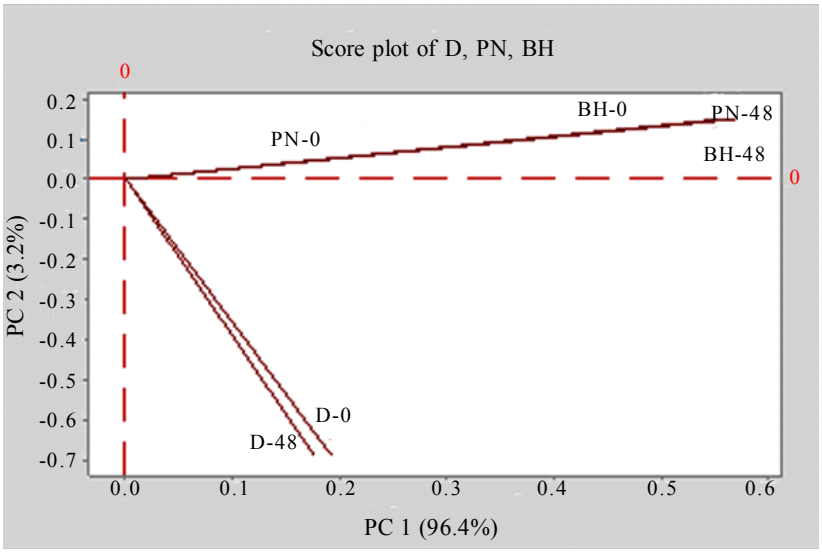

Figure 5. Loading plot for distinguishing the dry and semi-dry processes of the first two main components (PC1 and PC2)

Table 4. Profile of selected aroma compounds of roasted coffee beans (dry, pulped natural, black honey) with OAV values > 1 analyzed by SPME GC-MS.

Chemical group	Compounds	CAS number	LRI Exp[a]	LRI Ref[b]	Odor threshold (ppb)[c]	Concentration (ppb)						OAV[d]						Aroma description[e]
						D-0	D-48	PN-0	PN-48	BH-0	BH-48	D-0	D-48	PN-0	PN-48	BH-0	BH-48	
Aldehide	2-Methylbutanal	96-17-3	< 1042	925	1	32.5	26.4	102.5	49.39	76.9	58.0	32.5	26.4	102.5	49.4	76.9	58.0	Cocoa, almond, malt, chocolate
Furan	Furfural	98-01-1	1459	1459	3000	166	157	464	228	369	415	0.06	0.05	0.15	0.08	0.12	0.14	Sweet, woody, almond, bread
Ketone	2,3-Butanedione	431-03-8	< 1042	995	15	14.1	19.1	35.0	15.2	19	15.2	0.94	1.27	2.34	1.01	1.27	1.01	Buttery, creamy
Ketone	2,3-Pentanedione	600-14-6	1059	1073	20	9.1	9.5	63.1	30.5	47.3	49.3	0.30	0.32	2.10	1.02	1.58	1.64	Buttery, creamy
Ketone	Furaneol	3658-77-3	2033	1485	5	17.8	15.1	35.3	11.5	29.2	27.9	0.57	0.49	1.14	0.37	0.94	0.90	Caramel, sweet
Phenol	4-Vinylguaiacol	7786-61-0	2188	2186	0.75	77.3	58.1	232.8	77.6	180.5	239.3	25.8	19.4	77.6	25.9	60.2	79.8	Clove, curry
Phenol	Guaiacol	90-05-1	1851	1--848	3	11.9	11.6	29.3	13.0	24.9	25.1	3.97	3.88	9.78	4.36	8.28	8.35	Phenolic, burnt, smoky, sweet, savory, woody
Pyrazine	Methylpyrazine	109-08-0	1263	1262	60	67.3	86.5	196.4	103.4	160.6	7.1	1.12	1.44	3.27	1.72	2.68	0.12	nutty, cocoa-like, roasty
Pyrazine	2-Ethyl-6-methylpyrazine	13925-03-6	1383	1383	30	8.2	6.9	48.5	25.2	47.3	39.2	0.27	0.23	1.62	0.84	1.58	1.31	Flowery, fruity, hazelnut-like
Pyrazine	2,6-diethylpyrazine	13067-27-1	1433	1444	6	0.00	0.00	7.59	3.12	8.25	8.72	0.00	0.00	1.27	0.52	1.38	1.45	Nutty
Pyrazine	3-ethyl-2,5-dimethyl-pyrazine	13360-65-1	1446	1431	1	53.9	62.4	144.3	72.1	118.6	125.5	6.3	7.3	16.8	8.4	13.8	14.6	Earthy, roasted
Pyrazine	2-Ethyl-3,5-dimethyl-pyrazine	13925-07-0	1461	1457	0.04	5.73	7.93	11.58	6.79	12.36	11.50	5.73	7.93	11.6	6.79	12.4	11.5	Earthy, roasted
Pyrrole	2-Formyl-1-methylpyrrole	1192-58-1	1608	1601	40	6.8	8.9	25.7	12.4	23.9	20.6	0.17	0.22	0.64	0.31	0.60	0.52	Roasted, nutty
Terpene	β-damascenone	23726-93-4	1810	1813	0.00075	2.93	2.66	8.47	3.41	7.56	8.63	3907	3547	11293	4547	10080	11507	Honey-like, fruity, apple, rose

Notes: LRI (linier retention indices) experiment was determined from the calculation of the retention time of each compound with the C_{10}-C_{30} series alkane standard; LRI Reference were taken from Kulapichitr *et al*. (2019), Caporaso *et al.* (2018), Cheong *et al.* (2013), Lee *et al.* (2017); Odor threshold were taken from Caporaso *et al*. (2018), Kulapichitr *et al.* (2019); OAV (order activity value) was calculated as the ratio between compound concentration (ppb) and threshold (ppb); Aroma description were taken from Caporaso *et al*. (2018), Kulapichitr *et al*. (2019), Grosch (1998).

CONCLUSIONS

The fermentation treatment could significantly increase the total score of sensory analysis in the pulped natural process by 85.58±1.01 with the characteristics of nutty, fruity (pineapple, sweet orange), floral, and caramelly. Citric, malic, and lactic acids identified by the HPLC system were the acids that dominated on roasted coffee beans which contribute positively to the flavor. Analysis of volatile compounds using SPME-GCMS produced 20 volatile compounds that have an order activity value >1. β-damascenone was the compound that has the highest order activity value which has a honey-like, fruity, apple aroma, and rose aroma. Pulped natural process was a process that was suitable to be applied in Gayo Arabica coffee processing without washing steps in humid climate conditions and high altitudes.

REFERENCES

Ahmed, T.; R. Kanwal & N. Ayub (2006). Influence of temperature on growth pattern of *Lactococcus lactis*, *Streptococcus cremoris* and *Lactobacillus acidophilus* isolated from camel milk. *Biotechnology*, 5(4), 481–488.

Amanpour, A. & S. Selli (2016). Differention of volatile profiler and order activites value of Turkish coffee and Frech fress coffee. *Jouenal of Food Processing and Preservation,* 40(5), 1116-1124.

Avallone, S.; J.M. Brillouet; B. Guyot; E. Olguin & J.P. Guiraud (2002). Involvement of pectolytic micro-organisms in coffee fermentation. *International Journal of Food Science and Technology*, 37, 191–198.

Avallone, S.; J.-M. Brillouet; E. Olguin; B. Guyot; J.P. Guiraud & S. Avallone (2001). Microbiological and biochemical study of coffee fermentation. *Current Microbiology*, 42(4), 252–256.

Balzer, H. (2001). Acids in coffee. p. 18–32. ***In:*** *Coffee. Recent Developments*. Blackwell Science, London.

Bertrand, B.; R. Boulanger; S. Dussert; F. Ribeyre; L. Berthiot; F. Descroix & T. Joët (2012). Climatic factors directly impact the volatile organic compound fingerprint in green Arabica coffee bean as well as coffee beverage quality. *Food Chemistry*, 135(4), 2575–2583.

Blank, I.; A. Sen & W. Grosch (1992). Potent odorants of the roasted powder and brew of Arabica coffee. *Zeitschrift für Lebensmittel-Untersuchung und-Forschung*, 195(3), 239–245.

BPS (2018). *Indonesian Coffee Statistics*. Badan Pusat Statistik, Indonesia.

Bressani, P.; A. Paula; S. Juliana; S. Reis; D. Ribeiro & R. Freitas (2018). Characteristics of fermented coffee inoculated with yeast starter cultures using different inoculation methods. *LWT-Food Science and Technology*, 92, 212–219.

Caporaso, N.; M.B. Whitworth; C. Cui & I.D. Fisk (2018). Variability of single bean coffee volatile compounds of Arabica and Robusta roasted coffees analysed by SPME-GC-MS. *Food Research International*, 108, 628–640.

Cheong, M.W.; K. Hin; J. Jian; M. Ong; S. Quan; P. Curran & B. Yu (2013). Volatile composition and antioxidant capacity of Arabica coffee. *Food Research International*, 51(1), 388–396.

Clarke, R & R. Macrae (1985). *Coffee: Chemistry*. 1st eds. Elsevier Applied Science Publishers, London.

DGEC (2017) *The Crop Estate Statistics of Indonesia*. Directorate General of Estate Crops, Ministry of Agriculture, Jakarta.

Duarte, G.; A. Pereira & F. Adriana (2010). Chlorogenic acids and other relevant compounds in Brazilian coffees processed by semi-dry and wet post-harvesting methods. *Food Chemistry*, 118(3), 851–855.

Evangelista, S.R.; C.F. Silva; M.G.P.C. Miguel; C.S. Cordeiro; A.C.M. Pinheiro; W.F. Duarte & R.F. Schwan (2014). Improvement of coffee beverage quality by using selected yeasts strains during the fermentation in

dry process. *Food Research International*, 61, 183–195.

Evangelista, S.R.; M.G.P.C. Miguel; C.S. Cordeiro; C.F. Silva; A.C.M. Pinheiro & R.F. Schwan (2014). Inoculation of starter cultures in a semi-dry coffee (*Coffea arabica*) fermentation process. *Food Microbiology*, 44, 87–95.

Flament (2001) .The Individual Constituent: Structure, nomenclature, origin, chamical and organoleptic properties. p. 81–335. ***In:*** *Coffee Flavor Chemistry*. John Wiley and Sons, New York.

Giacalone, D.; T.R. Fosgaard; I. Steen & M. Münchow (2016). Quality does not sell itself. *British Food Journal*, 118(10), 2462–2474.

Ginz, M.; H.H. Balzer; A.G.W. Bradbury & H.G. Maier (2000). Formation of aliphatic acids by carbohydrate degradation during roasting of coffee. *European Food Research and Technology*, 211(6), 404–410.

Grosch, W. (1998). Flavour of coffee. A review, *Nahrung - Food*, 42(6), 344–350.

Hulupi, R.; D. Nugroho & Yusianto (2013). Keragaman beberapa varietas lokal kopi Arabika di dataran tinggi Gayo. *Pelita Perkebunan*, 29(2), 69–81.

Joët, T.; A. Laffargue; F. Descroix; S. Doulbeau; B. Bertrand; A. De & S. Dussert (2010). Influence of environmental factors, wet processing and their interactions on the biochemical composition of green Arabica coffee beans, *Food Chemistry*, 118(3), 693–701.

Knopp, S.; G. Bytof & D. Selmar (2006). Influence of processing on the content of sugars in green Arabica coffee beans. *European Food Research and Technology*, 223, 195–201.

Kulapichitr, F.; C. Borompichaichartkul; I. Suppavorasatit & K.R. Cadwallader (2019). Impact of drying process on chemical composition and key aroma components of Arabica coffee. *Food Chemistry*, 291, 49–58.

Lee, L.W.; G. Yu; M. Wai; P. Curran & B. Yu (2017). Modulation of the volatile and non-volatile profiles of coffee fermented with *Yarrowia lipolytica*: I. Green coffee. *LWT - Food Science and Technology*, 77, 225–232.

Lee, L. W.; M.W. Cheong; P. Curran; B. Yu & S.Q. Liu (2015). Coffee fermentation and flavor-An intricate and delicate relationship. *Food Chemistry*, 185, 182–91.

Martinez, J.; A. Paula; P. Bressani; M. Gabriela; P. Miguel; D. Ribeiro & R. Freitas (2017). Different inoculation methods for semi-dry processed coffee using yeasts as starter cultures. *Food Research International*, 102, 333–340.

Masoud, W. & L. Jespersen (2006). Pectin degrading enzymes in yeasts involved in fermentation of *Coffea arabica* in East Africa. *International Journal of Food Microbiology*, 110(3), 291–296.

Masoud, W.; L. Poll & M. Jakobsen (2005). Influence of volatile compounds produced by yeasts predominant during processing of *Coffea arabica* in East Africa on growth and ochratoxin A (OTA) production by *Aspergillus ochraceus*. *Yeast*, 22(14), 1133–1142.

Mazzafera, P. & R.P. Padilha (2004). Post harvest processing methods and alterations in coffee fruit. *Proceedings of 20th Colloque Coffee*. India.

Mori, E.E.M.; N. Bragagnolo; M.A. Morgano; V.D.A. Anjos; K. Yotsuyanagi & V.E. Faria (2003). Brazil coffee growing regions and quality of natural, pulped natural and washed coffees. *Food and Food Ingredients Journal of Japan*, 208(1), 416–423.

Neta, E.R.D.C.; S.D. Johanningsmeier & R.F. Mcfeeters (2007). The chemistry and physiology of sour taste - A Review. *Journal of Food Science*, 72(2), 33–38.

Pereira, G.V. de-M.; V.T. Soccol; A.L. Gollo; A. Pandey; C.R. Soccol; A.B.P Medeiros & J.M.R.A Lara (2014). Isolation, selection and evaluation of yeasts for use in fermentation of coffee beans by the

wet process. *International Journal of Food Microbiology*, 188, 60–66.

Pereira, G.V. de-M.; V.T. Soccol; S.K. Brar; E. Neto & C.R. Soccol (2017). Microbial ecology and starter culture technology in coffee processing. *Critical Reviews in Food Science and Nutrition*, 57(13), 2775–2788.

Ribeiro, L.S.; M.G.da-C.P. Miguel; S.R. Evangelista; P.M.M. Martins; J. van-Mullem; M.H. Belizario & R.F. Schwan (2017). Behavior of yeast inoculated during semi-dry coffee fermentation and the effect on chemical and sensorial properties of the final beverage. *Food Research International*, 92, 26–32.

Ribeiro, J.S.; R.F. Teófilo; F. Augusto & M.M.C. Ferreira (2010). Simultaneous optimization of the microextraction of coffee volatiles using response surface methodology and principal component analysis. *Chemometrics and Intelligent Laboratory Systems*, 102(1), 45–52.

Samoggia, A. & B. Riedel (2018). Coffee consumption and purchasing behavior review: Insights for further research. *Appetite*, 129, 70–81.

Schwan, R.; C.S. Ferreira & L.R. Batista (2012). Coffee fermentation. p. 677–690. ***In:*** *Handbook of Plant-Based Fermented Food and Beverage Technology*. Boca Raton, FL.

Silva, C.F.; L.R. Batista; L.M. Abreu; E.S. Dias & R.F. Schwan (2008). Succession of bacterial and fungal communities during natural coffee (*Coffea arabica*) fermentation. *Food Microbiology*, 25(8), 951–957.

Silva, C.F.; W.F. Duarte; D.M. Vilela; D.R. Dias; R.F. Schwan & C.de-S. Cordeiro (2013). Evaluation of a potential starter culture for enhance quality of coffee fermentation. *World Journal of Microbiology and Biotechnology*, 29(2), 235–247.

Sivetz, M. (1963). *Coffee Processing Technology*. Westport, Connecticut.

Sunarharum, W.B.; D.J. Williams & H.E. Smyth (2014). Complexity of coffee flavor: A compositional and sensory perspective. *Food Research International*, 62, 315–325.

Tadesse, F.T.; Y. Jemal & H. Abebe (2015). Effect of green coffee processing methods and roasting temperatures on physical and cup quality of Sidama coffee, Southern Ethiopia. *Journal of Nutritional Ecology and Food Research*, 3(1), 44–50.

Toledo, P.R.A.B.; L. Pezza; H.R. Pezza & A.T. Toci (2016). Relationship between the different aspects related to coffee quality and their volatile compounds. *Comprehensive Reviews in Food Science and Food Safety*, 15(4), 705–719.

Velmourougane, K. (2013). Impact of natural fermentation on physicochemical, microbiological and cup quality characteristics of Arabica and Robusta coffee. *Proceedings of the National Academy of Sciences India Section B - Biological Sciences*, 83(2), 233–239.

Wang, C.; J. Sun; B. Hassobliere; B. Yu; Zhoi; Feifei; Fongjun; Y. Chen & S. Hin (2019). Potential of lactic acid bacteria to modulate coffee volatiles and effect of glucose supplementation: Fermentation of green coffee beans and impact of coffee roasting. *Journal of the Science and Agriculture,* 99(1), 409–420.

Yusianto; R. Hulupi; S. Mawardi & C. Ismayadi (2007). Mutu fisik dan cita rasa beberapa varietas kopi Arabika harapan pada beberapa periode penyimpanan. *Pelita Perkebunan*, 3(90), 205–230.

0