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# Sensory and Physicochemical Characteristics of Two Common Roast Defects in Robusta Coffee

Wenny B Sunarharum [a,*], Tunjung Mahatmanto [a], Dego Y Ali [a], Yuniar P Prananto [b], Novianus Efrat [c], Ngesti E Asih [a], Nindya G Mirillia [a]

[a] *Department of Agricultural Product Technology, Universitas Brawijaya, Jalan Veteran, Malang, 65145, Indonesia*
[b] *Department of Mathematics and Natural Sciences, Universitas Brawijaya, Jalan Veteran, Malang, 65145, Indonesia*
[c] *University of Missouri, Columbia, United States*
*Corresponding author: *wbsunarharum@ub.ac.id*

***Abstract*— Roasting is an important coffee processing step to generate coffee aroma and flavor. Because roasting is time-temperature dependent, the variation of time and temperature applied may influence the structural properties, visual appearance, and chemistry of coffee. Improper roasting creates roast defects that reduce coffee quality and acceptance. Despite this importance, studies on coffee roast defects, particularly in Robusta coffee is limited. This study aims to characterize two common roast defects, i.e., underdeveloped and overdeveloped, compared with medium roast in Robusta coffee. Sensory evaluation by trained panelists and physicochemical evaluation reveal that the two common roast defect in Robusta coffee can be distinguished clearly through differences in sensory (aroma defect) characteristics as well as physicochemical properties. The overdeveloped roast defect produced darker coffee with the highest pH and total dissolve solids (TDS), and can be characterized by pyridine, furan, phenol and pyrrole derivatives. The carbony and ashy notes of the overdeveloped coffee were potentially contributed by phenol and polyphenol derivatives. In contrast to the overdeveloped coffee, the underdeveloped coffee is markedly characterized by higher concentration of aliphatic acids and higher concentration of pyrazines that contributes to raw nut-like notes. The combination of time and temperature during roasting influences the breakdown of chemical compounds through complex mechanisms involving proteins, carbohydrates and polyphenols degradation. Thus, roasting process variations that determine coffee cup quality and in turn drive consumer acceptance should be controlled.**

***Keywords*— Aroma; coffee; roasting defect; sensory; volatile.**

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## I. INTRODUCTION

Coffee is one of the most popular commodities worldwide consumed due to its pleasant flavor and caffeine stimulation effect. Indonesia is the fourth largest coffee-producing country after Brazil, Vietnam, and Colombia, producing around 11.2 million 60-kg bags in 2019 [1]. Coffee consumption has been continuously growing in the last decade, including producing countries such as Indonesia. Indonesian coffee production is dominated by Robusta coffee, reaching 83% of the total coffee produced in the country [2].

The growing consumption of coffee has led to increased demand for high-quality coffee. Many factors influence the coffee quality, from on-farm agroecological factors post-harvest processing to final product preparation. These factors include biochemical composition, genetic variation, growing conditions, the origin of coffee beans, post-harvest handling, roasting, grinding and brewing methods, and serving conditions. Readers are referred to a review by Sunarharum *et al*.[3]. Among coffee processing steps, roasting is regarded as one of the key processes where the most coffee aroma is created [4]. Roasting is an intricate process that is dependent on time and temperature. In addition, the speed of roasting influences the composition of volatile coffee compounds [5]. Roasting is not only influencing the degree of dehydration and internal temperature of coffee beans but also changes the chemical composition and microstructural properties of coffee beans, leading to the development of specific coffee color, aroma, and flavor [3], [6]–[13]. In principle, roasting can be classified into three levels, i.e., light, medium, and dark roasting [2], [14]. Different roasting levels can create different aroma profiles and influence cup quality and consumer perception [15]. The different aroma profiles created by different roasting levels can be assessed through cupping by coffee experts following standard protocols developed by the

Specialty Coffee Association America (SCAA) or through detailed sensory profiling by a group of trained panelists using laboratory-based sensory descriptive analysis. Medium roasted coffee generally exhibits a more complex aroma than light and dark roasted coffees. Lightly roasted coffee has a sweet, cocoa, and nutty aroma, whereas dark roasted coffee has burnt/acrid, ashy/sooty, sour, pungent, and roasted notes [16]. The coffee aroma profiled will be influenced by the specific coffee used, roasting time-temperature parameters, differences between flavor and orthonasal aroma testing, lack of panelist training, and many other variables [17].

Although several factors might cause defects in coffee cup quality, improper roasting is one of the most important ones. Problems during coffee roasting can create off-flavors or defects in the coffee brew, indicated by its aroma [7]. Previous studies have shown a close relationship between the roasting process and specific coffee aroma profiles [7], [15], [18]. Common roast defects in Arabica coffee include dark, light, scorched, baked, and underdeveloped. The aroma profiles also differ for each roast defect such as hay/straw, burnt, mothball, smoky, spicy, bitter, astringent, roasted, ethereal or solvent-like pungent aroma and burnt, licorice, and tobacco flavor as influenced by their volatile compound composition [7], [15]. Despite extensive studies on coffee roasting, information regarding roast defects is still limited. Few articles published have reported common roast defects on Arabica coffee [7], [15], [19]–[22]. However, Robusta coffee appears to be less explored, probably due to the higher popularity of Arabica coffee. This study investigates the effects of two common roasting process defects, i.e., underdeveloped and overdeveloped roastings, on Robusta coffee's sensory and physicochemical characteristics.

## II. Materials and Methods

Green coffee beans were collected and roasted with two roast defect profiles, i.e., underdeveloped and overdeveloped. Medium roasting was used as a comparison. Here, underdeveloped roasting is defined as coffee roasting that is ended before the first crack, whereas overdeveloped roasting is defined as coffee roasting that is ended after the second crack continues. Further processing includes grinding and brewing. The round-roasted coffee and brew were analyzed for physical and chemical characteristics. The coffee brew was further analyzed for sensory profile (Fig. 1). The experimental steps are described in the following.

### *A. Coffee Sample and Preparation*

Robusta green coffee beans (natural) used in this study were obtained from Sridonoretno coffee farmer, Dampit, Malang, East Java. Green coffee beans were roasted at three levels (underdeveloped, medium, and overdeveloped) using W1A Giesen coffee roaster, The Netherlands (drum speed: 55 Hz; airflow: 110 Pa; temperature in 175°C; turning point: 1 min 30 s). Roasting time was 8 min 24 s (underdeveloped), 10 min 31 s (medium), and 11 min 9 s (overdeveloped). The development time (and development percentage) for medium and overdeveloped roasting was 1 min 38 s (15.5%) and 2 min 27 s (22.0%). The temperature for underdeveloped, medium, and overdeveloped roasting was 176°C, 194°C, and 206°C, respectively. Before further analysis, the three roasted coffee beans were packed in sealed coffee bags.

For sensory evaluation, samples were freshly ground to a medium-coarse size. Brewing was performed using the French press method [30], where 11 g of ground coffee sample was brewed in 200 mL hot water (± 95°C).

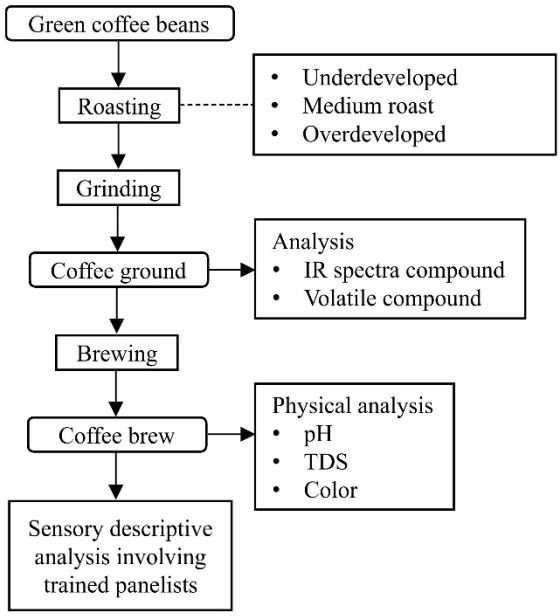


Fig. 1 General research scheme

### *B. Physical Analysis*

The pH of the coffee brew samples was analyzed using a hand digital pH meter (ATC). Total dissolved solids (TDS; expressed as % Brix) were analyzed using a VST Lab coffee refractometer. The color was analyzed using a color reader (Konica Minolta). Samples were analyzed in triplicates.

### *C. Sensory Evaluation*

Sensory evaluation was performed using the sensory descriptive analysis method [23], employing trained panelists. For this purpose, selected panelists were required to follow training before formal analysis, where the coffee brew samples were evaluated. The evaluation focuses on coffee defect aroma.

*1) Panelist Screening and Training:* Sensory panelists were screened based on the requirement of the study. Nine panelists were selected and trained in several sensory sessions, 1–2 h per session, for at least 24 hours in full training. Such training is important to minimize any evaluation bias from subjective perception. The training involved performance analysis (the ability to discriminate coffee samples, reproducibility, and consistency). It was followed by a series of focus group discussions (FGD), briefing and introduction to the coffee aroma, references, samples, and the method used. Sensory descriptive analysis [23] was introduced, focusing on the description of coffee defect aroma (vocabulary) generated during FGD.

*2) Formal Evaluation:* Coffee brew samples (25 mL) were served in 50 mL paper cups covered with watch glasses. Panelists were required to wear red glasses to mask the color of the coffee brew during the assessment, as is practiced by the Specialty Coffee Association. This method was done to

reduce the influence of color on aroma perception. Unstructured 0–15-line scales with 0.5 increments were used to score selected aroma attributes of the samples. Analysis was performed in triplicates. Sensory evaluation design and data collection were performed using RedJade Sensory Software (Martinez, CA, USA).

### *D. Infrared Spectroscopy Analysis*

For Fourier-transform infrared spectroscopy (FTIR) analysis, 20 mg of KBr was added to 40 mg of coffee ground and pressed into a pellet. Pellet was placed in a sample plate for measurement using FT/IR-6800 type A with TGS detector. Spectra were recorded in the frequency of 4000-400 $cm^{-1}$ at 4 $cm^{-1}$ resolution, 16 gain, and 2 mm/s scanning speed. The IR spectra are presented as curves between % transmittance (Y-axis) and wavenumber (X-axis).

### *E. Volatile Compounds Analysis*

Aroma analysis was performed using Headspace Solid Phase Micro Extraction (HS-SPME) coupled with Gas Chromatography-Mass Spectrometry (GC-MS) [24]. Samples were analyzed in triplicates.

*1) Sampling and Extraction:* Approximately 5.0 g of coffee ground was placed into a 22 mL SPME vial, added with 0.2 ul of 0.01% ISTD (ISTD: 2,4,6-Trimethyl pyridine) analyzed within 30 min of preparation. HS-SPME method was optimized for the sampling method and amount of sample, fiber length, incubation time and temperature, and desorption time and temperature. The SPME fiber used was a 2 cm length, gray, 50/30 µm DVB/Car/PDMS (Supelco Inc., Bellefonte, PA). Equilibrium and extraction were set at 60°C, 20 min in Memmert waterbath. After extraction, the SPME fiber was moved to the split less injection port of the GC for manual injection. The PTV injection port for SPME sampling was equipped with a 0.75 mm ID borosilicate glass SPME inlet liner (Sigma-Aldrich Co., LLC).

*2) GC-MS:* GC-MS analysis was performed using a GC Agilent 7890A coupled with an MS Agilent 5975C, Inert XL, EI/CI MSD. An Agilent J&W DB-WAX column (30 m x 250 µm x 0.25 µm, Agilent Technologies Inc., California, USA) was used with a split less mode and an inlet temperature of 250°C. The initial temperature was set at 40°C, held for 1 min, then ramped at 4°C/min to 150°C for 0 min, then 30°C/min to 230°C for 5 min. The transfer line temperature was set at 250°C. The gas carrier used was helium (ultra-high purity), set at a flow rate of 1 mL/min, the average velocity of 36.44 cm/s, and a pressure of 6.96 psi. The setting for MS source was 230°C, max. at 250°C, MS Quad 150°C, max. at 200°C, with 29-550 amu scan range (MS, 70 eV). Data were collected from Enhanced ChemStation software MSD ChemStation G1701EA revision E.02.02. Identification was performed using NIST 14 MS database and linear retention indices (LRI) calculation based on n-alkane mixtures. Semi-quantification was calculated by internal standard (ISTD).

### *F. Statistical Data Analysis*

All data collected were tabulated in Microsoft Excel 2013. Descriptive statistics, analysis of variance (ANOVA), and post-hoc analysis (Fisher's least significant difference test) at 95% confidence interval were performed using Minitab 17 Statistical Software (Minitab Inc., State College, Pennsylvania, USA). Multivariate and principal component analyses (PCA) were performed using XLSTAT version 2015 (Addinsoft, New York, USA).

## III. Results and Discussion

### *A. Physical Profile of Robusta Coffee Brew*

The coffee samples of three roasting levels were visually different. The underdeveloped coffee beans were lightest in color and remained structurally intact as compared to the medium roast level. This result shows that the roasting time and temperature were not sufficient for heat transfer needed to promote an intense brewing reaction and to dehydrate and change the structure of the coffee beans. In contrast, the overdeveloped coffee had the darkest color, indicative of the presence of oil on the surface of the beans. Oil migration is a common phenomenon in roasted coffee beans where mobile lipids migrate from the endosperm to the surface of coffee beans [25].

The color, pH, and Brix (%) of the Robusta coffee brew–important parameters of coffee cup quality–are presented in Table I. Roasting level significantly changed the color of the coffee beans. As the degree of roasting increased, a significant decrease in all color parameters' values was observed. The increased roasting time temperature, thus the roasting level, created darker coffee beans and their corresponding brew. The color of the coffee brews differed, particularly in the L* and b* values, where the overdeveloped roasting produced brews with a darker color. Intense roasting reduced the L*, a*, and b* values of the coffee beans due to non-enzymatic browning and pyrolysis reactions such as Maillard browning during roasting [8], [10], [26].

TABLE I
PHYSICOCHEMICAL CHARACTERISTICS OF ROASTED ROBUSTA COFFEE BREW

| Parameter | Underdeveloped roasting | Medium roasting | Overdeveloped roasting |
|---|---|---|---|
| Lightness (L*) | 25.63 ± 0.06 $^{a}$ | 25.47 ± 0.25 $^{ab}$ | 25.27 ± 0.06 $^{b}$ |
| Redness (a*) | -1.50 ± 0.00 $^{a}$ | -1.60 ± 0.20 $^{a}$ | -1.70 ± 0.10 $^{a}$ |
| Yellowness (b*) | 4.60 ± 0.17 $^{a}$ | 4.20 ± 0.27 $^{ab}$ | 4.13 ± 0.15 $^{b}$ |
| pH | 5.27 ± 0.06 $^{b}$ | 5.30 ± 0.00 $^{b}$ | 5.47 ± 0.06 $^{a}$ |
| TDS (% Brix) | 1.40 ± 0.00 $^{b}$ | 1.43 ± 0.06 $^{b}$ | 1.50 ± 0.00 $^{a}$ |

Notes: Data have been presented as means ± standard deviation (n=3). The different notation indicates significant difference (Fisher LSD, α=0.05)

The coffee brew is typically associated with acidity (may be measured as pH as an alternative to total acids, which have more influence on coffee acidity) and the amount of dissolved solids (measured as % Brix). Based on the result, overdeveloped roasting produced significantly higher pH and % Brix values compared to the other two roasting levels. It is well established that roasting degrades reducing sugar, organic acids, and chlorogenic acids, while crude fat and caffeine content increases as roasting degree increases [27]. Typically, there is a formation of quinic acids (quinic acid and *syllo*-quinic acid) as the main degradation products of chlorogenic acids as the degree of roasting increases [10]. Thus, the higher roasting temperature may generate certain aliphatic acids, although other organic acids are degraded.

The increase in pH with increasing roasting level was also observed in light-to-dark coffee beans roasted at 205°C [9], which arises mainly due to the formation of aliphatic acids (formic, acetic, glycolic, and lactic acid) during roasting, a mechanism that is dependent on precursor compounds such as sucrose [28]. During roasting, citric acid, malic acid, and chlorogenic acids decrease while quinic acids increase due to the degradation of chlorogenic acids. Up to medium roasting degree, the yields of formic and acetic acids increase but then begin to fall as roasting continues. In contrast, glycolic and lactic acids yields continue to increase even at dark roasting [29].

The TDS (% Brix) of Robusta coffee brews significantly increased as the roasting level increased. A similar result was observed by Rao et al. [9]. During the roasting process, decomposition occurs and leads to the loss of soluble solids, but pyrolysis occurs and leads to the formation of new compounds.

### *B. Aroma Attributes*

A total of 46 aroma vocabulary for coffee were obtained from FGD. These aroma attributes were sorted, and six dominant attributes were identified as defects in roasted Robusta coffee (Table II) and used in the formal evaluation of the coffee brews. This selection was based on the aroma identified as a defect from consensus among trained panelists while also considering a reasonable number of attributes that will not overwhelm the objectivity of the panelist evaluation.

TABLE II
AROMA ATTRIBUTES OF ROASTED ROBUSTA COFFEE

| Attribute | Definition | Intensity | Reference |
|---|---|---|---|
| Raw nut-like | A light brown, slightly sweet, and musty aromatic commonly associated with raw peanuts. | 6.89 | 2.5 g mashed raw peanut mixed with 1 mL water in a 25 mL covered cup. |
| Grainy | A light brown, musty, dusty, aromatic associated with refined rice seeds and corn seeds. | 9.39 | 2.5 g raw Basmati rice in a 25 mL covered cup. |
| Grassy | A light green aromatic associated with freshly cut green grass. | 7.72 | 0.5 g freshly cut green grass in a 25 mL covered cup. |
| Hay-like | A dark brown, dry aromatic associated with dry rice straw. | 9.00 | 0.3 g dry cut rice straw in a 25 mL covered cup. |
| Ashy | A bitter, dusty, phenolic aromatic associated with cigarettes ashes. | 7.33 | 0.3 g cigarettes ashes in a 25 mL covered cup. |
| Carbony | A bitter, burnt aromatic associated with black charcoal. | 1.78 | 0.5 g black charcoal mixed with 1 mL water in a 25 mL covered cup. |

### *C. Sensory Evaluation*

Nine trained panelists scored the three roasted Robusta coffee samples for raw nut-like, grainy, grassy, hay-like, ashy, and carbony attributes. Based on the result (Fig. 2), it can be seen that the three samples showed different aroma profiles. The underdeveloped roasted coffee brew was dominated by high intensity of raw nut-like and grainy aroma, whereas overdeveloped roasted coffee was dominated by high intensity of carbony and ashy aroma.

It is well established that differences in coffee species, post-harvest processing, preparation, and evaluation method can yield different results. However, in general, it can be observed that, to some extent, the roast defect aroma profile of the Robusta coffee being studied here is similar to that of Arabica coffee previously reported by Giacalone et al. [15]. In their study, dark roasting produced certain characters such as strong, sharp, burnt, and tobacco-like aroma, while the underdeveloped coffee has a hay/straw-like aroma and appears to have a less intense character [15]. Medium roasting, as expected, had a balanced aroma intensity, and normal roasting had a 'clean cup' profile with an average score of aroma intensity and an absence of defects [15]. Because of the limitation of the current study, having only six selected aroma attributes and the different methods used, a direct comparison could not be made.

As there was no or shorter development time in the underdeveloped roasting, the coffee aroma and flavor did not fully develop, resulting in raw-like coffee bean character, as indicated by the nutty, grainy, and grassy notes. In addition, the low roast intensity is associated with higher levels of organic acids (acetic acid, butanoic acid, hexanoic acid).

Prolonged roasting time and development time extension in the overdeveloped roasting produced coffee with a 'darker' character with identified defects such as carbony and ashy aroma (Fig. 2). Dark roasting is known to increase flavor compound concentration, yielding an increase in the intensity of bitterness and astringency and burnt, licorice, and tobacco aroma [15].

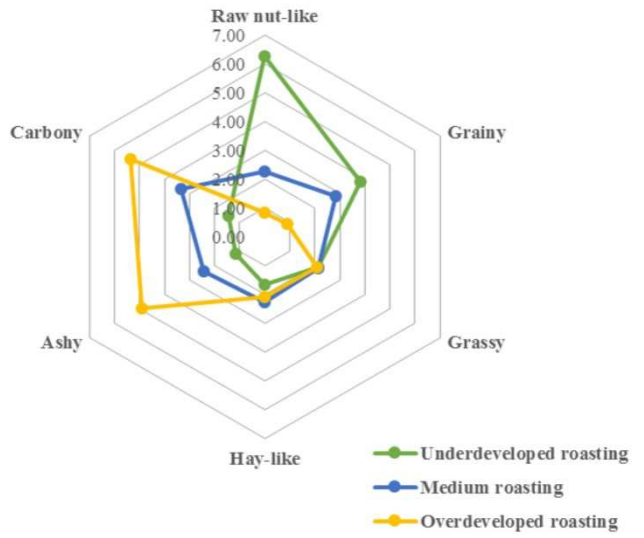


Fig. 2 Mean intensity score for six aroma attributes of roasted Robusta coffee

The two common roast defects and the medium roast had different aroma profiles. Sensory analysis shows that trained panelists could distinguish the different aroma profiles of the samples clearly and that the six major attributes can be used for aroma differentiation. However, we note that the use of red glasses to substitute red light might not be the best method to compensate for the influence of coffee brew color on aroma perception and expectation. The influence of color on sensory perception and experience is recognized and may be influenced, for example, by the color of the cups being used

for serving the beverage [30], [31]. Color differences can be linked to consumer's perception of certain sensory attributes [32]. Although the use of red glasses in this study shows the potential to minimize the perceptual bias of panelists, further studies will be needed to understand its effect. This can be done, for example, by comparing the effect of using red sunglasses vs red lighting on coffee brew perception to evaluate its effectiveness in disguising coffee brew color. Such studies could enable the standardization of glasses used as a red-light substitute for sensory evaluation.

### *D. Infrared Spectroscopy*

The infrared spectra of roasted Robusta coffee are shown in Fig 3. In general, we can see that the spectra of the three samples roasted at different levels are relatively similar, with negligible spectral differences.

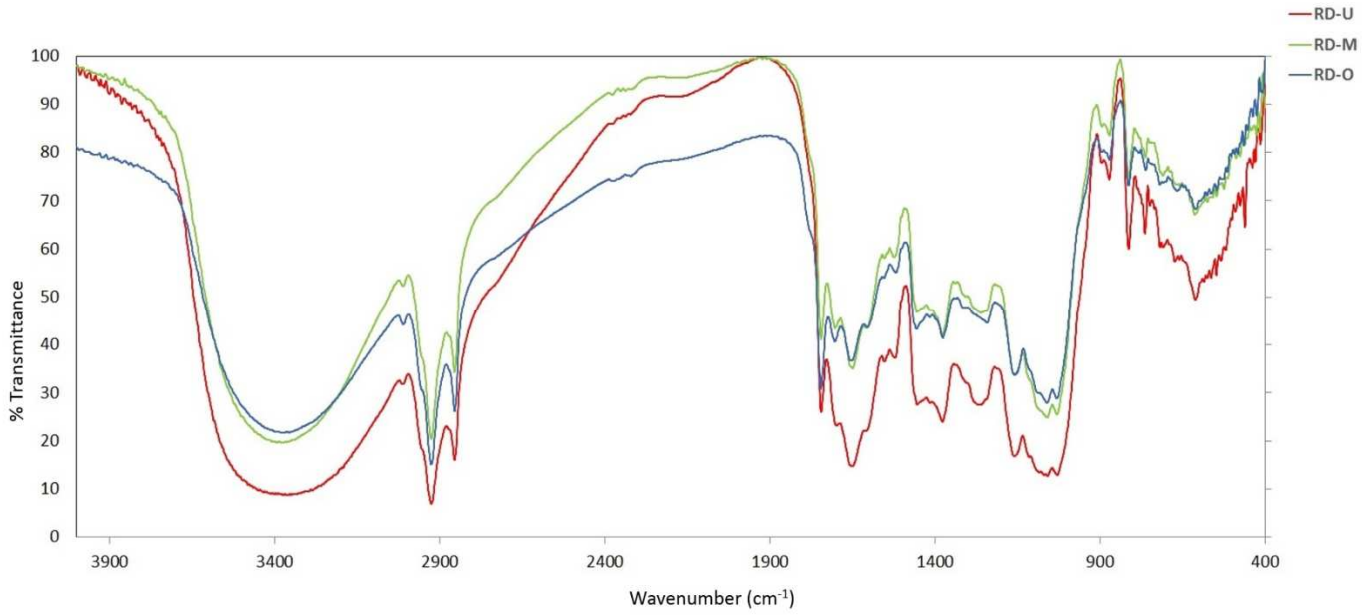


Fig. 3 The infrared spectrum of roasted coffee beans, RD-U (underdeveloped), RD-M (medium), RD-O (overdeveloped)

Functional group absorption of roasted Robusta coffee shows a peak width around 3200-3500 $cm^{-1}$, indicating the presence of hydroxide groups of alcohol compounds as well as water content in the sample. This wide peak may mask the absorption of primary and secondary amine groups, which usually occurs at 3200-3300 $cm^{-1}$. The two sharp absorptions around 2800-3000 $cm^{-1}$ indicate a significant stretch of alkyl group (C-H). The sharp peak around 1600-1700 $cm^{-1}$ is a typical stretch of carbonyl group (C=O), which can arise from aldehyde compounds, ketones, carboxylic acids, or esters. The peak width around 1400-1600 $cm^{-1}$ is an overlapping area of several functional groups such as alkenes (C=C), aromatic rings (C-C), asymmetric nitro (N-O), and bending of tertiary amine groups. The very sharp absorption around 1600 $cm^{-1}$ is an indication of a nitro group. The peak width around 1000-1400 $cm^{-1}$ is an overlapping area of several functional groups such as C-O, symmetrical nitro (N-O), and aliphatic amines (C-N). Since some of the absorptions that appears around 1600 $cm^{-1}$ is very sharp and there is asymmetrical vibration of the nitro group, the absorption around the 1000-1400 cm-1 area likely indicates the presence of C-O groups and nitro (N-O) symmetry. The absorption around 700-800 $cm^{-1}$ is indicative of alkene vibration, thus the absorption around 1400-1600 $cm^{-1}$ is estimated to be an overlap between alkene and nitro groups. Overall, the FTIR data reveal no significant difference of functional group variation between underdeveloped, medium, and overdeveloped roasting defects. To identify the presence of trace compounds, we performed further chemical analysis.

### *E. Volatile Compounds*

Chemical analysis of the samples revealed the presence of 70 volatiles (Table III) with a concentration range in parts per billion (ppb). The volatile compounds belong to several chemical functional groups, including hydrocarbons, alcohols, aldehydes, ketones, carboxylic acids, esters, pyrazines, pyrroles, pyridines, sulfur-containing compounds, furans, furanone, and phenols. The major aroma compounds found in roasted Robusta coffee were pyrazines and furans. Thus, it is not surprising that Dampit Robusta coffee is characterized by a nutty, earthy, roasty, and malty aroma since the pyrazines and furans exhibit those aromas.

As shown in Table III, the difference in roasting level, i.e., underdeveloped, medium, and overdeveloped roasting, appears to yield a significant effect on 25 volatile compounds. These compounds are 1,3-diazine; pyridine, 3-ethyl-); furan, 2-[(methylthio)methyl]-); pyrrole; pyrazine, (1-methylethenyl)-); furan, 2,2'-methylenebis-; butyrolactone; benzenamine, 4-methoxy-); furan, 2-(2-furanylmethyl)-5-methyl-; 1-(6-methyl-2-pyrazinyl)-1-ethanone; 2,5-furandione, 3,4-dimethyl-; 2(3H)-furanone; methyl salicylate; 1,2-cyclopentanedione, 3-methyl-; 2-cyclopenten-1-one, 2-hydroxy-3-methyl-; 2-cyclopenten-1-one, 3-ethyl-2-hydroxy-; phenylethyl Alcohol; 2-thiophenemethano; furan, 2,2'-[oxybis(methylene)]bis-; phenol; 1H-pyrrole-2-carboxaldehyde; phenol, 4-ethyl-2-methoxy-; 2,5-dimethyl-4-hydroxy-3(2H)-furanone; nonanoic acid; and 4H-pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl-. Since the concentrations of the compounds extracted from the

headspace were low (in ppb) and some compounds were only present in traces, the semi-quantification was somewhat difficult. However, the semi-quantification revealed that some compounds, such as those formed through amine-sugar or other chemical reactions due to high temperature processing such as pyrrole, pyrazine, pyridine, furan, thiols or sulfur-containing compounds, increased in concentration.

PCA was further performed to explore volatiles data. The first three principal components (PCs) can explain 93.53% variation in the dataset (Fig. 4). The three samples with different roasting profiles can be discriminated as indicated by a clear sample grouping. PC1 (40.36%) and PC2 (39.20%) differentiated between the overdeveloped and underdeveloped samples while PC 3 (13.96%) differentiated the medium roasted sample.

TABLE III
VOLATILE COMPOUNDS IDENTIFIED IN ROBUSTA COFFEE ROASTED UNDER DIFFERENT ROASTING PROFILE (UNDERDEVELOPED, MEDIUM, AND OVERDEVELOPED)

| Compounds | CAS No. | RT | LRI | Underdeveloped | | Medium | | Overdeveloped | | Sig.* |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Mean (ppb) | CV (%) | Mean (ppb) | CV (%) | Mean (ppb) | CV (%) | |
| 2,3-Butanedione | 000431-03-8 | 3.48 | 984 | 0.00 | 0.00 | 0.09 | 72.33 | 0.00 | 0.00 | N |
| Pyridine, 1,2,3,6-tetrahydro-1-methyl- | 000694-55-3 | 4.52 | 1042 | 0.00 | 0.00 | 0.00 | 0.00 | 0.63 | 124.46 | N |
| 2,3-Pentanedione | 000600-14-6 | 4.87 | 1060 | 0.80 | 59.48 | 0.59 | 83.05 | 0.45 | 81.70 | N |
| Cyclopentasiloxane, decamethyl- | 000541-02-6 | 7.66 | 1174 | 1.43 | 68.11 | 0.93 | 31.01 | 0.95 | 63.47 | N |
| Pyridine | 000110-86-1 | 7.92 | 1184 | 1.23 | 63.89 | 3.17 | 9.06 | 6.94 | 65.66 | N |
| 1,3-Diazine | 000289-95-2 | 8.68 | 1210 | 0.00 | 0.00 | 0.00 | 0.00 | 1.18 | 67.52 | Y |
| Pyrazine, methyl- | 000109-08-0 | 10.33 | 1262 | 7.56 | 68.10 | 7.41 | 10.70 | 1.18 | 67.52 | N |
| 2-Propanone, 1-hydroxy- | 000116-09-6 | 11.29 | 1291 | 2.22 | 72.17 | 1.52 | 25.34 | 8.36 | 67.01 | N |
| Pyrazine, 2,5-dimethyl- | 000123-32-0 | 12.09 | 1317 | 6.02 | 70.43 | 4.29 | 19.00 | 3.87 | 69.31 | N |
| Pyrazine, 2,6-dimethyl- | 000108-50-9 | 12.29 | 1323 | 5.62 | 70.98 | 4.75 | 18.83 | 4.74 | 68.94 | N |
| Pyrazine, ethyl- | 013925-00-3 | 12.43 | 1327 | 2.88 | 70.65 | 2.48 | 17.61 | 2.62 | 69.20 | N |
| Pyrazine, 2,3-dimethyl- | 005910-89-4 | 12.81 | 1340 | 1.19 | 71.01 | 1.14 | 19.89 | 1.45 | 69.25 | N |
| Cyclohexasiloxane, dodecamethyl- | 000540-97-6 | 13.07 | 1348 | 1.99 | 66.80 | 1.78 | 36.35 | 1.92 | 69.80 | N |
| 1-Hydroxy-2-butanone | 005077-67-8 | 13.58 | 1364 | 0.19 | 93.58 | 0.00 | 0.00 | 0.00 | 0.00 | N |
| Pyridine, 3-ethyl- | 000536-78-7 | 13.94 | 1375 | 0.00 | 0.00 | 0.00 | 0.00 | 0.94 | 67.92 | Y |
| Pyrazine, 2-ethyl-6-methyl- | 013925-03-6 | 14.07 | 1379 | 4.65 | 73.17 | 3.85 | 25.70 | 3.53 | 68.15 | N |
| Pyrazine, 2-ethyl-5-methyl- | 013360-64-0 | 14.25 | 1385 | 3.68 | 72.80 | 2.60 | 23.03 | 2.47 | 67.19 | N |
| Pyrazine, trimethyl- | 014667-55-1 | 14.69 | 1399 | 4.14 | 97.42 | 0.00 | 0.00 | 0.00 | 0.00 | N |
| 2-Furfurylthiol | 000098-02-2 | 15.46 | 1424 | 0.00 | 0.00 | 0.22 | 30.92 | 0.33 | 68.61 | N |
| Acetic acid | 000064-19-7 | 15.9 | 1438 | 3.77 | 73.11 | 0.00 | 0.00 | 0.00 | 0.00 | N |
| Pyrazine, 3-ethyl-2,5-dimethyl- | 013360-65-1 | 16.01 | 1442 | 6.74 | 73.17 | 5.87 | 16.28 | 6.24 | 63.24 | N |
| Furfural | 000098-01-1 | 16.37 | 1453 | 5.34 | 67.98 | 4.49 | 19.99 | 2.29 | 65.00 | N |
| Pyrazine, 2,6-diethyl- | 013067-27-1 | 16.46 | 1456 | 0.91 | 71.95 | 0.00 | 0.00 | 0.00 | 0.00 | N |
| Pyrazine, 2-ethyl-3,5-dimethyl- | 013925-07-0 | 16.45 | 1456 | 1.00 | 98.38 | 0.00 | 0.00 | 0.77 | 87.19 | N |
| Furan, 2-[(methylthio)methyl]- | 001438-91-1 | 17.07 | 1476 | 0.00 | 0.00 | 0.27 | 20.43 | 0.96 | 68.64 | Y |
| Pyrazine, 2-ethenyl-6-methyl- | 013925-09-2 | 17.19 | 1480 | 0.45 | 70.40 | 0.39 | 22.63 | 0.44 | 64.83 | N |
| Ethanone, 1-(2-furanyl)- | 001192-62-7 | 17.61 | 1494 | 1.32 | 70.86 | 2.13 | 19.95 | 2.78 | 67.51 | N |
| Pyrrole | 000109-97-7 | 17.98 | 1506 | 0.38 | 44.82 | 0.31 | 8.89 | 0.00 | 0.00 | Y |
| 2-Furanmethanol, acetate | 000623-17-6 | 18.73 | 1531 | 2.00 | 56.34 | 8.10 | 21.17 | 14.74 | 67.03 | N |
| 2-Furancarboxaldehyde, 5-methyl- | 000620-02-0 | 19.7 | 1563 | 4.10 | 72.32 | 7.27 | 21.77 | 4.77 | 69.25 | N |
| Pyrazine, (1-methylethenyl)- | 038713-41-6 | 20.36 | 1585 | 0.00 | 0.00 | 0.50 | 24.52 | 0.53 | 62.96 | Y |
| Ethenone, 1-(2-pyridinyl)- | 001122-62-9 | 20.47 | 1589 | 0.32 | 94.91 | 0.00 | 0.00 | 0.00 | 0.00 | N |
| 1H-Pyrrole-2-carboxaldehyde, 1-ethyl- | 002167-14-8 | 20.65 | 1595 | 0.25 | 78.96 | 0.00 | 0.00 | 0.00 | 0.00 | N |
| Furan, 2,2'-methylenebis- | 001197-40-6 | 20.79 | 1600 | 0.00 | 0.00 | 0.84 | 21.95 | 2.40 | 64.76 | Y |
| 1H-Pyrrole-2-carboxaldehyde, 1-methyl- | 001192-58-1 | 21.02 | 1608 | 0.00 | 0.00 | 1.37 | 23.10 | 1.60 | 71.68 | N |
| Butyrolactone | 000096-48-0 | 21.22 | 1615 | 0.00 | 0.00 | 0.00 | 0.00 | 5.48 | 64.33 | Y |
| Benzenamine, 4-methoxy- | 000104-94-9 | 22.04 | 1643 | 0.00 | 0.00 | 0.93 | 23.66 | 0.00 | 0.00 | Y |
| 2-Furanmethanol | 000098-00-0 | 22.5 | 1660 | 12.66 | 74.11 | 18.15 | 25.92 | 24.31 | 64.60 | N |
| 1-(5-Methyl-2-pyrazinyl)-1-ethanone | 022047-27-4 | 22.83 | 1671 | 0.67 | 95.87 | 0.00 | 0.00 | 0.00 | 0.00 | N |
| Furan, 2-(2-furanylmethyl)-5-methyl- | 013678-51-8 | 22.84 | 1671 | 0.00 | 0.00 | 0.00 | 0.00 | 2.77 | 61.48 | Y |
| 1-(6-Methyl-2-pyrazinyl)-1-ethanone | 022047-26-3 | 23.15 | 1682 | 1.42 | 61.48 | 0.00 | 0.00 | 0.00 | 0.00 | Y |
| Pyrazinamide | 000098-96-4 | 23.82 | 1706 | 1.32 | 104.91 | 0.00 | 0.00 | 0.00 | 0.00 | N |
| 2,5-Furandione, 3,4-dimethyl- | 000766-39-2 | 24.03 | 1714 | 0.00 | 0.00 | 0.58 | 23.09 | 0.00 | 0.00 | Y |
| 2(3H)-Furanone | 020825-71-2 | 24.66 | 1737 | 0.22 | 48.13 | 0.00 | 0.00 | 0.00 | 0.00 | Y |
| Methyl salicylate | 000119-36-8 | 25.25 | 1758 | 0.34 | 69.82 | 0.00 | 0.00 | 0.00 | 0.00 | Y |
| 2-Butenoic acid, 3-methyl- | 000541-47-9 | 26.1 | 1789 | 1.04 | 70.61 | 1.04 | 14.20 | 0.00 | 0.00 | N |

| | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| Benzoic acid, 2-hydroxy-, ethyl ester | 000118-61-6 | 26.22 | 1794 | 0.10 | 72.99 | 0.00 | 0.00 | 0.00 | 0.00 | N |
| Pyridine, 1-acetyl-1,2,3,4-tetrahydro- | 019615-27-1 | 26.38 | 1800 | 0.16 | 72.50 | 0.00 | 0.00 | 0.00 | 0.00 | N |
| 1H-Pyrrole, 1-(2-furanylmethyl)- | 001438-94-4 | 26.82 | 1817 | 0.74 | 61.03 | 0.83 | 38.61 | 1.35 | 58.90 | N |
| 1,2-Cyclopentanedione, 3-methyl- | 000765-70-8 | 26.92 | 1821 | 0.00 | 0.00 | 0.77 | 25.26 | 0.00 | 0.00 | Y |
| Hexanoic acid | 000142-62-1 | 27.48 | 1843 | 0.17 | 70.92 | 0.00 | 0.00 | 0.00 | 0.00 | N |
| Phenol, 2-methoxy- | 000090-05-1 | 27.68 | 1850 | 0.68 | 63.88 | 1.41 | 24.77 | 4.53 | 59.98 | N |
| 2-Cyclopenten-1-one, 2-hydroxy-3-methyl- | 021835-01-8 | 28.65 | 1889 | 0.00 | 0.00 | 0.00 | 0.00 | 1.24 | 59.70 | Y |
| 2-Cyclopenten-1-one, 3-ethyl-2-hydroxy- | 021835-01-8 | 28.65 | 1889 | 0.00 | 0.00 | 0.33 | 25.20 | 0.69 | 56.28 | N |
| Phenylethyl Alcohol | 000060-12-8 | 29 | 1905 | 0.00 | 0.00 | 0.36 | 26.45 | 0.00 | 0.00 | Y |
| Benzeneacetaldehyde, alpha. -ethylidene- | 004411-89-6 | 29.21 | 1921 | 0.18 | 60.56 | 0.15 | 24.75 | 0.00 | 0.00 | N |
| 2-Thiophenemethanol | 000636-72-6 | 29.48 | 1943 | 0.00 | 0.00 | 0.17 | 36.49 | 0.07 | 79.18 | Y |
| Maltol | 000118-71-8 | 29.73 | 1963 | 0.52 | 95.56 | 0.91 | 34.85 | 1.44 | 53.33 | N |
| Ethanone, 1-(1H-pyrrol-2-yl)- | 001072-83-9 | 29.81 | 1968 | 0.87 | 52.53 | 1.13 | 25.08 | 1.60 | 57.82 | N |
| Furan, 2,2'-[oxybis(methylene)] bis- | 004437-22-3 | 29.94 | 1979 | 0.00 | 0.00 | 0.28 | 24.24 | 0.88 | 54.44 | Y |
| Phenol | 000108-95-2 | 30.16 | 1996 | 0.00 | 0.00 | 0.58 | 19.29 | 1.30 | 55.07 | Y |
| 1H-Pyrrole-2-carboxaldehyde | 001003-29-8 | 30.32 | 2028 | 0.00 | 0.00 | 0.67 | 25.59 | 0.52 | 55.48 | Y |
| Phenol, 4-ethyl-2-methoxy- | 002785-89-9 | 30.38 | 2041 | 0.28 | 98.93 | 0.71 | 35.00 | 2.84 | 53.65 | Y |
| 2,5-Dimethyl-4-hydroxy-3(2H)-furanone | 003658-77-3 | 30.45 | 2058 | 0.88 | 52.92 | 0.71 | 24.11 | 0.00 | 0.00 | Y |
| Octanoic Acid | 000124-07-2 | 30.64 | 2102 | 0.50 | 89.48 | 0.00 | 0.00 | 0.00 | 0.00 | N |
| Nonanoic acid | 000112-05-0 | 31.34 | 2189 | 0.44 | 66.77 | 0.00 | 0.00 | 0.00 | 0.00 | Y |
| 2-Methoxy-4-vinylphenol | 007786-61-0 | 31.52 | 2211 | 3.79 | 70.52 | 3.27 | 23.99 | 3.35 | 52.53 | N |
| 4H-Pyran-4-one, 2,3-dihydro-3,5-dihydroxy-6-methyl- | 028564-83-2 | 32.01 | 2270 | 0.57 | 66.03 | 0.00 | 0.00 | 0.00 | 0.00 | Y |
| 3-Pyridinol | 000109-00-2 | 32.98 | 2432 | 0.00 | 0.00 | 0.00 | 0.00 | 0.07 | 71.15 | N |
| Indole | 000120-72-9 | 33.21 | 2465 | 0.12 | 69.03 | 0.06 | 35.51 | 0.08 | 63.97 | N |

Data are presented as means across all samples: underdeveloped, medium, and overdeveloped roasted coffee (n=3); CAS No. refers to Chemical Abstract Service number; RT refers to retention time; LRI refers to Linear Retention Indices; CV refers to correlation coefficient; Mean concentrations are in parts per billion (ppb); *Significant (Y=yes , at α=0.05).

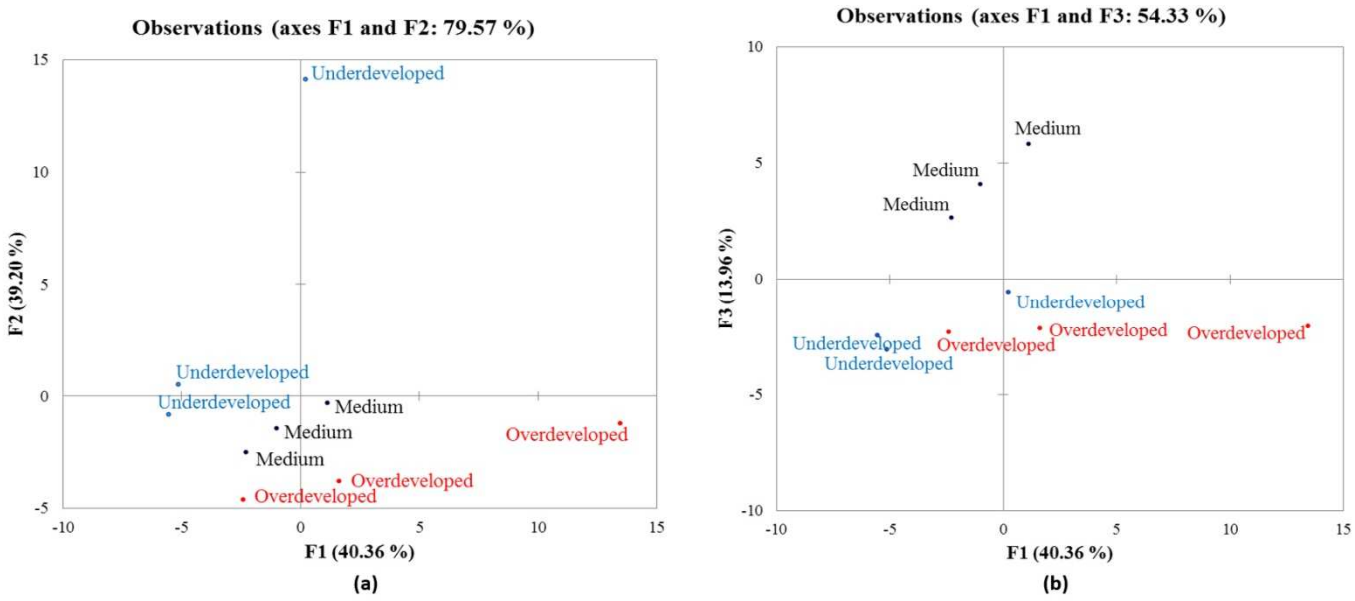


Fig. 4 PCA plot, PC 1 vs PC2 (a), PC1 vs PC3 (b)

The overdeveloped roast defect can be characterized by pyridine, furans, phenol, and pyrrole derivatives. This roast defect created coffee with carbony and ashy characteristics, presumably due to the phenol and polyphenol derivatives produced such as phenol, 2-methoxy-; phenol, 4-ethyl-2-methoxyl; and 2-methoxy-4-vinylphenol that contributes to harsh and smoky characteristics of roasted coffee. On the other hand, the underdeveloped coffee can be characterized by the presence of acids (acetic, hexanoic, octanoic and nonanoic) and certain pyrazines such as pyrazine, 2,6-diethyl; and pyrazine, trimethyl- that may contribute to the raw nut-like aroma. Of note, 2-furfurylthiol was found in the medium roasted coffee and its concentration increased in the overdeveloped roasted coffee.

The 2-furfurylthiol compound is a key compound in coffee aroma formed through roasting. The formation of thiol groups is related to roasting time and temperature [33]. Since the formation of thiols involves high temperature roasting, the concentration of 2-furfurylthiol compound increases along with the roasting level until a dark roast is achieved, before it decreases should the roasting continue [18]. The degradation of 2-furfurylthiol compound due to high temperature roasting has also been reported by Moon and Shibamoto [34]. In addition, high temperature roasting also yields more lactones such as butyrolactones. This compound has caramel-like, fatty, creamy, and oily aroma [35] as well as a butter/coconut-like flavor [34].

The medium roasted coffee can be discriminated due to the presence of furanones such as 2,5-dimethyl-4-hydroxy-3(2H)-furanone or strawberry furanone; ketones such as 2,3-butanedione and 2,3-pentanedione; phenylethyl alcohol; pyrazines; furfural and furan derivatives. The medium roasted coffee appears to be quite balanced with furanones that are responsible for certain sweet and caramel notes.

Roasting temperature influences the breakdown of volatile precursors through complex mechanisms, perhaps most importantly are the reactions that involve protein, carbohydrate, and polyphenol degradation. Physical and chemical changes that occur during coffee roasting are typically driven by Maillard reaction (the reaction between amino acids and reducing sugars), Strecker degradation (the reaction which converts α-amino acids into aldehydes), and thermal degradation [36]. Maillard reaction produces heterocyclic components that contribute to coffee flavor [37]. Heterocyclic nitrogen compounds such as pyridines and pyrroles originate from different degrees of breakdown during high temperature processing [38]. Thus, in general, higher time-temperature roasting will create these chemical groups.

The pyrazines and alkyl pyrazines identified in the roasted samples include pyrazine, 2,5-dimethyl-; pyrazine, 2,6-dimethyl-; pyrazine, ethyl; pyrazine, 2,3-dimethyl-; pyrazine, 2-ethyl-6-methyl-; pyrazine, 2-ethyl-5-methyl-; pyrazine, trimethyl-; pyrazine, 3-ethyl-2,5-dimethyl-; pyrazine, 2,6-diethyl-; pyrazine, 2-ethyl-3,5-dimethyl-; pyrazine, 2-ethenyl-6-methyl- and pyrazine, (1-methylethenyl)-. Pyrazines are commonly associated with heat processing that involves sugar-amine reaction leading to Maillard reaction and Strecker degradation of amino acids [26]. However, certain pyrazines decreased along with the increase of roasting level, suggesting thermal degradation as the roasting continues [18], [7]. These pyrazines might elicit a range of aroma such as coffee, nutty, musty, roasty, earthy, cocoa, and beef notes [39], [40], which suggests their contribution to the major nutty and raw character observed in our sensory study.

## IV. Conclusion

Based on the results of our study, the two common roasting defects, i.e., underdeveloped and overdeveloped, produced Robusta coffee with different sensory and physicochemical characteristics. The two common roasted coffee defects have different aroma profiles that expand from 'raw-like' character for the underdeveloped sample due to failure in aroma and flavor development to 'darker' character with strong carbony and ashy notes for the overdeveloped sample due to over roasting. The overdeveloped roast defect had higher pH and TDS values compared to the underdeveloped roast defect and the medium roast.

Evaluation of the volatile compounds shows clear grouping between the three roasted samples. Beside 2-furfurylthiol that increased along with roasting level, other volatile compounds belonging to pyridine, furan, phenol, and pyrrole derivatives were found to characterize the overdeveloped defect sample. The underdeveloped coffee can be characterized by a higher concentration of aliphatic acids and pyrazines contributing to raw nut-like aroma whereas the medium roasted coffee appears to be quite balanced. Our understanding of defects related to coffee production may aid in the control and improvement of coffee quality.

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