https://doi.org/10.17221/60/2024-CJFS

Effect of acetic acid immersion on the taste and aroma quality of immature Robusta coffee beans

Deden Fardenan[1,2], *Dian Angraini Suroto*[1], *Supriyadi Supriyadi*[1]*

[1]*Department of Food and Agricultural Product Technology, Faculty of Agricultural Technology, Universitas Gadjah Mada, Yogyakarta, Indonesia*
[2]*Standardization Agency for Agricultural Instruments, Ministry of Agriculture of the Republic of Indonesia, Jakarta, Indonesia*

**Corresponding author: suprif248@ugm.ac.id*

Citation: Fardenan D., Suroto D.A., Supriyadi S. (2024): Effect of acetic acid immersion on the taste and aroma quality of immature Robusta coffee beans. Czech J. Food Sci., 42: 391–404.

Abstract: The presence of immature coffee beans reduces the quality of the coffee brew. This study evaluated how the acetic acid affected the aroma and taste of immature compared to mature Robusta coffee. The naturally processed immature Robusta green beans were immersed in acetic acid (0–5%) for 30–90 min. The naturally processed mature Robusta green beans were used as a control treatment. The samples were roasted at a medium level (240 °C, 14 min). The sensory analysis was evaluated by the cupping test by a trained and certified panellist (Q-Grader). Acetic acid immersion significantly improved the sensory quality of immature beans ($P < 0.05$). Specifically, immersion in 3% acetic acid for 90 min yielded the highest sensory score of 84.92. According to the Coffee Quality Institute (CQI) classification, this score falls into the 'Fine' Robusta classification. The 0% 30-min, 3% 30-min, and 3% 90-min treated immature samples and an untreated control mature sample were analysed for pH, caffeine, chlorogenic acid, and volatile compounds. The treatment significantly decreased the chlorogenic acid ($P < 0.05$) and increased the volatile compound concentration of furans, ketone, pyrrole, aldehyde, and ester while decreasing pyrazines, sulphur, phenol, and alcohol. This study effectively enhanced immature Robusta taste and aroma quality to a degree comparable with mature Robusta coffee beans.

Keywords: cupping test; flavour; Fine Robusta coffee; pre-treatment; volatile compounds

Indonesia is the world's fourth largest coffee bean producer, primarily consisting of Robusta coffee (Slavova and Georgieva 2019). However, the taste and aroma quality of Indonesian Robusta coffee tend to be lower because many local coffee farmers in Indonesia often neglect the maturity level of coffee fruit during harvesting (Sunarharum et al. 2021). Coffee quality is impacted by various factors such as coffee variety, geographical conditions, harvesting methods, and post-harvest processing (Poltronieri and Ros-

Supported by the Standardization Agency for Agricultural Instruments, Ministry of Agriculture of the Republic of Indonesia. Commitment Making Official (CMO) No. 1297/Kpts/KP.320/H.1/9/2023.

https://doi.org/10.17221/60/2024-CJFS

si 2016; Halagarda and Obrok 2023). Robusta coffee is renowned for its bitterness, body, and robustness compared to Arabica (Caporaso et al. 2018). The quality of Robusta coffee will decline more significantly with the existence of immature coffee beans, resulting in an unpleasant flavour (Rabelo et al. 2021). Immature beans are coffee beans harvested before they reach their optimal maturity. Desirable coffee flavour characteristics include caramel-like, fruity, and naturally sweet taste (Kreuml et al. 2013). These characteristics are produced by the flavour precursors in beans during the roasting and brewing process (Velásquez and Banchón 2023). The concentration and composition of coffee flavour precursors, including proteins, sugars, lipids, and chlorogenic acids (CGAs), play a crucial role in shaping the complex flavours of coffee (Seninde and Chambers 2020; Velásquez et al. 2019). In contrast to immature beans, mature coffee beans yield a more pleasant aroma and flavour.

Roasted immature Robusta beans exhibit lower furan levels, fructose, glucose and higher pyrazine concentration compared to Arabica beans (Liu et al. 2019; Osorio Pérez et al. 2023). The furan compounds group contributes to the preferred sweet, caramel-like, and nutty aroma. Conversely, compounds in the N-heterocycles group contribute to the less preferred, including earthy, leather-like, burnt notes (Liu et al. 2019). Due to its elevated caffeine and chlorogenic acid content, Robusta coffee imparts an astringent and bitter taste that significantly influences aftertaste and overall beverage quality (Lang et al. 2020). In contrast to Arabica beans, Robusta beans contain approximately double the caffeine content, which makes them more bitter and more robust in taste (Olechno et al. 2021; Belgis et al. 2023).

The taste and aroma of coffee are related to the degradation of precursor composition due to reactions like the Maillard reaction, caramelisation, and pyrolysis, which are affected by roasting time and conditions, temperature, as well as the brewing process (Velásquez et al. 2019). The Maillard reaction is a chemical process in which reducing sugars react with amino acids. Next, caramelisation occurs when sucrose is heated, transforming it into simpler compounds. Lastly, pyrolysis is the thermal decomposition of organic compounds without oxygen, as observed in coffee roasting. The volatile compounds are classified as furans, pyrazines, pyrroles, pyridines, aldehydes, ketones, esters, alcohols, acids, phenols, and sulphur compounds that contribute to unique aroma characteristics, from sweet and fruity to earthy and spicy (Seninde and Chambers 2020). These flavour compounds determine the quality of the aroma and taste of coffee.

Numerous studies have investigated the methods to enhance the flavour profile of Robusta coffee, including steam pressure treatment, enzymatic processes, and acetic acid immersion (Kraehenbuehl et al. 2017; Kalschne et al. 2018; Liu et al. 2019). However, these studies have not specifically addressed the handling of immature Robusta coffee beans, which exhibit inferior aroma and taste characteristics compared to fully mature beans. This research aimed to enhance the sensory attributes of immature Robusta beans to resemble those of mature Robusta beans by immersing green immature beans in an aqueous acetic acid solution.

MATERIAL AND METHODS

Material. Mature and immature Robusta coffee beans were obtained from a local farmer in West Lampung Regency, Indonesia. The coffee cherries were handpicked and sorted based on red (mature) and green (immature) colour. The coffee cherries were dry-processed and dried until the moisture content reached 11%. Subsequently, the outer skin was removed, yielding green coffee bean samples. The samples were stored at a cool temperature (4 °C). The chemicals used were glacial acetic acid (100063), caffeine (C0750) and chlorogenic acid (C3878) standards purchased from Merck (Germany).

Acetic acid treatment. The green bean samples (25 g) were immersed in 100 mL (ratio 1 : 4) of an acetic acid solution of different concentrations (0, 3, and 5%) for 30, 60, and 90 min. The 0% acid-treated sample was treated with water only. The untreated mature green beans were used as the control sample. The treatments were done in triplicate. Following the immersion period, the acetic acid solution was discarded. The samples were transferred to a container, rinsed with distilled water while stirring, and drained. Subsequently, the samples were dried in a cabinet dryer (50 °C, 6 h) until the moisture content reached 11%. The dried samples were then stored in a stand up coffee pouch within a freezer at –17 °C until the pH analysis of both green and roasted beans could be conducted.

Roasting and milling process. The samples were roasted in a coffee roaster (Gene Café CBR-101; Gene Café, Korea) to medium roast at 240 °C for 14 min (Agtron Scale 55). The Agtron Scale measures coffee bean roast levels using a spectrophotometer to analyse their colour, giving a numerical value from 0 to 100 – lower for darker roasts, higher for lighter

https://doi.org/10.17221/60/2024-CJFS

ones. The green and roasted beans were ground using a coffee grinder (Latina N600; Yang-Chia Machine Works, Taiwan), fineness setting number 1, and sifted through a 20-mesh sieve. All the samples were stored in a stand up coffee pouch in a freezer at –17 °C until further analysis.

Sensory evaluation. Sensory evaluation was conducted at the Indonesian Coffee and Cocoa Research Institute (ICCRI) in Jember, Indonesia. The assessment employed the cupping test method and three expert panellists, Q-Graders, were engaged. These professionals are skilled coffee tasters and graders who have mastered the ability to comprehensively and objectively evaluate coffee based on its physical and sensory attributes. The sample (8.25 g) was brewed with 150 mL of hot water (93 °C) and left for 4 min. The coffee was ready for assessment after the temperature reached 70 °C to 73 °C. The scores and tasting notes were written in the cupping form. The hedonic scale scores were between 6.00 (good) and 9.00 (excellent) to assess aroma, flavour, aftertaste, acidity, sweetness, body, balance, overall experience, uniformity, and cleanness (Hetzel 2015). The final score was the sum of the total scores of all the parameters assessed. The 0% 30-min, 3% 30-min, and 3% 90-min treated immature samples and the control mature sample were analysed for pH, caffeine, chlorogenic acid, and volatile compounds.

pH measurement. Green and roasted bean powder (2 g) was boiled with 15 mL of distilled water. Whatman® filter paper No. 4 was used to filter the extract. The pH meter (Mettler Toledo F20; Mettler-Toledo, Switzerland) was used to measure pH.

Colour measurement. Green and roasted bean powder was ground. A chromameter CR-400 (Konica Minolta, USA) was used to perform colour analysis in triplicate.

Caffeine and chlorogenic acid measurement. Roasted bean powder (2 g) was boiled with 100 mL of distilled water for 5 min while stirred and then chilled for a few minutes. The brewed coffee was filtered using 0.45 µm nylon syringe filters. The filtrate was analysed with an LC20-AD high-performance liquid analyser (HPLC; Shimadzu Corporation, Japan). The caffeine standard powder (0.01 g) was dissolved in 100 mL of distilled water, and serial dilutions of standards (2.5, 5, 10, 50, and 100 ppm) were prepared. The method for preparing the standard chlorogenic acid solution followed the same procedure as for caffeine, using methanol as the solvent. The conditions of the HPLC were as follows: Shim Pack GIST C18 column, 150 × 4.6 mm; column temperature was 80 °C; PDA detector, wavelength set at 190–800 nm; the pressure, 150 kgf·cm^{-2}; mobile phase was water (799 mL) : acetic acid (1 mL) : methanol (200 mL), the flow rate was 1 mL·min^{-1} for 25 min. The volume of the injected samples and standards was 20 µL. The standard caffeine and chlorogenic acid solutions were analysed under the same conditions as the samples. Each standard solution was used to create an individual standard curve. The caffeine and chlorogenic acid levels of the samples were determined by comparing the area under the caffeine and chlorogenic acid standard curves with the area under the curve obtained from the samples.

Volatile aroma measurement. A 22 mL glass vial was filled with the 3.5 g sample of ground coffee, and the activated solid phase micro extraction (SPME) needle was inserted and incubated (45 min, 85 °C). The SPME needle was 2 cm Divinylbenzene/Carboxen/Polydimethylsiloxane (DVB/CAR/PDMS) SPME fibre assembly (Supelco; Sigma Aldrich, UK). The internal standard used was 2,4,6-trimethyl pyridine 0.2 µL 0.001%. GC-MS (GC 7890A; Agilent, USA) paired with a DB-Wax column (30 cm × 0.25 mm × 0.25 µm) was used to analyse the volatile chemical compounds. The injector temperature was 250 °C in split mode (25 : 1). Carrier gas: helium, 1 mL·min^{-1}. The conditions of the operation: the initial temperature was 40 °C for 5 min, increased by 3 °C per min to 180 °C, then 8 °C per min to 240 °C, and held for 7 min. Volatile compounds were found using the full scan mode (mass range from m/z 29 amu to 550 amu). They were identified by comparing their mass spectra and linear retention indicators (LRI) under the experimental conditions with reference libraries (NIST14; National Institute of Standards and Technology, USA).

Statistical analysis. This study was carried out using a completely randomised design involving two factors: concentrations of acetic acid (0, 3, and 5%) and immersion time (30, 60, and 90 min), and it was done in triplicate. SPSS (version 26) and Microsoft Excel (version 2021) software were used for statistical analysis. The mean value and standard deviation (SD) are given. Two-way analysis of variance (ANOVA) was used to test the sensory analysis to see the effect between treated samples (Table 1). Student's t-test was used to compare the control mature coffee beans with the best-treated sample, with the highest sensory score (Table 2) and volatile compound analysis (Table 3). One-way ANOVA was used to test other analyses. The Duncan multiple range test (DMRT) determined a significant difference at 5%.

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Table 1. Sensory evaluation of immature Robusta bean immersion in an acetic acid solution

Treatments	Parameters										
	aroma	taste	aftertaste	acidity	sweetness	body	balance	overall	uniformity	cleanness	final score
0% 30-min	6.67 ± 0.14^{aA}	6.83 ± 0.14^{aA}	6.58 ± 0.14^{aA}	7.42 ± 0.14^{aA}	7.58 ± 0.14^{bA}	7.58 ± 0.14^{bA}	6.83 ± 0.14^{aA}	6.67 ± 0.14^{aA}	10.00 ± 0.00^{aA}	10.00 ± 0.00^{aA}	76.17 ± 0.80^{aA}
0% 60-min	6.83 ± 0.14^{aB}	6.83 ± 0.14^{aB}	6.67 ± 0.14^{aA}	7.58 ± 0.14^{aA}	7.67 ± 0.14^{bA}	7.58 ± 0.14^{bA}	6.67 ± 0.14^{aA}	7.08 ± 0.14^{aB}	10.00 ± 0.00^{aA}	10.00 ± 0.00^{aA}	76.92 ± 0.63^{aB}
0% 90-min	6.50 ± 0.00^{aB}	6.75 ± 0.25^{aB}	6.75 ± 0.25^{aB}	7.50 ± 0.25^{aB}	7.67 ± 0.14^{bB}	7.58 ± 0.14^{bB}	6.83 ± 0.14^{aB}	6.50 ± 0.00^{aB}	10.00 ± 0.00^{aA}	10.00 ± 0.00^{aA}	76.08 ± 0.76^{aC}
3% 30-min	6.67 ± 0.14^{bA}	6.67 ± 0.14^{bA}	6.67 ± 0.14^{bA}	6.67 ± 0.14^{aA}	6.67 ± 0.14^{aA}	6.75 ± 0.00^{aA}	6.67 ± 0.14^{bA}	6.75 ± 0.00^{bA}	10.00 ± 0.00^{aA}	10.00 ± 0.00^{aA}	73.50 ± 0.50^{bA}
3% 60-min	7.58 ± 0.14^{bB}	6.67 ± 0.14^{bB}	7.42 ± 0.14^{bA}	7.33 ± 0.14^{aA}	7.17 ± 0.14^{aA}	7.42 ± 0.14^{aA}	7.25 ± 0.00^{bA}	7.33 ± 0.14^{bB}	10.00 ± 0.00^{aA}	10.00 ± 0.00^{aA}	79.00 ± 0.25^{bB}
3% 90-min	8.00 ± 0.00^{bB}	7.50 ± 0.00^{bB}	8.08 ± 0.14^{bB}	8.50 ± 0.00^{aB}	8.25 ± 0.25^{aB}	8.00 ± 0.00^{aB}	8.00 ± 0.00^{bB}	8.08 ± 0.14^{bB}	10.00 ± 0.00^{aA}	10.00 ± 0.00^{aA}	84.92 ± 0.29^{bC}
5% 30-min	8.08 ± 0.14^{cA}	8.00 ± 0.00^{cA}	8.08 ± 0.14^{cA}	8.00 ± 0.00^{bA}	7.67 ± 0.14^{bA}	8.00 ± 0.00^{bA}	8.00 ± 0.00^{cA}	8.00 ± 0.00^{cA}	10.00 ± 0.00^{aA}	10.00 ± 0.00^{aA}	83.75 ± 0.25^{cA}
5% 60-min	7.75 ± 0.00^{cB}	7.92 ± 0.14^{cB}	7.58 ± 0.14^{cA}	7.58 ± 0.14^{bA}	7.42 ± 0.14^{bA}	7.42 ± 0.14^{bA}	7.50 ± 0.00^{cA}	7.58 ± 0.14^{cB}	10.00 ± 0.00^{aA}	10.00 ± 0.00^{aA}	80.50 ± 0.25^{cB}
5% 90-min	7.67 ± 0.14^{cB}	7.67 ± 0.14^{cB}	7.75 ± 0.00^{cB}	8.25 ± 0.25^{bB}	7.92 ± 0.14^{bB}	7.67 ± 0.14^{bB}	7.75 ± 0.00^{cB}	7.67 ± 0.14^{cB}	10.00 ± 0.00^{aA}	10.00 ± 0.00^{aA}	82.33 ± 0.58^{cC}

$^{a–c}$ The difference designated with lowercase letters within the same column signifies concentration; $^{A–C}$ uppercase letters within the same column signify time; these different letters indicate statistically significant differences ($P < 0.05$); the data are presented as mean ± standard deviation (n = 3 panellists)

Table 2. Comparison of the sensory evaluation between the control mature coffee beans and the highest score of the treated sample

Treatments	Parameters										
	aroma	taste	aftertaste	acidity	sweetness	body	balance	overall	uniformity	cleanness	final score
Control mature	7.58 ± 0.14^{a}	7.67 ± 0.14^{a}	7.58 ± 0.14^{a}	7.42 ± 0.14^{a}	7.67 ± 0.14^{a}	7.83 ± 0.29^{a}	7.67 ± 0.14^{a}	7.67 ± 0.14^{a}	10.00 ± 0.00^{a}	10.00 ± 0.00^{a}	81.08 ± 0.95^{a}
3% 90-min	8.00 ± 0.00^{b}	7.50 ± 0.00^{b}	8.08 ± 0.14^{b}	8.50 ± 0.00^{b}	8.25 ± 0.25^{b}	8.00 ± 0.00^{a}	8.00 ± 0.00^{b}	8.08 ± 0.14^{b}	10.00 ± 0.00^{a}	10.00 ± 0.00^{a}	84.92 ± 0.29^{b}

$^{a, b}$ Different lowercase letters within the same column indicate statistically significant differences ($P < 0.05$); the data are presented as mean ± standard deviation (n = panellists)

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Table 3. The quantity of volatile compounds in the control mature and selected treated immature samples

Compounds	CAS	Concentrations (ppb)				Aroma notes*
		0% 30-min	3% 30-min	3% 90-min	control mature	
Furan						
5-methyl-furfural	620-02-0	16.51	7.97	10.45	17.92	spice, caramel, maple
Furfuryl-ether	4437-22-3	3.00	7.97	5.06	3.73	coffee, nutty, earthy
Furfuryl-methyl sulphide	1438-91-1	0.77	0.72	1.00	1.06	onion, sulphury, pungent, vegetable, horseradish
2-propionylfuran	3194-15-8	0.67	0.63	1.36	1.66	slight fruity
2-methyl furan	534-22-5	0.67	1.18	1.49	1.27	ethereal, acetone, chocolate
2-acetylfuran	1192-62-7	0.41	0.88	4.19	3.88	sweet, balsam, almond, cocoa, caramel, coffee
2-(methoxymethyl)-furan	13679-46-4	0.40	0.60	0.71	0.56	roasted coffee
2-furfurylfuran	1197-40-6	0.36	2.11	2.22	1.77	rich roasted
2-pentyl-furan	3777-69-3	0.21	0.16	0.28	0.34	fruity, green, earthy, beany, vegetable
2-butyl- furan	4466-24-4	0.18	0.61	0.70	0.40	mild, fruity, wine, sweet, spicy
Total		23.18	22.83	27.46^{a}	32.61^{b}	
Pyrazines						
3-ethyl-2,5-dimethyl pyrazine	13360-65-1	28.96	26.47	0.62	28.56	potato, cocoa roasted, nutty
2-methyl pyrazine	109-08-0	23.45	14.89	13.97	16.20	nutty, cocoa roasted, chocolate, peanut
2,5-dimethyl-pyrazine	123-32-0	13.96	7.62	7.53	10.28	cocoa, nuts roast, beef, woody, grass, medical
2-ethyl-6-methyl-pyrazine	13925-03-6	9.86	6.40	5.98	5.15	roasted potato
Ethyl-pyrazine	13925-00-3	7.38	4.87	4.85	5.19	butter, musty, nutty, woody, roasted cocoa, peanut
2-ethyl-5-methyl-pyrazine	13360-64-0	6.82	4.37	4.40	5.15	coffee bean, nutty, grassy, roasted
Trimethyl-2,3,5 pyrazine	14667-55-1	8.54	5.42	4.89	6.68	nutty, earthy, baked potato, roasted peanut, musty, cocoa
6-acetyl-2-methyl pyrazine	22047-26-3	4.35	3.49	3.85	5.91	roasted coffee, cocoa, popcorn
5-acetyl-2-methyl pyrazine	22047-27-4	2.78	2.71	3.17	3.84	popcorn
2,3-dimethyl-pyrazine	5910-89-4	2.12	2.44	2.40	3.18	nutty, cocoa, peanut, butter, coffee, walnut, caramel, roasted
2-ethenyl-5-methyl-pyrazine	13925-08-1	0.55	1.70	0.61	2.74	coffee
1-methyl ethenyl-pyrazine	38713-41-6	1.77	2.05	2.57	2.66	caramel-like, chocolate, nutty, roasted
Pyrazine	290-37-9	1.48	1.57	1.82	2.64	pungent, barley, sweet corn-like, roasted hazelnut
2,6-diethyl-pyrazine	13067-27-1	1.68	1.69	1.67	2.25	nutty, hazelnut
2-methyl-6-propyl-pyrazine	29444-46-0	1.11	0.70	0.41	0.72	burnt, hazelnut
2-(N-propyl)-pyrazine	18138-03-9	0.59	0.52	0.54	0.69	green vegetable, nutty
2-ethyl-3-methyl-pyrazine	15707-23-0	1.78	0.68	1.00	0.59	nutty, peanut, musty, corn raw bread
Total		117.19	87.60	60.26^{a}	102.43^{b}	

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Table 3. To be continued

Compounds	CAS	Concentrations (ppb)				Aroma notes*
		0% 30-min	3% 30-min	3% 90-min	control mature	
Sulphur						
Trimethyl-oxazole	20662-84-4	0.23	0.22	0.30	0.47	nutty, nut skin roasted, wasabi, mustard, vegetable
Total		0.23	0.22	0.30[a]	0.47[b]	
Phenol						
2-methoxy-4-vinyl phenol	7786-61-0	41.75	36.54	30.15	34.94	sweet, spicy, phenolic, clove carnation, peppery, woody, smoky, powdery
4-ethylguaiacol	2785-89-9	4.48	7.87	10.22	7.23	sweet, fruity
Phenol, 2-methoxy-	90-05-1	4.18	5.13	7.00	5.25	phenolic, smoke, spice, vanilla, woody
Phenol	108-95-2	3.60	4.94	6.39	4.37	phenolic, plastic rubber
Total		54.01	54.48	53.76[a]	51.79[b]	
Pyridine						
Pyridine	110-86-1	7.02	11.53	13.19	15.45	sour, putrid, fishy, amine
2-methyl-pyridine	109-06-8	0.32	0.36	0.51	0.63	sweat
2-acetylpyridine	1122-62-9	0.42	0.54	0.58	0.63	popcorn, corn chips, fatty tobacco
Total		7.77	12.43	14.29[a]	16.70[b]	
Pyrrole						
2-acetylpyrrole	1072-83-9	5.40	1.45	2.09	6.98	musty
2-formylpyrrole	1003-29-8	4.26	3.36	4.60	4.11	musty, beefy coffee
1-furfurylpyrrole	1438-94-4	2.40	2.42	4.67	1.98	waxy, fruity, coffee vegetable
2-acetyl-1-methyl pyrrole	932-16-1	0.62	1.13	1.48	1.41	earthy
Pyrrole	109-97-7	1.02	0.88	0.82	1.25	sweet, warm, nutty, ethereal
2-acetylpyrrole	1072-83-9	0.98	9.17	12.49	1.18	musty, nut skin cherry, liquorice, walnut, bready
Total		14.68	18.42	26.15[a]	16.90[b]	
Alcohol						
Acetyl carbinol	116-09-6	8.03	–	–	6.80	sweet, herbal, oily, nutty
Maltol	118-71-8	2.41	2.83	3.02	2.74	savoury, roasted coffee
Phenylethyl alcohol	60-12-8	1.68	2.59	4.13	1.81	floral, rose, dried rose
Isobutenyl carbinol	763-32-6	0.85	1.26	1.69	1.43	spicy, smoky bacon, phenolic clove
3-pentanol	584-02-1	0.87	0.62	0.73	1.11	pungent, sweet, caramelly, ethereal
2-thiophenemethanol	636-72-6	0.87	0.66	0.58	0.89	sweet, caramel, cotton candy, fruity jam, baked bread
Total		14.70	7.96	10.15[a]	14.78[b]	

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Table 3. To be continued

Compounds	CAS	Concentrations (ppb)				Aroma notes*
		0% 30-min	3% 30-min	3% 90-min	control mature	
Ketone						
2-methyloxolan-3-one	3188-00-9	3.10	1.38	1.74	4.50	sweet, bread, buttery, nutty
Furaneol	3658-77-3	2.95	1.10	1.35	2.85	sweet, cotton candy, caramel, strawberry, sugar
Acetone	67-64-1	2.19	2.14	2.80	3.23	solvent, ethereal, apple, pear
3-hexanone	589-38-8	1.91	1.09	1.50	3.36	sweet, fruity, waxy, rummy, grape
Ethyl cyclopentenolone	21835-01-8	1.86	1.87	2.81	2.47	sweet, caramel, maple, fenugreek
Acetoin	513-86-0	1.82	1.26	1.53	2.04	sweet, buttery, creamy, dairy, milky fatty
1-acetoxyacetone	592-20-1	0.82	0.44	13.13	0.61	fruity, buttery, dairy, nutty
Furfural acetone	623-15-4	0.79	0.99	1.30	1.07	sweet, spicy, warm, balsam, cinnamon, vanilla
Total		15.43	10.27	26.17[a]	20.14[b]	
Aldehyde						
3-furaldehyde	498-60-2	9.30	6.03	10.39	14.92	almond-like odour
Acetaldehyde	75-07-0	3.35	2.76	4.68	3.95	pungent, ethereal, aldehydic, fruity
α-methylbutanal	96-17-3	1.55	1.44	1.84	2.45	musty, phenolic, cocoa, coffee, nutty, alcoholic, fermented, fatty
2,4-hexadienal, (E,E)-	142-83-6	1.47	1.71	1.78	2.00	sweet, green, spicy, floral, citrus
Isovaleral	590-86-3	0.73	0.80	1.30	0.97	chocolate, peach, ethereal, aldehydic, fatty
Pentanal	110-62-3	0.71	0.38	0.77	0.82	berry, fermented, bready, fruity, nutty
Butanal	123-72-8	0.47	0.42	0.47	0.55	pungent, cocoa, musty, malty, bready
Hexanal	66-25-1	0.35	0.42	0.38	0.37	fresh, fatty, aldehydic, green grass, sweaty, leafy, fruity
Total		17.94	13.95	21.61[a]	26.03[b]	
Ester						
2-furfuryl-acetate	623-17-6	6.68	15.91	22.79	13.62	sweet, fruity, banana, horseradish
Methyl-acetate	79-20-9	0.73	1.34	2.06	1.02	ether, sweet, fruity
Total		7.42	17.26	24.85[a]	14.64[b]	

*Aroma description sourced from The Good Scents Company (2021); [a, b] the use of lowercase letters within the rows indicates statistically significant differences ($P < 0.05$); CAS – chemical abstracts service

RESULTS AND DISCUSSION

Sensory assessment. The cupping test was used to assess the quality of immature beans by identifying aroma, taste defects, and the degree of sensory change in response to acetic acid immersion compared to the mature beans. Table 1 shows the result of the sensory assessment of the treated beans.

The sensory analysis revealed that immersing immature Robusta beans in an acetic acid solution significantly enhanced most sensory parameters of the coffee brew ($P < 0.05$), except for the 3% 30-min sam-

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ple. The parameters include aroma, which refers to the smell of brewed coffee. It includes both the initial scent and the lingering aromas. Flavour describes the taste profile of the coffee. It encompasses various flavour notes like fruity, nutty, or chocolatey. Aftertaste is the taste that lingers on the palate after swallowing. A pleasant aftertaste is desirable. Acidity is not related to pH; it refers to the perceived brightness or liveliness of the coffee. Acidity can be citrusy, wine-like, or crisp. Sweetness is the natural sweetness perceived in the coffee. It balances other flavours. Body describes the thickness or viscosity of the coffee. Light-bodied coffee feels thin, while full-bodied coffee is more substantial. Balance is the harmony of flavours – no single aspect dominates. Overall, it is the holistic impression of the coffee, considering all sensory attributes. Uniformity is consistency in flavour across multiple cups. Cleanness refers to the absence of off-flavours or defects. The final score is the cumulative rating based on all parameters, often expressed on a scale of 0 to 100 (Lingle and Menon 2017).

The range of the final score of the treated beans was from 73.50 ± 0.50 to 84.92 ± 0.29, with the highest score obtained by the 3% 90-min samples (84.92 ± 0.29) and the lowest received by the 3% 30-min samples (73.50 ± 0.50). When coffee beans are immersed in acetic acid, the acidity in the beans increases. Excess acidity may disrupt the balance of flavours and reduce the sensory value. However, this depends on the duration and concentration of the immersion as well as the preferences of the panellists. Panellists may perceive that the 3% 30-min sample has an unbalanced sensory value due to the lack of immersion time. Table 2 shows the outcomes of the sensory analysis of the control mature beans and the highest score of treated immature beans immersed in an acetic acid solution.

The 3% 90-min treatment sample showed an increase in sensory values for all parameters except body, uniformity, and cleanness ($P > 0.05$). The 3% 90-min sample had a final score of 84.92 ± 0.29, while the control mature sample received 81.08 ± 0.95. This final score indicates an increase in the sensory quality of immature Robusta beans immersed in 3% acetic acid for 90 min, so that it could approach and even exceed the control mature sample.

The final score referenced a way to describe the range of coffee quality, with scores > 80 equating to Fine Robusta coffees. Based on the final score, the Robusta quality is classified as triage (< 10), off grade (< 20), below grade (< 30), exchange grade (< 40), commercial (40–50), usual good quality (50–70), premium (70–80), fine (80–90), and very fine (90–100) (Hetzel 2015).

Immersing in 3% acetic acid for 90 min or 5% acetic acid for at least 30 min could make the coffee brew obtain a Fine classification. Thus, immersion in 3% acetic acid for 90 min was selected as an alternative method to improve the quality of immature Robusta coffee beans. This condition also resulted in the highest final score, i.e. 84.92. Table 4 shows the final score classification and tasting notes of coffee samples.

Quality description evaluates sensory aspects like aroma, taste, aftertaste, acidity, sweetness, and body. Classification identifies physical defects in beans like breakage and insect damage.

The 3% 90-min sample improved every evaluated characteristic (Table 1) and had the most complete aroma compared to the other treatments (Tables 3 and 4). In terms of aroma, this group even showed much better

Table 4. Classification and tasting notes of coffee samples based on the final score

Samples	Final score	Quality description	Classifications	Flavour notes
Control mature	81.08	fine	fine	chocolaty, sweet, corn-like, bitter, nutty, ginger, cereal-like
0% 30-min	76.17	very good	premium	nutty, cereal-like, soybean aroma, grassy
0% 60-min	76.92	very good	premium	nutty aroma, cereal-like, sweet aroma, soybean aroma, grassy
0% 90-min	76.08	very good	premium	cereal-like, sweet, nutty aroma, soybean aroma
3% 30-min	73.50	very good	premium	chocolaty, somewhat burnt
3% 60-min	79.00	very good	premium	brown sugar aroma, nutty
3% 90-min	84.92	fine	fine	nutty aroma, brown sugar aroma, acidity, sweet aftertaste
5% 30-min	83.75	fine	fine	coconut milk aroma, nutty, peanut
5% 60-min	80.50	fine	fine	chocolaty, astringent aftertaste
5% 90-min	82.33	fine	fine	brown sugar, chocolaty aroma, acidity

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results than the mature coffee beans. In contrast, the 3% 30-min sample experienced the fewest improvements. The character of these tasting notes is due to the composition of non-volatile and volatile compounds, which is influenced by fruit maturity and green bean processing (Velásquez et al. 2019; Osorio Pérez et al. 2023). During roasting, excessive acetic acid might decrease the reactivity of amino groups (Liu et al. 2019). The reduced reactivity of amino groups is believed to be one of the reasons why the aroma of coffee soaked in 5% acetic acid is incomplete. Figure 1 shows the distribution of the sensory attributes of selected samples by the final scores.

A mature coffee brew with a high sensory score signifies that the panellists find the flavour characteristics preferable. This preference is often due to the fully developed chemical components of mature beans contributing to outstanding sensory scores (Osorio Pérez et al. 2023). The increase in the sensory scores of the 3% 90-min sample was possibly due to changes in the composition of flavour precursors in beans when immersed in an acetic acid solution. The composition of precursors such as proteins, carbohydrates and acids determines the resulting sensory characteristics through the Maillard reaction during roasting (Bastian et al. 2021). Weak acids and conjugate bases can accelerate the Maillard reaction and caramelisation during increasing sugar enolisation, which is more easily dehydrated and fragmented than a sugar-shaped ring (Wolfrom et al. 1947).

The duration of immersion may play a crucial role in altering the precursor composition within coffee beans. This process is closely related to hydrolysis, where acetic acid interacts with bean components. Based on the sensory analysis, the immersion time of 90 min results in good sensory quality. During immersion, acetic acid causes hydrolysis reactions in complex compounds like carbohydrates and proteins (Bastian et al. 2021). Carbohydrates are broken down into simple sugars, while proteins are broken down into amino acids. These hydrolysis results affect the quality of the coffee beans and produce compounds that contribute to changes in taste during the roasting and brewing process.

pH value. The acidity attribute is one of the important sensory qualities of coffee and is related to pH in the cupping test. Figure 2 shows the pH values of the samples.

The pH of the green beans in this study was higher than that of the roasted beans. Specifically, the green and roasted 3% 90-min sample exhibited pH values of 5.32 and 5.02, respectively. This finding contrasts with Liu et al. (2019), who reported higher values in the roasted beans. They reported pH values of 4.47 and 5.13 for mature green and roasted Robusta beans, respectively, using 2% acetic acid for 2-h treatment. This difference may be due to acid accumulation in beans during the roasting process. During coffee roasting, the choice of equipment and roasting method significantly influence the bean chemical composition (Diviš et al. 2019). This study used a coffee roaster (Gene Café CBR-101) that operated at a medium-roast level (240 °C for 14 min). In contrast, Liu et al. (2019) utilised a convection oven for dark-roast coffee (200 °C for 20 min). There was no significant dif-

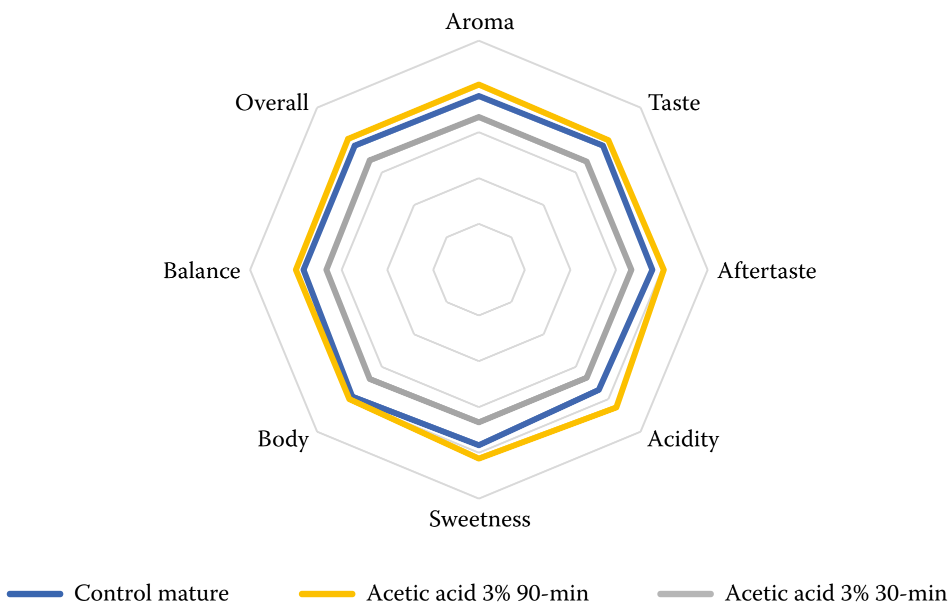

Figure 1. Distribution chart of the sensory characteristics of the control mature and selected treated samples

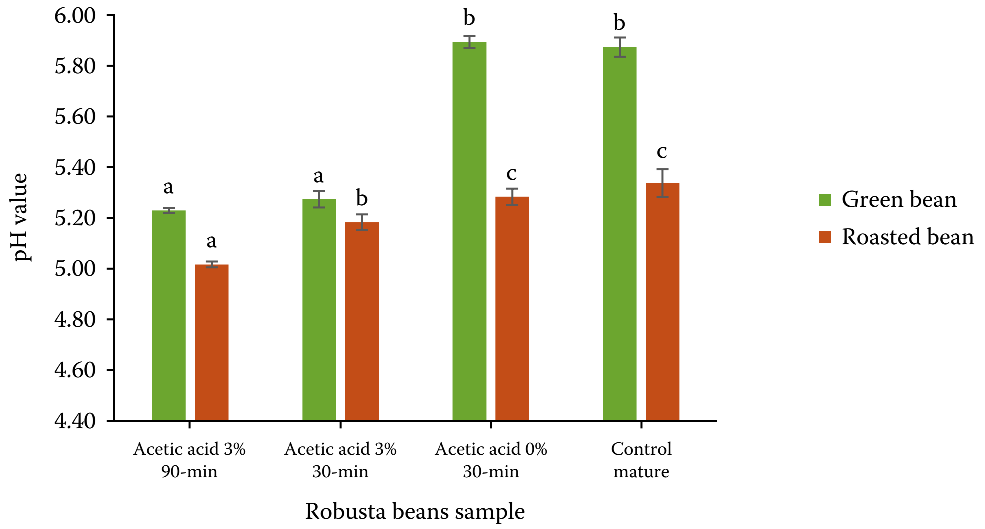

Figure 2. The pH values of the control mature and treated immature samples

[a–c] Different lowercase letters on the same colour bar indicate statistically significant differences ($P < 0.05$)

ference in pH values between the green and roasted control mature and 0% 30-min samples. However, there was a considerable difference between the control mature and acetic acid-immersed samples. When more and more acetic acid is added, the pH will decrease. During the immersion, both water and acetic acid permeated into the beans. As a result, their acid concentration increased, leading to a subsequent decrease in pH.

Various concentrations and immersion times have changed the acidity sensory attributes. Thus, the resulting taste of samples immersed in acetic acid was more acid than in the 0% 30-min samples immersed in water only and in the control mature sample. The decrease in pH during roasting may be attributed to the formation of compounds like citric acid and chlorogenic acid and to the breakdown of organic acids during the first roasting phase (Galarza and Figueroa 2022).

The pH value is closely related to the acidity parameter in the cupping test. An excessively acidic pH results in a lower score. Based on the results of the pH analysis in Figure 2, it is known that the pH of the roasted powder is the lowest, namely at the 3% 90-min treatment. However, based on the sensory analysis score in Table 1, the panellists perceived the acidity score on the 3% 90-min sample as an acidity taste. Hence, it got a high acidity score, and conversely, the 3% 30-min sample was perceived by the panellists as having an unpleasant sour taste, so it got a low acidity score.

Colour index values. A chromameter was used to quantify the colour components. Luminosity (L^*) measures the lightness of the colour, ranging from 0 (black) to 100 (white). The red-green (a^*) value indicates the hue intensity from green (–120) to red (+120), while the yellow-blue (b^*) value shows the hue intensity from blue (–120) to yellow (+120) (Brühl and Unbehend 2021). The colour difference values of the samples are shown in Table 5.

Table 5. Colour index values of the control mature and treated immature samples

Treatments	Colour index					
	green bean			roasted bean		
	L^*	a^*	b^*	L^*	a^*	b^*
Control mature	66.74 ± 0.15^d	-1.91 ± 0.07^a	19.00 ± 0.08^c	40.91 ± 0.25^c	4.62 ± 0.12^b	16.10 ± 0.46^d
Acetic acid 0% 30-min	63.21 ± 0.36^c	-1.06 ± 0.04^c	16.81 ± 0.20^a	48.58 ± 0.39^d	4.93 ± 0.22^c	22.52 ± 0.44^c
Acetic acid 3% 30-min	66.28 ± 0.27^a	-3.43 ± 0.05^c	18.15 ± 0.05^b	39.09 ± 0.11^b	3.93 ± 0.04^a	12.10 ± 0.22^a
Acetic acid 3% 90-min	68.23 ± 0.34^b	-2.69 ± 0.03^b	19.07 ± 0.18^b	38.06 ± 0.28^a	4.23 ± 0.04^{ab}	11.56 ± 0.07^a

[a–d] Different lowercase letters within the same column indicate statistically significant differences ($P < 0.05$); the data are presented as mean ± standard deviation (n =3 samples); L^* – luminosity; a^* – redness; b^* – yellowness

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The colour index values (L^*, a^*, and b^*) show a difference between immature and mature green coffee beans in green and roasted beans ($P < 0.05$). The control mature sample of green beans has heightened brightness and a more pronounced yellow hue than the acetic acid 0% 30-min sample. Notably, the acetic acid immersion treatment enhances brightness and yellowness in green beans. This proves that the difference in maturity in green beans can be seen from the difference in brightness levels and colours. However, identifying quaker beans within a batch of green beans remains challenging due to their colour similarity.

In roasted beans, the L^* and b^* values of the acetic acid 0% 30-min sample were higher than those of the control mature sample. The resulting colour exhibited a light brown hue. Notably, this light brown shade indicates the presence of quaker beans, which fail to fully develop during the roasting process (Rabelo et al. 2021). Consequently, our findings demonstrate that the acetic acid immersion treatment effectively reduces both brightness and yellowness in immature beans during roasting.

Caffeine and chlorogenic acid content. Caffeine and chlorogenic acid are essential substances in coffee beans, classified as phenolic compounds, which contribute to a bitter taste in coffee (Kreuml et al. 2013). Figure 3 shows the caffeine and chlorogenic acid analysis.

The samples treated with 3% acetic acid for 90 min exhibited the lowest caffeine content. However, statistical analysis indicated that the caffeine concentration did not significantly differ between all samples ($P > 0.05$). In contrast, a notable difference was observed in chlorogenic acid concentration between the treated immature samples and the control mature samples ($P < 0.05$). Specifically, the 3% 90-min treatment led to the most substantial reduction in chlorogenic acid.

Caffeine, chemically known as 1,3,7-trimethyl xanthine or methyl theobromine, is a type of purine alkaloid compound that is transparent, tastes bitter, and is stable when roasted (Grzelczyk et al. 2022). Heating will degrade caffeine compounds and turn them into caffeine and CO_2 gas, while H ions (acids) will be neutralised by OH ions (hydroxides). Acidic boiled water will cause caffeine to become unstable salts so that it dissolves easily in water (Olechno et al. 2021). Caffeine significantly influences coffee aftertaste and overall sensory attributes. Simultaneously, chlorogenic acid during roasting

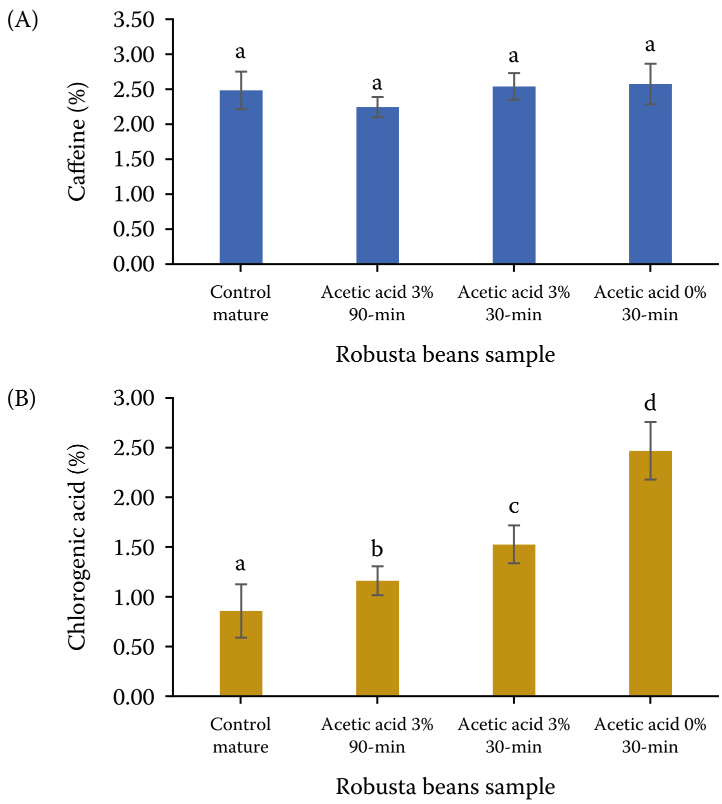

Figure 3. (A) Caffeine and (B) chlorogenic acid concentrations of the control mature and selected treated immature samples

[a–d] Different lowercase letters on the same colour bar indicate statistically significant differences ($P < 0.05$)

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will be degraded and will produce phenolic compounds that play a role in contributing to bitterness, acidity, and astringency (Sunarharum et al. 2014; Lemos et al. 2022).

The sensory result (Table 1) shows that the acetic acid treatment with 0% concentration for 30 min resulted in lower aftertaste, overall, bitterness, acidity, and astringency scores compared to the control group of mature beans. These reduced scores are closely associated with caffeine and chlorogenic acid levels. The caffeine content in coffee beans shows an increasing trend along with the level of fruit maturity, although not significantly (Hu et al. 2020). The caffeine content does not depend entirely on the degree of bean maturity but rather it is more influenced by the type of coffee. On the contrary, the immature coffee cherries contain higher levels of CGAs, which decrease as the coffee cherries mature (Bastian et al. 2021; Lemos et al. 2022). Applying acetic acid at 3% concentration for 90 min can effectively enhance the aftertaste, overall, bitterness, acidity, and astringency scores. This improvement occurs by mitigating the bitter taste attributed to chlorogenic acid (Table 1).

Volatile compounds. The amount and type of volatile compounds significantly affect the aroma produced during roasting (Caporaso et al. 2018). Table 3 shows the quantity of volatile compounds.

There were changes in volatile compounds in the treated samples compared to the mature control samples ($P < 0.05$), where a number of volatile compounds in the 3% 90-min sample had the highest total concentration. This is directly proportional to the aroma parameters of the sensory test results, which have a high score. Mature beans will produce the best combination of aromas (Velásquez et al. 2019). Hence, the immature Robusta beans immersed in 3% acetic acid for 90 min will produce better aromas because the total volatile compounds increase. The thermal degradation of non-volatile compounds like proteins, carbohydrates, fats, caffeine, and chlorogenic acid produces volatile compounds like furan, pyrazine, pyrrole, and others (Seninde and Chambers 2020). The volatile compounds created during roasting are crucial in creating the coffee aroma.

Most individuals enjoy the aroma of coffee that is typically fresh, fragrant, and different, while rancid, musty, or rotten aroma is generally disliked (Seninde and Chambers 2020). The variety, growing conditions, post-harvest handling, roasting, and brewing techniques all affect the amount and composition of volatile compounds (Velásquez et al. 2019; Seninde and Chambers 2020).

Table 3 indicates an improvement in the concentrations of desirable volatile chemicals, including pyridine, furan, pyrrole, ketone, aldehyde, and ester, as well as a decrease in the concentrations of less preferable volatile compounds such as pyrazine, phenol, and alcohol by acid treatment. The acetic acid treatment enhanced total volatile compounds of immature Robusta beans to a degree comparable with mature Robusta beans.

There was an 18.46% increase in the total concentration of furan compounds in the 3% 90-min sample. The most dominant compound was 5-methyl-furfural, with flavour notes like spice, caramel, and maple. A study by Liu et al. (2019) also indicated increasing furan concentrations using acid pre-treatment. The breakdown of ascorbic acid, carbohydrate components, or unsaturated fatty acids throughout the thermal process forms furan and furfural, which produce a sweet, fruity, and caramel-like aroma (Sunarharum et al. 2014).

There was a 48.58% decrease in the total concentration of pyrazine in the 3% 90-min sample. The most dominant compound was 3-ethyl-2,5-dimethyl-pyrazine, which has flavour notes like potato-like, roasted cocoa, and nutty aroma. In contrast, 2-methyl-6-propyl-pyrazine, which has flavour notes like burnt and hazelnut, shows the lowest concentration in the 3% 90-min sample. The Schiff base is produced at the initial stage of pyrazine synthesis through the condensation of carbonyl and amine groups. The pH of the sample significantly impacts the amine reaction rate. The rate of carbonyl-amine condensation reactions decreases with decreasing pH because of the protonated amine group weak reactivity (Hodge 1953).

There was a 0.46% decrease in the total concentration of phenol in the 3% 90-min sample, while 2-methoxy-4-vinylphenol that has flavour notes like sweet, spicy, phenolic, clove-carnation-like, woody, peppery, smoky, powdery was the most dominant compound. The formation of a phenol group, which can occur through the breakdown of quinic acid, is subject to various reactions such as pyrolysis, hydrolysis, oxidation, or decarboxylation. Several factors, including the type of catalyst, temperature, solvent, and the presence of other compounds influence this process (Wang et al. 2013).

Only one sulphur compound group was detected, i.e. trimethyl-oxazole, which has flavour notes like nut, roasted nut skin, wasabi, mustard, and vegetable-like aroma. There was a 30.43% decrease in the total concentration of sulphur in the 3% 90-min sample. When coffee is roasted and brewed, amino acids, proteins, and other compounds break down, forming the sulphur compound group. These compounds can affect the aroma of coffee in different ways, depending on the concentration, structure, and interaction with other compounds (Toci and Farah 2008; Wang et al. 2013).

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There was a 69.60% increase in the total concentration of ketone in the 3% 90-min sample. The 2-methyloxolan-3-one was the most dominant compound with flavour notes like sweet, bread-like, buttery, and nutty. The ketone compound group can be formed from the breakdown of sugars, lipids, and amino acids during roasting and brewing (Pinheiro et al. 2021).

Pyrrole and pyridine compounds result from the breakdown of trigonelline or proline compounds during the Maillard reaction (Seninde and Chambers 2020). There was a 78.13% and 83.91% increase in the total concentration of pyrrole and pyridine, respectively, in the 3% 90-min sample. The most dominant pyrrole compound was 2-acetyl pyrrole, which has flavour notes like musty. Meanwhile, pyridine was the dominant compound with flavour notes like sour, putrid, fishy, and amine. Pyrrole and pyridine are aromatic heterocycles containing one or more non-carbon atoms in the ring. Pyrrole has a five-membered ring with one nitrogen atom, while pyridine has a six-membered ring with one nitrogen atom (Pinheiro et al. 2021).

The total alcohol concentration in the 3% 90-min sample decreased by 30.95%, while 2-furan methanol was the most dominant compound, with flavour notes like sweet, herbal, oily, and nutty. The alcohol compound group is generally associated with aroma characteristics like alcoholic, fruity, sweet, and spicy (Afriliana et al. 2019).

Aldehyde and ester compound groups are generally associated with preferable aroma characteristics (Seninde and Chambers 2020). The respective concentrations of aldehydes and esters increased by 20.46% and 234.91% in the 3% 90-min sample. The most dominant aldehyde compound was 3-furaldehyde, which has almond-like flavour notes. The most dominant ester compound was 2-furfuryl-acetate, which has flavour notes like sweet, fruity, banana, and horseradish. Coffee roasting and brewing can break amino acids, sugars, and fats, forming aldehyde and ester chemical groups (Velásquez et al. 2019; Pinheiro et al. 2021).

CONCLUSION

Numerous coffee plantations in Indonesia continue to be locally managed, yet challenges persist in post-harvest handling and production processes at the farmer level. These challenges contribute to inconsistent coffee quality, including issues related to immature beans. This study established a treatment method that improves the aroma and taste of immature Robusta beans. Sensory analysis revealed that immersing these beans in a 3% acetic acid solution for 90 min resulted in the most significant improvement. This treatment decreased chlorogenic acid concentrations, increased preferable compounds, and reduced less preferable volatile compounds. Consequently, the immature Robusta coffee quality was improved significantly, even achieving the 'Fine Robusta' classification. Thus, acetic acid treatment holds promise for enhancing the taste and aroma quality of immature Robusta beans to a level comparable with the mature Robusta beans.

Acknowledgement. The authors are grateful to the Standardization Agency for Agricultural Instruments, Ministry of Agriculture of the Republic of Indonesia, for the scholarship.

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Received: March 27, 2024
Accepted: October 31, 2024
Published online: November 29, 2024