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Original Research Article

Volatile Compound Analysis of 'Monthong' Durian Harvested at Different Maturity Stages using Flash Gas Chromatography Electronic Nose

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ABSTRACT

This research investigated the effects of maturity stage on the development of volatile compounds during ripening of 'Monthong' durian. Eighty-six raw durians were obtained from orchards, then stored at 30°C and 70% relative humidity to allow the fruits to ripen. Dry matter (DM) content of each ripe durian was determined using an oven method and then the DM content was used to classify each fruit into one of the four groups, with group 1 to group 4 having DM contents of <26.00%, 26.00-29.99%, 30.00-33.99% and >34.00%, respectively. The DM contents of durians in group 1 to group 4 corresponded to 60, 70, 80 and 90% maturation levels, respectively. Volatile compounds of ripe durian flesh from each fruit were identified and quantified using a flash gas chromatography electronic nose equipped with MXT-5 and MX-1701 columns and AroChembase software. Results showed that, based on DM contents, 22, 17, 23 and 24 durian samples were categorized into group 1 to group 4, respectively and that 14 volatile compounds (5 alkanes, 2 ketones, 3 alcohols, 3 esters, and 1 alkanethiol) were detected in all durian groups at varying concentrations. These compounds mainly described fruity, sweet and sulfurous characteristics of the samples. The highest concentration was detected for decane, followed by 2-(5H)-furanone and 1-hydroxy-2-(methylthio)-ethane, respectively. Durian samples in group 1 tended to have lower volatile compound concentrations than those in group 2, although the differences were not significant ($p>0.05$). Contrarily, differences were evident between durians in group 2 and 3, with the former having lower ($p<0.05$) concentrations of methyl cyclohexanecarboxylate, butanoic acid, octadecane, decane, benzyl alcohol, eicosane and ethyl tetradecanoate than the latter. When comparing between the samples in group 3 and 4, concentrations of all volatile compounds were not significantly different ($p>0.05$). Results suggest that maturity stage at harvest is a critical factor that determines eating quality of durian and that the fruit should be harvested after reaching at least 80% maturation level (group 3) so that volatile compounds can be fully developed during ripening. Sensory evaluation should be further performed to substantiate the results of the present study.

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INTRODUCTION

Durian (Durio Zibethinus), the family of Bombacaceae, is one of the most well-known tropical fruits with unique aroma and taste (Chin et al., 2007). It is widely grown in Southeast Asian countries, including Thailand. In 2021, the total area of durian cultivation covered more than 1.3 billion square meters in eastern and southern parts of Thailand such as Chanthaburi, Rayong, Chumphon, Nakhon Si Thammarat, and Yala provinces, producing more than 1.28 million tons of durian. Currently, Thailand is the largest exporter of durian accounting for 77.33% of global export value of fresh durian with an average exporting value more than 3 billion baht per year (Department of Trade Negotiations, 2021; Office of Agricultural Economics, 2022).

Durian fruit is popular owing to its unique sensory property that combine a strong odor, sweet taste and creamy texture. Li et al. (2012) characterized odor property of 'Monthong' durian, one of the most popular durian cultivars, as a combination of an intense sulfury, roasted onion-like odor with fruity, sweet, caramel-like and soup seasoning-like notes. Since durian is a climacteric fruit, harvesting practices and maturity at harvest are the key factors that affect the fruit quality. Durian fruit that is harvested at immature stage often has poor eating quality as flavor and texture of the fruit are not fully developed during ripening. According to Thai Agricultural Standard (TAS 3-2013, 2013), percentage of dry matter (% DM) is commonly used as an index of maturation and durian flesh must constitute at least 32% DM to be classified as "mature". Currently, Thai durians for export contain a large percentage of immature fruits and this becomes a major issue encountered by durian exporting business (Department of agriculture, 2022a, 2022b). Nonetheless, limited studies have been conducted using instrumental analysis to monitor the effects of maturity stage at harvest on volatile compound profile of ripe durian fruit.

Electronic nose is an alternative method which can be used in food quality control and traceability of aroma change as an objective and rapid instrumental sensory analysis. The instrument uses artificial sensors to detect volatile compounds in test samples with high sensitivity and accuracy. It has been used to determine stage of ripeness of fruits such as banana (Chen et al., 2018), kiwifruit (Du et al., 2019) and peach (Voss et al., 2020), among others. Recently, Heracles NEO, an electronic nose based on dual flash gas chromatography technology, is developed and has been dedicated to smell and aroma analysis (Kovac et al., 2020).

The objective of this research was to determine the effects of maturity stage at harvest on the development of volatile compounds during ripening of 'Monthong' durian using flash gas chromatography electronic nose.

MATERIALS AND METHODS

Chemicals compounds

Chemical compounds used in this research were alkane standard (C8-C22) (Aldrich Chemical Co., USA), thiophene (Merck, Darmstadt, Germany) and methanol (Fisher Scientific Limited, UK). All of them had purity grade of higher than 98%.

Durian samples

Eighty-six ripe 'Monthong' durian flesh samples were used in this study. They were from durian fruits harvested from Chanthaburi province at varying maturity stages from 90 to 120 days after anthesis or approximately 60-90 % maturity and then stored at 30°C and 70% relative humidity for 4-8 days (depending on the maturity stages) to allow the fruits to ripen. All durian samples were obtained from a research project entitled "A correlation analysis between efficiency of sorting experts and quality and maturation of `Monthong' durian fruit" funded by Agricultural Research Development Agency (ARDA).

Determination of dry matter content

Dry matter content of each ripe durian flesh sample was determined according to the procedure described by Onsawai and Sirisomboon (2015). Three grams of durian flesh were weighed into an aluminum can with an aluminum lid. Moisture content was analyzed in triplicate using a hot air oven (RL11-10894, redLINE, Germany) at temperature 70±2 °C until a constant weight was reached. Percentage of moisture content (% MC) was calculated using Equation (1) and percentage of dry matter content (% DM) was calculated using Equation (2).

$$MC(\%)=((W_1-W_2)\times100)/W_1 \quad (1)$$

where W_1 is the wet weight (g), and W_2 is the oven-dried weight (g)

$$DM(\%)=100-MC(\%) \quad (2)$$

Based on % DM, each durian flesh sample was classified into one of the four groups as follows: Group 1 (the least mature): <26.00% DM, Group 2: 26.00-29.99% DM, Group 3: 30.00-33.99% DM and Group 4 (the most mature): >34% DM.

Analysis of volatile compounds

Durian flesh samples were kept frozen (-20□C) until used and thawed before analyzed for volatile compounds. Each sample (5 g) was blended and quickly transferred into a 20 mL vial. One microliter of thiophene solution (thiophene:methanol = 1:10) was spiked into the sample as an internal standard, then the vial was crimp-sealed with an aluminum cap with Teflon septum. Heracles NEO 300 electronic nose (Alpha MOS SA, Toulouse, France) with Alphasoft version 2021 was used for volatile compound analysis. The sample vial was incubated at 40∘C for 10 min with 500 rpm agitation speed. Headspace autosampler was used to inject the headspace gas under the following conditions: injection volume 5000 µL, injection speed 1000 µL/s, injection with a split mode at rate 10 mL/min, trapping temperature 50 □C. Separation of volatile compounds was achieved with two metal capillary columns (MXT-5 and MXT-1701, 2 x10 m x 0.18 mm i.d. x 0.4 µm film of absorbing polymer thickness) under oven temperature program as follows: 40 □C initially, held for 3 s, then increased to 250□C at a rate of 2 □C /s and held for 10 s. Hydrogen gas was used as a carrier gas at 0.4 mL/s constant flow rate. The flame ionization detector (FID) was operated at 260 □C. Each sample was analyzed in duplicate. The retention time (RTs) of eluted volatile compounds were recorded, then the RTs from a series of aliphatic alkanes (C8-C22) were used to calculate retention indices (RIs) for all identified compounds using AroChemBase library software. The relative concentration of each volatile compound was calculated from ratio of its peak area to internal standard's peak area using a response factor of 1.

Statistical data analysis

Analysis of variance (ANOVA) was performed to determine if volatile compound concentrations were significantly different among the four durian groups of varying maturation levels. Duncan's multiple range test (DMRT) was then used to compare concentration means of each volatile compound that were significant at 95% confidence level ($p<0.05$). Principal component analysis (PCA), with varimax rotation, was performed to group significant volatile compounds into principal components and to disclose relationship between durian samples and volatile compounds. The statistical software used for ANOVA and DMRT was IBM SPSS Statistics version 28.0 (Thaisoftup Co. Ltd., Thailand) and that used for PCA was XLSTAT statistical software version 19.6 (Addinsoft, New York, USA).

RESULTS AND DISCUSSION

Dry matter content

According to dry matter content, 22, 17, 23, 24 durian flesh samples were classified into group 1, 2, 3 and 4, respectively. Table 1 shows minimum, maximum and mean values of dry matter content of durian samples in each group. The DM contents of durians in group 1 to group 4 corresponded to 60, 70, 80 and 90% maturation levels, respectively. The samples in group 4 had the highest %DM ($P<0.05$), followed by those in group 3, 2, and 1, respectively. Therefore, the samples in group 4 were from the most mature fruits, while those in group 1 were from the least mature fruits. Results confirmed that durian samples used for volatile compound analysis in this study covered a wide range of maturation levels.

Volatile compounds

A total of 14 volatile compounds consisting of 5 alkanes, 2 ketones, 3 alcohols, 3 esters, and 1 aldehyde were identified in durian flesh samples based on the retention indices obtained from the AroChemBase library (Table 2). The results were in accordance with those of Aziz and Jalil (2019) who found that durian was rich in volatile esters, alcohols, ketones and sulfur. Pokhum et al. (2010) also detected esters, sulfur compounds, alcohols, ketone and organic acid in durian. The ester and sulfur-containing compounds, as the major volatile compounds in durian, were responsible for the sweet fruity and onion-sulfur odors, respectively (Chin et al., 2007, Pokhum et al., 2010).

Eight volatile compounds, including 1-hydroxy-2-(methylthio)-ethane, 2-(5H)-furanone, 3-methyl-3-sulfanylbutanol-1-ol, methyl cyclohexanecarboxylate, butanoic acid, dodecyl ester, octadecane, 2-hexadecanone and 1-Hexadecanol, were identified by the MXT-5 column. While six volatile compounds, including decane, benzyl alcohol, eicosane, ethyl tetradecanoate, 7-methyl octadecane and 9-methyl nonadecane, were identified by the MXT-1701 column (Table 2). According to an AroChemBase library, these identified volatile compounds were characterized as caramelized, fatty, floral, fruity, sulfurous, sweet and waxy notes. The sensory descriptions agreed with Li et al. (2012) who described odor characteristics of 'Monthong' durian as a combination of an intense sulfury, roasted onion-like odor with fruity, sweet, caramel-like and soup seasoning-like notes.

Among the 14 volatile compounds, the highest concentration was detected for decane (alkane, fruity, fuel, sweet; 207.89 – 734.02 µg/kg), followed by 2-(5H)-furanone (butter; 198.34 - 674.85 µg/kg) and 1-hydroxy-2-(methylthio)-ethane (meaty, sulfurous; 75.64 – 150.55 µg/kg), respectively (Table 2). Maturity levels of durian significantly ($p<0.05$) affected the concentration of almost all volatile compounds, except 2-hexadecanone and 7-methyl octadecane. Durian samples in group 1 (<26% DM) tended to have lower volatile compound concentrations than those in group 2 (26.00-29.99 %DM), although the difference was not significant ($p>0.05$). Significant differences became evident between durians in group 2 (26.00-29.99 %DM) and 3 (30.00-33.99 %DM), with the former having lower ($p<0.05$) concentrations of methyl cyclohexanecarboxylate, butanoic acid, octadecane, decane, benzyl alcohol, eicosane and ethyl tetradecanoate than the latter. When comparing between the samples in group 3 and 4, results showed that concentrations of all volatile compounds were not significantly different ($p>0.05$).

Table 1. The minimum, maximum, and mean values of dry matter content of 'Monthong' durian samples used in this research

Samples	% Dry matter			
	Criteria	Minimum	Maximum	Mean ± SD
Group 1 (n=22)	< 26.00	7.23	25.82	17.62 ± 4.85^{d}
Group 2 (n=17)	26.00-29.99	26.10	29.42	27.78 ± 1.12^{c}
Group 3 (n=23)	30.00-33.99	30.00	33.95	31.89 ± 1.27^{b}
Group 4 (n=24)	$\geq$ 34.00	34.46	42.53	37.96 ± 2.57^{a}

$^{a-d}$ Means within column with different superscript letters are significantly different ($p\leq 0.05$).

Table 2. Volatile compounds, their sensory descriptors and concentrations detected in 'Monthong' durian samples of different maturity stages using Flash Gas Chromatography Electronic Nose and AroChemBase software

Retention index	Chemical formula	Chemical name	Relevance index	Sensory descriptors	Concentration (µg/kg) detected in durian samples			
					Group 1	Group 2	Group 3	Group 4
MXT-5 column								
831	C_3H_8OS	1-hydroxy-2-(methylthio)-ethane	50.47	meaty, sulfurous	75.64^b	99.46^{ab}	150.55^a	143.22^a
900	$C_4H_4O_2$	2-(5H)-furanone	89.85	butter	198.34^b	337.66^{ab}	674.85^a	517.40^{ab}
984	$C_5H_{12}OS$	3-methyl-3-sulfanylbutanol-1-ol	30.20	broth, chervil, meat (cooked), meat broth, meaty, onion (cooked), spicy, sweet, tartare, vegetable	27.54^c	66.77^{bc}	124.33^{ab}	147.76^a
1029	$C_8H_{14}O_2$	methyl cyclohexanecarboxylate	76.72	berry, ester, fruity, pineapple	7.77^c	10.87^{bc}	26.23^a	20.67^{ab}
1766	$C_{16}H_{32}O_2$	butanoic acid, dodecyl ester	46.40	fatty, fruity, rancid	4.09^c	5.89^{bc}	13.10^a	10.28^{ab}
1815	$C_{18}H_{38}$	octadecane	74.19	alkane, fruity, fuel, fusel, sweet	8.81^b	14.94^b	34.43^a	29.85^a
1825	$C_{16}H_{32}O$	2-hexadecanone	75.67	fruity	11.02^a	7.83^a	11.19^a	10.66^a
1899	$C_{16}H_{34}O$	1-hexadecanol	74.85	faint, floral, oily, sweet, waxy	7.93^b	14.37^{ab}	23.27^a	22.21^a
MXT-1701 column								
998	$C_{10}H_{22}$	decane	64.89	alkane, fruity, fusel, sweet	207.89^c	371.13^{bc}	734.02^a	609.42^{ab}
1232	C_7H_8O	benzyl alcohol	64.39	aromatic, balsamic, faint, floral, fruity, phenolic, rose, sweet	7.53^b	10.62^b	24.03^a	18.64^{ab}
2010	$C_{20}H42$	eicosane	82.99	alkane, fruity, odorless, sweet, woody	36.39^c	48.18^{bc}	94.95^a	75.31^{ab}
1850	$C_{16}H_{32}O_2$	ethyl tetradecanoate	94.62	etheral, oily, orris, sweet, violet, waxy	3.91^c	5.19^{bc}	11.51^a	8.86^{ab}
1816	$C_{29}H_{60}$	7-methyl octadecane	81.01	-	11.49^a	7.02^a	12.02^a	11.60^a
1925	$C_{20}H_{42}$	9-methyl nonadecane	78.86	-	5.32^b	11.54^{ab}	18.71^a	19.04^a

Groups of durians were classified according to dry matter content (DM): Group1(< 26.00% DM), Group 2 (26.00-29.99% DM), Group3 (30.00-33.99% DM), and Group 4 (≥ 34% DM).

[a-c] Means within rows with different superscript letters are significantly different (p≤ 0.05).

Aziz and Jalil (2019) analyzed a fatty acid profile of 'Monthong' durian and identified 10 fatty acids which consisted of decanoic, dodecanoic, tetradecanoic, hexadecenoic, cis-9-hexadecenoic, octadecanoic, cis-9-octadecenoic, cis-9, 12-octadecadienoic, cis-6, 9, 12-octadecatrienoic and eicosanoic. These fatty acids were related to the volatile compounds identified based on an AroChemBase library in the current study including octadecane, eicosane, and decane whose concentrations tended to increase with increased % DM of durian samples.

A principal component analysis (PCA) was performed on volatile compounds that exerted significant differences ($p \leq 0.05$) to depict a map as shown in Figure 1. The first two principal components (PCs) explained a total of 83.76% of the variance. The first PC (PC1), with 44% explained variance, grouped together the following volatile compounds: decane, 2-(5H)-furanone, 1-hydroxy-2-(methylthio)-ethane, 1-hexadecanol, 9-methyl nonadecane, 5-methylfurfural, octadecane and eicosane. Sensory descriptors of the volatile compounds in PC1 were characterized as butter, meaty, sulfurous, sweet, waxy and onion (cooked). While for PC2, with 39% explained variance, the volatile compounds ethyl tetradecanoate, butanoic acid, dodecyl ester, methyl cyclohexanecarboxylate and benzyl alcohol were grouped together.

As seen in Figure 1, most of the durian samples in group 3 and 4 were located closely to all volatile compounds, while those in group 1 and 2 were far away in an opposite direction. Overlapping of durian samples in group 1 & 2 and group 3 & 4 was probably due to moderate sensitivity of electronic nose instrument. Pokhum et al. (2010) found that analysis of volatile compounds using gas chromatography mass spectrometry was less sensitive than descriptive sensory analysis performed by a highly trained panel in discriminating durian samples with different ripeness.

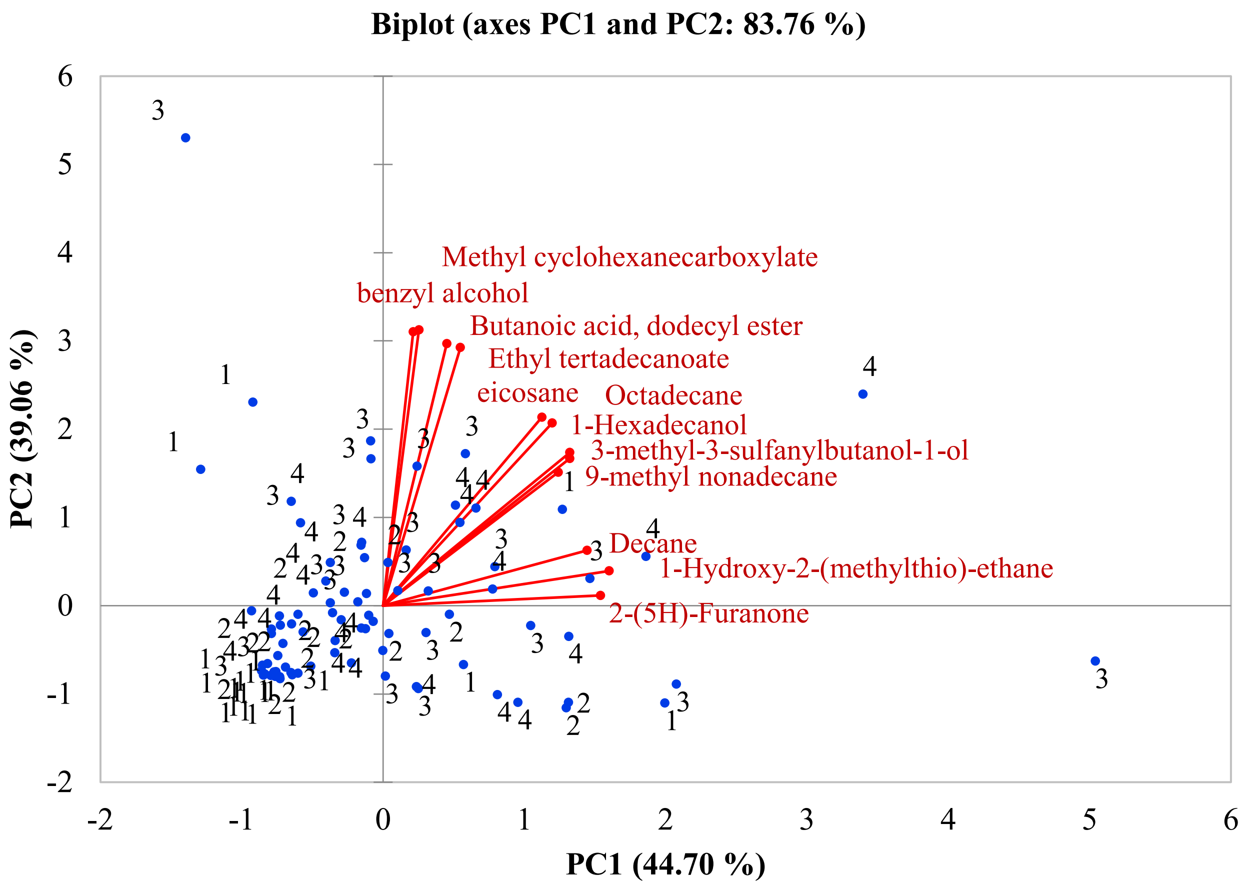

Figure 1. Principal component biplot after Varimax rotation showing relationship between volatile compounds and four durian groups based on dry matter (DM) content. 1 = Group 1 (< 26.00% DM), 2 = Group 2 (26.00-29.99% DM), 3 = Group 3 (30.00-33.99% DM), and 4 = group 4 (⩾ 34% DM).

CONCLUSIONS

Fourteen volatile compounds consisting of 5 alkanes, 2 ketones, 3 alcohols, 3 esters, and 1 alkanethiol were detected in all 'Monthong' durian samples at varying concentrations as determined by a flash gas chromatography electronic nose. The highest concentration was detected for decane, followed by 2-(5H)-furanone and 1-hydroxy-2-(methylthio)-ethane, respectively. Durian samples in group 1 (<26% DM) tended to have lower volatile compound concentrations than those in group 2 (26.00-29.99 %DM), although the differences were not significant ($p>0.05$). Contrarily, differences were evident between durians in group 2 (26.00-29.99 %DM) and group 3 (30.00-33.99% DM), with the former having lower ($p<0.05$) concentrations of volatile compounds than the latter. When comparing between the samples in group 3 (30.00-33.99% DM) and group 4 (>34% DM), concentrations of all volatile compounds were not significantly different ($p>0.05$). Results suggest that maturity stage at harvest is a critical factor that determines eating quality, especially flavor, of durian and that the fruit should be harvested after reaching at least 80% maturation level (group 3) so that volatile compounds can be fully developed during ripening. Sensory evaluation should be further performed to substantiate the results of the present study.

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