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Characterisation of Malaysian durian (*Durio zibethinus* Murr.) cultivars: Relationship of physicochemical and flavour properties with sensory properties

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Abstract

The physicochemical (pH, soluble solids, titratable acidity, sugars and organic acids), flavour and sensory properties of five Malaysian durian cultivars (D2, D24, MDUR78, D101 and Chuk) were studied. There were significant differences ($P < 0.05$) among the five cultivars in terms of all physicochemical characteristics tested with the exception for D2 and MDUR 78, which had similar physicochemical characteristics. Twenty two esters, 14 sulphur compounds, 7 alcohols, 3 aldehydes and 1 ketone were detected in the durian pulp of the five different cultivars using solid-phase microextraction coupled to gas chromatography-time of flight mass spectrometry. Diethyl disulphide, ethyl-*n*-propyl disulphide, diethyl trisulphide and ethanethiol were the predominant sulphur-containing compounds in all the cultivars. The major esters present in durian were either ethyl propanoate, ethyl-2-methyl butanoate, or propyl-2-methylbutanoate and their levels varied within cultivars. Principal component analysis applied to the data differentiated all cultivars based on 29 volatile flavour compounds exhibiting significant differences ($P < 0.05$) between cultivars. Principal components 1 and 2 explained 89% of the total variance. A strong correlation was observed between sensory properties with flavour compound and physicochemical characteristics of the fruit.

Keywords: Durian; Volatile compounds; SPME; GC-TOFMS; Physicochemical; Sensory

1. Introduction

The durian fruit (*Durio zibethinus* Murr.) is one of the most important seasonal fruit in tropical Asia. It is a climacteric fruit (Booncherm & Siriphanich, 1991; Tongdee, Suwanagul, Neamprem, & Bunruengsri, 1990) belonging to the family *Bombacaceae* (Martin, 1980). In this family, the genus *Durio* consists of 27 species, of which six produced edible fruit (Watson, 1984). The durian cultivars grown commercially in ASEAN countries are derived from *D. zibethinus* Murray originating in the Malay Peninsula. The existing commercial cultivars arise from chance seeding, selection by growers and later by breeders in government institutions (Nanthachai, 1994).

Durian grows in warm, wet conditions of the equatorial tropics and is cultivated in Southeast Asia, particularly Malaysia, Indonesia, Thailand, and the Philippines. In Malaysia, about 80% of the total area is planted with seedlings of indigenous cultivars while 20% of the area is planted with clones (Nanthachai, 1994), although there are more than 100 durian clones registered by the Malaysian Agriculture Department. Eight main durian clones widely cultivated are D2, D10, D24, D99, D145, MDUR 78, MDUR 79 and MDUR 88. The D24 clone, which is

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recommended by Malaysian Agricultural Research and Development Institute (MARDI), is extensively cultivated, occupying 70–80% of the total clones planting area (Nanthachai, 1994).

Solid phase microextraction (SPME) is a simple, solvent-free method for isolation and concentration of volatile compounds present in the headspace without modification of these compounds due to temperature or solvent effect (Harmon, 1997; Pawliszyn, 2001). Over the past few years, method based on headspace solid phase microextraction (HS-SPME) has been used extensively in the analysis of volatile compounds in fruits (Chen, Yan, Feng, Xiao, & Hu, 2005; Kourkoutas, Elmore, & Mottram, 2006; Riu-Aumatell, Castellari, López-Tamames, Galassi, & Buxaderas, 2004). A gas chromatograph detector utilizing time-of-flight mass spectrometer (TOFMS), when coupled with SPME sampling technology reduces chromatography time by an order of magnitude without affecting analytical performance (Song, Fan, & Beaudry, 1998). According to these authors, the speed of TOFMS permitted identification and quantification of compounds having chromatographic peak width of only a fraction of a second. In addition, with the use of TOFMS coupled with SPME, an unskewed nature of fragmentation patterns obtained allow individual component spectral characterization of unknown compounds even when they are not fully chromatographically separated.

The durian is characterized by a penetrating, sulphury, often objectionable odour described to be close to that of a rotten onion (Martin, 1980). Several studies on the volatile fractions of different durian cultivars show great variability with regards to the nature and concentration of aroma compounds isolated and determined using GC–MS (Baldry, Dougan, & Howard, 1972; Moser, Duvel, & Greve, 1980; Naf & Velluz, 1996; Weenen, Koolhaas, & Apriyantono, 1996; Wong & Tie, 1995) and fast GCMS (Chin et al., 2007). However, studies devoted to volatile compounds involved in durian flavour are limited (Weenen et al., 1996). Quantitative descriptive analysis (QDA) concerning durian has not, to-date, been reported. To the best of our knowledge, there has been no reports correlating physicochemical and flavour compounds with sensory properties for the characterization of durian aroma.

Research on the physiology, ripening and senescence changes in different durian cultivars had been mainly carried out in Thailand (Booncherm & Siriphanich, 1991; Imsabai, Ketsa, & van Doorn, 2002; Ketsa & Daengkanit, 1998; Ketsa & Pangkool, 1995). It is well known that varietal, geographical, seasonal, and maturity differences greatly influence composition of the fruit. While information on durian fruit grown in Malaysia is available, knowledge about its composition, in terms of individual sugars, organic acids and flavour compounds, is limited. The selection of different cultivars, could be interesting, based on their agronomical (good disease tolerance and early fruiting, ease of vegetative propagation and good field adaptability) as well as their keeping quality and sensory profile. Objective analytical determination of critical components should be coupled with subjective evaluations by a taste panel to yield useful and meaningful information about edible quality of fresh fruit.

The present work aims to characterize and compare the physicochemical properties and detection of headspace volatile compounds using SPME and GC-TOFMS, of five different durian cultivars Chuk Kiok (Chuk), D101, D2, D24 and MDUR78 from Malaysia. Sensory profiling of four cultivars was also carried out to determine how sensory differences between the four cultivars could be correlated with the physicochemical properties and volatiles composition.

2. Materials and methods

2.1. Plant material

Durian fruit (*D. zibethinus*) cultivars (D2, D24, D101, MDUR78 and Chuk) used in this study was obtained from a farm in Bentong, Pahang Darul Makmur, Malaysia (in mid August, 2005). The five cultivars selected were recognized as having typical aroma and were highly priced (Lim, 2005, personal communication). The first four cultivars are widely popular, while Chuk is an unregistered highly priced cultivar favoured by consumers in northern Malaysia. For each cultivar, three batches of 15 fruit were sampled in three replicates. Ripened durian fruit that dropped naturally was collected and transported within 6 h (30 ± 2 °C) to the laboratory. Fruit were selected for uniformity of size and free of visual defects. Durian was dehusked (cut open along the rind), by cutting along the suture on the back of the locules.

2.2. Chemicals

Volatile aroma compounds (propanethiol, propyl propanoate, diethyl disulphide, ethyl propanoate, ethyl 2-methylbutanoate and thiophene with purity $\geqslant 98\%$) of authentic GC standards were purchased from Sigma–Aldrich Company Ltd. (Milwaukee, WI), while sodium chloride was purchased from Merck (Darmstadt, Germany).

2.3. Standard mixture

Standards preparation was carried out according to Chin et al. (2007). In this study, the target aroma compounds previously reported to be key compounds for durian were propanethiol, propyl propanoate, diethyl disulphide, ethyl propanoate and ethyl 2-methylbutanoate. Stock solution of the internal standard (IS), thiophene and other GC standards of propanethiol, propyl propanoate, diethyl disulphide were prepared in methanol at a concentration of 1000 mg/l and ethyl propanoate as well as ethyl 2-methylbutanoate standards at 5000 mg/l.

2.4. Isolation of volatile compounds using headspace-solid-phase microextraction (HS-SPME)

A 50/30 μm divinylbenzene/carboxen on polydimethylsiloxane (DVB/CAR/PDMS) fiber (Supelco, Bellefonte, PA) was used in this study, as it was found to be a suitable fiber for extracting durian volatiles according to Chin et al. (2007). The fiber was conditioned prior use according to supplier's instructions, 30 min at 250 °C. Fifty grams of durian pulp were blended with 100 ml distilled cooled ice water in a Waring blender for 1 min. Blended pulp (15 ml) was quickly transferred into a 30 ml vial containing 5.0 g NaCl and a magnetic stirring bar. Thiophene (15 μg) (Sigma, UK) was spiked into the sample before the vial was crimp-sealed with Teflon septum. After equilibration for 1 h at 30 °C in a water bath, headspace sampling was performed at the same temperature for 30 min under stirring condition. Desorption of the analytes from the fiber coating was made at the injection port of GC at 250 °C for 5 min. Each analytical sample was measured in triplicate.

2.5. Gas chromatography-time of flight mass spectrometry (GC-TOFMS) conditions

An Agilent 6890N gas chromatography system (Wilmington, DE) equipped with electron ionization-Time-of-Flight Mass Spectrometer (Pegasus III, Leco Corp., St. Joseph, MI, USA) was used. Volatile compounds were separated using a Supelcowax-10 (Supelco, Bellefonte, PA, USA) capillary column (10 m × 0.10 mm, 0.10 μm film thickness) with the injector and detector maintained at 250 °C. The injection port was operated at splitless mode with purified helium as the carrier gas flowing at 0.4 ml/min. The oven temperature program was: isothermal at 40 °C for 1.5 min, ramped to 240 °C at 50 °C/min, and then held at this temperature for 2 min. The interface temperature was 240 °C and the ionizing voltage was 70 eV. The mass spectrometer was operated in a scan mode from 35 to 350 amu, and mass spectra collected at a rate of 60 spectra/s. Data were analyzed using the LECO deconvolution software (Chroma-TOF version 2.4).

Identification of aroma compounds was initially accomplished by matching mass spectra with the NIST v2.0 library (Palisade Corp., Newfield, NY) values. Only compounds with a similarity factor more than 800 were chosen. When available, confirmation of the identity of the major volatiles was performed by injecting standard aqueous solutions of each compound using headspace SPME under the same conditions used for the samples. Quantification was carried out by comparing peak areas of analytes to that of thiophene added as internal standard to the samples. The results were expressed as follows:

Peak area/internal standard (IS) area × 1000

2.6. Physicochemical determinations

Soluble solids concentration (SSC) was measured according to Booncherm and Siriphanich (1991) using a hand held refractometer (Atago Co. Tokyo, Japan). The blended pulp was filtered through two layers of Muslin cloth before subjected to SSC determination to give SSC (%) at 20 °C. The pH of slurry was measured using an electrode pH meter (Mettler Toledo). Durian pulp (10 g) was homogenized in a Waring blender with 100 ml of distilled water for 1 min before subjected to pH measurement. After pH determination, the solution was titrated against 0.1 N NaOH to an end point of pH 8.1. Results were expressed as percentage of malic acid (g malic acid/100 g fresh weight).

The method of Hunt, Jackson, Mortlock, and Kirk (1977) was used to carry out sugar extraction. The durian pulp (10 g) was blended and 100 ml of 85% methanol were added. The sample was heated in steam bath for 30 min and filtered. The residue was re-extracted twice with 75 portion of methanol. The collected supernatant was then evaporated using a rotary evaporator and made up to 10 ml using deionised water. Finally, the extracted sample was filtered through Sep-Pak cartridges (Waters Associates, Milford, MA) to remove phenolic compounds, and through a membrane filter of 0.45 μm (Whatman) before injecting to a HPLC system with a RI-1530 (Jusco) detector. The analytical column was Supelcosil™ LC–NH_2 column (5 μm, 25 cm × 4.6 mm ID). The injected volume was 20 μl. The mobile phase used was 75:25 v/v acetonitrile and deionized water filtered through a 0.45 μm filter and degassed ultrasonically. The flow rate was adjusted to 1.0 ml min^{-1}.

The method of Sturm, Koron, and Stampar (2003) with a slight modification was used for organic acid analysis. The durian pulp (5 g) was blended and diluted to 100 ml with deionized water and centrifuged at 4 °C, 12,000*g* for 20 min. The supernatant was filtered through C18 Sep-Pak cartridges (Waters Associates, Milford, MA), to remove the phenolic compounds, and through a 0.45 μm membrane filter paper (Whatman) before injecting to a Shimadzu HPLC system attached with a UV-spectrophotometric detector (SPD-6A, Shimadzu). The analytical column used was Aminex HPX-87H column (300 mm × 7.8 mm). Isocratic elution was performed using 0.008 M H_2SO_4 solution at a flow rate of 0.6 ml/min. Identification and quantification were done by comparison of sample peaks with those of external standards.

2.7. Sensory evaluation

Quantitative descriptive analysis (QDA) was used for sensory evaluation by 12 trained judges (4 females, 8 males) based on the method by Stone, Sidel, Oliver, Woolsey, and Singleton (1974). Subjects were screened and selected (2 sessions) for their sensory ability and trained (6 sessions)

for descriptive analysis according to the guidelines in ISO 8586-2 (1994).

To generate a descriptive language for durian, panelists were provided with three different durian samples (one per session) and were asked to list the sensory characteristics that they considered important in describing the samples. Terminology characterising sensory attributes were developed from panelists' opinion. Descriptors with similar meaning were grouped, and finally a consensus list of 14 terms was chosen to describe durian. A sensory score sheet with 15 cm unstructured scale lines (0–15), each with anchored terms at both ends, was used to indicate the intensity of each attribute by placing a vertical line on the scale. Assessors sat in individual booths and were asked to score the sensory properties of durian, using the 14 terms. For each session, four to five durians of each cultivar were dehusked, their pulp separated and presented to the assessors in randomly numbered capped containers. Three cultivars were assessed at each session, and three sessions were carried out to obtain duplicate value for each cultivar. Panelists were provided with water and unsalted crackers to clear their palates in between samples. At intervals, panelists were advised to take some fresh air before proceeding to the next sample to prevent saturation. Results were quantified by measuring the distance from zero to the vertical line.

2.8. Statistical analysis

The data obtained from the SPME-GC-TOFMS analyses and sensory data were compared for the different cultivars using analysis of variance (ANOVA). ANOVA was performed using the Minitab statistical software (Version 13.32, State College, PA, USA). Principle component analysis (PCA) was carried out using UNSCRAMBLER (Version 7.6, CAMO A/S, Trondheim, Norway) on sensory attributes and components exhibiting significant difference ($P < 0.05$) using ANOVA were used to reduce the data set and to study the interrelationship among the different attributes.

3. Result and discussion

3.1. Physicochemical properties

There were significant differences ($P < 0.05$) among the five cultivars in terms of all physicochemical characteristics tested (Table 1). The three major sugars detected in durian were sucrose, fructose and glucose. Sucrose, the major sugar in all cultivars studied, had mean values of 76.97, 60.33, 80.20, 55.70 and 106.47 g kg^{-1} for D2, D24, MDUR78, D101 and Chuk, respectively. There was no significant difference in the concentration of glucose and fructose in D24, while the ratio of glucose: fructose in the other four cultivars were 1.5. Chuk cultivar had significantly ($P < 0.05$) higher sucrose, total sugar, SSC, citric acid, malic acid, succinic acid, and pH values with the lowest titratable acidity value. The concentration of glucose, fructose, malic and succinic acids was significantly ($P < 0.05$) lower in D24 cultivar. There was no significant difference in all physicochemical characteristics tested in D2 and MDUR78 cultivars. Voon et al. (2006) reported that organic acids present D24 cultivar were malic, citric, tartaric and succinic acids. In this study, tartaric acid was only detected in the D24 cultivar. Acetic and lactic acids reported in durian previously (Brown, 1997) were not detected in this study in all cultivars. In D2, MDUR78, D101 and Chuk cultivars, the predominant organic acid was malic acid, followed by succinic and citric acid. In D24 cultivar, the concentration of citric acid was almost equivalent ($P < 0.05$) to that of malic acid, while the concentration of succinic and tartaric acids were significantly ($P \leqslant 0.05$) lower. MDUR78 and Chuk had significantly higher pH values than other cultivars, while titratable acidity of Chuk was significantly lower compared to the other four cultivars.

Table 1
Composition of varietal durian and significance level for statistical evaluation

Parameter[A]	Durian cultivars[B]					*P*-level[C]
	D2	D24	MDUR78	D101	Chuk	
Sucrose (g kg^{-1})	76.97 ± 11.97^{b}	60.33 ± 6.24^{c}	80.20 ± 4.68^{b}	55.70 ± 7.52^{c}	106.47 ± 4.77^{a}	***
Glucose (g kg^{-1})	25.13 ± 7.06^{ab}	7.34 ± 1.53^{c}	27.70 ± 2.45^{a}	19.70 ± 0.61^{b}	18.67 ± 2.69^{b}	***
Fructose (g kg^{-1})	16.63 ± 5.52^{a}	7.63 ± 1.15^{b}	18.23 ± 1.70^{a}	12.87 ± 0.84^{ab}	12.77 ± 0.42^{ab}	***
Total sugars (g kg^{-1})	118.73 ± 23.96^{ab}	75.30 ± 8.71^{b}	126.13 ± 8.44^{a}	88.27 ± 6.52^{b}	137.90 ± 3.82^{a}	***
SSC (%)	34.0 ± 1.73^{b}	32.0 ± 0.87^{b}	33.0 ± 0.0^{b}	33.0 ± 0.0^{b}	41.0 ± 0.87^{a}	***
Citric acid (g kg^{-1})	0.79 ± 0.72^{ab}	1.78 ± 0.81^{ab}	0.81 ± 0.68^{ab}	0.15 ± 0.13^{b}	2.63 ± 1.76^{a}	***
Malic acid (g kg^{-1})	12.41 ± 0.99^{a}	1.66 ± 0.48^{b}	10.52 ± 0.60^{a}	9.76 ± 0.17^{a}	12.86 ± 2.98^{a}	***
Succinic acid (g kg^{-1})	2.39 ± 0.18^{a}	0.81 ± 0.25^{b}	2.61 ± 0.21^{a}	1.95 ± 0.02^{ab}	3.17 ± 1.10^{a}	***
Tartaric acid (g kg^{-1})	0.00 ± 0.00^{b}	0.76 ± 0.30^{a}	0.00 ± 0.00^{b}	0.00 ± 0.00^{b}	0.00 ± 0.00^{b}	***
pH	7.17 ± 0.09^{b}	6.95 ± 0.03^{b}	7.35 ± 0.06^{ab}	6.88 ± 0.28^{b}	7.60 ± 0.11^{a}	***
Titratable acidity	0.22 ± 0.03^{a}	0.19 ± 0.03^{a}	0.26 ± 0.03^{a}	0.20 ± 0.05^{a}	0.09 ± 0.05^{b}	***

SSC, soluble solids concentration.

[A] Mean ± SD group comparisons by means of parametric LSD test.

[B] $^{a–c}$means within rows with changed letter are significantly different according to LSD test ($P \leqslant 0.05$).

[C] ***Significant at $P \leqslant 0.05$. NS, non-significant at $P \leqslant 0.05$.

Table 2
Correlation matrix of physicochemical variables

	1	2	3	4	5	6	7	8	9	10	11
1. Sucrose	1.000										
2. Fructose	0.321	1.000									
3. Glucose	0.319	**0.992**	1.000								
4. Total sugar	**0.903**	0.694	0.694	1.000							
5. Citric acid	0.689	−0.433	−0.456	0.319	1.000						
6. Malic acid	0.625	0.770	**0.823**	**0.839**	−0.090	1.000					
7. Succinic acid	**0.822**	0.698	0.736	**0.951**	0.176	**0.937**	1.000				
8. Tartaric	−0.436	**−0.816**	**−0.879**	−0.719	0.314	**−0.959**	**−0.868**	1.000			
9. SSC	**0.846**	−0.177	−0.143	0.571	**0.854**	0.366	0.558	−0.165	1.000		
10. pH	**0.982**	0.413	0.401	**0.928**	0.625	0.614	**0.833**	−0.455	0.755	1.000	
11. Titratable acidity	−0.580	0.474	0.417	−0.242	−0.787	−0.152	−0.291	−0.018	**−0.924**	−0.451	1.000

Absolute linear correlation $\geqslant|0.8|$ are marked in bold.

Interdependence of the physicochemical variables was investigated by the analysis of correlation (Table 2). Titratable acidity of the fruit correlated better ($R = -0.787$) with citric acid concentration as compared to the other organic acids or pH. A high titratable acidity value was always associated with a low soluble solids concentration ($r = -0.924$) (Table 2). All the 11 variables tested were highly correlated and greatest correlation being between fructose and glucose ($r = 0.992$). The fruit with high sucrose and total sugar content had higher pH value ($r = 0.982$). Interestingly, malic acid was present in higher amounts when tartaric acid was absent and vice versa ($r = -0.959$). The measurement of SSC was also found to be a better indicator of sucrose content than other sugars ($R = 0.846$).

3.2. Volatile composition of five durian cultivars

Table 3 presents the volatile compounds of five different durian cultivars. Forty-seven different volatile compounds from the headspace of durian were detected by GC-TOFMS, of which 17 were identified (Table 3). The volatiles detected included 22 esters, 14 sulphur compounds, 7 alcohols, 3 aldehydes and 1 ketone. All compounds detected were identical with previous studies (Baldry et al., 1972; Chin et al., 2007; Naf & Velluz, 1996; Wong & Tie, 1995) except for propanal and 1-methylethyl propyl disulphide (isopropyl disulphide), which is reported for the first time in durian. The presence of hydrogen sulphide reported by Baldry et al. (1972) and Moser et al. (1980) and indole derivatives reported by Stanton (1966) were not detected in this study. Results from this study are in agreement with those of Wong and Tie (1995) and Chin et al. (2007), who also did not detect hydrogen sulphide and indole derivatives.

Sulphur compounds (49.7–56.8%) were the predominant compounds followed by ester compounds (14.3–33.6%) and alcohols (7.4–26.2%) present in all cultivars studied except for D2. The predominant compounds in D2 were esters (52.0%) followed by sulphur compounds (28.8%) and alcohols (15.2%). Similarly, Wong and Tie (1995) reported that esters were the major volatile compounds detected in three Malaysian durian cultivars (No. 15, No. 28 and No. 74) and contributed up to 49.25% and 57.88% of the total volatiles. Therefore, it may be concluded that the major compounds contributing to durian volatiles were either ester or sulphur compounds.

In this study, out of the 14 sulphur compounds detected in durian, eight compounds were common in all five cultivars. They included ethanethiol, ethyl methyl disulphide, diethyl disulphide, ethyl *n*-propyl disulphide, diethyl trisulphide, two isomers of 3,5-dimethyl-1,2,4-trithiolane and 1,1-bis(ethylthio)-ethane. All these compounds were previously reported by Wong and Tie (1995) and Chin et al. (2007). Diethyl trisulphide and 1,1-bis(ethylthio)-ethane, were not reported by Wong and Tie (1995). However, Naf and Velluz (1996) and Chin et al. (2007) reported the presence of these two compounds in durian. The eight compounds found in this study might serve as character-impact compounds in durian that contribute to its sulphur note. In this study, diethyl disulphide, ethyl *n*-propyl disulphide, diethyl trisulphide and ethanethiol were predominant among the sulphur containing-compounds with no significant difference detected among the cultivars. However, Chin et al. (2007) reported significant difference in ethyl *n*-propyl disulphide and diethyl trisulphide content in the three durian cultivars studied. This may be due to geographical difference that resulted in the difference in their flavour profile. According to Mattheis and Fellman (1999), besides genetics, environmental, cultural practices, agrichemicals and nutrition will also influence the flavour of the crops through their effects on plant development. In this study, the presence of other sulphur compounds varied with cultivars. For instance, dipropyl trisulphide and 1-(methylthio)-propane were only detected in Chuk and D24 cultivars while 1-methyl ethyl propyl disulphide was only detected in D101 cultivar. Both isomers of 3,5-dimethyl-1,2,4-trithiolane were present in equivalent amounts in all cultivars and this was in agreement with findings by Wong and Tie (1995) and Chin et al. (2007).

The major esters in D2, D24, MDUR78 and Chuk were either ethyl propanoate or ethyl-2-methylbutanoate,

Table 3
Relative amounts of volatile compounds in the headspace of five different durian cultivars

Peak no.	RT	Compound	Relative amount in headspace						
			Chuk	D101	D2	D24	MDUR78	ID	Reference[A]
Aldehyde									
1	38.8	Acetaldehyde	88.3[a]	75.5[a]	271.2[a]	211.2[a]	34.2[a]	A	a
3	43.9	Propanal	57.0[a]	49.9[a]	n.d.[b]	55.6[a]	n.d.[b]	B	–
19	122.4	2-Methylbut-2-enal	54.4[b]	n.d.[b]	n.d.[b]	122.2[a]	n.d.[b]	B	c
		Total	199.7	125.4	271.2	389.0	34.2		
Ketone									
34	182.5	3-Hydroxybutan-2-one	66.5[bc]	115.9[b]	232.0[a]	n.d.[c]	n.d.[c]	B	c
Alcohol									
8	67.1	Ethanol	283.2[a]	612.8[a]	1204.2[a]	688.7[a]	722.2[a]	A	a, f
15	104.0	1-Propanol	291.0[a]	104.5[a]	163.8[a]	n.d.[a]	158.7[a]	B	a, c
24	143.3	1-Butanol	26.3[b]	39.1[b]	219.6[a]	24.9[b]	15.8[b]	B	c
28	157.3	2-Methylbutan-1-ol	30.7[a]	98.2[a]	40.5[a]	n.d.[a]	173.9[a]	B	c
30	158.1	3-Methylbutan-1-ol	47.3[a]	38.5[a]	184.8[a]	n.d.[a]	35.7[a]	B	a, c
36	187.6	1-Hexanol	n.d.[d]	28.5[a]	16.7[b]	n.d.[d]	7.9[c]	B	c
43	231.5	Butane-2,3-diol	n.d.[c]	27.2[b]	77.4[a]	n.d.[c]	n.d.[c]	B	c
		Total	678.5	948.8	1907.0	713.6	1114.2		
Sulphur containing compound									
2	40.1	Ethanethiol	405.3[a]	42.4[a]	313.9[a]	625.5[a]	40.9[a]	B	a, c, f
4	46.8	1-Propanethiol	275.9[ab]	n.d.[b]	73.9[b]	334.4[a]	50.5[b]	A	a, c, f
7	60.1	Methyl propyl sulphide	30.8[a]	n.d.[a]	n.d.[a]	39.7[a]	n.d.[a]	B	f
21	131.9	Methyl ethyl disulphide	83.7[b]	43.5[b]	99.9[b]	179.4[a]	62.7[b]	B	c–e
26	149.7	Diethyl disulphide	1139.1[a]	840.1[a]	989.1[a]	1796.6[a]	1217.4[a]	A	b–f
27	154.0	Methyl propyl disulphide	81.0[b]	12.2[b]	553.0[a]	n.d.[b]	34.0[b]	B	c, e, f
31	170.3	Ethyl propyl disulphide	674.7[a]	337.9[a]	188.0[a]	450.5[a]	507.0[a]	B	b, c, e, f
35	185.6	Dipropyl disulphide	121.6[a]	n.d.[b]	16.7[b]	n.d.[b]	43.6[b]	B	e, f
37	189.2	1-Methylethyl propyl disulphide	n.d.[b]	41.9[a]	n.d.[b]	n.d.[b]	n.d.[b]	B	–
40	213.1	Diethyl trisulphide	583.0[a]	331.0[a]	842.2[a]	1016.5[a]	298.7[a]	B	b, c, e, f
42	229.3	3,5-Dimethyl-1,2,4-Trithiolane (isomer 1)	118.9[ab]	56.8[b]	198.2[ab]	353.0[a]	269.7[ab]	B	c, d, f
44	232.5	3,5-Dimethyl-1,2,4-Trithiolane (isomer 2)	128.9[ab]	65.1[b]	230.2[ab]	395.3[a]	288.5[ab]	B	c, d, f
46	239.0	Dipropyl trisulphide	28.8[ab]	n.d.[b]	n.d.[b]	31.2[a]	n.d.[b]	B	e, f
47	251.9	1,1-Bis(ethylthio)-ethane	66.8[bc]	27.8[c]	114.5[b]	95.4[bc]	221.8[a]	B	e, f
		Total	3129.0	1798.7	3619.7	5317.5	3034.8		
Esters									
5	55.1	Ethyl acetate	4.47[a]	141.0[a]	96.7[a]	23.9[a]	77.7[a]	A	a–d, f
6	58.4	Methyl propionate	284.3[a]	26.3[a]	67.0[a]	203.1[a]	206.6[a]	A	a, c, d, f
9	70.9	Ethyl propanoate	507.2[b]	96.8[b]	1091.3[ab]	1528.6[a]	264.7[b]	A	a, c, f
10	74.2	Ethyl 2-methylpropanoate	25.6[a]	n.d.[a]	71.0[a]	205.8[a]	n.d.[a]	A	a, c, d, f
11	80.6	Methyl buatanoate	n.d.[b]	n.d.[b]	64.10[a]	53.7[a]	n.d[b]	A	c, f
12	89.1	Methyl 2-methylbutanoate	222.2[ab]	n.d.[b]	195.2[a]	140.4[ab]	n.d.[b]	A	a, c, d, f
13	100.0	Ethyl butanoate	n.d.[a]	n.d.[a]	257.2[a]	68.7[a]	n.d.[a]	A	a, c, f
14	103.9	Propyl propanoate	131.7[a]	n.d.[a]	63.5[a]	196.6[a]	n.d.[a]	A	a, c, f
16	107.4	Propyl 2-methylpropanoate	n.d.[b]	n.d.[b]	n.d.[b]	132.8[a]	n.d.[b]	B	c
17	108.9	Ethyl 2-methyl butanoate	103.4[b]	82.4[b]	2030.5[a]	348.8[b]	42.5[b]	A	a–f
18	112.8	Ethyl 3-methylbutanoate	n.d.[b]	n.d.[b]	n.d.[b]	71.0[a]	n.d.[b]	A	a, c, f
20	130.0	Propyl butanoate	n.d.[a]	n.d.[a]	273.4[a]	45.9[a]	n.d.[a]	A	c, f
22	136.4	Propyl 2-methylbutanoate	121.8[a]	259.4[a]	338.8[a]	235.5[a]	35.9[a]	B	a, c–f
23	140.8	Ethyl but-2-enoate	19.5[ab]	n.d.[b]	187.1[a]	n.d.[b]	n.d.[b]	B	c, f
25	146.3	Methyl hexanoate	n.d.[b]	n.d.[b]	98.3[a]	n.d.[b]	n.d.[b]	A	c, f
29	157.9	Ethyl hexanoate	n.d.[b]	n.d.[b]	587.5[a]	n.d.[b]	n.d.[b]	B	c–f
32	178.1	Propyl hexanoate	n.d.[a]	n.d.[a]	446.8[a]	n.d.[a]	n.d.[a]	A	f
33	181.0	Ethyl heptanoate	n.d.[b]	n.d.[b]	101.3[a]	n.d.[b]	n.d.[b]	B	c, f
38	196.2	Methyl octanoate	n.d.[b]	4.3[ab]	56.3[a]	n.d.[b]	n.d.[b]	B	c, f
39	203.9	Ethyl octanoate	14.2[b]	21.3[b]	419.0[a]	n.d.[b]	58.1[b]	B	c, e, f
41	217.7	Ethyl 3-hydroxybutanoate	n.d.[b]	n.d.[b]	58.3[a]	n.d.[b]	n.d.[b]	B	c
45	235.4	Ethyl decanoate	n.d.[c]	n.d.[c]	34.0[a]	n.d.[c]	15.3[b]	B	c
		Total	1434.4	631.5	6537.3	3254.8	700.8		

RT, retention time on a Supelcowax-10 capillary column.
ID: A, GC retention and MS data in agreement with that of authentic reference; B, tentatively identified by MS matching with library spectra only. Results are the means of triplicate analyses.
Letters [a–b] indicate there are no significant difference ($P < 0.05$) with the same letter using Fisher's least significance difference among the samples.
[A] Volatile reported previously in durian. Letter corresponds to reference: a, Baldry et al. (1972); b, Moser et al. (1980); c, Wong and Tie (1995); d, Weenen et al. (1996); e, Naf and Velluz (1996); f, Chin et al. (2007).

followed by propyl 2-methylbutanoate and their predominance varied with cultivars. This result was also in agreement with Wong and Tie's study (1995), who suggested that these two compounds were the major ester compounds among durian volatiles. Study by Chin et al. (2007) showed that ethyl-2-methylbutanoate and propyl 2-methylbutanoate were the major esters present in all (D101, D2 and D24) Malaysian durian cultivars. Propyl 2-methylbutanoate was the predominant ester in D101 cultivar. Naf and Velluz (1996) reported that propyl 2-methylbutanoate was the second most abundant ester extracted from durian, next to ethyl 2-methylbutanoate. Ester compounds other than ethyl propanoate, ethyl-2-methylbutanoate and propyl 2-methylbutanoate detected in all five cultivars were ethyl acetate, methyl propionate, ethyl propanoate, ethyl 2-methylbutanoate and propyl 2-methyl butanoate. Their presence was also confirmed in previous studies by Wong and Tie (1995) and Chin et al. (2007). Thus these five esters might serve as the character-impact compounds that contribute to the fruity note in durian. Ethyl but-2-enoate, an unsaturated ester, was only detected in Chuk and D2 cultivars. Chin et al. (2007) had also reported this compound in D2 cultivar using fast GCMS. Other unsaturated ester reported previously, E-2-methylbut-2-enoate (Wong & Tie, 1995) was not detected in this study.

Wong and Tie (1995) reported α-hydroxyketone as the second most abundant class of compounds extracted when using dichloromethane as the extracting solvent and this compound accounted for about one third of the total volatiles of durian. In this study, 3-hydroxybutan-2-one, which was reported to be the dominant α-hydroxyketone, by Wong and Tie (1995), was detected only in small amounts in Chuk, D101 and D2 cultivars. This could be attributed to the different extraction method used to extract the flavour compounds.

Seven alcohol compounds were detected in this study, with ethanol and 1-butanol present in all the five cultivars studied (Table 2). All these alcohol compounds were also detected in Wong and Tie's study (1995), except for ethanol which was detected by Chin et al. (2007) by using fast GCMS. Although all the alcohol compounds detected had been previously reported, their presence was controversial (Baldry et al., 1972; Chin et al., 2007; Wong & Tie, 1995). Ethanol was the predominant alcohol present in all the durian cultivars. Wong and Tie (1995) did not detect ethanol in their study and suggested that its presence was an indication of fermentation occurring during storage of durian. However in our opinion, the presence of ethanol was more likely due to accumulation during ripening process. The accumulation of ethanol during ripening had been reported in kiwi (Young & Peterson, 1985) and tomato (Ratanchinakorn, Klieber, & Simons, 1997). SPME used in this study was found to be a more sensitive method in extracting ethanol as compared to solvent extraction and steam distillation methods.

Principal component analysis (PCA) was applied to the data to identify the most important factors of variability and also to describe the relationship between variables and observations. The 29 volatile components exhibiting significant difference in ANOVA were subjected to grouping by PCA (Fig. 1). The first two principal components (PC1 and PC2) accounted for 79% of the variation in the data. PC1 and PC2 displayed 49% and 30% variance, respectively. Cultivar D2 was separated from the other four cultivars across PC1. The volatile compounds associated with D2 included a sulphur compound (methyl propyl disulphide), 2 alcohols (1-butanol and 2,3-butanediol) and 11 esters compounds. Cultivar D24 was separated from Chuk and D101 across PC2. Four sulphur compounds (1-propanethiol, methyl ethyl disulphide, two isomers of 3,5-dimethyl-1,2,4-trithiolane and dipropyl trisulphide), two esters (ethyl propionate and propyl 2-methylpropanoate) and 2-methylbut-2-enal were associated with D24 while 1-hexanol and 1-methylethyl propyl disulphide were associated with D101. Meanwhile, cultivar Chuk was associated with propanal and methyl propyl disulphide.

3.3. Sensory characteristics of four durian cultivars by QDA

Selection of similar terms used by the panelists resulted in a final list of 14 descriptors out of 70 descriptors for durian (Fig. 2). The colour of fruit ranged from whitish to golden yellow. Six aroma descriptors generated included were sweet, fruity, sulphur, alcohol, nutty and green notes. The green aroma perceived was reminiscent to a fresh cucumber-like aroma as described by the panelists. Creamy, sticky and moist and smooth sensations were used to describe the texture properties of the fruit, while sweet and bitter were employed to describe the taste of durian fruit. The sour note was not generated despite the presence of organic acids in durian. This was probably masked by the presence the high amount of sugar in the fruit. Martin (1980) reported that durian had two distinguishable notes, which were strong and onion-like, and delicate and fruit-like. These two descriptors were similar to the sulphur and fruity aroma terms generated in this study.

Generally, cultivar Chuk was most intense in colour (orangish yellow), and had the smoothest, stickiest and creamiest texture, with greatest green aroma as compared to others. The panelists preferred this cultivar mainly due to its physical character instead of its aroma intensity. In terms of odour, cultivar D2 was associated with the highest sulphur notes, while D24 possessed the highest sweet and nutty notes with the strongest bitter taste. Cultivar D101 did not have a distinguishable note, which separated it from other cultivars.

The intensities of sweet aroma of the four cultivars as determined by the panelists in descending order were: D24, D101, D2 and Chuk. The intensity of fruity aroma was the least in Chuk, while no significant difference was detected in the other cultivars. The strongest sulphur aroma was perceived in D2 followed by Chuk and D24 (with no significant difference between them). Despite the highest

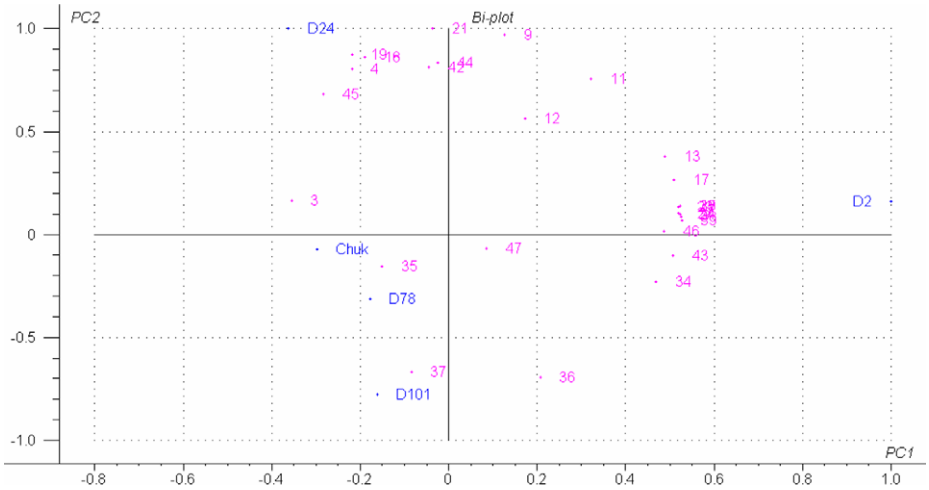

Fig. 1. Principal component plot (PC1 versus PC2) of five durian cultivars, showing correlations with statistically significant volatile compounds (numbers on plot refer to compound in Table 1). PC1 and PC2 are 49% and 30% of variation, respectively.

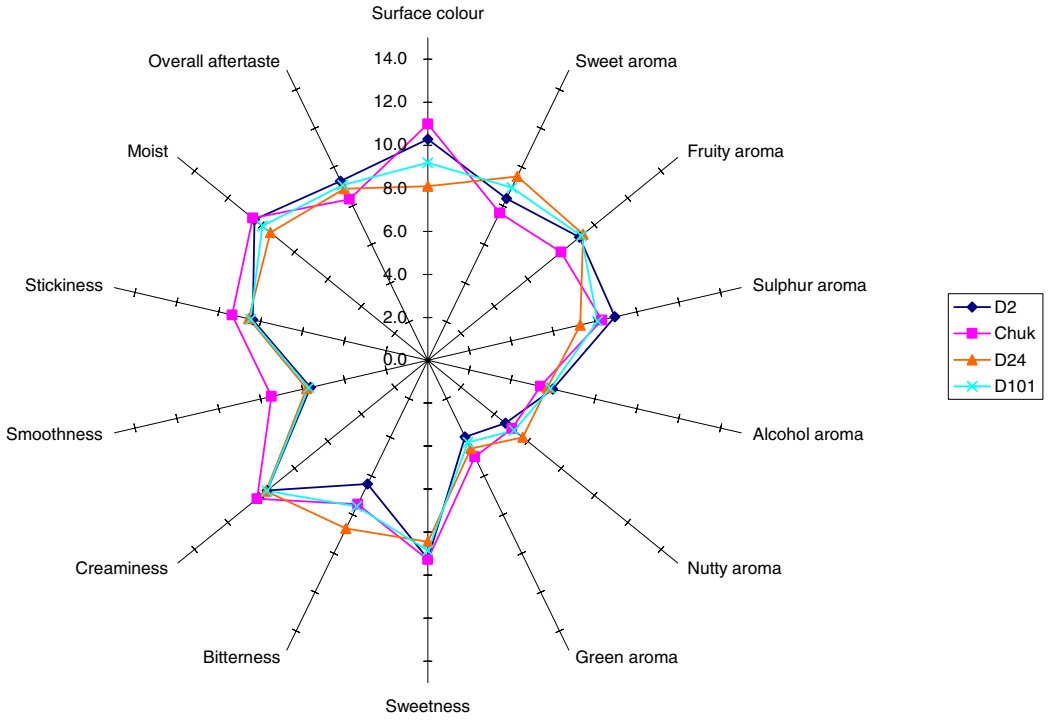

Fig. 2. Comparison of sensory profiles composed of average scores of 14 attributes identified in four durian cultivars.

concentration of total sulphur compounds in D24, the sulphur aroma detected was the lowest. This suggested that the total sulphur-containing compounds were not the major contributor to sulphur aroma. Instead, the intensity of sulphur aroma perceived in D2 was most probably due to methyl propyl disulphide, as it was the major sulphur compound that separated D2 from other cultivars (Fig. 1). In addition, this compound had the strongest correlation with sulphur note ($R = 0.756$) as compared to other sulphur compounds ($R \leqslant 0.205$) (Table 4).

The alcohol aroma detected in durian was higher in D2, D24 and D101 than Chuk although there was no significant difference ($P < 0.05$) in their ethanol content. Correspondingly, the total ethanol content in Chuk cultivar was lowest compared to other cultivars. The nutty aroma that was strongly perceived by the panelists in cultivar D24 was not as intense in other cultivars while green aroma was most prevalent or intense in Chuk, followed by D24, D101 and D2 cultivars. Besides the descriptors in this study, other potential descriptors for durian aroma generated that included woody, waxy and pungent, may require more intensive training before panelists are able to discriminate their intensity difference between durian samples.

Table 4
Correlation between sensory descriptors and flavour compounds

Variable	Aroma perceived					
	Sweet	Fruit	Sulphur	Alcohol	Green	Nutty
Propanal	−0.055	−0.442	−0.726	−0.846	0.896	0.749
1-Propanethiol	0.540	0.316	−0.698	−0.068	0.141	0.649
Ethyl propanoate	**0.940**	**0.949**	−0.439	0.713	−0.642	0.409
Methyl butanoate	0.798	**0.971**	−0.051	**0.950**	**−0.913**	0.016
Methyl 2-methylbutanoate	0.174	0.302	0.165	0.395	−0.400	−0.177
Ethyl butanoate	0.354	0.694	−0.489	**0.970**	**−0.990**	−0.519
Propyl 2-methyl-propanoate	**0.908**	0.669	**−0.905**	0.174	−0.071	0.889
Ethyl 2-methyl butanoate	0.220	0.587	0.606	**0.927**	**−0.961**	−0.633
Ethyl 3-methyl butanoate	**0.908**	0.669	**−0.905**	0.174	−0.071	0.889
2-Methylbut-2-enal	0.722	0.436	**−0.906**	−0.062	0.157	0.899
Propyl butanoate	0.256	0.617	0.576	**0.940**	**−0.970**	−0.604
Methyl ethyl disulphide	**0.952**	0.832	−0.694	0.454	−0.364	0.671
Ethyl but-2-enoate	0.042	0.431	0.736	0.841	−0.892	−0.759
1-Butanol	0.054	0.443	0.729	0.849	−0.899	−0.753
Methyl hexanoate	0.093	0.478	0.704	0.870	**−0.917**	−0.728
Methyl propyl disulphide	0.009	0.400	0.756	0.821	−0.875	−0.778
Ethyl hexanoate	0.093	0.478	0.704	0.870	**−0.917**	−0.728
Ethyl heptanoate	0.093	0.478	0.704	0.870	**−0.917**	−0.728
3-Hydroxybutan-2-one	−0.356	0.032	**0.927**	0.545	−0.627	**−0.938**
Dipropyl disulphide	−0.506	−0.527	0.205	−0.418	0.382	−0.188
1-Hexanol	−0.457	−0.301	0.525	−0.013	−0.045	−0.520
1-Methylethyl propyl disulphide	−0.500	−0.573	0.101	−0.522	0.494	−0.081
Methyl octanoate	0.056	0.444	0.728	0.850	**−0.900**	−0.751
Ethyl octanoate	0.052	0.441	0.732	0.849	**−0.900**	−0.755
Ethyl 3-hydroxybutanoate	0.093	0.478	0.704	0.870	**−0.917**	−0.728
3,5-Dimethyl-1,2,4-trithiolane, (isomer 1)	**0.980**	0.899	−0.634	0.555	−0.468	0.607
Butane-2,3-diol	−0.088	0.293	0.784	0.728	−0.788	−0.802
3,5-Dimethyl-1,2,4-trithiolane, (isomer 2)	**0.983**	**0.914**	−0.611	0.583	−0.497	0.585
Dipropyl trisulphide	0.402	0.127	−0.731	−0.276	0.346	0.733
Ethyl decanoate	0.093	0.478	0.704	0.870	**−0.917**	0.728
1,1-Bis(ethylthio)-ethane	0.661	0.852	0.050	0.887	−0.862	−0.081
Total alcohol	0.009	0.395	0.746	0.811	−0.864	−0.768
Total sulphur-containing compound	**0.928**	0.846	−0.608	0.517	−0.434	0.583
Total esters	0.482	0.785	0.355	**0.987**	**−0.992**	−0.387
Total aldehyde	**0.961**	**0.906**	−0.572	0.597	−0.515	0.545

Absolute linear correlation $\geqslant|0.9|$ are marked in bold.

3.4. Correlation of sensory scores with objective measurements

Table 4 shows the correlation between selected sensory attributes and flavour compounds with significant difference ($P \leqslant 0.05$) among cultivars. Sweet notes correlated strongly with both ester and sulphur containing compounds as well as total aldehyde. Meanwhile, ester and total aldehyde compounds correlated well with fruity notes perceived. However, sulphur compounds did not correlate strongly with the intensity of sulphur notes perceived. The perceived alcohol notes correlated strongly with some esters, while the presence of esters and sulphur compounds seemed to mask the green note perceived. There are no direct correlation between nutty notes and flavour compounds. However, nutty notes correlated negatively with the concentration of 3-hydroxybutan-2-one.

Although 3,5-dimethyl-1,2,4-trithiolane was reported to contribute to a strong durian note by Weenen et al. (1996), their presence did not correlate well with the intensity of sulphur notes in this study. Likewise, ethyl 2-methylbutanoate, which was found to have the highest odour impact among the non-sulphurous odourants in durian (Weenen et al., 1996), did not correlate well with the fruity aroma perceived. This could be explained by the fact that the increase in perceived intensity of different odourants with increasing concentration may not be a linear function (Frijters, 1979). The concentration of 3,5-dimethyl-1,2,4-trithiolane and ethyl 2-methylbutanoate in this study may be at a level where changes in concentration do not result in significant aroma changes. Instead, sweet aroma showed a strong correlation with both isomers of 3,5-dimethyl-1,2,4-trithiolane ($R > 0.98$), methyl ethyl disulphide ($R = 0.952$) and three other esters but to a lesser degree. Although 3,5-dimethyl-1,2,4-trithiolane and methyl ethyl disulphide did not contribute directly to sweet notes, these compounds may have synergistic effects on other flavour compounds that contribute to sweet notes. Dramatic synergistic effects between unrelated volatiles have been suggested by Fu, Yoon, and Bazemore (2002). Similarly,

Table 5
Correlation between sensory descriptors and objective measurements

Variable	Sweetness	Bitterness	Moist	Overall aftertaste
Sucrose	0.735	−0.345	0.781	−0.723
Glucose	0.858	**−0.957**	0.834	0.310
Fructose	0.859	**−0.966**	0.833	0.349
Total sugar	**0.943**	−0.663	**0.957**	−0.456
SSC	0.547	−0.054	0.587	**−0.909**
Citric acid	0.126	0.327	0.168	−0.871
Malic acid	**0.966**	−0.870	**0.962**	−0.096
Succinic acid	**0.953**	−0.694	**0.964**	−0.414
Tartaric acid	−0.865	0.833	−0.855	−0.021
pH	0.741	−0.327	0.770	−0.737
Titratable acidity	−0.362	−0.158	−0.408	**0.980**

Absolute linear correlation $\geqslant |0.9|$ are marked in bold.

3,5-dimethyl-1,2,4-trithiolane (isomer 2) correlated strongly with fruity aroma in durian, together with ethyl propanoate and methyl butanoate.

Table 5 shows the correlation between selected sensory attributes and physicochemical properties among cultivars. The sweetness of durian correlated well with total sugar of the fruit but not with SSC. Hence, SSC in this study was not a good indicator of sweetness of durian. Instead, the intensity of sweetness was enhanced by the presence of malic and succinic acids with *R*-value of 0.966 and 0.953, respectively. The concentration of glucose and fructose, on the other hand, correlated negatively with the bitterness perceived. Durian with a bitter taste tended to have a lower glucose and fructose concentration. This could either be due to the presence of reducing sugar that exerted a masking effect on bitterness or the bitter fruit tended to have low fructose and glucose content. The concentration of total sugar, malic and succinic acids correlated strongly with the intensity of moistness perceived (*R*-value 0.957–0.964). Higher titratable acidity was associated with more persistent aftertaste of the fruit ($R = 0.980$).

4. Conclusions

The sweetness in durian was mainly contributed by sucrose that differentiated the Chuk cultivar from the other four cultivars. The sour note was not detected despite the presence of organic acids. Mixture of esters, sulphur compounds and alcohol are responsible for the fruity and sulphur notes, which are important characteristics of the Malaysian durian aroma. Differences perceived among the five cultivars are related to the levels and types of volatiles identified as key aroma compounds by the SPME-GC-TOFMS analysis. Sensory attributes generated to describe durian included sweet, sulphur, fruity, alcohol, nutty and green aroma, sticky, creamy, and smooth texture, together with sweet, bitter and overall aftertaste. Sensory descriptive analysis indicated that sulfur compounds were involved in the typical, basic durian flavour, although these compounds do not correlate directly with the intensity sulphur notes perceived. Several compounds, like ethyl propanoate, methyl butanoate, propyl-2-methyl propanoate, ethyl-3-methyl-butanoate, were highly correlated with the sweet and fruity notes of different durian cultivars. The characterization of aroma for durian cultivars using SPME coupled with GC-MS and with GC-O in order to identify volatile compounds with olfactive impact will be the subject of further investigation.

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