Volatile flavour compounds and sensory properties of minimally processed durian (*Durio zibethinus* cv. D24) fruit during storage at 4 °C

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Abstract

Flavour volatile compounds and sensory attributes in minimally processed durian (*Durio zibethinus* cv. D24) fruit stored at 4 °C for 42 days were examined. The volatile compounds were extracted by solid-phase microextraction (SPME) and analysed by gas chromatography–time of flight mass spectrometry (GC–TOFMS). Forty compounds were identified, among which sulfur compounds, esters, and alcohols were found to be the major constituents. During storage of minimally processed durian at 4 °C, decreases in levels of the majority of ester compounds were observed after 14 days of storage. All ester compounds decreased significantly ($P \leq 0.05$) after 1 week of storage except for ethyl acetate that decreased after 2 weeks. Ethanethiol, 1-propanethiol, and both isomers of 3,5-dimethyl 1,2,4-trithiolane decreased significantly after 7 days of storage. Total sulfur content of fruit remained unchanged after 42 days of storage. Benzyl alcohol was produced after 4 weeks of storage and increased thereafter. Principal component analysis (PCA) applied to the data differentiated the fruit over the storage period based on 22 compounds exhibiting significant changes between samples and explained 86% of the total variance with two principal components. Quantitative descriptive analysis (QDA) was carried out using sixteen descriptors to describe the surface colour, odour, flavour and texture of fruit during storage. Fruit could be stored for 21 days, after which the green aroma became too intense and rendered the fruit unacceptable. Sulfur notes decreased gradually throughout storage while off odours developed on day 21 and increased to an unacceptable level on day 28 of storage. Sweet and fruity aroma correlated strongly with some ester and aldehyde compounds, while correlations between perceived sulphur aromas and sulphur compounds were poor. A green note and off-odours correlated well with benzyl alcohol and 1-hexanol.

Keywords: Durian; Minimally processed; Flavour; Sensory; Storage

1. Introduction

Durio zibethinus, commonly known as durian, is an exotic, seasonal fruit enjoyed in South East Asia due to its unique aroma, taste and texture. It is a climacteric fruit (Tongdee et al., 1990; Booncherm and Siriphanich, 1991) belonging to the family *Bombacaceae* (Martin, 1980) that grows in the warm, wet conditions of the equatorial tropics. The durian cultivars grown commercially in South East Asian countries are derived from *Durio zibethinus* Murray originating in Peninsular Malaysia.

Durian is a large heavy fruit, covered with a thick husk with sharp hexagonal thorns, which makes peeling a difficult task for untrained people. The fruit can be opened easily by cutting at abscission zones, which develop along the suture at the middle of each locule as the fruit ripen (Siriphanich, 1994). The fruit normally contains five locular units with between 1 and 5 pulps per unit. The pulp unit consists of seed which is completely covered by a creamy, white, yellow or golden yellow aril, the edible portion of the fruit (Nanthachai, 1994).

Most recent fresh-cut research has focused on quality retention based upon visual appearance and rapid biochemical analyses. This approach is questionable with respect to the flavour of fruit and vegetables with high water contents. Hence, shelf-life in terms of flavour quality is becoming an active area of research for fresh-cut produce (Beaulieu and Baldwin, 2000). Very often, the development of off-odours is one of the factors limiting the shelf-life of fresh-cut produce, as shown in honey dew melons (Bai et al., 2003), orange (Rocha et al., 1995), and watermelon (Abbey et al., 1998). Cut fruit products rapidly lose their typical flavour, even when stored under refrigerated conditions. Fresh-cut can-

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taloupe melon, reported by Lamikanra and Richard (2002), developed staleness or loss of freshness within a day of refrigerated storage. Inferior tomato flavour was reported after short-term refrigerated storage of ripe tomatoes (Maul et al., 2000).

Booncherm and Siriphanich (1991) reported that fresh-cut durian could be kept longer than its intact form at low temperature, contrary to common practice with other fruit and vegetable produce. The pulp was found to be less susceptible to chilling injury than the husk. This finding and the fact that durian has a high husk to pulp ratio of 2:1, makes storage of durian pulp instead of the whole fruit more promising.

Storage research has shown that matured but unripened whole durian fruit could be stored at 4 °C for only 20 days (Praditdoung, 1986), while the pulp could be stored at 5 °C for up to 8 weeks, with slight chilling injury observed after 4 weeks of storage (Booncherm and Siriphanich, 1991). Other research has shown that durian pulp could be stored at 4 °C for up to 30 days (Salunkhe and Desai, 1984; Praditdoung, 1986), with the main problems encountered being chilling injury at the base of the seed and contamination by fungi. Refrigerated minimally processed durian is very promising since temperatures that cause a slight amount of chilling injury are preferred over temperatures that cause rapid senescence and microbial deterioration (Watada and Qi, 1999).

Work has been carried out on the effect of storage on physicochemical, microbial and sensory qualities of minimally processed durian at 4 °C for 28 days (Voon et al., 2006). Storage of durian pulp at 4 °C effectively retained fruit firmness, maintained the pH at neutral, increased glucose, fructose and sucrose contents, and maintained the organic acid content of the pulp. Chilling also slowed down the growth of microorganisms, but fruit underwent losses in aroma and developed off-odours on day 21, increasing thereafter and rendering the pulp unacceptable in terms of overall aroma on day 28 of storage (Voon et al., 2006). Thus, the objective of this study was to identify changes that occurred in volatile aroma compounds during storage of minimally processed refrigerated durian and their contribution to sensory attributes.

2. Materials and methods

2.1. Preparation of minimally processed durian

Durian fruit (*Durio zibethinus*) cultivar D24 used in this study were obtained from a farm in Bentong, Pahang Darul Makmur, Malaysia, in mid August 2005. Ripened durian fruit that dropped naturally were collected and transported within 2 h on the same morning (at 30 ± 2 °C). Fruit were selected for uniformity of size and freedom of visual defects, and were dehusked (the rind was cut open), by cutting along the suture on the back of the locules. Upon cutting, 4–6 separated fruit arils (350–420 g) were packed together. Care was taken not to break the epidermis of the pulp when removing it from the husk. The pulps were placed on polystyrene trays and wrapped in a commercially available low-density polyethylene (LDPE) cling film (20 μm) with an oxygen transmission rate of 4.86×10^{-6} nmol s^{-1} mm^{-2} Pa^{-1} at 25 °C and 75% RH. Dehusking was performed manually in an air-conditioned room (20 °C) using good manufacturing practices. Only pulps with no external injuries (epidermis intact) were selected. Each replicate was composed of pulps taken from three trays and all data are the mean of three replicates. Samples were packaged at 25 °C and stored at 4 °C for 42 days. Flavour volatile compounds and sensory properties of the fruit were evaluated initially and at 7-day intervals.

2.2. Chemicals

Fifteen volatile flavour compounds (acetaldehyde, ethyl acetate, 1-propanethiol, methyl propionate, ethanol, methyl butanoate, methyl 2-methylbutanoate, ethyl butanoate, propyl propanoate, diethyl disulfide, ethyl propanoate, ethyl 2-methylbutanoate, ethyl 3-methylbutanoate, propyl butanoate, 1-butanol, and thiophene, as authentic GC standards with purity ≥98%, were purchased from Sigma–Aldrich Company Ltd. (Milwaukee, WI), while sodium chloride was purchased from Merck (Darmstadt, Germany).

2.3. 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 previously to be suitable for extracting durian volatiles (Chin et al., 2007). The fiber was conditioned prior to use according to the supplier's recommendations, for 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 a 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 with stirring. Desorption of the analytes from the fiber coating was made at the GC injection port at 250 °C for 5 min. Each analytical sample was measured in triplicate.

2.4. Gas chromatography–time of flight mass spectrometry (GC/TOFMS) conditions

An Agilent 6890 N gas chromatography system (Wilmington, DE) equipped with electron ionisation–time-of-flight mass spectrometer (Pegasus III, Leco Corp., St. Joseph, MI, USA) was used. Volatile compounds were separated in a 10 m × 0.10 mm, 0.10-μm film thickness Supelcowax-10 (Supelco MA) capillary column with the injector and detector maintained at 250 °C. The injection port was operated at a 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 were collected at a rate of 60 spectra/s. Data were analyzed using the LECO deconvolution software (ChromaTOF 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 of more than 800 were chosen. When available, confirmation of the identity of 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 an internal standard to the samples. The results were expressed as peak area/internal standard (IS) area × 1000.

2.5. Sensory evaluation

A panel of 12 assessors (4 females, 8 males) evaluated fresh durian pulp as well as samples stored for 7, 14, 21 and 28 days using quantitative descriptive analysis (QDA) according to Stone et al. (1974). Acceptability of each attribute was evaluated at the same time based on a nine-point hedonic scale (results not shown). The analysis was terminated at day 28 due to unacceptable durian flavour. Subjects were screened and selected (two sessions) for their sensory ability and trained for descriptive analysis according to the guidelines in ISO 8586-2 (1994). The assessors had been previously trained over six sessions to evaluate different durian cultivars (Voon et al., 2007). For this study, a total of three 2 h sessions were used for vocabulary elicitation, definitions and panel training. To generate a descriptive language for fresh and stored durian, panelists were provided with fresh and 28-day samples and were asked to list the sensory characteristics that they considered to be important in describing the samples. Terminologies characterizing sensory attributes were then developed and descriptors with similar meaning were grouped. Selection of similar terms used by the panelists resulted in a consensus list of 16 descriptors with their agreed definitions. The colour of durian fruit ranged from whitish to golden yellow. Eight aroma descriptors were generated, namely sweet, fruity, sulfur, alcohol, nutty, green notes, off-odour and overall aroma intensity. Creamy, sticky and moist sensations were used to describe the texture properties of the fruit, while sweet, bitter, grassy and overall aftertastes were employed to describe the taste of durian fruit. Descriptors for different attributes are summarized in Table 1.

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 16 terms. For each session, five to six trays of durian were removed from the refrigerator and presented to the assessors in randomly numbered capped containers. Samples were prepared and stored every week for 4 consecutive weeks in order to obtain samples of different storage periods on the same day. Products were evaluated in duplicate over four different sessions. Two or three samples from different storage periods were assessed at each session. In the first two sessions, fresh samples and samples stored for 7 and 14 days were evaluated. Samples stored for 21 and 28 days were evaluated in the last two sessions. 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. Expectoration of the sample was allowed. Quantification of results was obtained by measuring the distance from zero to the vertical line.

Table 1
Sensory attributes used for quantitative descriptive analysis of minimally pocessed durian

Attributes	Definition
Appearance	
Colour	The colour of durian surface ranging from white to golden yellow
Odour	Ranging from none to strong
Sweet aroma	Aroma associated with sweet candy or cotton candy
Fruit aroma	Displaying aroma and flavours suggestive of fruit
Sulphur aroma	Aroma associated with sulphur, onion like
Alcohol aroma	Aroma associated with alcohol or fermented odour
Green aroma	Aroma associated with odours reminiscent of fresh green grass or herbs, green beans or unripe fruit
Nutty aroma	The non-specific nut odour that is characteristics of several different nuts, e.g. peanuts, hazelnuts, pecans, almonds
Overall aroma	Overall intensity of aroma
Off-odour	Any undesirable aroma that present including musty, sweat and other aroma with revolting characteristic
Flavour	
Sweetness	Fundamental taste sensation of which sucrose is typical
Bitterness	Taste sensation of which caffeine and quinine are typical
Grassiness	Green flavour associated with green grass or cucumber
Texture	
Creaminess	The extent to which the sample resembles cream-ranges from none to strong
Stickiness	Amount of product that adheres to oral surfaces, ranging from not sticky to very sticky
Moist	The perceived moisture content of the durian. Ranging from very dry to very moist
Overall aftertaste	The effect produced after the sample as swallowed, ranging from none to strong

2.6. Statistical analysis

The data obtained from the HS-SPME–GC–TOFMS analyses and the sensory data were analyzed using analysis of variance (ANOVA). One-way ANOVA was performed using the Minitab statistical software (Version 13.32, USA). Principle component analysis (PCA) was carried out using the UNSCRAMBLER software (Version 7.6, CAMO A/S, Trondheim, Norway) on sensory attributes and flavour volatile compounds that exhibited significant difference ($P < 0.05$) in the ANOVA to reduce the data set and study the interrelation among the different attributes. In order to understand the relationship between the headspace flavour profile and descriptive sensory profile, PLS2 were performed using the UNSCRAMBLER software (Version 7.6, CAMO A/S, Trondheim, Norway). Volatile flavour compound concentrations were assigned as *x*-variables and the sensory data set was designated as the *y*-variables in PLS2 analysis under the assumption that the volatile flavour profile mainly affects the sensory perception of durian. The Pearson correlation coefficients between sensory and instrumental data were calculated using the Minitab statistical software (Version 13.32, USA).

3. Results and discussion

3.1. Changes in volatile compounds during storage

Table 2 presents the volatile composition of the headspace of minimally processed durian cultivar D24 during storage at 4 °C. Forty volatile flavour compounds from the headspace of durian were detected by GC–TOFMS, of which 15 were identified. The volatiles detected included 15 esters, 12 sulfur compounds, 9 alcohols, 3 aldehydes and 1 ketone. All compounds detected were identical with those from previous studies (Baldry et al., 1972; Wong and Tie, 1995; Naf and Velluz, 1996; Chin et al., 2007). The predominant volatile compounds in cultivar D24 were sulfur compounds (54.6%) followed by ester compounds (33.2%) and alcohols (8.2%).

In this study, the sulfur compounds in durian mainly comprised diethyl disulfide and diethyl trisulfide, as reported by Naf and Velluz (1996). As for ester compounds, the major contributors were ethyl propanoate (43.7%), followed by ethyl 2-methylbutanoate (9.4%) and propyl 2-methylbutanoate (9.1%). Likewise, the first two ester compounds have been reported by Wong and Tie (1995), as quantitatively the most important esters in durian. Among the ester compounds, ethyl 2-methylbutanoate was reported to have the highest odour impact among the non-sulfurous odorants in durian (Weenen et al., 1996). The alcohol compounds present in durian were almost solely comprised of ethanol (78%). Other compounds that were detected included acetaldehyde, propanal, 2-methylbut-2-enal and 3-hydroxybutan-2-one, which accounted for 4.0% of the total volatile compounds detected.

Storage of durian pulp for 1 week at 4 °C resulted in a sharp decrease (53%) in the total volatile compounds (Table 2), with the loss of 77.3% esters in the fruit. Five out of 15 esters were totally depleted after 7 days of storage, namely methyl butanoate, methyl 2-methylbutanoate, ethyl butanoate, ethyl 3-methylbutanoate, and propyl butanoate. Other ester compounds decreased gradually throughout storage to undetectable levels after 21 days of storage. Lamikanra and Richard (2002) reported that the reduction of esters was an important early reaction step in the loss of freshness during storage of fresh-cut cantaloupe. Similarly, short-term storage of ripe tomatoes at 3–5 °C was reported to result in inferior tomato flavour due to a reduction in important aroma volatiles (Maul et al., 2000). Esters were most probably hydrolyzed to their corresponding alcohols and acids during storage. However, it was noteworthy that fruity and sweet notes could still be perceived by the panelists even when ester concentrations were not detectable using SPME–GC–TOFMS, as shown in Fig. 1. Aldehyde compounds that included 2-methylbut-2-enal, acetaldehyde and propanal decreased significantly ($P \leq 0.05$) after 7, 14 and 21 days of storage, respectively. These aldehyde compounds may account for the fruity note in durian that was comparable to fresh durian even when most of the other ester compounds were not detected after 1 week of storage (Fig. 1).

There were no significant changes in total sulfur compounds in this study. Methyl ethyl disulfide, diethyl disulfide, and ethyl propyl disulfide were found to be the most stable sulfur compounds, and concentrations of these compounds were main-

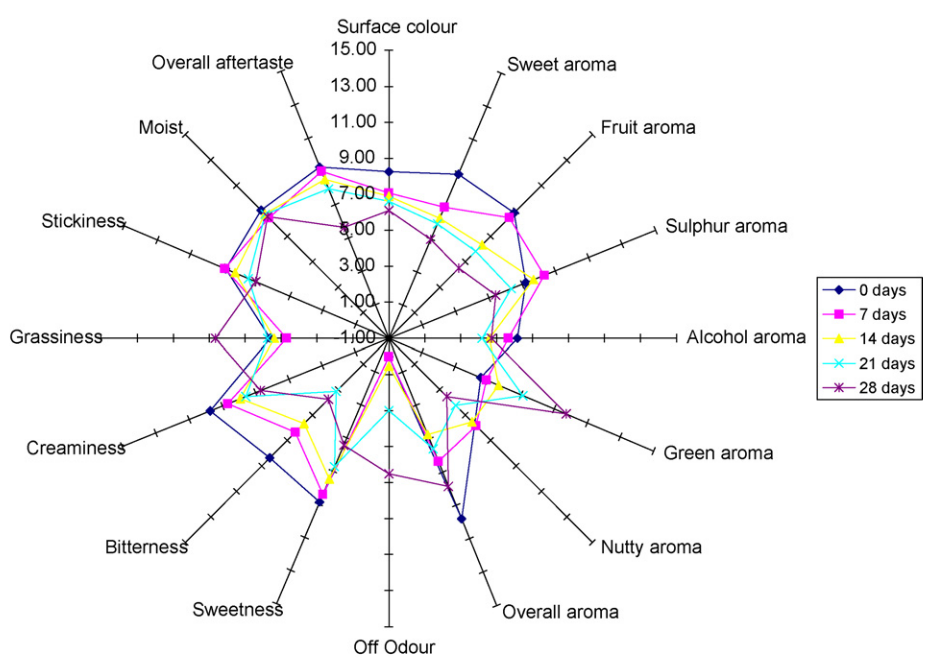

Fig. 1. Sensory profiles of minimally processed durian during storage at 4 °C over 28 days presented as a spider diagram.

Table 2
Relative concentration of aroma volatiles in fresh and stored minimally processed durian (ng/g)

Peak no.	Flavour compound	Storage period (days)*						
		0	7	14	21	28	35	42
Aldehyde								
1	Acetaldehyde	204.7^{a}	289.5^{a}	59.9^{b}	51.1^{b}	55.7^{b}	60.0^{b}	38.0^{b}
3	Propanal	53.7^{ab}	37.4^{ab}	105.3^{a}	n.d.	10.3^{b}	6.8^{b}	n.d.
18	2-Methylbut-2-enal	127.5^{a}	70.8^{b}	7.77^{c}	n.d.	n.d.	n.d.	n.d.
Total		385.8^{a}	397.7^{a}	172.9^{b}	51.1^{bc}	66.1^{bc}	66.8^{bc}	38.0^{c}
Ketone								
30	3-Hydroxybutan-2-one	48.4^{ab}	58.0^{ab}	10.31^{a}	41.7^{ab}	n.d.	14.6^{b}	19.0^{b}
Alcohol								
7	Ethanol	697.6^{a}	644.2^{b}	739.1^{a}	639.9^{a}	393.7^{a}	839.8^{a}	551.8^{a}
13	1-Propanol	n.d.	85.1^{b}	236.6^{ab}	138.0^{b}	157.1^{ab}	315.7^{a}	133.4^{b}
24	1-Butanol	20.0^{a}	11.9^{a}	53.6^{a}	61.2^{a}	58.2^{a}	22.8^{a}	22.2^{a}
27	2-Methylbutan-1-ol	106.4^{b}	134.0^{b}	585.1^{a}	188.7^{b}	162.3^{b}	106.4^{b}	187.4^{b}
28	3-Methylbutan-1-ol	55.3^{a}	17.2^{a}	49.3^{a}	21.1^{a}	8.4^{a}	49.0^{a}	56.4^{a}
31	1-Hexanol	n.d.	n.d.	n.d.	2.6^{a}	9.5^{a}	6.7^{a}	18.0^{a}
33	1-Heptanol	n.d.	6.9^{bc}	59.1^{a}	31.2^{b}	5.9^{c}	n.d.	n.d.
36	Butane-2,3-diol	n.d.	n.d.	30.5^{b}	54.2^{ab}	10.7^{b}	22.9^{b}	90.4^{a}
40	Benzyl alcohol	n.d.	n.d.	n.d.	n.d.	29.8^{c}	102.5^{b}	327.3^{a}
Total		883.4^{a}	899.2^{a}	1753.4^{a}	1137.0^{a}	835.4^{a}	1465.8^{a}	1386.9^{a}
Sulphur containing compound								
2	Ethanethiol	759.9^{a}	45.1^{b}	53.4^{b}	n.d.	n.d.	n.d.	n.d.
4	1-Propanethiol	330.9^{a}	69.9^{b}	60.1^{b}	n.d.	n.d.	n.d.	n.d.
20	Methyl ethyl disulfide	182.1^{a}	161.6^{a}	128.8^{a}	152.0^{a}	133.9^{a}	179.6^{a}	76.5^{a}
25	Diethyl disulfide	2130.6^{a}	1470.8^{a}	1228.3^{a}	1966.0^{a}	1494.4^{a}	2868.7^{a}	1158.4^{a}
26	Methyl propyl disulfide	n.d.	15.6^{ab}	55.0^{ab}	104.7^{a}	58.3^{ab}	103.5^{a}	46.2^{ab}
29	Ethyl propyl disulfide	447.4^{a}	459.7^{a}	707.2^{a}	1002.5^{a}	717.2^{a}	1040.5^{a}	709.7^{a}
32	Dipropyl disulfide	n.d.	n.d.	116.8^{b}	220.3^{a}	115.7^{b}	118.5^{b}	66.5^{bc}
34	Diethyl trisulfide	1183.1^{a}	417.6^{b}	1321.5^{a}	332.2^{b}	302.6^{b}	382.1^{b}	523.2^{b}
35	3,5-Dimethyl-1,2,4-Trithiolane (isomer 1)	371.2^{a}	86.2^{b}	127.6^{b}	114.4^{b}	70.1^{b}	163.4^{ab}	54.3^{b}
37	3,5-Dimethyl-1,2,4-Trithiolane (isomer 2)	398.7^{a}	89.8^{b}	125.4^{b}	124.4^{b}	70.0^{b}	154.0^{b}	89.0^{b}
38	Dipropyl trisulfide	36.5^{b}	29.0^{b}	80.2^{a}	32.4^{b}	34.0^{b}	52.6^{ab}	23.6^{b}
39	1,1-Bis(ethylthio)-ethane	64.27^{b}	40.8^{b}	117.7^{ab}	205.9^{a}	111.5^{ab}	222.1^{a}	129.9^{ab}
Total		5904^{a}	2886^{a}	4122^{a}	4255^{a}	3108^{a}	5285^{a}	2877^{a}
Esters								
5	Ethyl acetate	38.6^{a}	23.8^{ab}	9.43^{b}	1.53^{b}	n.d.	n.d.	n.d.
6	Methyl propionate	202.0^{a}	42.0^{b}	n.d.	n.d.	7.2^{c}	11.1^{bc}	n.d.
8	Ethyl propanoate	1570.5^{a}	422.1^{b}	n.d.	57.7^{c}	3.8^{c}	n.d.	n.d.
9	Ethyl 2-methylpropanoate	212.0^{a}	30.9^{b}	n.d.	n.d.	n.d.	n.d.	n.d.
10	Methyl butanoate	57.4^{a}	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
11	Methyl 2-methylbutanoate	129.33^{a}	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
12	Ethyl butanoate	76.6^{a}	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
14	Propyl propanoate	213.2^{a}	17.0^{b}	n.d.	n.d.	n.d.	n.d.	n.d.
15	Ethyl 2-methyl butanoate	339.3^{a}	31.9^{b}	n.d.	n.d.	n.d.	n.d.	n.d.
16	Propyl 2-methylpropanoate	136.3^{a}	42.4^{b}	12.8^{bc}	n.d.	n.d.	n.d.	n.d.
17	Ethyl 3-methylbutanoate	76.4^{a}	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
19	Propyl butanoate	50.7^{a}	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
21	Propyl 2-methylbutanoate	327.9^{a}	162.7^{b}	65.5^{bc}	55.6^{bc}	29.8^{c}	16.0^{c}	n.d.
22	Propyl 3-methylbutanoate	107.5^{a}	42.4^{b}	36.5^{b}	n.d.	n.d.	n.d.	n.d.
23	Ethyl but-2-enoate	53.1^{a}	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Total		3590.8^{a}	815.0^{b}	124.3^{c}	114.8^{c}	40.8^{c}	27.1^{c}	n.d.

n.d.: not detected.
* Values in the same row with the same letters are not significantly different (level of significance 5%).

tained throughout 42 days of storage at 4 °C. Meanwhile, sulfur compounds that included ethanethiol, 1-propanethiol, diethyl trisulfide, and both isomers of 3,5-dimethyl-1,2,4-trithiolane, decreased significantly ($P \leq 0.05$) after 7 days of storage. Although the concentration of ethanethiol and 1-propanethiol further decreased to an undetectable level on day 21 of storage, the two isomers of 3,5-dimethyl-1,2,4-trithiolane did not change significantly from day 7 onwards. Methyl propyl disulfide and 1,1-bis(ethylthio)-ethane increased significantly after 21 days of storage and remained unchanged thereafter. The concentration of other sulfur compounds, namely, dipropyl disulfide and dipropyl trisulfide increased before decreasing again during storage. The initial increase may be attributed to the biosynthesis of these sulfur compounds, which continued to take place in durian during refrigerated storage.

Ethanol and 1-butanol initially present in durian did not change significantly throughout 42 days of storage. One-propanol that was initially not detected, increased significantly ($P \leq 0.05$) to 85.1 ng/g after 7 days of storage and further increased thereafter, reaching a maximum relative concentration (315.7 ng/g) on day 35 of storage. Other alcohol compounds that were not detected in fresh durian, namely 1-heptanol, butane-2,3-diol and benzyl alcohol, were detected after 7, 14 and 28 days of storage, respectively. All alcohol compounds were identical to those found in a previous study (Wong and Tie, 1995), except for benzyl alcohol, which is for the first time reported in durian fruit. This compound was probably produced only after prolonged refrigerated storage of durian pulp. Benzyl alcohol was detected in the headspace of durian after 28 days of storage at 4 °C and increased drastically thereafter. Correspondingly, some of the panelists described durian pulp as having an aroma reminiscent of fragrant or essential oil when stored for 21 days, which in time became more intense after the fruit was subjected to 28 days storage. Benzyl alcohol has been previously reported in other fruit such as papaya (Almora et al., 2004), grapes (Rosillo et al., 1999), and passionfruit (Chassagne et al., 1996). This compound was reported to have a pleasant fruity odour and a slight pungent sweet taste (Burdock, 2005) and provided satisfactory antimicrobial efficacy against bacteria and fungi at 0.5% concentration (Gupta and Kaisheva, 2003). Alcohols typically make a minor contribution to flavour unless present in relatively high concentration (ppm) or if they are unsaturated (Heath and Reineccius, 1986). This may explain why alcohol compounds present in this study did not correlate well with the intensity of perceived alcohol aromas (Table 3). The alcohol aroma detected by the panelists was most probably due to the presence of some compounds with sharp or pungent notes such as acetaldehyde and propanal as well as compounds with odour reminiscent of brandy or rum such as ethyl acetate, methyl propionate, ethyl propanoate and propyl 2-methylbutanoate, since these compounds were found to be highly correlated with the perceived alcohol note (Table 3). When PCA was carried out on volatile flavour compounds that exhibited significant differences ($P \leq 0.05$), samples from day 21 onwards until day 42 could not be separated well due to the undetectable concentration of most flavour compounds after day 21 of storage. Samples stored for more than 28 days were also no longer acceptable. Therefore, a more meaningful plot was obtained when PCA was performed on samples stored from day 0 until day 28. The PCA biplot of all flavour compounds with significant differences in samples stored at different periods is shown in Figs. 1 and 2. The first two principal components (PCs) explained 86% of the variance in the data. The first PC separated fresh durian (stored 0 days) from samples stored for 14, 21 and 28 days, whereas the second PC separated the day 14 samples from day 21 and 28 samples (Fig. 2). Fresh samples had highly positive loadings across PC1 due to the higher content of all ester compounds, and some sulfur compounds, namely ethanethiol, 1-propanethiol, and both isomers of 3,5-dimethyl 1,2,4-trithiolane. The negative loadings across PC1 of samples stored for 14, 21 and 28 days were attributed to the higher concentrations of methyl propyl disulfide, dipropyl trisulfide, and 3 alcohol compounds (1-propanol, butane-2,3-diol, and 1-heptanol). PC2 separated the day 14 sample from day 21 and 28 samples based on the higher concentrations of propanal, 1-heptanol, diethyl trisulfide and dipropyl trisulfide. The day 21 and 28 samples had negative loadings along PC2 due to the presence of benzyl alcohol. Samples stored for 7 days at low temperature failed to be sepa-

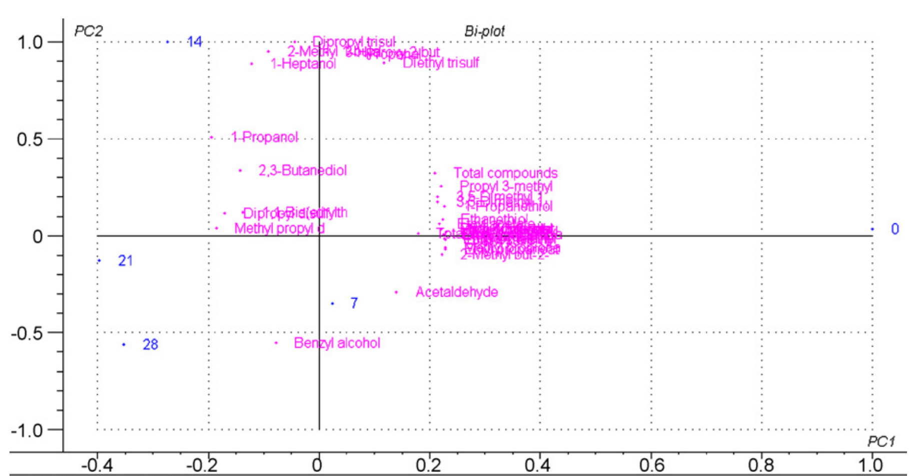

Fig. 2. PCA scores and loadings of volatile aroma compounds of minimally processed durian stored for 0, 7, 14, 21, and 28 days of storage at 4 °C.

Table 3
Correlation coefficients between sensory descriptors and flavour compounds

Variables	Aroma perceived							
	Sweet	Fruit	Sulfur	Alcohol	Green	Nutty	Overall	Off-odour
Acetaldehyde	0.531	**0.729**	0.523	**0.855**	−0.568	0.482	0.331	−0.468
Ethanethiol	0.530	0.430	0.068	**0.723**	−0.365	0.318	0.557	−0.334
Propanal	0.076	0.136	0.469	0.179	−0.433	**0.668**	−0.211	−0.436
1-Propanethiol	**0.747**	0.658	0.230	**0.804**	−0.542	0.538	**0.633**	−0.540
Ethyl acetate	0.573	**0.722**	0.501	**0.900**	**−0.642**	**0.733**	0.397	**−0.648**
Methyl propionate	**0.759**	0.647	0.117	**0.812**	−0.440	0.388	**0.736**	−0.398
Ethyl Propanoate	**0.720**	0.659	0.210	**0.838**	−0.478	0.408	**0.617**	−0.439
Ethyl 2-methylpropanoate	**0.804**	**0.657**	0.141	**0.721**	−0.446	0.371	**0.673**	−0.404
Methy-butanoate	**0.733**	0.562	0.058	**0.720**	−0.400	0.326	**0.683**	−0.354
Methyl 2-methybutanoate	**0.679**	0.524	0.050	**0.738**	−0.388	0.318	**0.663**	−0.342
Ethyl butanoate	**0.665**	0.515	0.048	**0.739**	−0.385	0.316	**0.657**	−0.339
1-Popanol	**−0.819**	**−0.757**	−0.243	**−0.708**	0.545	−0.089	−0.436	0.267
Propyl propanoate	**0.650**	0.535	0.078	**0.780**	−0.405	0.343	**0.654**	−0.362
Ethyl 2-methylbutanoate	**0.697**	0.568	0.106	**0.775**	−0.420	0.349	**0.648**	−0.374
Propyl 2-methylpropanoate	**0.664**	**0.635**	0.244	**0.835**	−0.485	0.559	**0.627**	−0.519
Ethyl 3-methylbutanoate	**0.723**	0.555	0.057	**0.726**	−0.399	0.325	**0.680**	0.352
2-Methyl but-2-enal	**0.721**	**0.771**	0.482	**0.791**	−0.545	0.567	0.428	−0.566
Propyl butanoate	**0.680**	0.525	0.050	**0.738**	−0.389	0.319	**0.664**	−0.342
Propyl 2-methylbutanoate	**0.876**	**0.791**	0.358	**0.724**	−0.561	0.430	0.509	−0.497
Propyl 3-methylbutanoate	**0.836**	**0.769**	0.372	**0.769**	**−0.644**	0.564	0.541	**−0.664**
Methyl propyl disulfide	−0.372	−0.281	−0.263	−0.524	0.364	−0.325	−0.346	0.235
2-Methyl butan-1-ol	−0.261	−0.308	0.206	−0.416	−0.013	0.270	−0.427	−0.250
3-Hydoxybutan-2-one	0.160	0.272	**0.664**	0.049	−0.481	**0.701**	−0.356	**−0.717**
Dipropyl disulfide	**−0.607**	**−0.774**	−0.458	**−0.787**	0.457	**−0.615**	−0.510	**0.632**
1-Heptanol	−0.298	−0.314	0.248	−0.556	−0.038	0.227	**−0.619**	−0.253
Diethyl trisulfide	0.380	0.282	0.366	0.148	−0.403	0.589	0.037	**−0.628**
3,5-Dimethy-1,2,4-trithiolane (isomer 1)	**0.651**	0.489	0.160	**0.665**	−0.455	0.414	0.495	−0.410
Butane-2,3-diol	−0.487	−0.492	−0.075	−0.449	0.120	−0.087	−0.570	0.244
3,5-Dimethy-1,2,4-trithiolane (isomer 2)	0.588	0.436	0.132	**0.651**	−0.438	0.381	0.473	−0.391
Dipropyl trisulfide	−0.246	−0.194	0.204	−0.089	−0.094	0.396	−0.335	−0.161
1-1-Bis(ethylthio)- ethane	−0.470	**−0.632**	−0.576	**−0.635**	0.567	**−0.684**	−0.233	0.492
Benzyl alcohol	−0.534	**−0.614**	−0.683	−0.166	**0.756**	−0.700	0.208	**0.873**
1-Hexanol	−0.513	−0.568	**−0.702**	−0.280	**0.809**	−0.667	0.228	**0.849**
Total aldehyde	**0.608**	**0.778**	**0.662**	**0.885**	**−0.699**	**0.726**	0.286	**−0.638**
Total esters	**0.756**	**0.661**	0.189	**0.820**	−0.482	0.411	**0.646**	−0.441
Total alcohol	−0.419	−0.365	0.023	−0.271	0.043	0.460	−0.158	−0.225

Correlation coefficients with $P \leq 0.05$ are marked in bold.

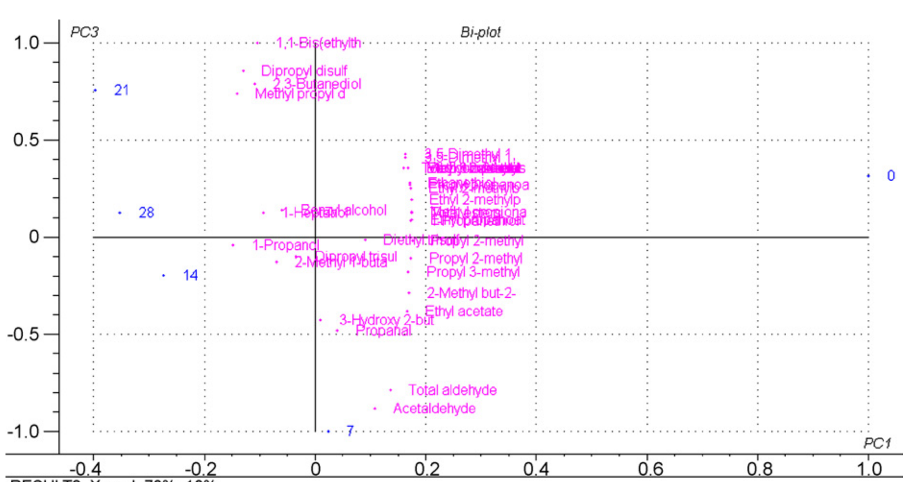

Fig. 3. PCA scores and loadings of volatile aroma compounds of minimally processed durian stored for 0, 7, 14, 21, and 28 days of storage at 4 °C.

rated clearly from other samples by the first two PCs. However, the third PC (Fig. 3) that explained 10% of the variance of data could separate the day 7 from the day 21 sample. The day 7 sample was associated with higher concentrations of acetaldehyde, 2-methylbut-2-enal and total aldehyde content together with propyl 3-methyl butanoate. The day 21 sample was differentiated from the day 7 sample due to the higher concentrations of 3 sulfur compounds (1,1-bis(ethylthio)-ethane, dipropyl trisulfide and methyl propyl disulfide) and 2,3-butanediol present in the former sample. From the PCA, the sample stored for 7 days was not well differentiated from that stored for 14 days, whereas the sample stored for 21 days could not be differentiated from that stored for 28 days. This suggested that fresh durian was very different in terms of volatile flavour profile from the stored samples, while the volatile flavour profiles of samples stored for 7 and 14 days, as well as 21 and 28 days were very similar.

3.2. *Changes in sensory characteristics during storage*

The mean intensities for surface colour, odour, flavour and texture properties of minimally processed durian during storage at 4 °C are given in Fig. 1. Generally, fruity, sulfur, alcohol, and nutty aroma, sweetness, bitterness, and overall aftertaste decreased gradually during storage, concomitant with the increment in green aroma and off-odour. No significant changes in surface colour, sweet and overall aroma, and grassiness were detected throughout the entire storage period. In terms of texture, creaminess decreased gradually during storage while there was no significant change in stickiness and moistness of fruit throughout storage.

After 7 days of storage, no significant changes were detected in all sensory attributes tested except for bitterness of the pulp. A strong reduction in bitterness (23.8%) was detected following 7 days of storage. This bitterness further decreased significantly ($P \leq 0.05$) to 3.15 (37.7% of original bitterness) on day 21 of storage before remaining stable again. This bitter note was most probably attributed to the presence of some amino acids in durian such as alanine, proline, phenylalanine and isoleucine (Zanariah and Noor Rehan, 1987), which have been reported to contribute to bitter taste (Fisher and Scott, 1997).

On day 14 of storage, the perceived alcohol aroma decreased significantly ($P \leq 0.05$) from 6.15 to 4.59 (1.8% per day). Fruity-, sulfur-, and nutty-aromas decreased significantly (25–49%) after 28 days of storage at a rate of ~0.9–1.8% per day. The intensity of green notes increased gradually (~4% per day) throughout the storage period and approached unacceptable levels (113%) on day 21 of storage. This increase in green notes was most probably due to the increase in 1-hexanol, which correlated well with the green aroma (Table 3). On day 28, most panelists detected an undesirable off-odour besides the green note. By now, the fruit was totally unacceptable due to its undesirable odour. As no new volatile aroma compound was detected during storage besides benzyl alcohol, the off-odour was most probably due to the low levels of ester and aldehyde compounds (Table 2), which resulted in a shift in flavour balance during storage. The presence of esters and aldehydes probably played an important role in providing a typically pleasant durian odour when in combination with sulfur, ethanol and acetaldehyde compounds. When sulfur, ethanol or acetaldehyde compounds were perceived individually, they may present an undesirable odour. Some of the sulfur compounds present in durian are reported to give rise to strong objectionable flavours (rubber odour, cooked cabbage, burnt rubber, etc.), even at extremely low concentrations (e.g. methanethiol, ethanethiol, 1,1-bis(ethylthio)-ethane) when present individually. The loss of esters and aldehyde probably unveiled these notes, resulting in durian with an undesirable odour. Although ethanol and acetaldehyde did not result from fermentation in this case, their presence with low levels of ester and aldehyde compounds may also contribute to the undesirable off-odours.

Sulfur aroma decreased significantly ($P \leq 0.05$) after 28 days of storage. Although Weenen et al. (1996) have reported that 3,5-dimethyl-1,2,4-trithiolane was the sulfur compound with the highest odour impact in durians, the concentration remain unchanged despite the significant ($P \leq 0.05$) decrease in sulfur note perceived by the panelists. This suggested that 3,5-dimethyl-1,2,4-trithiolane was not responsible for the decrease in sulfur notes during storage in this study. Meanwhile, alcoholic aroma decreased gradually from day 0 until day 14 of storage and remained stable thereafter (Fig. 1). Overall aftertaste of the fruit decreased gradually reaching a significant level ($P \leq 0.05$) after 28 days of storage (Fig. 1).

Partial least square (PLS2) regression models were run in an attempt to correlate the sensory scores with the chromatographically detected volatile compounds. The x matrix contained the volatile compounds, and the y matrix contained the sensory scores for each attribute. PLSR was unable to sufficiently predict the effect of flavour compounds on sensory attributes of durian flavour due to the drastic loss of flavour compounds during the first 2 weeks of storage. It is thus recommended that sensory and flavour determinations should be carried out between 2 and 3 days in the first 2 weeks of storage to yield a better correlation. The Pearson correlation coefficient was further calculated to examine the correlation between sensory and flavour profile.

3.3. *Correlation of sensory score with flavour profile measurements*

Table 3 shows the correlation between selected sensory attributes and flavour compounds with significant ($P \leq 0.05$) differences among samples stored at different days. Generally, sweet notes correlated strongly with most esters, an aldehyde compound (2-methyl but-2enal), as well as some sulfur compounds. Meanwhile, fruity notes also correlated well with some esters and aldehyde. Esters contributed to the sweet note to a greater extent than aldehyde, while aldehyde contributed more to the fruity note. The fruity note perceived by panelists remained the same after 7 days of storage as compared to the fresh fruit, and this could be attributed to the aldehyde content that remained high, although most of the other ester compounds had decreased or dissipated totally after 7 days of storage (Fig. 1, Table 2). The decreased sweet notes perceived after 7 days of storage were clearly attributed to the loss of esters. Ethyl 2-methylbutanoate, which was found to have the highest odour impact among the non-sulfurous odourants in durian (Weenen et al., 1996), correlated strongly with sweet and alcohol notes, together with overall aroma intensity.

3,5-Dimethyl-1,2,4-trithiolane did not correlate well with the intensity of sulfur notes in this study although it was reported to contribute to a strong durian note (sulfur note) by Weenen et al. (1996). This was further supported in the sensory study (Fig. 1), where panelists continued to perceive a drop in sulfur notes despite no change in the concentration of 3,5-dimethyl-1,2,4-trithiolane from 7 days of storage onwards. This was possibly due to the presence of esters and aldehydes that exerted a synergistic effect on the intensity of sulfur notes. Strong synergistic effects between unrelated volatiles have been suggested in a previous study on bamboo shoot (Fu et al., 2002). The effect of esters and aldehyde present on the sulfur notes warrants further investigation.

The perceived alcohol note correlated strongly with some aldehyde, ester as well as some sulfur compounds but not with any alcohol compounds (Table 3). The alcohol note was probably associated with the pungent note and brandy or rum odour as they correlated strongly with the compounds responsible for these notes (acetaldehyde, ethyl acetate, methyl propionate, ethyl propanoate and propyl 2-methylbutanoate) (Table 3).

The green note correlated well with the concentrations of 1-hexanol and benzyl alcohol. However, 1-hexanol was most probably the compound responsible for the green note as it has been reported to contribute to a green, fruity odour (Burdock, 2005). The green note, which was undesirable when present at a high intensity, seemed to be masked by the presence of ethyl acetate and propyl-3-methyl butanoate as these compounds correlated negatively with green notes (Table 3).

Although no compound directly responsible for a nutty note was discovered in this study, the perceived nutty note seemed to correlate well with propanal, ethyl acetate, and total aldehyde. Overall aroma intensity correlated strongly with the ester compounds together with 1-propanethiol, while off-odour correlated well with dipropyl disulfide, benzyl alcohol and 1-hexanol.

4. Conclusion

During storage of minimally processed durian at 4 °C, the main changes in volatile flavour profile involved the loss of most ester compounds after 14 days of storage. This result correlated well with the decrease in intensities of fruity aroma perceived by panelists during storage. Total sulfur content of the fruit remained unchanged after 42 days of storage, although the sulfur notes perceived by the panelists decreased significantly ($P \leq 0.05$) at the end of storage. Sensory analyses revealed that the fruit could be stored for 21 days, after which the green aroma became too intense and rendered the fruit unacceptable. Correlation analysis suggested that sweet and fruity aroma correlated strongly with some ester and aldehyde compounds while sulphur aroma did not correlate well with any sulphur compounds detected.

Acknowledgements

Financial support (Grant 03-02-04-0159-EA001) from the Ministry of Science, Technology and Innovation, Malaysia is fully appreciated. Special thanks are due to the 12 panelists for their participation in this study.

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