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FULL PAPER

Determination of shelf-life using accelerated shelf-life testing (ASLT) method and characterization of the flavour components of freeze-dried durian (*Durio zibethinus*) products

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Abstract

Durian is a unique tropical fruit that has a strong smell and distinctive taste. It is a seasonal fruit and has a few days shelf-life. Freeze drying is known for preserving foods while maintaining its original shape and provide excellent rehydrated products. This study aimed to determine the shelf-life of freeze-dried (FD) durian products using the accelerated shelf-life testing (ASLT) method and to assess the flavour changes in fresh and freeze-dried durian products. The parameters used to determine shelf-life were moisture content and L* a* b* colour values of FD durian products for 28 days of storage at 30, 40, and 50°C. Flavour analysis using Solid Phase Microextraction (SPME) and Gas Chromatography-Mass Spectrometry (GCMS) was carried out on fresh durian pulp, FD durian for 30 hrs, and FD durian for 36 hrs. The estimation of shelf-life of FD durian products at storage temperatures of 25 and 30°C, respectively, were based on the following parameters: (1) moisture content: 41 and 37 days, (2) L*(brightness): 467 and 311 days, (3) a* (redness): 144 and 171 days, and (4) b*(yellowness): 43 and 46 days. A total of twenty-four volatile compounds contributed to the flavour of fresh durian fruit and five of them had concentrations of more than 10 ppm. The losses percentage of these five volatile compounds were in a range of 78-95% (FD durian for 30 hrs) and a range of 0-100% (FD durian for 36 hrs). Freeze-drying technique on durian was able to extend shelf-life and preserve flavour compounds.

1. Introduction

Durian (*Durio zibethinus*) family Bombacacea is a seasonal tropical fruit from Southeast Asia (Malaysia, Thailand, Philippines and Indonesia (Ho and Bhat, 2015). Ripe durian fruit has a distinctive aroma, and taste, therefore it is called the 'king fruit' (Ho and Bhat, 2015). Durian fruit requires some form of handling process before consumption. For example, separating the edible (fleshy) part from the non-edible portion, separating the adherent waxy layer. Durian also requires separating the pulp from the skin, which is similar to jackfruit. Some studies reported that exotic fruits such as durian contain bioactive components/phytochemicals (Gorinstein *et al.*, 2011; Ho and Bhat, 2015). Durian fruit is also rich in micro and macronutrients, contains essential minerals and vitamins A, C, and E (Ho and Bhat, 2015; Charoenkiatkul *et al.*, 2016).

As a prevalent fruit in Southeast Asia, durian is commonly consumed in the form of fresh pulp. The market of durian fruit is very limited in the sense that it depends on the fruit harvest season and the constraints on marketing fresh fruit are also very limited due to the durian's shelf-life of 2-5 days at ambient temperature (Chin *et al.*, 2008). Therefore, the processing of fresh durian fruit into dry whole fruit is more necessary because drying of the fruit yields several advantages including reduced transportation costs, practical consumption, and product stability against microorganisms and adverse biochemical reactions (Chin *et al.*, 2010).

Above all types of drying methods for agricultural products that have been engineered up to date, freeze-drying is a drying method that is widely applied in the food industry where several drying principles are used (Chin *et al.*, 2008). Drying agricultural products through freeze-drying techniques uses the ice sublimation mechanism under vacuum conditions. Freeze drying can slow down the deterioration of the product by

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minimization of loss of flavour and aroma, maximization of nutrient retention because of the low temperature involved in the process (Ratti, 2013).

The freeze-drying method for durian fruit into powder/durian powder has been carried out by several researchers (Chin *et al.*, 2008; Chin *et al.*, 2010) but the application of freeze-drying to produce dried durian pulp has not been carried out by other researchers. An important parameter that needs to be determined in the production of freeze-drying durian is the possibility of reducing volatile durian flavour compounds due to the effect of drying (Chin *et al.*, 2010). The difference in the aroma characteristics of several varieties of durian is a key factor in consumer acceptance (Belgis *et al.*, 2017).

The quality of freeze-dried durian is also governed by its shelf-life. Shelf-life can be determined by observing the product during storage until changes occur that are no longer acceptable to consumers (Asiah *et al*., 2018). Determination of shelf-life using the ASLT (Accelerated Shelf-life Testing) method for pineapple, papaya, cempedak, and shallot products has been studied in other studies (Arif, 2016; Setyadjit *et al*., 2017). To the best of our knowledge, the determination of the shelf-life of freeze-dried durian products has not been performed by other researchers.

In the light of the above, the objective of this study was to determine the shelf-life of freeze-dried durian using the ASLT method and to assess changes in the flavour of freeze-drying durian products due to the drying process applied.

2. Materials and methods

2.1 Chemical and standards

The chemicals used are NaCl (Merck, Germany), Alkenes (C8-C28) and 1,4-dichlorobenzene (Aldrich, Germany) as standard internal solutions. Standard flavour solutions (maltol, ethyl-2-butyrate, diethyl disulphide, hexanal, acetyl-6-pyridine, and 2-methyl-butanol) were obtained from PT. Mane Flavour and Fragrance, Jakarta, Indonesia

2.2 Preparation of frozen durian

Durian was selected from local market in Cianjur, West Java, and the pulp was then separated from the seeds for crushing using a food processor (Russel Hobs, UK) at 300 rpm for 5 mins into durian pulp. Durian pulp was removed into a plastic food container until the thickness level of 2 cm and was formed into a 3 cm^2 square. The durian pulp was then placed into a commercial freezer (GEA, Indonesia) at -40°C for 24 hrs.

2.3 Freeze-drying condition

The freeze-drying process followed the methods of Ceballos *et al.* (2012) and Darniadi *et al.* (2019) with slight modifications. The frozen durian pulp samples were transferred to a stainless-steel pan, then placed into a Freeze-Dryer (Buchi Lyovapor L-200, Switzerland) at -55°C, vacuum pressure 0.04 m bar for 30 and 36 hrs. The dried durian samples were removed from the freeze-dryer and packed in aluminum foil for analysis.

2.4 Moisture content

A HB-120 Halogen Moisture Analyzer (Mettler Toledo, UK) set at 105°C was used to measure the moisture content of FD durian. A total of 1 g of freeze-drying durian powder sample was placed on the sample pan, then the lid of the moisture analyzer was lowered until it was tightly closed. The drying time ranged from 2 to 3 mins for each sample.

2.5 Colour measurement

Colour measurement followed the method by Faridah *et al.* (2013). The colour value of FD durian was measured using a Chromameter (Konica Minolta) which produced the Hunter L* (brightness), a* (redness), and b* (yellowness) values. Where L*: Value (+) = light colour, (-) = dark colour; a*: Value (+) = red, (-) = green and b*: Value (+) = yellow, (-) = blue

2.6 Accelerated shelf-life testing of FD durian

ASLT method was done according to Arif (2016). FD durian for 36 hrs was chosen for accelerated shelf-life testing. The samples packed in aluminium foil were placed in the storage incubator at 30, 40, and 50°C. A set of sampling was done every 7 days, from day 0 to 28. The physicochemical properties data obtained were plotted against time, then the linear regression equation was calculated for the three acceleration temperatures, using the equation Y = a + bx, where Y = Physical properties of FD durian, x = storage time (days), b or k = rate of change. The value of b is also called the slope, which is the rate of deterioration. Zero order is determined by relating between the physical value (At) and the storage time (t), while the first order is calculated by relating ln value (ln At) with storage time (t). The degradation of the zero-order is expressed as the following equation:

$$At - A_0 = -kt \quad (1)$$

Where At = Amount A at time t, A_0 = The initial number A, k = rate of deterioration and t = storage time

The equation for quality degradation with time first order is formulated by the equation:

$\ln At - \ln A_0 = -kt$ (2)

After obtaining the slope, intercept, and R^2 (zero order and first order), then the equation that has the largest R^2 was chosen for calculating the product shelf-life using the Arrhenius Model:

$\ln k = \ln k_0 - (Ea/R)(1/T)$ (3)

Where $\ln k_0$ = intercept, Ea/R = slope, Ea = activation energy, R = ideal gas constant = 1.986 cal/mol °K and T = temperature (°K)

The relationship between the ln k and (1/T) was formed as a graph. The K_0 and the Ea were obtained from the graph. The key parameters were determined from the smallest activation energy (Ea). The shelf-life of the product was estimated by calculating the difference between the initial rate (A_0) and the value the rate at a certain time (At) using the following equation:

$ts = [\ln (N_o\text{-}Nt)]/kT$ for first order (4)

$ts = (N_0\text{-}Nt)/kT$ for for zero order (5)

Where ts = storage time, N_0 = the initial rate at t_0, Nt = the rate after storage time t (critical limit) and KT = k at storage temperature T

2.7 Flavour analysis

2.7.1 Solid-phase micro extraction (SPME)

SPME was carried out according to Zhang *et al.* (2007). A total of 50 g of fresh durian pulp and FD durian were homogenized with 100 mL of NaCl solution (0.08 g/mL) and stirred for 1 min. Following this, 12.5 mL homogenates were put into a 22 mL vial with the top cap number 2713 and put into the vial the magnetic stirrer (p × d = 10 mm × 3 mm). Next, 0.5 µL of internal standard (IS) 1,4-dichlorobenzene 0.01% (w/v) was spiked into the sample and the vial was tightly closed. SPME fiber syringe (divinylbenzene-carboxen-polydimethylsiloxane 85 µm, Supelco Inc., PA, USA) was manually inserted to the vial headspace at 30°C for 30 minutes and injected into GC-MS.

2.7.2 Gas chromatography-mass spectrometry (GC-MS)

GC-MS analysis was conducted using GC-MS Agilent 7890A-5975C based on the method of Belgis *et al.*, (2017). Chromatographic separation was performed using an HP-INNOWAX column (30m × 0.25 mm i.d. 0.25 µm film thickness) with the following equipment conditions: helium as carrier gas at a constant flow of 1.0 mL/min, P: 60 kPa; electron ionization voltage 70 eV; injector temperature 250°C; oven initial temperature to 40°C and increased to 80°C in increments of 3°C/min., hold for 1 min, increased to 130°C in increments of 2°C/min, hold for 2 mins, increased to 240°C in increments of 6°C/min, mass scanning to charge ratio 33-400 m/z. The identification of volatile compounds was based on comparison of the mass spectra with those shown in the NIST 2.0 database and confirmed by reference publications. The linear retention index (LRI) was calculated using the linear retention data of the alkane solution (C8-C28) in n-hexane. The relative amount of volatile compounds was calculated by comparing the peak area and peak IS area, where 5 µL Is with 50 g of sample. The data were analysed as the mean value of two replications.

3. Results and discussion

3.1 The shelf-life of FD durian based on moisture content

The moisture content of FD durian products during storage (28 days) is shown in Figure 1. The initial moisture content of FD durian products was 9.9% and increased during storage at 30, 40, and 50°C. From Figure 1, it can be seen that the moisture content of FD durian ranged from 9.9–13.9%. The highest increasing in moisture content was shown at of 30 °C, followed by at 40 and 50 °C, respectively.

The R^2 of the linear equations for orders 0 and 1 was shown in Table 1. The R^2 of zero order was higher than first order. Therefore, the calculation of shelf-life based on the moisture content was done for zero order. The moisture content slopes of zero order were 0.136; 0.087; and 0.093, stored at 30, 40, and 50°C, respectively (Table 2). By plotting the inverse value of absolute temperature (1/T) against k (slope), the Arrhenius graph was obtained. The Arrhenius equation obtained was ln k = - 8.353 + 1902 1/T, where the $\ln k_0$ = -8.353, (Ea/R) = 1902.

Table 1. R^2 (determination) of linear regression for zero order and first order order obtained from different storage temperatures and parameters

Parameter	Storage temperature (°C)	R^2 (Determination)	
		Zero order	First order
Moisture content	30	0.97	0.95
	40	0.99	0.98
	50	0.94	0.94
L* (Brightness)	30	0.87	0.86
	40	0.83	0.84
	50	0.93	0.95
a* (Redness)	30	0.77	0.76
	40	0.93	0.93
	50	0.99	0.99
b* (Yellowness)	30	0.98	0.99
	40	0.93	0.91
	50	0.96	0.95

The shelf-life of FD durian was calculated employing the input storage temperature of 5°C = 278°K,

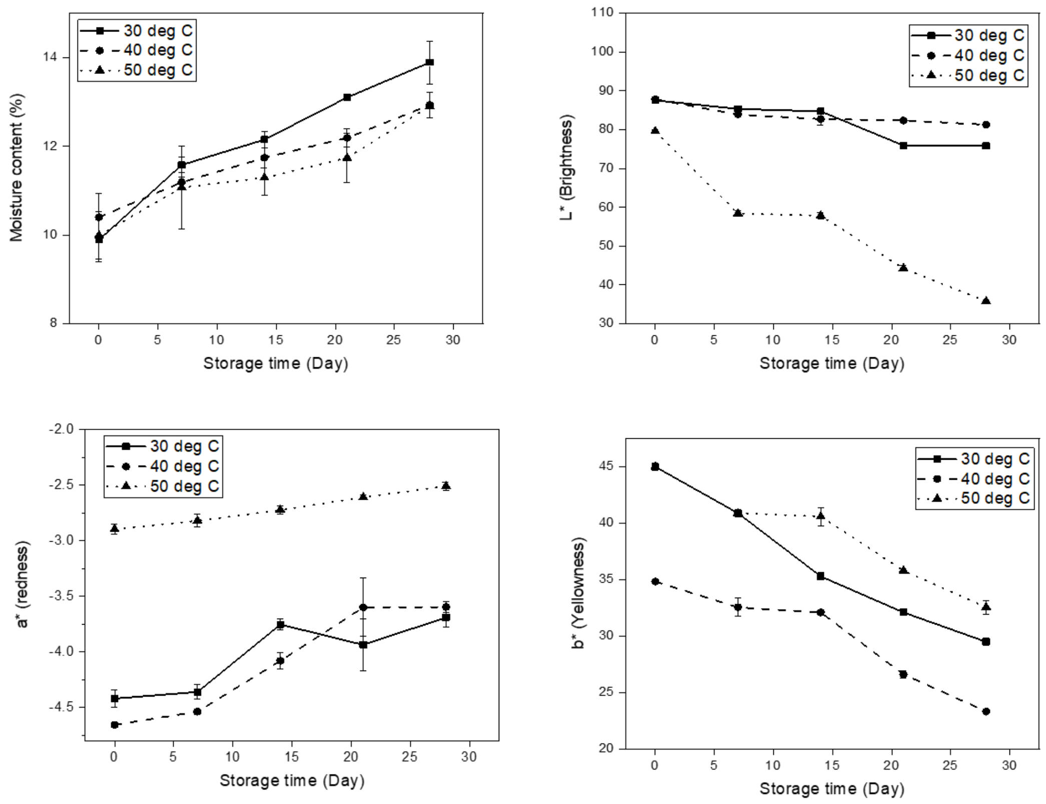

Figure 1. Moisture content and L*a*b* of freeze-dried durian stored at 30, 40, and 50°C

Table 2. Slope and ln k of linear regression of zero order and first order obtained from different storage temperatures and parameters

Parameter	Temperature		1/T	Zero order			First order		
	°C	°K		Slope	k	ln k	Slope	k	ln k
Moisture content	30	303	0.00330	0.136	0.136	-1.955	0.012	0.012	-4.468
	40	313	0.00319	0.087	0.087	-2.447	0.007	0.007	-4.900
	50	323	0.00310	0.093	0.093	-2.378	0.008	0.008	-4.810
L* (Brightness)	30	303	0.00330	-0.470	0.470	-0.756	-0.006	0.006	-5.153
	40	313	0.00319	-0.209	0.209	-1.567	-0.003	0.003	-6.003
	50	323	0.00310	-1.455	1.455	0.375	-0.027	0.027	-3.618
a* (Redness)	30	303	0.00330	0.027	0.027	-3.616	-0.006	0.006	-5.020
	40	313	0.00319	0.044	0.044	-3.131	-0.011	0.011	-4.538
	50	323	0.00310	0.014	0.014	-4.625	-0.005	0.005	-5.253
b* (Yellowness)	30	303	0.00330	-0.568	0.568	-0.566	-0.016	0.016	-4.166
	40	313	0.00319	-0.413	0.413	-0.884	-0.014	0.014	-4.246
	50	323	0.00310	-0.428	0.428	-0.849	-0.011	0.011	-4.498

then ln k = -1.5112 or k = 0.22063 was obtained, meaning that the increase in moisture content was 0.22063 units per day. Therefore, the total quality units until expiry date were calculated by subtracting the initial moisture content (9.89) from the critical limit moisture content (15), resulting in 5.2 units. The estimated shelf-life of FD durian products were 37 and 41 days, at 25 and 30°C respectively (Table 3).

3.2 The shelf-life of FD durian based on L (brightness)*

Figure 1 shows the change in brightness of FD durian during 28 days of storage. They were in a range 34.74 to 87.82. The sharp decrease of L* occurred for samples stored at 50°C, i.e., from 79.6 to 35.7. Different conditions appeared for samples stored at 30 and 40°C, the decrease was relatively moderate, from 87.5 to 75.8, and from 87.8 to 81.3, respectively. The conditions described above, indicate the phenomenon of non-enzymatic browning or the Maillard reaction, where heat react with carbohydrates and proteins, and had changed the colour to brown (dark colour).

The R^2 of orders 0 and 1 was shown in Table 1. The R^2 of first order was higher than zero order. Therefore,

Table 3. Shelf-life of freeze-dried durian calculated from moisture content and colour values

Prediction Temperature (°C)	Shelf-life based on properties (days)			
	Moisture content	L*/Brightness	a*/redness	b*/yellowness
-5	66.9	7365.0	45.2	25.4
5	61.1	2749.5	68.4	30.7
10	55.7	1724.4	83.3	33.5
15	50.7	1099.1	100.7	36.6
20	45.9	711.5	121.0	39.7
25	41.4	467.3	144.4	43.1
30	37.3	311.2	171.4	46.6
35	33.5	210.0	202.3	50.2
40	29.9	143.5	237.5	54.0
45	26.6	99.2	277.4	57.9
50	23.6	69.4	322.4	62.0
55	18.3	49.1	373.1	66.3

the calculation of shelf-life based for the L* value was performed for the first order. The L* slopes of the first order were -0.006; -0.003; and -0.027, stored at 30, 40, and 50°C, respectively (Table 2). By plotting the absolute temperature inverse value (1/T) against k, the Arrhenius graph was obtained. The Arrhenius equation obtained was ln k = 16.57 + 5388 1/T, where the value of ln k_0 = 16.57, (Ea/R) = 5388.

The shelf-life of was calculated through the input storage temperature of 5°C = 278°K, then ln k = -2.8113 or k = 0.060 was generated, meaning that the decrease in L* value was 0.060 units per day. Therefore, the total quality units until expiration were calculated by subtracting the initial L*, which was 87, from the critical limit value of 30, resulting in 57 units. The estimated shelf-life of FD durian products was 467 and 311 days, at 25 and 30 °C, respectively (Table 3).

3.3 The shelf-life of FD durian based on a (redness)*

Figure 1 shows the change of redness of FD durian during 28 days of storage. The a* of FD durian ranges from -4.66 to. -2.51 which were greenness. It can be seen that during 28 days of storage, the a* value of FD durian was increase. The a* of FD durian stored at 50°C was the highest value compared to those stored at 30 and 40°C.

The R^2 of orders 0 and 1 was shown in Table 1. The R^2 of zero order is higher than the first order. Therefore, the calculation of shelf-life based on the a* was carried out for zero order. The a* slopes of the zero order were 0.027; 0.044; and 0.014, stored at 30, 40, and 50°C, respectively (Table 2). By plotting the absolute temperature inverse value (1/T) against k, the Arrhenius graph was obtained. The Arrhenius equation obtained was ln k = -13.56 + 3093 1/T, where the value of ln k_0 = -13.56, (Ea/R) = 3093.

The shelf-life of FD durian was calculated through the input storage temperature of 5°C = 278°K, then ln k = -2,434 or k = 0.087 was obtained, meaning an increase of the a* was 0.087 units per day. Therefore, the total quality units to expiry date were calculated by subtracting the initial a*, which was -4.66, from the critical limit value of 2, resulting in 6.66 units. The estimated shelf-life of FD durian based on the a* were 144 and 171 days, stored at 25 and 30°C, respectively (Table 3).

3.4 The shelf-life of FD durian based on b (yellowness)*

The yellowness value of FD durian during 28 days of storage is shown in Figure 1. The b* of FD durian was positive, which was yellow (the colour of fresh durian pulp). The yellowness durian of FD during storage ranged from 23.3 - 45, with the initial b* value of 45. During storage, the b* was decrease and the lowest level occurred in the samples stored at 40 °C, i.e., 23.3.

The R^2 of orders 0 and 1 was shown in Table 1. The R^2 of zero order is higher than first order. Therefore, the calculation of shelf-life based on the b* is carried out in zero order. The b* slope of the zero order were 0.568; -0,413; and -0.42, stored at 30, 40, and 50 °C, respectively (Table 2). By plotting the absolute temperature inverse value (1/T) against k, the Arrhenius graph was obtained. The Arrhenius equation obtained was ln k = - 5,262 + 1406 1/T, where the value of ln k_0 = -5,262, (Ea/R) = 1406.

The shelf-life of FD durian was calculated by employing the input storage temperature of 5°C = 278°K, then ln k = -0.204 or k = 0.815 was obtained, meaning that the decrease of b* was 0.815 units per day. Therefore, the total quality units to expiry date can be calculated by subtracting the initial b*, which was 45, from the critical limit value of 20, resulting in 25 units. The estimated shelf-life of FD durian based on the b* were 43 and 46 days, stored at 25 and 30°C, respectively

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(Table 3).

3.5 Freeze-dried (FD) durian flavour properties

The edible portion of the durian fruit is reported to have a unique and strong aroma, associated with esters and volatile sulfur compounds (diethyl disulphide and propanethiol, ethyl 2-methylbutanoate). From the fresh durian pulp, FD durian 30 hrs, and FD durian 36 hrs were identified as 24, 17 and 18 volatile compounds, respectively (Table 4). The most volatile compounds were sulfur (12), followed by alcohols (5), acids (5), esters (4), terpenes (1), ketones (1) and aldehydes (1). Fresh durian was relatively high in diethyl disulfide, limonene, ethyl-1-methylethyl disulfide and diethyl

Table 4. Identification of volatile compounds from fresh and freeze-dried (FD) durian pulp using SPME coupled to GC-MS

No	RT (minute)	Compounds	Group	LRI Experiment	LRI Reference	Volatile amount (ppm)			Aroma
						Fresh pulp	FD durian 30 hrs	FD durian 36 hrs	
1	2.002	Ethanethiol[B]	sulfur 1			9.343	3.532	14.620	
2	2.276	Dimethyl sulfide[B]	sulfur 2			6.014	2.177	8.091	cabbage, sulfur, gasoline (12)
3	2.484	Ethyl acetate[B]	acid 1			6.215	5.148	25.200	sweet, fruity (8)
4	2.656	Ethanol[B]	alcohol 1			10.318	2.306	16.371	sweet (13)
5	3.085	Ethyl-2-methylbutanoate[B]	ester 1			3.804	1.606	6.976	
6	3.562	Methyl ethyl disulfide[A]	sulfur 3	1235		4.199	0.000	0.000	
7	3.756	Limonene[A]	terpen	1291	1192(1)	32.887	1.760	12.711	ethereal, citrus-like (14)
8	3.776	Diethyl disulfide[B]	sulfur 4	1297	1217(2)	36.755	2.013	19.238	sulfury, roasty (2)
9	4.036	Acetoin[A]	ketone	1378	1272(3)	0.000	2.398	14.774	butterscotch (3)
10	4.089	Ethyl-1-methylethyl disulfide[B]	sulfur 5	1395		17.478	1.074	6.046	
11	4.179	Hexanol[A]	alcohol 2	1425	1360(4)	1.238	0.000	0.000	green, floral (4)
12	4.326	Dipropyl disulfide[A]	sulfur 6	1475	1370(5)	1.313	0.000	0.000	
13	4.345	Nonanal[A]	aldehid	1482	1385(4)	0.000	0.000	trace	soapy (4)
14	4.433	Ethyl octanoate[A]	ester 2	1513	1458(6)	0.438	0.000	0.000	floral, fruity, musty (6)
15	4.458	Acetic acid[A]	acid 2	1522	1427(7)	3.526	83.819	35.988	vinegar (7)
16	4.69	Ethyl-3-hydroxybutyrate[B]	ester 3	1608		0.576	0.234	0.000	
17	4.704	Propanoic acid[A]	acid 3	1613	1523(7)	0.936	0.000	0.000	Soy (7)
18	4.719	*2,3-butanediol[A]	alcohol 3	1619	1538(6)	0.000	6.455	27.526	floral, fruity (6)
19	4.755	Diethyl trisulfide[A]	sulfur 7	1633		12.963	0.000	0.000	
20	4.803	*2,3-Butanediol[A]	alcohol 4	1652	1538(6)	0.000	0.809	32.561	floral, fruity (6)
21	5.041	2-Methylbutanoic acid[A]	ester 4	1747	1670(8)	8.486	7.573	32.338	
22	5.082	3,5-Dimethyl-1,2,4-trithiolane[A]	sulfur 8	1764	1602(9)	6.290	0.000	0.000	
23	5.166	Dipropyl trisulfide[A]	sulfur 9	1799	1658(5)	0.663	0.000	0.000	
24	5.272	**3-Ethyl-5-methyl-1,2,4- trithiolane[B]	sulfur 10	1842		0.859	0.000	0.000	
25	5.283	Bis[1-(methylthio)ethyl]-disulfide[B]	sulfur 11	1847		1.114	0.000	0.000	
26	5.307	**3-Ethyl-5-methyl-1,2,4- trithiolane[B]	sulfur 12	1856		0.488	0.000	0.000	
27	5.481	Hexanoic acid[A]	acid 4	1926	1863(10)	1.804	3.047	14.601	cheese (10)
28	5.724	Phenylethyl alcohol[A]	alcohol 5	2021	1910(11)	0.000	0.619	1.363	
29	6.069	Octanoic acid[A]	acid 5	2140	2083(10)	0.649	1.116	4.124	cheese (10)

All data are the means of two replicates; *2,3-Butanediol and **3-Ethyl-5-methyl-1,2,4- trithiolane has two isomers or stereoisomers; A: identified by mass spectrum and confirmed by retention index reported in literature, i.e., (1) Umano and Shibamoto (1987), (2) Selli *et al*. (2006), (3) Lee and Noble (2003), (4) Schieberle and Grosch (1987), (5) Takahashi and Shibamoto (2008), (6) Miranda-Lopez *et al*. (1992), (7) Lee and Noble (2003), (8) Dregus and Engel (2003), (9) Chung *et al.* (2004), (10) Ferreira *et al. (*2001), (11) Dregus and Engel (2003), (12) Peterson and Reineccius (2003), (13) Chung, Eiserich and Shibamoto (1993), (14) Schieberle and Grosch (1988); B: identified by mass spectrum trisulfide, while FD durian 36 hrs contained relatively high acetic acid, two isomers of 2,3 butanediol, 2 methylbutanoic acid, diethyl disulfide, ethanol, ethanethiol, acetoin and limonene. Compared to the volatile profile of fresh durian, the volatile profile of FD durian 30 hrs was more similar to that of FD durian 36 hrs, but with a lower content of volatile compounds except acetic acid. Among all samples, FD durian 30 hrs contained the most acetic acid compounds. Voon *et al.*, (2007) reported that from durian pulp had been identified 22 esters, 14 sulfur compounds, 7 alcohols, 3 aldehydes and 1 ketone. Furthermore, the main sulfur compounds in durian pulp were diethyl disulphide, ethyl-n-propyl disulphide, diethyl trisulphide and ethanethiol, while the main ester compounds were ethyl propanoate, ethyl-2-methyl butanoate, or propyl-2-methylbutanoate.

The freeze-drying process caused the loss of 11 volatile compounds (methyl ethyl disulfide, hexanol, dipropyl disulfide, ethyl octanoate, propanoic acid, diethyl trisulfide, 3,5-dimethyl-1,2,4-trithiolane, dipropyl trisulfide, two 3-ethyl-5-methyl-1,2,4-trithiolane isomers, bis [1- (methylthio) ethyl] -disulfide) and the formation of four new compounds (acetoin, two 2,3-butanediol isomers, and phenyl ethyl alcohol) in durian pulp. Chin *et al.*, (2008) reported that the freeze-drying process in durian pulp caused the loss of ethyl thioacetate, propyl butanoate, propyl 2-methylbutanoate and propyl 3-methylbutanoate.

Similar to the freeze-drying process, at 4°C of minimally processed durian pulp, most of the ester compounds were lost after 14 days of storage. This result was related to the decline in fruity aroma intensity detected by the panelists during storage. The total durian sulfur content remained unchanged after 42 days of storage, although the sulfur notes perceived by panelists decreased significantly at the end of storage (Voon *et al.*, 2007).

Weenen *et al.* (1996) reported that two potent odourants in Indonesian durian pulp, e.g., sulfur compounds 3,5-dimethyl-1,2,4-trithiolane, and nonsulfur ethyl compounds 2-methylbutanoate. They also reported that the potent odorant compounds in Monthong durian pulp were ethyl (2S) -2-methylbutanoate, ethanethiol, 1-(ethylsulfanyl) ethane-1-thiol, methanethiol, ethane-1,1-dithiol, ethyl 2- methylpropanoate. Furthermore, based on aroma simulations and omission tests, durian aroma, in general, can be formed from a mixture of two compounds, e.g., ethyl (2S) -2-methylbutanoate and 1-(ethylsulfanyl) ethane-1-thiol at certain concentrations.

Table 5 shows the amount of representative volatile components in ppm in the samples of fresh and freeze-dried examined using GC-MS. Diethyl disulfide was the most abundant volatile among the determined analytes with 36.75 ppm in fresh durian pulp. Chin *et al.* (2008) reported that the amount of Diethyl disulphide from fresh and freeze-dried durian (Variety D24) were 40.85 µg/g and 1.15 µg/g, respectively. Freeze-drying caused major reduction of major aroma volatile that ranged from 78-100% (FD durian 30 hrs) and 0-100% (FD durian 36 hrs). The reduction of aroma volatile in freeze-dried durian 36 hrs was lower than other freeze-dried durians in a previous study conducted by Chin *et al.* (2008), i.e. 72-97%. This result may be due to the drying time, where this study employed longer drying time (36 hrs) compared to a previous study (12 hrs). Furthermore, volatile compounds were influenced by the ice sublimation stage in freeze-drying (Krokida and Philippopoulos, 2006).

4. Conclusion

Product of dried durian had been succefully obtained by using laboratory-scale freeze-dryer with two different drying times i.e., 30 and 36 hrs. The shelf-life of FD durian at 25 and 30°C were based on the following parameters: (1) moisture content 41 and 37 days, (2) L*/ brightness 467 and 311 days, (3) a*/redness: 144 and 171 days, and (4) b*/yellowness 43 and 46 days. There were 24 volatile compounds that contribute to the flavour of fresh durian pulp and five of them had concentrations of more than 10 ppm. The losses percentage of these five volatile compounds were in a range of 78-95% (FD durian for 30 hrs) and a range of 0-100% (FD durian for 36 hrs).

Conflict of interest

The authors declare no conflict of interest.

Table 5. Concentration of target analytes from fresh and freeze-dried (FD) durian (ppm) determined by SPME coupled to GC-MS

Volatile compounds	Volatile amount (ppm)			Volatile Loses (%)	
	Fresh pulp	FD durian 30 hrs	FD durian 36 hrs	FD durian 30 hrs	FD durian 36 hrs
Ethanol	10.31	2.30	16.37	78.00	0.00
Limonene	32.88	1.75	12.71	95.00	61.40
Diethyl disufide	36.75	2.01	19.23	94.60	47.70
Ethyl 1-methyethyl disulfide	17.47	1.07	6.04	93.80	64.40
Diethyl trisulfide	12.96	0.00	0.00	100.00	100.00

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