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Original Research Article

Influence of harvest maturity and storage condition on changes on volatile compounds of 'Phulae' pineapple fruit

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ABSTRACT

Nowadays, the number of consumers asking for aroma and flavour quality were increasing. However, no previous study about influences of harvest maturity, storage condition on changes in volatile organic compounds of 'Phulae' pineapple fruits. Objectives of this research were investigations of volatile profiles, effect of harvest maturity (between green and full mature) and storage condition (10°C, 85-90% RH for 14 days before moved to room temperature) on volatile compounds in the pineapple fruits by simulation of fruit logistics. The results showed that 18 volatile compounds were identified in full mature stage included 10 esters, 2 terpenes and terpenoids, 1 alcohols and phenols, 1 aldehydes, 3 miscellaneous, and 1 unknown. On the other hand, 7 volatile compounds were detected. 2 esters, 2 terpenes and terpenoids, 1 alcohols and phenols, and 2 miscellaneous were identified. Volatile compounds in green mature stage tended to be increased after storing under the post-harvest storage included detection of volatile compounds in group of aldehydes, ketones, and lactones after 14 days of storage. In conclusion, harvest maturity and storage condition affected the development of volatile compounds in the pineapple fruits. Full mature fruits contained higher amounts of volatile compounds than green mature fruits.

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INTRODUCTION

Pineapples normally grow in tropical areas in the world. There are many cultivars of pineapple, but only a few varieties are sold commercially. In Chiang Rai province, Thailand, there is an economic cultivar of pineapple name 'Phulae' that plays an important role in geographical indication product. 'Phulae' pineapple refers to the Queen varieties (Kongsuwan et al., 2009). Nowadays, there are two ways to export the fresh fruits in long marketing chain. The first way growers harvest fruits at green mature stage and export these fruits by sea freight. However, low temperature for long time storage during transportation is harmful for the fruit from chilling injury that cause internal browning in the fruit. Second way, fruits are harvested at fully mature stage and exported as fast as possible by air freight that is an alternative way to transport fully mature fruits because fully mature fruits have limitation of shelf life and storage life. Although, air freight is more acceptable than sea freight, but boundary of marketing is only in high-end price sector (Steingass et al., 2014). Nearby difference of variety and stage of harvest maturity, quality of fruit is affected by postharvest handling resolutely (Steingass et al., 2014). Aroma and flavor of fruits formed by volatile compounds such as alcohols, esters, aldehydes, ketones, acids, furans, terpenes, etc. that generated by metabolic pathways during harvest, postharvest, storage, and factors related to species, variety, and processing (Kaewtathip and Charoenrein, 2012). Previous study showed that 144 and 127 volatile compounds were detected in green and postharvest ripened fruits respectively (Umano et al., 1992b). Nowadays, the number of consumers asking for horticultural products with higher quality not only physical appearance and nutrients, but aroma quality is also increasing (Ponce-Valadez et al., 2016). However, there is no previous study about influences of harvest maturity, storage condition on changes in volatile organic compounds of 'Phulae' pineapple fruit. Objectives of this research were investigation of volatile profiles, effect of harvest maturity and storage condition on volatile compounds in 'Phulae' pineapple by simulation of fruit logistics. Volatile profiles and influences of harvest maturity on volatile compounds of 'Phulae' pineapple was also investigated in this study as same as physicochemical qualities such as total soluble solids (TSS), titratable acidity (TA), TSS/TA, peel color, flesh color, and vitamin C.

MATERIALS AND METHODS

Plant materials and experimental design

'Phulae' pineapple fruits with uniformity and no defects (250g per fruit, without crown) were purchased from orchard in Nanglae district, Chiang Rai, Thailand at green mature stage and full mature stage (fruit peel was more than 75% yellow). The fruits were cleaned with blower and separated into 2 parts. First, full mature and green mature pineapples were measured on their volatile compounds within one day after harvest. For part 2, green mature pineapple fruits were packed into corrugated box (20 fruits per box). Then, it was kept under cooling room at 10°C, 85-90 %RH for 14 days. After that, the fruits were moved to store at room temperature (25°C) for 12 days. In this part, volatile compounds were analyzed on every 2 days after fruits were moved to store at 25°C. TSS, TA, peel color, flesh color, and vitamin C were measured as physico-chemical properties and given as supplementary data.

Volatile profiling

Volatile compounds profiles were obtained by the method of Steingass et al. (2014) with some modification. One hundred and fifty grams of fresh pineapple pulp was blended with household blender (HR2118/01, Philips) at speed level 1 for 1 min with 10 mL deionized water (DI H_{20}) and 30 g sodium chloride. 100 µL of 2-Methyl-1-pentanol solution (3.30 g/L) were added to the sample before blending as an internal standard (IS). Total ion chromatogram peak area of the IS was able to use for semi-quantitative determination of individual compounds. Blended sample (10.0 ± 0.1 g) was filled into a 30-mL amber vial and covered with Teflon coated silicone rubber septum and black phenolic hole cap (SU23228, Sigma–Aldrich). Vials were incubated for 30 min in a water bath preserved temperature at 40°C. Then, volatile compounds were extracted by headspace solid phase micro-extraction (HS-SPME) technique using a divinylbenzene/carboxen/polydimethylsiloxane fiber (50/30 µm DVB/CAR/PDMS StableFlex®/SS, Supelco 57328-U, Sigma-Aldrich) for 30 min. Prior to volatile compound extraction, the SPME fiber was preconditioned at 270°C for 60 minutes and penetrated into the headspace of the sample vials manually. Volatile compounds were analyzed by a Trace 1300 gas chromatograph in splitless mode connected with ISQ QD csingle quadrupole mass spectrometer (Thermo Fisher Scientific Inc.). A liner sealing ring and 0.8 mm ID SPME liner (Thermo Fisher Scientific Inc.) were used for focusing volatile compounds. Volatile compounds were desorbed for 1 min at 270°C. Prior to the latter extraction, the SPME fiber was preheated for 15 min at 270°C. Carrier gas was helium and flow rate were constant at 1.5 mL/min. The analytical column was a 5% phenyl methylpolysiloxane (TG-5MS, 30 m x 0.25 mm, df = 0.25 µm, Thermo Fisher Scientific Inc.). The oven temperature initiated from 60°C. After 1 min, temperature was increased at 3°C/min to 110°C and continuously increased to a final temperature of 250°C at 125°C/min and held at the final temperature for 7 min. Mass spectrometer recorded spectra and total ion chromatograms (TIC) by electron impact positive mode (EI+) by range of scan mode at m/z 40-270 using energy of electron at 70 eV. Source and transfer line temperatures were in range of 150-230°C and total run time was 45 min. Each individual volatile compound identification was done by comparing each mass spectra to NIST library (NIST 14) on Qual Browser Thermo Xcalibur, Version 4.0.27.10 (Thermo Fisher Scientific Inc.) and available literature (Stein, 1994) including volatile compounds reported by other study (Elss et al., 2005; Stein, 1994; Umano et al., 1992a; Zheng et al., 2012). Linear retention indices according to Van Den Dool and Dec. Kratz (1963), it was calculated relative to mixtures of n-alkanes (C8–C20) to compare with values from the literature. Five fruits were used for volatile compounds analysis on each replication.

Physicochemical properties

Peel and flesh color were measured on top, central, and basal part of the fruit by colorimeter (Konica Minolta cr-10, Japan) using the CIE system (L*, a*, and b*). After that, hue angle was calculated. Pineapple were peeled and removed core and fruitlet manually. Fresh pineapple pulp was squeezed by hand squeezer to collect pineapple juice for total soluble solids (TSS), titratable acidity (TA), and vitamin C content. TSS was determined from 1 mL of pineapple juice by using digital refractometer (PAL-1 model, ATAGO, Japan) and data was shown as %Brix. TA was determined by titration of pineapple juice with 0.1 N NaOH and using phenolphthalein as an indicator. The results were expressed as citric acid in grams per 100 g of fresh weight (FW). TSS/TA ratio was included in this part. Determination of vitamin C content was followed the method of AOAC (1990), 2 mL of pineapple juice mix with 5 mL of meta-phosphoric acetic acid solution and titrate with 2,6-dichloroindophenol until light pink color appear for 5 sec. Volume of the sample juice used for the titration with indophenol solution was recorded and calculated for vitamin C content.

Statistical analysis

Data were analyzed using the one-way analysis of variance (ANOVA). HSD Tukey's test ($p<0.05$) was applied to compare the mean values of the data using the SPSS program, version 23.0 (SPSS Inc., Chicago, IL, USA).

RESULTS AND DISCUSSION

Physicochemical properties

According to McGuire (1992), hue angle was used to indicating differences of color in ripen fruits from green, yellow, red, and blue. Table 1 was shown for physicochemical properties between the pineapples harvested at full mature and green mature stage. Peel and flesh of full mature pineapples have hue angle value significantly lower than the green mature pineapples at 0 day of storage, which indicated that peel and flesh of full mature pineapples have yellowish color more than peel of green mature pineapples. TSS of full mature pineapples was higher than green mature pineapples at 0 day of storage. However, there was no significant difference between TSS and TA of full mature and green mature pineapples. For TSS/TA ratio, no significant difference was observed in full mature pineapples and green mature pineapples as same as vitamin C content.

Table 1. Physicochemical properties of 'Phulae' pineapple harvested at full mature and green mature

Quality Characteristic	Harvest maturity	
	Full mature	Green mature
Hue value (Peel)	70.96 ± 1.96^{b}	76.76 ± 1.37^{a}
Hue value (Flesh)	83.79 ± 0.69^{b}	89.07 ± 0.86^{a}
TSS	16.24 ± 1.13^{a}	14.02 ± 0.95^{ab}
TA	0.41 ± 0.08^{a}	0.41 ± 0.04^{a}
TSS/TA ratio	39.44 ± 0.18^{a}	34.71 ± 1.27^{b}
Vitamin C	17.64 ± 2.46^{a}	21.55 ± 4.59^{a}

After storage at 10°C for 14 days and moved to store at 25°C for 12 days, hue angle of peel and flesh of pineapples decreased (Figure 1) due to its natural ripening process. Figure 2 was shown for change in TSS, TA, TSS/TA ratio, and vitamin C content for green mature 'Phulae' pineapple fruit during the storage condition. There were an decreasing trend of TSS during storage. Rohrbach and Paull (1982) indicated that high storage temperature activates decreasing of TSS value. After storage, the result showed that TA slightly increased and slightly decreased after 14 days of storage at 10°C. Decreasing of TA might be due to organic acids in fruit were substrates of respiration and sugar that usually decrease during maturity (Hong et al., 2013). Reduction of TSS and acid contents may be caused by volatile compound formation because both contents are substrate of volatile compounds (Dirinck et al., 1989; Rohrbach and Paull, 1982; Salunkhe and Y Do, 1976; Song and Forney, 2008). Vitamin C trended to decrease after the fruits were move to store at 25°C. It might cause from water loss according to previous report by Lee and Kader (2000).

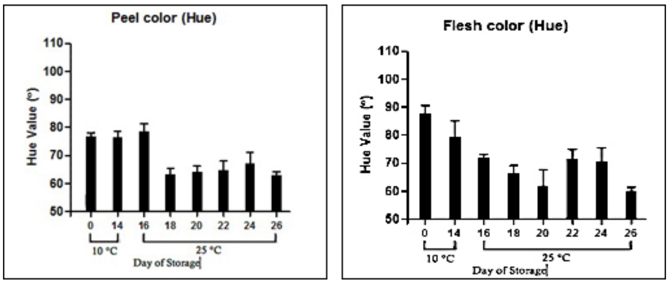

Figure 1. Change in hue angle of peel for green mature 'Phulae' pineapple fruit during each storage condition. Data are mean ± SD of n = 5

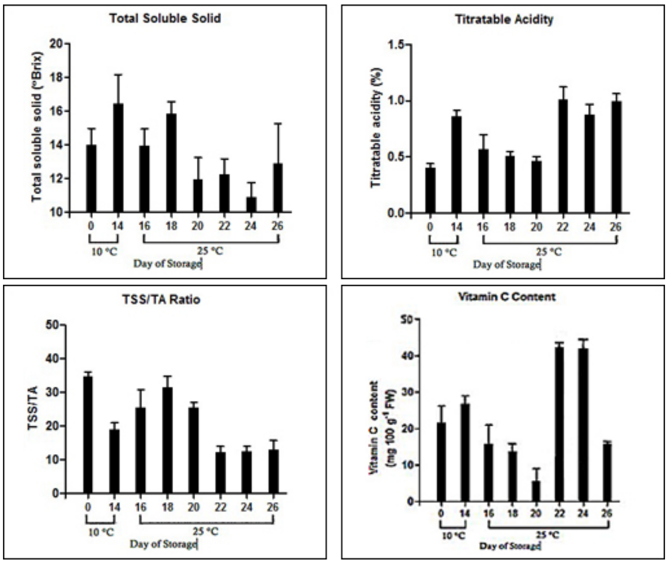

Figure 2. Change in TSS, TA, TSS/TA ratio, and vitamin C content for green mature 'Phulae' pineapple fruit during each storage condition. Data are mean ± SD of n = 5

Identification of volatile compounds

Volatile compounds identified in each maturity stages of 'Phulae' pineapples were shown in Table 2-10. 18 volatile compounds were identified in the headspace of full mature pineapples (Table 10). Refer to the literature data, 10 esters, 2 terpenes and terpenoids, 1 alcohols and phenols, 1 aldehydes, 3 miscellaneous, and 1 unknown. In green mature pineapples, 7 volatile compounds were detected. 2 esters, 2 terpenes and terpenoids, 1 alcohols and phenols, and 2 miscellaneous were identified (Table 2). At 14 days of storage at 10°C of green mature pineapples, 28 volatile compounds were detected such as 15 esters, 2 terpenes and terpenoids, 2 alcohols and phenols, 3 miscellaneous, and 4 unknowns were identified (Table 3). 43 volatile compounds were identified in green mature pineapples stored for 14 days at 10°C + 2 days at 25°C (Table 4) such as 6 esters, 14 terpenes and terpenoids, 3 alcohols and phenols, 3 aldehydes, 8 miscellaneous, and 9 unknowns. 44 volatile compounds were found in green mature pineapples stored for 14 days at 10°C + 4 days at 25°C (Table 5) such as 29 esters, 8 terpenes and terpenoids, 2 alcohols and phenols, 1 lactone, 3 miscellaneous, and 1 unknown. There were 47 volatile compounds in green mature pineapple stored for 14 days at 10°C + 6 days at 25°C (Table 6). 32 compounds were esters, 8 terpenes and terpenoids, 1 alcohols and phenols, 1 ketone, 4 miscellaneous, and 1 unknown. 49 volatile compounds were identified in green mature pineapples stored for 14 days at 10°C + 8 days at 25°C (Table 7.) such as 25 esters, 9 terpenes and terpenoids, 2 alcohols and phenols, 1 aldehyde, 1 ketone, 1 lactone, 5 miscellaneous, and 3 unknowns. 50 volatile compounds of green mature pineapples stored for 14 days at 10°C + 10 days at 25°C were showed on (Table 8.) such as 24 esters, 13 terpenes and terpenoids, 1 alcohols and phenols, 2 ketones, 4 lactones, 5 miscellaneous, and 1 unknown. 44 volatile compounds were found in green mature pineapples stored for 14 days at 10°C + 12 days at 25°C (Table 9.) such as 25 esters, 9 terpenes and terpenoids, 1 alcohols and phenols, 4 lactones, 4 miscellaneous, and 1 unknown. Esters were represented as the largest group in both qualitative and quantitative except green mature pineapples stored for 0 day and 14 days at 10°C +2 days at 25°C. Berger (2007) El Hadi et al. (2013) reported that esters were predominant compound of other volatile compounds in MD2 'Extra Sweet' pineapple (*Ananas comosus* L. Merr). Major key odorants of the pineapple such as methyl 3-methylthiopropanoate, ethyl 3-methylthiopropanoate, 2-methylbutanoate, 2-methylhexanoate, γ-octalactone, δ-octalactone, γ-nonalactone, and 1,3,5,8-undecatetraene (Berger, 2007; Flath, 1980; Umano et al., 1992a). Methyl 3-methylthiopropanoate, which is an aroma characteristic in pineapple (*Ananas comosus* L. Merr) were found on green mature 'Phulae' pineapple stored for 14 days, 14 days at 10°C + 4 days at 25°C, 14 days at 10°C + 6 days at 25°C, 14 days at 10°C + 8 days at 25°C, 14 days at 10°C + 10 days at 25°C, and 14 days at 10 °C + 12 days at 25 °C as same as γ-octalactone that found on 22 and 24 days of storage, γ-nonalactone that found on 26 days of storage, and 1,3,5,8-undecatetraene that found on the pineapples stored for 14 days at 10°C + 8 days at 25°C, 14 days at 10°C + 10 days at 25°C, and 14 days at 10°C + 12 days at 25°C.

Table 2. Identification of volatile compounds in 'Phulae' pineapple pulp at green mature stage

RT(s)	Name	SI	Concentration (g/L)	LRI	Classification
3.87	2-Methyl-1-pentanol (internal standard)	728	3.30	802	Alcohols and phenols
5.59	Methyl 12-(2-octylcyclopropyl)dodecanoate	928	0.02	848	Esters
8.70	(3Z)-1,4,6,9-Nonadecatetraene	777	0.05	937	Terpenes and Terpenoids
9.06	α-Ocimene	780	0.32	1046	Terpenes and Terpenoids
22.11	Methyl 12,15-octadecadiynoate	830	0.03	1057	Esters
35.98	Hexadecanoic acid[a,b]	767	0.04	1385	Miscellaneous
36.00	Hexadecanoic acid[a,b]	773	0.02	1756	Miscellaneous

[a]Unknown isomer.
[b]Tentatively identified.
SI = Similarity to reference spectrum on a scale of 0–999, with higher scores indicating greater similarity.

Table 3. Identification of volatile compounds in 'Phulae' pineapple pulp stored for 14 days at 10°C

RT(s)	Name	SI	Concentration (g/L)	LRI	Classification
1.74	2,5-Difluoro-α,3,4-trihydroxy-N-methyl benzeneethanamine[a,b]	749	0.01	-	Miscellaneous
1.94	Methyl acetate	859	0.11	-	Esters
2.19	Methyl propanonate	869	0.16	-	Esters
2.43	Methyl 2-propanoate	766	0.03	-	Esters
2.48	Dimethylsilanediol	778	0.02	-	Alcohols and Phenols
2.51	2,5-Difluoro-α,3,4-trihydroxy-N-methyl benzeneethanamine[a,b]	664	0.01	-	Miscellaneous
2.67	Methyl butanoate	934	0.21	-	Esters
3.13	Methyl 2-methylbutanoate	912	1.35	-	Esters
3.40	Hexanal	832	0.11	818	Aldehydes
3.49	Hexamethylcyclohexasiloxane	693	0.02	824	Miscellaneous
3.85	2-Methyl-1-pentanol (internal standard)	920	3.30	847	Alcohols and phenols
5.20	Unknown	688	0.01	922	Unknown
5.54	Methyl hexanoate	925	0.82	935	Esters
6.13	Methyl 4-methylpentanoate	688	0.01	959	Esters
6.70	2-Methylpentyl acetate	747	0.03	982	Esters
8.39	Methyl 5-methylhexanoate	726	0.01	1037	Esters
8.62	Methyl 3-methylthiopropanoate	827	0.13	1043	Esters
9.04	(3Z)-1,4,6,9-Nonadecatetraene	752	0.03	1056	Terpenes and Terpenoids
9.29	Unknown	679	0.01	1063	Unknown
10.72	Methyl 2,3-dimethylbutanoate	653	0.20	1105	Esters
11.21	Unknown	707	0.01	1117	Unknown
11.70	Methyl 4Z-octenoate	792	0.05	1130	Esters
11.98	Methyl octanoate	896	0.19	1137	Esters
12.83	Unknown	681	0.02	1159	Unknown
19.44	Methyl 12,15-octadecadiynoate	764	0.02	1320	Esters
19.56	Methyl 4-decenoate	815	0.03	1323	Esters
19.69	9,12,15-Octadecatrienal	777	0.05	1326	Aldehydes
22.11	α-Ylangene	842	0.03	1385	Terpenes and Terpenoids

[a]Unknown isomer.
[b]Tentatively identified.
SI = Similarity to reference spectrum on a scale of 0–999, with higher scores indicating greater similarity.

Table 4. Identification of volatile compounds in 'Phulae' pineapple pulp stored for 14 days at 10°C + 2 days at 25°C

RT(s)	Name	SI	Concentration (g/L)	LRI	Classification
1.59	Carbon dioxide	834	0.07	-	Miscellaneous
1.72	Unknown	667	0.02	-	Unknown
1.82	4,4'-Diisothiocyanostilbene-2,2'-Disulfonic Acid	702	0.03	-	Miscellaneous
1.91	Unknown	693	0.02	-	Unknown
1.95	Unknown	726	0.01	-	Unknown
2.01	Unknown	656	0.02	-	Unknown
2.17	Dimethylsilanediol	724	0.01	-	Alcohols and Phenols
2.23	Methyl 2-propanoate	641	0.01	-	Esters
2.45	Methyl butanoate	771	0.02	-	Esters
2.90	Methyl 2-methylbutanoate	897	0.16	-	Esters
3.15	Hexanal	739	0.01	801	Aldehydes
3.55	2-Methyl-1-pentanol (internal standard)	929	3.30	827	Alcohols and phenols
4.53	Unknown	734	0.01	892	Unknown
5.21	Methyl hexanoate	872	0.08	922	Esters
8.10	2-Ethylhexanol	870	0.05	1028	Alcohols and Phenols
10.08	Methyl 2,3-dimethylbutanoate	647	0.07	1087	Esters
10.79	Nonanal	771	0.01	1107	Aldehydes
13.92	1-Methylene-1H-indene	884	0.06	1186	Miscellaneous
14.20	(-)-α-Gurjunene	922	0.66	1193	Terpenes and Terpenoids
15.81	5-Hydroxymethylfurfural	847	0.23	1233	Aldehydes
16.79	α-Copaene[a,b]	734	0.02	1256	Terpenes and Terpenoids
19.15	α-Ylangene[a,b]	771	0.02	1313	Terpenes and Terpenoids
19.81	α-Ylangene[a,b]	797	0.04	1329	Terpenes and Terpenoids
21.05	Unknown	851	0.05	1359	Unknown
21.39	Globulol	732	0.04	1368	Miscellaneous
21.73	γ-Amorphene	906	0.22	1376	Terpenes and Terpenoids
21.97	α-Cadinene	891	0.14	1382	Terpenes and Terpenoids
23.04	α-Ylangene[a,b]	851	0.03	1408	Terpenes and Terpenoids
23.57	α-Muurolene	902	0.21	1421	Terpenes and Terpenoids
23.99	Desulphosinigrin[a,b]	722	0.05	1432	Miscellaneous
24.01	Desulphosinigrin[a,b]	735	0.01	1432	Miscellaneous
24.03	Desulphosinigrin[a,b]	741	0.02	1433	Miscellaneous
24.06	Desulphosinigrin[a,b]	738	0.02	1434	Miscellaneous
24.27	α-Copaene[a,b]	859	0.09	1439	Terpenes and Terpenoids
24.82	trans-Calamenene	804	0.02	1453	Terpenes and Terpenoids
24.95	cis-Calamenene	836	0.04	1456	Terpenes and Terpenoids
25.08	δ-Amorphene	861	0.06	1459	Terpenes and Terpenoids
26.02	4,5,9,10-Dehydro-isolongifolene	705	0.03	1483	Terpenes and Terpenoids
27.64	Unknown	658	0.02	1524	Unknown
29.32	Unknown	770	0.03	1568	Unknown
30.97	1-Decanethiol acetate	674	0.02	1611	Esters
32.58	Guaiazulene	762	0.02	1655	Terpenes and Terpenoids
35.04	Unknown	633	0.02	1726	Unknown

[a]Unknown isomer.
[b]Tentatively identified.
SI = Similarity to reference spectrum on a scale of 0–999, with higher scores indicating greater similarity.

Table 5. Identification of volatile compounds in 'Phulae' pineapple pulp stored for 14 days at 10°C + 4 days at 25°C

RT(s)	Name	SI	Concentration (g/L)	LRI	Classification
1.54	Carbon dioxide	815	0.09	-	Miscellaneous
1.72	Methyl acetate	919	0.15	-	Esters
1.76	Thiourea	637	0.04	-	Miscellaneous
1.90	Ethyl acetate	760	0.04	-	Esters
1.96	Methyl propanonate	894	0.44	-	Esters
2.19	Methyl 2-propanoate	908	0.29	-	Esters
2.41	Methyl butanoate	945	1.31	-	Esters
2.68	Ethyl 2-methylpropanoate	701	0.02	-	Esters
2.86	Methyl 2-methylbutanoate	906	7.21	-	Esters
3.10	Methyl pristanate	683	0.06	-	Esters
3.40	Methyl pentanoate	881	0.10	818	Esters
3.51	2-Methyl-1-pentanol (internal standard)	920	3.30	825	Alcohols and phenols
3.77	Ethyl 2-methylbutanoate	928	0.35	842	Esters
4.25	2-Methylbutyl acetate	686	0.02	873	Esters
4.91	Methyl 5-hexenoate	711	0.04	910	Esters
5.18	Methyl hexanoate	960	16.87	921	Esters
5.36	Methyl 3-hexenoate[a,b]	815	0.12	928	Esters
5.45	Methyl 3-hexenoate[a,b]	783	0.18	932	Esters
6.22	Methyl 2-hexenoate	737	0.02	963	Esters
6.29	2-Methylpentyl acetate	820	0.14	965	Esters
7.15	Ethyl hexanoate	846	0.17	1000	Esters
7.94	Methyl heptanoate	873	0.21	1023	Esters
8.02	Methyl 3-methylthiopropanoate	888	0.54	1026	Esters
8.08	2-Methyl-2-hexanethiol	654	0.06	1028	Alcohols and Phenols
8.38	α-Pinene	695	0.02	1036	Terpenes and Terpenoids
8.55	Unknown	654	0.09	1041	Unknown
8.74	(Z)-β-Ocimene	811	0.07	1047	Terpenes and Terpenoids
10.06	Methyl 2,3-dimethylbutanoate	658	1.98	1086	Esters
10.58	Methyl 2,3-dimethylbutanoate	662	0.16	1101	Esters
11.21	Methyl 4Z-octenoate	898	0.97	1117	Esters
11.53	Methyl Octanoate	913	2.95	1125	Esters
11.74	Methyl (Z)-4-octenoate	792	0.03	1131	Esters
12.29	Methyl 7-octynoate	724	0.05	1145	Esters
13.47	(E)-5-Undecen-3-yne	732	0.03	1175	Miscellaneous
13.58	6-(Z)-1-Butenyl-1,4-cycloheptadiene	740	0.05	1178	Terpenes and Terpenoids
14.89	Methyl 3-hydroxyhexanoate	682	0.13	1210	Esters
16.87	3-Methoxy-3-methyl-tetrahydro-pyran-2-one	685	0.11	1258	Lactones
19.10	Methyl (E)-4-decenoate	866	0.12	1312	Esters
19.18	(3Z)-Heptadeca-1,8,11,14-tetraene	762	0.60	1314	Terpenes and Terpenoids
19.72	Methyl 8-methylnonanoate	827	0.09	1327	Esters
21.83	α-Copaene[a,b]	926	0.27	1378	Terpenes and Terpenoids
22.50	(-)-β-Elemene	839	0.08	1395	Terpenes and Terpenoids
23.22	α-Longipinene	810	0.05	1413	Terpenes and Terpenoids
26.87	α-Copaene[a,b]	805	0.07	1504	Terpenes and Terpenoids

[a]Unknown isomer.

[b]Tentatively identified.

SI = Similarity to reference spectrum on a scale of 0–999, with higher scores indicating greater similarity.

Table 6. Identification of volatile compounds in 'Phulae' pineapple pulp stored for 14 days at 10°C + 6 days at 25°C

RT(s)	Name	SI	Concentration (g/L)	LRI	Classification
1.58	Carbon dioxide	813	0.07	-	Miscellaneous
1.67	Nitrosomethane	791	0.05	-	Miscellaneous
1.76	Methyl acetate	913	0.14	-	Esters
1.80	Thiourea	653	0.04	-	Miscellaneous
1.94	Ethyl acetate	881	0.25	-	Esters
2.00	Methyl propanoate	886	0.31	-	Esters
2.22	Methyl 2-methylpropanoate	904	0.15	-	Esters
2.36	Ethyl propanoate	713	0.02	-	Esters
2.44	Methyl butanoate	961	1.09	-	Esters
2.71	Ethyl 2-methylpropanoate	796	0.06	-	Esters
2.89	Methyl 2-methylbutanoate	905	4.82	-	Esters
3.13	Ethyl butanoate	884	0.14	-	Esters
3.42	Methyl pentanoate	854	0.08	819	Esters
3.53	2-Methyl-1-pentanol (internal standard)	928	3.30	826	Alcohols and phenols
3.79	Ethyl 2-methylbutanoate	961	1.50	843	Esters
4.23	3-Methylbutyl acetate	729	0.02	872	Esters
4.28	2-Methylbutyl acetate	831	0.05	875	Esters
4.92	Methyl 3-cyclopropylpropanoate	742	0.03	910	Esters
5.20	Methyl hexanoate	962	12.70	922	Esters
5.38	Methyl 3-hexenoate[a,b]	828	0.11	929	Esters
5.46	Methyl 3-hexenoate[a,b]	819	0.13	932	Esters
6.23	Methyl 2-hexenoate	768	0.03	963	Esters
6.93	α-Myrcene	717	0.03	991	Terpenes and Terpenoids
7.16	Ethyl hexanoate	916	0.93	1000	Esters
7.94	Methyl heptanoate	913	0.38	1023	Esters
8.02	Methyl 3-methylthiopropanoate	919	1.04	1026	Esters
8.39	α-Pinene	776	0.05	1037	Terpenes and Terpenoids
8.55	Unknown	644	0.12	1041	Unknown
8.74	α-Ocimene	864	0.14	1047	Terpenes and Terpenoids
10.06	Methyl 2-methylacetoacetate	669	1.40	1086	Esters
10.57	Methyl 2,3-dimethylbutanoate	671	0.20	1101	Esters
11.20	Methyl 4Z-octenoate	891	0.77	1117	Esters
11.52	Methyl Octanoate	917	2.97	1125	Esters
11.74	Methyl (Z)-3-octenoate	835	0.07	1131	Esters
12.28	Methyl 7-octynoate	715	0.04	1145	Esters
12.71	Methyl 8-hydroxyoctanoate	670	0.06	1155	Esters
13.46	(E)-5-Undecen-3-yne	771	0.06	1175	Miscellaneous
13.57	1,3,5,8-Undecatetraene	844	0.16	1177	Terpenes and Terpenoids
14.44	Ethyl octanoate	821	0.08	1199	Esters
14.88	Methyl 3-hydroxyhexanoate	674	0.18	1210	Esters
16.01	Methyl-2-methoxyoct-2-enoate	666	0.06	1237	Esters
16.85	3-Methoxy-3-methyl-tetrahydro-pyran-2-one	671	0.18	1258	Ketones
19.17	(3Z)-Heptadeca-1,8,11,14-tetraene	768	0.41	1314	Terpenes and Terpenoids
19.71	Methyl decanoate	825	0.08	1327	Esters
21.81	α-Copaene	934	0.27	1378	Terpenes and Terpenoids
22.47	(-)-β-Elemene	827	0.06	1394	Terpenes and Terpenoids
26.85	α-Ylangene	831	0.06	1503	Terpenes and Terpenoids

[a]Unknown isomer.

[b]Tentatively identified.

SI = Similarity to reference spectrum on a scale of 0–999, with higher scores indicating greater similarity.

Table 7. Identification of volatile compounds in 'Phulae' pineapple pulp stored for 14 days at 10°C + 8 days at 25°C

RT(s)	Name	SI	Concentration (g/L)	LRI	Classification
1.60	Carbon dioxide	847	0.05	-	Miscellaneous
1.78	Methyl acetate	917	0.15	-	Esters
1.96	Ethyl acetate	846	0.09	-	Esters
2.01	Methyl propanoate	859	0.14	-	Esters
2.24	Methyl 2-methylpropanoate	832	0.03	-	Esters
2.46	Methyl butanoate	956	0.59	-	Esters
2.91	Methyl 2-methylbutanoate	922	1.51	-	Esters
3.44	Methyl pentanoate	833	0.04	820	Esters
3.55	2-Methyl-1-pentanol (internal standard)	912	3.30	827	Alcohols and phenols
3.81	Ethyl 2-methylbutanoate	856	0.05	844	Esters
3.84	1-Hepten-4-ol	698	0.03	846	Alcohols and Phenols
4.25	3-Methylbutyl acetate	835	0.03	873	Esters
4.30	2-Methylbutyl acetate	893	0.10	876	Esters
5.22	Methyl hexanoate[a,b]	958	10.52	922	Esters
5.40	Methyl hexanoate[a,b]	786	0.03	930	Esters
5.48	Methyl 3-hexenoate	771	0.04	933	Esters
6.95	β-Pinene[a,b]	807	0.06	992	Terpenes and Terpenoids
7.18	Ethyl hexanoate	885	0.23	1001	Esters
7.97	Methyl heptanoate	916	0.32	1024	Esters
8.04	Methyl 3-methylthiopropanoate[a,b]	916	0.95	1026	Esters
8.10	Methyl 3-methylthiopropanoate[a,b]	685	0.07	1028	Esters
8.15	D-Limonene	692	0.02	1030	Terpenes and Terpenoids
8.41	α-Pinene[a,b]	836	0.11	1037	Terpenes and Terpenoids
8.56	Butanedioic acid, 2-hydroxy-2-methyl-, dimethyl ester, (2R)-	652	0.16	1042	Miscellaneous
8.76	α-Ocimene	897	0.19	1048	Terpenes and Terpenoids
9.10	γ-Hexalactone	733	0.06	1058	Lactones
9.25	4-Methoxy-2,5-dimethyl-3(2H)-furanone	821	0.09	1062	Lactones
9.69	Butane-2,3-diyl diacetate	703	0.06	1075	Esters
10.08	Methyl 2-methylacetoacetate	668	1.04	1087	Esters
10.59	Methyl 2,3-dimethylbutanoate	671	0.21	1102	Esters
11.22	Methyl 4Z-octenoate	882	0.44	1118	Esters
11.55	Methyl octanoate	916	5.94	1126	Esters
13.20	Unknown	725	0.04	1168	Unknown
13.25	Endo-borneol	807	0.04	1169	Miscellaneous
13.49	(E)-5-Undecen-3-yne	770	0.05	1175	Miscellaneous
13.60	1,3,5,8-Undecatetraene	845	0.18	1178	Terpenes and Terpenoids
13.82	Methyl 5-cyclopropylidenepentanoate	744	0.05	1184	Esters
13.93	1-Methylene-1H-indene	838	0.08	1187	Miscellaneous
14.46	Ethyl octanoate	732	0.05	1200	Esters
14.90	Methyl 3-hydroxyhexanoate	690	0.26	1210	Esters
16.03	Methyl-2-methoxyoct-2-enoate	665	0.09	1238	Esters
16.86	3-Methoxy-3-methyl-tetrahydro-pyran-2-one	662	0.28	1258	Ketones
17.09	γ-Octalactone	810	0.09	1264	Lactones
19.19	Methyl 8,11,14,17-eicosatetraenoate	764	0.28	1314	Esters
19.72	Methyl decanoate	864	0.14	1327	Esters
21.83	α-Copaene	944	0.31	1378	Terpenes and Terpenoids
22.50	β-Elemene	871	0.07	1395	Terpenes and Terpenoids
24.22	(+)-Sativene	821	0.04	1438	Terpenes and Terpenoids
26.88	α-Cadinene	821	0.09	1504	Terpenes and Terpenoids

[a]Unknown isomer.

[b]Tentatively identified.

SI = Similarity to reference spectrum on a scale of 0–999, with higher scores indicating greater similarity.

Table 8. Identification of volatile compounds in 'Phulae' pineapple pulp stored for 14 days at 10°C + 10 days at 25°C

RT(s)	Name	SI	Concentration (g/L)	LRI	Classification
1.61	Carbon dioxide	883	0.11	-	Miscellaneous
1.74	Acetone	785	0.03	-	Ketones
1.79	Methyl acetate	920	0.15	-	Esters
1.83	Thiourea	719	0.08	-	Miscellaneous
2.03	Methyl propanoate	878	0.13	-	Esters
2.25	Methyl 2-methylpropanoate	818	0.03	-	Esters
2.47	Methyl butanoate	956	0.59	-	Esters
2.92	Methyl 2-methylbutanoate	933	1.95	-	Esters
3.45	Methyl pentanoate	854	0.05	821	Esters
3.56	2-Methyl-1-pentanol (internal standard)	927	3.30	828	Alcohols and phenols
3.85	Methyl 2-hydroxy-2-methylbutanoate	761	0.04	847	Esters
4.25	3-Methylbutyl acetate	830	0.04	873	Esters
4.30	2-Methylbutyl acetate	887	0.13	876	Esters
5.22	Methyl hexanoate	964	19.71	922	Esters
5.49	Methyl 3-hexenoate	758	0.04	933	Esters
6.32	2-Methylpentyl acetate	774	0.05	967	Esters
6.95	β-Pinene[a,b]	790	0.08	992	Terpenes and Terpenoids
7.18	Ethyl hexanoate	838	0.09	1001	Esters
7.96	Methyl heptanoate	918	0.56	1024	Esters
8.04	Methyl 3-methylthiopropanoate	916	1.41	1026	Esters
8.15	D-Limonene	715	0.03	1030	Terpenes and Terpenoids
8.40	α-Pinene[a,b]	854	0.13	1037	Terpenes and Terpenoids
8.56	Butanedioic acid, 2-hydroxy-2-methyl-, dimethyl ester, (R)-	655	0.22	1042	Miscellaneous
8.76	α-Ocimene	918	0.37	1048	Terpenes and Terpenoids
8.97	2,5-Dimethylfuran-3,4(2H,5H)-dione	791	0.08	1054	Ketones
9.09	γ-Hexalactone	792	0.13	1057	Lactones
9.25	4-Methoxy-2,5-dimethyl-3(2H)-furanone	826	0.10	1062	Lactones
9.69	Butane-2,3-diyl diacetate	691	0.07	1075	Esters
10.07	Unknown	664	1.03	1086	Unknown
10.59	Methyl 2,3-dimethylbutanoate	673	0.32	1102	Esters
10.77	4-[[(2-Methoxy-4-octadecenyl)oxy]methyl]-2,2-dimethyl-1,3-dioxolane	667	0.09	1106	Miscellaneous
11.22	Methyl 4Z-octenoate	884	0.59	1118	Esters
11.54	Methyl octanoate	911	9.12	1126	Esters
11.75	Methyl (Z)-4-octenoate	808	0.05	1131	Esters
13.48	(E)-5-Undecen-3-yne	790	0.10	1175	Miscellaneous
13.59	1,3,5,8-Undecatetraene	854	0.29	1178	Terpenes and Terpenoids
14.89	Methyl 3-hydroxyhexanoate	700	0.66	1210	Esters
16.01	Methyl-2-methoxyoct-2-enoate	654	0.25	1237	Esters
16.85	3-Methoxy-3-methyl-tetrahydro-pyran-2-one	668	0.76	1258	Lactones
17.08	γ-Octalactone	815	0.21	1263	Lactones
19.18	Methyl stearidonate	804	0.40	1314	Esters
19.72	Methyl decanoate	869	0.25	1327	Esters
20.06	(+)-Sativene	799	0.06	1335	Terpenes and Terpenoids
21.82	α-Copaene[a,b]	946	0.75	1378	Terpenes and Terpenoids
22.49	β-Elemene	884	0.20	1394	Terpenes and Terpenoids
23.22	α-Longipinene	821	0.09	1413	Terpenes and Terpenoids
24.00	α-Copaene[a,b]	863	0.06	1432	Terpenes and Terpenoids
24.21	(+)-Sativene	817	0.12	1437	Terpenes and Terpenoids
25.91	α-Copaene[a,b]	863	0.08	1480	Terpenes and Terpenoids
26.88	α-Muurolene	879	0.26	1504	Terpenes and Terpenoids

[a]Unknown isomer.
[b]Tentatively identified.
SI = Similarity to reference spectrum on a scale of 0–999, with higher scores indicating greater similarity.

Table 9. Identification of volatile compounds in 'Phulae' pineapple pulp stored for 14 days (10°C) + 12 days (25°C)

RT(s)	Name	SI	Concentration (g/L)	LRI	Classification
1.60	Carbon dioxide	872	0.11	-	Miscellaneous
1.78	Methyl ethanoate	929	0.16	-	Esters
1.82	Thiourea	674	0.05	-	Miscellaneous
1.96	Ethyl acetate	925	1.14	-	Esters
2.02	Methyl propanoate	823	0.30	-	Esters
2.13	Isopropyl acetate	802	0.05	-	Esters
2.24	Methyl 2-methylpropanoate	880	0.09	-	Esters
2.38	Ethyl propanoate	794	0.08	-	Esters
2.46	Methyl butanoate	923	0.51	-	Esters
2.73	Ethyl 2-methylpropanoate	837	0.15	-	Esters
2.87	Isobutyl acetate	807	0.03	-	Esters
2.91	Methyl 2-methylbutanoate	927	2.87	-	Esters
3.15	Ethyl butanoate	893	0.10	801	Esters
3.56	2-Methyl-1-pentanol (internal standard)	926	3.30	828	Alcohols and phenols
3.81	Ethyl 2-methylbutanoate	948	1.48	844	Esters
4.25	3-Methylbutyl acetate	933	0.20	873	Esters
4.30	2-Methylbutyl acetate	919	0.36	876	Esters
5.22	Methyl hexanoate	950	12.82	922	Esters
6.32	2-Methylpentyl acetate	846	0.11	967	Esters
6.95	β-Pinene[a,b]	866	0.18	992	Terpenes and Terpenoids
7.19	Ethyl hexanoate	909	0.78	1001	Esters
7.43	(Z)-3-Hexenyl acetate	802	0.09	1008	Esters
7.97	Methyl heptanoate	893	0.46	1024	Esters
8.05	Methyl 3-methylthiopropanoate	878	0.50	1027	Esters
8.11	2-Ethylhexyl acetate	686	0.16	1028	Esters
8.16	D-Limonene	831	0.29	1030	Terpenes and Terpenoids
8.42	α-Pinene[a,b]	891	0.30	1038	Terpenes and Terpenoids
8.57	Dimethyl 2-hydroxy-2-methylbutane-1,4-dioate	649	0.12	1042	Esters
8.77	α-Ocimene	921	0.30	1048	Terpenes and Terpenoids
9.11	γ-Hexalactone	786	0.08	1058	Lactones
9.25	4-Methoxy-2,5-dimethyl-3(2H)-furanone	842	0.23	1062	Lactones
9.70	2,3-Diacetoxybutane	871	0.23	1075	Miscellaneous
10.09	Methyl 2,3-dimethylbutanoate	657	0.91	1087	Esters
10.23	Terpinolene	811	0.05	1091	Terpenes and Terpenoids
10.60	Methyl 2,3-dimethylbutanoate	670	0.38	1102	Esters
11.23	Methyl 4Z-octenoate	876	0.30	1118	Esters
11.55	Methyl octanoate	918	7.65	1126	Esters
12.73	Ethyl 2-methylacetoacetate	659	0.13	1156	Esters
13.21	Unknown	682	0.11	1168	Unknown
13.49	(E)-5-Undecen-3-yne	804	0.10	1175	Miscellaneous
13.60	1,3,5,8-Undecatetraene	860	0.25	1178	Terpenes and Terpenoids
14.47	Ethyl octanoate	854	0.16	1200	Esters
14.91	Methyl 3-hydroxyhexanoate	682	0.14	1211	Esters
16.88	3-Methoxy-3-methyl-tetrahydro-pyran-2-one	652	0.12	1258	Lactones
17.11	γ-Nonalactone	777	0.07	1264	Lactones
19.19	Methyl stearidonate	801	0.28	1314	Esters
19.73	Methyl decanoate	852	0.12	1327	Esters
21.84	α-Copaene	943	0.58	1379	Terpenes and Terpenoids
22.50	β-Elemene	853	0.09	1395	Terpenes and Terpenoids
26.89	α-Muurolene	884	0.13	1504	Terpenes and Terpenoids

[a]Unknown isomer.

[b]Tentatively identified.

SI = Similarity to reference spectrum on a scale of 0–999, with higher scores indicating greater similarity.

Table 10. Identification of volatile compounds in 'Phulae' pineapple pulp at full mature stage

RT(s)	Name	SI	Concentration (g/L)	LRI	Classification
1.74	2-Bromooctadecanal	738	0.04	-	Aldehydes
1.92	Unknown	699	0.18	-	Unknown
2.18	Methyl 12-(2-octylcyclopropyl)dodecanoate[a,b]	725	0.04	-	Esters
2.66	Methyl butanoate	869	0.51	-	Esters
3.14	Methyl 2-methylbutanoate	796	0.34	801	Esters
3.86	2-Methyl-1-pentanol (internal standard)	923	3.30	848	Alcohols and phenols
5.55	Methyl hexanoate	922	2.09	936	Esters
6.71	Hexyl octadec-9-enoate	756	0.04	982	Esters
8.63	Methyl 12-(2-octylcyclopropyl)dodecanoate[a,b]	705	0.17	1044	Esters
8.69	(3Z)-1,4,6,9-Nonadecatetraene	782	0.14	1046	Terpenes and Terpenoids
9.05	α-Ocimene	892	0.94	1056	Terpenes and Terpenoids
10.77	Z-8-Metyl-9-tetradecenoic acid	717	0.06	1106	Miscellaneous
11.99	Methyl octanoate	790	0.27	1137	Esters
19.44	Methyl 12,15-octadecadiynoate	782	0.04	1320	Esters
19.69	(E)-10-Heptadecen-8-ynoic acid methyl ester	789	0.05	1326	Esters
22.11	Methyl 12,15-octadecadiynoate	784	0.10	1385	Esters
30.04	9-Hexadecenoic acid[a,b]	799	0.10	1587	Miscellaneous
30.06	9-Hexadecenoic acid[a,b]	789	0.03	1587	Miscellaneous
30.08	9-Hexadecenoic acid[a,b]	797	0.05	1588	Miscellaneous

[a]Unknown isomer.
[b]Tentatively identified.
SI = Similarity to reference spectrum on a scale of 0–999, with higher scores indicating greater similarity.

Effect of harvest maturity and storage condition on changes of volatile compounds

Normally, fruit should be harvest at optimal maturity stage to get higher amounts of volatile compounds. The fruit was harvested at optimal maturity stage was contain higher amounts of volatile compounds (Fellman et al., 2003). In vice versa, immature fruit will produce low quantity of volatile compounds and initial precursor of volatile compounds in unsuitable harvest fruit was changed from ester to fatty acid. In the immature fruit, aldehyde and alcohol were produced from enzymatic breakdown of fatty acids without ester. During ripening, level of C_6 compounds decreased rapidly with increased ester and lactone (Ménager et al., 2004). According to the literatures, the result showed that, the pineapples harvested at full maturity stage had esters higher than fatty acids in both qualitative and quantitative when compared to the pineapples harvested at green mature stage. Additionally, the results also showed that aldehyde and alcohol were detected in green mature pineapples stored for 14 days at 10°C and after moving to store at 25°C for 2 and 4 days by amount of aldehyde and alcohol were higher than full mature pineapples. For 'Phulae' pineapples, which is a chilling sensitive fruit. Low temperature also affected to accumulation of terpenes and terpenoids including its derivatives as showed on Table 4 by the pineapples stored for 14 days at 10°C + 2 days at 25°C had terpenes and terpenoids higher than esters (Tietel et al., 2012).

CONCLUSIONS

Harvest maturity and storage condition affected to development of volatile compounds in the pineapple fruit especially types of volatile compounds and their derivatives. Full mature fruit contained higher amounts of volatile compounds, when compared with amounts of volatile compounds in green mature fruit. Major volatile compounds, which is characteristics of pineapple such as Methyl 3-methylthiopropanoate, γ-octalactone, γ-nonalactone, 1,3,5,8-undecatetraene were detected in green mature pineapples. In vice versa, major volatile compound in full mature pineapples were Methyl hexanoate.

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