http://www.e-journals.in
DOI:10.7598/cst2015.1068

Chemical Science Transactions
2015, **4(3),** 663-667

RESEARCH ARTICLE

GC-MS and ^{1}H NMR Analysis of Fatty Acids in Monthong Thai Durian (*Durio Zibethinus,* Murr)

WEERACHAI PHUTDHAWONG[1], PERMSAK CHAIRAT[2], KRITCHASORN KANTAROD[2] and WAYA PHUTDHAWONG[*2]

[1]Department of Chemistry, Faculty of Liberal Arts and Science, Kasetsart University, Kamphaeng Saen Campus, Kamphaeng Saen, Nakhon Pathom, 73140, Thailand

[2]Department of Chemistry, Faculty of Science, Silpakorn University, Nakhon Pathom, 73000, Thailand

sengwaya@su.ac.th

Received 21 May 2015 / Accepted 4 June 2015

Abstract: This study was undertaken to determine the fatty acid composition of Monthong durian (*Durio zibethinus,* Murr.) clones. The extract of Monthong Thai durian was analyzed by ^{1}H NMR and combined gas chromatography and mass spectrometry (GC-MS). The GC-MS data were used to identify 15 methyl esters of the durian extract after transesterification using sodium in methanol. The most prominent components found were 9-octadecenoic acid(Z) methyl ester (26.22%), cholest-5-en-3-ol(3.beta) (24.95%), vitamin E (15.40%), hexadecanoic acid methyl ester (6.37%), 9,12-octadecenoic acid(Z,Z) methyl ester (5.88%), 9-octadecenoic acid(E) methyl ester (5.00%) and ethyl oleate (4.08%). The ^{1}H NMR data, signals for a methoxy group of transesterification oil at δ 3.60-3.75 was readily discernible.

Keywords: Durian, Fatty acids, GC-MS, ^{1}H NMR

Introduction

Durian (*Durio zibethinus,* Murr.) is a perennial tree that can be cultivated in tropical rain forest especially in the Eastern and Southern of Thailand. Monthong (Golden pillow) is the most popular variety and it was reputed to be the “King of tropical fruits” due to it is a highly prized fruit gastronomically, culturally, and economically in Thailand[1]. Borneo is thought to be the origin center and diversity of durian; however, it has spread throughout South-East Asia and is now extensively cultivated in Malaysia, Thailand, Indonesia, Brunei, Cambodia, Vietnam, and the Southern Philippines[2]. Several papers have reported their chemical content[3-6] including fatty acids[7-12], including the determination and relation of lipid content and fatty acid composition. The report was found that durian contained a higher proportion of lipids but lower unsaturated fatty acid contents such as palmitic and palmitoleic acid[13]. However, as far as we know, no study has yet been carried out on Thai durian Monthong variety especially using ^{1}H NMR combined GC-MS. We report here the fatty acid analysis results of 'Monthong' durian by ^{1}H NMR and GC–MS.

Experimental

'Monthong' durian fruits were obtained from a local market in Nakorn Pathom, Thailand, which was supplied from Supanburi province in February 2015.

Extraction

Durian clones without seed were digested and 50 g of the digested aril were extracted with 150 mL of hexane by soxhlet extraction for 10 h. The solvent was dried over anhydrous magnesium sulphate ($MgSO_4$) and evaporated solvent out to obtain 0.099 g of oil (0.198%).

Transesterification

0.099 g of oil with excess methanol (10 mL) was added a small amount of sodium (~0.05 g) and left stirred 6 h. The solution was extracted with 3×20 mL of dichloromethane and washed with water several times and dried over anhydrous magnesium sulphate ($MgSO_4$). The dichloromethane layer was evaporated to give 0.062 g of oil which was a mixture of methyl esters.

GC-MS Analysis

The oil obtained from the transesterification was analyzed by GC–MS using an Agilent 6890(GC)/HP 5975(MS) GC-MS system. Separation was achieved using helium as the carrier gas (ca.1 mL/min) with a fused silica capillary column (DB-5), 30 m long, 0.25 mm *i.d.* and 0.25 µm film thickness. The injector and detector temperatures were 250 °C and 260 °C respectively; oven temperature programme: 1 min isothermal at 40 °C, then at 10 °C/min to 260 °C (5 min isothermal). The MS instrument was operated in full scan and in electron impact ionization mode (70 eV).

^{1}H NMR Analysis

^{1}H NMR spectra of the oil obtained from the transesterification was recorded in (D) chloroform solutions at 300 MHz with a Bruker AVANCE 300 spectrometer. Tetramethylsilane was used as the internal standard.

Results and Discussion

The ^{1}H NMR data, signals for a methoxy group of transesterification oil was readily discernible at δ 3.60-3.75. This data was confirmed that the transesterification occurred completely. A typical gas chromatogram of the transesterified oil is shown in Figure 1. The compounds corresponding to the various peaks are listed in Table. Their chemical structures are also shown below.

It is clear from Table 1 and Table 2 that, in the Monthong durian (*Durio zibethinus*, Murr.) oil after transesterification, 15 methyl esters prominent peaks can be distinguished and identified. The most prominent components found were 9-octadecenoic acid(Z) methyl ester (26.22%), cholest-5-en-3-ol(3.beta) (24.95%), vitamin E (15.40%), hexadecanoic acid methyl ester (6.37%), 9,12-octadecenoic acid(Z,Z) methyl ester (5.88%), 9-octadecenoic acid(E) methyl ester (5.00%) and ethyl oleate (4.08%). The ^{1}H-NMR data, signals for a methoxy group of transesterification oil at δ 3.60-3.75 was also readily discernible, which was confirmed that the transesterification occurred completely.

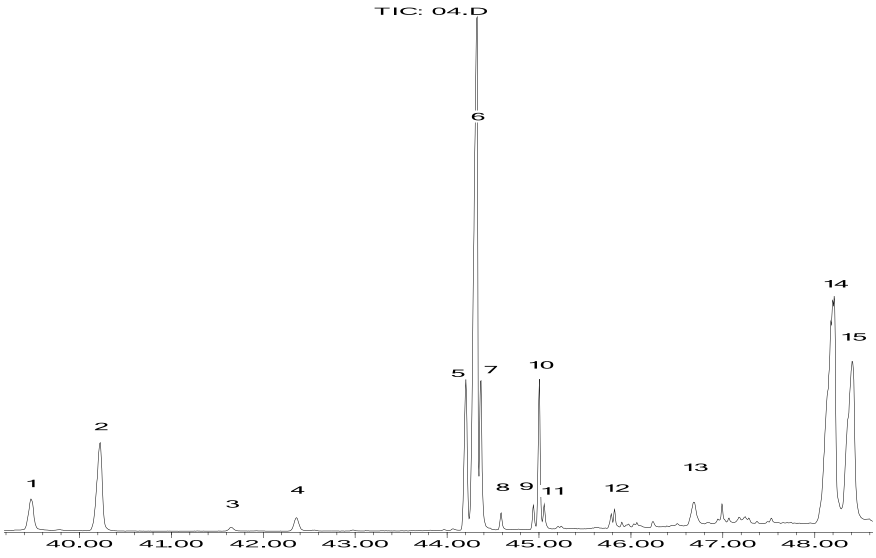

Figure 1. Gas chromatogram of the transesterified durian oil

Table 1. Components in the transesterified of durian oil

Peak no.	Compounds	RA[a] %	MW[b]	Quality %[c]	Identification[d]
1	9-Hexadecenoic acid(Z) methyl ester (Palmitoleic acid methyl ester)	2.239	268.24	99	1,2
2	Hexadecanoic acid methyl ester (Palmitic acid methyl ester)	6.379	270.26	99	1,2
3	Ethyl-9-hexadecenoate	0.259	282.26	99	1,2
4	Hexadecanoic acid ethyl ester (Palmitic acid ethyl ester)	0.864	284.27	99	1,2
5	9,12-Octadecenoic acid(Z,Z) methyl ester Linoleic acid methyl ester	5.884	294.26	99	1,2
6	9-Octadecenoic acid(Z) methyl ester(6) Oleic acid methyl ester	26.225	296.27	99	1,2
7	9-Octadecenoic acid(E) methyl ester Oleic acid methyl ester	5.000	296.27	99	1,2
8	Octadecanoic acid methyl ester Stearic acid methyl ester	0.526	298.29	98	1,2
9	9,12-Octadecenoic acid ethyl ester Linoleic acid ethyl ester	0.665	308.27	99	1,2
10	Z-Ethyl oleate	4.081	310.29	99	1,2
11	E-Ethyl oleate	0.764	310.29	99	1,2
12	5,8,11,14,17-Eicosapentaenoic acid(all-Z) methyl ester	0.542	316.24	91	1,2
13	4,7,10,13,16,19-Docosahexaenoic acid(all-Z) methyl ester	0.718	342.26	95	1,2
14	Cholest-5-en-3-ol(3.beta)	24.951	386.36	99	1,2
15	VitaminE	15.408	430.38	99	1,2

[a]RA, relative area (peak area relative to total peak area) [b]molecular weight from GC-MS (EI) data [c]MS quality comparison with database [d]1, based on comparison of mass spectra from Wiley library; 2, based on comparison of mass spectra from NIST library

The mass spectra were recorded on a Polaris Q or Hewlett Packard 5973 mass spectrometer. The EI-MS fragmentation data of the compounds from Table 1 are shown in Table 2.

Table 2. EI-MS fragmentation data of the compounds

Peak no.	The EI-MS fragmentation data (Fragmentation : Percentage ratio)
1	32(10.8),41(74.4),55(100),69(61.6),74(56.0),83(39.2),91(4.0),96(34.4),101(5.6), 110(16.0),115(5.6),123(12.8),128(4.0),137(7.2),142(3.2),147(3.2),152(10.4),165 (2.4),194(10.4),207(2.4),236(16.0),268(3.2)
2	43(35.5),55(26.4),69(13.6),74(100),81(8.2),87(64.0),97(5.6),129(5.6),143(13.6), 171(3.2),185(4.0),199(3.2), 227(8.8),239(4.0),270(7.2)
3	32(83.2),39(16.0),44(100),55(86.4),60(10.4),65(3.6),69(56.0),73(18.4),79(10.4), 83(37.6),88(38.4),93(5.6),97(39.2),101(27.2) , 107(3.2),111(16.0),115(8.0),119(4.8),123(14.4),129(4.8),133(4.0),137(8.0),141 (4.0),147(3.2),152(7.6),194(11.6),207(10.4), 237(12.8),281(4.8)
4	32(12.8),39(4.4),43(41.2),55(30.4),61(12.0),69(16.0),73(19.2),83(10.0),88(100), 97(7.2),101(52.0),111(2.4),115(5.6), 129(2.4),143(4.8),157(12.0),185(2.8),199(2.8),213(2.4),241(8.0),284(8.0)
5	41(60.8),45(2.4),55(66.4),67(100),71(3.2),77(12.8),81(81.6),87(7.2),91(10.0),95 (52.8),105(2.4),109(22.8), 115(2.4),123(15.2),135(8.8),150(8.8),164(5.6),178(3.2),263(6.4),294(8.0)
6	41(73.6),45(2.4),55(100),59(20.0),65(3.2),69(62.4),74(50.0),79(13.6),83(45.6),87 (38.4),93(6.4),97(39.2),101(6.4),110(22.4),115(6.4), 123(14.4),128(4.0),133(3.2),137(8.4),141(6.4),147(2.4),152(5.6),166(4.8),180 (9.6), 222(15.2),235(2.4),264(20.8),296(3.2)
7	41(67.2),45(2.4),55(100),59(18.4),65(2.4),69(62.4),74(49.6),83(40),87(34.4),93 (4.0),97(36.0),101(4.8),110(16.0),115(4.8),119(2.4),123(10.4),129(3.2),137(7.2), 141(5.2),152(5.2),166(4.0),180(7.2),222(9.6),264(16.8),296(2.4)
8	32(14.4),43(41.6),55(32.0),69(16.0),74(100),81(4.0),87(64.8),97(8.0),111(3.2),129 (5.6),143(15.2),185(2.4),199(6.4),213(2.4),241(2.4),255(9.6),267(4.0),298(10.8)
9	32(14.11),41(64.12),55(74.71),67(100),81(78.24),95(50.59),109(25.29),123(11.76), 135(10.59),150(8.82),164(5.88),178(4.12)207(4.12),220(3.53),263(8.82), 281(2.35), 308(5.88),355(0.58)
10	41(69.41),55(100),69(61.76),83(47.06),97(38.82),111(17.65),123(12.35),137(7.65), 155(6.47),166(3.53),180(9.41),222(10.59),264(16.47),310(3.53)
11	32(14.71),41(67.65),55(100),69(67.65),83(47.06),97(41.18),111(17.06),123(11.76), 137(5.88),155(5.88),180(8.24),207(2.35),222(10),265(14.11),281(2.94)
12	32(15.88),14(65.88),55(44.12),67(74.12),79(100),91(88.24),105(37.06),117(34.12), 131(22.94),145(14.12),159(9.41),171(5.88),185(5.29),199(4.71),200(14.12),281 (8.24),416(20)
13	32(16.0),41(64.8),55(44.8),67(77.4),73(7.20),79(100),85(16.0),91(89.6),97(8.8), 105(40),111(5.6),117(32.0),123(3.2),131(24.0),137(2.4),145(12.8),171(6.4),185 (4.8),199(4.0),207(16.8),281(8.0)
14	43(100),55(67.06),67(38.24),81(54.71),95(52.94),105(55.88),119(36.47),133 (32.35),145(47.06),159(32.35),173(16.47),185(10.59),199(12.94),213(35.29), 231(16.47),247(10.59),255(2.35),275(55.88),301(47.06),326(7.06),353(32.35), 368(36.47),386(77.65)
15	41(20.59),57(10),69(5.29),81(4.12),91(4.12),107(1.76),121(6.47),136(3.53),149 (1.76),165(100),177(1.76),189(1.76),205(10),430(94.12)

Acknowledgement

The authors are grateful to the Biology Department, Maejo University for the provision of the GC-MS facilities. We would also like to thank the development and promotion for Science and technology talents project and the science achievement scholarship of Thailand for providing a grant to P. C. and K. K., a final year chemistry undergraduate students.

References

1. Jingtair S, Postharvest Biology and Technology of Tropical and Subtropical Fruits: Cocona to Mango. 2011, A volume in Woodhead Publishing Series in Food Science, *Technology and Nutrition*, 80–114.
2. Nanthachai S, *ASEAN Food Handling Bureau*, 1994.
3. Voon Y Y, Hamid N S A, Rusul G, Osman A, Quek S Y, *Food Chem.,* 2007, **103**, 1217-1227; DOI:10.1016/j.foodchem.2006.10.038
4. Stanton W R, *Trop Sci.,* 1966, **8**, 6-10.
5. Liu Q, Zhou J, Xie M D, Zhu Y F and Huang W Y, *Fenxi Ceshi Xuebao*, 1999, **18**, 58.
6. Weenen H, Koolhaas W E and Apriyantono A, *J Agri Food Chem.,* 1996, **44(10)**, 3291-3293; DOI:10.1021/jf960191i
7. Siondalski P and Lysiak-Szydlowska W, Food components in the protection of the cardiovascular system. In Sikorski Z E, (Ed.), Chemical and functional properties of food components, Boca Raton, FL: CRC Press LLC, 2007, 3rd Ed., 439-450.
8. Berry S K, *J Am Oil Chem Soc.,* 1981, **58(6)**, 716-717; DOI:10.1007/BF02899460
9. Berry S K, *Lipids,* 1980, **15(6)**, 452-455.
10. Eni S R, *Farmasi Indonesia,* 2001, **12**, 66.
11. Naef R and Velluz A, *Flavour Fragr J.,* 1996, **11(5)**, 295-303; DOI:10.1002/(SICI)1099-1026(199609)11:5<295::AID-FFJ585>3.0.CO;2-4
12. Lee-Hoon H and Rajeev B, *Food Chem.,* 2015, **168**, 80-89; DOI:10.1016/j.foodchem.2014.07.020
13. Liu Q, Zhou J, Xie M, Zhu Y and Huang W, *Fenxi Huaxue,* 1999, **27**, 320.