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Change in fatty acid profile, volatile compounds and FTIR spectra of samrong seed oil during storage

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Abstract Samrong (*Sterculia foetida* Linn.) is a wild plant. It is used in traditional medicine. The kernels of samrong seeds have been reported to contain a high amount of oil. The changes in fatty acid profile, volatile compounds, and fourier transform infrared (FTIR) spectra of oil from samrong seed kernel during storage (48 days) were investigated. With increasing storage time, the content of saturated fatty acid (SFA) and polyunsaturated fatty acid (PUFA) decreased. There was little change in fatty acid profile at the end of storage. As to FTIR spectra, a slight change in wave number from 3600 cm^{-1} to 3200 cm^{-1} and wave number 1711 cm^{-1} was observed with increasing storage time. After storage, the results observed 12 volatile lipid oxidation compounds. However, slight changes in abundance of volatile compounds were found. Therefore, samrong seed kernel oil had good oxidative stability.

Keywords: Samrong seed, Fatty acid profile, Volatile compound, FTIR

Introduction

Samrong (*Sterculia foetida* Linn.) is a wild plant found in tropical and subtropical countries. It is good potential raw material for the production of oil. Oil can be prepared from the kernels of samrong seeds (Figure 1) and this oil can be used as medicine as it exhibits antimicrobial, antiviral, antitumor, and insecticidal properties (Guerere *et al.*, 1985). Samrong seed kernels have been reported to contain about 53.65% golden yellow oil. The main constituents found in oil from samrong seed kernels were poly-unsaturated fatty acid (PUFA), followed by saturated fatty acid (SFA) and monounsaturated fatty acid (MUFA), respectively (Chanyawiwatkul *et al.*, 2018). Moreover, Kale *et al.* (2011) presented that the main unsaturated fatty acids (UFA) and SFA found in samrong seed oil were oleic acid (20.50%) and palmitic acid (11.87%), respectively. PUFAs are very susceptible to oxidative deterioration. It results in changes in

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physicochemical properties of oil (Fennema, 1996). Lipid oxidation is the main problem of oil, which is related to the shelf-life of oils. However, Chanyawiwatkul *et al.* (2018) reported that oil from samrong seed kernels contained α-tocopherol (0.594 ± 0.1 mg/kg of oil) and phenolic compounds (7.2 ±0.15 mg GAE/g oil). The presence of indigenous α-tocopherol and phenolic compounds plays a crucial role as these are antioxidants and might affect the oxidative stability of samrong seed oil during storage. There are several methods for monitoring the oxidation of oils that indicate changes in chemical and physical reactions of oils or analyze oxidation compounds at the different reaction stages of the process (Crapiste *et al.*, 1999). Gas chromatography (GC) with flame ionization detector (FID), GC with mass spectrometry (MS) and fourier transform infrared (FTIR) spectroscopy were used as effective analytical tools to determine the oxidation deterioration of oil during storage (Yun and Surh, 2012; Kim *et al.*, 2018). The fatty acid profile and the volatile compounds were determined using GC-FID and GC-MS, respectively. For FTIR spectroscopy, FTIR was used to identify the type and functional groups in oil. However, a little information about the use of GC-FID, GC-MS, and FTIR spectroscopy for the determination of compounds in samrong seed kernel oil has been studied. Thus, this study aimed to investigate the changes in fatty acid profile, volatile lipid oxidation compounds, and FTIR spectra on the oxidation of samrong seed kernel oil during a storage period of 48 days at 30 ℃.

Figure 1. Samrong seed kernels

Materials and Methods

Chemicals

Hexane was procured from Labscan Asia Co, Ltd (Bangkok, Thailand). The other reagents with the analytical grade were used.

Preparation of samrong seed oil

Samrong seeds were obtained from a Siamjannin company, Thailand. Shells of samrong seeds were removed from the kernels. Seed kernels were finely ground using a Phillips blender for 1 min. The seed kernel powder was kept at -18 ℃. The seed kernel powder was subjected to chemical analyses.

Extraction of samrong seed oil

Oil of samrong kernel was drawn out from the seed kernel powder following the procedure of Chanyawiwatkul *et al.* (2018). Seed kernel powder (50 g) was mixed with cold hexane (250 ml) at the speed of 9500 rpm for 3 min using a homogeniser. The oil extracts were filtered through filter paper no.4 (Whatman International Ltd., Maidstone, England). The residue was dissolved in cold hexane (two times). All extracts were added with anhydrous sodium sulphate (3-6 g) and poured into a round-bottom flask using Whatman no.4 filter paper. The hexane was evaporated at 45 ℃ using a rotary evaporator (Tokyo Rikakikai, Co. Ltd, Tokyo, Japan) and the residual hexane was eliminated by nitrogen flushing. The obtained seed kernel oils were placed at 30±1 ℃ and were taken for analyses at day 0 and day 48 of storage.

Fatty acid profile

Fatty acid profile in samrong seed kernel oil was analysed as fatty acid methyl esters (FAMEs) according to the method described by AOAC (2000). FAMEs of samples were injected to the gas chromatogram (Shimadzu, Kyoto, Japan) equipped with the flame ionisation detector (FID). Retention times of FAMEs in the oil sample were determined using the retention times of FAME standards. The standards were used to calculate as g fatty acid/100 g oil based on its peak area.

Fourier transform infrared (FTIR) spectra

The spectra of the oil samples were acquitted on a horizontal attenuated total reflectance (ATR)-Fourier transform infrared (FTIR). IR spectra were recorded in the mid-IR region of 500-4000 cm^{-1} with a resolution better than 4 cm^{-1} in 16 scans. The oil sample (200 µl) was place on the plate crystal cell. The cell was fixed with the mount of the FTIR spectrometer. Data analysis was done by the software program (Bruker Co., Ettlingen, Germany).

Volatile compounds

Oil samples were analysed for the volatile compounds at day 0 and day 48 of storage. A headspace solid-phase micro-extraction gas chromatography mass spectrometry (SPME GC/MS) was conducted according to the method of Iglesias and Medina (2008) with slight modification. Identification of the components was carried out using library data from the software of GC/MS system. This results were performed based on the retention time. Quantitative analyses of samrong seed kernel oil components were reported in terms of abundance of each identified lipid oxidation compound.

Statistical analysis

The data were subjected to ANOVA procedure. Comparison of means was analysed using Duncan's multiple range test. The t-test was used for pair comparison. Statistical analysis was performed using the Statistical Package for Social Science (SPSS for windows, SPSS, Inc., Chicago, IL, USA).

Results

Fatty acid profiles

Table 1 presents the fatty acid profiles of oils from samrong seed kernels stored for 0 and 48 days at 30 ℃. Oil from samrong seed kernel contained a total of monounsaturated fatty acid (MUFA) (5.30%), saturated fatty acid (SFA) (27.32%) and polyunsaturated fatty acid (PUFA) (55.95%) at day 0. Oils from seed kernel contained gamma-linoleic acid (C18:2) (47.80%). It was the major abundant fatty acid, followed by palmitic acid (C16:0) (16.49%), stearic acid (C18:0) (10.45%), linoleic acid (C18:2) (6.48%) and oleic acid (C18:1) (4.96%). Oils from samrong seed kernel had eicosapentaenoic acid (EPA) contents of 0.55 g/100 g oil. After 48 days of storage, SFA MUFA and PUFA in samrong seed kernel oil were 26.59%, 5.71%, and 54.67%, respectively. Gamma-linoleic acid was the major component in samrong seed kernel oil at 48 days of storage, having a concentration of 44.36%, followed by palmitic acid (16.49%), stearic acid (9.71%), linoleic acid (8.13%), and oleic acid (5.36%).

FTIR spectra

The entire FTIR spectra range of samrong seed kernel oil stored in room temperature at day 0 and day 48 are shown in Figure 2. There was a major absorption peaks at a spectral range of 3050-2800 cm^{-1}. Additionally, the peaks can be detected at 1117 and 1099 cm^{-1}. At 48 days of storage, a

larger peak at interval of 3400-3600 cm^{-1} was found. The lower amplitude of the peak at wave number 2853 cm^{-1} was found after storage. Peaks at wave number 1743 cm^{-1} were found in samrong seed kernel oil.

Table 1. Fatty acid profile of samrong seed kernel oil before and after storage for 48 days at 30 ℃

Fatty acids (g/100 g oil)	Samrong seed kernel oil	
	Day 0 ¨	Day 48
Myristic acid (C14:0)	0.12 ±0.00*a+	0.12 ±0.00a
Pentadecanoic acid (C15:0)	0.03 ±0.00a	0.03 ±0.00a
Palmitic acid (C16:0)	16.49 ±0.10a	16.49 ±0.06a
Palmitoleic acid (C16:1, n-7)	0.17 ±0.00a	0.18 ±0.01a
Heptadecanoic acid (C17:0)	0.06 ±0.00a	0.06 ±0.00a
Steric acid (C18:0)	10.45 ±0.19a	9.71 ±0.41b
Oleic acid (C18:1, n-9)	4.96 ±0.03b	5.36 ±0.02a
Linoleic acid (C18:2, n-6)	6.48 ±0.03b	8.13 ±0.01a
α-Linolenic acid (C18:3 n-3, ALA)	0.17 ±0.00a	0.19 ±0.02a
γ-Linolenic acid (C18:3 n-6)	47.80 ±1.34a	44.36 ±0.25b
Arachidic acid (C20:0)	0.10 ±0.00a	0.10 ±0.01a
Gadoleic acid (C20:1, n-9)	0.13 ±0.00a	0.13 ±0.00a
Eicosadienoic acid (C20:2, n-6)	0.78 ±0.05b	1.09 ±0.12a
Dihomo-gamma-linolenic acid (C20:3, n-6)	0.03 ±0.01a	0.04 ±0.01a
Arachidonic acid (C20:4 n-6, ARA)	0.14 ±0.01a	0.13 ±0.00a
Eicosapentaenoic acid (C20:5 n-3, EPA)	0.55 ±0.07a	0.66 ±0.22a
Behenic acid (C22:0)	0.02 ±0.00b	0.03 ±0.00a
Lignoceric acid (C24:0)	0.04 ±0.00a	0.04 ±0.00a
Nervonic acid (C24:1)	0.04 ±0.00a	0.03 ±0.00a
Saturated fatty acid (SFA)	27.32 ±0.20a	26.59 ±0.16b
Mono-unsaturated fatty acid (MUFA)	5.30 ±0.20b	5.71 ±0.17a
Poly-unsaturated fatty acid (PUFA)	55.95 ±0.19a	54.67 ±0.14b

* Values are expressed as means ± standard deviation (n = 3).
+Different letters in the same row indicate significant differences ($p < 0.05$).
¨Referred from result of Chanyawiwatkul *et al.* (2018)

Volatile compounds

The volatile compounds in samrong seed kernel oil at 0 and 48 days of storage is presented in Table 2. A total of 14 volatile compounds was used as indicators of oxidation. The oxidation volatile compounds found in this study were 1) alcohols (2-methyl-3-hexanol, 2-hexanol, 1-methyl-cyclopentanol, 1-hexanol, nonanol, 1-octanol, and 1-nonanol); 2) aldehydes (octanal and benzaldehyde); and 3) ketones (2-hexanone, 2-heptanone, 2-octanone, 2-decanone and 5-ethyldihydro-2(3H)-furanone). At day 0, twelve volatile compounds were identified in oil from samrong seed. The major volatile compounds found in samrong seed kernel oil were 1-octanol, followed by 2-methyl-3-hexanol and 2-hexanol. However, octanal benzaldehyde and 1-nonanol were obtained only in the initial sample. After 48 days at 30°C, 2-heptanone and 2-octanone were identified as new volatiles in samrong seed oil. 2-octanone, 2-methyl-3-hexanol, and 1-octanol were the dominant volatile compounds.

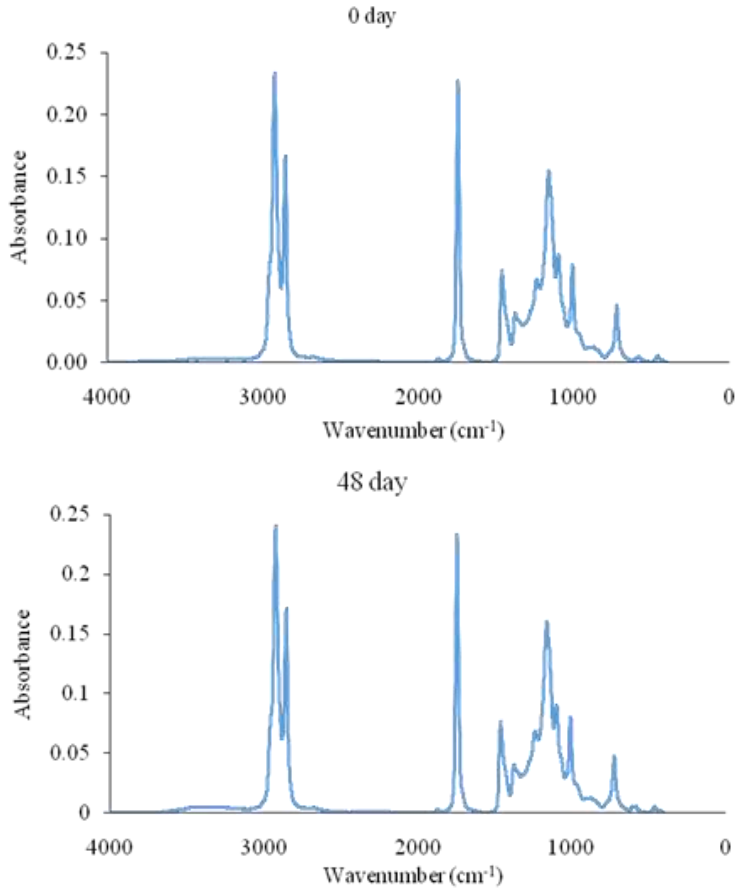

Figure 2. FTIR spectra of oils extracted from samrong seed kernel stored at 30 ℃ for 0 and 48 days

Table 2. Volatile compounds in samrong seed kernel oil stored at 30 ℃ for 0 and 48 days

Compounds	**Peak area (Abundance) × 10^5**	
	Day 0	**Day 48**
2-Hexanone	3.25	6.15
2-Heptanone	ND	4.5
2-Methyl-3-hexanol	23.23	38.96
2-Hexanol	11.87	13.93
1-Methyl-cyclopentanol	2.8	3.19
2-Octanone	ND	41.21
Octanal	4.75	ND
1-Hexanol	3.92	4.81
Nonanol	1.37	0.99
2-Decanone	2.03	1.82
Benzaldehyde	2.39	ND
1-Octanol	35.18	24.76
1-Nonanol	0.47	ND
5-ethyldihydro-2(3H)-Furanone	0.62	0.82

ND: non-detectable

Discussion

Fatty acid profiles

Oil from samrong seed kernels had the highest PUFA. Orsavova *et al.* (2015) reported that the major fatty acids of plant oils was PUFAs. Linoleic acid was also found as the predominant PUFA in oil from samrong seed kernel, while palmitic acid was the major SFA. Vipunngeun and Palanuvej (2009) reported palmitic acid as the major fatty acid. The study of Kale *et al.* (2011) found that the major SFA and unsaturated fatty acids (UFA) of samrong seed oil were palmitic acid and oleic acid, respectively. Genetic and environmental factors affect the fatty acid composition in plant oils (Hemingway *et al.*, 2015). Moreover, the extraction method and the solvent used also influence fatty acid composition in oil (Ghazani *et al.*, 2014).

At 48 days of storage, little changes in fatty acid profiles were observed. Oleic acid, linoleic acid, eicosadienoic acid and behenic acid increased as storage period increased. This coincided with decreases in steric acid (SFA) and γ-linolenic acid (PUFA). A decrease in PUFA, especially γ-linolenic acid, was found during storage. γ-Linolenic acid decreased by 7.20% at day 48. The decrease in γ-linolenic acid content was due to susceptibility of samrong seed kernel oil to lipid oxidation during storage period. At day 48, SFA and PUFA contents decreased by 2.67% and 2.29%, respectively, whereas MUFA content increased by 7.74%, compared with that observed at day 0. PUFAs were generally more towards oxidation than MUFAs. Changes in fatty acids occurred in oil from samrong seed kernels. PUFAs slightly decreased during storage for 48 days at 30 ℃. Takeungwongtrakul *et al.* (2012) indicated that PUFA in oil decreased during oil storage because of lipid oxidation of oil. However, oils from samrong seed kernels showed slight changes in fatty acid profile.

FTIR spectra

Mid-infrared spectra in 2800-3050 cm^{-1} region were observed as the prominent peaks, corresponding to the stretching vibrations of C-H, which overlap with a spectral range of 2400 – 3100 cm^{-1}. This range is assigned to the -OH group in carboxylic acids. The peaks intensities at 1163 and 1237 cm^{-1} were due to the stretching vibration of the C−O ester groups and the bending vibration of the CH_2 groups. The stretching vibration of the C−O ester groups appeared at 1118 cm^{-1} and 1097 cm^{-1} (Guillen and Cabo, 1997). The spectral analysis indicated the presence of ester bonds between long chain fatty acids and a glycerol backbone of phospholipids or triglyceride. The peak intensities around 3442 cm^{-1} and 3470 cm^{-1} showed the −OO−H stretching vibrations of hydroperoxide group as the oxidative products formed (Ogbu and Ajiwe, 2016; Rohman and Che-Man, 2013).

The results showed that the absorbance changes of peaks between 3462 cm^{-1} and 3470 cm^{-1} were increased. This suggested that the hydroperoxides were found in samrong seed kernel oil within the first 48 days of storage. The ratio between the absorbance band at 2854 cm^{-1} and the absorbance band at 3600-3100 cm^{-1} ($A_{2854}/A_{3600–3100}$) could determined the oxidation of oil (Guillén and Cabo, 2004). A little change was found in seed kernel oil after 48 days of storage at 30 ℃ due to the lower oxidation rate. After 48 days, the lower peak amplitude at wave number 2853 cm^{-1} was found. The amplitude in this peak indicated the amounts of aldehyde compounds in the oil samples. The results indicated that samrong seed kernel oil had low amounts of aldehyde compounds at the end of the storage period.

The absorption of ester carbonyl group of triglyceride was presented at wave number 1741-1746 cm^{-1}. Triglyceride peak of samrong seed kernel oil was considered at wave number 1743 cm^{-1}. After 48 days of storage, this peak intensities became lower. This change was in accordance with an increase in peak at wave number 1711 cm^{-1}, which represents the C=O carboxylic group of free fatty acids (Guillen and Cabo, 1997). The results suggested that hydrolysis and lipid oxidation occurred in oil from samrong seed kernels with increased storage time. However, slight changes in absorbance peaks were found. Thus, oils from samrong seed kernels have high oxidative stability during storage.

Volatile compounds

Samrong seed kernel oil contained lower amounts of volatile compounds compared with oil kept at 30 ℃ for a longer time. Some volatile compounds (octanal, benzaldehyde, and 1-nonanol) were found only in the initial sample. This was due to the volatilization or the decomposition of those volatile lipid oxidation compounds. The lower abundance of compounds during storage is possibly due to the oxidative changes in oils or the reactions of volatile compounds with the other substances (Andrés *et al.* 2004). Oils from samrong seed contained UFAs; linoleic acid was the dominant fatty acid, followed by linoleic acid and linoleic acid (Table 1). These fatty acids are very susceptible to oxidative deterioration. After storage of 48 days, new volatiles (2-heptanone and 2-octanone) in samrong seed oil were identified. It is presumed that carbonyl compounds such as aldehydes, ketones, and alcohols can be formed by the oxidation of UFAs (Ahmed *et al.,* 2016). From the results, 2-octanone, 2-methyl-3-hexanol, and 1-octanol can be seen as the dominant volatile compounds in samrong seed kernel oil stored at 30 ℃ for an extended time. The formation of volatile compounds coincidentally occurred during oxidation (Ahmed *et al.,* 2016). However, there were slight changes in abundance of volatile compounds found.

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References

Ahmed, M., Pickova, J., Ahmad, T., Liaquat, M., Farid, A. and Jahangir, M. (2016). Oxidation of lipids in foods. Sarhad Journal of Agriculture, 32:230-238.

Andrés, A., Cava, R., Ventanas, J., Muriel, E. and Ruiz, J. (2004). Lipid oxidative changes throughout the ripening of dry-cured Iberian hams with different salt contents and processing conditions. Food Chemistry, 84:375-381.

AOAC (Association of Official Analytical Chemists) (2000). Official methods of analysis., Washington, D. C.: AOAC.

Chanyawiwatkul, J., Supapvanich, S. and Takeungwongtrakul, S. (2018). Physicochemical properties and oxidative stability of oils from samrong (*Sterculia foetida*) seed. International Journal of Agricultural Technology, 14:1097-1106.

Crapiste, G. H., Brevedan, M. I. V. and Carelli, A. A. (1999). Oxidation of sunflower oil during storage. Journal of the American Oil Chemists' Society, 76:437-1443.

Fennema, O. R. (1996). Food chemistry. 3rd ed. New York: Marcel Decker.

Ghazani, S. M., Garcá-Llatas, G. and Marangoni, A. G. (2014). Micronutrient content of cold-pressed, hot-pressed, solvent extracted and RBD canola oil: Implications for nutrition and quality. European Journal of Lipid Science and Technology, 116:380-387.

Guerere, M., Mondon, J. and Pujaniaye, A. (1985). Physicochemical composition of some seeds from plants of the isle of Re-union. Annales des falsifications et de l'expertise chimique et toxicologique, 78:281-286.

Guillén, M. and Cabo, N. (2004). Study of the effects of smoke flavourings on the oxidative stability of the lipids of pork adipose tissue by means of Fourier transform infrared spectroscopy. Meat Science, 66:647-657.

Guillen, M. D. and Cabo, N. (1997). Infrared spectroscopy in the study of edible oils and fats. Journal of the Science of Food and Agriculture, 75:1-11.

Hemingway, J., Eskandari, M. and Rajcan, I. (2015). Genetic and environmental effects on fatty acid composition in soybeans with potential use in the automotive industry. Crop Science, 55:658-668.

Iglesias, J. and Medina, I. (2008). Solid-phase microextraction method for the determination of volatile compounds associated to oxidation of fish muscle. Journal of Chromatography A, 1192:9-16.

Kale, S., Darade, V. and Thakur, H. (2011). Analysis of fixed oil from *Sterculia foetida* Linn. International Journal of Pharmaceutical Sciences and Research, 2:2908-2914.

Kim, D. S., Kim, H. S., Lee, K. T., Hong, D. L., Cho, S. R., Pan, J. H., Park, Y. B., Lee, Y. B., Kim, J. K. and Shin, E. C. (2018). Chemical characterization and oxidative stability of medium-and long-chain fatty acid profiles in tree-borne seed oils. Journal of Analytical Methods in Chemistry, 2018:1-9.

Orsavova, J., Misurcova, L., Ambrozova, J. V., Vicha, R. and Mlcek, J. (2015). Fatty acid composition of vegetable oils and its contribution to dietary energy intake and dependence of cardiovascular mortality on dietary intake of fatty acids. International Journal of Molecular Sciences, 16:12871-12890.

Ogbu, I. M. and Ajiwe, V. I. E. (2016). FTIR studies of thermal stability of the oils and methyl esters from *Afzelia africana* and *Hura crepitans* seeds. Renewable Energy, 96:203-208.

Rohman, A. and Che Man, Y. B. (2013). Application of FTIR spectroscopy for monitoring the stabilities of selected vegetable oils during thermal oxidation. International Journal of Food Properties, 16:1594-1603.

Takeungwongtrakul, S., Benjakul, S. and Aran, H. (2012). Lipids from cephalothorax and hepatopancreas of Pacific white shrimp (*Litopenaeus vannamei*): Compositions and deterioration as affected by iced storage. Food Chemistry, 134:2066-2074.

Vipunngeun, N. and Palanuvej, C. (2009). Fatty acids of *Sterculia foetida* seed oil. Journal of Health Research, 23:157.

Yun, J. M. and Surh, J. (2012). Fatty acid composition as a predictor for the oxidation stability of Korean vegetable oils with or without induced oxidative stress. Preventive Nutrition and Food Science, 17:158-165.

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