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LWT - Food Science and Technology 41 (2008) 1871–1875
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Effects of sesamol, sesamin, and sesamolin extracted from roasted sesame oil on the thermal oxidation of methyl linoleate

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Received 30 August 2007; received in revised form 26 November 2007; accepted 29 November 2007

Abstract

This study investigated the effects of lignan compounds extracted from roasted sesame oil, which were sesamol, sesamin, and sesamolin, on oxidation of methyl linoleate (ML) during heating. These compounds were added at 500 or 1000 mg/kg to ML, and α-tocopherol was used as a reference antioxidant. The ML added with lignans or α-tocopherol was heated at 180 °C for 60 min. Thermal oxidation of ML was evaluated by conjugated dienoic acid (CDA) contents, *p*-anisidine value (PAV), and ML retention. Contents changes of lignan compounds or α-tocopherol in ML during heating were monitored by high-performance liquid chromatography. CDA contents and PAV of samples increased and ML decreased with heating time at 180 °C. Samples added with lignan compounds showed lower CDA contents and PAV but higher ML retention than samples without lignan compounds. The antioxidant activity of sesame oil lignan compounds in ML oxidation during heating tended to be higher than that of α-tocopherol. The contents of lignan compounds in samples decreased with heating time due to their degradation, but the degradation rates were lower than that of α-tocopherol. This study suggested that sesame oil lignan compounds be used as antioxidants in oil at high temperatures for deep-fat frying due to their higher effectiveness and stability than α-tocopherol.
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Keywords: Sesame oil lignan compounds; Methyl linoleate; Heating; Antioxidant; Stability

1. Introduction

Food lipids undergo a variety of chemical reactions such as accelerated oxidation, thermolysis, and polymerization on heat exposure (Fritsch, 1981; Stevenson, Vaisey-Genser, & Eskin, 1984). The oxidation of oils gives undesirable flavors and taste to foods, destroys essential fatty acids, and sometimes produces toxic compounds (Auroma, 1998). Oxidized polymers can change the functionality of lipids (Min & Boff, 2001). The oxidation of oil during heating proceeds faster than the oxidation at room temperature. Antioxidants such as tocopherols and butylated hydroxyanisole are sometimes added to the oil to decrease the oil oxidation during frying (Orthoefer & List, 2007).

Tocopherols decrease lipid oxidation by donating hydrogen to lipid peroxy radicals (Verleyen et al., 2002) and inhibit degradation and isomerization of methyl linoleate hydroperoxides (Frankel, 1996). β-Carotene can also donate hydrogen to lipid peroxy radical, but with low possibility (Beutner et al., 2001). There have been growing interests on antioxidants of plant origin due to a broad application in food industry (Pokorny, Trojakova, & Takacsova, 2000) as well as significant impacts on health improvement and disease prevention (Deshpande, Desphande, & Salunkhe, 1996; Virgili, Scaccini, Packer, & Rimbach, 2001; Yanishlieva, Marinova, & Pokorny, 2006). Methanol extracts of tea seed oil decreased the free radical autoxidation by scavenging radicals and inhibited red blood cell homolysis and low density lipoprotein oxidation (Lee & Yen, 2006). Roasted sesame oil, which is manufactured by pressing the roasted sesame seeds, is very stable to the oxidation (Chung, Lee, & Choe, 2006; Kim & Choe, 2005) and the addition of roasted sesame oil significantly decreased the oxidation of soybean oil during heating at 160 °C (Chung & Choe,

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doi:10.1016/j.lwt.2007.11.019

2001). Sesame oil contains sesamol, sesamin, and sesamolin which decreased the autoxidation of oil and methyl linoleate at relatively low temperatures (Lee & Choe, 2006; Suja, Jayalekshmy, & Arumughan, 2005).

Although many compounds have shown antioxidant activities *in vitro* or *in vivo* at 20–40 °C, it is more demanding for the antioxidants in high temperature food processing since large portion of oil consumption occurs in deep-fat frying. Antioxidants for deep-fat frying should also possess stability as well as effectiveness at high temperatures over 160 °C. Frying oil mostly consists of triacylglycerols having high portions of polyunsaturated fatty acids, especially linoleic acid (Firestone, 2006), and it also contains minor compounds such as phospholipids, pigments, and tocopherols which affect the oil oxidation. Methyl linoleate which does not have any other compounds thus has been used as a model compound to study the oil oxidation (Endo, Usuki, & Kaneda, 1984; Lee & Choe, 2006; Terao & Matsushita, 1986). This study therefore was performed (1) to investigate the effectiveness of sesamol, sesamin, and sesamolin extracted from roasted sesame oil to decrease thermal oxidation of methyl linoleate and (2) to monitor their stabilities at deep-fat frying temperatures to apply them to natural antioxidants in deep-fat frying.

2. Materials and methods

2.1. Materials and chemicals

Sesame oil, which was manufactured by roasting sesame seeds at 200 °C, pressing and filtering over a screen, was obtained from CJ Co. (Seoul, Korea). Methyl linoleate (ML), sesamol, α-tocopherol, and *p*-anisidine were purchased from Sigma–Aldrich Co. (St. Louis, MO, USA). Methanol, water, *n*-hexane, isopropanol, and isooctane in HPLC grade were purchased from J.T.Baker (Phillipsburg, NJ, USA). All other chemicals were of analytical grade.

2.2. Sample preparation and oxidation

ML (200 mg) was put into a rancimat reaction vessel, and 0, 0.1, or 0.2 mg of sesame oil lignans were added to have 0, 500, or 1000 mg/kg concentration. Sesame oil lignans were sesamol, sesamin, and sesamolin extracted from roasted sesame oil by preparative HPLC and identified as described previously (Lee & Choe, 2006). α-Tocopherol was separately added to ML at the same concentrations for a reference antioxidant. Control samples, ML containing neither sesame oil lignan compounds nor α-tocopherol, were also prepared. The reaction vessels containing ML were capped with paper for free flow of air through the vessels, wrapped with aluminum foil, and placed in an oven at 180 °C for 60 min in the dark and taken out every 20 min for analyses. All samples were prepared in duplicates.

2.3. Analysis of oxidation of samples

Oxidation of ML during heating was evaluated by conjugated dienoic acid (CDA) contents and *p*-anisidine values (PAV) by AOCS (1990) methods Ti 1a-64 and Cd 18-90, respectively. Retention of ML after the oxidation was determined by gas chromatography (Lee & Choe, 2003). The sample was dissolved in isooctane and the solution (0.2–0.4 μl) was injected into a gas chromatograph (Younglin M600D, Younglin Co., Seoul, Korea), equipped with a Supelcowax™ capillary column (30 m × 0.53 mm, 1.0 μm thickness: Supelco, Bellefonte, PA, USA) and a flame ionization detector. Temperatures of the oven, injector, and detector were 200 °C, 280 °C, and 280 °C, respectively. Nitrogen flow rate was 5 ml/min, and the split ratio was 33:1. ML in the GC chromatogram was quantified from the calibration curve of standard ML.

2.4. Determination of sesamol, sesamin, sesamolin, and tocopherol in samples during heating of methyl linoleate

Contents of sesamol, sesamin, and sesamolin in samples were analyzed by high-performance liquid chromatography (HPLC; Lee & Choe, 2006). Samples were dissolved in methanol and filtered through a polytetrafluoroethylene membrane filter (0.2 μm × 13 mm; National Scientific Co., Lawrenceville, GA, USA). The filtrate (20 μl) was injected into a Younglin SP 930D HPLC equipped with a C18 symmetry reverse column (4.6 mm × 150 mm; id, 5 μm; Waters Co., Milford, MA, USA). The eluting solvent was a mixture of methanol and water (70:30, v/v) at a flow rate of 1.0 ml/min and a UV detector was set at 288 nm. Sesamol, sesamin, and sesamolin were quantified from the calibration curves of respective standard compounds. α-Tocopherol was determined by HPLC (Kim & Choe, 2005) with the same HPLC equipped with a μ-porasil column (3.9 mm × 300 mm; Waters Co.) and a fluorescence detector set at 300 nm for the excitation and 338 nm for the emission. The eluting solvent was isopropanol in *n*-hexane (0.5:99.5, v/v), and the concentration of α-tocopherol was determined from the calibration curve of standard α-tocopherol.

2.5. Statistical analysis

Statistical differences among samples were analyzed by the SAS package program (SAS Institute, Inc., Cary, NC, USA), which includes Duncan's multiple range test, regression analysis and one-way analysis of variance (ANOVA) at a 5% level of significance.

3. Results and discussion

3.1. Effects of lignan compounds extracted from roasted sesame oil on the thermal oxidation of ML

Effects of added sesamol, sesamin, sesamolin, or α-tocopherol on the CDA formation in ML during heating at 180 °C for 60 min are shown in Table 1. CDA contents of samples increased with heating time due to the transformation of nonconjugated double bonds in ML to thermodynamically more stable conjugated double bonds by the oxidation (Chung &

Table 1
Effects of sesame oil lignan compounds and α-tocopherol on conjugated dienoic acids contents (%) of methyl linoleate during heating at 180 °C

Additive level (mg/kg)	Additive	Heating time (min)			
		0	20	40	60
0	No (control)	0.63 ± 0.01 l	2.36 ± 0.23 fg	2.96 ± 0.14 bc	3.92 ± 0.26 a
500	Sesamol	0.63 ± 0.01 l	1.03 ± 0.02 kl	2.49 ± 0.31 ef	2.38 ± 0.26 fg
	Sesamin	0.63 ± 0.01 l	0.67 ± 0.05 l	1.96 ± 0.07 i	2.34 ± 0.00 fg
	Sesamolin	0.63 ± 0.01 l	0.73 ± 0.02 l	1.15 ± 0.12 k	2.31 ± 0.26 fg
	α-Tocopherol	0.63 ± 0.01 l	1.14 ± 0.04 k	2.06 ± 0.17 hi	2.70 ± 0.24 de
1000	Sesamol	0.63 ± 0.01 l	0.67 ± 0.03 l	2.27 ± 0.01 fgh	2.92 ± 0.11 bc
	Sesamin	0.63 ± 0.01 l	1.12 ± 0.06 k	1.55 ± 0.02 j	2.33 ± 0.03 fg
	Sesamolin	0.63 ± 0.01 l	0.65 ± 0.15 l	2.01 ± 0.25 i	2.80 ± 0.38 cd
	α-Tocopherol	0.63 ± 0.01 l	1.71 ± 0.13 j	2.17 ± 0.13 hi	3.14 ± 0.10 b

Mean ± standard deviation.
Different letters mean significant differences among samples at $\alpha = 0.05$.

Choe, 2001). CDA contents of samples without sesame oil lignan compounds increased from 0.63 to 3.92% after 60 min heating at 180 °C. CDA values of samples added with sesamol, sesamin, and sesamolin at 500 mg/kg were 2.38, 2.34, and 2.31% after 60 min heating, which were significantly lower than the value of control samples (3.92%). This indicates that sesamol, sesamin, and sesamolin decreased the oxidation of ML during heating. The antioxidative activities of sesamol, sesamin, and sesamolin were significantly higher than that of α-tocopherol; however, the addition level of lignan compounds did not show differences in making CDA contents lower than those of samples without sesame oil lignan compounds during heating at 180 °C. Sesamin and sesamolin tended to show higher effect than sesamol in decreasing CDA values of samples during heating. Sesamin was reported to lower the stress caused by reactive oxygen species (Hou, Huang, Tzen, & Jeng, 2003) and have anticarcinogenic effects in experimental mammalians (Hirose et al., 1992).

Table 2 shows the PAV changes in samples containing sesamol, sesamin, sesamolin, or α-tocopherol during heating at 180 °C for 60 min. PAV of samples containing no sesame oil lignan compounds was 4.1 before heating and significantly increased to 175.8, 389.8, and 854.7 after heating for 20, 40, and 60 min, respectively, due to the decomposition of ML hydroperoxides to aldehyde compounds (Danowska-Oziewicz & Karpinska-Tymoszcyk, 2005). Addition of roasted sesame oil lignan compounds decreased PAV of samples during 20 min heating at 180 °C; PAV of samples containing sesamol, sesamin, and sesamolin at 500 mg/kg was 7.2, 27.0, and 40.1, which were significantly lower than that of a sample containing no lignan compounds. However, PAVs of samples containing sesamol, sesamin, and sesamolin were significantly higher than those of a sample containing no sesame oil lignans after 40 min heating, which could not be explained. PAV tells the contents of nonvolatile 2-alkenals in the oil and ML can produce 2-heptenal (trace), 2-octenal (2.7%), 2-nonenal (1.4%), 2,4-decadienal (14%) as well as hexanal (15%) and pentane (9.9%) which are more volatile to the headspace of the samples by the autoxidation (Frankel, 1985). Also high contents of 2,4-decadienal in a sample containing no sesame oil lignans might be decomposed either to the 2,3-epoxy or the 4,5-epoxy derivative which was not contributed to PAV, and then further decomposed to 2-octenal (Boskou, Salta, Chiou, Troullidou, & Andrikopoulos, 2006). As a result, PAV of a sample containing no sesame oil lignans could show lower PAV and then higher values than PAV of samples containing sesame oil lignans or tocopherol. Further study might be needed.

Retention of ML affected by the addition of sesame oil lignan compounds or α-tocopherol during heating at 180 °C for 60 min is shown in Fig. 1. The amounts of ML decreased with heating time showing its oxidation, as shown in other studies (Chen, Tai, Chen, & Chen, 2001; Waltking & Zmachinski,

Table 2
Effects of sesame oil lignan compounds and α-tocopherol on *p*-anisidine value of methyl linoleate during heating at 180 °C

Additive level (mg/kg)	Additive	Heating time (min)			
		0	20	40	60
0	No (control)	4.1 ± 0.6 t	175.8 ± 2.2 p	389.8 ± 17.1 m	854.7 ± 7.7 ab
500	Sesamol	4.1 ± 0.6 t	7.2 ± 0.5 t	466.6 ± 4.9 k	721.4 ± 15.2 g
	Sesamin	4.1 ± 0.6 t	27.0 ± 5.7 s	514.5 ± 2.7 i	826.0 ± 3.7 d
	Sesamolin	4.1 ± 0.6 t	40.1 ± 4.8 r	541.1 ± 6.4 h	844.1 ± 13.4 bc
	α-Tocopherol	4.1 ± 0.6 t	56.5 ± 11.9 q	394.3 ± 5.0 m	809.2 ± 12.1 e
1000	Sesamol	4.1 ± 0.6 t	8.0 ± 0.6 t	342.3 ± 15.1 o	756.8 ± 18.8 f
	Sesamin	4.1 ± 0.6 t	41.8 ± 3.2 r	436.1 ± 10.8 l	857.0 ± 5.9 a
	Sesamolin	4.1 ± 0.6 t	21.8 ± 1.6 s	486.1 ± 17.2 j	841.5 ± 5.8 c
	α-Tocopherol	4.1 ± 0.6 t	39.5 ± 5.2 r	357.2 ± 13.7 n	823.8 ± 6.4 d

Mean ± standard deviation.
Different letters mean significant differences among samples at $\alpha = 0.05$.

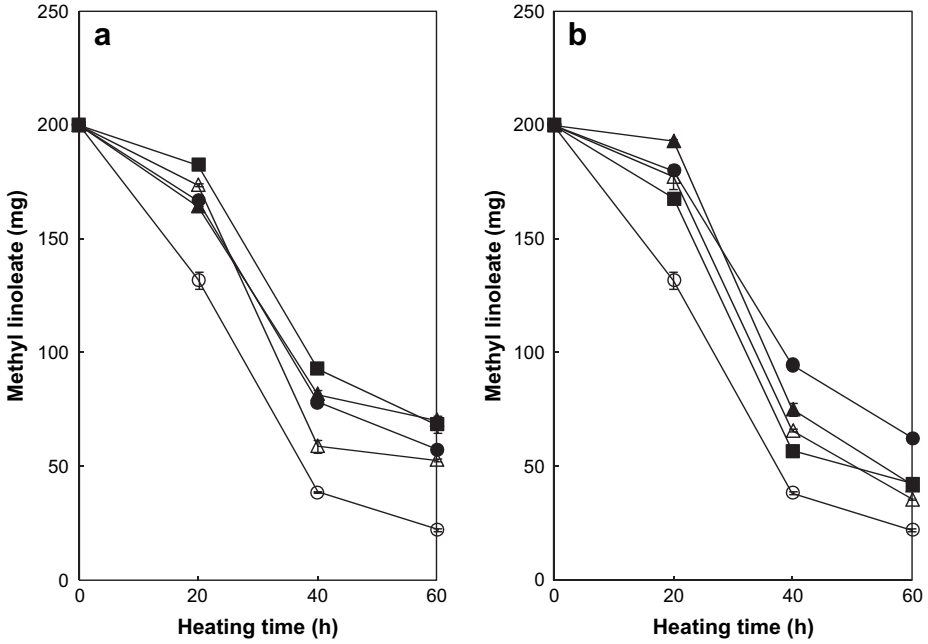

Fig. 1. Effects of lignan compounds on methyl linoleate remained during heating 180 °C for 60 min (a, 500 mg/kg; b, 1000 mg/kg; —○—, no additives; —▲—, sesamol; —●—, sesamin; —■—, sesamolin; —△—, α-tocopherol).

1970). Significantly higher amounts of ML were retained in samples containing sesamol, sesamin, and sesamolin than in samples without them. This clearly shows that sesame oil lignan compounds protected ML from oxidation during heating at 180 °C. Protection of ML from oxidation by α-tocopherol was comparable to that by sesame oil lignans at 20 min heating as shown in CDA formation, but it was lower than that by lignans after 40 min heating.

The results clearly show that sesamol, sesamin, and sesamolin decreased the oxidation of ML during heating, and suggest a possible use of roasted sesame oil lignan compounds as an antioxidant in the oil for high temperature food processing such as deep-fat frying.

3.2. Stability of lignan compounds added to methyl linoleate during heating

Changes in contents of sesame oil lignan compounds added to ML during heating at 180 °C for 60 min are shown in Table 3. Contents of sesamol, sesamin, and sesamolin decreased due to their degradation as the heating time of samples increased. Sesamol contents decreased from 1000 mg/kg to 818.2 (81.8%), 579.8 (58.0%), and 376.7 mg/kg (37.7%) after 20, 40, and 60 min heating, respectively. Sesamolin and sesamin showed less degradation than sesamol.

Since two levels of each lignan compound and α-tocopherol were added to samples, the relative retention (%) to the initial value was considered to compare the degradation rate among lignan compounds and α-tocopherol as shown in Table 4. The degradation rates of lignan compounds in samples during heating are represented as "a" values in the regression equations between retention (%) of lignan compounds and the heating time in minutes. Sesamol was degraded at the rate of 1.054%/min at the addition level of 1000 mg/kg while the degradation rates of sesamin and sesamolin were significantly low at 0.755 and 0.758%/min, respectively. This means that sesamol in samples was degraded faster than sesamin or sesamolin. There was no significant difference in

Table 3
Contents changes in lignan compounds added to methyl linoleate during heating at 180 °C for 60 min

Additive level (mg/kg)	Additive	Heating time (min)			
		0	20	40	60
500	Sesamol	500.0 ± 2.9 a	400.0 ± 10.4 c	269.5 ± 12.6 e	186.5 ± 21.9 g
	Sesamin	500.0 ± 11.1 a	438.1 ± 16.2 b	362.0 ± 10.3 d	248.4 ± 11.4 f
	Sesamolin	500.0 ± 1.2 a	438.4 ± 12.9 b	347.0 ± 21.8 d	260.7 ± 5.7 ef
	α-Tocopherol	500.0 ± 10.2 a	395.1 ± 11.8 c	255.1 ± 10.4 ef	180.3 ± 9.7 g
1000	Sesamol	1000.0 ± 27.6 a	818.2 ± 23.8 cd	579.8 ± 11.9 f	376.7 ± 21.4 h
	Sesamin	1000.0 ± 14.2 a	913.8 ± 6.0 b	761.8 ± 29.5 e	547.4 ± 10.2 gf
	Sesamolin	1000.0 ± 2.9 a	855.4 ± 43.8 c	764.6 ± 24.8 e	524.7 ± 17.0 g
	α-Tocopherol	1000.0 ± 21.5 a	799.1 ± 13.7 de	541.3 ± 20.6 gf	248.0 ± 11.1 i

Mean ± standard deviation.
Different letters mean significant differences among samples within the same additive level at $\alpha = 0.05$.

Table 4
Regression analysis between relative retention of sesame oil lignan compounds or α-tocopherol added to methyl linoleate and heating time at 180 °C

Addition level (mg/kg)	Additive	Regression parameters[a]		
		a	*b*	r^2
500	Sesamol	−1.071 c	99.9	0.993
	Sesamin	−0.831 b	102.3	0.980
	Sesamolin	−0.809 b	101.5	0.993
	α-Tocopherol	−1.099 c	99.5	0.988
1000	Sesamol	−1.054 c	100.9	0.997
	Sesamin	−0.755 a	103.2	0.965
	Sesamolin	−0.758 a	101.3	0.963
	α-Tocopherol	−1.256 d	102.4	0.993

Different letters mean significant differences among samples at $\alpha = 0.05$.
[a] Relative retention (%) of lignan compounds or α−tocopherol to the initial level = $a \times$ heating time (min) + b, r; correlation coefficient.

degradation during heating at 180 °C between sesamin and sesamolin. Kikugawa, Arai, and Kurechi (1983) reported that degradation of sesamol was faster than that of sesamolin in sesame oil at 98 °C. Degradation rates of sesame oil lignan compounds were significantly ($p < 0.05$) lower than that of α-tocopherol (1.256%/min) at the addition level of 1000 mg/kg. Degradation rates of sesame oil lignan compounds were lower in samples added with 1000 mg/kg than in samples with 500 mg/kg.

In conclusion, ML oxidation during heating could decrease by addition of roasted sesame oil lignan compounds whose antioxidant activities and stability during heating at 180 °C for 60 min were higher than that of α-tocopherol, and this study suggests a potential use of sesame oil lignan compounds as antioxidants in oil at high temperatures for deep-fat frying.

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