Comparison between different sesame oil production techniques for lignans

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Outline

- Introduction
- Objective
- Experimental design
- Materials and methods
- Results
- Conclusions

Introduction

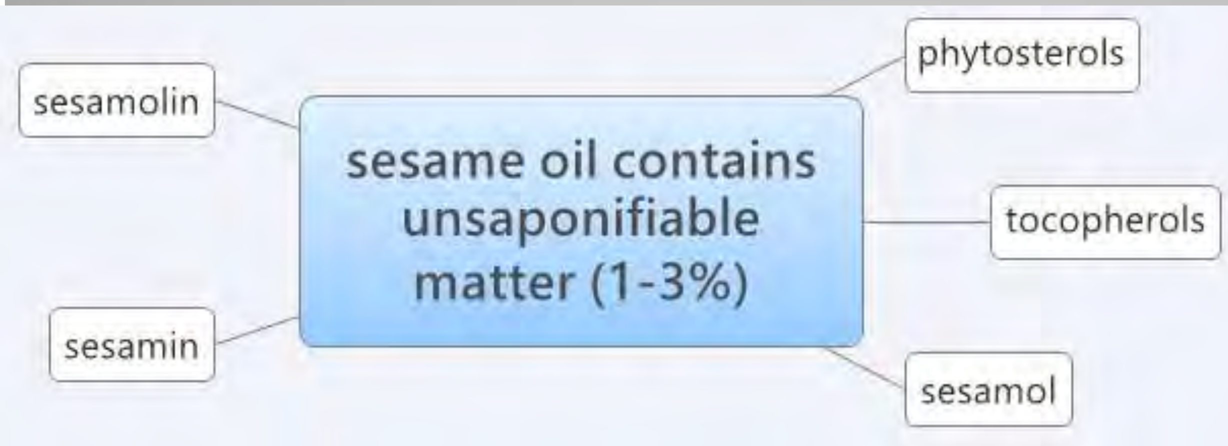
Introduction

- Sesame is an important oilseed crop in the world and provides a good source of edible gourmet oil.
- Sesame serves as a nutritious food for humans.

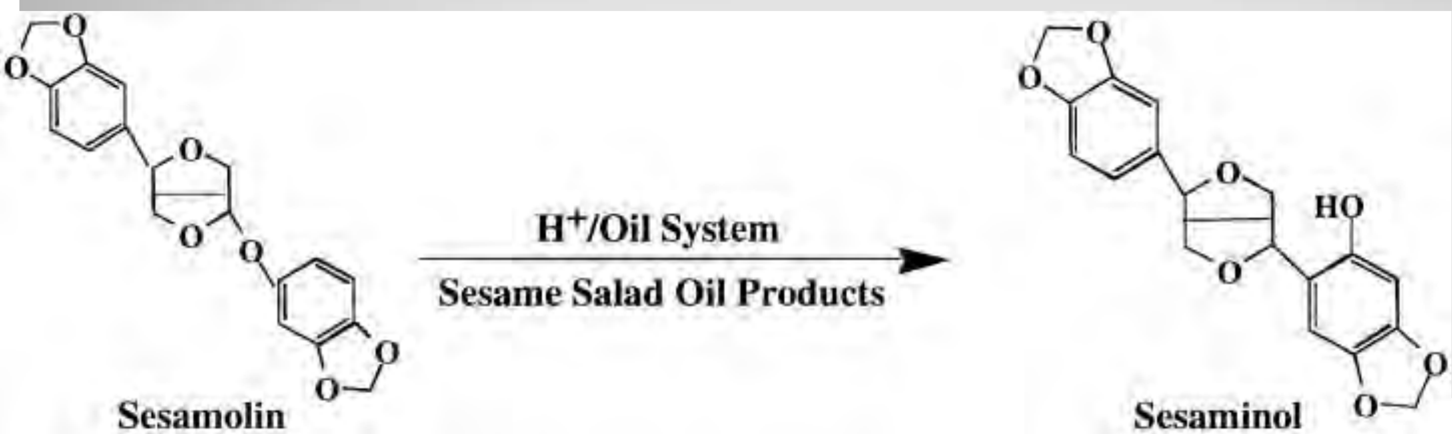
(Chen *et al*., 2005)

(http://realfoodforlife.com/20-health-benefits-of-sesame/) (http://eat.sh/8769.html)

Sesame oil



(Kochhar, 2002)



(Karnika and Naik, 2014)

Introduction

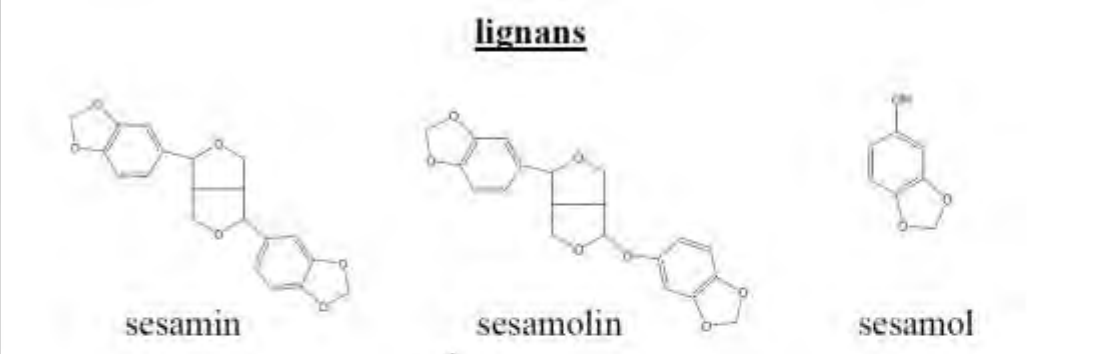
- Lipid oxidation has been recognized as the major problem affecting edible oils, as it is the cause of important deteriorative changes in their chemical, sensory and nutritional properties.
- The remarkable stability of sesame oil may be due to the presence of unique unsaponifiable constituents namely **lignans** and **tocopherols**.
- ... are unstable ... Therefore, ... oxidative stage of oil requires three different oxidation parameters.

(Velasco et al., 2002) *(Suja et al., 2004)*

- Ses... resistant to oxi...

... *et al.*, 2005)

- According to the previous research, the most lignans in sesame is sesamin, sesamolin and sesamol.
- After heating or acid processing ,the sesamolin will turn into the other kinds of lignans - sesamol. As a result, the sesamol is mainly exist in the hot processing sesame and sesame oil.



(Kamal-Eldin *et al*., 1995)

- **Cold-pressing** is a special processing procedure commercially used to produce edible oils.

- **Therefore, cold-pressed oils rich in α-linolenic acid may be an excellent dietary source of n-3 PUFAs. In addition to their fatty acid contents, shelf stability is a critical factor for the potential food applications of the cold-pressed oils.**

(Yu *et al*., 2002)

- **Cold-pressed oils may retain a greater level of natural antioxidants than conventionally processed oils and exhibit acceptable oxidative stability and improved safety without added synthetic antioxidants.**

(Parker *et al*., 2003)

- **Compare with the traditional sesame oils, cold-pressed oils have awesome characteristics, like light color, high nutritional value. The use of cold-pressed sesame oil as frying oil, not only increase the nutritional value of fried foods also fried foods will have a special sesame oil flavor.**

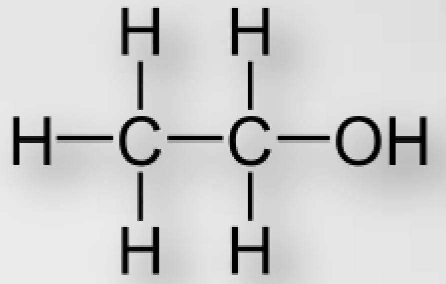
- **After 24-hours frying test, the results showed that the content of benzopyrene was no significant difference, which was about 1.16 µg/kg (far lower than the international standard value of 10 µg/kg). And after 18 hours , carbonyl value reached 59.01 meq/kg (more than the international standard value of 50 meq/kg), so the results of cold-pressed sesame oil for fry should be set up at 17 hours.**

(Huang et al., 2009)

- **Ethanol is recognized as non-toxic and has less handling risks than other solvent such as hexane and acetonitrile .**

- **In addition to triglycerides , during the extraction process other compounds such as polyphenols, pigments are extracted jointly.**

(Yu *et al*., 2002)



(wikipedia)

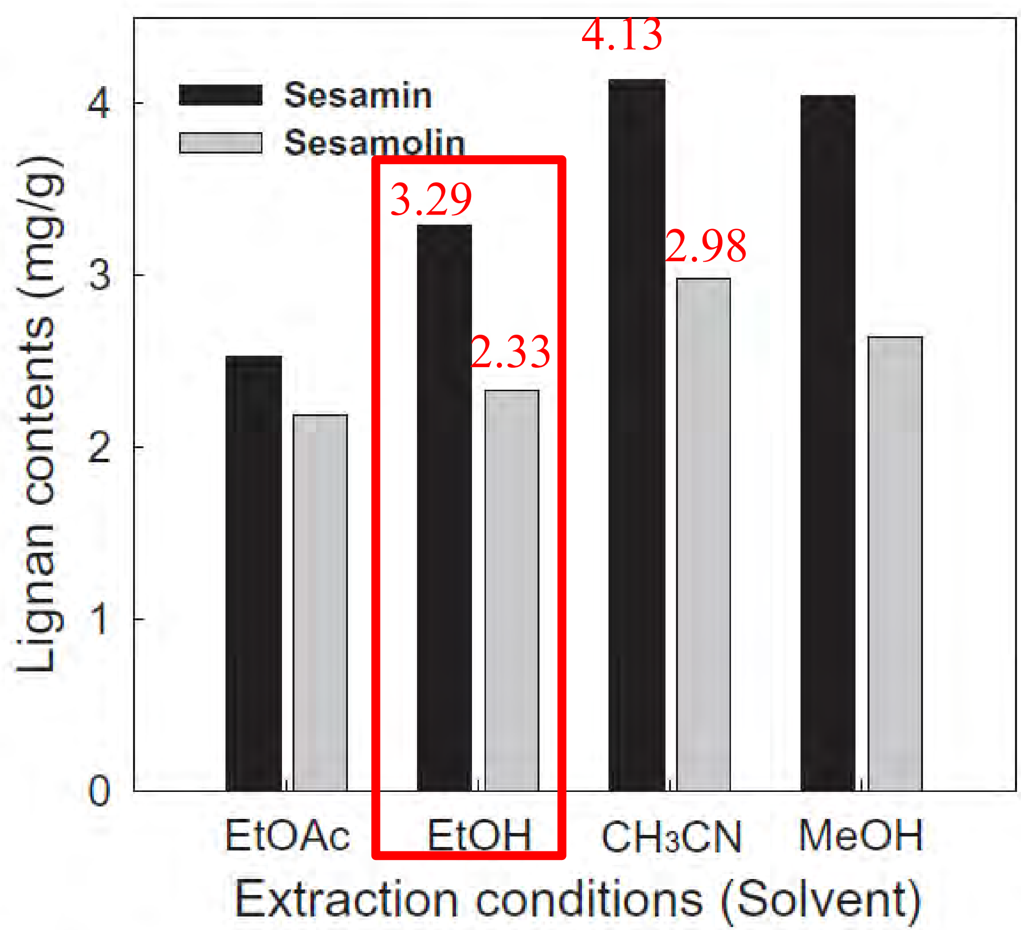


Fig 1. · **Comparison of lignan contents in four different solvent extracts from sesame seeds.**

(Kim et al.,2014_

Antioxidant activity of defatted sesame meals . . .

Table 1. Effect of heat treatments with different temperatures and periods on total phenolic contents of methanol extracts from defatted sesame meals

	Roasting time (min)[a]								
Temp (°C)	0 (μM)	10 (μM)	20 (μM)	30 (μM)	40 (μM)	60 (μM)	90 (μM)	120 (μM)	SEM
50	35.6d	27.2fz	39.3bx	34.9dz	33.0ez	33.5ez	37.1cy	40.5az	0.2
100	35.6d	33.2ey	35.7dy	39.7cy	40.1cy	47.0ay	35.4dz	43.8 by	0.4
150	35.6f	38.0ex	33.4gz	43.5dx	83.6aw	84.4ax	46.7cx	67.9bx	0.3
200	35.6f	50.7ew	73.8bcw	72.3cdw	69.0dx	87.4aw	70.9cdw	76.8bw	1.1
SEM	0.1	0.9	0.4	1.1	0.4	0.5	0.4	0.3	

[a]Different letters (a through f) within a row indicate significant differences ($P < 0.05$); $n = 3$. Different letters (x through w) within a column with same color value indicate significant differences ($P < 0.05$).

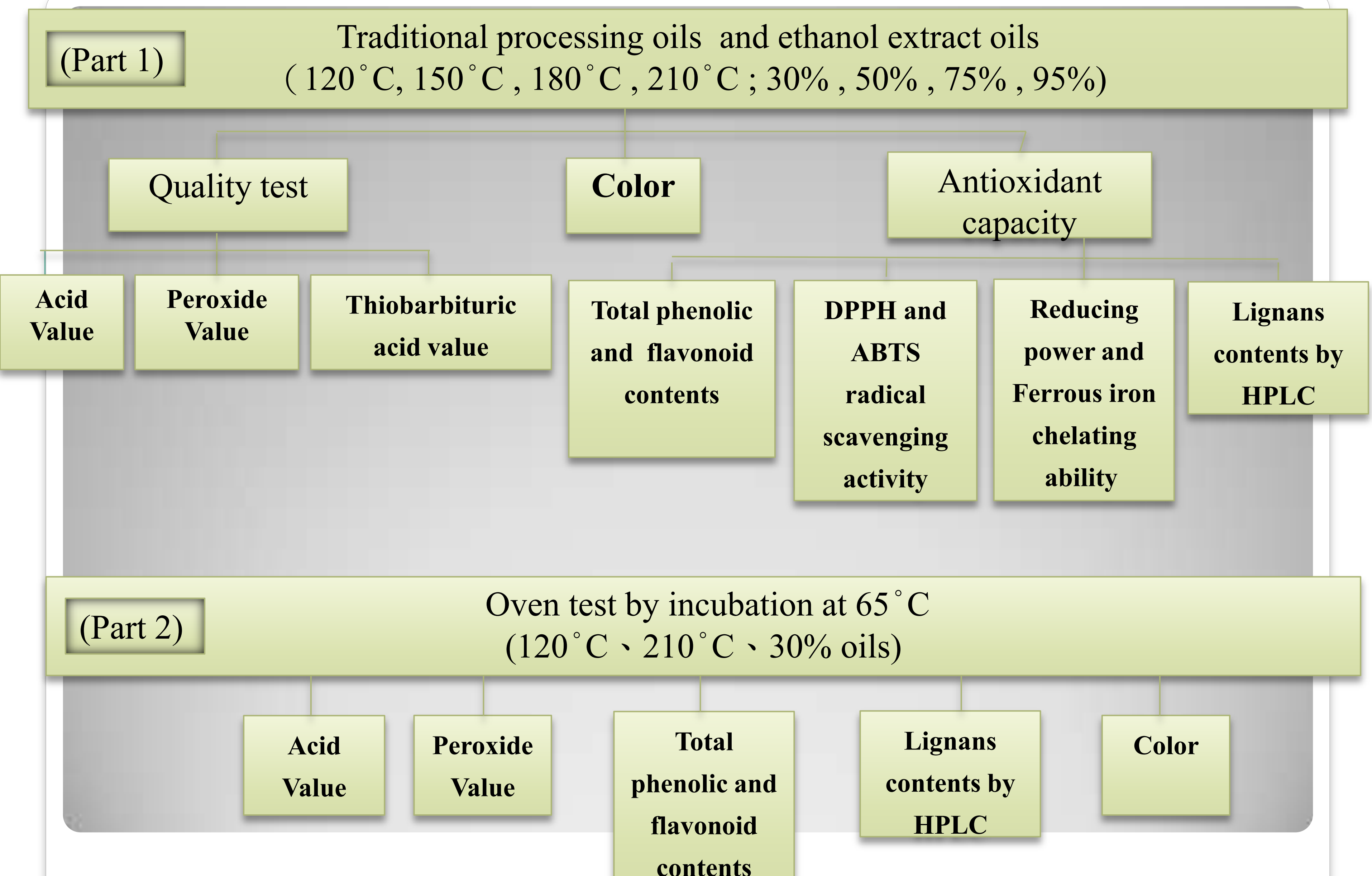
(S.-M. JEONG *et al.*, 2004)

Objective

- **The objective is to evaluate the shelf life of sesame oils from difference extraction methods.**

- **Ethanol extraction method can increase the antioxidants contents which was compared to the tradition heating process in sesame oil.**

Experimental design


(Part 1)
Traditional processing oils and ethanol extract oils
(120˚C, 150˚C , 180˚C , 210˚C ; 30% , 50% , 75% , 95%)
Quality test
Color
Antioxidant capacity
Acid Value
Peroxide Value
Thiobarbituric acid value
Total phenolic and flavonoid contents
DPPH and ABTS radical scavenging activity
Reducing power and Ferrous iron chelating ability
Lignans contents by HPLC
(Part 2)
Oven test by incubation at 65˚C
(120˚C、210˚C、30% oils)
Acid Value
Peroxide Value
Total phenolic and flavonoid contents
Lignans contents by HPLC
Color

Materials and methods

Materials and Methods

Sample preparation

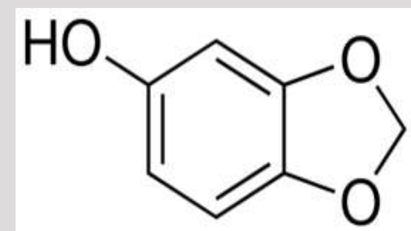
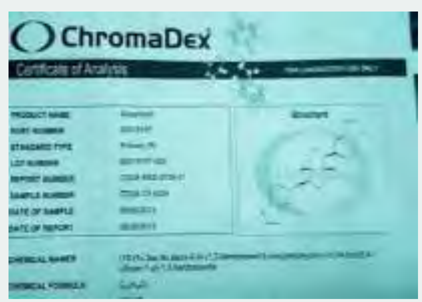
White Sesame (*Sesamum indicum L*.) from India

Traditional processing oils

- ✓ Roast
- ✓ Squeezing

Ethanol extract oils

- ✓ Crushed
- ✓ Soaked with ethanol
- ✓ Filter with filtering cloth
- ✓ Centrifuge

Sesamin	Sesamolin	Sesamol
5,5'-(1S,3aR,4S,6aR)-tetrahydro-1H,3H-furo[3,4-c]furan-1,4-diylbis(1,3-benzodioxole)	5-[4-(1,3-benzodioxolol-5-yloxy)tetrahydro-1h,3h-furo[3,4-c]furan-1-yl]-1,3-benzodioxole	3,4-(Methylenedioxy)phenol, 5-Benzodioxolol
$C_{20}H_{18}O_6$	$C_{20}H_{18}O_7$	$C_7H_6O_3$
		
		

Results-part 1

Table 1. Effect of heating temperature and ethanol extract concentration of traditional heating process and ethanol extract sesame oil on acid value, peroxide value and thiobarbituric acid value

	Acid value (mg KOH/g)	Peroxide value (meq/kg oil)	Thiobarbituric acid value (μg/g)
120℃	1.65±0.02 [a]	1.8±0.20 [cd]	0.14±0.08 [e]
150℃	1.60±0.03 [b]	1.5±0.12 [de]	0.57±0.34 [cd]
180℃	1.40±0.03 [c]	1.3±0.12 [de]	1.21±0.12 [a]
210℃	1.36±0.02 [d]	1.1±0.12 [e]	1.26±0.25 [a]
30%	0.59±0.01 [e]	3.2±0.24 [a]	0.96±0.13 [ab]
50%	0.51±0.01 [f]	2.4±0.12 [ab]	0.78±0.18 [bc]
75%	0.41±0.01 [g]	2.0±0.20 [bc]	0.48±0.13 [cde]
95%	0.41±0.01 [g]	1.6±0.12 [cde]	0.31±0.13 [de]

Each value is express as means ± standard deviation from three data. [a-e] Means followed by the different letters in the same column are significantly different (p <0.05).

Total Phenolic Contents

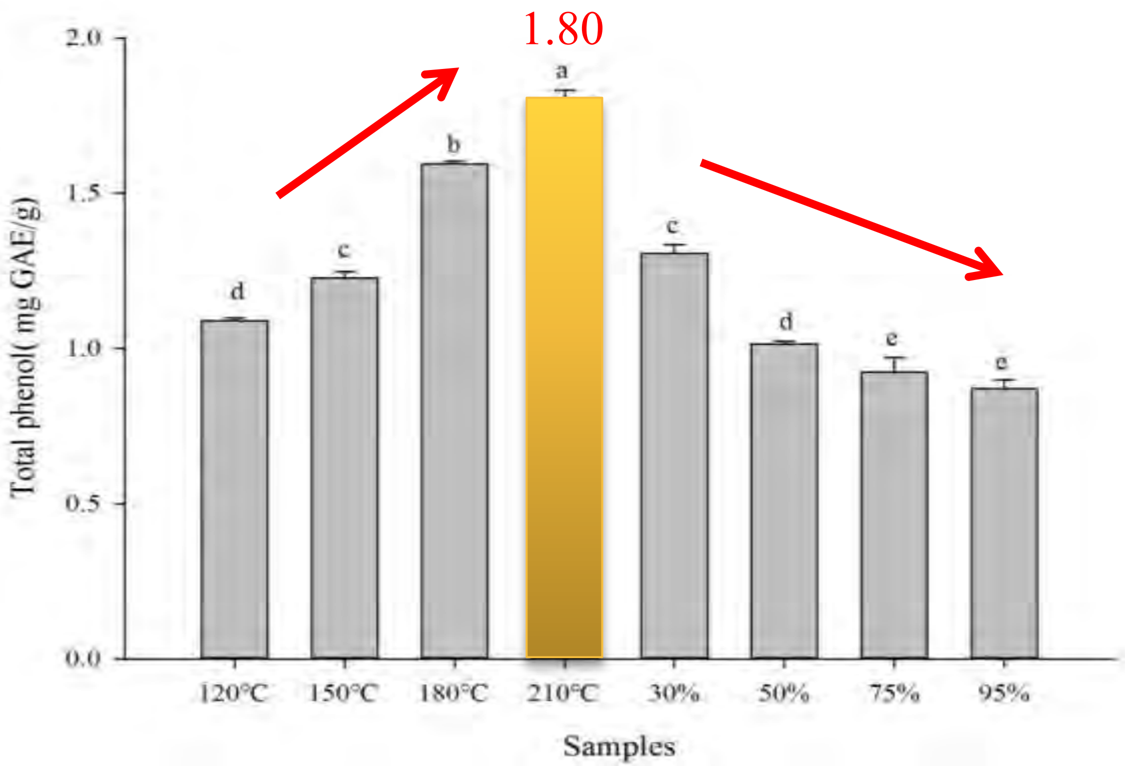


Figure 1. Effect of heating temperature and ethanol extract concentration on total phenolic contents of traditional heating processing sesame oil and ethanol extract sesame oil. Each value is express as means ± standard deviation from three data. a-e Means followed by the different letters in the same column are significantly different ($p < 0.05$).

Total Flavonoid Contents

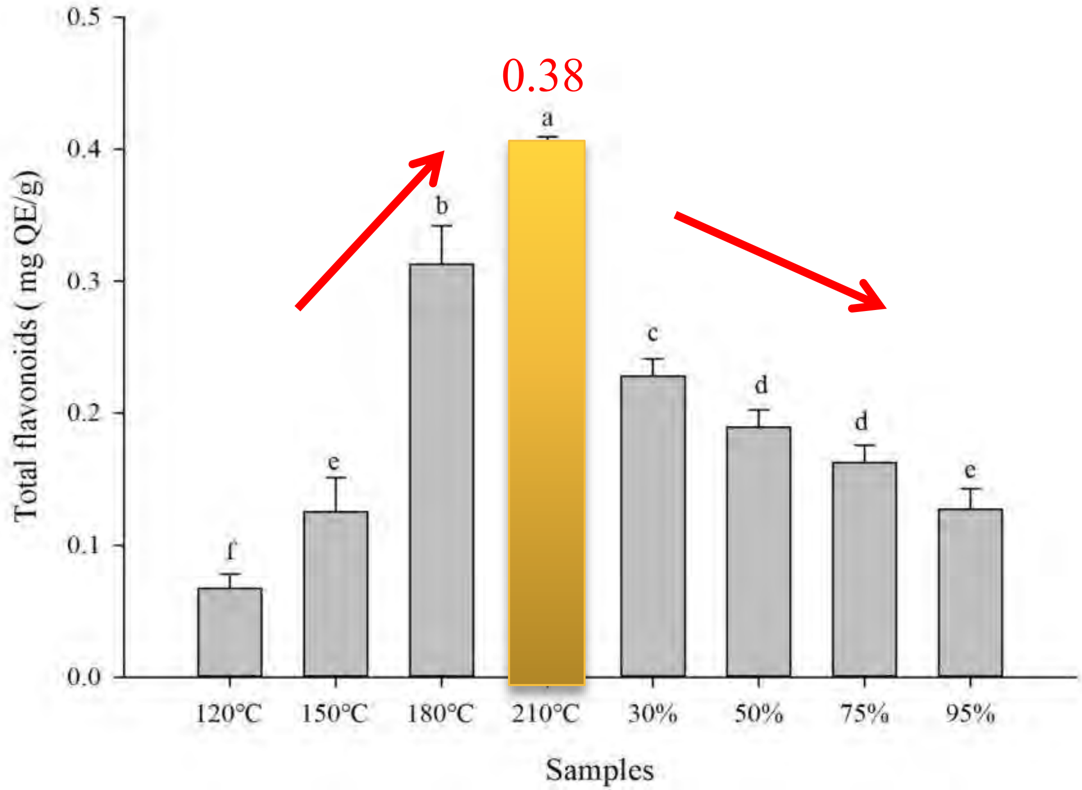


Figure 2. Effect of heating temperature and ethanol extract concentration on total flavonoid contents of traditional heating processing sesame oil and ethanol extract sesame oil. Each value is express as means ± standard deviation from three data. [a-f] Means followed by the different letters in the same column are significantly different (p <0.05).

DPPH Scavenging Activity

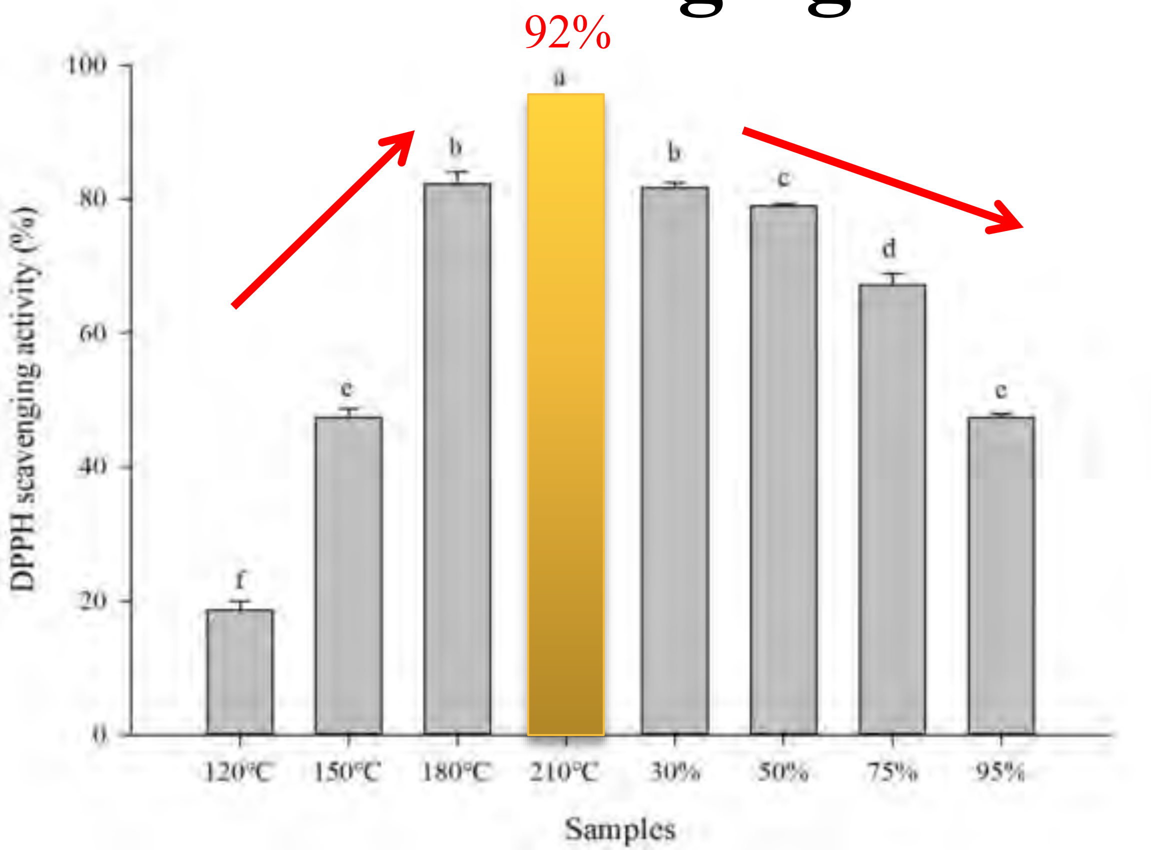


Figure 3. Effect of heating temperature and ethanol extract concentration on DPPH scavenging activity of traditional heating process sesame oil and ethanol extract sesame oil. Each value was expressed as means ± standard deviation from three data. [a-f] Means followed by the different letters in the same column are significantly different (p <0.05).

ABTS Scavenging Activity

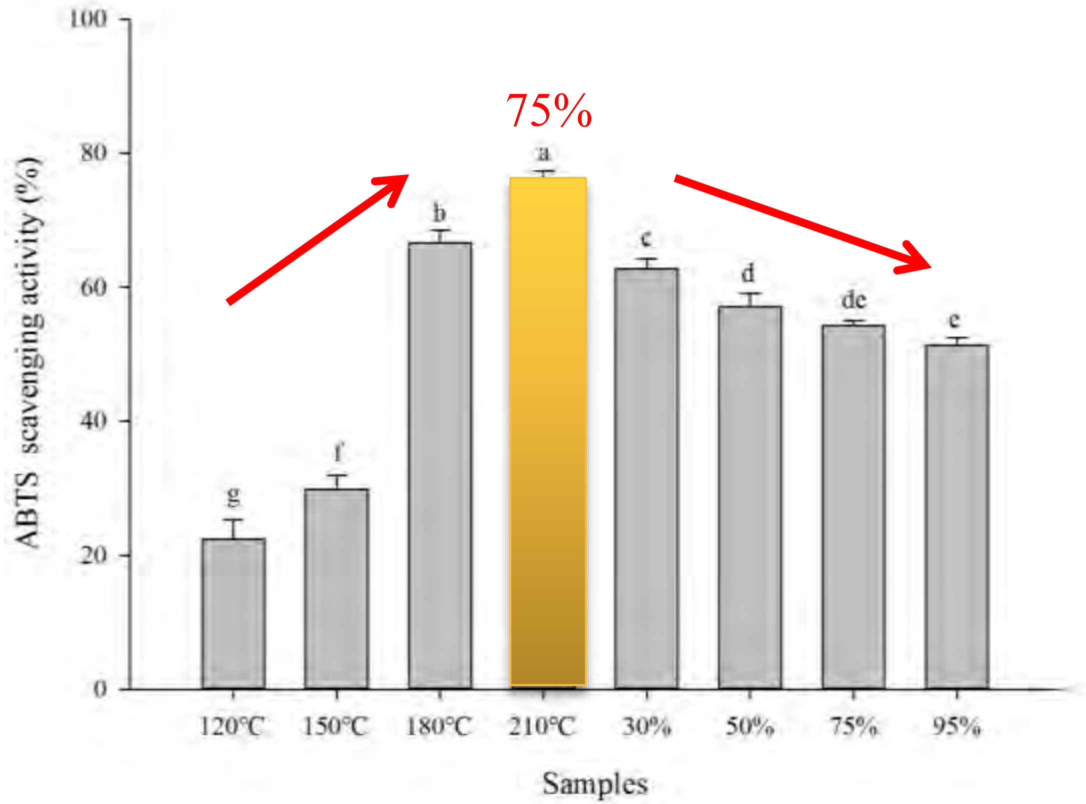


Figure 4. Effect of heating temperature and ethanol extract concentration on ABTS scavenging activity of traditional heating process sesame oil and ethanol extract sesame oil. Each value was expressed as means ± standard deviation from three data. [a-g] Means followed by the different letters in the same column are significantly different (p <0.05).

Reducing Power

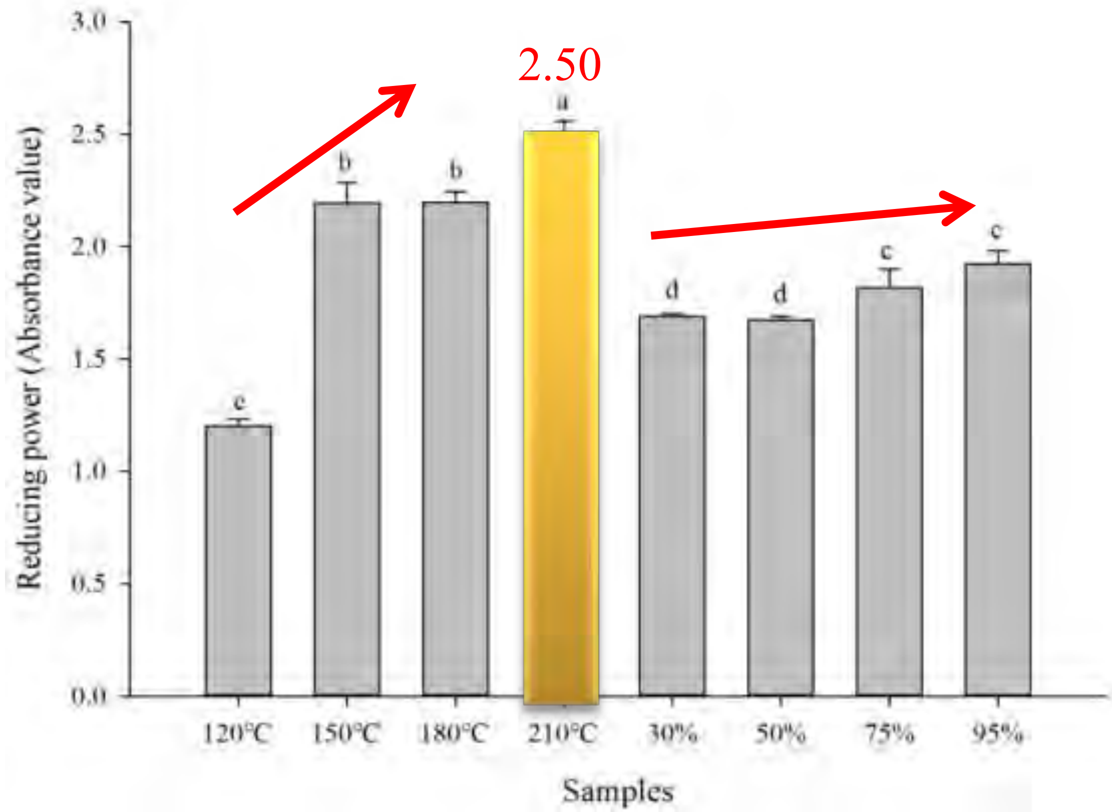


Figure 5. Effect of heating temperature and ethanol extract concentration on reducing power of traditional heating processing and ethanol extract sesame oil. Each value was expressed as means ± standard deviation from three data. [a-e] Means followed by the different letters in the same column are significantly different (p <0.05).

Ferrous iron Chelating Ability (%)

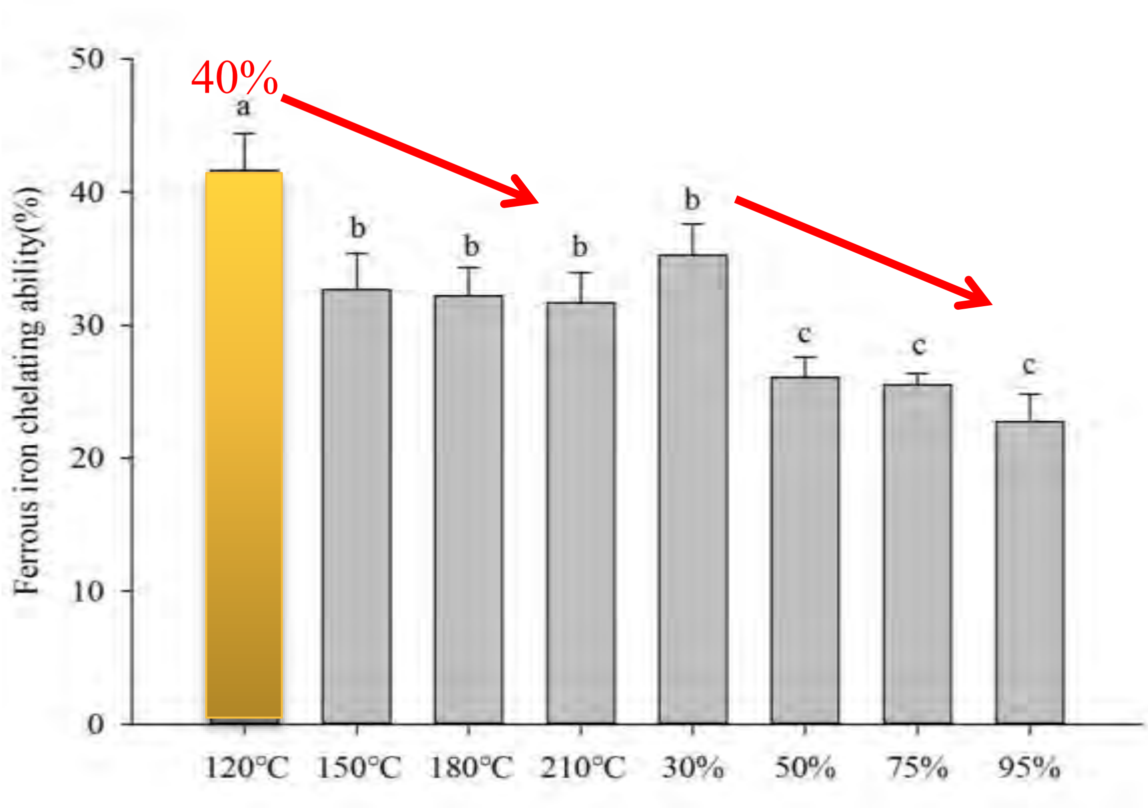


Figure 6. Effect of heating temperature and ethanol extract concentration on Ferrous iron chelating ability of traditional heating processing and ethanol extract sesame oil. Each value was expressed as means ± standard deviation from three data. [a-c] Means followed by the different letters in the same column are significantly different ($p < 0.05$).

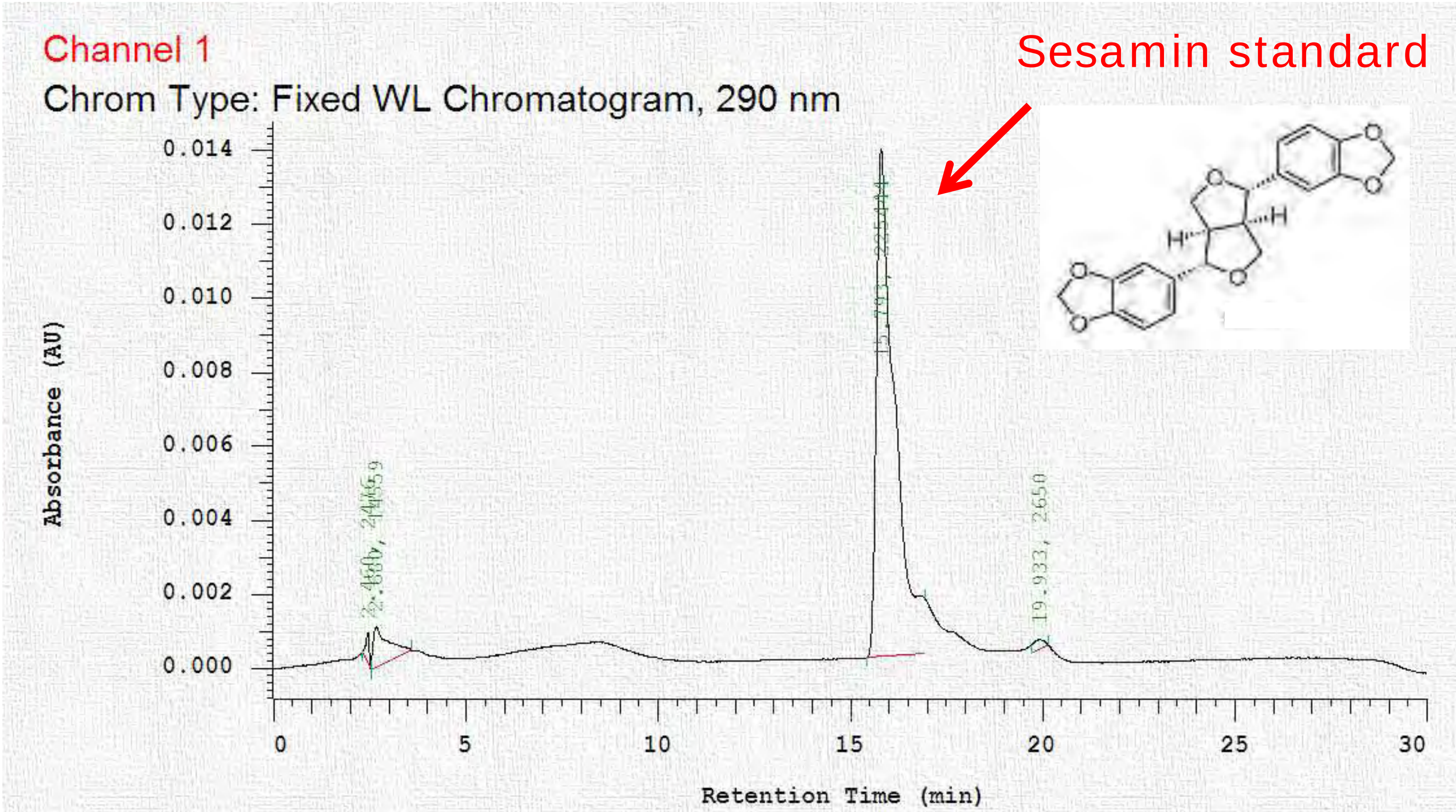


Figure 7 . HPLC profile of sesamin standards showing one sharp peak corresponding to sesamin with retention times of 15.7 .

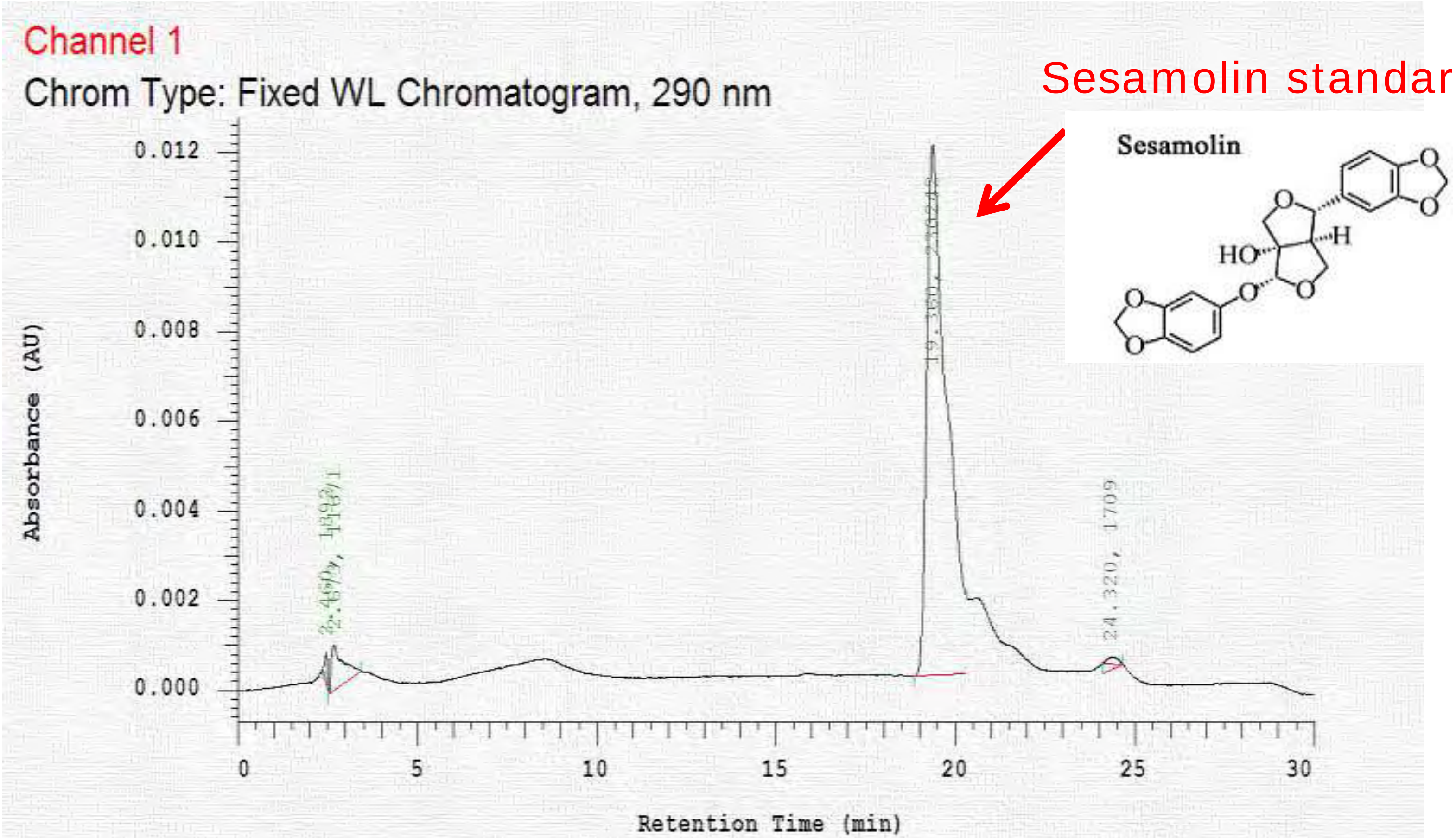


Figure 8 . HPLC profile of sesamin standards showing one sharp peak corresponding to sesamolin with retention times of 19.3 .

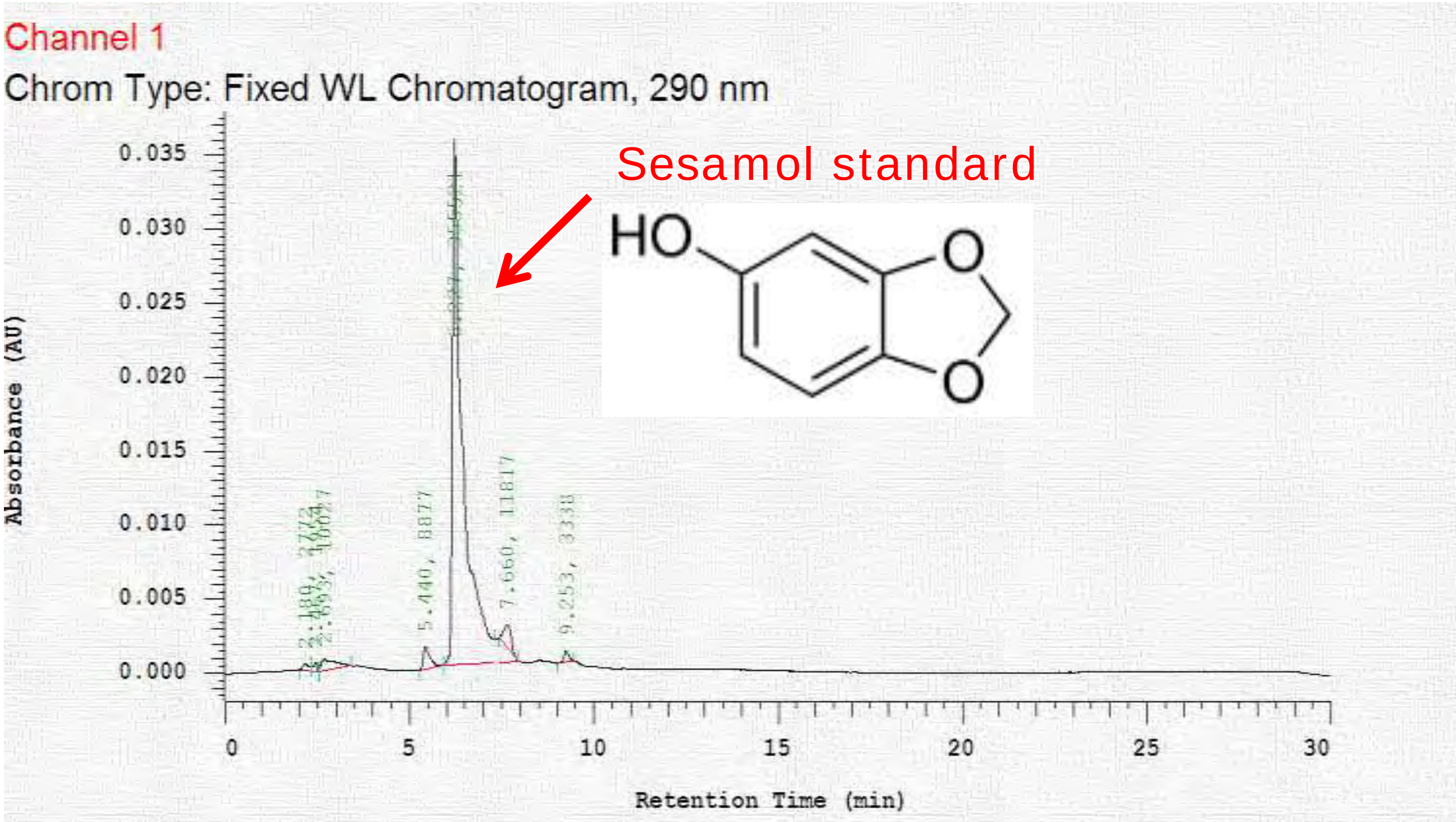


Figure 9 . HPLC profile of sesamol standards showing one sharp peak corresponding to sesamol with retention times of 6.2 .

Table 2. Effect of heating temperature and ethanol extract concentration of traditional heating processing and ethanol extract sesame oil on lignan

	Sesamin (mg/g)	Sesamolin (mg/g)	Sesamol (mg/g)	Total (mg/g)	Sesamolin + sesamol (mg/g)
120℃	20.37±1.01 [a]	6.74±0.22 [b]	0.13±0.01 [c]	27.25±1.23 [a]	6.87±0.23 [b]
150℃	17.38±1.10 [b]	5.75±1.01 [bc]	0.14±0.01 [c]	23.28±2.09 [bc]	5.90±1.02 [bc]
180℃	16.74±0.61 [b]	5.06±0.57 [cd]	0.27±0.02 [b]	22.08±1.20 [cd]	5.34±0.59 [cde]
210℃	16.08±0.48 [bc]	3.98±0.42 [d]	0.52±0.05 [a]	20.59±0.94 [cd]	4.51±0.47 [e]
30%	18.34±0.67 [ab]	8.58±0.83 [a]	ND	26.92±1.50 [ab]	8.58±0.83 [a]
50%	15.64±2.72 [bc]	6.90±0.53 [b]	ND	22.54±3.25 [cd]	6.90±0.53 [b]
75%	13.29±2.54 [c]	5.79±0.76 [bc]	ND	19.09±3.25 [d]	5.79±0.76 [bcd]
95%	10.31±1.42 [d]	4.57±0.65 [cd]	ND	14.88±2.03 [e]	4.57±0.65 [de]

Means followed by the different letters in the same column are significantly different (p <0.05).

Appendix 1. Effect of lignans on total phenol, ABTS scavenging activity and ferrous iron chelating activity.

	total phenol (μg GAE/g)	ABTS (%)	ferrous iron chelating activity (%)
sesmin	58.4±0.9 [c]	13.1±4.6 [c]	14.5±2.3 [a]
sesamolin	113.1±1.4 [b]	24.0±3.3 [b]	7.1±1.8 [b]
sesamol	261.9±5.5 [a]	58.5±2.0 [a]	10.3±1.4 [ab]

Each value is express as means ± standard deviation from three data. [a-c] Means followed by the different letters in the same column are significantly different (p <0.05).Total phenol assay(100 μg/g lignans), ABTS scavenging activity assay(20 μg/g lignans) , Ferrous iron Chelating Activity(10 μg/g lignans).

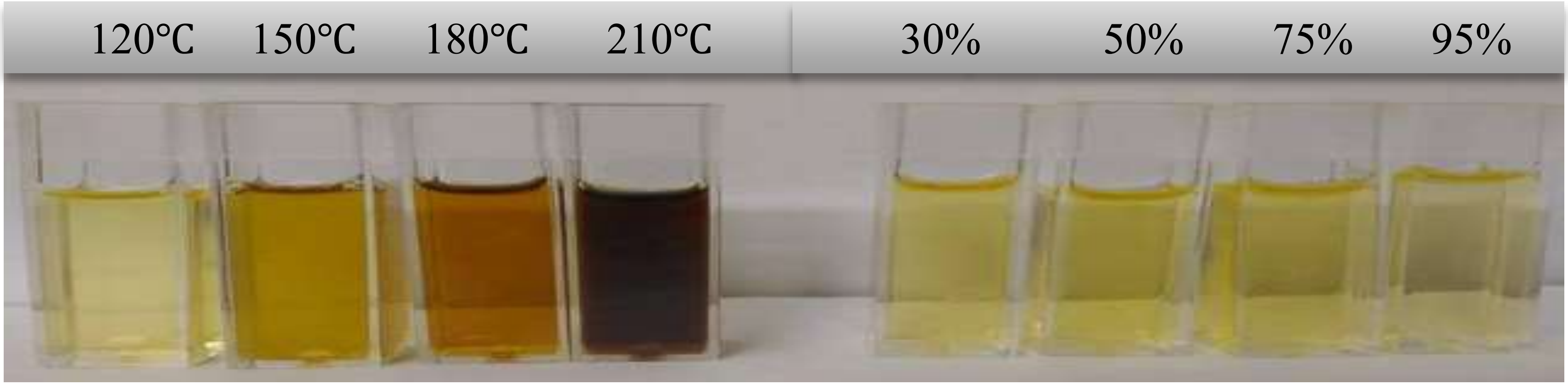


Table 3. Effect of heating temperature and ethanol extract concentration of traditional heating processing and ethanol extract sesame oil on color

	L	a	b
120℃	40.90±0.21 ^{e}	6.66±0.05 ^{d}	42.30±0.43 ^{a}
150℃	29.20±0.01 ^{f}	13.66±0.05 ^{c}	40.06±0.15 ^{d}
180℃	20.06±0.11 ^{g}	20.83±0.05 ^{b}	25.46±0.46 ^{f}
210℃	14.20±0.01 ^{h}	21.90±0.10 ^{a}	14.06±0.47 ^{h}
30%	53.06±0.60 ^{d}	2.83±2.83 ^{e}	41.26±2.82 ^{b}
50%	54.40±0.43 ^{b}	1.75±1.75 ^{f}	39.83±0.15 ^{e}
75%	53.50±0.43 ^{c}	2.66±2.66 ^{e}	40.93±0.05 ^{c}
95%	56.93±0.20 ^{a}	-0.33±-0.33 ^{g}	23.73±0.25 ^{g}

Each value was expressed as means ± standard deviation from three data.
$^{a-h}$ Means followed by the different letters in the same column are significantly different (p <0.05).

Results-part 2

Storage Test-Acid Value

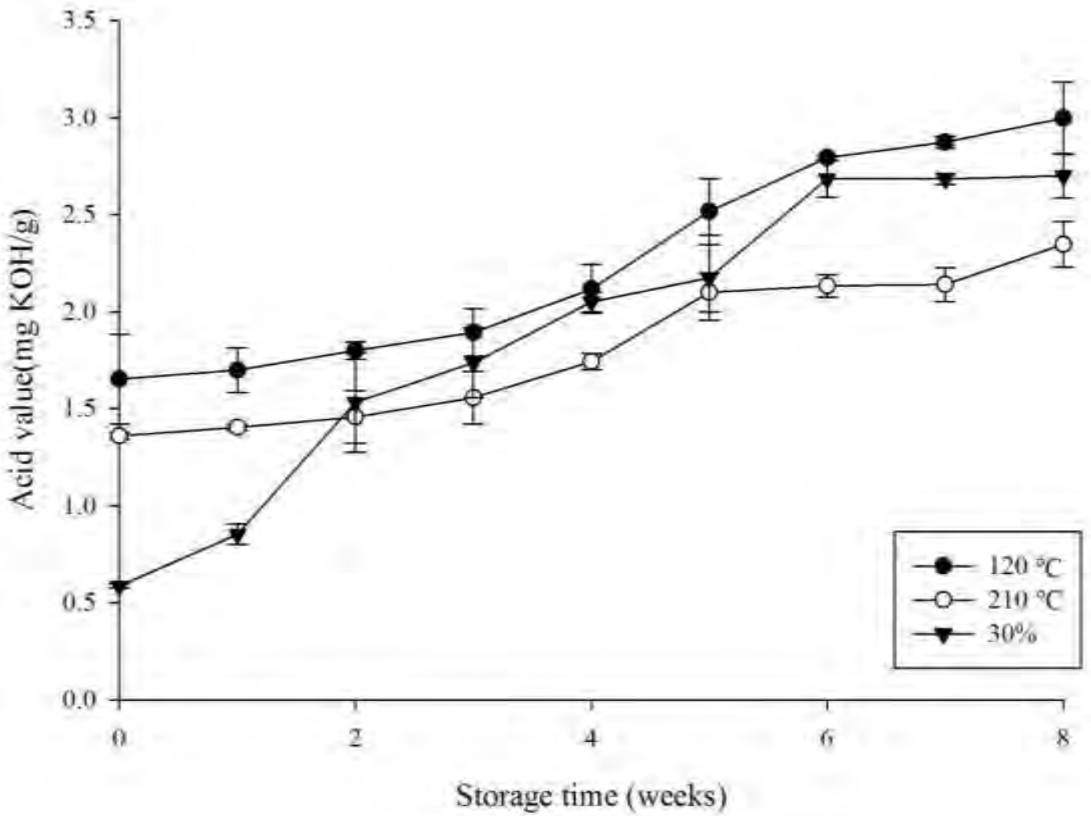


Figure 10. Change on acid value of heating temperature and ethanol extract concentration of traditional heating processing and ethanol extract sesame oil during the incubation at 65℃. Each value was expressed as means ± standard deviation from three data.

Storage Test-Peroxide Value

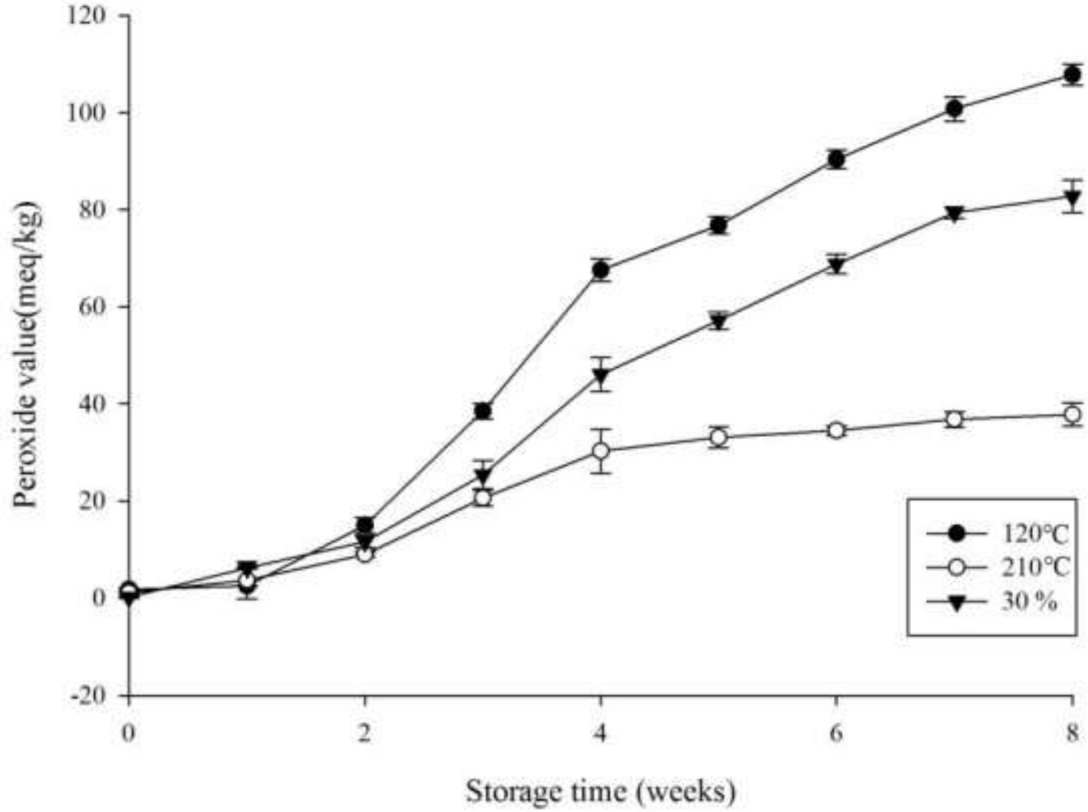


Figure 11. Change on peroxide value of heating temperature and ethanol extract concentration of traditional heating processing and ethanol extract sesame oil during the incubation at 65°C. Each value was expressed as means ± standard deviation from three data.

Storage Test-Total Phenol Contents

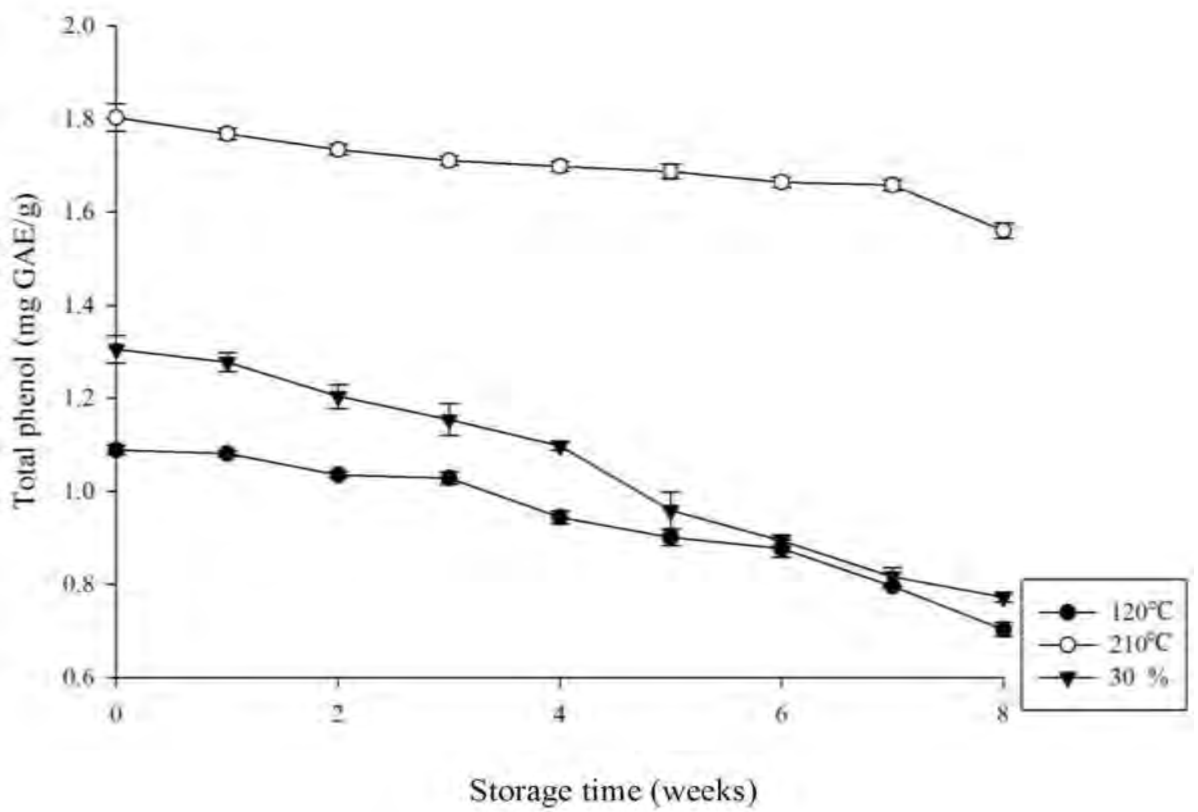


Figure 12. Change on total phenol contents of heating temperature and ethanol extract concentration of traditional heating processing and ethanol extract sesame oil during the incubation at 65°C. Each value was expressed as means ± standard deviation from three data.

Table 4. Change on lignans of heating temperature and ethanol extract concentration of traditional heating processing and ethanol extract sesame oil during the incubation at 65℃ for eight weeks

		Sesamin (mg/g)	Sesamolin (mg/g)	Sesamol (mg/g)	Total (mg/g)	Sesamolin + Sesamol (mg/g)
120℃	0 weeks	20.37±1.01 [a]	6.74±0.22 [b]	0.13±0.01 [b]	27.25±1.23 [a]	6.87±0.23 [b]
	8 weeks	15.04±0.73 [c]	6.68±0.22 [b]	0.13±0.01 [b]	21.86±0.95 [b]	6.81±0.22 [b]
210℃	0 weeks	16.08±0.48 [c]	3.98±0.42 [c]	0.52±0.05 [a]	20.59±0.94 [b]	4.51±0.47 [c]
	8 weeks	12.42±1.56 [d]	3.64±0.41 [c]	0.46±0.41 [a]	16.53±1.93 [c]	4.10±0.36 [c]
30%	0 weeks	18.34±0.67 [b]	8.58±0.83 [a]	ND	26.92±1.50 [a]	8.58±0.83 [a]
	8 weeks	14.69±1.25 [c]	7.39±0.43 [b]	ND	22.08±1.68 [b]	7.39±0.43 [b]

Each value was expressed as means ± standard deviation from three data.

[a-d] Means followed by the different letters in the same column are significantly different (p <0.05).

Storage Test-color analysis-L value

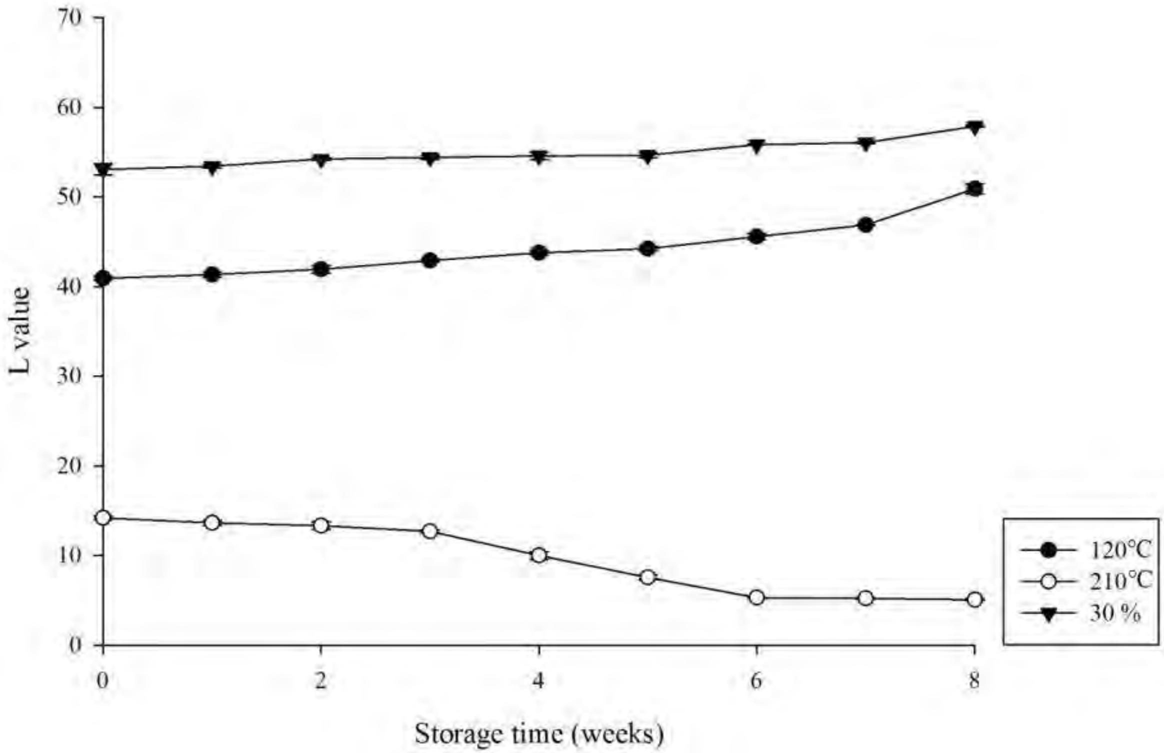


Figure 13. Change on L value of heating temperature and ethanol extract concentration of traditional heating processing and ethanol extract sesame oil during the incubation at 65°C. Each value was expressed as means ± standard deviation from three data.

Storage Test-color analysis-a value

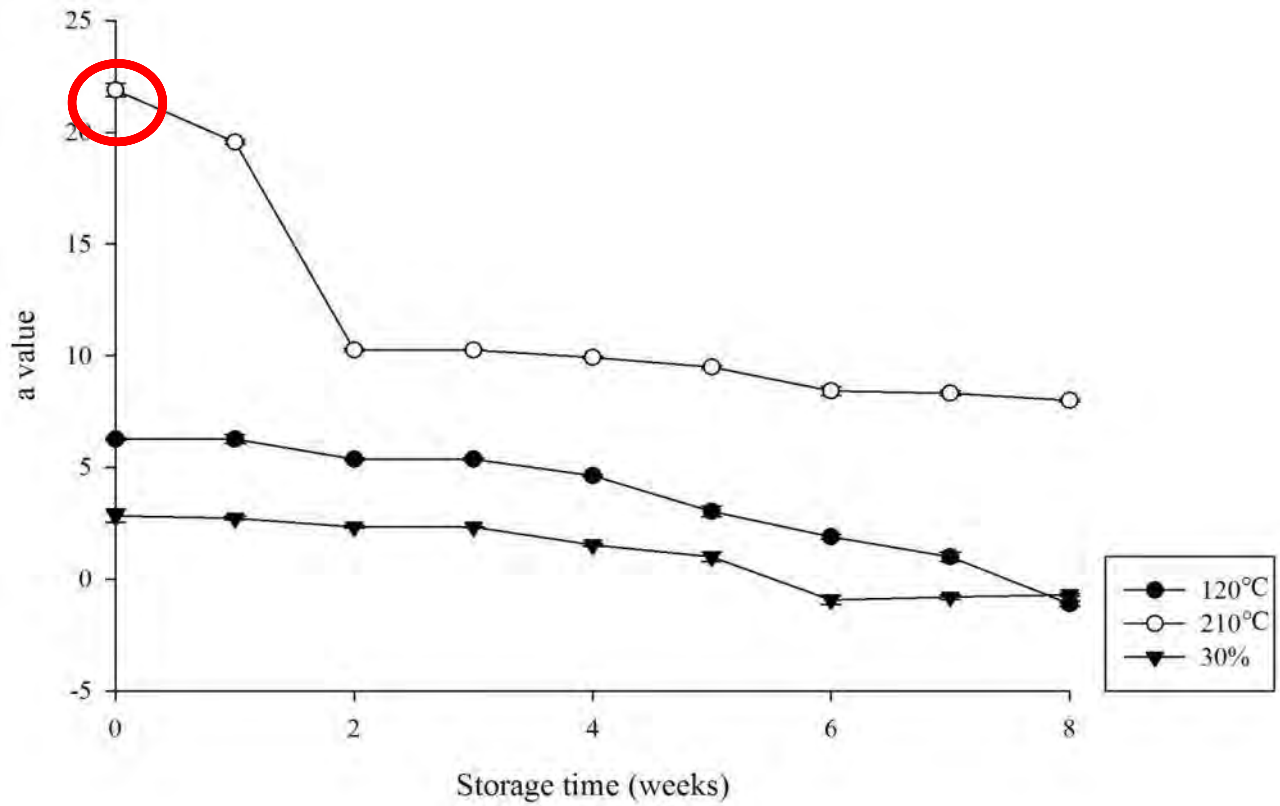


Figure 14. Change on a value of heating temperature and ethanol extract concentration of traditional heating processing and ethanol extract sesame oil during the incubation at 65°C. Each value was expressed as means ± standard deviation from three data.

Storage Test-color analysis-b value

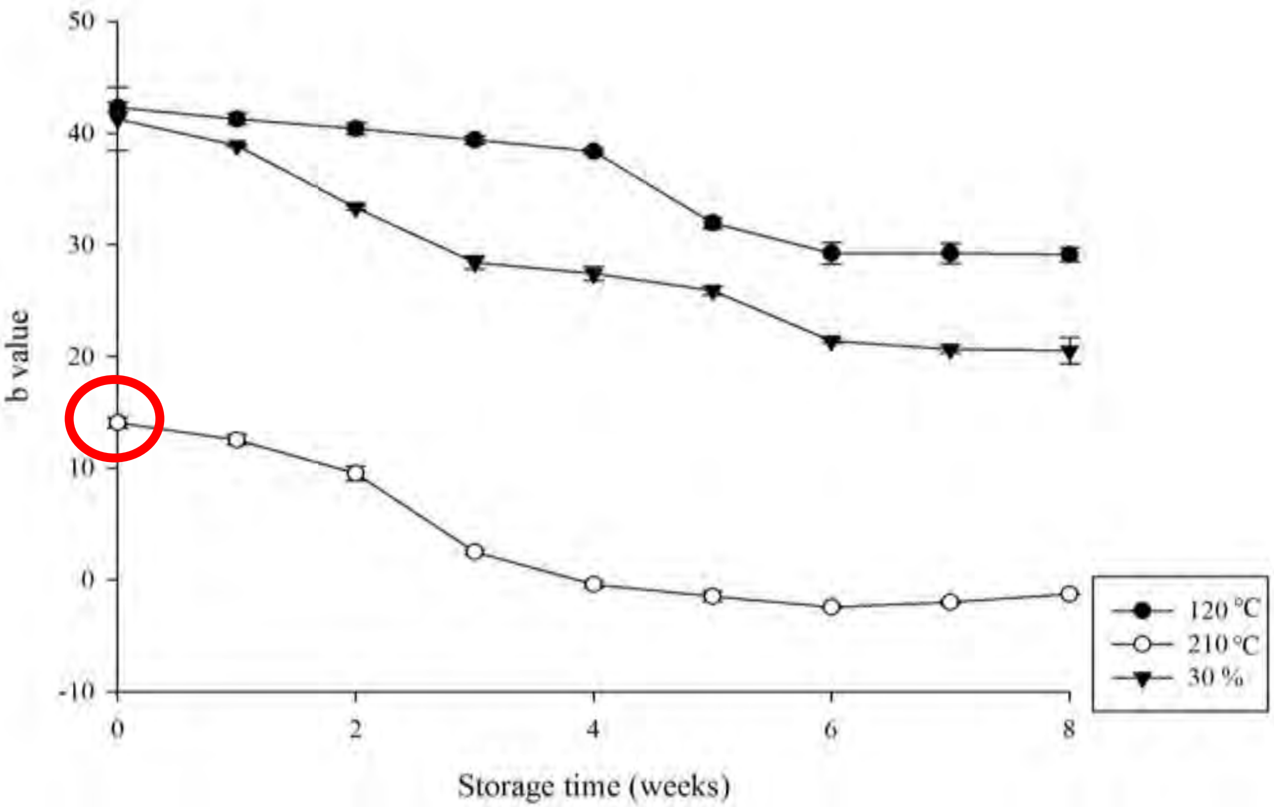
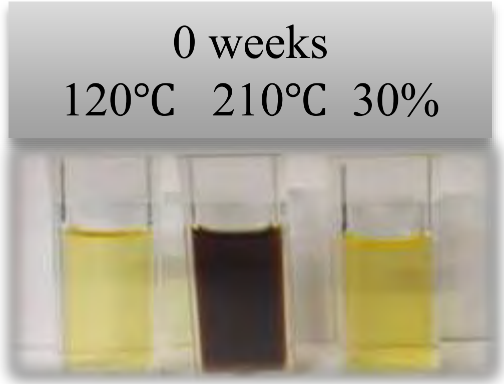


Figure 15. Change on b value of heating temperature and ethanol extract concentration of traditional heating processing and ethanol extract sesame oil during the incubation at 65°C. Each value was expressed as means ± standard deviation from three data.



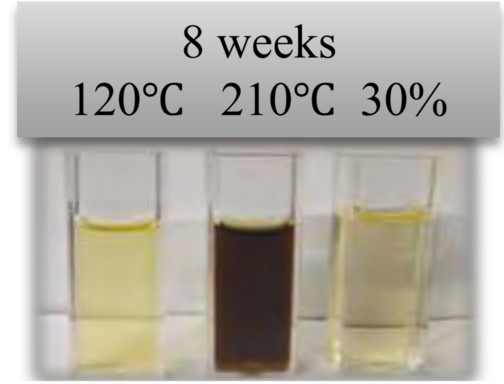


Table 5. Change on ΔE* of heating temperature and ethanol extract concentration of traditional heating processing and ethanol extract sesame oil during the incubation at 65℃ for eight weeks

ΔE*			
	120℃	210℃	30%
4 weeks	5.22±0.08 [c]	19.21±0.15 [a]	12.52±0.72 [b]
8 weeks	18.21±1.35 [a]	19.96±0.62 [a]	20.17±1.32 [a]

Each value was expressed as means ± standard deviation from three data.
[a-c] Means followed by the different letters are significantly different ($p < 0.05$).

Conclusions

- **On quality analysis, 95% ethanol extract oils shows best performance on lowest acid vale, peroxide value and thiobarbituric acid value.**

- **While on antioxidant content analysis, 210 ℃ traditional process oil shows the highest concentration of phenolic contents , flavonoid and scavenging activity.**

- **30% ethanol extract oils shows quite good antioxidant capacity and favorite amber color .It has potential to replace low temperature roasted sesame oil for blended edible oil, salad and sauces of the use in the near future.**


Thank You!

Acid value

Determination of free fatty acid.

(CNS 3647-N 6082)

Peroxide value

Determination of peroxides which be stated an indicator of the primary level of oil oxidation.

(CNS 3650-N 6085)

Hunter L, a, b

- L scale: Dark vs. Light where a low number (0-50) indicates dark and a high number (51-100)indicates light.
- a scale: Red vs. Green where a positive number indicates red and a negative number indicates green.
- b scale: Yellow vs. Blue where a positive numbe rindicates yellow and a negative number indicates blue.

Total phenolic contents

The amount of total phenolic compounds was measured using the method. Sample solution was added to 2% Na_2CO_3 and Folin-ciocalteu reagent was added. Absorbance was measured at 760nm. And the results were expressed as gallic acid.

(Tahvanainen *et al.*,2000)

Total flavonoid contents

Sample solution, mixed with then 5% $NaNO_2$ solution and $AlCl_3$ solution was added. Then, 1 mol/L NaOH were added, absorbance was measured at 510 nm. The total flavonoid content was expressed as quercetin.

(Shen *et al.*,1999)

DPPH radical scavenging activity

DPPH radical is scavenged by antioxidants through the donation of proton forming the reduced DPPH. The color changes from purple to yellow after reduction, which can be quantified by its decrease of absorbance at wavelength 517 nm.

(Shimadaet *al.*,1992)

ABTS radical scavenging activity

Measure the loss of color when an antioxidant is added to the blue–green ABTS+. The antioxidant reduces ABTS+ to ABTS and decolorize it. Trolox, a water-soluble subtance of vitamin E, can be used as an antioxidant standard. Absorbance was measured at 734 nm.

(Luypaert *et al*.,2004)

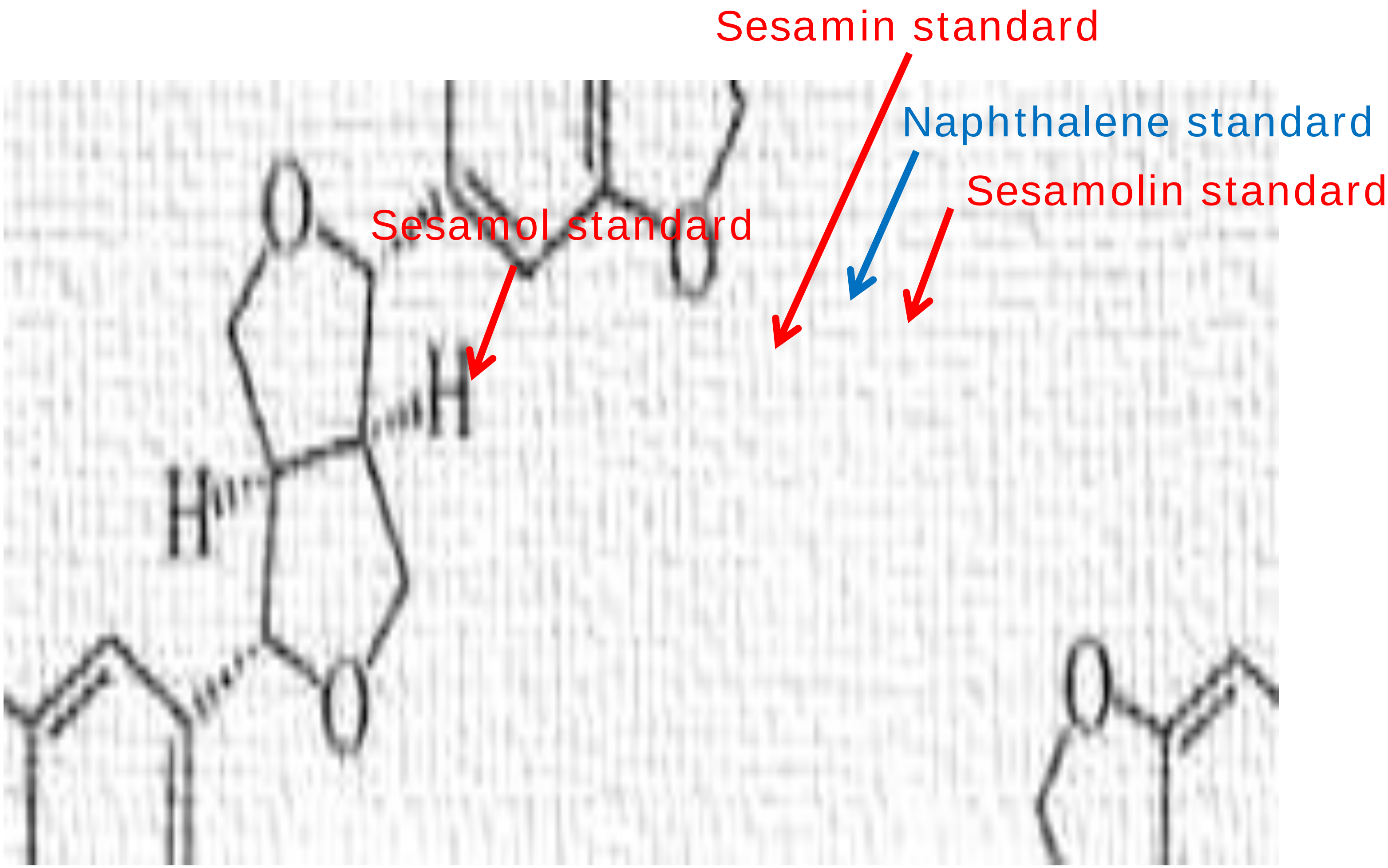
Reducing power

Solution were mixed with 0.2 M phosphate buffer and 1% potassium ferricyanide. Then, 10% trichloroacetic acid were added, and the tubes were centrifuged. Thenthe upper layer were mixed with methanol and 0.1% ferric chloride, and the absorbance of the reaction mixtures was measured at 700 nm.

(Oyaizu et al.,1986)

Ferrous iron chelating ability

The ferrous ion was monitored by measuring the formation of a red ferrous ion-ferrozine complex . Samples solution were mixed with Ferrous Chloride(2 mM) and ferrozine added to a concentration of 5 mM to start the reaction. The absorbance of the solution was measured at 562 nm. (Decker and Welch,1990)



圖十六、sesamin, sesamolin, sesamol, naphthalene 標準品分別有一顯著波峰的高效液相層析圖

Figure 16 . HPLC profile of sesamin, sesamolin, sesamol, naphthalene standards showing one sharp peak with Individual retention times .

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表1 食用芝麻油品質要求(續)

項目	等級	
	壓榨芝麻油	精製芝麻油
不皂化物(%m/m)	3.0以下	2.0以下
過氧化價 (milliequivalents of active oxygen/kg Oil)	15以下	10以下

表 1 食用芝麻油品質要求

項目	等級	
	壓榨芝麻油	精製芝麻油
一般性狀	大致澄清，有芝麻油特有之香味	透明澄清，風味良好
顏色	具芝麻油特有顏色	
水分及揮發物(%m/m)	0.25 以下	0.2 以下
夾雜物(%m/m)	0.1 以下	0.05 以下
比重(20 ℃/20 ℃)	0.915～0.924	
折射率(ND 40 ℃)	1.465～1.469	
碘價	104～120	
酸價(mg KOH/g Oil)	4.0 以下	0.6 以下
皂化價(mg KOH/g Oil)	186～195	

	Press rate %
120°C	40
150°C	42
180°C	44
210°C	46
30%	30
50%	25
75%	23
95%	20

	Water content (%)
Sesame (raw material)	6.50±0.15