

Chemical composition and oxidative stability of safflower oil prepared from safflower seed roasted with different temperatures

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Abstract

The chemical composition and oxidative stability of safflower oil prepared from the seed roasted, at different roasting temperatures (140–180 °C), were evaluated and compared with those of unroasted safflower oil. The colour development and phosphorus content of oils increased significantly as roasting temperature increased. The fatty acid compositions of safflower oils did not change with roasting temperature. The major fatty acid was linoleic acid (ca. 80%). Four phospholipid classes, namely, PE, PI, PA and PC, were identified. The major phospholipid component of safflower seed oil is PI. However, the proportion of PI in the safflower oil increased significantly as roasting temperature increased ($P < 0.05$), but, PE in safflower oil decreased significantly as roasting temperature increased ($P < 0.05$). Tocopherol and tocotrienol homologues were identified, namely, α -, β -, and γ -tocopherols, and γ - and δ -tocotrienols, whereas no δ -tocopherol, or α -, and β -tocotrienols were detected. The major tocopherol in safflower oil was α -tocopherol. The content of α -tocopherol in safflower oil gradually increased from 441 to 520 mg/kg as roasting temperature increased from 140 to 180 °C. The oxidative stability showed that, as the roasting temperature increased, the oxidative stability of safflower oil increased.

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1. Introduction

Safflower oil is used as a condiment oil, along with sesame, red pepper and perillar oils in Korea. Traditionally, these condiment oils are prepared by extracting the roasted seed with a mechanical press or expeller after the seeds have been roasted at the appropriate temperatures (Kim, Kim, & Lee, 1998; Yoshida & Takagi, 1997). During the roasting process, pleasant aroma or taste (nut-like or peanut butter-like), that transfers to the oil during extraction, is developed. The conventional method for the preparation of condiment oils, such as sesame, red pepper and perillar oils, involves cleaning, roasting and pressing but not refining (Kim et al., 2002). The roasting process is the key step

for making condiment oil, since the colour, flavour, composition and quality of the oil are all influenced by the conditions of the process. Some researchers (Jung, Bock, Baik, Lee, & Lee, 1999; Kim et al., 2002; Yen, 1990; Yoshida & Takagi, 1997) reported that the chemical composition of an oil is independent of the roasting temperature used for preparing it. However, little investigation has been conducted on the effects of roasting on the chemical composition or oxidative stability of safflower oil. Recently, roasted safflower seed was investigated as medicinal food for bone formation in Korea. The powder of roasted safflower seed influenced the recovery of bone fracture by accelerating the process of bone repair in rat (Kim et al., 1998).

The objective of this study was to investigate the changes in fatty acid composition, colour formation and minor components, such as tocopherol, tocotrienol, and phospholipids, and oxidative stability of the oil prepared from safflower seed roasted at different temperatures using an electric roaster.

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2. Materials and methods

2.1. Safflower and reagents

Safflower used in this study, was purchased from Kyungbook Agricultural Cooperative Federation (Kyungbook, Korea). Tocopherol homologues were purchased from Merck (Darmstadt, Germany) and tocotrienol homologues were purchased from Calbiochem (Calbiochem-Novabiochem Co., San Diego, CA). Phospholipid standards were purchased from Sigma Chemical Company (St. Louis, MO). Other chemicals used in this study were of analytical grade.

2.2. Preparation of safflower oil

Safflower seeds (1 kg) were roasted in an electric roaster equipped with a stirrer and a temperature controller. Safflower seeds were roasted with constant stirring up to 140, 160 and 180 °C. Times reached up to 140, 160, and 180 °C were 16.05 ± 0.30 , 19.36 ± 0.32 and 24.08 ± 1.02 min, respectively. The roasted safflower seeds were immediately transferred to the mechanical press (Dongkwang Oil Co, Seoul, Korea) and then pressed at 600 kg/cm² for 20 min to obtain the safflower oil. The unroasted safflower oil was prepared by the same procedure as described above but without roasting. The extracted safflower oil was filtered with cheese cloth under vacuum to remove particles.

2.3. Determination of colour development

As an index of colour development (Yoshida, Takagi, & Mitsuhashi, 1999), the absorbance at 420 nm of 5.0% (w/v) solutions of oils in chloroform was determined with a spectrophotometer (UV-900; JASCO, Tokyo, Japan).

2.4. Fatty acid composition

Oils were esterified according to the AOCS standard method Ce 2-66 (1990). Methyl esters of fatty acids (FA) were extracted with hexane. Then 1- μ l aliquots of the extracts were injected into a gas chromatograph (Varian 3800; Varian Inc., Walnut Creek, CA) equipped with a FID. The column used was a Supelcowax 10 fused-silica capillary column (30 m \times 0.32 mm i.d.; Supelco, Bellefonte, PA). The carrier gas was helium, and the total gas flow rate was 20 ml/min. The injector, oven, and detector temperatures were 240, 190, and 260 °C, respectively.

2.5. Phospholipid analysis

To analyze phospholipids by HPLC (Kim et al., 2002), phospholipids were isolated from the oils as follows:

triplicate samples of 2 g oil were fractionated on a 20 g column of silicic acid (100–200 mesh, Sigma) with sequential elution by 200 ml chloroform, 100 ml acetone, and 200 ml methanol. The methanol was removed in a rotary evaporator at 35 °C; the sample residues were then frozen (–60 °C) until analysis. Phospholipid analyses were performed with a HPLC (PU-1580; JASCO), connected to a Rheodyne injector, with a 20 μ l sample loop and an ELSD (Shedex 55; Richard Scientific, Novata, CA). Phospholipids isolated by silicic acid column were analyzed on a normal-phase column, Lichrospher Si-60 (250 \times 4.6 mm i.d.; Merck Co.), and a linear gradient elution from (A) chloroform/tertiary-butylmethyl ether (75:15, v/v) to (B) methanol/ammonium hydroxide/chloroform (92:7:1, by vol.) at 0.5 ml/min for 30 min and held at (B) for 10 min. This was followed by a reverse linear gradient to the starting solvent (A) at 0.5 ml/min for 10 min.

2.6. Tocopherol and tocotrienol contents

Tocopherol and tocotrienol contents were measured according to the method of Kim et al. (2002). One gram of the safflower oil, 4 ml of 5% pyrogallol solution in ethanol, and a few boiling chips were placed in a 120-ml round-bottomed flask fitted with a reflux condenser and heated on a hot plate. When the mixture started boiling, the condenser was removed and 1 ml 50% aqueous potassium hydroxide solution was added. The sample was saponified for 5 min. After saponification, the flask was placed in an ice bath and 20 ml water and diethyl ether were added. The mixture was transferred to a 250-ml separatory funnel. Extraction of sample with 30 ml diethyl ether was repeated twice. The pooled diethyl ether layer was washed three times with 20 ml distilled water, filtered through anhydrous sodium sulfate, and then evaporated at 30 °C. The remaining sample was diluted with 10 ml *n*-hexane and filtered through a Milipore 0.45 FH membrane and injected into the liquid chromatograph (LC). The LC system consisted of a HPLC (PU-1580; JASCO) connected to a Rheodyne (Rohnert Park, CA) injector with a 20 μ l sample loop and a fluorescence detector (FP-1520; JASCO) with excitation set at 298 nm and emission set at 325 nm. A Lichrospher Si-60 column (250 \times 4.6 mm i.d.; Merck Co.) was used. The mobile phase was *n*-hexane/2-propanol (99:1 by vol.) at 1.0 ml/min.

2.7. Oxidative stability of unroasted and roasted safflower seed oils

To study the oxidative stability of unroasted and roasted safflower seed oils, 60 g of oil were transferred, in triplicate, to a 100 ml capacity glass beaker. The samples were stored in forced-draft air oven at 60 °C for 26 days. The oxidative stabilities of oils were studied by

measuring the increase in peroxide content according to the AOCS standard method Cd 8-53 (1990) and conjugated diene content in the oils (Jung et al., 1999). For the determination of conjugated diene, samples were diluted with 2,2,4-trimethylpentane. The absorptivities of the prepared samples were then measured at 233 nm, and the contents of the conjugated diene were expressed as absorptivities of the 1% safflower seed oils in 2,2,4-trimethylpentane at 233nm.

2.8. Statistical analysis

Each reported value is the mean of determinations for triplicate samples prepared from each roasting condition, and the data were analyzed by ANOVA and Duncan's multiple range test (Duncan's test). Statistical significance was accepted at a level of $P < 0.05$ (SAS Institute, 1996).

3. Results and discussion

3.1. Colour development

The colour development of safflower oil prepared at different roasting temperatures is shown in Fig. 1. With increasing the roasting temperature, browning substances were developed, resulting in significant ($P < 0.05$) increase of the absorbance at 420 nm. The colour of oils from safflower seed changed gradually from light-yellow (absorbance; 0.093) before roasting to brown (absorbance; 0.202 and 0.239) at 140 and 160 °C and finally to deep-brown (absorbance; 0.332) at 180 °C. Therefore, the colour formation in the oil was influenced by roasting temperatures. The formation of browning substances in several thermally processed foods results from Maillard-type non-enzymatic reactions between reducing sugars and free amino acids or amides

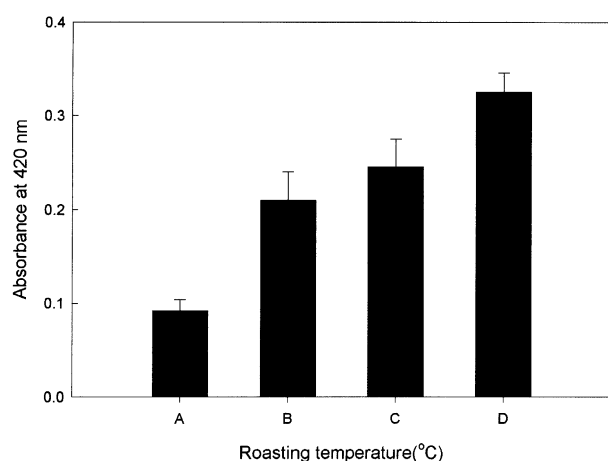


Fig. 1. Changes in absorbance (colour) of safflower oils prepared from safflower seed, unroasted or roasted at 140, 160, and 180 °C in an electric oven. A: Unroasted B: 140 °C, C: 160 °C, D: 180 °C.

Table 1
Fatty acid composition of safflower oils prepared from safflower seed, unroasted or roasted at 140, 160, and 180 °C in an electric oven

Fatty acid (%)	Roasting temperature (°C)			
	Unroasted	140	160	180
C16:0	5.5±0.1c	5.3±0.1c	5.8±0.4c	5.0±0.3c
C18:0	1.6±0.3d	1.7±0.1d	1.6±0.3d	1.7±0.2d
C18:1	11.1±1.3e	11.5±0.6e	10.9±0.3e	10.8±0.9e
C18:2	81.4±3.7f	81.0±3.9f	81.1±4.3f	82.0±2.3d
C18:3	0.4±0.1g	0.5±0.0g	0.6±0.3g	0.5±0.1g

Mean values±SD of determinations for triplicate samples. Value of the unroasted samples and values in the same line (within the same subgroup) with different letters (c–g) are significantly different ($P < 0.05$) as measured by Duncan's test.

(Koechler & Odell, 1970). The increase in colour of oils with increasing roasting temperature seemed to be due to non-enzymatic browning at the elevated roasting temperatures. Previous studies (Kim et al., 2002; Yen, 1990; Yosida, 1994) have reported that an increase in the roasting temperature of seeds, such as rice germ and sesame seed, resulted in significant increase in colour of oils, which is consistent with our results.

3.2. Fatty acid (FA) composition

FA composition of an oil can be an indicator of its stability, physical properties, and nutritional value. There were almost no differences in FA composition of safflower oils prepared at different roasting temperatures (Table 1). Safflower oil (unroasted) consisted of 5.53% palmitic, 1.62% stearic, 11.00% oleic, 81.5% linoleic, and 0.40% linolenic acids. Safflower oil from seeds roasted at 180 °C consisted of 4.99% palmitic, 1.69% stearic, 10.9% oleic, 82.0% linoleic, and 0.41% linolenic acids. Previous studies (Kim et al., 2002; Yen, 1990; Yosida, 1994) have reported that no differences in FA compositions of rice germ and sesame seed oils prepared at different roasting temperatures and times. Common commercial safflower, with oil containing about 80% linoleic acid (high linoleic acid) is the leading type of safflower grown; about 80% of all safflower grown in the United States is of this type (Knowles, Bell, & Ruckman, 1965). Another type of safflower (high oleate), with oil containing about 80% oleic acid, may be of increasing interest because it is mono-unsaturated (Knowles et al., 1965). However, commercial safflower, with oil containing about 80% linoleic acid (high linoleic acid) is the leading type of safflower grown in Korea from our results.

3.3. Phospholipid contents

The phosphorus contents and phospholipid distributions determined by HPLC-ELSD for safflower germ

Table 2

Changes in phospholipid composition and phosphorus contents of safflower oils prepared from safflower seed, unroasted or roasted at 140, 160, and 180 °C in an electric oven

Roasting temperature (°C)	Phosphorus content (ppm)	Phospholipid classes (%)			
		PE	PI	PA	PC
Unroasted	19.4±1.2b	59.9±2.1 b	32.6±2.2 b	4.9±0.1 b	2.6±0.2 b
140	111±2.3c	22.1±3.2 c	60.8±1.3 c	1.5±0.2 c	15.6±0.9 c
160	141±2.4d	17.8±1.2 d	64.5±1.3 d	1.9±0.3c	15.8±1.1 c
180	175±4.3e	12.4±0.6 e	70.9±2.2 e	1.3±0.2 c	15.4±1.4 c

Mean values±SD of determinations for triplicate samples. Value of the unroasted samples and values in the same column (within the same subgroup) with different letters (b–e) are significantly different ($P < 0.05$) as measured by Duncan's test.

oils prepared at different roasting temperature are presented in Table 2. There were significant ($P < 0.05$) differences in the phosphorus content of oils prepared at different roasting temperatures. With increasing roasting temperature, phosphorus contents significantly increased. Phosphorus contents of oils prepared from safflower seeds roasted at 140, 160, and 180 °C were 100, 140 and 175 ppm, respectively, whereas that of oil prepared from unroasted safflower seed was 19 ppm. Veldsink et al. (1999) reported that the phosphorus content of rapeseed and sunflower oils significantly increased as the preheating temperature of the oilseeds increased. Clark and Snyder (1991) also reported that, at a higher pretreatment temperature, a large amount of phosphorus was extracted. Our results confirmed these observations. Four phospholipid classes, such as phosphatidylethanolamine (PE), phosphatidylinositol (PI), phosphatidic acid (PA) and phosphatidylcholine (PC), were identified. The major phospholipid component of safflower seed oil is PI. However, the proportion of PI in the safflower oil increased significantly ($P < 0.05$) as roasting temperature increased. Kim et al. (2002) also reported that the PI content of rice germ oils significantly increased as the roasting temperature and time of rice germ increased. However, PE in safflower oil decreased significantly as roasting temperature

increased ($P < 0.05$). The amount of PE relative to other phospholipids may decrease, but this is mainly due to the increase in PI.

3.4. Tocopherol and tocotrienol contents

The contents of the individual tocopherols and tocotrienols in oils prepared at various roasting temperatures are given in Table 3. Three tocopherol homologues, i.e. α -, β -, and γ -tocopherols, and two tocotrienol homologues, i.e. γ - and δ -tocotrienols, were identified, whereas no δ -tocopherol, or α - or β -tocotrienols were detected. The major tocopherol in safflower oil was α -tocopherol. The content of α -tocopherol in safflower oil gradually ($P < 0.05$) increased as roasting temperature increased. For example, the contents of α -tocopherol in safflower oils roasted at 140, 160, and 180 °C were 441±12.6, 477±10.1, and 520±12.3 mg/kg. Similar trends were observed in β -, and γ -tocopherols. However, there were no significant differences in the contents of tocotrienols. Yoshida et al. (1995) reported that the content of tocopherol in sesame oils prepared by microwave oven heating decreased over time. On the other hand, Yen (1990) reported that the level of tocopherol in sesame oils prepared by electric oven heating was increased by roasting at temperatures

Table 3

Changes in tocopherol and tocotrienol compositions of safflower oils prepared from safflower seed, unroasted or roasted at 140, 160, and 180 °C in an electric oven

Tocopherol homologues (mg/kg oil)	Roasting temperature (°C)			
	0	140	160	180
α -Tocopherol	386±12.7b	441±12.6c	477±10.1d	520±12.3e
β -Tocopherol	8.9±1.1b	8.6±1.4b	11.8±0.2c	12.4±0.9c
γ -Tocopherol	2.4±0.5b	4.0±1.8b	6.5±0.1bc	7.7±0.5c
δ -Tocopherol	NDc	NDc	NDc	NDc
α -Tocotrienol	NDc	NDc	NDc	NDc
β -Tocotrienol	NDc	NDc	NDc	NDc
γ -Tocotrienol	5.2±0.6c	3.8±1.5c	7.0±0.1d	5.8±0.6c
δ -Tocotrienol	8.4±0.8c	7.8±1.3c	7.5±0.6c	7.5±0.1c

Mean values±SD of determinations for triplicate samples. Value of the unroasted samples and values in the same line (within the same subgroup) with different superscript letters (b–e) are significantly different ($P < 0.05$) as measured by Duncan's test.

up to 200 °C. Lane, Quereshi, and Salsler (1997) also reported that a heat pre-treatment (over range of 100–175 °C) by a convection oven caused an increase in level and yield of tocopherol in rice bran oil. Kim et al. (2002) also reported that the content of α -tocopherol in rice germ oil increased as roasting temperature and time increased. This results suggested that damage to the membrane by heating allow increased release of tocopherols.

3.5. Oxidative stability of safflower oils

The oxidative stability tests clearly showed that, as the roasting temperature increased, the oxidative stability of safflower oil increased. Fig. 2 shows the changes in peroxide values in unroasted and roasted safflower oils during storage at 60 °C. Peroxide value in unroasted safflower oil increased ($P < 0.05$), resulting in 241 ± 7.84 meq/kg oil after 15 days of storage at 60 °C. Peroxide values of the oils obtained from safflower seeds roasted at 180 °C, however, increased slowly, resulting in less than 64.2 ± 3.23 meq/kg oil after 15 days of storage at 60 °C. After 15 days of storage, peroxide values of the oils from safflower seeds, unroasted and roasted at 140, 160, and 180 °C were 241 ± 7.84 , 169 ± 6.20 , 123 ± 5.37 and 64.2 ± 3.23 meq/kg oil, respectively. Fig. 3 shows the changes in the conjugated diene contents in safflower oils during storage at 60 °C. Conjugated diene contents of safflower oils increased gradually ($P < 0.05$) as the storage time increased. Oxidative stability of safflower oils, based on the changes of conjugated diene contents, were in an agreement with those estimated by peroxide value development. Therefore, the oils from safflower seeds roasted at higher temperatures had a much greater oxidative stability than oils from safflower seeds unroasted or roasted at lower temperature. Our

present results were in accordance with previously reported results for sesame oils (Yen & Shyu, 1989), showing that oxidative stability of sesame seed oil increased with increasing roasting temperature. Probably the greater antioxidative stability of safflower oil prepared from safflower seeds roasted at higher temperatures was due to non-enzymatic reaction products formed during the roasting process. As seen in Fig. 1, the higher the roasting temperature, the darker the oil; i.e. the higher the roasting temperature, the greater the formation of non-enzymatic reaction products. Maillard reaction products, formed through the interaction of proteins with reducing sugars, reportedly show strong antioxidant activities (Beckel & Waller, 1983; Elizade, Rosa, & Lerici, 1991; Elizade, Bressa, & Rosa, 1992; Jung et al., 1999; Lee, 1992).

In summary, the chemical composition and oxidative stability of safflower oil prepared from the seed roasted at different roasting temperatures (140–180 °C) were evaluated and compared with that of unroasted safflower oil. Colour phosphorus, phospholipid, tocopherol, and tocotrienol contents of oils varied with the roasting temperature. However, fatty acid compositions of safflower oils did not change with roasting temperature. Commercial safflower, with oil containing about 80% linoleic acid (high linoleic acid), is the leading type of safflower grown in the Korea. The major phospholipid component of safflower seed oil is PI. However, the proportion of PI in the safflower oil increased significantly as roasting temperature increased ($P < 0.05$). Tocopherol and tocotrienol homologues were identified, namely α -, β -, and γ -tocopherols, and γ - and δ -tocotrienols, but no δ -tocopherol, or α -, and β -tocotrienols were detectable. The oxidative stability showed that, as the roasting temperature increased, the oxidative stability of safflower oil increased.

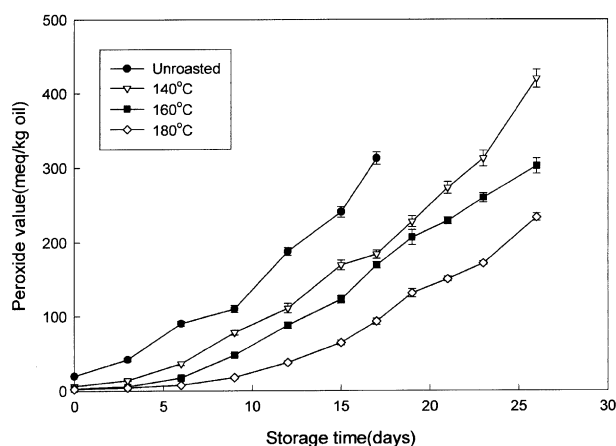


Fig. 2. Changes in peroxide values of safflower oils prepared from safflower seed, unroasted or roasted at 140, 160, and 180 °C in an electric oven during storage.

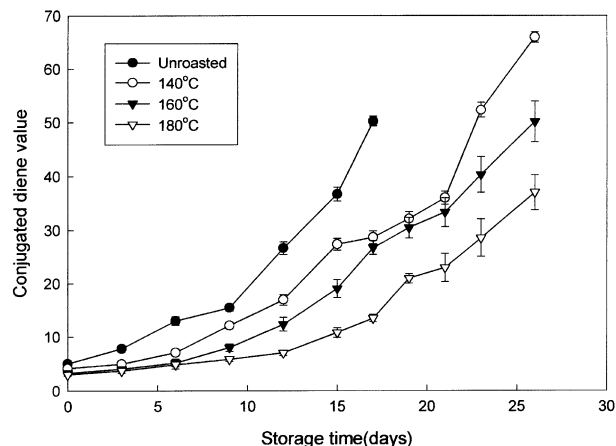


Fig. 3. Changes in conjugated diene values of safflower oils prepared from safflower seed, unroasted or roasted at 140, 160, and 180 °C in an electric oven during storage.

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