JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

Quantitation of the Main Constituents of Some Authentic Sesame Seed Oils of Different Origin

COLIN CREWS,*,† PATRICK HOUGH,† PAUL BRERETON,† JOHN GODWARD,† MICHELLE LEES,‡ SEBASTIEN GUIET,‡ AND WILFRIED WINKELMANN§

Central Science Laboratory, Sand Hutton, York YO411LZ, United Kingdom, Eurofins Scientific, Rue Pierre Adolphe Bobierre, BP 42301, 44323 Nantes Cedex 3, France, and Eurofins/Wiertz-Eggert-Jorissen, Stenzelring 14 b, D-21107 Hamburg, Germany

This paper describes the composition of sesame seed oils obtained from seeds collected from five countries that are major suppliers of traded sesame seed oil. Oils were extracted from the seeds using small-scale industry pressing equipment and analyzed using standard methods for fatty acids, fatty acids in the triglyceride 2-position, tocopherols and tocotrienols, triglycerides, sterols, steradienes, and iodine value. Values for the composition of the sterols, triglycerides, fatty acids, iodine value, and tocopherol composition were generally in good agreement with the results published elsewhere. All of the oils from roasted seeds contained low levels of the sterol degradation products stigmasta-3,5-diene and campesta-3,5-diene, which were probably formed by dehydration of the parent sterols during roasting.

KEYWORDS: Sesame seed oil; *Sesamum indicum*; fatty acids; triacylglycerol 2-position; tocopherols; tocotrienols; triacylglycerols; sterols; steradienes; iodine value

INTRODUCTION

Sesame, *Sesamum indicum* L., is an annual herb native to the tropics, which was formerly classified as *Sesamum orientale* L. The seeds contain about 40−60 wt % of oil, which is obtained by pressing, and further oil can be obtained by solvent extraction of the hull. Sesame seeds are sometimes roasted prior to pressing in order to produce an oil with a very distinctive flavor. The oil is used, mainly in the areas of production, for cooking and as a salad oil, for which purposes it is usually refined. Oil from roasted seeds is more popular in Europe, where it is used in the preparation of oriental style meals.

World production of sesame seed was about 3 million metric tons in 2004. The major producers were India, China, and Myanmar with 21%, 20%, and 17%, respectively, of the total (*1*). Although not a major grower of sesame, Japan is a major seed importer and produces about 6% of the world's sesame seed oil.

Sesame contains significant amounts of the lignans sesamin and sesamolin. These compounds have beneficial effects on serum lipid levels and liver function and give sesame seed oil a marked antioxidant activity. The lignans are also responsible for the great stability of sesame seed oil to oxidation.

Databases of the composition of edible vegetable oils such as that prepared by the Codex Alimentarius Commission (*2*) can be used to monitor authenticity and obtain information on the quality of the oil in terms of storage stability, flavor, taste, and nutrition. Such information can help in assessing dietary intake of beneficial components of the oil.

This work is part of a project to study the composition of authentic oils obtained from seeds and nuts collected in the major producing countries and analyzed by well-established methods using stringent quality control procedures. Previous publications described the composition of hazelnut (*3*) and walnut oils (*4*). The current paper describes the results for a study of sesame seed oils obtained and analyzed under similar conditions.

The production uses and composition of sesame seed oils have been reviewed (*5*−*7*). Several papers have studied the fatty acid composition of sesame seed oils (*8*−*14*), often as part of experiments into the effects of roasting. The published work shows that sesame contains a high proportion of unsaturated acids with oleic and linoleic acids in equal amounts. However, with the exception of earlier authenticty-related work (*7*) none of these studies include a large enough number of samples to produce reliable tables of composition with respect to the minor acids. The situation with the sterol composition is similar with very few recent publications (*7*, *15*, *16*) available and only the single larger survey (*7*) covering commercially traded seeds. Levels of tocopherols and tocotrienols have been measured (*7*, *10*, *16*−*21*), but again in almost all of these reports few samples were used with the emphasis frequently being on other components, other oils, or non-commercial varieties.

This paper reports the results of a survey which comprised a large number of samples (*33*) collected at the source and analyzed using a full audit trail to ensure authenticity. This is

* To whom correspondence should be addressed. E-mail: c.crews@csl.gov.uk.
† Central Science Laboratory.
‡ Eurofins Scientific.
§ Eurofins/Wiertz-Eggert-Jorissen.

10.1021/jf0603578 CCC: $33.50
Published on Web 07/15/2006

the first survey reporting results for seeds collected from countries chosen to reflect the current market share for sesame seed oil production and extracted under conditions very similar to those used in industry. Unlike most previous reports the determinations were made using using official methods with rigorous quality assurance procedures in place. Sesame seed oil is frequently used as the flavored product of roasted seeds; therefore, additional portions of some of the collected seed batches were roasted in the laboratory prior to expression of the oil sample for analysis.

MATERIALS AND METHODS

Sesame Seed. Sesame seeds were collected by the authors directly from growers in Burkina Faso (3 samples), China (6), Egypt (4), India (6), Japan (1), Mexico (4), Myanmar (1), and Thailand (8). The seeds included principally white and black varieties but with no obvious mixtures. Seeds were collected on various dates between 2000 and 2002.

The seeds were packed in secure heavy-duty polyethylene containers and sealed with numbered tamper-proof plastic tags with a unique number that was used at all stages from collection through analysis, allowing a detailed audit trail to be established. They were stored at 4 °C prior to oil pressing or roasting.

Roasted Sesame Seeds. To obtain samples of oil from roasted seeds, portions of some samples were heated in a modified laboratory oven as described previously (*3*). Precise details of the industry roasting conditions for sesame were not available. The temperatures and times used were therefore based on industry advice and published scientific work (*22*−*25*) in which conditions used were very variable. Typical light roasting procedures used 100−150 °C for 10 min, and for deep roasting temperatures were increased to 260 °C for 30 min.

Sesame Seed Oil. The oil was extracted soon after the seeds were received. A cold-press expeller (Statfold Seed Oils, Tamworth, U.K.) operating at a temperature close to 37 °C was used. The extracted oil was filtered at low pressure but not processed further. Normal steam cleaning procedures were carried out between samples to ensure that there was no adventitious mixing. The oils were then filtered and stored in polypropylene bottles under nitrogen at 4 °C in the dark prior to analysis.

Analysis. *Standards*. Standards of the sterols stigmasterol (95%) and sitosterol (containing campesterol), and of fatty acid methyl esters, were obtained from Sigma, Poole, U.K. The steradienes stigmasta-3,5-diene (69%) with campesta-3,5-diene (13%) and stigmasta-3,5,22-triene (97%) were obtained from Chiron AS, Trondheim, Norway. The internal standard cholesta-3,5-diene (95%) was obtained from Sigma. Tocopherols and tocotrienols (>95%) were obtained from Merck Biosciences, Schwalbach, Germany. The CBR reference sample RM162 (soya and maize oil) was purchased from the Community Bureau of Reference, Brussels, Belgium.

Analytical Methods. Determinations of the composition of the sterols, steradienes, FA, calculated iodine, FA in the triacylglycerol (TAG) 2-position, TCN, and tocopherol and tocotrienol composition and all statistical analyses were carried out as described previously (*3*).

RESULTS AND DISCUSSION

Oil Extraction. It was not certain that the volumes of oil returned from the press were quantitative; therefore, no data are presented for the oil content of the samples.

Quality Assurance. For the data reported all results for the replicate injection and analysis samples were within the warning limits of Shewart control charts, and all results for the reference materials were within specified limits.

Sterol Composition. The sterol composition of sesame seed oils expressed in milligrams per 100 g of oil is given in **Table 1**.

The total levels ranged from 324 to 798 mg/100 g with an average value of 531 mg/100 g. These values have a range similar to those reported in a previous survey (*7*); however, the mean value is much smaller. The samples contained surprisingly high levels of 24-methylenecholesterol (up to 22.9 mg/100 g) that has not been reported elsewhere. The identity of the 24-methylenecholesterol peak was confirmed by comparison of its retention time with that of a rapeseed oil known to contain 24-methylenecholesterol and by full scanning mass spectrometry. It is possible that in earlier studies the compound had not been resolved from campesterol. Brassicasterol was not detected in any unroasted sample; a small response for one roasted sample was not confirmed as brassicasterol.

Table 1. Sterol Composition of Sesame Seed Oils (mg/100 g)

sterol	unroasted oils	roasted oils
cholesterol	nd[a]–2.2	nd–2.5
cholestanol	nd–0.8	nd–0.4
brassicasterol	nd	nd–0.4
24-methylenecholesterol	5.1–22.9	7.0–13.8
campesterol	51.3–134.2	82.0–106.4
campestanol	nd–1.9	nd–0.8
stigmasterol	19.2–47.1	30.0–45.0
Δ7-campesterol	nd–2.9	nd–4.6
Δ5,23-stigmastadienol	nd–8.6	nd–3.4
clerosterol	2.3–22.4	2.7–7.8
β-sitosterol	190.9–428.7	303.1–379.6
β-sitostanol	nd–3.9	0.4–2.4
Δ5-avenasterol	31.2–81.8	45.7–67.8
Δ5,24-stigmastadienol	nd–7.1	0.6–11.5
Δ7-stigmastenol	nd–16.1	2.2–4.7
Δ7-avenasterol	nd–9.4	3.1–6.5
total sterols	324–798	503–647

[a] nd = less than 0.1.

Table 2. Sterol Composition of Sesame Seed Oils (%)

sterol	unroasted oils	roasted oils
cholesterol	nd[a]–0.4	nd–0.4
cholestanol	nd–0.2	nd–0.1
brassicasterol	nd	nd–0.1
24-methylenecholesterol	0.9–4.4	1.3–2.4
campesterol	14.0–21.1	15.2–17.7
campestanol	nd–0.4	nd–0.1
stigmasterol	4.9–8.6	5.5–7.0
Δ7-campesterol	nd–0.4	nd–0.8
Δ5,23-stigmastadienol	nd–1.4	nd–0.6
clerosterol	0.4–3.0	0.5–1.2
β-sitosterol	48.7–67.2	58.5–64.8
sitostanol	nd–0.7	0.1–0.5
Δ5-avenasterol	6.9–15.3	8.7–12.6
Δ5,24-stigmastadienol	nd–1.2	0.1–2.1
Δ7-stigmastenol	nd–2.6	0.4–0.7
Δ7-avenasterol	nd–1.8	0.6–1.2

[a] nd = less than 0.1.

Values for the sterols' percentage composition are given in **Table 2**.

They were generally in good agreement with the published results (*2*, *7*, *15*, *16*). Values for campesterol ranged from 14.0% to 21.1% (average = 17.7%) and for stigmasterol from 4.9% to 8.6% (average = 6.1%). These ranges are narrower than reported in other major surveys (*2*, *7*) whereas the range for the major sterol, β-sitosterol, agrees with those reports. Values for Δ5-avenasterol ranged from 6.9% to 15.3%, broadly in agreement with one previous survey (*7*) at 4.5−14.5%; however, the Codex range (*2*) is narrower and lower (6.2−7.8%).

A single sample, from seeds collected in Thailand, did not contain detectable Δ7-avenasterol or Δ7-stigmastenol. In the remainder the range for the latter was 0.1−2.6% (average = 0.7%). This was lower and narrower than the ranges given in elsewhere (*2*, *7*) (0.2−15.1%).

Table 3. Fatty Acid Composition of Sesame Seed Oils (%)

FA	unroasted oils	roasted oils
myristic acid C14:0	nd[a]–0.1	nd
pentadanoic acid C15:0	nd	nd
palmitic acid C16:0	8.4–11.0	9.5–10.9
palmitoleic acid C16:1	0.1–0.2	0.1–0.2
margaric acid C17:0	nd–0.1	nd–0.1
heptadecenoic acid C17:1	nd	nd
stearic acid C18:0	4.5–6.5	5.0–6.6
oleic acid C18:1	34.1–44.7	39.5–43.7
linoleic acid C18:2	36.9–47.8	38.8–43.9
linolenic acid C18:3	0.2–1.0	0.3
arachidic acid C20:0	0.5–0.7	0.6–0.7
eicosenoic acid C20:1	0.1–0.2	0.1–0.2
behenic acid C22:0	nd–0.2	nd–0.1
docosenoic acid C22:1	nd	nd
lignoceric acid C24:0	nd–0.2	0.1

[a] nd = less than 0.1.

Sterediene Composition. No steredienes (stigmastadiene, campestadiene, stigmastatriene, and campestatriene) were detected in oils from any of the unroasted seeds.

All of the eight oils from roasted seeds analyzed contained the major steradienes 3,5-stigmastadiene and 3,5-campestadiene. These are dehydration products of the major sterols β-sitosterol and campesterol. The levels of stigmastadiene ranged from 0.12 to 0.75 mg/kg and were mostly close to the EC limit of 0.15 mg/kg for this compound in virgin olive oil (*26*). Levels of campestadiene were 0.10−0.43 mg/kg. It is most likely that the parent sterols were dehydrated during the roasting operations, and the finding confirms that steredienes can be formed on heating oils in the absence of acid bleaching earths.

FA Composition. The FA composition of the sesame seed oils is given in **Table 3**.

The values obtained were in reasonable agreement with the ranges reported in earlier studies (*7*−*14*), but as might be expected for the increased sample numbers, the ranges for most acids were slightly expanded. Slightly higher proportions of C18:1 (46.8−53.9%) have been reported in two samples (*10*). A single sample, from Thai seeds, had an exceptionally high C18:3 at 1% where the range for the remaining Thai samples was 0.3−0.4%, with an average of 0.3%. The high value of the unusual sample was confirmed by repeated analysis. There was no clear difference in the FA profile of oils from roasted seeds, the results for which fell within ranges similar to those of literature reports (*8*, *24*, *25*, *27*).

Iodine Value. The mean calculated iodine values ranged from 104 to 115 in oil from unroasted seeds and 106 to 111 in oil from roasted seeds. Values per country for unroasted seed oils were Burkina Faso (106−107), China (111−115), Egypt (104−110), India (107−110), Japan (114), Mexico (110−112), Myanmar (112), and Thailand (112−114).

The results were comparable to those published elsewhere (*7*, *25*). Roasting of the seeds under the conditions used did not have a detectable effect on the iodine value.

FA in the Triglyceride 2-Position Composition. The triglyceride 2-position FA composition of sesame seed oils is given in **Table 4**.

The ranges for the major acids were slightly broader than the single reported literature values (*7*). For example, C18:1 ranged from 36.9% to 55.2% and C18:2 from 43.1% to 60.8% compared with the literature value of C18:1, 36.4−45.2%, and C18:2, 52.3−61.8%.

TCN. The TCN composition of the sesame seed oils is given in **Table 5**.

Table 4. Triacylglycerol 2-Position Fatty Acid Composition of Sesame Seed Oils (%)

FA	unroasted oils	roasted oils
myristic acid C14:0	nd[a]	nd
palmitic acid C16:0	0.8–1.8	1.0–2.9
palmitoleic acid C16:1	nd–0.2	0.1–0.2
margaric acid C17:0	nd–0.1	nd–0.1
stearic acid C18:0	0.3–0.8	0.4–1.2
oleic acid C18:1	36.9–55.2	41.3–47.5
linoleic acid C18:2	43.1–60.8	48.9–55.1
linolenic acid C18:3	nd–2.2	0.3–0.4
eicosenoic acid C20:1	nd–0.3	nd–0.1
others	nd–0.4	nd–0.4

[a] nd = less than 0.1.

Table 5. Triacylglycerol Carbon Number Composition of Sesame Seed Oils (%)

TCN	unroasted oils	roasted oils
TCN 48	nd[a]–0.3	nd
TCN 50	2.0–4.0	3.2–3.3
TCN 52	22.8–29.5	24.4–28.0
TCN 54	65.5–73.6	67.5–69.5
TCN 56	0.7–3.8	1.3–2.0
TCN 58	nd–2.1	nd–0.7
TCN 60	nd–0.6	nd

[a] nd = less than 0.1.

Table 6. Tocopherol and Tocotrienol Composition of Sesame Seed Oils (mg/kg)

tocol	unroasted oils	roasted oils
α-tocopherol	nd[a]	nd
β-tocopherol	nd	nd
γ-tocopherol	430–717	410–676
Δ-tocopherol	i[b]	i
α-tocotrienol	nd	nd
β-tocotrienol	nd	nd
γ-tocotrienol	nd	nd
Δ-tocotrienol	i	i
total	430–717	410–676

[a] nd = less than 10 mg/kg. [b] i = interference.

The results are similar to those reported earlier (*7*) apart from two samples, one from Burkina Faso and one from Thailand, that contained higher levels of C56 (3.0% and 3.8%, respectively) compared with the literature maximum of 1.9%.

The roasted samples showed a possible slight decrease in the levels of C52 when compared to their unroasted counterparts. A significantly greater loss of C54 and C52 containing unsaturated acids compared with those with saturated acids (C50 and C48) has been reported at roasting temperatures in excess of 180 °C (*28*).

Tocopherol Composition. The tocopherol composition of sesame seed oils is given in **Table 6**.

The only tocopherol detected in the sesame oils was γ-tocopherol. Chromatographic interferences for all samples prevented the detection of Δ-tocopherol and Δ-tocotrienol. The γ-tocopherol content ranged from 410 to 717 mg/kg with α- and β-tocopherols and tocotrienols not detected.

The range of tocopherol values was in broad agreement with the Codex range (*2*) and with literature values (7, *10*, *16*−*19*, *21*). Low levels of α-tocopherol and Δ-tocopherol have been reported in a small number of samples (*16*, *17*).

Roasting seeds under the conditions used in this work had no significant effect on the tocopherol composition of the oil.

This is in agreement with other studies (*21*, *25*). It has been reported that the γ-tocopherol content of oil from sesame seeds increases with roasting temperature up to about 200 °C (*25*) but decreases rapidly above 240 °C. Conversely, a decrease in total tocopherols was reported for roasting both white and brown seeds at 180 °C for 30 min (*10*).

Statistical Analysis. Statistical analysis based on ANOVA, pairwise *t*-tests, and principal component analysis carried out using the CSL-Metabolab visualization toolbox as described previously (*3*) was unable to separate the sesame samples by seed color. Factors relating to FA data were the most significant (>95%), but no data had over 99% significance.

Country of Origin. Comparison of the compositional data for each country shows overlapping of the ranges for most analytes; only a few factors showed significant (>99%) separation according to country of origin. These were cholestanol; FAs C16:1, C18:0, C18:1, and C18:2; 2-position FAs C16:1, C18:1, C18:2, and C20:1; and triacylglycerol TCN 52. For example, ANOVA applied to the cholestanol data was able to show that, for the limited number of data points for Burkina Faso, oils from that country could be distinguished from those of China, India, Mexico, and Thailand with high probability.

Using data for C18:1, Mexican oils were distinguished from those of Burkina Faso and Egypt, and using data for C18:2 Burkina Faso oils were distinguished from all but Egyptian oils.

In all statistical analyses there was a distinct pairing of the results for the four samples from Egypt. This was not associated with the source of seeds, two samples from Upper Egypt being placed in opposite pairs, but was possibly due to unidentified varietal or cultivation differences.

Factors Affecting the Composition of Sesame Seed Oil. The FA composition of sesame has been shown to vary with species (*11*), cultivar (*29*), and maturity (*30*).

Roasting seeds at temperatures between 180 and 260 °C for 30 min has been reported to cause a decrease in the total quantity of FAs and in the proportions of oleic and linoleic acids (*25*). Conversely, others have found similar conditions to have little (*8*) or no (*24*) effect on the acid composition, and these findings are confirmed in the study reported here. Roasting sesame seeds has been shown to have little effect on the tocopherol composition of the oil (*8*, *21*), although there is some evidence of a decrease at temperatures above 260 °C (*25*).

It was not possible to identify the species, maturity, or growing conditions etc. of the current samples. They did, however, reflect the types of seed traded for oil manufacture and industry production conditions. This paper adds to and updates the available compositional tables for sesame seed oils. The analyses have been performed using modern equipment and validated methods incorporating stringent quality control measures based on the use of fully characterized reference materials. The geographical origin of the seeds has been confirmed by collection from the harvest site and the authenticity of the seeds and oil by the use of an audit trail.

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Received for review February 7, 2006. Revised manuscript received May 3, 2006. Accepted May 25, 2006. We thank the U.K. Food Standards Agency for funding this project.

JF0603578