High-Linoleic Oils from Five Species of Japanese Plants

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ABSTRACT

Oils extracted from the achenes of *Boehmeria longispica* Steud., *B. spicata* Thunb., and *B. nivea* Gaud. var. *concolor* Makino (Urticaceae) and those from the seeds of *Tricyrtis affinis* Makino and *Hosta longipes* Matsum. (Liliaceae) were surveyed for their characteristics and fatty acid composition. All the oils contained linoleic acid in high levels (80.2-83.5%) as determined by gas liquid chromatography of the derived methyl esters.

INTRODUCTION

Previously, we found exceedingly high levels of linoleic acid in the seed oils of 3 species of deciduous tree, *Aphananthe aspera* (1) and *Celtis sinensis* var. *japonica* (2) of the Ulmaceae family and *Betula platyphylla* var. *japonica* (3) of the Betulaceae family.

This paper describes some data on the high-linoleic oils from 5 additional species of perennial plants, i.e., one shrub (*B. spicata*) and 4 herbs, growing wild in Japan (Table I). Among these species, only *B. nivea* has been studied for the seed oil (4,5).

EXPERIMENTAL PROCEDURES

Materials

All of the seed materials analyzed were collected from plants grown during the 1979 season at 3 different geographical locations in Saitama-ken, Japan.

Extraction of Oil

Achenes of the 3 Urticaceae species and seeds of the 2 Liliaceae species were air-dried and cleaned to remove fragments and foreign material. The cleaned seeds (or achenes) were separately ground in a mortar and subjected to extraction with ethyl ether in a Soxhlet apparatus. After 6 hr, the solvent was removed from the extract by heating and evaporating under a stream of nitrogen. The resulting oil was treated with hexane in the manner previously reported (1), and the hexane-soluble fraction was used for analysis.

Preparation and Gas Liquid Chromatography (GLC) of Methyl Esters

Each sample oil was saponified in the usual manner. After removal of the unsaponifiable matter, the mixed fatty acids were converted to methyl esters by an H_2SO_4-methanol procedure.

The methyl esters were analyzed on a Hitachi 163 gas chromatograph equipped with a hydrogen flame ionization detector and a Takeda Riken TR-2217 automatic integrator. A 4 m × 3 mm stainless steel column packed with 5% diethylene glycol succinate polyester on 60/80 mesh Chromosorb G AW was used; flow rate of nitrogen was 20 mL/min; temperature of column, 200 C; temperature of injection port and detector, 300 C. Identification of the components was based on the comparison of their retention times with those of the authentic materials.

TABLE I

Characteristics of Oils and Their Mixed Fatty Acids

Species	*Boehmeria longispica* Steud.	*B. spicata* Thunb.	*B. nivea* Gaud. var. *concolor* Makino	*Tricyrtis affinis* Makino	*Hosta longipes* Matsum.
Family	Urticaceae			Liliaceae	
Oil					
Yield (%)	12.1[a]	10.6[a]	17.5[a]	22.0[b]	28.5[b]
Color	Dark green	Dark green	Dark greenish brown	Green	Yellowish brown
Sp grav (20 C/4 C)	0.9236	0.9201	0.9255	–	0.9261
n_D^{20}	1.4785	1.4756	1.4768	1.4775	1.4767
Saponification value	193.0	187.1	189.2	193.3	194.1
Iodine value (Wijs)	149.9	152.2	147.0	151.2	152.4
Unsaponifiable matter (%)	3.15	3.87	3.61	1.84	4.62
Mixed fatty acids					
Neutralization value	204.0	202.3	203.1	202.5	201.7
Iodine value	158.6	159.1	155.0	158.6	158.8

[a]Achenes.
[b]Seeds.

TABLE II

Fatty Acid Composition of Oils Determined by Gas Liquid Chromatography

Species	Component acids (% by wt)											
	12:0	14:0	15:0	16:0	16:1	17:0	18:0	18:1	18:2	18:3	20:0	22:0
B. longispica	trace	trace	trace	5.2	–	trace	2.3	7.9	82.7	0.9	0.6	0.4
B. spicata	–	0.2	–	5.2	–	–	1.5	7.8	83.5	1.5	–	0.3
B. nivea var. *concolor*	–	trace	–	6.2	trace	0.1	2.7	9.4	80.4	1.2	–	–
T. affinis	trace	0.2	–	5.3	–	trace	1.4	11.4	80.2	1.4	–	0.1
H. longipes	–	trace	–	5.8	0.1	–	1.0	9.5	82.5	0.8	trace	0.3

RESULTS AND DISCUSSION

Yield and characteristics of the sample oils are shown in Table I. Each achene sample of the family Urticaceae had a lower oil content (11-18%), as compared to the 2 seed samples of the family Liliaceae (22 and 29%). UV and IR examinations of the oils revealed no evidence for conjugated unsaturation or unusual functional groups.

Fatty acid profiles of the oils, based on GLC analyses of their methyl esters, are given in Table II. All the oils were characterized by the presence of high levels of linoleic acid (80.2-83.5%). Another remarkable feature of these oils was the predominance of unsaturated components (91.0-93.0%) consisting exclusively of C_{18} acids, except for the *H. longipes* oil with 0.1% of C_{16}-monoenoic acid.

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REFERENCES

1. Tanaka, T., S. Ihara and Y. Koyama, JAOCS 54:269 (1977).
2. Ihara, S., and T. Tanaka, Ibid. 55:471 (1978).
3. Ihara, S., and T. Tanaka, Ibid. 57:421 (1980).
4. Popov, A., and St. Ivanov, CR. Acad. Bulg. Sci. 8:17 (1955); Chem. Abstr. 50:11035g (1956).
5. Popov, A., and St. Ivanov, Ibid. 10:221 (1957); Chem. Abstr. 52:3367e (1958).

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High Performance Reversed Phase Chromatography of Natural Triglyceride Mixtures: Critical Pair Separation

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ABSTRACT

The separation of the critical triglyceride pairs C48:0, C50:1, C54:3*ccc* and C54:3*ttt* as well as C54:2, C52:1 and C50:1 has been accomplished without the aid of any interacting ion such as silver. A theoretical carbon number (TCN) for the unsaturated triglycerides can be calculated from the carbon number (CN) and capacity factor (k′) relationship of the saturated triglycerides, and used to predict the separation of critical pairs. A mathematical equation was derived for the identification of not only the triglycerides by their carbon number and number of double bonds but also the possible acyl groups present in these triglycerides. The pattern of triglyceride elution sequence within each triglyceride category with the same equivalent carbon number (ECN) starts with the triglyceride with the highest number of double bonds and terminates with those with the lowest number of double bonds, with the lower ECN triglycerides eluting ahead of those with higher ECN. A possible mechanism for the separation of these triglycerides on highly efficient columns packed with 5 μ silica bonded with the octadecyl stationary phase is postulated.

INTRODUCTION

The increasingly efficient separation of individual triglycerides present in fats and oils which can now be carried out is gradually increasing the understanding of their structural composition. The availability of such data would facilitate the understanding of triglyceride biosynthesis and deposition in plant and animal cells.

Increasingly efficient separation of triglycerides by high performance reversed-phase chromatography (HPRC) has been achieved on μ-Bondapac C18 (1-5), Spherosorb 5-ODS (6), Vydac (6), Zorbax (7,8), Supelcosil LC-8, Supelcosil LC-18 and Partisil ODS-2 (8) column packings using various mobile phases. Triglyceride separations have been attempted with silica columns, but satisfactory separations (9) were not achieved. Mobile phases containing silver nitrate (10) have aided the separation of saturated and unsaturated triglycerides where highly efficient packings were not available. However, argentation liquid chromatography showed a lack of reproducibility of k′ value. Furthermore, possible silver mirror formation on the detector cell windows remains a strong deterrent toward general use of this type of approach. The effects of changes in mobile phase polarity and composition on the separation and resolution of triglycerides as well as the characteristics of the packing (e.g., particle size, bonded chain length, percent coverage) have also been reported (8). In the present study, the separation of critical pairs and triglyceride isomers has been accomplished with the aid of highly efficient octadecyl bonded column packings operated in the reversed phase mode. A possible mechanism for the separation of triglyceride critical pairs by reversed-phase chromatography is postulated.

METHODS

The instrument used consisted of a Tracor 995 isochromatographic pump (Tracor Instruments, Austin, TX), a Rheodyne loop (20 μL) injector (Model 7120), and a Waters R401 Differential Refractometer Detector (Waters Assoc., Milford, CT). Separations were recorded with the aid of a Hewlett Packard 3385A electronic integrator (Hewlett Packard, Palo Alto, CA) at a chart speed of 0.1-0.2 cm/min. Retention times were automatically printed by the recording integrator. Two 250 mm × 4.6 mm commercially packed columns from different manufacturers were used in this study: a Supelcosil LC-18 column with a 5-μ octadecyl bonded spherical silica (Supelco, Supelco Park, Bellefonte, PA) and a Zorbax ODS column with octadecyl bonded silica of 6-7-μ diameter (DuPont Co., Wilmington, DE). Mixtures of analytical grade acetone and glass-distilled acetonitrile (63.6:36.4 [v/v]) were used as the mobile phase. Triglycerides used as standards were obtained from Nu-Chek-Prep (Elysian, MN) and Supelco (Supelco, Bellefonte, PA). Triglycerides were solubilized in tetrahydrofuran (THF) or acetone at 100 mg/mL for each triglyceride. Vegetable oils were solubilized in THF at 200-250 mg/mL.

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