Chemistry of Natural Compounds, Vol. 44, No. 6, 2008

COMPOSITION OF SUPERCRITICAL FLUID EXTRACTS OF SOME *Xanthium* SPECIES FROM CHINA

Ting Han, Hong Zhang, Hui-liang Li, Qiao-yan Zhang, Han-chen Zheng, and Lu-ping Qin*

UDC 547.913

The genus *Xanthium* (Compositae) is represented by 25 species in the world and 3 species and 1 variety in China [1]. These are *X. sibiricum* Patr. (*X. strumarium* L.), *X. mongolicum* Kitag., *X. inaequilaterum* DC., and *X. sibiricum* var. *subinerme* (Winkl.) Widder. *Xanthium* species have been used as traditional herbal medicines for the treatment of nasal sinusitis, headache caused by wind-cold, urticaria, and arthritis for a long time in oriental countries [2]. The chemical composition of ent-kaurane diterpenoids, sesquiterpene lactones, caffeoylquinic acids, and thiazinediones from this genus (leaves or fruits) has been reported [3–8]. Chemical examination of the essential oil from the leaves of *Xanthium strumarium* showed that the main components of the essential oil were d-limonene and d-carveol, with a relative content of 35% and 25% (w/w), respectively [9]. The composition of the fruit essential oil of *Xanthium sibiricum* Patr. was reported in 1994 [10], and showed that saturated paraffin hydrocarbons such as eicosane and nonadecane were the major components. The fruit oil of *Xanthium* obtained by crushing were reported to have anti-thrombus effects and to decrease blood-fat in civil use [11], which were referred to the lipophylic extracts of *Xanthium fruits*.

The purpose of this research was to conduct a comparative analysis on the fatty oil composition from samples belonging to three *Xanthium* species growing in China: *X. sibiricum*, *X. sibiricum* var. *subinerme*, and *X. mongolicum*. The collected location and date of the three *Xanthium* species used for supercritical fluid extraction (SFE) and GC-MS analysis are listed in Table 1. This is the first report on the lipophylic extracts prepared by SFE of the three *Xanthium* species. The results of the fatty acid analysis by GC-MS and the oil yield of the taxa are presented in Table 1.

The extraction of essential oil components using solvents at high pressure, or supercritical fluids, has received much attention in the past several years, especially in the food, pharmaceutical, and cosmetic industries, because it presents an alternative to conventional processes such as organic solvent extraction and steam distillation [12, 13]. By comparing the composition of the product with hydrodistilled oil [14], higher levels of the volatile components such as d-limonene and d-carveol were found by using the hydrodistillation method [9]. However, these compounds were not found in the SFE products. In contrast, the fatty acids linoleic acid, palmitic acid, stearic acid, and some sterols were found in the SFE products, whereas negligible amounts were present in the distilled oils.

In this study, a total of 37 compounds of three *Xanthium* species in China was determined, of which aromadendrene, α-phellandrene, globulol, norphytane, tomentosin, etc. were identified from *Xanthium* genus for the first time. The fatty oil composition of the studied *Xanthium* genus was not uniform but overlap. A considerable content of linoleic acid and palmitic acid was noted in all samples, but small quantitative differences were also found. Linoleic acid was the predominant constituent of the studied fruit oil and had the relative content of 80.24% (*X. sibiricum*), 70.75% (*X. sibiricum* var. *subinerme*) and 77.71% (*X. mongolicum*), respectively. The amount of linoleic acid is important with respect to the quality of the oils consumed as a food resource [15]. Therefore, the fruits of the three studied *Xanthium* species can all be regarded as a new resource for linoleic acid production.

Stearic acid, linolenyl alcohol, *n*-tetracosane, *n*-pentacosane, and *n*-nonacosane, were present in both *X. sibiricum* var. *subinerme* and *X. mongolicum* fruit oil. The *X. sibiricum* var. *subinerme* oil composition was characterized by a large variation of the long-chain diolefins, including *n*-heptadecane, *n*-docosane, and all the linear-chain diolefin froms *n*-tetracosane to *n*-nonacosane. Only nine compounds of *X. mongolicum* fruit oil were determined, of which diisooctyl phthalate was the special component.

School of Pharmacy, Second Military Medical University, 325 Guohe Rd., Shanghai 200433, China, fax: +86 21 25070394, e-mail: qinsmmu@126.com. Published in Khimiya Prirodnykh Soedinenii, No. 6, pp. 655-656, November-December, 2008. Original article submitted July 21, 2007.

0009-3130/08/4406-0814

TABLE 1. Comparative Analysis of Fatty Oil Components in Three Different Species of *Xanthium* by GC-MS

Compound	Formula	Relative content, %		
		S-1	S-2	S-3
Hept-*cis*-2-enal	$C_7H_{12}O$	0.68	-	-
n-Caproic acid	$C_6H_{12}O_2$	0.14	-	-
α-Phellandrene	$C_{10}H_{16}$	0.07	-	-
n-Nonaldehyde	$C_9H_{18}O$	0.02	-	-
n-Caprylic acid	$C_8H_{16}O_2$	0.04	-	-
trans-2-Decenal	$C_{10}H_{18}O$	0.19	-	-
(*E,E*)-2,4-Decadienal	$C_{10}H_{16}O$	1.72	-	-
Aromadendrene	$C_{15}H_{24}$	0.40	-	-
Globulol	$C_{15}H_{26}O$	0.30	-	-
3,7,11,15-Tetramethyl-2-hexadecen-1-ol	$C_{20}H_{40}O$	0.14	-	-
Palmitic acid	$\mathbf{C_{16}H_{32}O_2}$	**1.02**	**10.25**	**7.84**
Palmitic acid, ethyl ester	$C_{18}H_{26}O_2$	1.63	-	-
Linoleic acid	$\mathbf{C_{18}H_{32}O_2}$	**80.24**	**70.75**	**77.71**
9,12-Octadecadienoic acid, ethyl ester	$C_{20}H_{36}O_2$	5.97	-	-
Stearic acid, ethyl ester	$C_{20}H_{40}O_2$	1.14	-	-
Tomentosin	$C_{15}H_{20}O_3$	3.30	-	-
Santalol	$C_{15}H_{24}O$	1.49	-	-
1,3-Dihydroxypropan-2-yl (9*E*,12*E*,15*E*)-octadeca-9,12,15-trienoate	$C_{21}H_{36}O_4$	1.51	-	-
n-Heptadecane	$C_{17}H_{36}$	-	0.07	-
Norphytane	$C_{19}H_{40}$	-	0.08	-
2,4-Diphenyl-4-methyl-2(*E*)-pentene	$C_{18}H_{20}$	-	0.05	-
Stearic acid	$C_{18}H_{36}O_2$	-	2.14	3.17
n-Docosane	$C_{22}H_{46}$	-	0.86	-
Linolenyl alcohol	$C_{18}H_{32}O$	-	1.06	2.55
8-Hydroxy-2,2,8-trimethyldeca-5,9-dien-3-one	$C_{13}H_{22}O_2$	-	1.57	-
Methyl hexadecatrienoate	$C_{17}H_{28}O_2$	-	1.24	-
n-Tetracosane	$C_{24}H_{50}$	-	0.95	0.38
Isopropyl linoate	$C_{21}H_{38}O_2$	-	0.84	-
n-Pentacosane	$C_{25}H_{52}$	-	1.52	0.52
2,4-di-(α-Methylstyryl)phenol	$C_{24}H_{26}O$	-	2.17	-
n-Hexacosane	$C_{26}H_{54}$	-	1.32	-
n-Heptacosane	$C_{29}H_{60}$	-	1.54	-
n-Octacosane	$C_{28}H_{58}$	-	1.34	-
n-Nonacosane	$C_{29}H_{60}$	-	1.83	0.72
(*Z,Z*)-5,7-Dodecadiene	$C_{12}H_{22}$	-	0.44	-
Diisooctyl phthalate	$C_{24}H_{38}O_4$	-	-	5.30
di(2-Ethylhexyl)isophthalate	$C_{24}H_{38}O_4$	-	-	0.34
Oil yield, wt %		11.5	15.0	7.0

S-1: *X. sibiricum* (Sunqiao, Shanghai) - 2003.11.04; S-2: *X. sibiricum* var. *subinerme* (Renqiu, Hebei Province) - 2003.11.13; S-3: *X. mongolicum* (Yantai, Shandong Province) - 2003.11.07.

Among the three *Xanthium* samples, the highest oil yield was obtained from *X. sibiricum* var. *subinerme* fruits (15.0%); the *X. sibiricum* fruits (11.5%) and the *X. mongolicum* gave the lowest oil yield (7.0%). Thus, *X. sibiricum* var. *subinerme* was considered to be a high-quality germplasm if the oil yield was used as the major standard of quality evaluation.

The results obtained from this study showed that *Xanthium* fruit oils are of the linolenic type, that is, the linolenic type fatty acids are the predominant components. It was reported that linolenic acid showed the effects of decreasing the content of cholesterin and reversing artherosclerosis [16], and the fruit oil, rich in unsaturated fatty acids and with high production, was often used as an edible oil [11].The evaluation of fatty acids in a wider range of species in Compositae is a powerful tool that might contribute to the characterization of new renewable resources.

REFERENCES

1. Y. L. Chen, in: *Flora Reipublicae Popularis Sinicae,* Vol. **75**, Science Press, Beijing, 1979, p. 324.
2. *Pharmacopoeia of P. R. China* (English Edition), Book. Chemical Industry Press, Beijing, 2000, p. 111–112.
3. Editorial Board of China Herbal. State Administration of Traditional Chinese Medicine. *China Herbal*, Vol. **7**, Shanghai Science and Technology Press, Shanghai, 1999, p. 1014.
4. J. Cumanda, G. Marinoni, M. De Bernardi, G. Vidari, and P. Vita Finzi, *J. Nat. Prod.,* **54**, 460 (1991).
5. Y. T. Ma, M. C. Huang, F. L. Hsu, and H. F. Chang, *Phytochemistry*, **48**, 1083 (1998).
6. T. Han, H. L. Li, Q. Y. Zhang, H. C. Zheng, and L. P. Qin, *Chem. Nat. Compd.*, **42**, 567 (2006).
7. L. P. Qin, T. Han, H. L. Li, Q. Y. Zhang, and H. C. Zheng, *Fitoterapia*, **77** (3), 245 (2006).
8. T. Han, H. L. Li, Y. Hu, Q. Y. Zhang, H. C. Zheng, K. Rahman, and L. P. Qin, *J. Chin. Integr. Med.*, **4** (2), 194 (2006).
9. H. A. Taher, G. O. Ubiergo, and E. C. J. Talenti, *J. Nat. Prod.*, **48**, 857 (1985).
10. Y. H. Guo, J. S. Li, J. G. Pan, and Z. L. Xu, *Chin. J. Chin. Mat. Med.*, **19** (4), 235 (1994).
11. S. H. Hu, *Special Economic Animal and Plant.*, **4** (6), 29 (2001).
12. M. H. Eikani, I. Goodarznia, and M. Mirza, *Flavour Fragrance J.*, **4**, 29 (1999).
13. L. M. Assis and F. M. Lancas, *J. Microcolumn Separat.,* **11**, 501 (1999).
14. Z. L. Li and S. Y. Liu, *Chin. J. Chromatogr.,* **15** (4), 344 (1997).
15. E. Bagci, *Chem. Nat. Compd.*, **42**, 645 (2006).
16. W. Jiang, *J. Chin. Cereals Oils Assoc.*, **3**, 21 (2006).