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Constituents of Essential Oil from *Rumex crispus**

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Composition of the essential oil of *R. crispus* L. was investigated. This oil was obtained by steam distillation of leaf materials from the plants growing in Higashiosaka-shi (Japan). By LC, GLC, GC-MS, IR, 60 components were identified. The principal components are 1,2-diacetin and *cis*-3-hexen-1-ol.

Introduction

It is very difficult to distinguish between *Rumex crispus* L. and *R. japonicus* Houtt. due to hybridization in both cases; however, according to T. Osada¹⁾, it is possible to discriminate *R. crispus* L. from *R. japonicus* Houtt. by the following: the shape of flower including the seeds of *R. crispus* L. is round without teeth-marks and that of *R. japonicus* Houtt. is triangular with teeth-marks on half of the flower. The composition of the essential oil of *R. crispus* L., which grows in the Higashiosaka area and is distinguished by the form of the inner perianth as indicated above, was investigated.

In our previous work²⁾, the essential oil of the aerial and root portions of *R. japonicus* Houtt. was investigated. Discussion was based on the position of Chemotaxonomy by the same distinguished *R. japonicus* L.

Results and Discussion

The essential oil was separated into neutral and acidic fractions by aqueous sodium hydroxide extraction. The neutral and acidic fractions were analyzed by spectrometric techniques, the 60 components listed in **Table-1** being identified. Each component was isolated by column chromatography and preparative GLC. The relative concentration was calculated on the basis of GLC peak areas. Thus, the oil from *R. crispus* L. was

clarified to contain 1,2-diacetin and *cis*-3-hexen-1-ol as its main components and β -pinene, nonanal, furfural, 1-dodecanol, and phenol as minor components. There were 24 hydrocarbons, 4 aldehydes 1 ketone, 12 alcohols, 6 phenolic compounds, 3 esters, and 10 fatty acids. The relative concentration and chemical components of the essential oils from *R. japonicus* Houtt. and *R. crispus* L. are shown in **Table-2**. The most noteworthy feature of this oil was the presence of furfural and *cis*-3-hexen-1-ol, 1-dodecanol and a rather large quantity of 1,2-diacetin. The hydroxy compounds consisted of aliphatic compounds, and they represented 48.7% of oil and the aromatic hydroxy compounds, 10.5%. The amounts of hydroxy compounds, ester and ketone compounds were greater than in *R. japonicus* Houtt, while the amounts of alkanes, aldehydes and acidic compounds were less. The acidic part examined after esterification was considerably less complex than carboxylic acid, and benzoic acid as the main acid, were found in *R. crispus* L. Thus, both essential oils were made up of the same components and there was only a small difference in their amounts.

Experimental

IR spectra were obtained as liquid films. GC-MS was carried out with a 3m $\times$ 3mm glass column with 5% PEG-20 M; temp. programmed 100~200°C at 5°C/min; He, 30 ml/min; the mass spectrometer was operated at 70 eV with the ion source at 210°C. Analytical GLC was performed on a Yanaco-G 8 instrument equipped

* Studies on Chemical Constituents of Naturalized Plants in Japan. Part IX, This paper was presented at the annual Meeting of the ACS/CSJ Chemical Congress, Hawaii U.S.A., April, 1979, for part VIII, see ref. 2).

Table-1 Chemical constituents of the essential oil of *R. crispus*.

Compounds	(%)	M.I.	Compounds	(%)	M.I.
(Hydrocarbons)			(Alcohols, Phenols and Esters)		
Decane	1.0	MS, t _R	Hexyl acetate	1.9	MS, t _R
Undecane	1.2	MS, t _R	1-Hexanol	0.7	MS, t _R
Dodecane	1.3	MS, t _R	<i>cis</i> -3-Hexen-1-ol	11.1	MS, t _R , IR
Tridecane	1.5	MS, t _R	1-Heptanol	2.2	MS, t _R
Tetradecane	1.2	MS, t _R	Linalool	0.9	MS, t _R
Pentadecane	1.8	MS, t _R	1-Octanol	1.9	MS, t _R
Hexadecane	0.9	MS, t _R	Furfuryl alcohol	1.2	MS, t _R
Heptadecane	0.8	MS, t _R	α -Terpineol	0.1	MS, t _R
Octadecane	0.9	MS, t _R	1-Decanol	0.4	MS, t _R
Nonadecane	1.0	MS, t _R	Benzyl alcohol	Tr.	MS, t _R
Icosane	1.2	MS, t _R	Dodecyl acetate	1.1	MS, t _R
Henicosane	1.1	MS, t _R	β -Phenylethyl alcohol	1.7	MS, t _R
Docosane	1.0	MS, t _R	1-Dodecanol	9.2	MS, t _R
Tricosane	0.8	MS, t _R	Nerolidol	Tr.	MS, t _R
Tetracosane	0.7	MS, t _R	Phenol	3.7	MS, t _R
Pentacosane	0.7	MS, t _R	<i>o</i> -Cresol	2.3	MS, t _R
Hexacosane	0.5	MS, t _R	<i>p</i> -Cresol	1.3	MS, t _R
Heptacosane	0.8	MS, t _R	<i>m</i> -Cresol	0.8	MS, t _R
Octacosane	0.9	MS, t _R	Thymol	0.2	MS, t _R
Nonacosane	0.8	MS, t _R	Carvacrol	2.2	MS, t _R
α -Pinene	Tr.	MS, t _R	1,2-Diacetin	22.6	MS, t _R , IR
Camphene	Tr.	MS, t _R	(Acidic compounds)		
β -Pinene	3.1	MS, t _R	Acetic acid	Tr.	MS, t _R
β -Myrcene	0.3	MS, t _R	Propionic acid	Tr.	MS, t _R
(Ketone and Aldehydes)			Hexanoic acid	Tr.	MS, t _R
Camphor	1.1	MS, t _R	Octanoic acid	Tr.	MS, t _R
Hexanal	2.1	MS, t _R	Decanoic acid	Tr.	MS, t _R
Nonanal	3.0	MS, t _R	Dodecanoic acid	Tr.	MS, t _R
Furfural	3.8	MS, t _R , IR	Tetradecanoic acid	Tr.	MS, t _R
Benzaldehyde	0.9	MS, t _R	Hexadecanoic acid	Tr.	MS, t _R
			2-Furan carboxylic acid	Tr.	MS, t _R
			Benzoic acid	0.1	MS, t _R

M.I.; Method of identification, Tr; trace amount.

with a flame ionization detector. The temperature of the injection block was maintained at 220°C and that of the detector at 250°C. Stainless steel columns (2.25 m × 3 mm) packed with 20% Carbowax-20 M on 80~100 mesh Chromosorb W were used. The temperature was programmed from 70 to 200°C at 2°C/min. The helium flow was maintained at 30 ml/min. Preparative GLC was performed with a Yanaco-G 800 instrument with a thermal conductivity detector.

Plant material and essential oil. The

plant materials were collected from some waste lands in Higashiosaka-shi in May, 1978. After steam distillation of the aerial parts, the essential oil (d_4^{25} 0.9910, n_D^{25} 1.4882, $[\alpha]_D^{25}$ -15.1°, Yield 0.001%) was obtained by extraction of the distillation with ether and evaporation under N₂.

Treatment of the essential oil. The essential oil (1.0 g) was processed for acidic and neutral fractions by the usual method. The neutral fraction was dissolved in hexane (1 ml) and was chromatographed on a column (20 mm × 2 m) of silica gel. Elution was performed

Table-2 Chemical components of the essential oils from *R. crispus* and *R. japonicus*.

Compound	[A] %	[B] %
Hydrocarbons	23.5	42.3
(Alkanes)	(20.1)	(40.1)
(Terpenes)	(3.4)	(1.6)
Ketones	1.1	0.9
Aldehydes	9.8	11.8
Alcohols and phenols	62.5	41.0
Esters	3.0	2.1
Acides	Tr.	0.2

[A]; *R. crispus*, [B]; *R. japonicus*

successively using hexane (100 ml), benzene (100 ml), ether (150 ml), and ethyl acetate (150 ml). Fractions (50 ml) were collected and monitored with analytical GLC. Each component was identified by MS², IR and retention time (t_R) on GLC compared with those of authentic samples and samples isolated from the plants reported in our previous papers.²⁾ The acidic fraction from the essential oil was treated with ethereal CH₂N₂ and then examined with GC-MS.

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References

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ナガバギシギシの精油成分

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ナガバギシギシの精油成分について研究した。東大阪市で採集した葉部を水蒸気蒸留することによって精油を得た。カラムクロマト, ガスクロマト, ガスクロマトグラフィー-質量スペクトル及び赤外スペクトルによって 60 成分を同定した。その主要成分は 1,2-ジアセチンと *cis*-3-ヘキセン-1-オールであった。