Volatile Composition of *Jasonia glutinosa* D. C.

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Using GC-MS the volatile composition of *Jasonia glutinosa* D. C., was studied by comparing two different methods for the isolation of a volatile fraction: distillation from the fresh plant in order to obtain the essential oil, and direct thermal desorption (DTD). Compared with essential oil extraction the main advantages of the DTD technique are the smaller sample amount required, and the increased range of volatile compounds which can be subsequently analysed by GC-MS.

Key words: Jasonia glutinosa, Direct Thermal Desorption, Volatile Compounds

Introduction

Jasonia glutinosa D. C. [*Chiliadenus saxatilis* Cass. (Brullo, 1979), *Chiliadenus glutinosus* (L.) Fourr. (Greuter *et al.*, 1989); *Inuleae*, Asteraceae] is a perennial species that grows in the east of Spain on alkaline soils. This plant, usually known in Spain as "rock tea", has a characteristic odour. It has been used in Spanish traditional medicine for its antispasmodic activity (Font Quer, 1992), but it is also widely consumed as an herbal tea. The volatile constituents of *Jasonia glutinosa* D. C. have been previously reported (Guillén and Ibargoitia, 1996). By GC-MS we studied the volatile composition of *Jasonia glutinosa* D. C., comparing two different methods for the isolation of a volatile fraction: distillation from the fresh plant to obtain the essential oil, and direct thermal desorption (DTD).

Experimental

Plant material

The aerial parts of *Jasonia glutinosa* D. C. were collected in San Andrés del Congosto (Guadalajara, Spain) in July 1997 and identified by Dr. Carmen Bartolomé Esteban. A voucher specimen (LV 91) is kept in the Department of Pharmacognosy, Faculty of Pharmacy, Alcalá de Henares (Spain).

Extraction of the essential oil and GC analysis

The fresh leaves (600 g) were submitted for 3 h to steam distillation in 2 l H_2O in a modified Clevenger apparatus with a water-cooled oil receiver to reduce artifact formation. The oil was stored under refrigeration until examined by GC-MS.

GC-MS analysis of the oil was carried out using Hewlett Packard (HP) 5890 Series II mass selective detector and a HP-1 crosslinked methyl silicone 30 m × 0.25 mm capillary column (0.25 mm film thickness). Helium low rate was 1 ml/min and the injection temperature was 250 °C. The column was held initially at 80 °C for 10 min, then the temperature was increased to 270 °C at a rate of 4 °C/min. Spectra were recorded in the EI mode, using 70 eV for the ionization energy.

Determination was carried out using mass spectral data from the NIST and Wiley libraries, and from peak time retention and area data.

Direct thermal desorption (DTD) coupled to GC-MS

An automatic thermal desorption unit (ATD-400 from Perkin-Elmer, Norwalk, CT) able to pro-

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cess automatically up to 50 samples was used. 20 mg of dry leaves were deposited in a Teflon liner which was introduced into a stainless-steel tube (0.25″ × 3.5″). Sample volatiles were thermally desorbed by heating the tube at 180 °C with a flow of carrier gas (He) for 15 min (primary desorption), and then adsorbed in a Tenax GC cold trap (– 30 °C) which was later heated at 30 °Cs^{-1} to 250 °C (secondary desorption), allowing their rapid transfer to the GC capillary column through a heated (250 °C) fused silica line. Details are given elsewhere (Esteban *et al.*, 1992).

The DTD-400 transfer line was connected to an OV-1 capillary column (50 mm × 0.25 mm) installed in a Fisons 8000 gas chromatograph (Fisons Instruments), equipped with a quadrupole mass detector MD 800 (Fisons, VG Masslab) operating in EI mode at 70 eV. Oven temperature was held at 70 °C for 5 min, then programmed at 5 °C min^{-1} up to 220 °C and held at this temperature for 20 min. Helium was used as desorb and carrier gas; column flow rate was 1 ml/min.

Results and Discussion

Determination results for both essential oil and thermal desorption fractions are shown in Table I. Thirty-four components were identified in the essential oil, representing 89.0% of the total volatile composition. The main constituents were camphor (31.5%), borneol (15.7%), caryophyllene oxide (11.4%), farnesol (8.6%) and bornyl formate (2.9%).

Direct thermal desorption coupled to GC-MS afforded the identification of twenty-two compounds, which only represented 15.5% of the total volatile composition. The most important components were camphor (7.4%), borneol (3.6%), caryophyllene oxide (2.5%), cadinol (1.8%) and spatulenol (1.3%). Twenty-one of these compounds have been identified for the first time in the essential oil of *Jasonia glutinosa* D. C.

The most striking difference between essential oil and DTD results is the presence of a very high amount of low-volatility unidentified compounds found by the latter technique which represent about 80% of the total volatile composition. When the effect on the percent results in Table I of these compounds is discounted, both compositions are similar; camphor, borneol and caryophyllene oxide being the most important constituents in both fractions. Table II lists the mass fraction values and the molecular weight (estimated from mass spectral data) for several unidentified components. The essential oil contains some unidentified sesquiterpene alcohols (molecular weight 220) in low concentrations, while the DTD frac-

Table I. Volatile composition of *Jasonia glutinosa* D. C.

Component	Essential oil (%)	DTD (%)
α-Pinene	1.5	0.3
Camphene	0.8	0.2
β-Pinene*	0.6	0.2
δ-3-Carene*	t	–
α-Phellandrene*	t	t
α-Terpinene*	t	t
Limonene*	0.5	–
Eucalyptol*	1.1	–
2-Hexenal	0.4	–
γ-Terpinene*	0.3	–
p-Cymene*	0.4	t
Terpinolene*	t	–
cis-3-Hexenol*	t	–
Cineole	–	0.2
Neroloxide	0.4	–
γ-Terpinene*	–	t
Sabinene hydrate	–	t
Linalool oxide	–	t
α-Campholene aldehyde	t	–
Linalool	–	0.2
Camphor	31.5	7.4
Linalool	1.7	–
2-Cyclohexen-1-ol	t	–
Bornyl formate	2.9	–
β-Caryophyllene	0.8	–
Terpinen-4-ol	0.2	0.2
Terpinen-1-ol*	2	–
Bornyl formate	–	0.3
trans-Pinocarveol*	0.3	–
β-Selinene*	0.9	0.3
Borneol	15.7	3.6
p-Menthadienol*	0.7	0.2
Geraniol*	0.9	–
β-Damascenone*	t	–
p-Cymen-8-ol*	t	–
Selinene	–	0.3
Nerolidol	–	0.1
Caryophyllene oxide*	11.4	2.5
Methyl eugenol*	0.8	–
Farnesol*	8.6	–
Cadinol	–	1.8
Spatulenol*	–	1.3
δ-Cadinene*	0.7	–

* Compounds identified for the first time in the volatile fraction of *Jasonia glutinosa* D. C.
t: < 0.1%.

Table II. Oxygenated sesquiterpene derivatives present in *Jasonia glutinosa* D. C.

Fraction	*M*	(%)
EO	220	1.1
EO	220	0.8
DTD	232	9.3
DTD	232	4
DTD	232	10.3
DTD	236	4.2
DTD	236	2
DTD	236	1.7
DTD	238	18.6

EO: Essential oil.
DTD: Direct thermal desorption.
%: Percent of total volatiles.

tion presents a high number of low-volatility compounds, some of them being major components of the fraction, whose molecular weight range from 232–238. From their molecular weight and mass spectral pattern, the DTD compounds seem to be oxygenated sesquiterpenes. The mass spectra of the most important component (18.6%, molecular weight 238) is similar to that published in Guillén and Ibargoitia (1996), for one of the major components (15.0%, compound 1c of that paper) of the *Jasonia glutinosa* pentane extract, tentatively characterized as a eudesmenediol.

Intermedeol, a common constituent of *Jasonia montana* (Del.) Botsch., and *Jasonia candicans* (Vahl.) Botsch., has been considered as a chemotaxonomical marker in the genus *Jasonia* (Hammerschmidt *et al.*, 1993), however, this compound has not been identified in *Jasonia glutinosa* D. C. The common presence of this compound could indicate that these two species present a close taxonomic relationship, or could be the result of their narrow growing range.

When compared with the essential oil extraction the main advantages of the DTD technique are the smaller sample amount required and the increased range of volatile compounds which can be subsequently analysed by GC-MS. Oxigenated sesquiterpene compounds in the 230–240 molecular weight range, which decompose in distillation, can be analysed by DTD-GC-MS using samples in the mg range. The technique is promising in the study of plants with complex volatile compositions as *Jasonia glutinosa.*

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