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Quality control of medicinal plants with an electronic nose

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Abstract

In the last decades, a new interest in active components of phytodrugs as well as in the control of their quality has pointed out new ways to identify and characterize natural plants which, till present, did not fulfill the claimed specifications—Valerians among them. Valerians exhibit numerous and traditional medical uses. In this paper, the use of an electronic nose to discriminate different varieties of Valerians, was investigated. The principal species—*valeriana officinalis* (VO) and *valeriana wallichii* (VW)—with different origins and, in some cases, different years of crop, were clearly separated with the electronic nose. The fact that valeric acid is present in VO variety and not in the VW and that there are differences in the valepotriates concentration in both varieties, determined the importance of discriminating both species, since they are active components exhibiting different therapeutic effect. The electronic nose has demonstrated to be a fast and effective tool to separate both species, giving besides other interesting results. Chemical chromatographic analysis was performed enabling the identification of species separated by the electronic nose.
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Keywords: Electronic nose; *Valeriana officinalis* (VO); *Valeriana wallichii* (VW)

1. Introduction

In the last decades, the direct employment of the medicinal plants has increased considerably [1–5], among them Valerians which are widely used medicinally [6–9]. The evaluation of active compounds of medicinal plants as well as the quality control of raw materials began to play an important role in the pharmaceutical and cosmetic industries. Besides, phytotherapy industry quality control demands the extraction of active species and the concentration of liquids in order to apply different chemical analysis techniques often implying a long and complicated task and, actually, modern techniques like the electronic nose, are being applied to study their properties [10,11].

Valerians belong to the Valerianaceus botanical classification. It is a perennial plant native to Europe, North and South America as well as parts of Northern Asia. Valerian bushes reach from 1 to 1.5 m height, growing in humid woods and coasts of streams and rivers. The most known variety is the *valeriana officinalis* (VO) following in importance the *valeriana wallichii* (VW). Valerian rhizomes are harvested manually or automatically, dried in a natural way in the shadow or in industrial driers (controlling the temperature to avoid the decomposition of active components as the valepotriates or the hydrolysis of esters). VO rhizome exhibits nearly clear yellow color and smooth consistency while the VW rhizome is dark yellow or brown with a ligneous consistency.

Chemical analysis of Valerians proved that they contain esters (bornyl acetate, bornyl-isovalerate, bornyl-formiate, eugenyl-isovalerate and isoeugenyle). Besides, camphene, α-therpineol, azulene, geraniol, borneol, β-caryophyllene, some ketons and the valeric and isovaleric acids were found. Valeric acid, which was found in the VO is absent in the VW and in other Valerian species. An important group of active compounds are the non-glycosidic iridoid esters known as valepotriates (like the valtrate, dihydro-valtrate, acevaltrate, etc. (Fig. 1). The valepotriates are found in higher concentration in the VW (3%) than in the VO (which only contains ∼1.2%). Cytotoxic and mutagenic effects of valepotriates have been evidenced, mainly in valtrate and hydro-valtrate. Valerians also contain alkaloids like chatinine, valerine, valerianine, isovaleriamide and actinidine.

With regards to Valerian applications, they are widely used at the present in phytotherapy, in allopathic and homeophatic medicine as sedative infusions and as restaurative of nervous system sicknesses. It was demonstrated in human experiments an anxiolytic effect. It differs from barbi-

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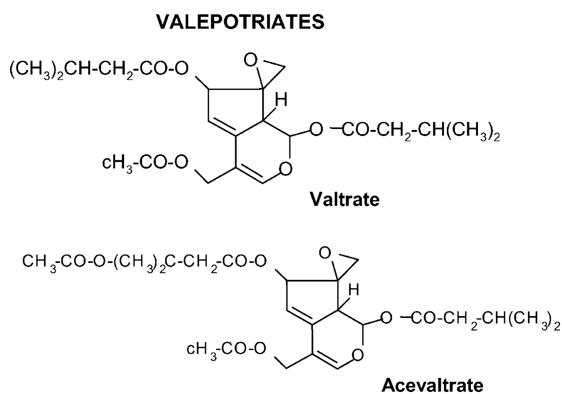

Fig. 1. Valepotriates structure.

turics because they do not interact with alcohol or produce hang-over when weaking up in the morning. It is also weakly hypotensor and antispasmodic (intestinal cramps, digestive problems, muscle cramps related to stresses, etc.) and exhibits diuretic properties.

In this paper, the use of an electronic nose to discriminate different varities of Valerians, was investigated. Valerians species with different origins and, in some cases, different years of crop, were separated with an electronic nose. The fact that valeric acid is present in VO variety and not in the VW and that, there are differences in the valepotriates concentration, determined the importance of discriminating both species exhibiting different therapeutic activity. The electronic nose has demonstrated to be a fast and effective tool to separate both species giving, besides, other interesting results.

2. Experimental

2.1. Electronic nose

Table 1 reports the abbreviated identification of the different analyzed species, the specimen types, origin of specimen (pointing out the crop year in case of Valerian from San Juan-Argentina) and commercial suppliers who were identified with numbers.

Table 1

Number	Variety	Origin	Supplier
VO-1	*Valeriana officinalis*	Bulgaria	1
VW-2	*Valeriana wallichii*	Pakistan	1
VO-3	*Valeriana officinalis*	San Juan-Argentina, crop 1997	2
NSV-4	Non-specified *valeriana*	China	3
VW-5	*Valeriana wallichii*	Pakistan	3
VO-6	*Valeriana officinalis*	San Juan-Argentina, crop 1999	2
NSV-07	*Valeriana* specified	Trujillo (Perú)	2
VO-8	*Valeriana officinalis*	Poland	4
VW-9	*Valeriana wallichii*	India	2

Rhizomes of different species were milled with a laboratory grinder and an equal weighed quantity of each specimen (1 g) was put in each 10 ml vial to be analyzed with the electronic nose MOSES II. Vials were placed in the DANI HSS 8650 headspacer of the nose and thermostated at 70 °C for an hour. Carrier gas was synthetic air (flow: 18 ml min^{-1}).

2.2. Chromatographic analysis

A gram of each specimen was extracted with dichlormethane, heated at 40 °C for 10 min, concentrating the extract to 1 ml final volume with CH_2Cl_2. Operating conditions of both used chromatographic systems are given:

2.2.1. TLC (thin layer chromatography)

- Stationary phase: silicagel chromatofolios 60 F 254, 0.2 mm thickness (Merck).
- Mobile phase: toluene-ethylacetate (75:25).
- Solvent front: 10 cm.
- Developer: chlorhydric acid/glac.acetic acid (2:8), heating at 105 °C for 10 min.

2.2.2. HPTLC (high performance thin layer chromatography)

- Stationary phase: silicagel 60 F 254, HTPLC (10 cm × 10 cm).
- Mobile phase: hexane/ethylacetate/glac.acetic acid (65:35:0.5).
- Solvent front: 5 cm.
- Developer: (a) UV 254 nm and; (b) anisaldehide/sulphuric acid, heating at 105 °C for 10 min.

3. Results and discussion

3.1. Electronic nose

Fig. 2 is the principal component analysis (PCA) plot, as obtained with the electronic nose data, of Valerian species (Table 1) showing a clear separation of different specimens—VO specimens of different origins (VO-1; VO-3; VO-6 and VO-8) are well-discriminated among them and from VW species. Besides, the discriminated specimens VO-3 and VO-6 belong to the same origin (San Juan, Argentina) but to crops harvested on different years. Data dispersion of the same VO variety is acceptable and assigned to the different geographic origin and harvest time. Specimens VW data appear in overlapped clusters with a minor dispersion. Fig. 3 is the magnification of the right zone of PCA plot of Fig. 2, showing that the overlapped clusters of VW-2 and VW-5 correspond to specimens with the same origin (but pro-

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