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Odor-active compounds of different lavender essential oils and their correlation with sensory attributes

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ABSTRACT

Volatile components of lavender (*Lavandula officinalis* L.) essential oils from five different cities were analyzed by using descriptive sensory analysis, gas chromatography-olfactometry (GC-O), gas chromatography- flame ionization detection (GC–MS) and statistics analysis. In order to assess the lavender essential oil, six sensory attributes (sweet, floral, woody, fruit, camphor and herb) were selected. Altogether 30 volatile compounds were identified and quantified by GC–MS, like 10 terpenes, 8 alcohols, 7 esters, 2 ketones and 3 other miscellaneous compounds. However, linalyl acetate occupied the largest chemical proportion among the volatiles of the essential oils. The relationship among samples, sensory attributes and aroma compounds was elaborated by using the partial least squares regression (PLSR). It turns out to be the case that limonene, linalool, linalyl acetate, camphor were typical aroma compounds covaried with characteristic aroma of lavender essential oils and the odor of the lavender essential oils was coincided with the geographical distribution.

1. Introduction

Lavender is an aromatic shrub used in the fragrance and alternative medicine industries. The lavender essential oil extracted from various kinds of lavender were recognized as a popular and easily recognizable fragrance. (Adam, 2006) reported that the lavender essential oils which were used as raw ingredients in industrial perfume and fragrance materials accounted for a large proportion of this market. Lavender, one of the oldest remedies, has been used in various kinds of forms like massage, aromatherapy, and medicinal baths for the calming, relaxing effect, abolishing nervous tension and sleep inducing properties (Aprotosoaie et al., 2014). High levels of linalool and linalyl acetate were identified in the lavender essential oil. More than 100 individual components contained in the lavender essential oil contributed to the chemical and sensory properties of the oils accompanying with many minor ones often unidentified and/or not quantified each. A volatile mixture of terpenes and their derivatives is generally charged with the characteristic fragrance of vegetable matters. The essential oil produced from L. Angustifolia is the most suitable for using in perfumes owing to its high linalool content (Lisbalchin and Lisbalchin, 2002). Studies on lavender essential oils have been reported previously. The comparison between the GC–MS results and the GC × GC–MS analysis of a lavender essential oil results have been conducted (Shellie et al., 2002). In this study, semi-quantitative analysis of the same nine lavender essential oil samples was applied by using gas chromatography (GC) with mass spectrometry detection (MS) and the components identified occupied over 95% of each sample. Also Bicchi et al. (2010) aimed at finding the best trade-off between separation of the most critical peak pair and analysis time, in enantioselective GC–FID and GC–MS analysis of lavender essential oil, using the GC method-translation approach. Zagorcheva et al. (2013) conducted a research on the comparative GC/MS analysis of lavender (*Lavandula angustifolia Mill.*) in florescence and essential oil volatiles. So far, the correlation among the lavender essential oil sample, sensory attributes and aroma compounds by PLSR and the GC − O analysis of the lavender essential coupled with the sensory evaluation have not been characterized.

Therefore, to identify which pivotal aroma compounds play a decisive role in the typical aroma of lavender essential oil, our study investigated the lavender essential oils from five different cities as following: (a) implementing a descriptive sensory evaluation to depict the aroma attributes and sort the aroma notes with different lavender essential oils; (b) identifying and quantifying odor-active compounds of lavender essential oils by GC–MS and GC-O; (c) using PLSR to establish the relationship among samples, sensory attributions, and aroma compounds. Reaching a better understanding of this study, the characteristic aroma of lavender essential oils will be improved by adjusting

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some certain compounds.

2. Materials and methods

2.1. Experimental Details

2.1.1. Experimental Materials

Lavender essential oils were purchased from five different cities of China, Sanya; Shanxi; Fujian; Xinjiang and Yunnan were obtained from the following companies, such as Sanya Rose Valley Aiwei Industrial Co., Ltd. (C1), Fujian Olydfjy Unilateral oil Co., Ltd. (C2), Puyang Henan Natural Medicine Essential Oil Co., Ltd. (C3), Xinjiang Scent Biotechnology Co., Ltd. (C4), Yunnan Sabossin Science and Technology Co., Ltd. (C5). The chosen manufacturers were verified and we are sure that the manufacturers were formal factories in China. Moreover, all the details of the five samples were confirmed with the manufacturers. The species of the lavender is Lavandula Angustifolia, the method of extraction is steam distillation, and the samples were not adulterated. Mehyl nonanoate (inter standard) and n − alkane standards (C_7-C_{30}) were obtained from Sigma- Aldrich Chemical Co (St. Louis, Mo). Pure authentic reference compounds were purchased from Shanghai Titan technology Co., LTD.

2.1.2. Experiment (i): sensory evaluation

Corresponding to the International Standard ISO8589:2007, the sensory evaluation of five lavender essential oils were conducted in a sensory laboratory. (Standards I, 2007 Sensory analysis, 2007) The sensory evaluation was carried out by an experienced panel insisting of eight members. Six sensory attributes of sweet, floral, woody, fruity, camphor and herb were developed and described (in Table 1) according to the China National Institute of Standardization (CNIS)GB/T10781-2006 and GB/T 26760 2011. According to the ISO standards, all panel members have passed screening tests and they had discussed aroma characteristic of the lavender essential oil samples thoroughly through three preliminary sessions before the quantitative descriptive sensory analysis. Firstly, descriptive terms of the lavender essential oils were generated by panelists. Secondly, a variety of aroma standards were presented and analyzed by panelists. After these analyses, six aroma terms (sweet, floral, woody, camphor, fruity, herb) were chosen for further descriptive research. Thirdly, the lavender essential oils were evaluated in triplicate with a 10 point interval scale (0 = none, 9 = extra strong). Then, scoring data for each assessor and the entire panel were analyzed using analysis of variance. Smelling strips were used for detecting the odor. One end of a smelling strip (about 1 cm) was dipped into the sample. Three deep and quick sniffs were achieved from the smelling strip and then remove the odor source. Clean air was obtained between each assessment. A gap of 20 s was sufficient to the individual odor assessments (Yoshikawa, 1989).

Table 1
The mean intensity values of the six attributes for the five lavender essential oil in descriptive sensory evaluation.

Sample	Mean score					
	Sweet	Floral	Woody	Camphor	Fruity	Herb
C1	4.13^{a}	3.50^{a}	7.50^{b}	7.50^{c}	1.88^{ab}	3.88^{a}
C2	6.63^{b}	5.50^{bc}	6.50^{b}	4.63^{a}	2.88^{bc}	6.25^{bc}
C3	4.62^{a}	5.25^{abc}	6.25^{b}	5.63^{ab}	2.75^{bc}	6.38^{bc}
C4	4.50^{a}	6.75^{c}	3.75^{a}	4.88^{bc}	3.50^{c}	7.50^{c}
C5	7.38^{b}	4.50^{ab}	4.63^{a}	4.38^{a}	1.00^{a}	5.38^{ab}

Mean scores for each attribute within a column with different letters are significantly different ($p < 0.05$) using Duncan's multiple comparison tests ($n = 24$; 8 panelists with three replications).

2.1.3. Experiment (ii): Gas chromatography-olfactometry (GC-O)

The GC-O was consisted of an Agilent 7890A Chromatography combined with a flame ionization detector (FID) and an ODP-2 Olfactory This system let us obtain a FID signal for the quantification and the odor characteristics of each compound. Samples were analyzed on a HP − INNOWAX column (60m × 0.25 mm ID. × 0.2 μm, J & W Scientific). At the end of the capillary column, the effluent was split into 2:1 for the FID and sniffing port, using deactivated and uncoated fused silica capillaries as transfer lines, and the sniffing cone was purged with humidified air to help maintaining olfactory sensitivity by reducing dehydration of mucous membranes in the nasal cavity. The flow rate of carrier gas (He) was 1 ml/min, the injection volume was 0.2 μL.

Following was the conditions for GC-O analysis: the oven temperature was first increased from 40 °C (6 min) to 100 °C at the speed of 3 °C/min, and then ramped at 5 °C/min to 230 °C in 20 min; the injector and FID detector temperatures were respectively set at 250 and 280 °C.

Six assessors including three males and three females with the average old of 25 years old were rigorously selected and trained for 3 months in GC-O by sniffing at least 30 odor-active reference compounds in a concentration 10 times above their odor thresholds in air (Gao et al., 2014; Fan and Qian, 2006). During a GC run described above, a panelist let his/her nose close to the sniffing port and then with a response to the aroma intensity of the stimulus, then wrote down the aroma descriptor, intensity value and the retention time. The aroma descriptors were determined according to an evaluation of the odor quality of reference odorants previously. The sniffers could use the vocabulary randomly but describe the odors as simple and precise as possible (Breme et al., 2010). A six point scale ranging from 0 to 5 was utilized for intensity judgment: 0 = none, 1 = very weak, 2 = weak, 3 = moderate, 4 = strong, and 5 = very strong. The mean intensity for each sample was an average result of the six panelists.

Peak identifications of the odorants were performed by comparison of mass spectra with those of the NIST 05 a. L database (Agilent Technologies Inc., Santa Clara, CA, USA). Positive identification was achieved by comparison of their odors, retention indices (RIs) and mass spectra with those of pure standards. RIs of the odorants were calculated from the retention times of n- alkanes (C5 − C30), according to a modified Kovats method (Cates and Meloan, 1963).

2.1.4. Experiment (iii): gas chromatography–mass spectrometer (GC–MS)

An Agilent 7890 system coupled with a mass spectrometer (GC–MS, HP, 5973C) was used to detect the samples. Separation of compounds was achieved on Innowax-Wax (60 m × 0.25 mm i.d. × 0.25 μm film thickness, Agilent Technologies) and DB-5 (60 m × 0.25 mm i.d. × 0.25 μm film thickness, Agilent Technologies). The mass detector was operated in scan mode. The Mass spectrum was operated in electron impact (EI) mode with an ionization voltage of 70 eV. The flow rate of carrier gas (He) was 1 ml/min (34 cm/s) in a split ratio of 20:1 and the injection volume was 0.2 μL.

Following was the conditions for GC–MS analysis: the oven temperature was first increased from 40 °C (6 min) to 100 °C at the speed of 3 °C/min, and then ramped at 5 °C/min to 230 °C in 20 min; the injector and MS detector temperatures were respectively set at 250 and 280 °C.

2.2. Statistical analysis

The descriptive analysis data was assessed by variance (ANOVA) accompanied with SAS v8 (SAS Institute Inc., Cary, NC, USA.). The discrepancies among individual sample for each sensory attribute were distinguished by ANOVA with the method of Duncan's multiple comparison tests. The relationship among the samples, the volatile compounds and sensory intensity attributes were evaluated by calculating the Spearman's rank order correlation coefficient.

The descriptive analysis data was also assayed by CA (Cluster Analysis) using XLATAT ver. 2010 (Addinsoft, New York, NY, USA). CA, using physical distances between trajectories, is applied to classified