



Chemometric analysis of French lavender and lavandin essential oils by near infrared spectroscopy



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ABSTRACT

Chemometric treatments of near infrared (NIR) spectra were used firstly to understand data structure by principal component analysis (PCA), to discriminate, by partial least squares-discriminant analysis (PLS-DA) regression, French lavender and lavandin essential oils (EOs) samples ($n = 160$) and the seven varieties (Abrial, Fine, Grosso, Maillette, Matherone, Sumian and Super) and to quantify the main compounds such as linalyl acetate, linalool, eucalyptol and camphor by PLS regression models. The study was carried out over three crop years (2012–2014) to take seasonal variations into account. French lavender and lavandin EOs and their varieties were well classified (100% for lavender/lavandin EOs and between 96 and 100% for varieties) by PLS-DA regression models. The calibration models obtained by PLS regression for the determination of the main compound contents revealed good correlation (≥ 0.97) between the predicted and reference values. In the case of major compounds including linalyl acetate and linalool, the relative error of prediction (REP) is close to 2.5%. Partial least squares regression vectors allowed us to identify lavandulyl acetate, eucalyptol, linalool, camphor, *trans*- β -ocimene, β -caryophyllene and linalyl acetate as metabolomic indicators of Fine, Maillette, Matherone, Abrial, Grosso, Super and Sumian varieties respectively. The use of NIR spectra allowed for an improvement in French lavender and lavandin EOs characterization, quality control and traceability.

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1. Introduction

The *lavandula* family (Lamiaceae) is composed of thirty nine *lavandula* species which are mostly of Mediterranean origin, such as *Lavandula angustifolia* (lavender) and numerous intraspecific taxa and hybrids such as lavandin. The smell of the *lavandula* family and its color are typical of the Mediterranean landscape, particularly in France. Its essential oil is used for cosmetics, perfumes, and in medicine (Cawthorn, 1995; Knowlton and Pearce, 1993; Lis-Balchin, 2003; Lis-Balchin and Hart, 1999; Piccaglia et al., 1993; Raut and Karuppayil, 2014; Vakili et al., 2014). The Lamiaceae family is classified in several species which are subdivided into varieties, each with its physical and environmental characteristics. The best known is the population lavender including *L. angustifolia*, and among its varieties (Maillette, Matherone and Fine), the Fine variety is the most famous thanks to its essential oil yield and its high linalool content. Lavandin is a hybrid between *L. angustifolia* P. Mill. and *L. latifolia* (L.f.) Medikus, whose culture is more recent and more focused on “industrial” production. Among the

main varieties of lavandin EOs (Abrial, Grosso, Sumian and Super), the Grosso variety is the most famous for its essential oil yield (Lis-Balchin, 2004). Lavender and lavandin oils contain more than one hundred compounds including linalyl acetate, linalool, camphor, borneol, eucalyptol and β -caryophyllene, each contributing to the chemical and sensory properties of the oils. The major distinction between varieties of lavandin essential oils is in their relative contents of linalyl acetate, linalool, eucalyptol and camphor. The chemical composition of lavandin has a higher camphor content than true lavender. Chemical composition can be revealed using gas chromatography (Bicchi, 2000; Can Başer and Özek, 2012; Cserhádi et al., 2005; Daferera et al., 2002; Marriott et al., 2001; Nikolić et al., 2014; Pallado et al., 1997; Ter Heide et al., 1970) and gas chromatography-sniffing (Chin and Marriott, 2015; Cserhádi et al., 2005; Marriott et al., 2001). These methods are usually applied for quality control purposes (Do et al., 2015) and selection of high-quality plants, but they are very time-consuming, and attempts have been made to find alternative analytical analysis methods. Also, to our knowledge, the quality of an essential oil depends on three main factors: quality of the plant, harvesting, distillation. However, on the basis of these criteria, only specialists are able to differentiate one variety from another. In this context, vibrational spectroscopic methods such as NIR spectroscopy in combination

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with chemometric treatments were successfully introduced for a non-destructive determination of metabolites occurring in essential oils, and to facilitate the implementation of this method on an industrial scale. Vibrational spectroscopy methods offer a global metabolic fingerprint of essential oils and allow the prediction of metabolite contents such as camphor (Allwood et al., 2007; Bombarda et al., 2008; Cozzolino, 2009; Dupuy et al., 2013, 2014; Maietti et al., 2013; Moco et al., 2007; Schulz et al., 2008; Tankeu et al., 2014).

The aim of this study was to show the advantages of NIR spectroscopy associated with chemometric treatments for direct and rapid test methods. These can be used for lavender and lavandin EOs characterization, quality control and traceability. This methodology can also be used to identify metabolomic indicators and establish a faster quality control for discrimination of lavender and lavandin essential oils based on their specificities.

To achieve these goals, in the first part of this study, data mining was carried out by principal component analysis (PCA) to evaluate differences and similarities between sample spectra; partial least squares (PLS) regression was used to develop lavender/lavandin EOs or varieties prediction models. Partial least squares regression was also used to quantify the main compounds such as linalyl acetate, linalool and camphor with gas chromatography as reference data. In the second part, the PLS regression vectors obtained for the prediction of the main compounds were compared with the PLS regression vectors obtained for the prediction of the EO varieties to identify the metabolomic indicators of each variety.

2. Materials and methods

2.1. Essential oil samples

One hundred sixty samples were analyzed including lavandin oil samples ($n=94$) and lavender oil samples ($n=66$) obtained from three French cooperatives (“Société Coopérative Parfums Provence Ventoux” in Sault, “Société Coopérative des Plantes à Parfums de Provence” in Simiane la Rotonde and “France Lavande” in Montguers), over three years of harvest (2012–2014), including various varieties and various French collect areas (Unknown department (00), Alpes-de-Haute-Provence (04), Ardèche (07), Drôme (26) and Vaucluse (84)). The lavender/lavandin essential oils studied were [*L. angustifolia* Miller] (lavender) and its hybrid [*L. angustifolia* Miller \times *L. latifolia* Linnaeus filis Medikus] (lavandin) type France; they were divided into varieties: Fine (FI, $n=21$), Maillette (MA, $n=29$) and Matherone (MT, $n=16$) for lavender samples and Abrial (AB, $n=16$), Grosso (GR, $n=37$), Sumian (SU, $n=19$) and Super (SP, $n=22$) for lavandin samples.

2.2. Pure standard samples

Pure standard substances linalyl acetate, linalool, camphor, eucalyptol, borneol, *trans*- β -ocimene, lavandulyl acetate and β -caryophyllene were purchased from Lavender France (Montguers, France), Fluka (Buchs, Switzerland), Alpha Aesar (Karlsruhe, Germany), Merck (Schuchardt, Germany), Alpha Aesar (Karlsruhe, Germany), Sigma–Aldrich (Steinheim, Germany), Adrian (Aix-les-Milles, France) and TCI Europe (Zwijndrecht, Belgium) respectively.

2.3. Gas chromatography (GC)

2.3.1. Gas chromatography coupled to a mass spectrometer (GC–MS) analysis

Gas chromatography coupled to a mass spectrometer analyses were performed on a 7890A GC system coupled to a 5975C VL mass spectrometer detector (Agilent Technologies) equipped with a HP-5MS capillary column (J&W Scientific, 30 m \times 0.25 mm,

0.25 μ m film thickness). Data acquisition and processing were performed using the MSD Chemstation E.01.01.335 (Agilent) software. One microliter of diluted essential oil (80 μ L in 1.5 mL of ethanol) was injected. The experimental conditions developed in the laboratory were: oven temperature program, 2 min at 80 °C, then 80 °C to 200 °C (5 °C/min), then 200 °C to 260 °C (20 °C/min), and held at final temperature for 5 min; temperatures injector (split ratio 60) and detector were set to 250 °C; carrier gas was helium at a flow rate of 1.2 mL/min; solvent delay 2 min; ionization voltage 70 eV; electron multiplier 1 kV.

2.3.2. Gas chromatography (GC) analysis

Gas chromatography analyses were performed on a 7890A GC (Agilent Technologies) system with a flame ionisation detector (FID) equipped with a HP5 capillary column (J&W Scientific, 30 m \times 0.25 mm, 0.25 μ m film thickness). The data acquisition and processing were performed using the Chemstation B.04.03-SP1 (87) (Agilent) software. The experimental conditions developed in the laboratory were the same as GC–MS except for carrier gas which was hydrogen. Linear retention indices were calculated with reference to *n*-alkanes (C8–C28).

2.4. Near infrared (NIR) spectroscopy

The spectra of each lavender or lavandin EOs and liquid standard compounds placed in a quartz cell (2 mm) were recorded from 4000 to 10,000 cm^{-1} using the software result integration 2.1 (Thermo Nicolet), with 4 cm^{-1} resolution and 64 scans on a Nicolet Antaris II spectrometer equipped with an InGaAs photodiode detector, an H₂ NIR source and a CaF₂-germanium beam splitter. An empty quartz cell was taken as reference for the background spectrum before analysis of each sample. The near infrared spectrometer was situated in an air-conditioned room (21 °C). Spectra of solid standard compounds (camphor and borneol) were recorded using an integrating sphere in diffuse reflectance mode from 4000 to 10,000 cm^{-1} , with 4 cm^{-1} resolution and 64 scans. A background spectrum was collected under the same conditions before measurement of each sample (interleaved mode).

2.5. Chemometric analysis

2.5.1. Principal component analysis (PCA)

Principal component analysis (PCA) (Cozzolino et al., 2011; Ebsensen et al., 2002; Kumar et al., 2014; Wold et al., 1987) is an unsupervised modeling method that allows for exploratory data analysis; it extracts information from data set and removes noise; it reduces the number of dimensions; and it allows for classification of samples by investigating similarities and differences between the samples. The principal component analysis projects into a smaller number of latent variables called principal components (PC). Each principal component explains part of the total information contained in the original data and the first PC is the one that contains the most information, followed in descending order in terms of information by PC2 and PC3 and so on. Plotting two PCs relatively to each other thus allows for interpretation of some groups, thanks to the similarities or differences between samples.

2.5.2. Partial least squares regression (PLS)

Partial least squares (PLS) regression (Daszykowski et al., 2007; Ebsensen et al., 2002; Kumar et al., 2014; Liang and Kvalheim, 1996; Martens, 1979; Sjöström et al., 1983) is a supervised method which is based on the relation between signal intensity (spectrum) and the characteristics of the sample (*Y* variable). Interference and overlapping information may be overcome by using a powerful multicomponent analysis such as PLS. The algorithm is based on the ability to mathematically correlate spectral data to a property

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