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Chemical composition and antifungal activity of *Anacyclus valentinus* essential oil from Algeria

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ABSTRACT

The chemical composition of *Anacyclus valentinus* essential oil from Algeria was determined and its antifungal effect *in vitro* toward 7 toxigenic fungal strains was evaluated. Essential oil yield of 0.13% (v/w) was obtained from the aerial parts of *A. valentinus* using hydrodistillation. The GC/MS analysis identified 29 components of which δ-3-carene (31%), spathulenol (14.2%), decanoic acid (5.5%), δ-cadinene (4.4%), anethole (3.4%) and aromadendrene (3.3%) were the major compounds of this oil. The results of the antifungal activity showed that the most sensitive fungal strains to *A. valentinus* oil were *Fusarium graminearum*, *Aspergillus parasiticus*, and *Penicillium expansum* with lower minimal fungicidal concentration (MFC) of 1.25 µl/ml (v/v). The oil also had a strong fungicidal effect against *A. flavus* and *F. moniliforme* at a MFC value of 2.5 µl/ml, while the oil concentration of 10 µl/ml was needed to show a fungicidal activity against *A. ochraceus* and *P. citrinum*. This study suggested that *A. valentinus* oil is a potential candidate to be used as a safe biocontrol agent to prevent food crops from fungal diseases and improve product quality and safety.

1. Introduction

The *Anacyclus* is a Mediterranean genus of mostly annual herbs, belongs to the *Asteraceae* family, comprising about 12 species distributed mainly in the North West of Africa, but it is also found in Southern Europe and the Middle East (Bremer & Humphries, 1993). Among them, *Anacyclus valentinus*, which is an annual specie found in different regions of Algeria, is commonly known as “ghertoufa” or valence anacycle (Side Larbi, Meddah, Tir Touil Meddah, & Sonnet, 2016). In Algeria, *A. valentinus* has been traditionally used in folk medicine (Selles et al., 2013) and as a food condiment in some parts of the country (Side Larbi et al., 2016). It is a rich source of terpenoids and flavonoids (Greger, 1978; Selles et al., 2013) and may have useful biological activities.

Mycotoxin-producing fungi, especially within *Aspergillus, Penicillium* and *Fusarium* species, are significant destroyers and contaminants of agricultural products and seeds in the field, and during processing and storage lead to significant economic losses and have serious health hazards for humans and animals (Anjorin et al., 2013; Jimoh & Kolapo, 2008). Moreover, chemical control of several crop diseases mainly involves the use of synthetic fungicides; however, the increasing concern of authorities and consumers about the possible effects of toxic residues on human health and the environment is leading to more strict control on the use of these hazardous chemicals (Anjorin, Salako, & Makun, 2013; Marin, Ramos, & Sanchis, 2008). Thus, different strategies have been proposed as alternatives to synthetic fungicides, among them the biological control using essential oils from plants and other natural products from bacteria and fungi which could be an alternative and complement to existing methods of control (Fallik, 2008).

Essential oils or some of their components are largely used for their antibacterial, antifungal and insecticidal activities, without showing the same secondary effects as synthetic chemicals (Bakkali, Averbeck, Averbeck, & Idaomar, 2008). Several essential oils of different *Anacyclus* species have been studied for their biological activities *in vitro*; however, there are few reports on the antimicrobial activities of *A. valentinus* essential oils, without characterizing their chemical composition. Therefore, the present study was carried out to determine the chemical composition of *A. valentinus* essential oil from Algeria and evaluate its antifungal effect *in vitro* toward 7 toxigenic fungal strains belonging to *Fusarium, Penicillium,* and *Aspergillus* species.

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2. Materials and methods

2.1. Plant material

Anacyclus valentinus samples (aerial parts) were collected from the region of Hassi R'mel, Laghouat, Algeria during the flowering stage from April to June, 2015. After transportation to the laboratory, the samples were air dried at room temperature (< 30 °C) in the dark and stored in paper bags until use, a maximum of two months. The plant was then confirmed by the botanists of the Department of Agriculture, Laghouat University, Laghouat, Algeria.

2.2. Analysis of essential oil using GC/MS

To obtain essential oil, the dried samples were subjected to hydrodistillation for approximately 4 h using a Clavenger-type apparatus (Schott Duran, Mainz, Germany), using the British Pharmacopoeia method (British Pharmacopoeia, 1990). A Perkin Elmer Clarus instrument model 500 GC/MS (Yokohama, Japan), equipped with a SGE capillary column (60 m × 0.25 mm ID, BPX5, film thickness 0.25 μm, Austin, TX, USA), was used to determine the chemical composition of the oil obtained. The GC/MS analysis was carried out with the following conditions: flow rate of carrier gas (helium, purity 99.95%) 1.5 ml/min; splitless injection mode with ionization voltage 70 ev; mass spectra range of 35–425 *m/z*; column oven temperature programmed at 60–250 °C (4 °C/min), maintained at 250 °C (10 min), and injector temperature 240 °C. The injection volume of diluted sample (1% n-hexane, v/v) was 1 μl. The oil components were identified by comparison of their mass spectra with those from NIST and Wiley libraries (http://chemdata.nist.gov and https://www.wiley.com) or reported in the literature (Adams, 2007). The relative percentage of the oil constituents were calculated from the peak area of the chromatogram using the software provided by the manufacturer and assuming peak areas for different compounds were all proportional to their molecular concentration.

2.3. Antifungal activity

The antifungal susceptibility testing of *A. valentinus* essential oil was done using a broth macrodilution method M38-A (CLSI Clinical and Laboratory Standards Institute, 2002) with 7 toxigenic fungal strains. One type strain was from the Central Bureau of Fungal Cultures (CBS) culture collections of micro-organisms (Utrecht, The Netherlands) (*Aspergillus parasiticus* CBS 100926), and two type strains were from the Agricultural Research Service (ARS) culture collection (United States Department of Agriculture, Washington, DC, USA) (*Aspergillus flavus* NRRL 3251, and *Aspergillus ochraceus* NRRL 3174). Four type strains were from the Belgian Coordinated Collections of Micro-organisms (Catholic University of Leuven, Belgium) (*Fusarium moniliforme* MUCL 53645, *Fusarium graminearum* MUCL 53452, *Penicillium expansum* MUCL 29192, and *Penicillium citrinum* MUCL 31475). In all experiments, RPMI-1640 medium (Roswell Park Memorial Institute medium, Sigma R6504, St. Louis, MO, USA) adjusted to pH 7.0 with MOPS (morpholinopropane-1-sulfonic acid, Sigma M3183) buffer was used. Briefly, serial doubling dilutions from 0.04 to 20 μl/ml were prepared in DMSO (dimethyl sulfoxide, Sigma 34943), and the final concentration of the DMSO was ≤ 1%. For each fungal strain, inoculum was prepared from 7 to 14 day cultures, adjusted to the density of a 0.5 McFarland standard at 530 nm wavelength using a spectrophotometer (Jenway, 6405 UV/VIS, Essex, UK) and diluted 1:100 to obtain a working suspension within the range of 0.4–5 × 10^4 CFU (colony-forming units)/ml. Then, the test tubes were inoculated in triplicate, including two control tubes (sterility and growth) per strain. After incubation at 35 °C for 48 h/72 h, the minimal inhibitory concentrations (MIC) were determined visually. From each negative tube, 20 μl aliquots were then transferred onto Sabouraud dextrose agar (Eur-Pharm, 1024.00, Madrid, Spain) plates, and the minimal fungicidal concentrations (MFC) were evaluated after 72 h incubation at 35 °C.

Table 1
Composition of *Anacyclus valentinus* essential oil characterized using GC/MS.

No.	Components	Retention time (min)[a]	% Composition *Anacyclus valentinus*	Identification[b]
1	δ-3-Carene	11.81	**31.0**	MS
2	Limonene	14.86	1.7	MS
3	*trans*-Ocimene	16.72	1.9	MS
4	*allo*-ocimene	19.98	1.3	MS
5	Naphthalene	21.64	0.8	MS
6	Estragole	22.25	1.4	MS
7	1-Methyldodecylamine	22.59	0.6	MS
8	Adrenalone	23.32	0.2	MS
9	Cuminaldehyde	23.61	2.5	MS
10	Carvone	23.70	2.7	MS
11	Anethole	24.93	**3.4**	MS
12	Thymol	25.47	2.1	MS
13	Decanoic acid	28.15	**5.5**	MS
14	Geranyl acetate	28.30	1.9	MS
15	Aromadendrene	28.56	**3.3**	MS
16	α-Curcumene	30.16	2.3	MS
17	Acenaphthylene	30.60	2.9	MS
18	β-Patchoulene	31.12	0.7	MS
19	δ-Cadinene	32.62	**4.4**	MS
20	Spathulenol	34.11	**14.2**	MS
21	Agarospirol	34.41	1.2	MS
22	Cis-α-Copaene-8-Ol	34.57	1.7	MS
23	α-Bisabolol	35.09	1.5	MS
24	Zizanyl Acetate	35.40	1.0	MS
25	β-Ionone	35.84	1.0	MS
26	Trimethyl-decalin	39.99	0.9	MS
27	Nerolidol	42.07	0.4	MS
28	Eicosane	47.68	0.5	MS
29	Tricosane	54.06	0.3	MS
	Total identified		93.3	
	Monoterpenes hydrocarbons		35.9	
	Oxygenated monoterpenes		14.0	
	Sesquiterpenes hydrocarbons		10.7	
	Oxygenated sesquiterpenes		19.0	
	Others		13.7	
	Oil yield (%)		**0.13**	

[a] Retention time (min) in elution order from the SGE column.
[b] MS: NIST and Wiley libraries, and the literature.

3. Results and discussion

An essential oil yield of 0.13% (v/w) was obtained from the aerial parts of *Anacyclus valentinus* using hydrodistillation (Table 1). Several studies have extensively analyzed the essential oils of different species of *Anacyclus*. However, little information is available on the chemical characterization of the essential oils obtained from different *A. valentinus*. In one study (Side Larbi et al., 2016), a yield of 0.63% was obtained from the aerial parts of *A. valentinus* grown in Algeria, which is noticeably higher than that obtained from the current samples. Table 1 also shows the qualitative and quantitative compositions of *A. valentinus* oil. The GC/MS analysis identified 29 components, corresponding to more than 93.3% of the total sample. δ-3-Carene, spathulenol, decanoic acid, δ-cadinene, anethole, and aromadendrene were the major compounds of *A. valentinus* oil. Other constituents were also identified in this oil with minor concentrations. Side Larbi (2016) reported that the main components of *A. valentinus* grown in Algeria were germacrene (15.5%), *trans*-chrysanthenyl acetate (12.4%), *trans*-thujone (7.3%), eudesma-4(15),7-diene-1-β-ol (6.2%), and β-biotol (5.1%). The composition of essential oils depends on several factors including