Industrial Crops & Products 119 (2018) 249–254

Contents lists available at ScienceDirect

Industrial Crops & Products

journal homepage: www.elsevier.com/locate/indcrop

Chemical profile and bioactive properties of the essential oil isolated from *Ammodaucus leucotrichus* fruits growing in Sahara and its evaluation as a cosmeceutical ingredient

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ARTICLE INFO

Keywords:
Ammodaucus leucotrichus
Essential oils
Bioactivity
Preservers
Cosmeceuticals

ABSTRACT

Ammodaucus leucotrichus is a medicinal plant commonly used in Algeria by the indigenous populations, especially due to its therapeutic effects. In this context, the aim of the present study was to chemically characterize the essential oil of *A. leucotrichus* fruits (EOALF) growing in Algerian Sahara, and to evaluate its bioactive properties (antimicrobial, antioxidant and anti-inflammatory). Considering the interest of the cosmetic industry for natural ingredients, and taking into account the obtained biological properties, the essential oil was also evaluated by incorporation in a base cosmetic (cream). The essential oil was extracted with a yield of 2.58 ± 0.17%, being perilla aldehyde identified as the main component, accounting for 85.6% of the total composition. Concerning the tested bioactivities, EOALF presented antioxidant potential, a strong anti-inflammatory activity, and was effective against the tested microbial strains (*Staphylococcus aureus*, *Eschericia coli* and *Pseudomonas aeruginosa*), being *S. aureus* the most sensitive bacteria. After incorporation in a base cosmetic, the developed formulation was able to preserve the EOALF bioactivities along 28 days under storage. The obtained results, with relevance for the strong-anti-inflammatory activity, pointed out the interest to exploit this essential oil as a cosmeceutical ingredient in the cosmetic industry.

1. Introduction

The Sahara is not only the largest desert, but also the most expressive and typical due to its extreme aridity, presenting a discontinuous and very irregular vegetation mat (Le Houerou, 1990; Boukerker et al., 2016). The Saharan climate is characterized, in particular, by a weak and irregular precipitation, intense brightness, high potential evapotranspiration and high thermal amplitude. These climatic characteristics create drastic conditions that cannot be easily tolerated by the living ecosystems, resulting in a phytochemical adaptation and the appearance of new protective molecules against this extreme environment (Sitouh, 1983; Koull and Chehma, 2015).

The status of the spontaneous flora in Sahara Desert, and its relationship with local populations, deserve special attention. In addition to their ecological importance, they found many traditional uses in terms of pharmaceuticals, food and other domestic applications (e.g. as garden decoration). These plants have the ability to synthesize many compounds called secondary metabolites constituting an immeasurable source of important molecules, including polyphenols and essential oils of high chemical diversity, and possessing a wide range of biological activities (Jean and Jiri, 1983; Baamer et al., 2015). Thus, studies on the biological effects of medicinal plants have increased remarkably in

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https://doi.org/10.1016/j.indcrop.2018.04.043
Received 28 March 2018; Accepted 15 April 2018

the recent years due to their potential to be used as sources of several drugs (Haddouchi et al., 2016).

Among the huge plant diversity of the Sahara Desert, *Ammodaucus leucotrichus* Cosson & Durieu, which belongs to the *Apiaceae* family (Umbelliferae), inhabits the maritime sands in the Saharan and sub-Saharan countries of North Africa, Morocco, Algeria and Tunisia, extending to Egypt and tropical Africa (Velasco-Negueruela et al., 2006). *A. leucotrichus,* known in Algeria as "KammÛnes-sofi", is a medicinal plant with an extended culinary use by the indigenous populations against stomach pain, indigestion, diarrhea, vomiting, fever, spasms and colic, intestinal worms, constipation (Merzouki et al., 2000; Didi et al., 2003; Benhouhou, 2005; Chehma, 2006; Hammiche and Maiza, 2006; Fakchich and Elachouri, 2014), in the treatment of allergy symptoms (Didi et al., 2003; Hammiche and Maiza, 2006) and also against coughing, as emmenagogue and against anorexia (Hammiche and Maiza, 2006). In Tassili (Algeria), the fruits and the leaves are commonly consumed in infusions, being the fruits most consumed for their bioactive capacity mainly in the treatment of heart palpitations (Jouad et al., 2001), and the leaves for their flavoring properties in tea. The powder form is also an appreciated spice for foodstuff (Benhouhou, 2005; Chehma, 2006; El-Haci et al., 2014).

A. leucotrichus from different regions has been studied regarding their bioactive properties. Dahmane et al. (2017) studied the antioxidant and the antimicrobial properties of *A. leucotrichus* essential oils from Algeria. Alaoui et al. (2014) reported also the antibacterial and antifungal activity of the essential oils obtained from this species from Morocco. The same species from Morocco were also studied as antifungal agents against postharvest phytopathogenic fungi in apples (Manssouri et al., 2016). Considering the application in cosmetics, as far as we know, there are no reports on the use of the essential oil of *Ammodaucus leucotrichus*.

In the cosmetic industry, the preservation against microbial contamination and oxidation is a highly relevant topic. In this context, the important antimicrobial activity and powerful antioxidant properties of some essential oils have led many researchers to propose their use in cosmetics as natural preservatives. In fact, different reports from the literature have shown the preservative efficacy of a high array of natural products in cosmetic formulations (Popescu et al., 2014; Patrone et al., 2010; Yorgancioglu and Bayramoglu, 2013; Kunicka-Styczyńska et al., 2009, 2011; Kerdudo et al., 2016). In addition, essential oils can be incorporated in cosmetic products due to several other associated properties such as anti-inflammatory, emollient and humectant capacity, dye power, wound healing, anti-mutagen, anti-aging, protective effect against UV-B damage and skin discoloration (Dreger and Wielgus, 2013). In this context, the aim of the present work was to chemically characterize the essential oil obtained from *Ammodaucus leucotrichus* fruits (EOALF), growing in Algerian Sahara, and study its biological activity, including antimicrobial, antioxidant, and anti-inflammatory. Furthermore, the essential oil was tested as a cosmeceutical ingredient and their biological efficacy in a base cream evaluated along storage time (7, 14, 28 and 43 days).

2. Material and methods

2.1. Standards and reagents

Methanol was of analytical grade and supplied by Pronalab (Lisbon, Portugal). *n*-Hexane (purity ≥ 99.0%) was purchased from Merck (Darmstadt, Germany). 2,2-Diphenyl-1-picrylhydrazyl (DPPH) was obtained from Alfa Aesar (Ward Hill, MA, USA). Dulbecco's modified Eagle's minimum essential medium (DMEM), fetal bovine serum (FBS), Griess reagent system (Promega), DMSO, lipopolysaccharide (LPS), were obtained from Sigma-Aldrich Co. (Saint Louis, MO, USA). The bacterial strains (*Eschericia coli* ATCC 10536, *Staphylococcus aures* ATCC 19213 and *Pseudomonas aeruginosa* ATCC 9027) were purchased from Liofilchem Bacteriology Products (Italy). The culture media Muller Hinton broth (MHB) and Tryptic Soy Broth (TSB) were obtained from Biomerieux (Marcy l'Etoile, France). Blood agar with 7% sheep blood and MacConkey agar plates were purchased from Biomerieux Marcy l'Etoile, France). The dye *p*-iodonitrotetrazolium chloride (INT) was purchased from Sigma-Aldrich (Spruce Street; St. Louis, MO, USA) and was used as a microbial growth indicator. Helium, hydrogen, and synthetic air were acquired from Air Liquide (Portugal). The base cream was purchased from Fagron Iberica S.A.U. (Barcelona, Spain).

2.2. A. leucotrichus fruits samples and essential oil extraction

A. leucotrichus fruits were collected in March of 2015 in Tiouliline (wilaya of Adrar), south of Algeria (27°52′N and 0°17′W). The plant material was identified by Dr. Tayeb Si Tayeb (Laboratory of Biotoxicology, Pharmacognosy and Biological recovery of plants, University of Moulay-Tahar, Saida, Algeria). A voucher specimen was deposited at the Herbarium of the Laboratory under the code number LBPBP-TS03-12. The plant fruits were dried in the dark at room temperature and preserved until extraction.

The essential oil was extracted using a Clevenger-type apparatus. Briefly, 400 g of the dried fruits were subjected to hydro-distillation with 4 L of distilled water during 4 h. The obtained essential oil was dried over anhydrous sodium sulphate and then stored in sealed glass vials at 4 °C prior to analysis.

2.3. Chemical characterization of A. leucotrichus

2.3.1. Gas chromatography (GC) analysis

Quantitative analysis of the essential oil from *A. leucotrichus* fruits was performed using a Varian CP-3800 chromatograph equipped with a flame ionisation detector (FID) and a CP-Wax 52 CB bonded fused silica polar column (50 m × 0.25 mm, 0.2 µm film thickness) from Varian. The oven temperature was set at 50 °C for 5 min, increasing by 2 °C/min to 200 °C and finally held isothermal for 20 min. The injector and detector temperatures were 240 and 250 °C, respectively. Helium (He N60) was used as the carrier gas at a constant flow rate of 1 mL/min. Samples were diluted in *n*-hexane (1:1) and injected (0.1 µL) using a split ratio of 20:1. The percentage composition of the components was calculated by normalisation of the GC peak areas without response factors. Reproducibility was verified by analysing the sample three times.

2.3.2. Gas chromatography–mass spectrometry (GC–MS) analysis

The essential oil of *A. leucotrichus* was analyzed using a Varian CP-3800 coupled with a Varian Saturn 2000MS ion-trap mass spectrometer (MS), a CP-Wax 52 CB bonded fused silica polar column (50 m × 0.25 mm, 0.2 µm film thickness) from Varian and a Rxi®-5Sil MSf used silica low-polar column (30 m × 0.25 mm, 0.25 µm film thickness) from Restek, and a Varian MS Workstation 6.9 software. The injector was set at 240 °C and the samples (diluted in *n*-hexane (1:1)) were injected (0.1 µL) using a split ratio of 50:1, with helium (He N60) at a constant flow rate of 1 mL/min. The oven temperature program was initially set at 50 °C for 5 min, then raised up to 200 °C at a rate of 2 °C/min, and finally held isothermal for 20 min. All mass spectra were acquired in electron impact (EI) mode. The transfer line, manifold and trap temperatures were 171, 83 and 150 °C, respectively. The mass to charge ratio, *m/z*, ranged from 80 to 500, the emission current was 10 µA, and the maximum ionisation time was 0.025 s. Reproducibility was verified by analysing the sample three times. The components were identified according to their retention indices relative to C_8−C_{40} *n*-alkanes and mass spectra, which were compared with those of the NIST98 Spectral Library, the mass spectral database of Flavors and Fragrances of Natural and Synthetic Compounds 2 (FFNSC2) from Wiley, an in-house library (with > 200 pure reference chemicals) and literature data (Adams, 2001; Babushok et al., 2011; Abu Zarga et al., 2013).