Nat. Volatiles & Essent. Oils, 2018; 5(4): 35-38

NVEO
Natural Volatiles & Essential Oils

RESEARCH ARTICLE

Characterization of *Opuntia ficus-indica* (L.) Mill. fruit volatiles and antibacterial evaluation

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Abstract

Opuntia ficus-indica (L.) Mill. fruits known as prickly pear cactus are used in folk medicine as well as food. In the present study it was aimed to extract the volatile constituents of *O. ficus-indica* fruits by *n*-hexane. The volatiles were subsequently analyzed by GC-FID and GC-MS, where a total of 14 compounds were identified. The main components were characterized as hexadecanoic acid 39.4%, heptacosane 12.3%, and methyl linoleate 6.8%, respectively. Further antimicrobial evaulation using a broth microdilution assay (Clinical and Laboratory Standards Institute method) against human pathogenic *Staphylococcus aureus* ATCC 6538, *Enterococcus faecalis* ATCC 29212*, Escherichia coli* NRLL B-3008, and *Pseudomonas aeruginosa* ATCC 10145. The minimum non-reproductive concentrations were determined as MIC, where *S. aureus* showed the most potent inhibition by 500 μg/mL.

Keywords: *Opuntia ficus-indica*, volatile compounds, chemical characterization, Cactaceae

Introduction

The genus *Opuntia* L. is represented by more than 1500 species in the world and this genus is an important member of the Cactaceae family. It is distributed in the Mediterranean and Aegean Region and consumed as food due to its unique taste and its natural antioxidants (Lee et al., 2002; Kabas et al., 2006)). The fruits and stems are used traditionally in folk medicine against burns, wounds, edema, bronchial asthma, diabetes, and indigestion (Ahn, 1998). It is also reported that fruit and stems extracts exhibit anti-inflammatory, analgesic, hypoglycemic, anti-ulcer, and anti-allergic actions (Ibanoz et al., 1979; Trejo-Gonzales et al, 1996; Galati et al., 2001; Lee et al., 2002; Lee et al., 2000; Park et al., 1998; Park et al., 2001).

The aim of the present study was to elucidate the *in vitro* antimicrobial activity of *O. ficus-indica* fruit volatiles and aroma components. The phytochemical composition was analyzed by GC-FID and GC/MS after lipophylic extraction. To the best of our knowledge, this is the first report on the volatiles of *O. ficus-indica* fruits.

Materials and Methods

Chemicals and plant material

O. ficus-indica fruits were collected from Turunç, Marmaris (Date: 04.08.2017), which was indentified by Derya Çiçek Polat, voucher specimens were deposited at Ankara University Herbarium. Pinkish fruits were thinly cut and dried. Samples were powdered and extracted with *n*-hexane on a magnetic stirrer where 200 g sample, was extracted 2 x 200 mL, followed by filtration. The *n*-hexane removed by *vacuo* by using a rotary evaporator.

GC-MS analysis

The GC-MS analysis was carried out with an Agilent 5975 GC-MSD system. Innowax FSC column (60 m x 0.25 mm, 0.25 µm film thickness) was used with helium as carrier gas (0.8 mL/min). GC oven temperature was kept at 60°C for 10 min and programmed to 220°C at a rate of 4°C/min, and kept constant at 220°C for 10 min and then programmed to 240°C at a rate of 1°C/min. Split ratio was adjusted at 40:1. The injector temperature was set at 250°C. Mass spectra were recorded at 70 eV. Mass range was from *m/z* 35 to 450 (McLafferty, 1989).

GC analysis

The GC analysis was carried out using an Agilent 6890N GC system. FID detector temperature was 300°C. To obtain the same elution order with GC-MS, simultaneous auto-injection was done on a duplicate of the same column applying the same operational conditions. Relative percentage amounts of the separated compounds were calculated from FID chromatograms. The analysis results are given in Table I.

Identification of the volatile components were carried out by comparison of their relative retention times with those of authentic samples or by comparison of their relative retention index (RRI) to series of n-alkanes. Computer matching against commercial (Wiley GC/MS Library, MassFinder Software 4.0) (1,2) and *in-house* "Başer Library of Essential Oil Constituents" built up by genuine compounds and components of known oils.

Antimicrobial activity

The antimicrobial activity was determined using the broth microdilution assay following the methods described by the Clinical and Laboratory Standards Institute (CLSI, 2006) to determine the minimum inhibitory concentrations (MIC) against the human pathogenic standard strains; *Pseudomonas aeruginosa* ATCC 10145, *Escherichia coli* NRLL B-3008*, Enterococcus faecalis* ATCC 29212*, Escherichia coli* NRLL B-3008*,* and *Staphylococcus aureus* ATCC 6538. All strains were grown in Mueller Hinton Broth (MHB, Merck, Germany) at 37°C in aerobic conditions for 24 h and standardized to 1×10^8 CFU/mL using McFarland No: 0.5 in sterile saline (0.85%). Test samples stock solution was prepared in dimethylsulfoxide (DMSO) and serial dilutions were prepared for each sample, antibaterial evaluations were in triplicates and reported as mean in Table 2.

Results and Discussion

The phytochemical constituents of the *n*-hexane extract were analyzed using GC-FID and GC-MS which led to the identification of a total of fourteen different compounds. The main components characterized were hexadecanoic acid 39.4%, heptacosane 12.3%, methyl linoleat 6.8%, hexacosane 5.8%, tricosane 5.1%, methyl hexadecanoate 4.2%, camphor 2.8%, borneol 2.5%, verbenone 1.8%, pentacosane 1.7%, α-terpineol 1.1%, respectively. To the best of our knowledge, this is the first report on the volatiles of *O. ficus-indica n*-hexane extract.

Table 1. The Volatile Composition of *Opuntia ficus-indica n*-hexane extract

RRI	Compound	%	Identification Method
1203	1,8-cineole	1.0	tR, MS
1400	Camphor	2.8	tR, MS
1466	Linalool	0.8	MS
1495	Isopinocamphone	0.4	MS
1497	α-terpineol	1.1	tR, MS

1535	Borneol	2.5	MS
1553	Verbenone	1.8	tR, MS
1868	Methyl Hexadecanoate	4.2	MS
1933	Tricosane	5.1	tR, MS
1957	Pentacosane	1.7	MS
1958	Methyl linoleate	6.8	tR, MS
2037	Hexacosane	5.8	MS
2041	Heptacosane	12.3	MS
2050	Hexadecanoic acid	39.4	MS
	Total	**85.7**	

RRI Relative retention indices calculated against *n*-alkanes. % calculated from FID data. tr Trace (< 0.1 %). tR, identification based on the retention times (tR) of genuine standard compounds on the HP Innowax column; MS, tentatively identified on the basis of computer matching of the mass spectra with those of the Wiley and MassFinder libraries and comparison with literature data.

Table 2. Antimicrobial activity of *O. ficus-indica* fruit *n*-hexane extract (MICs in μg/mL)

Sample	*E. coli*	*S. aureus*	*P. aeruginosa*	*E. faecalis*
***n*-hexane extract**	>1000	500	>1000	>1000
Chloramphenicol	8	8	>32	16
Tetracycline	16	0.25	>16	0.025

O. ficus-indica fruit volatiles *(n*-hexane extract) against bacterial strains were listed, in Table 2. The results revealed that *n*-hexane extract is effective on *S. aureus* at 500 μg/mL. However, the evaluated extract showed no inhibitory activity on bacteria at the tested concentrations suggesting further detailed biological evaluations. To the best of our knowledge, this is the first report on the volatiles and antibacterial evaluation of *O. ficus-indica* fruits. Our studies on *Opuntia* sp. species are ongoing.

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