Research Article

Sicilian *Opuntia ficus-indica* seed oil: Fatty acid composition and bio-economical aspects

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The fatty acid composition of the seed oil from the yellow fruit of *Opuntia ficus-indica* widely grown in Sicily shows several distinctive features. The oil obtained comprises significant amounts of vaccenic acid along with several other unsaturated fatty acids showing several health benefits, including linolenic, *trans*-13-octadecenoic, gondoic, 7Z,10Z-hexadecadienoic, and gadoleic acid. The economic analysis shows the significant advantage of carrying out the extraction from fruits considered unfit for consumption.

Practical applications: The fatty acid composition of Sicilian *Opuntia ficus-indica* seed oil is similar to that of fruits grown in Tunisia, while it has a completely different profile than the OFI fruits grown in Algeria and Morocco. Like the oil obtained in Tunisia, the Sicilian oil has a higher vaccenic acid content, but it comprises significant amounts of other unsaturated fatty acids showing highly beneficial health properties. Extracted from fruits considered unfit for consumption, its production generates €1220 additional revenues per tonne of unfit fruit thereby significantly improving the economics of OFI processing companies. The oil has several potential applications which go beyond cosmetics, especially as nutraceutical ingredient. With increasing usage and market expansion, we predict that cold press extraction will be replaced by advanced extraction methods such as extraction with supercritical CO_2 maximizing for example the biophenol amount in the oil.

Keywords: Cactus pear / Linoleic acid / *Opuntia ficus-indica* / Seed oil / Sicily

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1 Introduction

Mostly comprised of polyunsaturated fatty acid triglycerides [1], the *Opuntia ficus-indica* (OFI) seed oil is a good source of edible oil for human consumption [2]. The oil, furthermore, has a very high content of vitamin E (>600 mg/kg) and biophenols [3] (often in glycoside form) which imparts this valued bioproduct with antioxidant properties. The presence of β-sitosterol, in turn, induces remarkable anti-inflammatory activity [4]. Applied on the skin of the face, neck, and eye area, the OFI oil acts as an excellent anti-aging moisturizer and nourishes the skin cells [5], whereas when applied in small amounts onto impoverished hair, it promotes keratin regeneration. The nutritive value of OFI seeds and oil obtained in Turkey was lately confirmed with seeds being an important source of natural fiber and the oil, with its high (61%) linoleic acid content, recommended for use as a nutraceutical agent [6]. We have lately argued accordingly, that the oil will shortly be

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Abbreviations: MUFAs, monounsaturated fatty acids; **OFI,** Opuntia ficus-indica; **PFTBA,** perfluorotributylamine; **SFAs,** saturated fatty acids

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used not only in cosmetic products but also in food supplementation and nutraceutical products, in a typical case of innovation guided by the past, which has been a constant driving force of chemistry [7].

Sicily, with a production of about 87 000 tonnes from 8300 hectares [8], hosts most of Italy's *Opuntia* cultivation. Several companies use the fruits to produce jams and juices, after having removed the seeds that are usually disposed of as animal feed. In general, whereas significant differences are observed for biophenols content among different harvesting years [9], the fatty acid composition does not significantly change with harvesting years [10]. In 2013, a study addressing the composition of the oil obtained from the skin, pulp, and seeds of Sicilian cactus pears, identified 20 compounds in the oil from the seeds of red fruits and 16 compounds in the oil from yellow fruits [11]. However, the fatty acid composition was not further investigated. Now, we report the outcomes of such investigation. The results point to remarkable differences with similar fruits grown in semi-arid areas in other Mediterranean regions.

2 Experimental section

2.1 Seed oil extraction

OFI seed oil was extracted from the seeds via cold press using a proprietary hydraulic press unit is use at an OFI processing company based in Sicily (Bienesse, Italy) which commercializes also the oil. The oil was obtained from the seeds of yellow fruits (Surfarina cultivar) of ideal maturation degree harvested in Biancavilla, Sicily, at 400 m above the sea level over volcanic land. The seeds were cleaned with water only and left to dry at room temperature. No preservatives were added to the oil, which was immediately stored in small glass vials colored in brown to limit photooxidation in which it is usually commercialized.

2.2 Fatty acid analysis by GC-MS

The fatty acids composition of the oil was determined via GC-MS. The qualitative and quantitative assessment of fatty acids was carried out according to the official European Commission Regulation EEC methodology (2568/91) of July 11, 1991, on the characteristics of olive oil and olive-residue oil and on the relevant methods of analysis.

2.2.1 Methyl ester synthesis

The methyl esters of fatty acids (FAMEs) were obtained from the triglycerides by transesterification reaction treating 0.10 g of oil with 0.2 mL of a 2N KOH solution in MeOH. The resulting FAME solution was extracted with 2 mL of *n*-hexane. This fraction was then diluted 1:20 with *n*-hexane, before the GC-MS analysis.

2.2.2 GC-MS analysis

A 1 mL sample of the FAME solution was injected (split mode 1/100) into a ZB-5MS column (Zebron, 30 m $\times$ 0.25 mm id $\times$ 0.25 μm) installed on a Trace-ISQ GC/MS (q) equipped with an autosampler Triplus RSH Thermo Scientific, using He (5.0) as carrier gas at a flow-rate of 0.95 mL min^{-1}. The temperature ramp used was follows: After holding the column at $T = 165°C$ 10 min, the temperature was raised at 1.5°C/min rate up to 200°C, followed by an increment of 10°C/min up to 250°C, keeping the column at this temperature for 20 min. The overall time for the analysis was 37 min. The injector and transfer line temperature were 270°C and 265°C, respectively. The spectrometer was automatically tuned using perfluorotributylamine (PFTBA) according to manufacturer instructions. Following automatic tuning, the electron multiplier voltage was set at 70 eV EI (+). Full scan data were acquired over the range $m/z = 35$–500 at 0.73 s per scan, keeping the ion source at 260°C. The retention times and molecular fragment mass data obtained were processed with the instrument's software. Each measurement was repeated three times. Both chromatographic and spectrometric results showed excellent reproducibility (SD 4%). All FAME compounds were first identified by critical comparison with mass spectral data from NIST/EPA/NIH Mass Spectral Library 2005.

3 Results and discussion

Table 1 shows that the cactus pear seed oil from the yellow fruit grown in eastern Sicily (Biancavilla) is mainly (83.79%) composed of unsaturated fatty acids including polyunsaturated fatty acid (PUFAs), that is, linoleic acid, monounsaturated fatty acids (MUFAs), mostly oleic acid, and vaccenic acid, with a lower but significant fraction (16.31%) of saturated fatty acids (SFAs) mostly comprised of palmitic acid (12.29%) and stearic (3.92%) acid. Overall, 13 different fatty acids were identified and assessed (Table 1).

For comparison, Table 2 reports the fatty acid composition of the OFI oils obtained in Tunisia [10], Morocco [12], and Algeria [13], namely North African countries south of Sicily and Saudi Arabia where scholars in 1982 reported the first fatty acid composition of OFI essential oil [14].

Confirming previous results from OFI oil from geographically different Mediterranean countries and regions, the ω-6 polyunsaturated fatty acid, linoleic acid is the dominating fatty acid. However, contrary to what was observed in OFI seed oil from Algeria, in which vaccenic acid was only present in the green and orange varieties, and no vaccenic acid was found in the seed oils from yellow and red fruits [13], we observed a significant amount of vaccenic acid (6.29%) in the Sicilian oil obtained from the seeds of yellow fruits.

Scholars in Saudi Arabia could not identify neither linolenic acid nor fatty acids of chain length greater than C18

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Table 1. Fatty acid composition of yellow *Opuntia ficus-indica* seed oil grown in Sicily

	Lipid composition	Concentration (%)
	Myristic acid 14:0	0.10
	Palmitic acid 16:0	12.29
	Palmitoleic acid 16:1 (9Z)	0.75
	7Z,10Z-Hexadecadienoic acid, 16:2 (6Z,9Z)	0.05
	Stearic acid 18:0	3.92
Fatty acid	Oleic acid 18:1 (9Z)	17.61
	Vaccenic acid 18:1 (11E)	6.29
	13-Octadecanoic acid 18:1 (13E)	0.17
	Linoleic acid 18:2 (9Z,12Z)	57.98
	Linolenic acid 18:3 (9Z,12Z,15Z)	0.21
	Arachidic acid 20:0	0.33
	Gondoic acid 20:1 (11Z)	0.10
	Methyl-9-eicosenoate	0.22

in the oil extracted with ether from previously ground seeds [14]. Similarly, researchers in Morocco did not detect linolenic and oleic acids in the Soxhlet-extracted oil from the seeds with *n*-hexane, attributing the absence of monounsaturated fatty acids to the nature of the fruits harvested in the early phase of fruit maturation process [12].

The oil Soxhlet-extracted with petroleum ether from the ground seeds of OFI fruits harvested in Tunisia had a similar fatty acid composition to the oil extracted via cold pressing in Sicily. The amount of linolenic acid, an ω-3 fatty acid important in the secondary prevention of coronary heart disease and in the prevention of cancer [15], is twice higher than the amount found in Sicily (0.45% vs. 0.21%). However, the content of vaccenic acid is higher in the Sicilian bio-oil (6.29% vs. 4.83%). The latter fatty acid is the main *trans* fatty acid isomer present in milk fat and in human milk where it is the predominant component of the *trans* fatty acid part, which in the body is converted into a conjugated linoleic acid showing potent anticarcinogenic activity [16].

Similar to the Tunisian bio-oil, the Sicilian oil comprises appreciable levels of two eicosenoic acids, namely gondoic acid (0.10%) and methyl-9-eicosanoic acid (0.22%, in methyl ester form). The former is a monounsaturated omega-9 long chain fatty acid also found in jojoba oil [17]. The latter is the methyl ester of gadoleic (or 9-eicosenoic) acid, namely a monounsaturated omega-11 long chain fatty acid occurring in triglyceride form in whale, shark, cod, anchovy, mackerel, and herring oil [18], as well as in pomegranate, jojoba, borage, and peanut oils, having a remarkable negative correlation with immunoglobulin E (IgE) levels in cord blood of 35 children who subsequently developed allergic sensitization and atopic dermatitis [19].

7Z,10Z-hexadecadienoic acid, a conjugated dienoic fatty acid metabolite of conjugated linoleic acid, with potential beneficial effects on atherosclerosis, carcinogenesis, and obesity in humans [20], was detected in a small, yet appreciable level (0.05%). Finally, the *trans* isomer of 13-octadecanoic acid (13*t*-18:1), a C18 acid, well adsorbed by human plasma found in several medicinal plants [21], was present in the Sicilian OFI oil at 0.17% level.

The saturated fatty acids, myristic (0.10%), and arachidic acid (0.33%), but mostly palmitic acid (12.29%) and stearic acid (3.92%), that significantly correlated with the oxidative-stability index [22], were present in levels similar to those found in the OFI oil from Tunisia. The amount of palmitoleic acid was also very similar to that in the Tunisian oil (0.75% vs. 0.65%). This fatty acid behaves in the human body behaves like a saturated (and not a monounsaturated) fatty acid [23].

Table 2. Fatty acids composition of cactus pear seed oil grown in Sicily (present study) and in Saudi Arabia [14], Morocco [12], Algeria [13], and Tunisia [10]

Sicily (yellow fruit)	Algeria (yellow fruit)	Saudi Arabia	Morocco	Tunisia
Myristic acid (0.10%)	Stearic acid (3.6%) palmitic acid (13.4%)	Stearic acid (5.8%) palmitic acid (12%)	Stearic acid (1.50%) palmitic acid (11.18%)	Myristic acid (0.14%)
Palmitic acid (12.29%)				Palmitic acid (12.24%)
Stearic acid (3.92%)				Stearic acid (3.69%)
Oleic acid (17.61%)	Oleic acid (20.9%)	Oleic acid (8%)		Oleic acid (20.19%)
Linoleic acid (57.98%)	Linoleic acid (62.1%)	Linoleic acid (73.4%)	Linoleic acid (58.79%)	Linoleic acid (56.63%) palmitoleic acid (0.65%)
Vaccenic acid (6.29%)	Vaccenic acid (0.0%)			Vaccenic acid (4.83%)
Arachidic acid (0.33%)				Arachidic acid (0.36%)
Linolenic acid (0.21%)				Linolenic acid (0.45%)
gondoic acid (0.10%)				
Methyl-9-eicosenoate (0.22%)				Gondoic acid (0.24%)

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4 Economic aspects

Until a few decades ago, the *Opuntia ficus-indica* was used in Sicily only as a source of animal feed and to erect natural fences between relatively small land portions which are characteristic of agriculture in Sicily since the mid 1950s. Starting in the early 1990s, new entrepreneurial efforts were deployed aimed at the valorization of the fresh fruit for human consumption. Since then, Sicilian producers recorded small yearly increments without particular marketing strategies [24]. More recently, new production efforts are aimed to exploit the value the whole OFI plant in a bioeconomy perspective in that what was previously considered waste, now becomes a source of value due to new usages and applications. For example, along with the oil from the seeds, a nutraceutical sweet juice with antioxidant and anti-inflammatory properties due to the high indicaxanthin [25] concentration can be obtained from yellow fruits considered unfit for consumption. This gives an economic value of €0.2–0.5/kg to the previously discarded fruit and enables a conversion that would not be economically feasible using fruits of good aspect traded at twice the above price.

The costs of the fruit juice production, seed separation and oil extraction (energy, workforce, plant and equipment management, and amortization, etc.) amounting to €0.10–0.15/kg add to the cost of the raw material.

The first production line, starting from the fruit pulp produces a concentrated juice of 50–58 °B×, following centrifugation, pasteurization, concentration, and packaging. The overall yield of the juice is around 7%–8%, thus from 1000 kg of fruit, about 80 kg of concentrated juice are obtained. The juice is currently commercialized at €4/L.

In the second production line, the seeds isolated from the washed and peeled fruits via mechanical sieving, are further washed with water and left to dry at room temperature and atmospheric pressure. The clean dried seeds are then cold pressed with a hydraulic press to extract the oil under high mechanical pressure. The oil is separated from the cellulosic solid residue via simple filtration, and collected prior to packaging in 5 L bottles for industrial customers, and 10 mL vials for the pharmaceutical clientele. Managed by a dedicated worker, a typical cold extractor such as the one used in Sicily to extract the present OFI oil is able to extract 2 L of oil in 24 h.

Facing €600 overall costs to process 1000 kg of cactus pear fruit unfit for commercialization as fresh fruit, €320 of revenues originate from selling the concentrated juice and €1500 from selling the seed oil, mainly to pharmacies which further sell the OFI oil as an improved version of the Argan oil widely used in cosmetics for its restorative and antiaging properties for hair, skin and nails [26]. It is therefore clear why the bioeconomy approach to valorize all fruits and all fruit components is of crucial relevance to OFI processing companies.

5 Conclusions

The fatty acid composition of the seed oil from the yellow fruit of *Opuntia ficus-indica* Surfarina cultivar extensively grown in Sicily shows several distinctive features. The oil has a profile similar to that of similar fruits grown in Tunisia, while it has a completely different profile than the OFI fruits grown in Algeria and Morocco. Like the oil obtained in Tunisia, the Sicilian oil has a higher vaccenic acid content. Yet, the oil obtained in Sicily comprises significant amounts of other unsaturated fatty acids showing highly beneficial health properties, including *trans*-13-octadecenoic, 7Z,10Z-hexadecadienoic, and gadoleic acid (the latter in ester form).

The economical analysis showed the significant advantages offered by the oil (and juice) extraction from fruits considered unfit for consumption. Indeed, one tonne of fruit generates €1220 net revenues for the OFI processing company, translating into additional value for the fruit suppliers now selling a product previously dealt with as agricultural waste, mostly ending landfilled at significant economic cost.

The extraction method relies on an entirely green process, namely cold pressing carried out at industrial premises where the juice is extracted from the pulp and the oil from the seeds of fruits. Predicting further progress, we anticipate that instead of cold pressing, new extraction physical techniques will be applied and optimized to extract the oil in shorter time than in cold pressing, while still affording no solvent waste and toxic chemical residues. In fact, the first results of *Opuntia* seed oil extraction via alternative methods showed that the extraction with supercritical CO_2 affords an oil with >2.25 times higher phenolics content than oil extracted with *n*-hexane [27], whereas ultrasound-induced cavitation affords lower yields of an oil of lower antioxidant activity when compared to Soxleth-extracted oil with *n*-hexane [28]. In any case, the simplicity of the extraction, the growing extension of *Opuntia* cultivation (owing to its high water utilization efficiency, the plant provides an excellent feed source for livestock and effectively combats desertification in arid areas) [29], as well as the economic and environmental advantages of the bioeconomy approach described in this study, all support rapid increase in the availability and utilization of cactus-seed oil not only in cosmetics, but also in nutraceuticals, including sport and energy drinks [30], in all main *Opuntia* growing countries.

In conclusion, looking back at a seminal paper published in 2001 [31], Carle et al. concluded that “to improve the nutritional and economic feasibility of cactus pear,” besides nutritional and clinical investigations, an increase in the worldwide production for price reduction, breeding for a higher pulp yield, and optimization of harvest procedures, were necessary. In the subsequent fifteen years, the worldwide production has indeed increased, and cultivation procedures have improved with the use of drip irrigation and the expansion of organic farming [32]. The same scholars

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also clearly anticipated that "the trend towards natural ingredients and products promoting health and well-being" was likely to increase.

Fifteen years later, an unexpected bioeconomy approach has clearly emerged owing to which all fruits and all fruit components that are actually used to produce valued bioproducts in high and increasing demand, such as the *Opuntia* fruit peel rich in pectin of superior quality [33], driven by the very same societal trend toward health-beneficial natural products replacing synthetic functional substances. Hopefully, this study will contribute to further advance the bioeconomy of *Opuntia* in all countries where this important plant is cultivated.

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The authors have declared no conflict of interest.

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