An analysis of the similarities in the ATR-FTIR spectra from *Argania spinosa, Rosa rubiginosa* and *Elaeis guineensis* oils

Abstract

The attenuated total reflection Fourier transform infrared spectroscopy (ATR-FTIR) spectra of the essential oil from *Rosa rubiginosa* L. seeds and the vegetable oils from *Argania spinosa* L. kernel and *Elaeis guineensis* Jacq. pulp show important similitudes that hamper their identification by vibrational spectroscopy techniques if they are not complemented with well-established methods such as gas chromatography. Nevertheless, the observed similarities in structure-composition-traditional uses between *Argania spinosa* and *Rosa rubiginosa* oils suggest that they could be interchangeable when skin physicians, dermatologic-surgeons or cosmetologists perceive in their practice that one of the oils produces an allergic reaction or other side effects, although further activity studies are needed.

Keywords: argan; ATR-FTIR; African oil palm; oils; sweet briar.

1. Introduction

Infrared spectroscopy may be defined as the triggering of vibrations in molecules of a sample through irradiation with infrared light, with the aim of producing a fingerprint of the sample with absorption peaks that correspond to bonds between the atoms that make up the molecule. The use of FTIR spectroscopy has been deemed suitable for verifying the presence of functional groups or for structure elucidation. Moreover, when coupled with an ATR accessory, this technique shortens the time of sample preparation and eases spectral reproducibility, thus making sample analysis even faster. ATR-FTIR is used in vegetal oil chemistry as a rapid quantitative tool to determine the main components (i.e., saturated, *trans*, mono- and polyunsaturated fatty acids)

present in different edible or cosmetic oils. In spite of having some limitations in such particular application, this technique proves to be useful to show composition similitudes.

Back in 2014, Yildirim and Kostem (2014) reported a comparative study of 10 cosmetic oils including argan, almond, apricot seed, jojoba, wheat germ, sesame seed, avocado, cocoa, carrot and grapes seed oils. At around the same time, our working group initiated a study of oil-bearing seeds and oils from macauba palm, rosehip, rock rose, heather, chia, black seed and opium poppy (Carrión-Prieto, et al., 2017; Carrion-Prieto, et al., 2017; del Río, et al., 2016). The analysis of the ATR-FTIR features of these oils led us to observe a very high similarity between those from argan kernel, rosehip seeds and palm pulp, a correspondence that had –to the best of our knowledge– not been reviewed in the literature. We herein present a comparative study of the ATR-FTIR spectra of these three oils (taking gas chromatography data as a reference), with the aim of establishing structure-composition-properties that could justify *(i)* the similitudes observed and *(ii)* the presumable interchangeability of argan and rosehip oils in some common applications in Cosmetics and Dermatology.

Argan oil is produced from the kernels of the argan tree (*Argania spinosa* L. or *Argania sideroxylon* Roem. & Schult.), which is endemic to SW of Morocco and to the province of Tinduf in Algeria. The fruits of the argan tree are round and small, with a thick peel that covers the fleshy pulp, which in turn surrounds a hard-shelled nut. The nut, which contains several (one to three) argan-oil rich kernels, accounts for ca. 25% of the fresh fruit weight. The oil extraction yield from these kernels ranges from 30% to 50%, depending on the extraction procedure (Charrouf and Guillaume, 1999). Argan oil from non-roasted kernels is used for burns healing, to treat skin diseases and as a cosmetic resource against *acne vulgaris* or scaly, dry skin (El Babili, et al., 2010). The increasing popularity of argan oil has prompted the Moroccan government to plan for increased production, aiming to reach 4,000 tons by 2020. On November 27, 2014, UNESCO designated as Intangible Cultural Heritage the knowledge, skills and practices related to argan.

As regards *Rosa rubiginosa* L. or *R. eglanteria* L., it is a shrub from Great Britain that is frequent in Southern Alps and that grows massively in South America and in the temperate

regions of Australia (Hatton, 1989). It is commonly known in South America as ‘rosa mosqueta’ and in Australia as ‘sweet briar’. This member of the rose family has pink flowers which only live about 24 hours. When petals fall, the shrub develops enlarged floral cups (receptacles) that surround numerous small, hard dry fruits (achenes) commonly called ‘seeds’. Rose hips are bright orange and oval and become fleshy, but are not true fruits (Joublan, et al., 1996). The traditional rosehip seed vegetable oil is derived from cold pressing of hips, but Soxhlet extraction is nowadays the usual procedure (yielding an essential oil). *R. rubiginosa* oil has been traditionally used by local people for its soothing and moisturizing properties and is now used at industrial scale for its applications in skin care products. It has also been reported to be useful for the healing of open wounds (Marchini, et al., 1987; Moreno, et al., 1989).

On the other hand, palm oil is an edible vegetable oil derived from the fibrous flesh mesocarp (reddish pulp) of the fruit of the oil palms, primarily from the African oil palm *Elaeis guineensis* Jacq., by hot-squeezing. Palm oil is naturally reddish in colour because of its high beta-carotene content. This oil, whose content per fruit ranges between 40% and 70%, is one of the few highly saturated vegetable fats and is semi-solid at room temperature. Palm pulp oil is mostly employed in the making of soaps and cosmetics (Reeves and Weihrauch, 1979).

2. Experimental

There are two procedures for the extraction of *Argania* vegetable oil: either using the traditional method, in which roasted kernels are crushed and then kneaded into paste or dough with hot water and finally pressed by hand, or the half-industrialized or semiautomatic method, which is carried out by mechanical cold-pressing without water addition. The oil produced using the former method includes less linoleic and oleic acids than the oil produced using the later procedure. The *Argania* kernel vegetable oil used in this study was a virgin oil obtained from the first mechanical cold-pressing and it was supplied by Esential'arôms (Lleida, Spain). Routine GC/MS analyses of the oil were conducted *in-situ* to confirm its identity. Its composition is

reported in Table 1, in good agreement with the data reported by other authors (El Abbassi, et al., 2014; Khallouki, et al., 2003; Marfil, et al., 2011).

High-purity commercial *Rosa rubiginosa* seed essential oil, obtained by Soxhlet extraction, was supplied by Bariloche Silvestre (Provincia de Rio Negro, Argentina). The composition of this commercial oil, confirmed by GC/MS, is also presented in Table 1, and was in agreement with the characterization carried out for rosehip oils by Concha, et al. (2006), Pareja and Kehl (1990) and other authors (Adamczak, et al., 2012; Hornero-Mendez and Minguez-Mosquera, 2000; Martín-Ramos, et al., 2016).

High-purity *Elaeis guineensis* palm vegetable oil was supplied by Cia Refinadora da Amazônia (Belém, Pará, Brazil), part of Agropalma group. Its composition, confirmed *in-situ* by routine GC/MC analysis, is reported in Table 1.

A. spinosa, *R. rubiginosa* and *E. guineensis* oil FFA content was determined by AOAC method (protocol 940.28) using a chromatograph (Hewlett-Packard 5890 series II, Palo Alto, CA, USA) equipped with an FID and fitted with an SP-2330 column. After methylation, a hexane extract containing FAME was injected into the GC column. FAME were separated using a stated temperature program and its weights calculated on the basis of their relative area *vs*. tridecanoic acid.

The vibrational spectra of the materials in the 400-4000 cm^{-1} range were measured using a Thermo Scientific (Waltham, MA, USA) Nicolet iS50 FT-IR Spectrometer, equipped with an in-built diamond ATR system. The infrared spectra were collected with 2 cm^{-1} spectral resolution and 64 scans. The relative intensity shift introduced by the ATR technique was corrected using the Advanced ATR Correction Algorithm implemented in OMNIC software (Nunn and Nishikida, 2008). Measurements were conducted with a constant contact area, so that the peak intensity was proportional to concentration. Calibration curves (not shown) with excellent linearity were obtained, as it is usually the case for liquid samples.

3. Results

As it may be observed in Table 1, the composition of the three oils was very similar in terms of components and differed only in their relative proportions. With regard to the fatty acids, the percentages of polyunsaturated fatty acids (PUFA), monounsaturated fatty acids (MUFA) and saturated fatty acids (SFA) were more similar in the argan and rosehip oils that in the palm pulp oil. Among the families of chemical species, the most discrepant was that of carotenoids: *A. spinosa* was rich in xanthophylls; *E. guineensis*, in carotenes; and *R. rubiginosa* showed a mixed composition. Moreover, the concentration of carotenes and terpenes (mg/kg or ppm DM) of *A. spinosa* was five or six times higher than those of the other two species under study. On the other hand, *R. rubiginosa* had noticeably higher phenolic acids and sterols contents than the two other oils.

Nonetheless, above differences were barely reflected in the positions and intensities of the bands in the ATR-FTIR spectra (see Figure 1), due to the close structural relationship within a same family (the same family members share identical functional groups) and to the fact that the greatest differences would occur for components whose concentration was not representative.

From the analysis of the intensities of the peaks of the spectra, the main difference was observed in the intensity of the peak at 966 cm^{-1}, which was higher for *R. rubiginosa* oil than for the other two oils. Although this absorption is usually attributed to =CH out-of-plane deformation (Table 2), Yildirim and Kostem (2014) related it to carboxylic acids. In support of this later observation, it is worth noting that rosehip oil was, out of the three oils under study, the one with the highest content of free fatty acids (92%). On the other hand, differences in the relative intensity of the bands at 2920 cm^{-1} and 1740 cm^{-1} may be referred to the relative percentages of PUFA+MUFA *vs*. SFA in the three oils: the peak at 2920 cm^{-1} showed greater intensity than the one at 1740 cm^{-1} for *R. rubiginosa* and *A. spinosa* oils, whereas for *E. guineensis* it was just the opposite. The A_{2920}/A_{1740} ratios are reported in Table 3.

4. Discussion

The ATR-FTIR spectra of *Argania spinosa* L. kernel, *Rosa rubiginosa* L. seeds and *Elaeis guineensis* Jacq. pulp oils show important similitudes which evince the presence of dermatologically active molecules (viz. MUFA, PUFA, SFA, α-tocopherol, terpenes, phenols, flavonoids/vitamin C, carotenoids (xanthophylls and carotenes), trans-retinoic acid and sterols), much of them recognized by their antioxidant activity (Başer and Buchbauer, 2010; Darvin, et al., 2011; Edris, 2007). These composition similarities are particularly relevant in the case of argan oil and rosehip essential oil, in agreement with their analogous applications in Cosmetics and Dermatology.

In spite of the fact that the content of the PUFA (linoleic and linolenic acids) in the composition of argan oil is half of that of rosehip seed oil, it appears to be sufficient to ensure an active behavior in tissue regeneration (Guillaume and Charrouf, 2011). Besides, although rosehip oil is one of the most powerful skin regenerators known (and hence surgeons are increasingly employing it to treat the skin after surgery or in cases of burns, scars and stretch marks), the fact that Moroccans have also traditionally used the un-roasted argan oil to heal burns should not be obviated. In short, both oils would be suitable choices so as to help repair damaged first- and second-degree skin burns, those caused by the sun and even radiation burns.

It has been proven that vitamin A helps rejuvenate the skin by increasing cell renewal. The concentrations of *trans* retinoic acid in rosehip essential oil have been shown to reduce signs of aging as well as to reduce scar tissue. These benefits are also shared by argan oil, which -amongst its properties- features revitalizing cell effects that inhibit early skin aging. It is also worth noting that the high *trans* retinoic acid levels in argan oil have a sebum regulating action not referred for 'rosa mosqueta'.

Thanks to the high amounts of flavonoids and retinoic acid in argan oil and rosehip essential oil, both would be equally applicable in order to build and strengthen body tissues and would also be beneficial to the building and maintaining of a good blood vascular system (preventing and healing of fragile capillaries, bedsores, wrinkles and any related dermatological condition).

On the basis of vitamin E content, argan oil has been shown to help reduce wrinkles, soften the skin and increase elasticity, so rosehip essential oil may also fulfill the same function in an effective manner.

5. Conclusions

Although the analysis of the ATR-FTIR spectra from *Rosa rubiginosa* essential oil and *Argania spinosa* and *Elaeis guineensis* vegetable oils shows some limitations, it allows to obtain valuable information on dermatologically active molecules present in this triad, such as fenols, vitamin E and carotenoids. Their accumulation in human skin by topical application in the form of oils, creams and lotions is known to neutralize the attacks of free radicals, especially reactive oxygen species. The study on the relative concentrations of such active molecules in these oils has allowed to observe important similarities between *Argania spinosa* oil and *Rosa rubiginosa* essential oil. Such similarities could tentatively lead to replace one with the other in some Dermatology and Cosmetics applications when allergy or other side effects occur for one of them (although data of animal trials would be needed to confirm this claim).

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Figure legends

Figure 1. ATR-FTIR spectra of *A. spinosa* kernel (*solid line*), *R. rubiginosa* seed (*dashed line*) and *E. guineensis* pulp (*dotted line*) oils. Intensities have been normalized for comparison purposes.

Table headings

Table 1. Composition of the *A. spinosa* kernel oil, *R. rubiginosa* seed oil and *E. guineensis* pulp oil.

Table 2. Main bands in the ATR-FTIR spectra of *A. spinosa* kernel oil, *R. rubiginosa* seed oil and *E. guineensis* pulp oil (wavenumbers, in cm^{-1}) and their assignments.

Table 3. PUFA+MUFA to SFA ratios and intensity of the absorption band at 2920 cm^{-1} to intensity of the band at 1740 cm^{-1} ratios for the three oils under study.

Table 1. Composition of the *A. spinosa* kernel oil, *R. rubiginosa* seed oil and *E. guineensis* pulp oil.

Composition	*A. spinosa* kernel oil (Guillaume and Charrouf, 2011; Khallouki, et al., 2003; Marfil, et al., 2011; Reeves and Weihrauch, 1979)	*R. rubiginosa* seed oil (Adamczak, et al., 2012; Concha, et al., 2006; El Abbassi, et al., 2014; Grajzer, et al., 2015; Hornero-Mendez and Minguez-Mosquera, 2000; Martín-Ramos, et al., 2016; Pareja and Kehl, 1990)	*E. guineensis* pulp oil (Jacobsberg, 1974; Rukmini, 1994; Sambanthamurthi, et al., 2011)
MUFA and PUFA	79%	91%	47%
oleic acid, C18:1 n-9	45% (42-47.5%)	15% (10-20%)	40% (37-52%)
linoleic acid, C18:2 n-6	34% (31-37%)	47% (41-52%)	7% (5-9%)
linolenic acid, C18:3 n-3	0.3% (0.1-0.5%)	29% (21-37%)	
SFA	18.5%	6%	48%
palmitic acid, C16:O	13.5% (12-14%)	4% (3-5%)	43.5% (32-47%)
estearic acid, C18:O	5.5% (5-6%)	2% (1-3%)	4.3% (3.7-4.7%)
α-tocopherol/vitamin E (lipophilic antioxidants)	637-750 ppm	700-1100 ppm	500-800 ppm
Terpenes (recognized for their antiseptic and anti-viral activities)	squalene, 1,10-di-epi-cubenol, viridiflorol, cadinol and camphor	squalene (poor)	squalene
	1500-3200 ppm	<200 ppm	200-500 ppm
Phenols (responsible for antioxidant properties)	vanillic, ferulic and syringic acids, hydroxybenzoic acids, vanillin, oleuropein, cafeic acid, tyrosol, catechol, resorcinol, epicatechin and catechin	p-hydroxybenzoic acid, trihydroxybenzoic acid (gallic acid), vanillic acid, vanillin, ferulic acid, epicatechin, gallocatechin and p-coumaric	p-hydroxybenzoic acid, protocatechuic acid, vanillic, caffeic, syringic, p-coumaric and ferulic acids, caffeoylshikimic acid and hydroxytyrosol
	57 ppm	26-780 ppm	55-376 ppm
Flavonoids/vitamin C	myricetin and quercetin	glycoside derivatives of quercetin	
Carotenoids (xanthophylls and carotenes)	xanthophylls (β-cryptoxanthin and zeaxanthin) but no carotens	rubixanthin, gazaniaxanthin, β-cryptoxanthin, zeaxanthin, β-carotene and lycopene	α-carotene, β-carotene, and lycopene

		400-800 ppm	500-700 ppm
	3000 ppm		
trans-retinoic acid / pro-vitamin A (used to treat acne)	>400 ppm	357 ppm	>150 ppm
Sterols	spinasterol and schottenol	β-sitosterol and cycloarterol	
	1600 ppm	6000 ppm	326-527 ppm

SFA = saturated fatty acids; MUFA = monounsaturated fatty acids; PUFA = polyunsaturated fatty acids

Table 2. Main bands in the ATR-FTIR spectra of *A. spinosa* kernel oil, *R. rubiginosa* seed oil and *E. guineensis* pulp oil (wavenumbers, in cm^{-1}) and their assignments.

A. spinosa kernel oil	*R. rubiginosa* seed oil	*E. guineensis* pulp oil*	Assignments
3007	3009	3006	=CH stretching
2922	2923	2919	CH stretching from $–CH_2$
2853	2853	2851	CH stretching from $–CH_2$
1743	1742	1742	OC=O ester carbonyl of triglycerides
1653	1653	1648	C=C stretching of *cis* disubstituted olefins; olefinic terpenes
1464	1456	1456	CH asymmetrical bending of $–CH_3$
1418	1418	1417	CH in plane deformation of CH_2=CH- O-H in plane deformation
1399	1397	-	CH_2 in plane deformation
1377	1377	1377	CH symmetrical bending from $–CH_3$
1236	1238	1243	$-C(CH_3)_3$ skeletal; O-H in plane
1159	1160	1160	C-CH_3 of trans –CH-C(CH3) C=O stretching methyl ester
1118	-	1115	C-C stretching
1097	1098	1099	starch OH, cellulose
1032	1029	1031	ring resonance
962	966	961	CH_2 wagging of *trans* -CH=CH-
912	913	919	CH_2 wagging of CH_2-CH- unit
875	868	868	δ(C-O-C)
845	-	-	CH_2 wagging of trans CH=C(CH3)- CH wagging in R_1R_2C=CHR isopropylidine
722	723	718	$\rho(CH_2)_n$ skeletal
603	609	603	

540	-	544
513	511	514
491	493	488

* unrefined

Table 3. PUFA+MUFA to SFA ratios and intensity of the absorption band at 2920 cm^{-1} to intensity of the band at 1740 cm^{-1} ratios for the three oils under study.

	R. rubiginosa seed oil	*A. spinosa* kernel oil	*E. guineensis* pulp oil
PUFA+MUFA/SFA	15	4	1
A_{2920}/A_{1740}	1.5	1.2	0.87

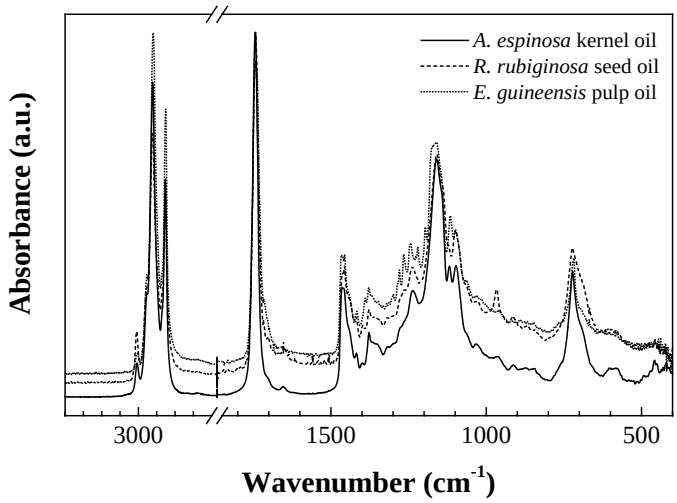

Figure 1. ATR-FTIR spectra of *A. spinosa* kernel oil (*solid line*), *R. rubiginosa* seed oil (*dashed line*) and *E. guineensis* pulp oil (*dotted line*). Intensities have been normalized for comparison purposes.