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Effect of extraction process on quality of oil from *Asphodelus tenuifolius* seeds☆

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Abstract – In this study, *Asphodelus tenuifolius* (ATO) seeds oils were extracted by three different extraction methods, namely Soxhlet extraction (SE), microwave-assisted extraction (MAE) and ultrasonic-assisted extraction (UAE). The physicochemical properties, fatty acids, sterols, tocols, total phenol content and antioxidant activity of ATO seed were examined and compared. The best extraction yield was from SE (21.97 ± 0.01%), followed by MAE (19.28 ± 0.13%) while that of UAE was the lowest (16.5 ± 0.1%). Analysis of the chemical composition of these oils showed that linoleic acid and β-sitosterol were the main fatty acid and sterol compounds, respectively. A large amount of fatty acids and sterols were not significantly affected by the three extraction methods. Nevertheless, the tocols, the total phenols contents and the antioxidant power showed a variation between these three extracted oils. Based on these results, it was found that this specie has great potential for industrial use.

Keywords: *Asphodelus tenuifolius* / extraction method / oil / quality

Résumé – Effet du procédé d'extraction sur la qualité de l'huile des graines d'Asphodelus tenuifolius. Dans cette étude, les huiles de graines d'*Asphodelus tenuifolius* (ATO) ont été extraites par trois méthodes d'extraction différentes, à savoir l'extraction par Soxhlet (SE), l'extraction assistée par micro-ondes (MAE) et l'extraction assistée par ultrasons (UAE). Les propriétés physicochimiques, les acides gras, les stérols, les tocols, la teneur totale en phénols et l'activité antioxydant des graines d'ATO ont été examinés et comparés. Le meilleur rendement d'extraction a été obtenu par SE (21,97 ± 0,01 %), suivi par MAE (19,28 ± 0,13 %), tandis que celui de UAE était le plus faible (16,5 ± 0,1 %). L'analyse de la composition chimique de ces huiles a montré que l'acide linoléique et le β-sitostérol étaient les principaux composés d'acides gras et de stérols, respectivement. Une grande quantité d'acides gras et de stérols n'a pas été affectée de manière significative par les trois méthodes d'extraction. Néanmoins, les tocols, les teneurs totales en phénols et le pouvoir antioxydant ont varié entre les trois huiles extraites. Sur la base de ces résultats, il a été constaté que cette espèce a un grand potentiel d'utilisation industrielle.

Mots-clés : Asphodelus tenuifolius / méthode d'extraction / huile / qualité

1 Introduction

Asphodelus tenuifolius, is an annual plant; belonging to the family Liliaceae. It is native to the Mediterranean region, as it can be found present in other countries, Malaysia, Australia, Chile, New Zealand, Mexico and the United States (IUCN, 2005). The leaves, seeds and roots of *A. tenuifolius* have traditionally been used to treat several diseases, such as inflammatory disorders, rheumatic pain, colds, diuretics and hemorrhoids (Kalim *et al.,* 2010; Panghal *et al.,* 2011). Moreover, the authors reported the extracts of *A. tenuifolius* displayed antimicrobial potential, antioxidant effects and anti-parasitic activity—trophozoites growth inhibition assay (Kalim *et al.,* 2010; Menghani *et al.,* 2012).There are many bioactive compounds in different vegetative structures of

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A.tenuifolius plants, such as flavonoids (AP: Aerial Part leaves, stems and flowers), carbohydrates; Amino acids, Chromone, Triterpenoids (WP: Whole plant; leaves, stems, flowers and root) Naphthalene derivatives, Triterpenoids (R: Root) and fatty acids; Ester (seeds) (Malmir *et al.,* 2018). The seed oils of *A. tenuifolius* had a hight amount of polyunsaturated fatty acids, especially Linoleic acids accounting for about 62.62% (Madaan *et al.,* 1973, Ahmad *et al.,* 1976)

In recent years, some extraction methods have attracted attention, such as microwave-assisted extraction (MAE) and ultrasonic-assisted extraction (UAE). Microwave-assisted solvent extraction is a new procedure that combines organic solvent extraction and microwave irradiation. In addition, it heats the extract quickly, accelerates adsorption and desorption of compounds (Koubaa *et al.,* 2016). MAE has various advantages, such as shorter extraction time and higher yield (Amarni and Kadi, 2010). A study compared the conventional solvent extraction to MAE achieved a higher yield in less time for the oil from the olive cake and cotton seed that was extracted by solvent (*n*-hexane**)** (Amarni and Kadi, 2010; Taghvaei *et al.,* 2014).

Ultrasonic extraction technologies have main advantages; specifically high extraction yields, low solvent volume, and short extraction times (Vuong *et al.,* 2015). Ultrasound waves generated by a water bath or an ultrasound probe produce a disruption of the cells and facilitate the extraction by the cavitation phenomenon (Zhao *et al.,* 2007). In an ultrasonic bath, Mohammadpour extracted oil from *Moringa peregrina* seeds using *n*-hexane as a solvent (Mohammadpour *et al.,* 2019). The UAE had the highest oil yield with the desirable antioxidant activity. However, other factors had negative effects on the reproducibility, such as the volume of water in the ultrasonic bath, the location and wall thickness of the extraction vessel used. Compared to an ultrasonic bath, the ultrasonic probe could avoid the attenuation of the propagation of ultrasound because it is directly inserted into the extraction vial. However, due to the powerful thermal effect created by the ultrasound probe, it was impossible to take measures to control the rapid temperature increase (Chemat *et al.,* 2017a; Chemat *et al.,* 2017b).

Many studies have been conducted on the impact of extraction methods on chemical composition such as fatty acids, sterols, tocopherols, phenolic compounds and antioxidant activity. (Zhong *et a*l., 2018; Dong *et al.,* 2021).

Until now, the use of microwave and ultrasound to extract *A. tenuifoluis* seed oil has not been reported yet and the study on *A. tenuifolis* is the first to be conducted in Morocco, Therefore, the objective of our work was to study the influence of microwave and ultrasound extraction methods on the yield, physico-chemical parameters, determine the composition of fatty acids, sterols, tocols, total phenol and antioxidant activity which was compared with the classical soxhlet method. The characteristics bands of long-chain fatty acids were identified by Infrared spectroscopy. Additionally, the data was subjected to principal component analysis (PCA) to identify different characteristics of *A. tenuifolius* seed oil.

2 Materials and methods

2.1 Materials and reagents

The black seeds of *A. tenuifolius* were harvested from the regions of Tinghir in Morocco. The samples were shelled, dried in the dark at room temperature until the moisture content is 5%, powdered, and then sieved through a set of standard size sieves for oil extraction. The Folin-Ciocalteu reagent, tocol, and gallic acid and other chemical reagents were supplied by Sigma-Aldrich Co. (Steinheim, Germany).

2.2. Oil extraction methods

2.2.1 Soxhlet extraction (SE)

Approximately 20 g of the powdered material and 200 mL of *n*-hexane were placed in a Soxhlet extractor at 80 °C for six hours. Following extraction, *n*-hexane was eliminated using a rotary evaporator at 50 °C under pressure. The oil was dried at 60 ± 5 °C for 15 min in a drying oven. The yield of oil was measured gravimetrically.

2.2.2 Microwave-assisted solvent extraction (MAE)

Microwave-assisted extraction was carried out using an assembly consisting of a domestic microwave oven (whirlpool MWD, China,119 WH, 20L, 700 W), directly connected to the cooling system through a hole to condense the distillate continuously.

A 20 g of *A. tenuifolius* seed powder was added in conical flack with the *n*-hexane. The flask was inserted into the microwave extractor cavity and connected to the cooling system through a hole there. A microwave power was set to 200 W, and the extract was obtained at 60 °C for 30 minutes. After 30 minutes, the contents of the flask were transferred to centrifuge tubes. The oil was then retained at 4 °C for further analysis after the filtrate was evaporated in a rotating evaporator at reduced pressure and 50 °C.

2.2.3 Ultrasound-assisted solvent extraction (UAE)

Twenty grams of ground of seeds were mixed with 250 mL of hexane in a 500 mL beaker. An ultrasound probe (UP100H ultrasonic system, Hielscher, Germany) with 7 mm tip diameter was immersed into the mixture and sonicated at frequency of 24 kHz and power of 130 W. After 10 min of extraction time, the extract was centrifuged for 10 minutes at 4500 rpm and evaporated in a rotary evaporator (model VV2000, Heidolph, Schwabach, Germany) under reduced pressure at 40 °C.

2.3 Physicochemical properties

The acid value, saponification index, iodine value, and specific extinction coefficients (K_{232} and K_{270}) were obtained using AOCS suggested protocols Ca 3a-63, Cd 1b-87, Cd 3b-76, and Ch 5-91, respectively (AOCS, 1998). The acid value and saponification index were represented in mg KOH/g, IV was expressed as mg I_2/100g of oil, and extinction coefficients (K_{232} and K_{270}) were calculated using a spectrometer as the specific extinction of a 1% (w/v) solution of oil in cyclohexane (LLG-uniSPEC 2UV). Carotenoids and chlorophylls were measured at 670 and 470 nanometers, respectively. One gram of oil was dissolved in 100 mL of cyclohexane, and the results were given in milligrams per kilogram (El Moudden *et al.,* 2020). The natural pigment content was determined as follows:

$$\text{Chlorophylls}\,(\text{mg/kg}) = (A_{670} \times 10^6)/(613 \times 100 \times S),$$

$$\text{Carotenoids}\,(\text{mg/kg}) = (A_{470} \times 10^{6})/(2000 \times 100 \times S).$$

Where A represents absorbance and S indicates spectrophotometer cell thickness (1cm). Chlorophyll and carotenoid concentration were measured in milligrams of pheophytin and lutein per kilogram of oil, respectively.

2.4 Fatty acid composition

Fatty acids were synthesized into fatty acid methyl esters, which were then analysed using gas chromatography (Varian CP-3800) with a flame ionization detector (FID) and a CP-wax 52CB type column (30 m ×0.25 nm diameters). Helium was used as the carrier gas at a flow rate of 1 mL/min. The oven temperature was set to 170 °C for 1 min before being increased to 230 °C at a rate of 4 °C/min (Idrissi *et al.*, 2022). The data was processed using a Varian Star workstation v 6.30. (varian Inc. Walnut Creek, CA, USA). The results were represented as the relative percentage of each particular FA present in the sample.

2.5 Sterols composition

According to ISO 6799 (1991), the extracted oils were saponified and assessed in a gas chromatograph equipped with a flame ionization detector utilizing a capillary column (30 m × 0.32 mm, DI: 0.25µm, phase: CPSIL8CB). The injector type 1079 (T= 300°C), a FID (T= 300°C). and the carrier gas is helium (flow rate: 1.5 mL/min).

2.6 Tocols composition

The tocols content of *A. tenuifolius* seed oils was determined using the ISO 9936 standard method. The analysis is performed by HPLC equipped with a silica column (25 cm× 4 mm) on a fluorometric detector (excitation wavelength 290 nm, emission wavelength 330 nm). A mixture of isooctane: isopropanol (99:1 v/v) at a flow rate of 1.2 mL/min was used for tocol elution. External standards α, β, γ, δ isomers of tocols (tocopherols and tocotrienols) were used for their quantification (ISO, 2006).

2.7 Total phenolic content (TPC)

Using a separate approach, phenolic chemicals were extracted from seed residue using 200 mL of methanol/water mixture (70:30 v/v). The extracts were treated with the Folin Ciocalteau reagent before being mixed with a satured sodium carbonate solution (7.5% w/v) (Guihua *et al*., 2008). A UV visible spectrophotometer was used to measure the absorbance of reaction mixtures and a blank solution at 720 nm. The phenolic component concentration was calculated using gallic acid equivalents (mg GAE/100 g oil).

2.8 Antioxidant activity

The antioxidant activity of the various methanolic extracts was determined using DPPH, with some changes to the methods reported by Debasis *et al*., 2017. To extract solutions containing concentrations ranging from 0.6 to 2 mg/mL, 0.5 mL of DPPH (0.2 mMol) in methanol was injected. After 30 minutes of incubation in the dark at room temperature, the absorbance was measured at 517 nm. As a negative control, a DPPH solution without the sample was utilized. The result was computed as a percentage inhibition of DPPH using the following equation:

$$\text{DPPH scavenging activity}\,(\%) = \frac{(\text{NC})\text{Abs} - (\text{sample})\text{Abs}}{(\text{NC})\text{Abs}}.$$

2.9 Fourier transforms infrared (FT-IR) spectral analysis

The Fourier Transform Infrared spectrometer was used to measure the infrared spectra of the various ATOs. All oils spectra were recorded at 4000-400 cm^{-1} with a resolution of 4 cm^{-1}. The oil was placed between two pellets of potassium bromide (KBr). Each band was analyzed using the Origin 8.0 program.

2.10 Statistical analysis

Using IBM SPSS Statistics Version 21 software, the measurements were performed in triplicate, and the outcomes were subjected to an analysis of variance (ANOVA) and Tukey's test (SPSS Inc., Chicago, IL, USA). Pearson's was also used for correlation analysis. The physicochemical indices, fatty acid, tocol, sterol, total phenol contents, and antioxidant activity were examined using principle components analysis (PCA) to find any differentiation between the samples in terms of extraction method.

3 Results and discussion

3.1 Physicochemical properties

The yield and physicochemical properties of *A. tenuifolius* seeds oils, extracted by different extraction methods are shown in Table 1. SE showed the highest oil extraction yield (21.97 ± 0.01 %), followed by MAE (19.28±0.13 %) and UAE (16.5 ± 0.1 %). The acid value is used to measure the amount of carboxylic fatty acids present in oil as well as the degree of degradation. Compared to oil extracted by MAE and SE, UAE had a significantly lower acid value, since a lower temperature was employed in the UAE. As a quality measure, the *Codex Alimentarius* Commission established a maximum acid number of 4.0 mg KOH/g for crude oil (Codex Alimentarius, 2015). All the extraction methods presented acid values lower than this standard, indicating that these oils are of good quality.

The level of unsaturation in oils is measured by the iodine value. The results reveal that all extraction methods generated an equal proportion of high iodine, suggesting that the extraction process had no influence on oil saturation. These high iodine values are caused by the presence of more unsaturated fatty acid bonds and tend to stay liquid at room temperature (Emil *et al.,* 2010). This property shows that these oils were drying and indicates their applicability in the paint and varnish sector (Hespel, 2013).

Table 1. Yield and physicochemical properties of *A.tenuifolius* seed oil.

Parameters	SE	MAE	UAE
Extraction yield(%)	21.97±0.01[a]	19.28±0.13[b]	16.5±0.1[c]
Acid value (mg KOH/g oil)	1.96±0.28[a]	1.4±0.28[a]	0.84±0.28[a]
Saponification index (mg KOH/goil)	200.64±0.24[a]	200.52±0.17[a]	200.81±0.21[a]
Iodine index (g I_2/100 g oil)	168.87±0.27[a]	169.65±0.74[a]	168.17±0.56[a]
K_{232}	1.17±0.001[a]	1.159±0.001[b]	1.12±0.001[c]
K_{270}	0.88±0.001[a]	0.67±0.001[b]	0.35±0.002[c]
Carotenoid (mg/kg)	1.79±0.001[a]	1.56±0.00[b]	1.29±0.001[c]
Chlorophyll (mg/kg)	0.87±0.007[a]	0.47±0.05[b]	0.26±0.002[c]

Results are given as the mean values ± standard deviation of the three replicates (mean ± SD, $n = 3$); (a–f) different letters within a row indicate significant statistical differences ($p \leq 0.05$). Extraction yield; acid value (AV); saponification index (SV); iodine index (IV); conjugated dienes (K_{232}); conjugated trienes (K_{270}), chlorophyll (mg/kg) and carotenoid (mg/kg).

Table 2. Fatty acid composition of *A. tenuifolius* seed oil (%).

Fatty acids	SE	MAE	UAE
Saturated fatty acids			
Myristic (C14:0)	0.045±0.005[a]	0.045±0.005[a]	0.04±0.01[a]
Palmitic (C16:0)	6.88±0.01[a]	6.81±0.01[a]	7.07±0.26[a]
heptadecanoic (C17:0)	0.07±0.01[a]	0.07±0.01[a]	0.08±0.01[a]
Stearic (C18:0)	2.3±0.01[a]	2.23±0.005[b]	2.37±0.00[c]
Arachidic (C20:0)	0.095±0.005[a]	0.085±0.005[a]	0.08±0.01[a]
Monounsaturated fatty acids			
Palmitoleic (C16:1)	0.08±0.005[a]	0.07±0.005[a]	0.075±0.005[a]
Heptadecenoic (C17:1)	0 .03±0.005[a]	0.02±0 .01[a]	0 .01±0.005[a]
Oleic acid (C18:1)	11.67±0.01[a]	11.26±0.01[b]	11.52±0.005[c]
Gadoleic (C 20:1)	0.20±0.005[ab]	0.18±0.005[a]	0.21±0.005[b]
Polyunsaturated fatty acids			
Linoleic acid (C18:2)	78.50±0.01[a]	79.04±0.005[b]	78.14±0.01[c]
Linolenic acid (C18:3)	0.07±0.005[a]	0.13±0.01[b]	0.12±0.01[ba]
SFA	9.39±0.01[a]	9.24±0.005[b]	9.9±0.005[c]
MUFA	11.99±0.01[a]	11.54±0.005[b]	11.83±0.01[c]
PUFA	78.57±0.01[a]	79.17±0 .01[b]	78.26±0.00[c]

Data are presented as the mean of individual duplicates (n = 2nd ± SEM), means followed by similar superscript lowercase letters in the same row are not different ($p < 0.05$).

The saponification index of the oils obtained by different extraction methods did not present a significant difference (200 mg KOH/g) and higher than those obtained for Groundnut and sunflower oils (Amira *et al.,* 2014; Cerchiara *et al.,* 2010) and lower than a *Phoenix dactylifera*, Kariya and *Thevetia peruviana* (Ali *et al.,* 2015; Okeleye and Betiku 2019; Yarkasuwa *et al.,* 2013). This high saponification value (200 mg KOH/g) indicates the existence of higher molecular weight triacylglycerols, which favors their use in shoe polish, shampoo, liquid soap and alkyd resin preparation.

The absorbance measurements at 232 nm (K_{232}) and 270 nm (K_{270}) indicate the presence of primary (conjugated dienes) and secondary (conjugated trienes) oxidation products, respectively. The oil obtained with MAE had higher values than the oil obtained by the other two procedures, suggesting that it contained more oxidation products.

Chlorophyll and carotenoid were measured in the highest amounts (0.47 ± 0.05 and 1.56 ± 0.00 mg/kg, respectively) in Microwave-extracted ATO and (0.87 ± 0.007 and 1.79 ± 0.001 mg/kg, respectively) in soxhlet, but found in lower values (0.26 ± 0.002 and 1.29 ± 0.001 mg/kg, respectively) in ultrasound. The amounts of chlorophylls was less than 2 mg/kg in MAE and UAE methods. With less chlorophyll, it is possible to prevent photooxidation and increase the shelf life and quality of the oil (Kiritsakis and Markakis 1988).

3.2 Fatty acid composition

It can be noticed that the all extraction processes presented similar percentages in the chemical composition of fatty acids (Tab. 2). The main fatty acid of all oils is linoleic acid

Table 3. Sterols (mg/100 g of oil) of *A. tenuifolius* seed oils.

Sterols	SE	MAE	UAE
Cholesterol	7.65±0.005[a]	1.42±0.025[b]	1.47±0.003[b]
Campesterol	24,65±0.15[a]	32,02±0,001[b]	33.92±0.002[c]
Stigmasterol	2.945±0.01[a]	2.70±0.001[b]	2.24±0.002[c]
β-Sitosterol	256.32±0.001[a]	359.68±0.001[b]	354.26±0.002[c]
Δ5-avenasterol	91.84±0.01[a]	129.10±0.001[b]	138.008±0.001[c]
Δ7-stigmastenol	6.35±0.11[a]	1.41±0.015[b]	1.53±0.002[b]
Δ7-avenasterol	2.7±0.01[a]	10.03±0.0015[b]	11.31±0.001[c]
Total	394.55±0.106[a]	536.40±0.011[b]	542.75±0.012[c]

Data are presented as the mean of individual duplicates ($n = 2$nd ± SEM), means followed by similar superscript lowercase letters in the same row are not different ($p < 0.05$).

(78.14 ± 0.01 – 79.04 ± 0.005 %), followed by oleic (11.26 ± 0.01–11.67 ± 0.01 %) and palmitic acids (6.81 ± 0.01–7.07 ± 0.26 %). It's worth noting that linoleic acid has a higher value than the other fatty acid, similar to those reported for the red pepper seed oils (Chouaibi *et al.,* 2019). Because of its high linoleic acid content, *A. tenuifolius* seed oil was particularly susceptible to oxidation. However, this compound may have advantageous nutritional and physiological effects in the prevention of cancer and both coronary heart disease (Oomah *et al.,* 2000).

These three oils extracted were characterized by a low proportion of saturated (SFA) and monounsaturated (MSFA) fatty acids, while the proportion of polyunsaturated (PUFA) was the highest of the total fatty acids .The MAE technique exhibited the lowest SFA level and the greatest PUFA content. Recent research has demonstrated the relevance of PUFA in human health, particularly in the prevention of the many disease such as ulcerative colitis, inflammatory, rheumatoid arthritis, renal and autoimmune disease (Abedi and Sahari 2014).

The fatty acid content of the oils extracted using the three ways did not differ, demonstrating that the extraction methods have no effect on the fatty acid composition of ATO. This finding is consistent with other studies published by Castejón *et al.* (2018) and Dong *et al.* (2021).

3.3 Sterols composition

Table 3 shows the sterol content of three distinct extraction methods. The total sterol concentration of ATO extracted by SE (394.55 ± 0.10 mg/100 g oil) was not statistically different between UAE and MAE in terms of sterol content, but it was noticeably lower than that of the oils extracted using the other two procedures ($p = 0.05$).The predominant sterol in AT oil seeds was β-sitosterol, which ranged from 256.32 ± 0.001 to 359.68 ± 0.001 mg/g. The MAE method yielded the highest value, followed by the UAE (354.26 ± 0.002 mg/g oil) and SE samples (256.32 ± 0.001 mg/g oil). In addition, Δ5-avenasterol was identified in ATO when the high content was found in the UAE and MAE methods. It is well known that the Δ5-avenasterol found in frying oils has antioxidant and antipolymerization properties (Gordon and Magos, 1983). In comparison to other vegetable oils, ATO had significantly greater sterol content than the *Maclura pomifera* oil (Saloua *et al.,* 2009) and kernel oils (*Zhiyong et al*., 2016).

3.4 Tocols composition

Six kinds of Tocol were identified, which γ-tocotrienol was the major tocol in the ATOs, ranging from 81.36±0.00 mg/100g oil to 106.86±0.01 mg/100 g oil. The lowest γ-tocotrienol level in the ATO was accounted for by SE, while the highest was accounted for by MAE. The γ-tocotrienol has a considerable impact on the prevention of colon cancer (*Guihua et al.,* 2008). Furthermore, the level of α-tocopherol in ATO extracted using various methods ranged from 8.66 ± 0.012 mg/100 g oil to 27.90 ±0.01 mg/100 g oil, with the UAE method producing the greatest content, followed by MAE and SE samples. SE has a lower total tocol content in ATO than the MAE and UAE techniques. When compared to tocopherols, tocotrienols showed strong anticancer, cholesterol-lowering, and neuroprotective effects (Weiping *et al.,* 1999; Chandan *et al.,* 2006). As indicated in Table 4, there were substantial changes ($p < 0.05$) between the ATOs extracted using various techniques, suggesting that the extraction method has a major impact on the tocol content.

3.5 Total phenolic content (TPC) and antioxidant activity (extract)

Table 5 shows the phenolic content of the methanolic extracts. For the three extracts tested the value total phenol content range from 12.68±0.13 to 18.51±0.19 mg GAE/g extract. The total phenol concentration of the three extracts studied was higher than that of *Echium vulgare* (11.94 mg GAE/g oil) (Eruygur *et al.,* 2012). In particular, the extract obtained by the UAE method had high levels of total polyphenols, which were then followed in decreasing order by the MAE and Soxhlet methods. The UAE method produces the highest quality extract because to its high value of total phenolic components of the extract, which improves the resistance of the extract to oxidation. It can be concluded that the content of polyphenolic substances is mainly influenced by the extraction technologies.

The DPPH free radical scavenging capacity of the ATO extracted by three methods presented in Table 5. The UAE approach extracted the ATO with the lowest concentration of 50% radical scavenging ability (503,199 ± 0.11 μg/mL), followed by the MAE (705.74 ± 0.02 μg/mL) and SE (982.47 ± 0.05 μg/mL) procedures. The UAE had more antioxidant capacity than the

Table 4. Tocols (mg/kg of oil) of *A. tenuifolius* seed oil.

Tocols	SE	MAE	UAE
α-Tocopherol	8.66±0.012[a]	9.02±0.00[b]	27.90±0.01[c]
β-Tocopherol	2.69±0.005[a]	3.35±0.00[b]	4.33±0.01[c]
γ-Tocopherol	0.706±0.001[a]	0.846±0.004[b]	1.71±0.001[c]
α-Tocotrienol	2.54±0.00[a]	5.68±0.005[b]	5.72±0.01[c]
γ-Tocotrienol	81.36±0.00[a]	106.86±0.01[b]	96.39±0.005[c]
δ-Tocotrienol	9.39±0.005[a]	9.09±0.00[b]	8.41±0.00[c]
Total	105.37±0.02[a]	134.86±0.02[b]	144.52±0.059[c]

Data are presented as the mean of individual duplicates ($n = 2nd \pm SEM$), means followed by similar superscript lowercase letters in the same row are not different ($p < 0.05$).

Table 5. TPC (mg GAE/g extract) and IC_{50} (μg/mL) of *A. tenuifolius* seed oil.

	SE	MAE	UAE
TPC(mg GAE/g extract)	12.68±0.13[a]	15.65±0.16[b]	18.51±0.19[c]
IC_{50} (μg/mL)	982.47±0.01[a]	705.74±0.01[b]	503.19±0.01[c]

Data are presented as the mean of individual duplicates ($n = 2nd \pm SEM$), means followed by similar superscript lowercase letters in the same row are not different ($P < 0.05$).

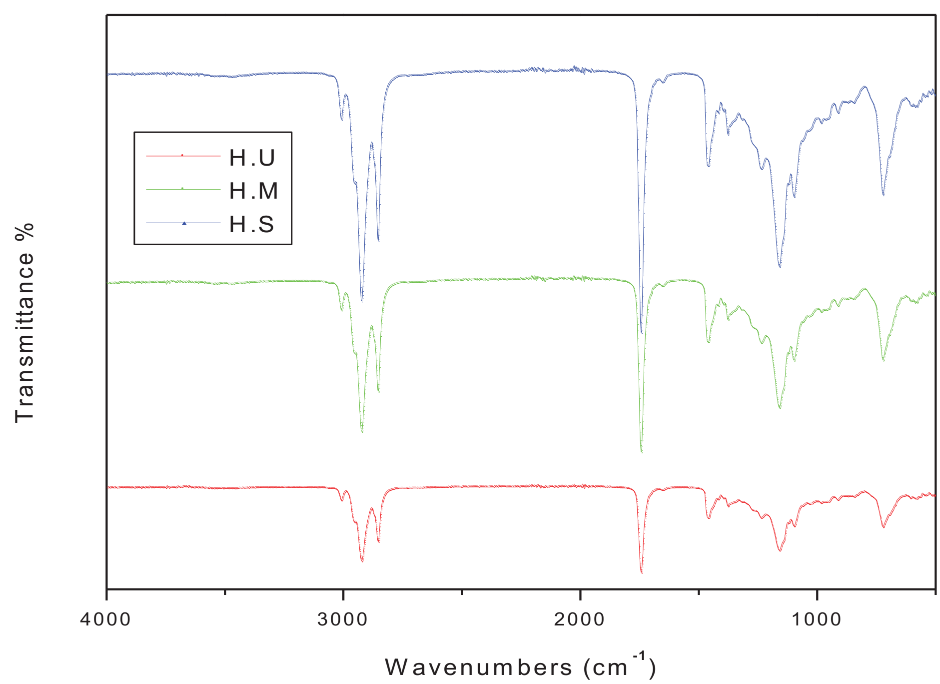

Fig. 1. FT-IR spectra of *Asphodelus tenuifolius* oils extracted by different methods.

other techniques. It indicated that more than 80% of free radicals were inhibited. These results suggested that the extraction technique is a key factor affecting the antioxidant activity of the extracts.

3.6 Fourier transform infrared (FT-IR) spectroscopy

All infrared spectra of the *Asphodelus* oils were similar because of the similar chemical composition (Fig. 1). The strong triplet peaks at 2980–2800 cm^{-1} are attributed to the C–H stretching of the methyl and methylene backbones of the oils. The C<O carbonyl stretching of fatty acid ester groups was related to the high absorption peak at 1745 cm^{-1}. A combined effect of methyl and methylene group deformation modes resulted in the overlapping peaks at 1460-1372 cm^{-1}. The peak at 1165 cm^{-1} was associated to diacyl glycerol ethers found in oils. Furthermore, at 721 cm^{-1}, all spectra showed a peak connected to cis C&9552;C out-of-plane bending (Vongsvivut *et al.,* 2014).

Table 6. Pearson's correlation matrix coefficient between the variables of the various seed oil samples.

Variables	K_{232}	K_{270}	Chlorophyll	Carotenoid	Linoleic acid	Linolenic acid	T. sterols	T. tocol	TPC	DPPH	Acid index	Iodine index	Saponification
K_{232}	1												
K_{270}	0.997	1											
Chlorophyll	0.926	0.953	1										
Carotenoid	0.986	0.996	0.976	1									
Linoleic acid	0.579	0.513	0.228	0.434	1								
Linolenic acid	−0.619	−0.678	−0.870	−0.742	0.283	1							
T. sterols	−0.768	−0.816	−0.953	−0.865	0.077	0.978	1						
T. tocol	−0.887	−0.921	−0.996	−0.952	−0.138	0.911	0.977	1					
TPC	−0.976	−0.990	−0.986	−0.999	−0.387	0.775	0.890	0.967	1				
DPPH	−0.994	−1.000	−0.961	−0.998	−0.488	0.699	0.832	0.932	0.994	1			
Acid index	0.978	0.992	0.984	0.999	0.397	−0.768	−0.884	−0.964	−1.000	−0.995	1		
Iodine Index	0.645	0.583	0.309	0.508	0.996	0.201	−0.007	−0.220	−0.462	−0.560	0.473	1	
Saponification	−0.739	−0.684	−0.430	−0.616	−0.977	−0.072	0.137	0.345	0.574	0.663	−0.583	−0.992	1

3.7 Correlation matrix

Table 6 displays the Pearson correlation, which aids in assessing the relationships between the variables investigated in this study. There was a substantial positive connection ($p < 0.05$) between K_{232} and K_{270} ($r^2 = 0.997$), as well as a very positive correlation (p-value < 0.05) between Carotenoid and Acid index ($r^2 = 0.999$). The correlations data of TPC with DPPH free radical scavenging effect were 0.994, this indicates that the polyphenol content of *ATO* contributes to its hydrogen electron donating abilities. Many authors, Amri *et al.* (2015) and Guettaf *et al.* (2016), have also demonstrated a linear positive correlation between polyphenol and antioxidant capacity. Furthermore, a substantial positive connection (p-value < 0.0001) was found between iodine value (IV) and the polyunsaturated fatty acid Linoleic (C18:2) (r^2= 0.996). These findings agree with those obtained with fluted pumpkin seed oil. (Samuel *et al.,* 2017).

3.8 Principal component analysis (PCA)

The variables evaluated on the F1–F2 factorial plan were projected using the PCA. Physicochemical characteristics, fatty acid compositions, tocols, sterols, total phenol, and ATO antioxidant activity are among the variables. As shown in Figure 2, the cumulative percentage of two principal components (Fs) was 100.00%, (F1: 76.46%; F2: 23.54%), it was more than 50%, indicating that it was representative of the variables. A plan generated by the F1 and F2 axes explained the associations between all variables investigated. Besides, the F1 axe was formed by the positive correlation between TPC, DPPH (1/IC_{50}), carotenoid, total tocol and chlorophyll. Axis F2 is produced by the inverse relationship between linolenic acid and saponification. The SE samples are distinguished by their high chlorophyll and carotenoid levels, as well as their acid index and extinction coefficient (K_{232} and K_{270}). For the MAE oil, it can be distinguished by the high content of iodine index and of polyunsaturated fatty acids Linoleic (C18:2). The UAE samples obtained significantly higher Saponification Index, polyunsaturated fatty acids (linolenic C18:3), Total sterol, total tocol and total polyphenol content (TPC), which indicates its great antioxidant power (Fig. 3).

4 Conclusions

The physicochemical and antioxidant characteristics of Moroccan *Asphodelus tenuifolius* seed oils extracted using various techniques, as well as the fatty acid, sterol and tocol, were investigated. The results revealed that the most abundant fatty acid, sterol and tocol were Linoleic acid (C18:2), β-sitosterol and γ-Tocotrienol. The yield and the acid index, sterol content, tocol, and total phenol contents were affected by the three extraction methods. However, minor differences existed in the fatty acid, and saponification index.

Chemometric analysis (PCA) was beneficial in visualizing the evaluation of *Asphodelus tenuifolius* oils obtained by diverse procedures. The UAE procedure is recommended for ATO extraction to obtain a product having a high total of Fatty acid, tocopherol, sterol, and polyphenol content (TPC). The MAE process is preferable for preparing oils rich in linoleic

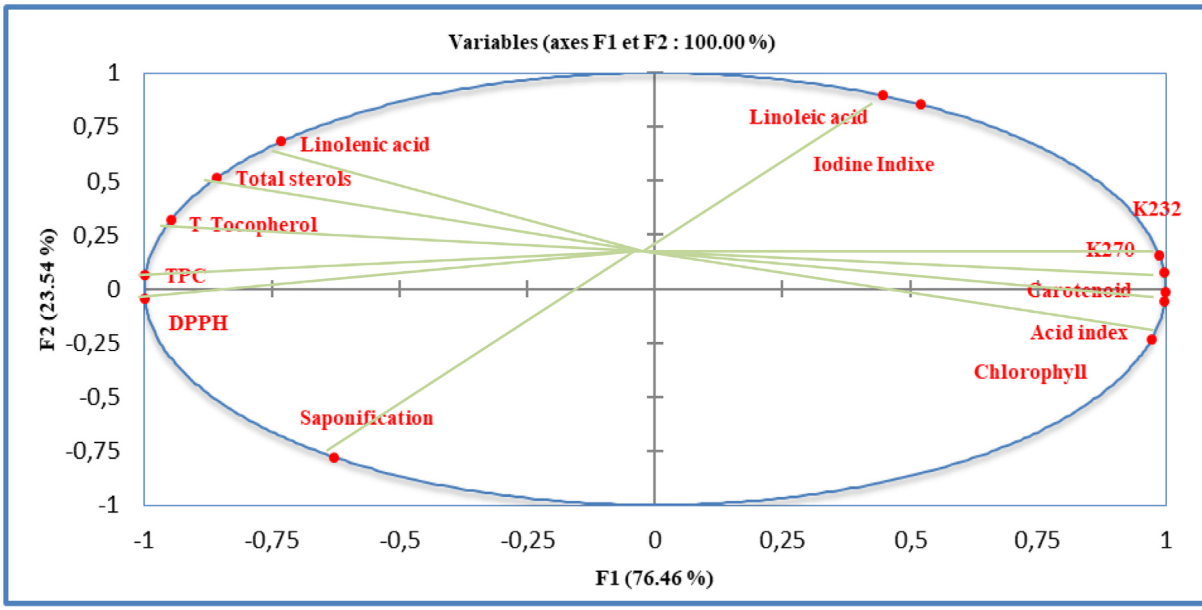

Fig. 2. Principal Component Analysis factorial plan carried out on the values of the different the variables: FFA, Total sterol, Phenolic compounds, antioxidant activity, Carotenoid, Total Tocol, Chlorophyll, K_{270}, K_{232}, PV, IV, Linoleic, Linolenic acid of the different samples of seeds oils.

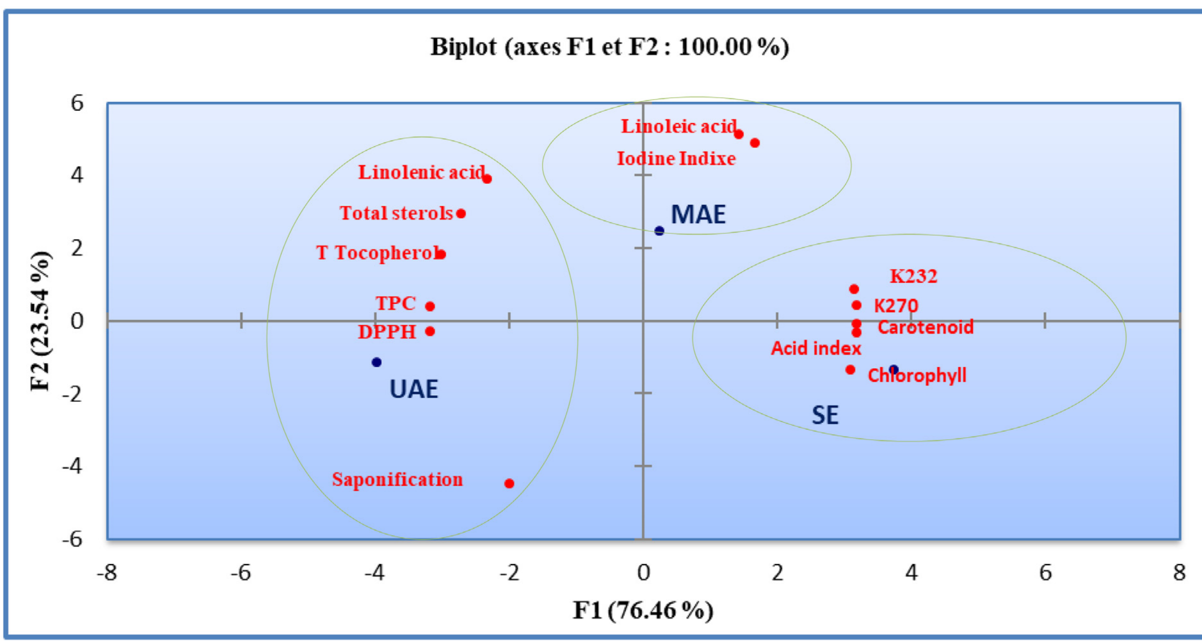

Fig. 3. Projection of individuals on the factorial plan (F1×F2). SE: MAE; UAE.

acid. Moreover, The SE approach is suggested to produce ATO with greater chlorophyll and carotenoid concentrations. Mostly, The UAE method is suggested for extracting high-quality *Asphodelus tenuifolius* oil for industrial use.

Authors contributions

Conceptualization, Fatima Ezzahra Eddaoudi and Mohamed Tabyaoui; **Data curation**, Yousra El Idrissi and Hicham Harhar; **Formal analysis**, Fatima Ezzahra Eddaoudi, Hamza El Moudden, and Mohammed Saber; **Investigation**, Chakir El Guezzane, Hicham Harhar and Mohammed Saber; **Methodology**, Fatima Ezzahra Eddaoudi, Hamza El Moudden, and Mohamed Tabyaoui; **Project administration**, Hamza El Moudden, Hicham Harhar and Mohamed Tabyaoui; **Resources**, Chakir El Guezzane and Hamza El Moudden; **Supervision**, Hicham Harhar and Mohamed Tabyaoui; **Validation**, Mohamed Tabyaoui; **Visualization**, Mohamed Tabyaoui; **Writing – original draft**, Fatima Ezzahra Eddaoudi, Chakir El Guezzane, and Mohamed Tabyaoui; **Writing – review &**

editing, Fatima Ezzahra Eddaoudi, Hamza El Moudden, Hicham Harhar, and Mohamed Tabyaoui.

Conflicts of Interest

The authors declare no conflict of interest.

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