Maceration, microwave assisted and ultrasound assisted extraction of phenolic compounds from Tunisian *Limoniastrum monopetalum* : kinetic modeling and chemical analysis

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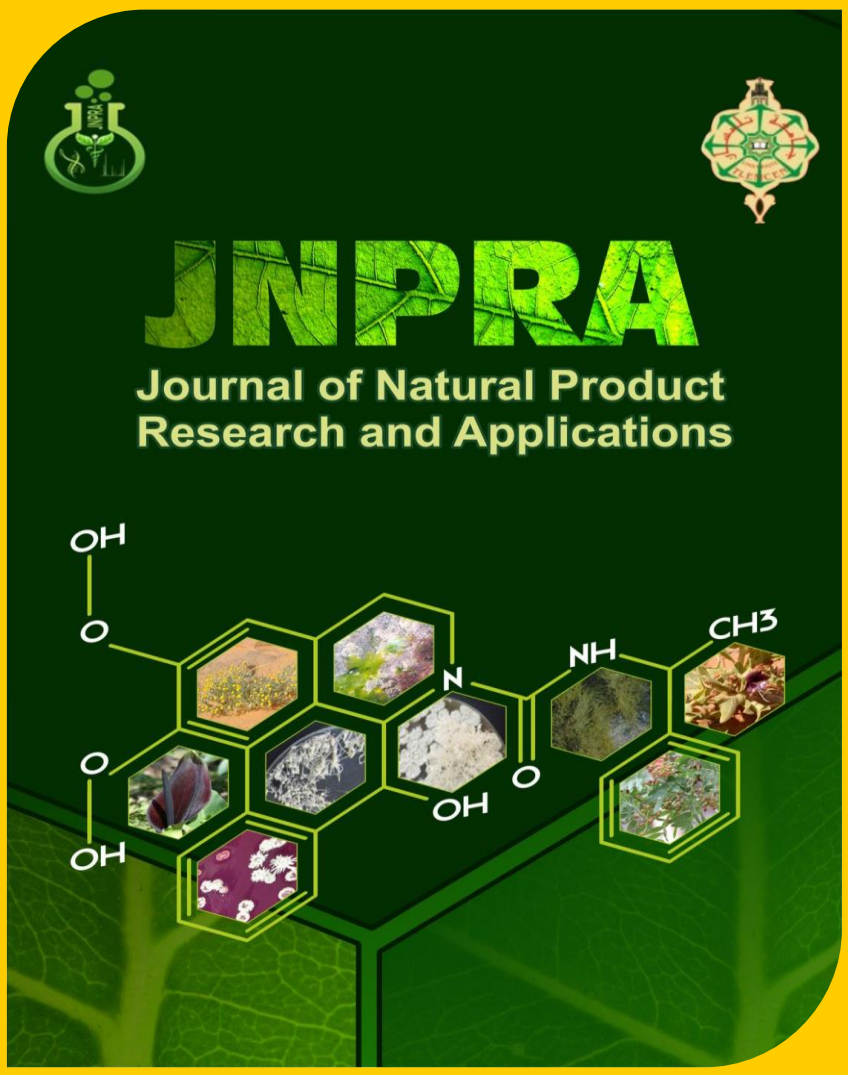

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Maceration, microwave assisted and ultrasound assisted extraction of phenolic compounds from Tunisian *Limoniastrum monopetalum*: kinetic modeling and chemical analysis

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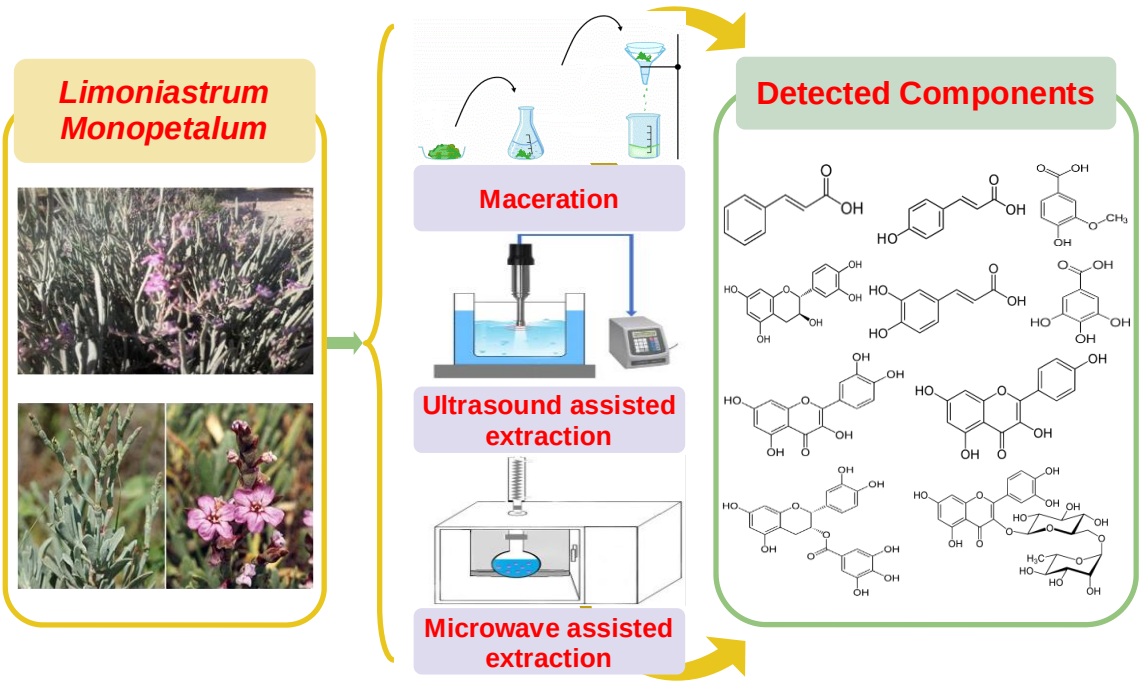
Highlights

- *Limoniastrum monopetalum* is a source of phenolic compounds.
- Patricelli's model provided an accurate modeling of polyphenols extraction kinetics.
- MAE method is the better method to reach a higher proportion of polyphenols.

Graphical abstract



Abstract
Maceration, ultrasound-assisted extraction (UAE), and microwave-assisted extraction (MAE) were investigated in this study, focusing on the selectivity towards phenolic compounds in ethanol extracts from *Limoniastrum monopetalum* leaves using the Folin-Ciocalteu method and kinetic models of extractions. Moreover, several phenolic compounds were identified by RP-HPLC. Results showed that total polyphenol compounds (TPC) under the MAE conditions (ethanol, 700 W microwave power, 60 s extraction time, and 1 mL/g solvent-to-solid ratio), were 75.707 mg EGA/100 g dried sample in only one minute of extraction. We conclude that MAE is a promising extraction method for polyphenols. Model results were correlated with mathematical models of the extraction process. Six phenolic acids and three flavonoids were identified by HPLC–DAD. MAE is a valuable and green analytical methodology for the investigation of phenolic components in natural plants.

Keywords: *Limoniastrum monopetalum*; RP-HPLC; phenolic compounds; UAE; MAE.

1. Introduction

Limoniastrum monopetalum L. Boiss is a green plumbaginaceae ranging from 50 to 120 cm in height. It grows in swamps and in sandy and rocky soils. Some researchers have found that this plant is a source of phenolic compounds (Trabelsi et al., 2010) and it is widely accepted that the significant antioxidant activity of plants is related to their high total phenolic content (Khedher et al., 2014). Plants represent a rich source of natural products with an almost infinite molecular diversity, of which the active ingredients of medicinal plants are mostly phenolic compounds. They are among the most important phytochemicals due to their antimicrobial, antiviral, anti-inflammatory, and anti-cancer properties and high antioxidant capacities (Touati et al., 2015; Dragsted et al., 1993; Krishnaswamy et al., 2013).
Therefore, in this work, for *Limoniastrum monopetalum*, the maceration, ultrasound-assisted (UAE) and microwave-assisted (MAE) extraction of phenolic compounds with ethanol were compared for the first time. In the first step, we established the kinetic models of these extractions. After that, we characterized the extracts by identifying several phenolic compounds by RP-HPLC.

2. Materials and Methods

2.1. Plant materials

The plant *Limoniastrum monopetalum* L. (LM), also named *Bubania monopetala* L. Girard or *Statice monopetala* in the international plant names index (also called *Limoniastrum* in the UK), was collected in March 2021, the flowering season, from Gafsa (Tunisia). The botanical identification was performed by G. Pottier–Alapetite and Abdessatar Ghobtane. Voucher specimens were conserved at the Herbarium of Montpellier University in France under the number MPU025549 for *Limoniastrum monopetalum* L. Boiss. The aerial part of *Limoniastrum monopetalum* was washed and air-dried for four weeks in the dark. Finally, the specimens were reduced to powder.

2.2. Extraction methods

2.2.1. Extraction by maceration

Extracts were obtained using ethanol. Ethanol was used as an environmentally preferable green solvent, and based on our previous studies (Elakremi et al., 2022a; Khedher et al., 2014; 2021; Nacer et al., 2023). Plant extract was obtained by magnetic stirring of 1 g of dry matter powder with 10 mL of solvent in a glass bottle for (20, 40, 60, 80, 100, 150, 500, 2880, 4320 min) at room temperature (25°C). Experiments were done at room temperature, and varying the duration of extraction for mathematical modeling using the Patricelli and Peleg models. The extract was filtered through filter paper, and the solvent evaporated under reduced pressure. The extract was stored at 4°C until tested (Elakremi et al., 2022a; Nacer et al., 2023).

2.2.2. Ultrasound assisted extraction (UAE)

An ultrasonic system with a working frequency fixed at 20 KHz (FALC ultrasonic UTA, Italy) was used for extracting secondary metabolites from the aerial part (leaves, flowers and seeds) of *Limoniastrum monopetalum*. Briefly, 1 g of dry powder was mixed with 10 mL of ethanol in a closed tube. The obtained suspension was exposed to acoustic waves in a bath of 3 L of capacity for (0, 5, 10, 15, 20, 30, 40, and 50 min) extraction time and at a temperature (25 ± 2°C). Each experiment was carried out in triplicate. After the UAE treatment, the extract was filtered through filter paper, and the solvent was evaporated under reduced pressure. Extract was stored at 4°C until tested (Elakremi et al., 2022a; Nacer et al., 2023).

2.2.3. Microwave assisted extraction (MAE)

A domestic microwave oven (Carrefour HMG20MD) with a cavity of 20 L and a working frequency of 2.45 GHz was used for extracting secondary metabolites from the aerial part (leaves, flowers and seeds) of *Limoniastrum monopetalum* powder. In a round bottom flask, 1 g of dry powder was mixed with 10 mL of ethanol by stirring as preparation for the extraction using the MAE system. The MAE extraction parameters were microwave power (700 W) and extraction time (5, 10, 20, 30, 45, and 60 s). Each trial was carried out in triplicate. After the MAE treatment, the extract was filtered through filter paper, and the solvent was evaporated under reduced pressure. The extract was stored at 4°C until tested (Elakremi et al., 2022a; Nacer et al., 2023).

2.3. Determination of total polyphenols content (TPC)

Phenolic content was determined according to the Folin–Ciocalteu method (Elakremi et al., 2022a; Nacer et al., 2023; Tao et al., 2014). 300 µL of diluted sample extract were added to 1500 µL of Folin–Ciocalteu reagent (10/100). After 1 min, 1200 µL of aqueous sodium carbonate (7.5 g/100 mL) were added. The mixture was vortexed and allowed to stand at room temperature in the dark for 120 min. The absorbance was read at 760 nm (Elakremi et al., 2022a; Khedher et al., 2021; Nacer et al., 2023), using a UV-visible spectrometer (BECKMAN DU 800) in a 10 mm quartz cuvette. The total phenolic content in the extract was calculated through the calibration curve, using gallic acid as a standard, and the results were expressed as mg of gallic acid equivalents (mg EGA) per 100 g dried sample. Three determinations were performed on each sample. For gallic acid, the curve of absorbance versus concentration is described by the equation: $Y = 8.9321\ X + 0.0102$ with $R^2 = 0.9987$.

2.4. Phenolic compounds identification by RP-HPLC

The phenolic compound content was analyzed using an HPLC system from Varian Proster. The column was a RP-C18 Zorbax (250 mm, 4.6 mm, 5µm) reverse-phase silica gel column.

Detector: DAD spectrophotometer at 280 nm or at 360 nm. All samples were tested in triplicate. The gradient elution method was performed with (A): water/acetic acid (98/2, v/v) and (B): water/acetonitrile/acetic acid (58/40/2, v/v/v). The solvent gradient in volume ratios were as follows: 0–10 min, 100% B; 10–20 min, 20% A–80% B; 20–35 min, 100% A.

The phenolic acids and the flavonoids were detected, respectively, at 280 nm and 360 nm. Each compound was identified by comparison to the retention time of a standard compound. Retention times of phenolic acids were 1-Gallic acid (6.5 min), 2-Catechin (15.8 min), 3-Caffeic acid (16.8 min), 4-Epicatechin (18.6 min), 5-Vanillic acid (20.8 min), 6-p-coumaric acid (26 min) and 7-Cinnamic acid (28 min), and of flavonoids: 8-Rutin (21.8 min), 9-Quercetin (30 min) and 10-Kampferol (33 min) (Elakremi et al., 2022a; 2023; Khedher et al., 2021; Yiin et al., 2016).

2.5. Mathematical modelling of extraction kinetics profile

The extraction curves of phenolic compounds obtained with the experiments of maceration, UAE, and MAE were fitted to the models derived by Patricelli et al. (1979) and Peleg (1988). The basis of Patricelli's model was to consider the extraction of active compounds as controlled by two phase boundaries. The first boundary explains the washing step, where the compounds are dissolved in bulk solvent whose temperature has been increased, thus reducing the mass transfer limitations and enhancing solvent penetration into ruptured wall cells. On the other hand, the second phase considers a diffusion step where the solutes from the internal wall cells diffuse into the solvent. Typically, this step is slower than the first extraction step due to mass transfer limitations.

The extraction yield of phenolic compounds for Patricelli's model is given by the following equation that expresses the yield of extraction (ρ) as a function of time (t) (Eq. (1)):

$$\boldsymbol{\rho = \rho_1[1 - \exp(-k_l t)] + \rho_d[1 - exp(-k_d t)]} \qquad Eq.(1)$$

Where $\boldsymbol{\rho_l}$ (mg GAE/100g dried sample) is the solute equilibrium yield at the washing phase, $\boldsymbol{\rho_d}$ (mg GAE/100 g dried sample) is the solute equilibrium yield at the diffusion step,

$\boldsymbol{k_l}$ (min^{-1}) is the mass transfer coefficient at the washing step, and $\boldsymbol{k_d}$ (min^{-1}) is the mass transfer coefficient at the diffusion step.

The derivation of polyphenol extraction yield rate estimation is expressed in Eq. (2).

$$\boldsymbol{V = \frac{d\rho}{dt} = \rho_1 k_l \exp(-k_l t) + \rho_d k_d \, exp(-k_d t)]} \qquad Eq.(2)$$

Values of $\boldsymbol{k_l}$ are usually higher than values of $\boldsymbol{k_d}$ due to the fast extraction rate at the beginning of the course. Thus, we determined the extraction rate of the process at the beginning of phase $\boldsymbol{V_0}$ at t = 0 (Eq. (3)). However, total equilibrium yield $\boldsymbol{\rho_e}$ can be determined from the summation of the equilibrium yield from both regions $\boldsymbol{\rho_l + \rho_d}$.

$$\boldsymbol{V_0 = \left(\frac{d\rho}{dt}\right)_{t=0} = \rho_1 k_1 + \rho_d k_d} \qquad Eq.(3)$$

In contrast, Peleg (1988) proposed a non-exponential empirical model where the absorption of solutes into the solvent was established as the basis of the development model. Peleg's model is described through the following equation (Eq. (4)).

$$\rho = \rho_0 + \frac{t}{(K_1 + K_2 t)} \qquad Eq.(4)$$

Where **ρ** is the yield of polyphenols at the time **t** of extraction. **ρ₀** is the initial yield of extraction. When t = 0, **K₁** is Peleg's constant rate (min.100g dried sample/mg GAE) and **K₂** is Peleg's capacity constant (100g dried sample/mg GAE). As for the experiments performed in this work, the initial polyphenol yield **ρ₀** is zero in all conditions, which was the working equation used to fit the experimental data (Eq. (5)).

$$\rho = \frac{t}{(K_1 + K_2 t)} \qquad Eq.(5)$$

The Peleg's constant rate **K₁** and capacity constant **K₂** are related to extraction rates at the very beginning course **V₀** (**t =0**) and the equilibrium yield **ρe** (**t** = ∞), respectively. At equilibrium yield, the content is considered to be at its maximum. Their relationships can be described as follows in Eqs. (6) and (7).

$$V_0 = \frac{1}{K_1} \qquad Eq.(6)$$

$$\rho_e = \frac{1}{K_2} \qquad Eq.(7)$$

All data fittings and analyses were done by the Solver program package implemented in Microsoft Office 2007. The parameters for the Patricelli and Peleg models were estimated based on the experimental data by means of non-linear regression, using the percentage average absolute relative deviation **(% AARD)** between the experimental and predicted extraction yield as the objective function for the minimization procedure (Eq. (8)).

$$\%\mathbf{AARD} = \frac{100}{N}\sum_{i=1}^{N}\frac{|\rho_{exp} - \rho_{pred}|}{\rho_{exp}} \qquad Eq.(8)$$

Where N is the number of experimental data points. The coefficient of determination R^2 was calculated to determine the quality of the fit residuals between experimental and calculated data from both models.

3. Results and discussion

Phenolic compounds are a class of chemical constituents containing one or more hydroxyl residues attached to an aromatic (phenyl) ring. They are very effective antioxidative constituents that contribute to the antioxidant activity of plant foods (Alara et al., 2021; Gil-Martin et al., 2022; Rashmi and Negi, 2020; Skendi et al., 2022; Yu et al., 2021). Hence, it was deemed important to quantify the phenolic content.

3.1. Polyphenols extraction

Extraction is an important step in the recovery and isolation of bioactive phytochemicals from plant materials before analysis. Maceration is the most commonly used procedure prior to the analysis of bioactive compounds in the natural matrix. In our study, ethanol extracts of the aerial part of *Limoniastrum monopetalum* in relation to the extraction methods UAE, MAE, and maceration were studied. It was evaluated by the yields of polyphenol extraction methods. The extraction curves obtained with the extraction approaches were fitted with Patricelli and Peleg's models (**Figure 1**).

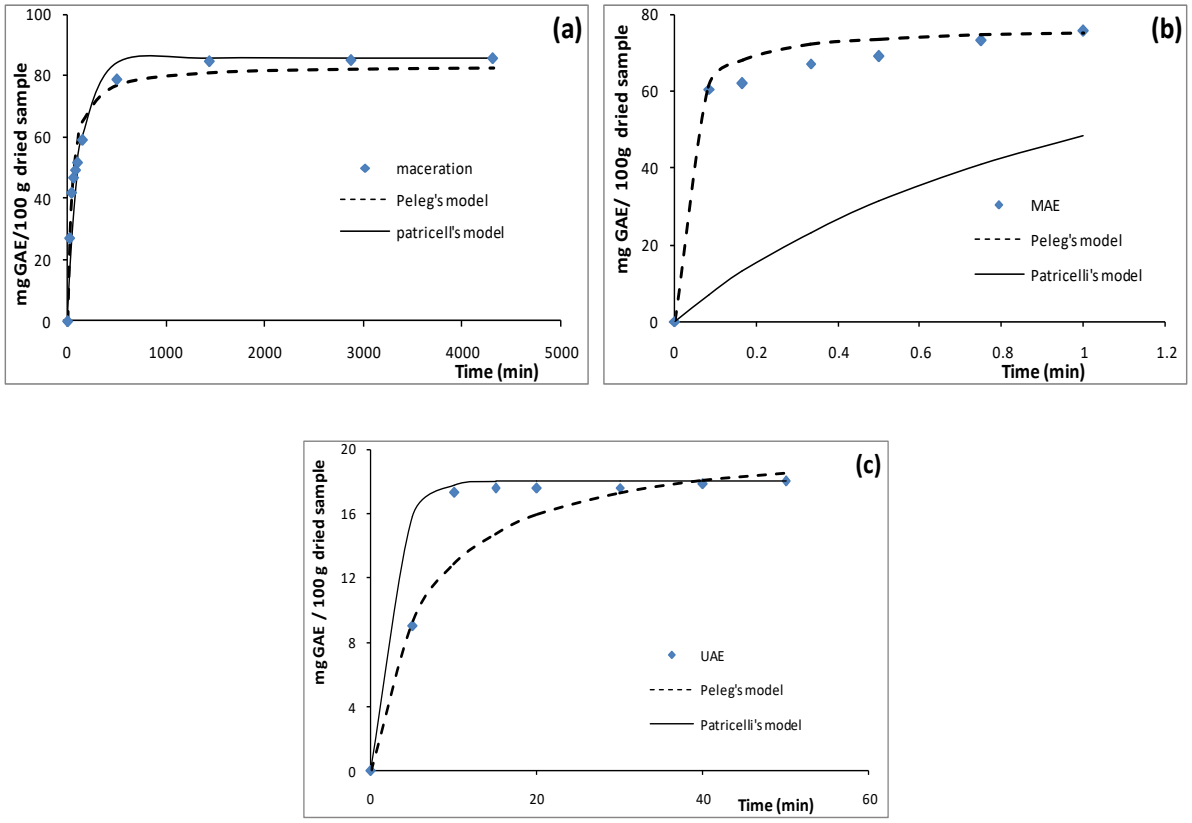


Figure 1. Variation of yield (in %) by time (in min): **(a)**: maceration extract, **(b)**: MAE extract, **(c)**: UAE extract

It is observed that the extraction with microwave irradiation resulted in a considerable increase in the extraction yield. Moreover, a significantly faster extraction was observed with MAE treated samples. Indeed, the conventional method needs more than 500 min to reach equilibrium yield, whereas with MAE-treated samples, the equilibrium yield was reached in less than 0.1 min. As a conclusion, MAE-treatment clearly increased the extraction yield rate of polyphenols from *Limoniastrum monopetalum*. The extraction of solutes in MAE was driven by electromagnetic waves that heated the whole sample simultaneously. As a result, localized heating occurred, leading to an expansion and rupture of cell walls and an improvement in the release of solutes from the material. However, in the conventional method, the extraction needed a longer period for the solutes to diffuse out and become solubilized in the solvent (Chan et al., 2011; Elakremi et al., 2022a; Molto-Puigmartí et al., 2011), whereas the UAE extraction yields did not present significant results (Figure 1).

The kinetic profiles of polyphenol yield extraction for different processes were correlated with Peleg's and Patricelli's models, as presented in Table 1.

Table 1. Mass transfert coefficient and yield of extraction by maceration, MAE and UAE fitted by Patricell's and Peleg's models.

Model's Patricelli	**K_l**	**K_d**	**ρ_l**	**ρ_d**	**ρ_e**	**R^2**
Maceration	0.008	0.008	78.712	6.888	85.6	0.952
MAE	0.935	3.911	69.2366	6.4634	75.7	0.537
UAE	0.416	0.767	17.577	0.483	18.06	0.875
Model's Peleg	**K_1**	**K_2**			**ρ_e**	**R^2**
Maceration	0.500	0.012			83.333	0.980
MAE	0.00028	0.013			76.923	0.991
UAE	0.295	0.048			20.833	0.927

Yield (ρ) is expressed as mg GAE/100 g dried sample.

The high value of the correlation coefficient R^2 and reasonable model characteristics of the Patricelli's model compared to Peleg's model indicate that the Patricelli's model is a useful tool to profile the UAE and MAE from *Limoniastrum monopetalum* for the conditions studied. In this case, the extraction process was controlled by two processes: washing and diffusion.
As for maceration, Peleg's model profiles the extraction. This result indicates that this model is based on desorption and sorption phenomena. The total phenolic content (TPC) changed significantly. It depends, importantly, on the extraction conditions (Table 2).

Table 2. Total phenolic compounds content (TPC) in different extraction methods.

Extracts	Maceration	MAE	UAE
TPC in mg EGA/100g dried sample	85.60	75.70	18.06

For instance, the results showed that TPC was extracted similarly by using MAE in ethanol at 700 W for 1 min and maceration for 36 hours at room temperature. From the results obtained, MAE can be considered a better method to reach a higher proportion of polyphenols (75.70 mg EGA/100 g dried sample) than UAE (18.06 mg EGA/100 g dried sample). Evidently, it was also observed that heat MAE was a better extraction method compared to maceration extraction at room temperature because of the shorter extraction duration. Indeed, the extraction was done in 0.1 min instead of 500 minutes.
To optimize UAE extraction, we can increase the temperature; however, this is not an appropriate measure for all plants. In fact, for *pomegranate seed* oil extraction by UAE, the yield decreases with temperature increase (Milic et al., 2013; Goula, 2012). The low extraction yield is probably due to the limited solubility of phenolic compounds, tannins, flavonoids, and polysaccharides in ethanol. This is in agreement with other works (Elakremi et al., 2022b; Özbek et al., 2020; Sun et al., 2015), which indicate high extraction yields using an ethanol:water ratio ranging from 50:50 (v:v).

3.2. Phenolic compounds studied by RP-HPLC

The HPLC phenolic profiles of *Limoniastrum monopetalum* were studied. Figure 2, presents the chromatograms of extracts obtained under optimal conditions for MAE, UAE, and maceration.

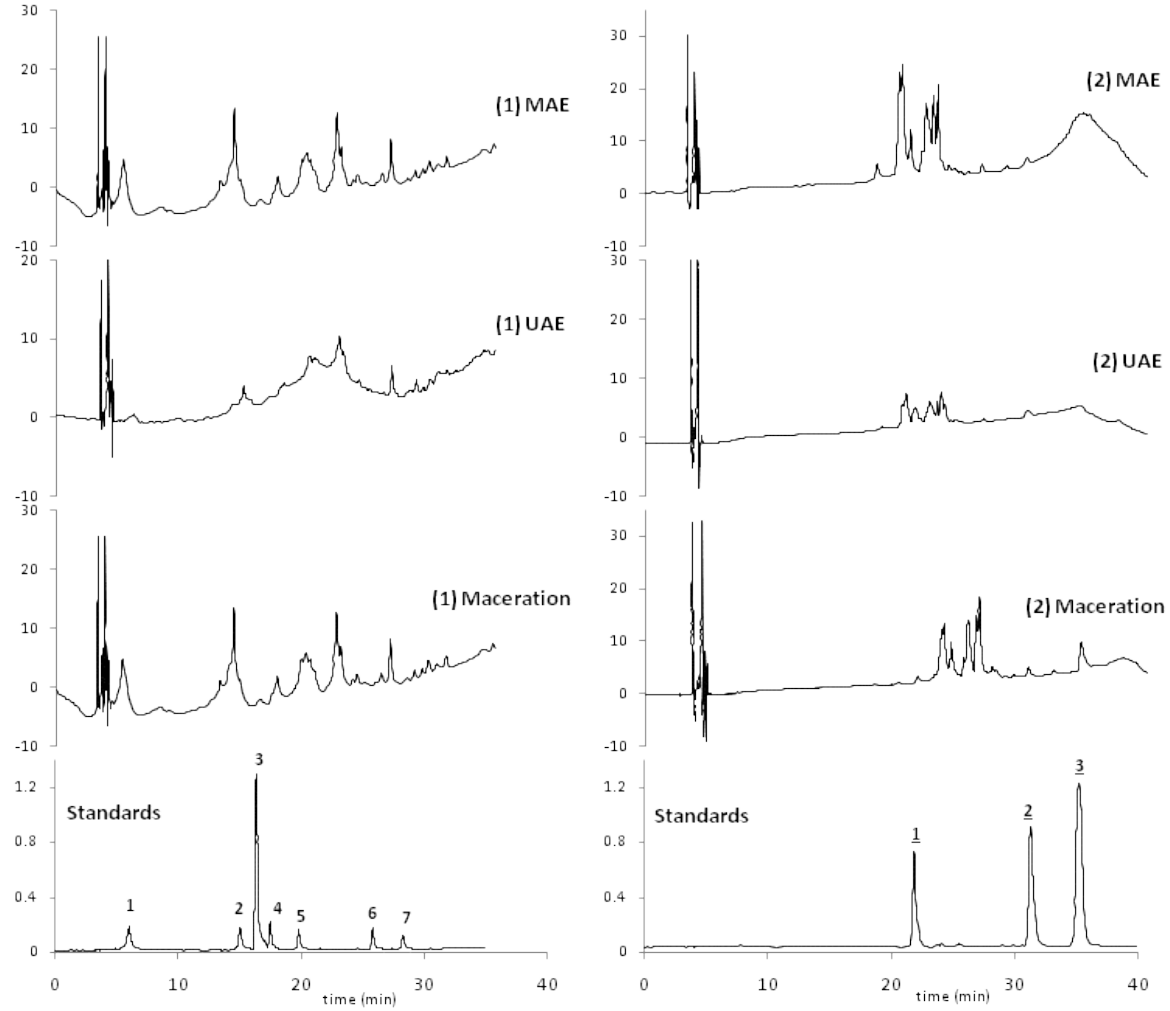


Figure 2. High performance liquid chromatography (HPLC) chromatograms of maceration, MAE and UAE ethanol extracts of *Limoniastrum monopetalum* aerial part: **(1)** phenolic acids (at λ= 280 nm) and **(2)** flavonoids (at λ= 360 nm). **1**-gallic acid; **2**-Catechin; **3**-caffeic acid; **4**-Epicatechin; **5**-Vanillic acid; **6**- p-coumaric acid; **7**-Cinnamic acid; **1**- Rutin; **2**- Quercetin; **3**-Kampferol.

The use of UAE decreases the content of phenolic compounds detected compared to the other extraction methods. The major components identified in the plant extracts were phenolic acids: gallic acid, coumaric acid, and flavonoids: quercetin and rutin. Different phenolic compounds can be delivered by MAE and maceration from plants. So, both methods can be used together to obtain the maximum yield.

4. Conclusion

MAE, UAE, and maceration extraction kinetics of ethanol from the aerial part of the *Limoniastrum monopetalum* system were reported. In terms of modeling, Patricelli's model

gave excellent profiling kinetic behavior and accurate predictions of TPC yield extraction with MAE and UAE. Microwave treatment substantially improved the extractability of polyphenols from *Limoniastrum monopetalum* and could be considered a promising approach to enriching compounds of interest in a shorter time. The maceration and UAE extraction are not effective in extracting polyphenols from *Limoniastrum monopetalum*. Also, the results revealed that the aerial part of *Limoniastrum monopetalum* is a source of phenolic compounds such as gallic acid, catechin, caffeic acid, epicatechin, vanillic acid, p-coumaric acid, cinnamic acid, rutin, quercetin and kampferol which are considered valuable natural antioxidants. RP-HPLC chromatograms revealed that the variability of polyphenol compounds is more observed in maceration and MAE extracts than in UAE extracts.

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Author Contribution Statement
Olfa Khedher: material preparation, conceptualization, investigation, data curation, writing – original draft preparation, methodology. Manel Elakremi: resources, methodology, investigation, formal analysis, conceptualization. Ridha Ben Salem: methodology, supervision, writing–review and editing. Younes Moussaoui: methodology, supervision, visualization, writing–review and editing

Conflict of interest
The authors declare that they have no conflict of interest.

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