

Myrtle Leaves (*Myrtus communis* L) and Olive Leaves (*Olea europaea*): Effect of Drying by Fluidization and Solar Methods on Key Bioactive Compounds Contents

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ABSTRACT

The present study aimed to evaluate the influence of drying by fluidization technology, compared to a traditional solar drying, on phenolic compounds of Moroccan *Myrtus communis* L. and *Olea europaea* L. species. Two main parameters of drying by fluidization (air speed (0.3-1 m/s for myrtle leaves and 1-3 m/s for olive leaves) and air temperature (40-60 °C for the two plants leaves)) were modified and controlled. Results showed that high loss values in total polyphenols (TPP) and flavonoids (TF) were observed at (60 °C; 0.3 m/s) for myrtle leaves and at (60 °C; 1 m/s) for olive leaves. However, these compounds were more stable at (40 °C; 1 m/s) for myrtle leaves and at (40 °C; 3 m/s) for olive leaves. Regarding the solar drying method, the results showed that, for myrtle leaves, high losses were observed in the TPP and TF contents, compared to the fluidization method for all processing parameters. For olive leaves, traditional solar drying gives products with phenolic contents similar to those obtained by fluidization drying, especially at (60 °C; 3 m/s). The findings indicated that employing the fluidization drying method might be a suitable approach for enhancing the conservation of bioactive compounds within myrtle and olive leaves.

Keywords: air drying parameters, flavonoids, fluidization method, myrtle leave, olive leaves, solar drying, total polyphenols.

INTRODUCTION

Myrtle (*Myrtus communis* L.) is a fragrant medicinal plant that is characteristic of the coastal areas in Mediterranean regions like North Africa and Southern Europe. However, it can also be found in South America, Australia, and certain regions of the Himalayas (Alipour et al., 2014; Jabri et al., 2018). It falls under the Myrtaceae family and grows naturally as an evergreen shrub or small tree. The diverse array and quantity of bioactive compounds, including polyphenols, flavonoids, anthocyanins, phenolic acids, lignans, tannins, antioxidants, organic acids, fatty acids, and minerals, are present in

various parts of the myrtle plant, particularly its leaves (Aleksic & Knezevic, 2014; Alipour et al., 2014; Sumbul et al., 2011).

Additionally, *Olea europaea* L., a perennial tree member of the Oleaceae family, covers a total expanse of 10.8 million hectares and has been cultivated across 41 countries, particularly those situated in the Mediterranean region (Cavaca et al., 2020). Among the most significant residues of olive cultivation are the olive leaves, constituting about 10% of the entire weight of the olive harvest and amounting to 25 kg per tree during pruning (Khemakhem et al., 2017). Furthermore, olive leaves are abundant in bioactive constituents, including a substantial quantity of secoiridoids,

hydroxytyrosol, polyphenols, triterpenes, and flavonoids (Kiritsakis et al., 2010). These compounds hold potential for adding value to the by-products and promoting a circular economy (Lorini et al., 2021; Medina et al., 2019).

The existing literature extensively covers the studies concerning myrtle and olive leaves, encompassing their extracts, analysis, health-beneficial attributes, and potential applications (Aleksic & Knezevic, 2014; Alipour et al., 2014; Dias et al., 2021; Saadati et al., 2021; Selim et al., 2022). Therefore, it becomes imperative to preserve the leaves of these plants in an optimal manner to harness their potential post-harvest benefits. Drying stands out as a potent technique used to convert perishable harvested crops into long-lasting products. By reducing the water activity of the product to a level where biochemical changes are impeded, drying effectively achieves this purpose (Chua et al., 2019). However, the drying process introduces modifications in the biochemical composition, mainly attributed to degradation. Certain drying methods could exacerbate the deterioration of essential bioactive constituents. Consequently, identifying an appropriate drying approach becomes crucial in order to ensure the optimal preservation of the bioactive content.

Previous studies have explored the impact of various drying methods on the dehydration of myrtle and olive leaves. For instance (Afaneh et al., 2015), examined the impact of drying temperatures (25 °C and 50 °C) on the oleuropein content extracted from olive leaves. Another investigation by Cör Andrejč et al., (2022) studied the antioxidant activity of olive leaves using three different drying techniques: room temperature drying, drying at 105 °C, and freeze drying. Similarly, Kamaran et al. (2015) delved into the recovery of phenolic compounds from fresh, air-dried, freeze-dried, and oven-dried (at 60 °C and 105 °C) olive leaves. Shifting focus to the drying of myrtle leaves, Saifullah et al. (2019) explored the effects of diverse methods, such as hot air drying, vacuum drying, microwave drying, sun drying, shade drying, and freeze drying, on parameters like total phenolic content, total flavonoids, proanthocyanidins, antioxidant capacity, and phenolic compounds of myrtle leaves.

Nonetheless, to the best of authors' knowledge, only a limited number of investigations have examined how drying conditions influence the phenolic content of both olive and myrtle leaves. Moreover, it appears that there is a scarcity of information

concerning the application of the fluidization drying method to Moroccan myrtle and olive leaves. Consequently, this study aimed to assess the effects of two different drying methods (fluidization drying and sun drying), as well as varying drying parameters (air velocity and temperature), on the comprehensive phenolic and flavonoid contents of Moroccan myrtle and olive leaves.

MATERIALS AND METHODS

Plant material

The plant materials of this study are fresh myrtle leaves (*Myrtus communis* L.) and olive leaves (*Olea europaea*) that were collected in March 2016. The myrtle leaves samples are from the regional forest of Benslimane in Morocco, whereas the olive leaves samples, originated from variety 'Picholine Marocain', are collected from different regions of Morocco. Fresh samples were transported to laboratory and immediately analyzed as well as dried using the processes studied in this work. The dried samples were milled to 0.5 mm using an electronic grinder (Retsch GmbH, Germany) to obtain a homogeneous fine powder and stored for further chemical analyses.

Drying

Drying device

The drying process is conducted using an electric pilot setup for fluidized bed drying (Deltalab-France), which delivers drying air with meticulous control over aerothermal conditions (Figure 1).

Drying protocol

The samples of myrtle leaves and olive leaves were dried by fluidization to constant weight at different air speeds (S_a) and temperatures (T_a), using a range of 40–60 °C (Uribe et al., 2016; Zecchi & Ivantysynova, 2011), for predetermined durations (t_d) during descriptive studies already conducted (Lekrati et al., 2017)

Table 1 shows the operating conditions of drying. Dried leaves were stored for chemical analyses to determine the polyphenols content and the flavonoids content variation due to drying parameter modification. Myrtle and olive leaves samples were dried in the open air away from

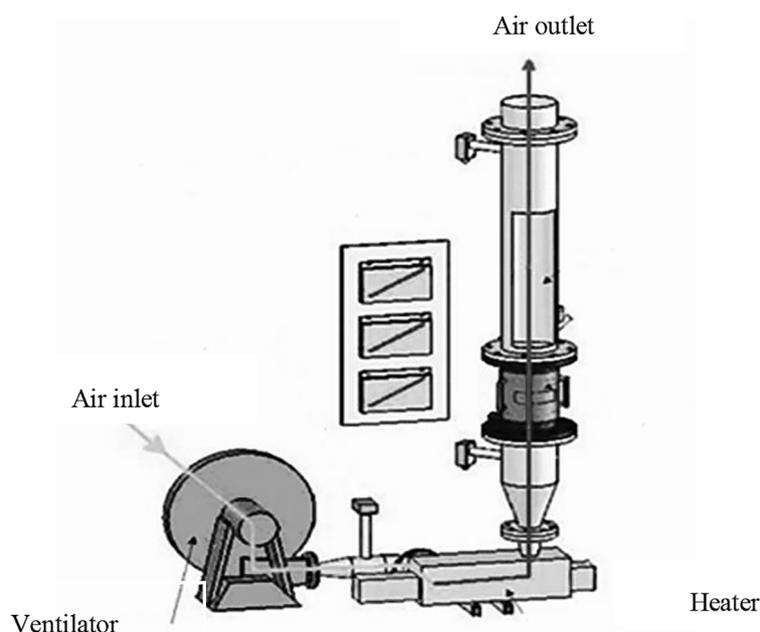


Figure 1. Pilot plant drying by fluidization (Deltalab-France)

Table 1. Operating drying conditions of olive leaves (a) and myrtle leaves (b)

A	Drying conditions			B	Drying conditions		
Treatment	T_a (°C)	S_a (m/s)	t_d (min)	Treatment	T_a (°C)	S_a (m/s)	t_d (min)
1	40	3.0	420	1	40	1.0	220
2	50	3.0	180	2	50	1.0	90
3	60	3.0	90	3	60	1.0	60
4	60	2.0	120	4	60	0.8	180
5	60	1.0	140	5	60	0.3	340

light at an average temperature of 25 °C in order to determine the initial values of polyphenols and flavonoids contents. In addition, a solar drying was carried out under traditional conditions during 2 days at an average temperature of 35 °C.

Determination of phenolic charge

Total polyphenols content (TPC)

- Preparation of phenolic extracts

The extraction of polyphenols was carried out cold by maceration of 2.5 g of dry vegetal powder in 25 mL of polar solvent (pure methanol) for 48 hours and in obscurity. The mixture was filtered and obtained filtrates were submitted to a vacuum evaporation at 50 °C.

- Dosage of total polyphenols

Under alkaline conditions, the polyphenols reduce this reagent to blue-colored tungsten oxide and molybdenum oxide. A sample of 1250 μ L of Folin-Ciocalteu reagent was brought into

contact with 250 μ L of phenolic extract and a sample of 1 mL of sodium carbonate was added. After incubation in the dark for 30 min, the reading of absorbance was done with a wavelength of 765 nm. The calibration range was prepared with gallic acid at variable concentrations of 10, 30, 50, 100, 200 mg/L. Polyphenols contents are expressed in mg of equivalent gallic acid per g of dry weight (mg EGA/g DW).

Total flavonoids content (TFC)

- Preparation of flavonoids extracts

The polyphenols extraction was obtained by plant material maceration (during 48 hours) of 5 g in a methanol/water mixture 80% (Basli et al., 2012). The macerate was filtered through Whatman paper N°1 and then the filtrate was evaporated to dryness. To obtain the flavonoids fraction, the residues were collected in hot water and were separated with n-butanol and ethyl acetate. The two fractions were dried.

- Dosage of total flavonoids

The total flavonoids dosage principle is to form a complex of flavonoids and aluminum chloride (Popova et al., 2004). In the study, 1 mL of AlCl₃ was added to 1 mL of methanolic extract. The absorbance was read at 420 nm during 40 min. The calibration range was prepared with rutin at variable concentrations from 50 to 500 mg/L. Flavonoids contents are expressed in rutin equivalent mg per g (mg eq RU/g DM).

Statistical treatment

Statistical analysis was carried out using Microsoft Excel version 2016 (XLSTAT) and the comparison of results of each treatment was made by one-way ANOVA test at the significance level 5%. The averages were expressed as mean ± standard deviation of three replications per sample.

RESULTS

Effect of drying air temperature and speed on myrtle and olive leaves drying kinetics

Olive leaves drying

The olive leaves drying curves describe the variation of water content as function of time (Figure 2). It was noted that the drying at 3 m/s was carried out more quickly at 60 °C than 50 °C and 40 °C, and the drying speed at 60 °C became more important at 3 m/s than 1m/s. It concludes that the olive leaves drying speed increases as speed and temperature air increase.

Myrtle leaves drying

Figure 3 presents the water content variation of myrtle leaves as function of time where it can be noticed that for the same air speed 1m/s, the

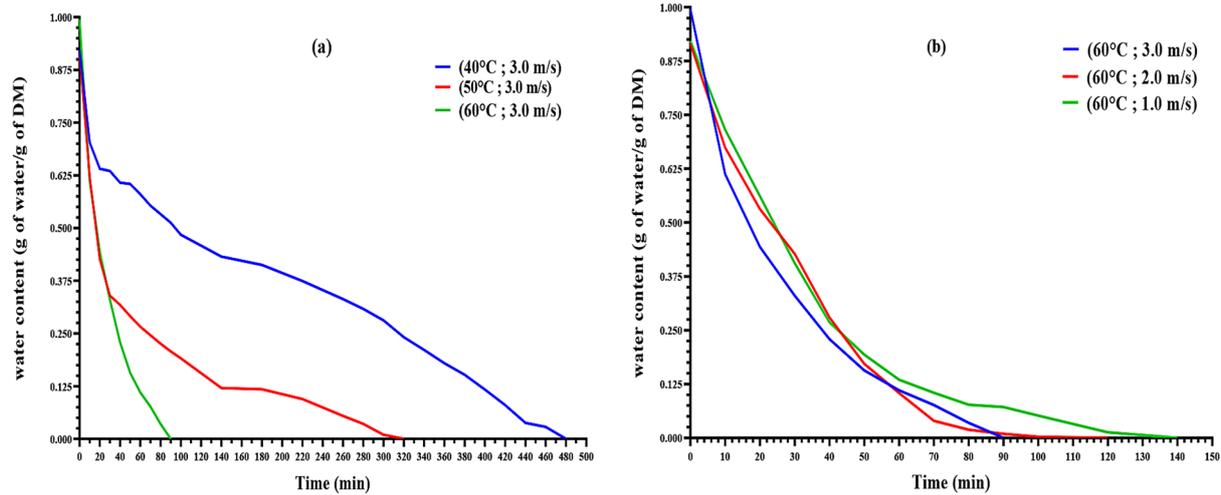


Figure 2. Effect of drying air temperature and speed on olive leaves drying kinetic: (a) at a variable temperature and fixed speed (3 m/s); (b) at a variable speed and fixed Temperature (60 °C)

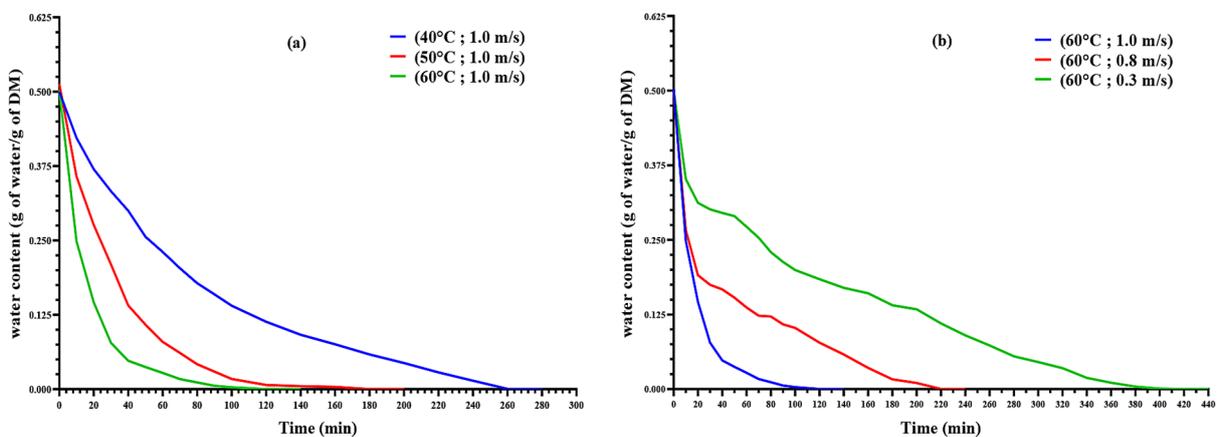


Figure 3. Effect of drying air temperature and speed on myrtle leaves drying kinetic: (a) at a variable temperature and fixed speed (1 m/s); (b) at a variable speed and fixed Temperature (60 °C)

rate of drying accelerates with the air temperature rising. The drying time at 60 °C decreases when the air speed changes from 1 m/s to 0.3 m/s. It concludes that the drying is done more quickly at higher air speed. Hence, the drying time is reduced when air temperature and air speed increase for the leaves of both studied plants, which goes hand in hand with the principles of heat transfer and mass transfer.

Influence of air parameters and drying method on TPC

Figure 4 shows that fresh myrtle leaves are less rich in TPC compared to olive leaves with values of phenolic content in fresh myrtle leaves of (136.26 mg EGA/g) that are almost the half of those in fresh olive leaves (230.22 mg EGA/g).

After drying, it can be noticed that TPC is markedly affected by the drying method. For fluidization method, it generally decreases with temperature increase and air speed decrease as shown in Figure 4. Also, solar method decreased highly the TPC for both leaves.

Moreover, the results indicate that the fluidization method is more preservative of the TPC content compared to the solar methods for myrtle

leaves. However, olive leaves seem to generally have high amount of TPC losses for both drying methods. The percentages of TPC losses for both myrtle and olive leaves were discussed through Table 2.

According to Table 2 results, the TPC of olive leaves are more sensitive to air temperature than to its speed for fluidization method. In fact, it was noted that these parameters have the same effect on polyphenols conservation for both olive leaves and myrtle leaves: the lower polyphenols loss is registered at the higher speed value (3 m/s) and the smaller temperature value (40 °C).

It is noticeable that the losses represent more than the half of the initial phenolic content of olive leaves (56.46%) at 40 °C; 3 m/s, and they are insignificant for myrtle leaves (4.69%) at 40 °C; 1 m/s: TPC in olive leaves went from 230.22 mg EGA/g to 100.24 mg EGA/g and that of myrtle leaves varied from 136.26 mg EGA/g to 129.87 mg EGA/g.

Moreover, the traditional solar drying method seems to be generally less effective compared to the fluidization method by causing high TPC losses for myrtles leaves. However, the solar drying impact on TPC losses for the olive leaves seems to be generally the same for the fluidization method

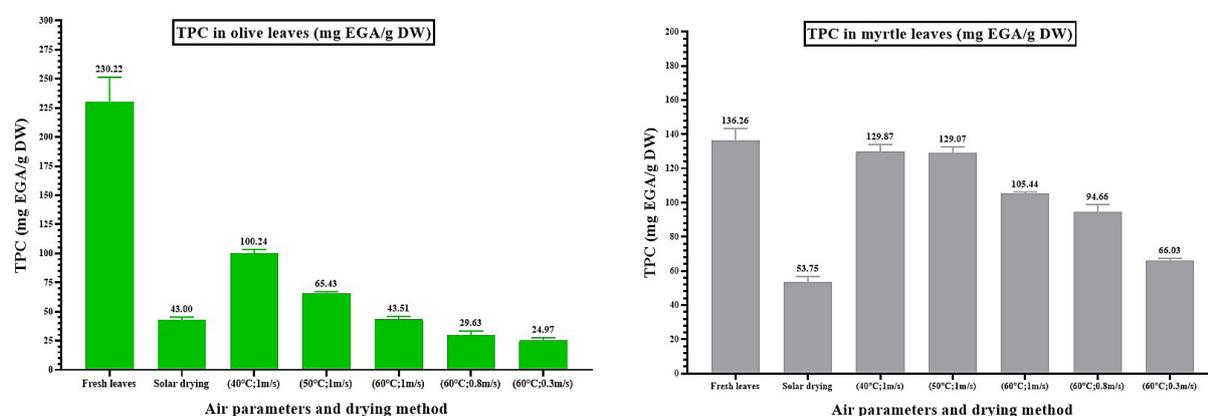


Figure 4. Influence of drying treatments on TPC in olive and myrtle leaves

Table 2. TPC losses of olive and myrtle leaves according to drying conditions

Olive leaves			Myrtle leaves		
Drying method	Treatment conditions	Loss in TPC (%)	Drying method	Treatment conditions	Loss in TPC (%)
Traditional	Solar drying	81.32	Traditional	Solar drying	60.56
Fluidization	(40 °C; 3 m/s)	56.46	Fluidization	(40 °C; 1 m/s)	4.69
	(50 °C; 3 m/s)	71.58		(50 °C; 1 m/s)	5.28
	(60 °C; 3 m/s)	81.10		(60 °C; 1 m/s)	22.62
	(60 °C; 2 m/s)	87.13		(60 °C; 0.8 m/s)	30.53
	(60 °C; 1 m/s)	89.16		(60 °C; 0.3 m/s)	51.54

for all studies speed drying at 60 °C with loss values of (81.32%) for solar drying and (89.16%; 87.13%; 81.10%) for fluidization drying at (60 °C; 1 m/s); (60 °C; 2 m/s); (60 °C; 3 m/s).

Furthermore, the phenolic content losses for olive leaves were generally higher than the phenolic content losses for myrtle leaves. This could be due to the treatment drying duration that was longer (480 min) for olive leaves compared to myrtle leaves (260 min).

Influence of air parameters and drying method on TFC

Figure 5 shows that fresh myrtle leaves are less rich in TFC (30.97 mg ERU/g) compared to olive leaves (118.11 mg ERU/g).

Results showed that the effects of the method and the parameters of drying on the TFC is significant: TFC mostly decreases with temperature increase and air speed decrease with fluidization method. Also, the drying using the solar method decreased highly the TFC for both leaves. The percentages of TFC losses were analyzed through Table 3.

According to Table 3, percentages of TFC losses with fluidization method show a similar value to

solar drying losses for both leaves at high temperature (60 °C) for all speed drying used with values of:

- Olive leaves : 53.46% for solar drying and (54.73%; 86.64%; 86.71%) for (3 m/s; 2 m/s; 1 m/s) at 60 °C with fluidization, respectively.
- Myrtle leaves : 58.99% for solar drying and (56.32%; 52.80%; 57.45%) for (3 m/s; 2 m/s; 1 m/s) at 60 °C with fluidization, respectively.

Moreover, fluidization method seems to have less degradation effect on TFC for olive using (40 °C; 3 m/s); (50 °C; 3 m/s) and for myrtles leaves using (40 °C; 1 m/s); (50 °C; 1 m/s) compared to solar drying losses.

It can be concluded that fluidization method gives us a better TFC losses when using a (40 °C; 3 m/s) condition for olive (26.02%) and myrtle leaves (37.23%) compared to solar drying (53.46%; 58.99%, respectively).

DISCUSSION

Following these results, heat effect on polyphenols and flavonoids can be explained by phenol structure damage at high temperatures. Many studies showed that total phenols are highly

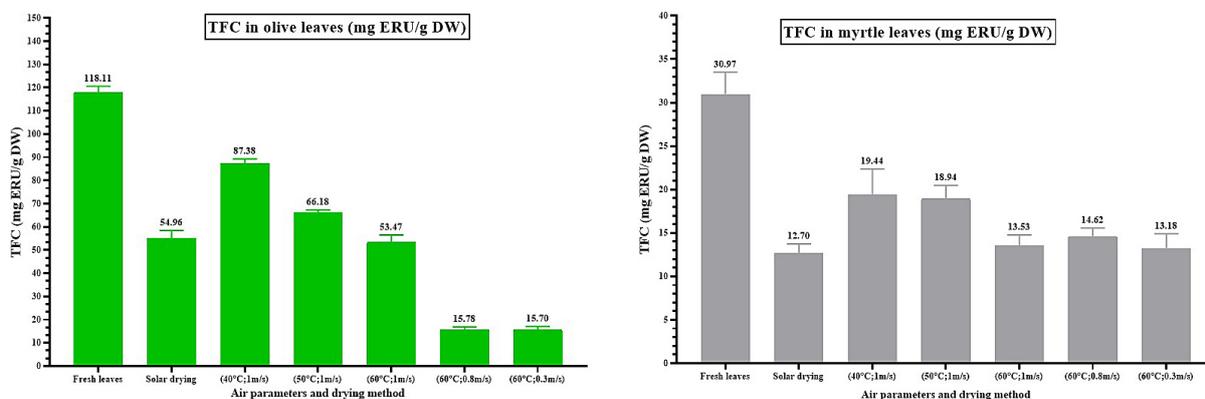


Figure 5. Influence of drying treatments on TFC in olive and myrtle leaves

Table 3. TFC losses of olive and myrtle leaves according to drying conditions

Olive leaves			Myrtle leaves		
Drying method	Treatment conditions	Loss in TFC (%)	Drying method	Treatment conditions	Loss in TFC (%)
Traditional	Solar drying	53.46	Traditional	Solar drying	58.99
	(40 °C; 3 m/s)	26.02		(40 °C; 1 m/s)	37.23
Fluidization	(50 °C; 3 m/s)	43.97	Fluidization	(50 °C; 1 m/s)	38.85
	(60 °C; 3 m/s)	54.73		(60 °C; 1 m/s)	56.32
	(60 °C; 2 m/s)	86.64		(60 °C; 0.8 m/s)	52.80
	(60 °C; 1 m/s)	86.71		(60 °C; 0.3 m/s)	57.45

affected by drying, and this effect is more pronounced when the drying temperature is increased (Orphanides et al., 2013; Sagrin & Chong, 2013). However, air speed influences TPP conservation indirectly through the acceleration of exchanges between leaves and dry air and thus reducing drying time. Therefore, a treatment by fluidization at a higher air speed allows reducing the residence time in the dryer where the leaves are subject to a heat treatment, which translates generally into a lower loss of polyphenols. Thus, losses of TPP and specifically of flavonoids depend on temperature-time couple.

As for solar drying, it gives the maximum of TPC and TFC losses against the fluidization treatments of myrtle leaves. However, this traditional mode of drying leads to a level of conservation comparable to that provided by the fluidization technique at 60 °C-3 m/s for olive leaves. Therefore, the solar drying presents a good performance comparing it with the treatments carried out at low speed and high temperature values for olive leaves. This was found also for spearmint (Orphanides et al., 2013) for which the solar drying presents a medium level of phenolic compounds conservation. Many studies show that the phenolic content is affected by the drying method (Lachowicz et al., 2019; Liu et al., 2020; Tan et al., 2021; Yap et al., 2020). This was illustrated also by Noutfia et al., (Noutfia et al., 2018; Noutfia et al., 2021) in his studies about fig drying, where the solar method has shown a good quality preservation.

CONCLUSIONS

Myrtle and olive leaves are recognized as abundant source of phytochemicals, particularly bioactive compounds. On the basis of this study, the fluidization, and solar drying methods, in general, had led to variable effects on the total phenolic content and flavonoids content in myrtle and olive leaves.

Moreover, it can be inferred that drying by fluidization at (40 °C; 3 m/s) for Moroccan Myrtle and (40 °C; 1 m/s) for Moroccan olive leaves could be considered as the most appropriate drying conditions for the preservation of high amount of total phenolic content and flavonoids contents compared to sun drying.

Optimization of various fluidization drying conditions should be carried out to ensure a high bioactive compounds preservation for olive and myrtle leaves.

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